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An American National Standard
Technical Association of Pulp
and Paper Industry
Test Method T 401 om-88

Standard Test Method for Fiber Analysis of Paper and Paperboard[1]

This standard is issued under the fixed designation D 1030; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the identification of the kinds of fibers present in a sample of paper and their quantitative estimation.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product

D 586 Test Method for Ash in Pulp, Paper, and Paper Products

D 1193 Specification for Reagent Water

2.2 *TAPPI Standards:* [3]

T 8 Identification of Wood and Fibers from Conifers

T 10 Species Identification of Nonwoody Vegetable Fibers

3. Summary of Test Method

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

4. Significance and Use

4.1 Many types of paper, particularly bonds, ledgers, index, and book papers are bought on the basis of fiber composition. This test method is used to evaluate the fibers in the paper and to ensure the purchaser that the composition and types of fibers are in accordance with the specifications. It will also show whether the composition is free of inferior fibers which the specifications particularly prohibit. It is also significant as to the structure and quality of the paper. In order that the examination may be interpreted into practical significance, it is important that the analyst should be experienced in the field of pulp and paper microscopy.

4.2 For accurate results, considerable training and experience are necessary. The analyst should make frequent use of standard samples of known composition or of authentic fiber samples and should become thoroughly familiar with the appearance of the different fibers and their behavior when treated with the various stains.

4.3 Morphological characteristics identify special fibers such as straw, flax, esparto, and certain types of wood, 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 helpful and, in some cases, essential for their identification. Some information on this subject is given in the Appendixes.

5. Apparatus and Materials

5.1 *Microscope*, compound, preferably of the binocular type, equipped with a mechanical stage and Abbe condenser. A magnification of approximately 100 diameters is recommended for observation of fiber colors, although a higher magnification may be desirable for studying morphological characteristics. If an apochromatic objective is used, it is desirable to have a compensating eye piece and an achromatic condenser. The eyepiece shall be provided with a cross hair, pointer, or dot for counting the fibers passing under it. Such an eyepiece can be

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[1] This test method is under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and is the direct responsibility of Subcommittee D06.92 on Standard Documents Relating to Paper and Paper Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

supplied by the manufacturers, or it may be prepared by the technician, positioning the point in the eyepiece so as to obtain its image in focus.

5.2 *Slides and Cover Glasses*—Standard slides 25 by 74-mm (1 by 3-in.) of clear, colorless glass, and No. 2 cover glasses (25-mm square).

5.3 *Dropper*—A glass tube approximately 100 mm (4 in.) long and 8 mm (5⁄16 in.) inside diameter, with one end carefully smoothed, but not constricted, and the other end fitted with a rubber bulb. The tube is graduated to deliver 0.5 mL.

5.4 *Warm Plate*—A plate with a plane, level top made of solid metal having black mat finish, and provided with a control to keep the temperature of the surface between 50 and 60°C.

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

5.6 *Glass-Marking Equipment*—Either a glass-marking pencil or an aluminum stearate solution (see Appendix X6) for marking lines on the slide.

5.7 *Light Source*—A 15-W "daylight" fluorescent tube or equivalent daylight source.

5.8 *Camel's-Hair Brush*, small.

5.9 *Miscellaneous*—50 or 100-mL beaker; test tube; glass beads, 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 in separating plies of cylinder board.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined in Specification D 1193.

6.3 *Graff "C" Stain*, suggested for general analysis, but when desirable, other stains, listed below, should be used for specific purposes or to confirm results obtained with the "C" stain.

6.4 *Herzberg Stain*, especially useful to differentiate between rag, groundwood, and chemical wood pulps.

6.5 *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.

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

6.7 *Green and Yorston Stain*, very useful for the detection of unbleached sulfite fibers.

6.8 *Du Pont Stains*, customarily used in sequence, may be very useful in fiber analysis.

6.9 Directions for preparing these stains and the directions for preparing and using other stains, are given in Annex A1. Directions for using spot stains for groundwood are given in Appendix X5.

7. Test Specimens

7.1 A single composite test specimen of approximately 0.2 g shall be selected so as to be representative of all the test units of the sample obtained in accordance with Practice D 585.

8. Disintegration of Specimens of Ordinary Papers

8.1 Handling the specimen with gloves, tear it into small pieces and place in a small beaker. Handling the specimen with gloves is required, as metalic salts on the skin may contaminate the specimen and give false reaction with stains. Cover with distilled water and bring to a boil on a hot plate. Decant the water, 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 has been thoroughly absorbed by the paper. Add a little more water, and shake well and again add some water and shake. Continue in this way until the paper has been thoroughly disintegrated. After 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 %. If the specimen is difficult to disintegrate, glass beads may be used in the test tube, but if this is done, it should be so stated in the report. Glass beads should not be used if the fibers are to be examined for degree of beating.

8.2 If the paper cannot be disintegrated by shaking in water, return the specimen to the beaker and cover it with 1 % sodium hydroxide (NaOH) solution, bring to a boil, decant the alkaline solution, and wash twice with water. Cover the specimen with 0.05 *N* hydrochloric acid (HCl), let stand several minutes, decant the acid, and wash several times with water. Roll into pellets and proceed as in 8.1.

NOTE 1—If it is known that the specimen will not disintegrate by the method described in 8.1, the analyst may start with that given in 8.2. Roofing papers and papers containing wool fibers, however, must not be so treated, because the alkali may dissolve the wool.

8.3 If the specimen cannot be disintegrated by either of the above methods, use one of the special methods given below.

9. Disintegration of Specimens of Specially Treated Papers

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

9.1.1 *Tar- and Asphalt-Treated Papers*:

9.1.1.1 *Method A*—Place the test specimen in a dish, cover with kerosine, and digest on a steam bath for 1 h. After this

[4] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

remove the specimen and press it between blotters, treat it again on the steam bath, and again press between blotters. Then extract with cold benzene 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).[5]

9.1.1.2 *Method B*—Fill several convenient containers (250-mL beakers) about one half full with carbon tetrachloride (CCl_4) (Note 2). Cut the test 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 scrim or mesh, which can then be treated separately if desired. Continue moving the specimen into fresh CCl_4 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.

9.1.1.3 *Method C*—Place the specimen in a Soxhlet or similar extractor and extract with chloroform, carbon tetrachloride, dioxane, trichloroethylene or similar solvent.

9.1.2 *Rubber-Treated Papers*—Extract the paper for 6 h in a Soxhlet extractor with cumene (isopropyl benzene), 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 cumene will take out about 98 % of the rubber (2).

9.1.3 *Parchment Papers*:

9.1.3.1 *Method A*—To 25 mL of water, add 25 mL of concentrated H_2SO_4 and cool to 50 to 60°C. Place the paper in the acid, and when the paper begins to disintegrate, stir quickly and empty into a 1-L beaker two thirds full of water (4).

9.1.3.2 *Method B*—Soak the specimen for about 5 min in concentrated HCl, wash, boil up 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 suitable wetting agent, and disintegrate (4).

9.1.4 *Pyroxylin-Treated Papers*—Extract or remove the pyroxylin with ethyl acetate, or amyl acetate.

9.1.5 *Wet-Strength Papers*:

9.1.5.1 *Method A*—Tear the paper into small pieces and place in a beaker; cover with 5 % aluminum sulfate solution and boil from 5 to 20 min, depending on the amount of wet strength present. Decant the alum solution, wash, and proceed as in 8.1.

9.1.5.2 *Method B*—When an estimation of the degree of beating of the fibers is not required, the test specimen may be disintegrated in water in a high-speed mixer.[6]

9.1.5.3 Samples containing alkaline-cured resins may be disintegrated at a pH of 10 and a temperature of 38°C. As little of 0.1 % sodium hypochlorite on a fiber weight basis may be effective in accelerating disintegration for some samples. Information on papers treated with PEl (also considered to be an alkaline curing resin) indicates that disintegration is most satisfactory under acid conditions.

9.1.6 *Highly Colored Papers*—If the paper is highly colored, remove the dye by one of the following methods, and then disintegrate by the usual procedure. The treatment selected depends on the characteristics of the dyes.

9.1.6.1 *By Solution*—Use alcohol, NH_4OH, acetic acid, or HCl.

9.1.6.2 *By Oxidation*—Use HNO_3 or bleach liquor. (sodium hypochlorite solution)

9.1.6.3 *By Reduction*—Use hydrosulphite, stannous chloride, or HCl and zinc (1).

10. Preparation of Slides

10.1 It is desirable to keep the slides and cover glasses in 50 % alcohol. After a slide has been dried and polished, draw lines 1 in. (25.4 mm) 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 a 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 square. Leave the slides on the warm plate until completely dry.

NOTE 2—A few drops of an acrylamide-type deflocculating agent[7] added to the fiber suspension is very effective in many cases.

11. Staining

11.1 To use the Graff "C" stain, Herzberg stain, Selleger's stain, or Wilson's stain, apply 3 drops of the stain to the fiber field on the slide, then place a cover glass over it in such a way as to avoid air bubbles. Allow the slide to stand 1 or 2 min, then drain off the surplus stain, preferably by tilting the long edge of the slide into contact with a blotter.

NOTE 3—Take care not to touch the unstained fibers on the slide with the fingers, since the fingers usually have various metallic salts on them which will be absorbed and later may give rise to puzzling stain reactions.

11.2 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 should check known samples to become familiar with their appearance.

11.3 *Graff "C" Stain*—When lignin is present, a yellow color is developed with the" C" stain. Groundwood gives a

[5] The boldface numbers in parentheses refer to a list of references at the end of this test method.

[6] A Waring Blendor, or equivalent device, has been found satisfactory for this purpose.

[7] Cytame, available from American Cyanamid Co., Paper Chemicals Div., Stamford CT, or its equivalent, has been found satisfactory.

very vivid yellow with a tendency toward orange. Unbleached jute stains much the same color, but the two fibers can easily be distinguished by their structural appearance. 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 pulp will stain a vivid yellow, but as the degree of cooking increases, it tends toward a greenish yellow. Unbleached sulfate pulp tends toward yellowish brown, while an unbleached alpha pulp is more brown than yellow. The hardwood pulps (Note 4) 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 4—Hardwood pulps are those from dicotyledons or broadleaved trees. Softwood pulps are those from conifers.

11.3.1 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, 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; therefore, hardwood alkaline pulps, even though bleached, show almost no red when stained. Unbleached hardwood alkaline pulps cannot be easily distinguished from the bleached pulps, nor can the hardwood kraft pulp be distinguished from hardwood soda pulp.

11.3.2 Some special fibers lend their own colors to the system. Thus abaca in the bleached state has a tendency towards purplish-gray; bleached jute is a light yellow-green; cereal straw, bamboo, sugar cane bagasse, flax hurds, and esparto tend towards 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 "C" stain has been published (**5**).

11.4 *Herzberg Stain*:

11.4.1 Being an iodine stain, the general color trends discussed under "C" stain will hold also for the Herzberg stain. However, in general, it gives 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. Groundwood 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, except for jute and abaca, their bleached pulps stain blue, as do chemical wood pulps. Bleached jute gives a strong greenish-yellow color. Abaca varies from purple to pink. The raw, unbleached wood pulps will also tend towards greenish-yellow if enough lignin is present.

11.4.2 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, groundwood, and chemical pulps. If the only interest is in the percentage of rag or percentage of groundwood, the counting is much easier with the Herzberg stain than with the "C" stain. Color charts showing the colors obtained with "C" stain and Herzberg stain have been published (**5**).

11.5 *Selleger's Stain*:

11.5.1 The reactions with Selleger's stain follow the general pattern for iodine stains but, in general, give redder colors than either the "C" or the Herzberg stain. Lignin-containing pulps, such as groundwood 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 hardwood give a purple or blue coloration that is easily distinguished from the colors given by other pulps.

11.5.2 Softwood alkaline pulps give a much lighter blue, but 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, but 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.

11.6 *Wilson's Stain*—In an effort to obtain more distinctive colors with less overlapping, the commonly used potassium iodide is replaced in this stain with cadium iodide and the hygroscopic zinc chloride is eliminated (**6**). In general, the colors obtained from the Wilson stain are similar to those of the "C" stain. A list of colors obtained is given in Appendix X7.

11.7 *Alexander's Stain*—This is a modification of the Herzberg stain which is sometimes useful for differentiating bleached sulfite, bleached sulfate, and bleached soda fibers. To use this stain, apply 2 drops of solution *A* and allow to remain for 1 min, after which carefully blot off the excess dye and allow the specimen to dry. Add 3 drops of Solution *B* and allow to remain 1 min; then, thoroughly mix 1 drop of Solution *C* with the solution on the slide. Apply a cover glass in the usual manner. Bleached sulfite stains red, bleached soda pulp stains blue, and bleached sulfate gives a bluish red.

11.8 *Du Pont Stains*—The various stains and their methods of use are described in Annex A1. These stains are intended to provide clear differentiation among the common paper-making fibers in all possible combinations (**7**).

12. Procedure for Qualitative Identification

12.1 For the proper differentiation of the colors in fiber analysis, and also to become accustomed to the colors developed, it is recommended that a daylight fluorescent lamp be used at all times, placed 10 to 12 in. (254 to 305 mm) from the mirror of the microscope (**8**). 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[8] for comparison with the sample.

[8] A catalog listing the pulps available may be obtained from the *TAPPI* Fibrarian. The Institute of Paper Chemistry, Box 1039, Appleton, WI 54912.

13. Quantitative Determination

13.1 *Preferred Method Using Cross Hairs*:

13.1.1 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 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, repeat passes may be made for each type of fiber count.

13.1.2 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 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 to a fiber, they can be recorded as one fiber.

13.2 *Alternative Procedure Using a Pointer*:

NOTE 5—This procedure has been reported to be less accurate than the cross hair method described in 13.2.

13.2.1 With the mechanical stage, move the field so that the pointer is 2 or 3 mm from atop 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 most convenient. Alternatively, if care is taken and the slide is not moved vertically, repeated passes may be made for each type of fiber counted.

13.2.2 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. Ignore very fine fragments, but 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.

13.2.3 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, each 5 mm apart, have been examined. If the slide has been prepared properly, a total fiber count of between 200 and 300 will have been made.

13.2.4 Multiply the total number of each kind of fiber by its respective weight factor (Table 1) to obtain the equivalent weights, and calculate their percentages by weight of the total fiber composition.

13.2.5 Examine both square fields. If the results for the two fields vary for any type of fiber present by more than the amount stated in Section 14, then prepare and examine one or more additional fields and include the results from all the fields in the reported average (**2**).

TABLE 1 Weight Factors

Fibers	Weight 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	1.20
Douglas fir	1.50
Southern pine	1.55
Alpha (northern)	0.70
Alpha (southern)	1.70
Hardwood	
Soda, sulfate, or sulfite (except gum and alpha)	0.60
Gum	1.00
Alpha (northern)	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

14. Calculation

14.1 Many of the weight factors given in Table 1 were determined by Graff (**9**). To a great extent they depend on the size of the elements included in the count; consequently, each analyst should determine his own values for each kind of pulp he is likely to encounter.

14.2 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.

14.3 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, 12**). Weight factors are related directly to the coarseness of the pulp.

15. Report

15.1 Report the proportions of the various fibers found in terms of weight percentages of the total fiber composition to the nearest whole number, 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 the accuracy is ±3 %, the report would read 23 ± 3 %. Report percentages less than 2 % as "traces." In case of dispute include the weight factors used.

16. Precision and Bias

16.1 *Repeatability (Within-Laboratory)*:

16.1.1 The precision depends upon the skill and experience of the operator and on the selection of the proper weight

factors. Provided the weight factors employed are reliable, competent workers may be expected to be able to check the composition of a chemical pulp furnish that is not too complex within the following tolerances:

Given Fiber in Total Furnish, %	Tolerance, ± % of Content
Under 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

16.1.1.1 Current experience indicates that mechanical pulps may show tolerances (± %) that are 1.5 to 2 times those shown below.

16.1.2 It is emphasized that to achieve the precision stated in 16.1, 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 pulp examined, it is possible for experienced analysts to check the composition of a furnish to within half the stated limits.

16.1.3 The data in 16.1.1 were obtained from historical data (**13**); however, it has been confirmed by recent tests in two laboratories.

16.2 *Compatibility (Between-Materials)*—Not applicable.

16.3 *Reproducibility (Between-Laboratories)*—Not known.

16.4 There is considerable variation in the precision to be expected in fiber analysis. The ability to differentiate between colors that are only slightly different is very important so that no matter how well the specimens are taken, slides prepared, and related statistics calculated, erroneous identification and improper separation can greatly influence the results.

17. Keywords

17.1 fiber analysis; groundwood fibers; hardwood fibers; microscopic examination (of paper); paper; paperboard; semi-chemical fibers; softwood fibers

ANNEX

(Mandatory Information)

A1. PREPARATION OF STAINS

A1.1 "C" Stain

A1.1.1 Prepared "C" stain can be purchased[9] or it may be prepared as follows (**5, 14**):

A1.1.1.1 *Solution A*—Prepare an aluminum chloride solution (sp gr 1.15 at 28°C) by dissolving about 40 g of $AlCl_3 \cdot 6H_2O$ in 100 mL of water.

A1.1.1.2 *Solution B*—Prepare a calcium chloride solution (sp gr 1.36 at 28°C) by dissolving about 100 g of $CaCl_2$ in 150 mL of water.

A1.1.1.3 *Solution C*—Prepare a zinc chloride solution (sp gr 1.80 at 28°C) by dissolving 50 g of dry $ZnCl_2$ (fused sticks in sealed bottles, or crystals) in approximately 25 mL of water. Do not use $ZnCl_2$ from a previously opened bottle.

A1.1.1.4 *Solution D*—Prepare an iodide-iodine solution, by dissolving 0.90 g of dry KI and 0.65 g of dry iodine in 50 mL of water. Dissolve the KI and iodine by first thoroughly intermixing and crushing together, then adding the required amount of water drop by drop with constant stirring.

A1.1.2 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 A1.1—The "C" stain is very sensitive to slight differences, and extreme caution must be exercised in its preparation and use. The solutions used for preparing all iodine stains should be of the exact specific gravity specified and should be 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. Fresh stain should be made every 2 or 3 months.

A1.2 Herzberg Stain (1)

A1.2.1 Prepare the following solutions:

A1.2.1.1 *Solution A*—Prepare zinc chloride solution (sp gr 1.80 at 28°C) by dissolving 50 g of dry $ZnCl_2$ (fused sticks in sealed bottles, or crystals) in approximately 25 mL of water.

A1.2.1.2 *Solution B*—Dissolve 0.25 g of iodine and 5.25 g of KI in 12.5 mL of water.

A1.2.2 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 A1.2—For special tests, the Herzberg stain is sometimes modified by adding more $ZnCl_2$ to make it bluer, or more iodine to make it redder. However, modification is not recommended for normal use.

A1.3 Selleger's Stain

A1.3.1 Prepare by either of the following methods:

A1.3.1.1 *Solution A*—Dissolve 100 g of $Ca(NO_3)_2 \cdot 4H_2O$ 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 1 week. The stain is then ready for use.

A1.3.1.2 *Solution B*—Dissolve 0.267 g of KI in 53 mL of water; add 1 g of iodine, and let stand for 2 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

[9] Prepared "C" stain is available from the Institute of Paper Chemistry, Appleton, WI.

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.)

A1.3.2 If the stain does not give the colors desired (Appendix X7), it may be modified by adding more $Ca(NO_3)_2$ 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.

A1.4 Wilson's Stain (6)

A1.4.1 Dissolve 1.5 g of iodine and 70.0 g of CdI_2 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 USP 37% formaldehyde, 140 g of $Ca(NO_3)_2 \cdot 4H_2O$, and 40 g of $CdCl_2 \cdot 2½\ H_2O$.

A1.4.2 Store the finished solution in an amber stock bottle. Titrate a portion of the stain with 0.01 *N* $Na_2S_2O_3 \cdot 5H_2O$ (2.482 g/L), adding starch indicator near the end point. Ten millilitres of stain solution should be equivalent to 12.0 ± 2.0 mL of 0.01 *N* $Na_2S_2O_3$ solution.

A1.4.3 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 min 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.

A1.5 Alexander's Stain

A1.5.1 Prepare the following solutions:

A1.5.1.1 *Solution A*—Dissolve 0.2 g of Congo red dye in 300 mL of water.

A1.5.1.2 *Solution B*—Dissolve 100 g of $Ca(NO_3)_2 \cdot 4H_2O$ in 50 mL of water.

A1.5.1.3 *Solution C*—Herzberg stain, as described in Section A3.2.

A1.5.2 The fibers on the slide are covered with 2 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 3 drops of Solution B and allowed to stand for 1 min; 1 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 min before covering.

A1.6 Kantrowitz-Simmons Stain (Modified Bright Stain) (13)

A1.6.1 Prepare the following solutions:

A1.6.1.1 *Solution A*—Dissolve 2.7 g of $FeCl_3 \cdot 5H_2O$ in 100 mL of water.

A1.6.1.2 *Solution B*—Dissolve 3.29 g of $K_3Fe(CN)_6$ in 100 mL of water.

A1.6.1.3 *Solution C*—Dissolve 0.5 g of benzopurpurin[10] in 100 mL of 50 % ethyl alcohol. Warm the solution until the dye is completely dissolved. (Some of the dye will precipitate on cooling.)

A1.6.2 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.

A1.6.3 This stain may be either applied to fibers on the slide, or 1.5 g of the fibers may be stained in 50 mL of the solution in a beaker. In either case, mix equal parts of Solutions A and B just before using; apply for 1 min at room temperature, thoroughly wash the stain mixture from the fibers, and then stain them for 2 min with Solution C. After staining, thoroughly wash the fibers again before observation.

A1.6.4 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 be red, while a poorly cooked, unbleached pulp will be 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.

A1.7 Lofton-Merritt Stain (15)

A1.7.1 Prepare the following solutions:

A1.7.1.1 *Solution A*—Dissolve 2 g of malachite green in 100 mL of water.

A1.7.1.2 *Solution B*—Dissolve 1 g of basic fuchsin in 100 mL of water.

A1.7.2 As in the case of the Kantrowitz-Simmons stain, the Lofton-Merritt stain may be applied either to the fibers on the slide or to fibers in a beaker. When staining in a beaker, add 1.5 g of fibers to a mixture of 15 mL of Solution A, 20 mL of Solution B, and 0.09 mL of concentrated HCl (sp gr 1.19). After 2 min 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 min 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.

A1.7.3 This stain is affected also by the amount of lignin present. If the pulp is free of lignin, the fibers will be colorless; if the pulp is highly lignified, they 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. Therefore, this stain has some value for their differentiation. However, any special treatment given to the pulp may interfere with the test, and

[10] DuPont Purpurin 4B concentrated, or its equivalent, is satisfactory for this purpose.

hence it should be used only as an indication of the presence of unbleached kraft or unbleached sulfite, and not as a conclusive test.

A1.8 Green-Yorston Stain (16)

A1.8.1 A stain that is very useful for the detection of unbleached sulfite is prepared by dissolving 15 mg of *p,ph* azodimethylaniline in 100 mL of glacial acetic acid. After the solution is complete, add 300 mL of distilled water, slowly, with agitation. Flood the fiber field with the stain, pour off after 2 or 3 min and replace with fresh stain.

A1.8.2 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 and may be used to differentiate these and kraft semichemical pulp.

A1.9 DuPont Stains (2, 7)

A1.9.1 The five stains to be described and their methods of application are claimed to provide a clear differentiation among all the common papermaking fibers in all possible combinations.

A1.9.1.1 *General Stain* may be used to identify groundwood rag and hardwood chemical pulps, and to establish the presence of but not differentiate coniferous wood pulp. Five drops of a stain made of 50 g of $ZnCl_2$ and 15 g of $CaCl_2$ made up to 100 mL with distilled water (*Chloride Stain No. 3*) are added to the slide and spread evenly. After 20 s, add one drop of stain made by carefully mixing 6 g of KI and 1.5 g of crystalline iodine in 100 mL of distilled water (*Modified Herzberg Stain No. 2*), and mix by tilting the slide. After 1 min from the time the iodine was added, drain the slide and add the cover glass.

A1.9.1.2 *V-stain* is used to determine if hardwood and coniferous wood chemical pulps have been bleached. Add 6 drops of stain made by dissolving 5 g of potassium ferricyanide in 50 mL of distilled water and 50 mL of alcohol (*Ferricyanide Stain No. 5*), add 3 drops of stain made by dissolving 5 g of $FeCl_3$ in 100 mL of distilled water (*Ferric Chloride Stain No. 6*) and mix by tilting the slide. After 1 min, wash lightly and blot. Add a few drops of stain made by dissolving 5 g of Du Pont Pontamine Bordeaux B in 100 mL of distilled water (*Bordeaux Stain No. 7*). After 1 min, wash and blot dry. Add 1 small drop of a solution of 50 mL of saturated NaCl solution in 50 mL of glycerin and add the cover glass.

A1.9.1.3 *W-Stain* is used to determine whether unbleached coniferous pulp is sulfite or kraft. Add a few drops of stain made by dissolving 2 g of basic orange dye in 50 mL of distilled water and 50 mL of alcohol (*W-Basic Orange Stain No. 8*). After 30 s, wash and blot. Then add a few drops of stain made by dissolving 0.75 g Du Pont brilliant green crystals in 25.5 mL of alcohol, 11.0 mL of distilled water, and 62.5 mL of the basic orange stain. After 30 s, wash and blot. Finally add 1 small drop of the salt-glycerin solution described earlier and mount the cover glass.

A1.9.1.4 *Y-Iodine Stain* is used to differentiate fully bleached kraft from bleached sulfite. Add a few drops of stain made by mixing 20 mL of distilled water, 40 mL of alcohol, and 40 mL of the W-basic orange stain No. 8 described above. After 30 s, wash and blot. Add a few drops of *Special Y-Iodine Stain*, prepared by mixing 1 mL of alcohol, 2 mL of Chloride Stain No. 3, 3 mL of Herzberg iodine stain (100 mL of distilled water, 2 g of KI, and 2 g of crystalline iodine); and 4 mL of saturated NaCl solution. Blot after 1 min. Add 1 drop of Chloride Stain No. 3 and add the cover glass. *The Special Y-iodine Stain must be prepared fresh.*

A1.9.1.5 *X-Stain* is used to differentiate some high partially bleached kraft pulps from bleached sulfite pulps. Add a few drops of stain made by dissolving 1.5 g Du Pont brilliant green crystals in 70 mL of alcohol and 30 mL of distilled water. Other sources of Color Index No. 42040 may be substituted for du Pont brilliant green crystals. After 30 s, wash and blot. Add a few drops of *Modified Herzberg Stain No. 2.* Blot after 30 s. Finally, add a drop of Chloride Stain No. 3, and mount a cover glass. The X-stain, or a modification of it, has been used to separate hardwood bleached NSSC pulps from bleached kraft pulps. Several drops of the brilliant green stain are added to the slide so that all fibers are thoroughly covered. After 1 min, pour off the stain, wash thoroughly with distilled water and blot carefully several times, using a clean area of the blotting paper each time. Stain with the modified Herzberg stain for 1 min and again blot thoroughly. Add several drops of the Chloride stain, apply the cover glass, and drain off the excess stain. The bleached kraft pulp was stained chiefly green-blue and the NSSC pulp yellow-green or blue-green, but some fibers in each pulp resembled the colors in the other type, which may interfere with a quantitative analysis of a mixture of the two pulps. When Fuchsine SP was substituted for the brilliant green used in the X-stain, similar results were obtained, although the color reactions were different, of course.

A1.10 NCR Stain (17)

A1.10.1 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, with the NSSC pulp staining different shades of green and the kraft pulp giving 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 to 5 min for development.

A1.10.2 *SC Stain* may be used separately for other fiber separations. It must be noted that the recipe for this stain has not been published and it is only available from the formulators.

APPENDIXES

(Nonmandatory Information)

X1. MORPHOLOGICAL CHARACTERISTICS

X1.1 The characteristics of common coniferous pulpwood fibers are discussed in TAPPI Test Method T 8 and in several readily available references (**18–21**). Pulp fibers from broadleaved trees are considered in various references (**18–21**) and those of other vegetable fibers in TAPPI Test Method T 10, as well as references (**19, 20, 22**). These morphological characteristics may be obscured by the action of swelling agents in the stains or modifications during refining.

X1.2 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.

X1.3 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 and 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 (**18–20**).

X1.4 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 which 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 and, 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 (**18–20**).

X1.5 *Groundwood*—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.

X1.5.1 As various 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. 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 jac, 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 is at times impossible. The color differentiation of western sulfite pulp with the "C" stain, and the tendency toward greater fiber width than eastern species may be useful. The identification of 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 TAPPI Test Method T 8 (species identification of Wood and Wood Fibers) and other references consulted (**18–21**).

X1.6 *Jute and Abaca*—Jute and abaca usually constitute the majority, of the "rope fibers" found in paper. 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 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 is a steel blue or gray. Abaca may vary from dark blue to light red (not so deep as for rag), depending on degree of cooking.

X1.7 *Rag Pulp*—Rag pulp consists of cotton and linen fibers. As rags usually undergo considerable treatment, 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, "rag." Pulp produced from cotton linters is also reported as rag. This pulp is composed of a mixture of lint fibers that are similar to rag, and fibers that 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 either are 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 (**23**). Some of these fibers show a decided twisted appearance at the base. The color of linters with Herzberg stain is red, although the red is darker and tends to give a 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 X2 for further information on these fibers.

X1.8 *Esparto, Cereal Straws, Cornstalks, Bamboo and Sugar Cane Bagasse*—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, and 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. 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 annual vessels, and vessel members are found in all. Most characteristics of esparto are small comma-shaped cells known as trichomes; but unless care is exercised and especially if the pulp has been well-washed, they may be overlooked.

X1.9 *Semichemical Pulps*—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; while if the neutral sulfite cook has been used, the tendency is toward the red.

X2. SYNTHETIC FIBERS

X2.1 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. Also, the intentional addition of such fibers to various grades of paper and such specialties as non-woven fabrics makes it desirable that the analyst should be alert for the many kinds of man-made fibers.

X2.2 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 (**24–26**).

X3. WOOL

X3.1 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 (**27**) has suggested a weight factor of 3.1 for a coarse wool.

X4. ALTERNATIVE PROCEDURE FOR QUANTITATIVE DETERMINATION OF GROUNDWOOD

X4.1 The quantitative analysis of groundwood-containing papers may be facilitated by the following procedure (**28**), 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.

X4.2 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. With the relative weights of chemical pulp and counter-weight pulp thus determined and knowing the weight of counter-weight pulp, the weight of chemical pulp in the paper sample can be calculated by proportion. The weight of groundwood in the paper sample is then determined by difference.

X4.3 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, but 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.

X4.4 Measure the moisture content of the cotton pulp and of the paper. Weigh 0.2 g of the paper on the analytical balance and 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 water as described in Section 7. Prepare slides, 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.

X4.5 Weight of chemical pulp = weight of cotton × relative weight of chemical pulp/relative weight of cotton. Then 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.

X4.6 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 Test Method D 586. 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.

X5. SPOT STAINS

X5.1 *Spot Stains for Groundwood*—To detect the presence of groundwood, 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 X5.1—When applying a spot stain to the surface of a colored paper, the dyes used may be sensitive to acids and 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.

X5.1.1 *Phloroglucinol* —**(29)** 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.

X5.1.2 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, however, does not necessarily prove its presence, as 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, so that in the case of strong papers especially, an indication of groundwood should be confirmed microscopically.

X5.1.3 *Aniline Sulfate*—**(30)** 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 it is easy to prepare and is less costly.

X6. PREPARATION OF ALUMINUM STEARATE SOLUTION

X6.1 To 600 mL of water, add 15 g of shavings from a good grade of plain soap and stir until the soap is dissolved completely. To the solution add 10 g of aluminum sulfate, $Al_2(SO_4)_3 \cdot 18H_2O$. A white precipitate of aluminum stearate forms immediately. Stir with a glass rod until the precipitate coagulates into a wax-like mass. With the stirring rod, lift out the precipitated aluminum stearate and place in a desiccator for 48 h. Store in a well-stoppered bottle to be used as needed.

X6.2 To 50 mL of benzene in a glass-stoppered bottle, add 0.7 g to the desiccated aluminum stearate. Shake well each day until completely dissolved. This usually requires about 10 days. The solution is then ready for use.

NOTE X6.1—If after several weeks it should be found that the solution has lost some of its capacity as a water repellent, add a small piece of aluminum stearate to the solution. This will correct the condition within a few hours.

X7. COLOR CHART FOR IODINE STAINS

X7.1 Highly purified pulps (such as alpha) are characteristically kinky in appearance. The word *raw* refers to unbleached pulp, raw or very lightly cooked. *Unbleached* and *bleached* refer to medium and well cooked pulps.

X7.2 *Graff "C" Stain (5)*:

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 dusky 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 and bleached: *Yellowish gray to weak blue and medium gray.*

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.*

X7.3 *Herzberg Stain (5)*:

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.*

X7.4 *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.*

X7.5 *Wilson's Stain*:

A. Groundwood:
1. Unbleached: *Bright yellow.*
2. Bleached: *Greenish yellow.*

B. Softwood pulps:
1. Sulfite:
a) Raw: *Very 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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