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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.