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FEDERAL SPECIFICATION

PAPER; GENERAL SPECIFICATIONS AND METHODS OF TESTING

This specification was approved by the Commissioner, Federal Supply Service, General Services Administration, for the use of all Federal agencies.

1. PURPOSE AND SCOPE

1.1 This specification covers the general requirements and methods of testing that are common to paper, paperboard, and converted paper products covered by Federal specifications. This specification does not include test methods applicable only to a specific product; such test methods are included in the detail specifications. In the case of conflict between the provisions of this specification and those of the detail specification for a particular material or product, the provisions of the latter shall take precedence.

1.2 The detail specifications for the various types of material represent the lowest quality that will be accepted. All detail specifications definitely state the minimum or maximum limit as to stock, strength, folding endurance, or other qualities which will be accepted. Contractors shall adjust their materials and processes so as to insure the delivery of finished paper which will fully comply with the requirements of the specifications.

2. GENERAL REQUIREMENTS

2.1 Curl.—Paper having a curl which cannot be overcome under reasonable working conditions may be rejected.

2.2 Size and trim.—Paper shall be full size when delivered. Coated book, writing, map, chart, lithograph, manifold, bond, parchment, ledger, and index paper shall be trimmed square on four sides. Other flat paper shall be cut square on four sides. A tolerance of one-sixteenth inch in size will be allowed, unless otherwise specified.

2.3 Count.—"Mill count," 500 perfect sheets to the ream, shall be accurate for all grades of paper. Verification of "mill count" will be made by the receiving office and deliveries may be rejected for incorrect count. Shortage will be deducted in case the delivery is accepted.

2.4 Roll winding.—Roll paper shall be tightly wound, at even tension, and shall contain the least possible number of splices. Splices shall be neatly made and be flagged at both ends of the roll with projecting colored markers. Any roll shall not contain more than four splices.

2.5 Basis weight tolerance. — Unless otherwise specified, any variation above or below the basis weight specified shall not exceed 5 percent.

2.6 Thickness tolerance.—Where an average thickness is specified, the following plus or minus tolerances will be allowed: up to .012 inch, tolerance .0005 inch; .012 to

.016 inch, tolerance .0008; above .016 inch, tolerance .001 inch.

2.7 Watermark.—Paper furnished under the specifications for 25-, 50-, and 100-percent bond, and ledger papers shall be watermarked with the seal of the United States, together with the symbol designating the grades, as provided in the detail specifications. The watermark shall be clear and legi , and shall appear on a basis of four times on sheets 16 by 21 inches, once in each quarter. Copies of full-size watermark design for each of these types of papers will be supplied upon request by the Public Printer, Government Printing Office, Washington 25, D. C.

2.7.1 Contractors will be permitted to sell mill sortings and rejected stock bearing the Government watermark. The Government watermark shall not be applied to any paper other than that ordered by a duly authorized officer of the United States Government.

2.8 Definitions.

2.8.1 *Tub-sized.*—The term "tub-sized" in detail specifications shall be construed to mean that the paper has been passed through a bath of sizing material.

2.8.2 *Ruling, writing, and erasing qualities.*—Where the requirement "shall be suitable for ruling and writing on with ink, and shall have good erasing quality" appears in a detail specification, it shall be construed to mean that lines ruled or characters written with ink shall be clear cut; and that after erasure of ruled lines and written or typed characters, the paper shall have retention of good writing and typing quality, and appearance of surface.

2.9 Sampling and inspection.

2.9.1 Unless otherwise specified, purchases shall be inspected and samples for test taken at the point of delivery. If the inspection and tests, whether preliminary or final, are to be made on the premises of the contractor or subcontractor, the contractor shall furnish, without additional charge, all reasonable facilities and assistance for the safe and convenient performance of the inspections and tests. The manufacturer and/or the contractor shall notify the purchasing officer sufficiently in advance of the completion of manufacture of material to permit arrangement for inspection and selection of samples.

2.9.2 The number of samples to be selected from each lot shall be as specified in the detail specification. The number of specimens to be tested in each type of test shall be as specified in the detail specification; if not so specified, at least five specimens shall be tested.

2.10 Results of tests.

2.10.1 Unless otherwise specified, the average of the results for the specimens tested shall be used to determine conformance of materials tested under this specification.

2.10.2 Unless otherwise specified, results that deviate from the mean value of all tests shall be rejected if the deviation of the doubtful value is more than five times the average deviation from the mean obtained by excluding the doubtful value. Additional specimens shall be tested in place of any for which the results are discarded in accordance with these provisions.

2.11 Latent defects.—If latent defects should be discovered after material has been accepted, the contractor shall, if required, replace the defective material without cost to the Government.

3. NOTES

3.1 Payment basis.—Roll paper will be paid for at gross weight, exclusive of returnable cores, unless more than 2½ percent over the ordered basis weight, in which case the weight in excess of 2½ percent will be deducted. Flat paper will be paid for at the ordered basis weight to the nearest 2½ pounds, unless more than 5 percent under weight, in which case payment will be made

for actual scale weight, including wrappers and twine.

3.2 Additional details of testing methods.—For further details of testing methods described herein, the methods of the Technical Association of the Pulp and Paper Industry (T.A.P.P.I.) listed should be consulted. These may be obtained from the Association at 122 E. 42nd Street, New York 17, N. Y.

(Activities outside the Federal Government may obtain copies of Federal Specifications and Standards as outlined under General Information in the Index of Federal Specifications and Standards and at the prices indicated in the Index. The Index, which includes cumulative monthly supplements as issued, is for sale on a subscription basis by the Superintendent of Documents, U. S. Government Printing Office, Washington, 25, D. C.

(Single copies of this specification and other product specifications required by activities outside the Federal Government for bidding purposes are available without charge at the General Services Administration Regional Offices in Boston, New York, Atlanta, Chicago, Kansas City, Mo., Dallas, Denver, San Francisco, Los Angeles, Seattle, and Washington, D. C.

(Federal Government activities may obtain copies of Federal Specifications and Standards and the Index of Federal Specifications and Standards from established distribution points in their agencies.)

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4. NAVY ACTIVITIES INTERESTED

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ADHESION OF PRESSURE-SENSITIVE TAPES

1. Scope

1.1 This method is intended for testing the adhesion of pressure-sensitive paper tapes and cellulose tapes.

2. Test specimen

2.1 The test specimen shall be about 203 millimeters (8 inches) long and not more than 25.4 millimeters (1 inch) wide.

3. Apparatus

3.1 A pendulum-type tensile testing machine as described in method 171.

3.2 A 5″ x 2″ x 1/16″ stainless-steel plate polished to a No. 4 finish (commercial polish ground) as specified in Federal Specification QQ–S–766.

3.3 A 4½-pound rubber-covered roller approximately 3½ inches in diameter by 1¾ inches in width, the surface of which has a durometer hardness value within the range 70–80.

4. Procedure

4.1 Before each test, wash the polished surface of the steel plate thoroughly with carbon tetrachloride, using a clean piece of lintless wiping tissue for each washing. After all traces of the carbon tetrachloride have evaporated, wipe the surface of the plate with a clean dry piece of the tissue. Apply the tape, adhesive side down, to the polished surface of the plate with a 5-inch section extending beyond one end of it. Press the tape down firmly by passing the roller twice over its surface in opposite directions at the rate of approximately 12 inches per minute, using the weight of the roller only. (Pumice, French talc, or a strip of paper may be applied to the adhesive side of the extended section of the tape to prevent adhesion during the rolling.) Immediately double-back the free end of the tape at an angle of 180° and peel 1 inch of the tape off the plate at the folded end. Clamp the adjacent end of the plate into the lower jaw of the pendulum-type tensile machine and the free end of the tape in the upper jaw. The pawls on the pendulum arm shall be taped back in an open position. The tensile tester shall be of such capacity that the test values will be read when the pendulum hangs between 9° and 45° from the vertical. After the first inch is removed from the plate during the test, the minimum tension required to remove the remainder of the tape, except for the final 1 inch, from the plate shall be taken as the adhesion value. The test shall be made with the stressing jaw moving at a speed of 12 inches per minute.

5. Report

5.1 The average adhesion value for not less than five specimens shall be reported in ounces per inch width to one decimal place.

ADHESION OF SEALS AND CLOSURES FOR PACKAGES

1. Scope

1.1 This method is applicable for testing the adhesive used in forming seals, closures, and seams of heavy paper or paperboard packages. It indicates the suitability of the adhesive for the specific material on which it is to be used.

2. Test specimens

2.1 If finished seals are not available, prepare a seal with the adhesive and with the material on which the adhesive is to be used. Condition two pieces of the material, each 12 by 3½ inches, and cement them together with the adhesive with a joint 2 inches wide along the 12-inch sides, applying the adhesive as in actual use. Allow to condition for 24 hours, then cut across the joint into strips 1 inch wide.

2.2 If finished seals or seams are available, cut strips 1-inch wide across them, adjusting the length, whenever possible, so that the material extends about 1½ inches beyond the cemented joint.

3. Procedure

3.1 Grasp each projecting end of the specimen between the thumb and forefinger of each hand, the forefingers resting against the cemented joint, one on each side. Separate the joint by gently pulling the ends apart with a rolling motion from the top downward with the second joints of the second and third fingers successively resting against each other to act as fulcrums. Estimate the percentage of separation of the joint that occurs without fiber separation from the surface of the paper or paperboard.

4. Report

4.1 The average percentage of separation without fiber failure, of the joints of not less than 10 strips shall be reported.

ATMOSPHERIC CONDITIONS

1. Humidity and temperature

1.1 Unless otherwise specified in the detail specification, all physical tests of paper and paper products shall be made on samples conditioned in an atmosphere maintained at 50 ± 2 percent relative humidity and 23° ± 2°C. (73° ± 3.5°F.), and the tests shall be made under these atmospheric conditions.

2. Conditioning

2.1 Suspend each test specimen of the sample so that the conditioning atmosphere will have free access to all surfaces. Means shall be provided for so circulating the air of the conditioning and testing chamber that its humidity and temperature will be uniformly maintained throughout the chamber. The conditioning time shall be sufficient for the moisture content of the specimens to attain equilibrium with the conditioning atmosphere. Determine this by conditioning until there is no significant change in the weight of the specimens (i.e. less than 1 part in 1,000). For papers that are not highly resistant to water vapor, weigh at intervals of not less than 2 hours; for highly resistant papers such as hard, surface-sized bond and ledger papers, at intervals of not less than 12 hours; for papers that have been given special treatment for resistance of water vapor, and for paperboards, at intervals of not less than 24 hours.

3. Determination of humidity and temperture

3.1 Determine the relative humidity of the conditioning atmosphere by means of either (1) a sling psychrometer, or (2) a stationary type of psychrometer having the air circulated over the bulbs of the thermometers mechanically. In both cases the circulation of air around the bulbs shall be at the rate of not less than 3 meters (10 feet) per second and the exposure not less than 60 seconds before the readings are taken. When the sling type is used, make the readings, especially of the wet bulb, as quickly as possible after bringing it to rest.

3.2 The thermometers used shall be accurately calibrated by comparison with certified standard thermometers and any corrections found necessary applied to the readings.

BASIS WEIGHT

1. Definition

1.1 The basis weight of paper and paperboards is the weight in pounds of a given area which varies for different kinds.

2. Test specimens

2.1 The specimens of paper for test shall consist, whenever possible, of not less than 10 sheets, each at least 100 square inches in area. The specimen of paperboard for test shall consist, whenever possible, of not less than 5 sheets, each at least 1 square foot in area.

3. Apparatus

3.1 The balance used shall have a sensitivity of not more than 0.25 percent of the load applied and shall be so graduated that readings of this degree of accuracy can be made. The balance shall have been calibrated not more than 30 days prior to the test, both with increasing and decreasing loads by applying accurate weights.

4. Procedure

4.1 The area of the specimen shall be determined to the nearest 0.25 percent of its total area, which requires a precision of 0.1 percent in measuring the dimensions. The weight of the specimen shall be determined to the nearest 0.25 percent of its total weight. Calculate the basis weight from the measured area of the specimen and its weight.

5. Report

5.1 Report the basis weight of paper in pounds per ream of sheets of stated number and size, and the basis weight of paperboard in pounds per 1,000 square feet. The weight shall be reported to three significant figures. /0

BRIGHTNESS

1. Definition

1.1 Brightness is the reflectance of white and near-white papers to light in the blue and violet portions of the spectrum. Brightness measurements of such papers correlate well with subjective estimates of their relative whiteness.

2. Test specimen

2.1 The test specimen shall consist of a pad of sheets of the size suitable for the apparatus used and the number of sheets being such that when arranged in a pad, the brightness of it is not changed by doubling its thickness. The area of the test specimen used for the measurement shall be free from watermark and any blemish that might affect the measurement.

3. Apparatus

3.1 The apparatus shall be a reflection meter of such design and adjustment that its readings correlate satisfactorily with readings made by the master instrument of the Technical Association of the Pulp and Paper Industry.

4. Procedure

4.1 All measurements shall be made on the felt side of the paper. Place the pad of paper over the sample aperture of the instrument with the machine direction of the paper parallel to the plane determined by the axes of the incident and reflected rays of light. Place a 1-kilogram weight having a plane base upon the pad. Measure and record the brightness reading to a 0.1 unit. Move the lower sheet of paper to the back of the pad and make another brightness determination. Repeat this procedure until six different sheets of the pad have been tested.

5. Report

5.1 The report shall state the average percentage of brightness (based on magnesium oxide = 100) to one decimal place.

BURSTING STRENGTH

1. Scope

1.1 This method is designed to measure the bursting strength of paper and paper products having a bursting strength of not over 200 points, and in the form of sheets not over 0.025 inch thick.

2. Test specimens

2.1 The test specimens shall be at least 63.5 by 63.5 millimeters (2.5 by 2.5 inches).

3. Apparatus

3.1 The testing machine shall have means for firmly clamping the test specimen without slippage during the test, between two annular, plane, unpolished (matte) surfaces, which may have fine, concentric tool marks not over 0.002 inch deep. The upper clamping surface (clamping ring) shall have a circular opening 1.200 ± 0.001 inches in diameter. The circular edges of the openings which are in contact with the specimen and rubber diaphragm during testing shall be rounded off to a radius of not over 1/64 inch to avoid any cutting action. The lower clamping surface (the diaphragm plate) shall be 0.125 inch thick and shall have an opening 1.25 ± 0.01 (1.24 recommended) inches in diameter. The clamping ring shall be connected to the clamping mechanism through a swivel joint to insure an even clamping pressure. During the tests the circular edges of the openings in the two clamping plates shall be substantially concentric, with no overlapping at any point.

3.2 A rubber diaphragm free from mineral loading material and .033 to .035 inch thick, shall be clamped between the lower clamping plate and the rest of the apparatus so that before the diaphragm is distended by pressure underneath it, the center of the upper surface is below the plane of the clamping surface.

3.3 There shall be means for applying controlled, increasing hydraulic pressure to the underside of the diaphragm until the specimen bursts. This pressure shall be generated by a piston forcing a liquid (usually glycerine) into the pressure chamber of the apparatus at the rate of 95 ± 10 millimeters per minute (motor-driven) or at the rate of 120 revolutions per minute (hand-driven).

3.4 The apparatus shall be equipped with a pressure gage of the Bourdon-tube maximum-reading type graduated in pounds per square inch and accurate throughout the entire range of its scale to within a value equal to 1.0 percent of its maximum capacity. This gage shall be such that the individual readings will be not less than 25 percent nor more than 75 percent of the total capacity of the gage. 12

4. Procedure

4.1 Clamp the specimen securely in position, apply the hydrostatic pressure as specified in 3.3 until the specimen ruptures, and record the maximum registered by the pressure gage. An equal number of tests shall be made on each side of the paper.

5. Report

5.1 Report the average of the test results of not less than 10 specimens in points (approximately pounds per square inch) to three significant figures.

CREASING FOR WATER AND WATER-VAPOR RESISTANCE TESTS

1. Scope

1.1 This method is to provide reproducibly creased specimens for tests of papers and other thin sheet materials used for packaging.

2. Test specimens

2.1 Whenever possible, the test specimen shall be 76 by 76 millimeters (3 by 3 inches).

3. Apparatus

3.1 The apparatus shall consist of a smooth, flat base plate having hinged to it at one edge a top plate capable of matching and surfacing the base plate, and weighing 10 pounds.

4. Procedure

4.1 Fold the test specimen without creasing it so that corners of the sheet diagonally opposite coincide. Place the folded specimen on the base plate of the apparatus with the fold towards the hinge. Position the top plate so that its center is directly above the fold, lower it on the specimen, and allow it to remain on the specimen for 30 seconds. At the end of this period, remove the specimen and repeat the procedure to make a crease on the other side of the specimen, and at right angles to the first crease.

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FINISH (SMOOTHNESS)

1. Scope

1.1 This method is for measurement of the finish of printing papers with reference to the printing smoothness of their surfaces.

2. Test specimens

2.1 The test specimens shall be 51 by 51 millimeters (2 by 2 inches), and shall be clean and free from watermark and from blemishes that might affect the test result, such as creases and wrinkles.

3. Apparatus

3.1 The apparatus shall be a Bekk smoothness tester conforming to the following:

3.1.1 A circular plane surface having an effective area of approximately 10 square centimeters, and a small circular aperture in the center.

3.1.2 A means of applying a direct pressure of 1 kilogram per square centimeter to this area and a vacuum of 380 millimeters of mercury.

3.1.3 A means of passing a known volume of air, 10 milliliters, controlled by the size of the air chamber, between the paper surface and the plane surface.

3.1.4 A means of measuring the volume and rate of change in vacuum from 380 millimeters to 360 millimeters in passing the air between the two surfaces.

3.2 The apparatus shall be tested frequently for leakage, and the capillary tube and the pad of the apparatus shall be kept clean.

4. Procedure

4.1 Set the stopcock of the tester to position "p" with gentle strokes of the vacuum pump, pull the mercury column slightly above 380 and turn the stopcock to position "o". Place the side of the specimen to be tested on the polished glass disc, then lay the square of soft gum rubber on the paper and on top of this, center the metal pressure disc. Bring the lever down to a horizontal position so that the leveling screw rests on the bore in the center of the metal disc. With the aid of the spirit level, and by means of the leveling screw, maintain the pressure bar in a level position throughout the test. Turn the stopcock counter-clockwise to position "M" and with the fine regulating vent at the base of the air chamber, by a gentle pulling-turning motion, permit the mercury column to drop to approximately 380. Just before the mercury column reaches 380, close the fine adjustment vent with a firm pushing-turning motion, and at the instant the 380 mark is reached, start the stop watch noting the time required for the mercury column to drop from 380 to 360. This figure represents the time necessary for 10 milliliters of air to pass between the plane surface and that of the test specimen. This is a numerical measure of the smoothness of the sample. Before removing the paper, turn the stopcock clockwise back to the "o" position. If the time required for the 20-millimeter drop is over 300 seconds, turn the stopcock clockwise to the 1/10 position and multiply the observed time, for the 20-millimeter drop, by 10. Test at least five specimens for the wire side and at least five for the felt side of the sample. Use separate specimens for each test since the pressure on the test specimen compresses the fibers in the sample.

5. Report

5.1 The average smoothness in seconds for not less than five tests on each side of the paper shall be reported.

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FOLDING ENDURANCE, SCHOPPER

1. Scope

1.1 This method is for testing the folding endurance of papers not more than 0.01 inch thick.

2. Test specimens

2.1 The test specimens shall be cut accurately in each principal direction of the paper with a width of 15 millimeters (0.59 inch) and in a length of 100 millimeters (4 inches). They shall be free from folds, wrinkles, or blemishes.

3. Apparatus

3.1 The apparatus shall be a Schopper type folding endurance tester conforming to the following:

3.1.1 Two horizontally opposed clamps, approximately 10 centimeters apart, provided with spring tension that varies during the folding cycle as a slotted folding blade, sliding back and forth between creasing rollers, folds the paper. The clamps while in motion are freely suspended between the tension springs, except that they are supported from below by rollers. The folding blade is 0.50 millimeter (0.02 inch) thick, and the edges of the vertical folding slot are cylindrical and extend somewhat above and below the normal position of the test specimen. The four creasing rollers, each approximately 6 millimeters in diameter and 18 millimeters long, are arranged symmetrically about the midposition of the folding slot, and are preferably provided with jewel bearings.

3.1.2 A means of imparting harmonic motion of constant period to the reciprocating blade. A power-driven apparatus is preferable.

3.1.3 A device to register the number of double folds, which stops automatically when the specimen breaks.

4. Procedure

4.1 With the vertical slot of the reciprocating blade in its central position, place the specimen in the slot and fasten the ends firmly and squarely in the clamps with the surface of the specimen lying wholly within one plane. Handle the specimen by the ends and do not touch it with the hands in the region which is to be folded. Then apply the specified tension and fold the specimen at a uniform rate of approximately 120 double folds per minute until it is severed at the crease. Record the number of double folds required to sever the specimen.

5. Report

5.1 Results shall be reported as Schopper folding endurance for each of the principal directions of the paper. The folding endurance reported shall be the average of not less than 10 tests in each direction. Tests run on strips having their length cut in the machine direction shall be designated as those of machine direction, and tests on strips cut at right angles to the machine direction shall be designated as those of cross direction. In reporting test results, all digits after the first two shall be rounded to zero.

NOTE 1.—In detail specifications, if the kind of folding endurance is not stated, it shall be understood to be Schopper.

FOLDING ENDURANCE, M.I.T.

1. Scope

1.1 This method is for testing the folding endurance of paper of any thickness.

2. Test specimens

2.1 All of the provisions given in 2.1 of method 131 shall apply except that test specimens of definite length are not required. They shall be cut of such length as to insure a firm grip in the clamps without buckling.

3. Apparatus

3.1 A loading clamp constrained to move without rotation in a direction perpendicular to the axis of rotation of the folding head specified below and having its clamping surfaces in the plane of this axis. The load is applied through a spring attached to the loading clamp which is easily adjustable to provide any desired tension on the specimen from 0 to 1.5 kilograms. The deflection of the spring when loaded shall not be less than 17 millimeters (0.67 inch) per kilogram.

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3.2 An oscillating folding head supporting two smooth, cylindrical folding surfaces parallel to, and symmetrically placed with respect to, the axis of rotation. The position of the axis of rotation should be approximately in the common tangent plane to the two folding surfaces in the conventional design and midway between them. The folding head is provided with a clamping device back of the axis of rotation and so designed that no clamping pressure is exerted nearer than 0.375 inch to the bending axis. The rotary oscillating movement of the folding clamp shall be such as to fold the paper through an angle of 135° ± 5° to both right and left of the position of zero fold.

3.3 Each of the two folding surfaces has a radius of curvature of 0.38 ± 0.015 millimeters (0.015 ± 0.001 inch) and a length of not less than 19 millimeters (0.75 inch). The distance separating the folding surfaces shall be greater than the uncompressed thickness of the paper being tested but shall not exceed it by more than 0.25 millimeter (0.01 inch).

3.4 A power-driven device for imparting a rotary oscillating motion of constant period to the folding clamp.

3.5 A device for registering the number of double folds required to sever the specimen.

4. Procedure

4.1 Place the oscillating folding head in the position of zero fold. Place on top of the plunger a weight equivalent to the tension desired on the specimen, and clamp the plunger in position when depressed under this load. Then clamp the specimen firmly and squarely in the jaws with the surface of the specimen lying wholly within one plane and not touching the jaw mounting-plate. The distances separating the folding surfaces shall be greater than the uncompressed thickness of the paper being tested, but shall not exceed it by more than 0.25 millimeter (0.01 inch). Handle the specimen by the ends and do not touch it with the hands in the region which is to be folded. Then apply the specified tension to the test strip by releasing the plunger. If the reading of the load indicator has changed, reset it by means of the adjusting screw to agree with the reading obtained when the weight was applied. Whenever possible, a tension of 1 kilogram shall be used, but if this does not afford practical test results, a greater or a lesser tension may be used. Fold the strip at a uniform rate of 175 ± 25 double folds per minute until it is severed at the crease. Record the number of double folds required to sever the specimen.

5. Report

5.1 Results shall be reported as M. I. T. folding endurance for each of the principal directions of the paper. The folding endurance reported shall be the average of not less than 10 tests in each direction. Tests run on strips having their length cut in the machine direction shall be designated as those of machine direction, and tests on strips cut at right angles to the machine direction shall be designated as those of cross direction. In reporting test results, all digits after the first two shall be rounded to zero. If the tension is other than 1 kilogram, the tension used shall be reported.

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MACHINE DIRECTION (GRAIN)

1. Definitions

1.1 The two major directions of paper are termed as follows:

Machine direction.—The direction of paper parallel to its forward movement on the paper machine. This is also termed "grain."

Cross direction.—The direction of the paper at right angles to the machine direction.

2. Test specimens

2.1 Cut the test specimens with sides parallel to the major directions of the original paper sample sheets. Mark the major directions of both the sample sheets and the test specimens so that they can be respectively identified. For the method in 3.1.1, use a piece approximately 50 millimeters (2 inches) square or a circular piece 2 inches in diameter, and for the method in 3.1.2, use two strips approximately 12.5 millimeters (0.5 inch) wide and 152 millimeters (6.0 inches) long, cut at right angles to each other.

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3. Procedure

3.1 A positive result obtained by one of the following methods shall be regarded as a conclusive determination:

3.1.1 Float the specimen on water and note the direction of the curl. The axis of the curl is parallel to the machine direction of the paper. Paper which absorbs water readily should not be exposed to the water for more than a few seconds.

3.1.2 Hold the two strips by the ends in a horizontal position, one over the other, placing first one and then the other on top. The strip cut in the cross direction will bend the more and fall away from the one cut in the machine direction.

3.1.3 Burst the specimen as described in method 112 for determination of bursting strength. The chief line of rupture will be at right angles to the machine direction of the paper.

OIL PENETRATION

1. Scope

1.1 This method is a measure of the receptivity of paper to printing inks having an oil vehicle. It is suitable only for easily permeable papers such as news, book, and mimeograph.

2. Test specimen

2.1 The test specimen shall be 51 by 51 millimeters (2 by 2 inches).

3. Apparatus

3.1 Box having an open front; a ground-glass top containing a 0.75-inch hole for observation of the specimen; a ground-glass partition parallel with the front side to prevent excess heat from affecting the test results; a 15-watt electric bulb placed back of the partition for illumination of the sample; and an adjustable mirror near the bottom of the box and centered on the hole in the top of the box for observation of the specimen.

3.2 Separatory funnel with a tip approximately 0.75 inch in length and of such diameter that 25 drops of distilled water delivered at 21°C. (70°F.) will have a volume of 1 milliliter. The funnel is suspended with the end of the tip approximately 1.75 inches above the test specimen and contains U.S.P. castor oil, the temperature of which is maintained during the test at 25° ± 2°C. (77° ± 3.5°F.).

4. Procedure

4.1 Place the specimen over the hole in the top of the box. Let a drop of the U.S.P. castor oil fall from the funnel upon the specimen and start a stopwatch the instant the drop strikes the specimen. Observe the under side of the specimen and measure the time interval from the instant of contact of the oil with the paper until the spot of oil reaches a uniform and maximum translucency. Covering the spot of oil with a cap having a black interior aids in the determination of the end point.

5. Report

5.1 The report shall state the average time of penetration of the oil in seconds to the nearest 5 seconds, for not less than five tests on each side of the paper.

OPACITY

1. Definition

1.1 The essential principle of the contrast-ratio method of determining the opacity of paper which is here specified, is as follows: The apparent reflectance of translucent paper when combined with a white backing is higher than its apparent reflectance when combined with a black backing because of the greater amount of light reflected from the white backing. The ratio of the lower reflectance to the higher, expressed in percentage, is taken as the opacity of the paper, this being 100 percent for perfectly opaque paper and nearly zero for perfectly transparent paper.

2. Test specimens

2.1 The test specimens shall be of suitable size to fit the holder of the apparatus. They shall be clean and free from folds and wrinkles.

3. Apparatus

3.1 The standard black backing shall have an apparent reflectance of less than 0.5 percent. It shall consist of a permanent diffusing surface of this apparent reflectance in contact with the sample, or of an equivalent backing, such as a cavity lined with black velvet or other material that will lead to an apparent reflectance of less than 0.5 percent.

3.2 The standard white backing shall have an apparent reflectance equal to 91.5 percent. It shall consist of a permanent diffusing surface of this apparent reflectance in contact with the sample, or of an equivalent cavity, such as that provided by a diffusing surface separated from the sample by a cover glass.

3.3 Measurements of apparent reflectance shall be by light from an incandescent lamp or lamps at a color temperature between 2,400 and 2,800 K (common electric bulbs of 100 watts or less, used at their designated voltage, are usually acceptable), and shall be by illumination which is in effect completely diffused. The direction of view shall be not more than 20° from the normal to the surface of the sample, or equivalent conditions of illuminating and viewing shall be used, such as unidirectional illumination at an angle of not more than 20° from the normal to the surface of the sample and observing all reflected light.

3.4 The holder for the specimen shall be so constructed that the sample is held flat to within 1 percent of the diameter of the area used for the measurements.

3.5 Observations shall be by the human eye or by an equivalent means, such as a photoelectric cell with a filter adjusting the spectral sensitivity to that of the human eye.

4. Procedure

4.1 The procedure appropriate to the apparatus shall be used and the results shall be calculated as a ratio expressed in percentage and recorded to three significant figures.

5. Report

5.1 The test results shall be reported as contrast-ratio opacity, calculated as a percentage to the nearest 0.5. The opacity of a sample shall be the average of the results obtained from testing not less than five specimens.

SAMPLING AND PREPARATION OF TEST SAMPLES

1. Scope

1.1 This method is for sampling sheet or roll paper and preparing the samples for testing.

2. Test sample

2.1 The test sample, unless otherwise specified, shall consist when possible of specimens each cut not less than 11 by 11 inches. This allows margin for trimming exactly 10 by 10 inches, which simplifies the calculation of basis weight determination. The specimens shall consist of a sufficient number to complete the tests. They shall be kept smooth and flat and protected from exposure to direct sunlight, contact with liquids, and other harmful influences. Care should be exercised in handling the specimens if optical, surface, acidity, or other physical or chemical characteristics affected by the moisture of the hands are to be determined. In the case of a test for moisture the test sheets shall be placed, immediately after sampling, in a can made airtight by means of a cover and kept in this condition until the moisture test is performed. When an accurate moisture test is important, it is desirable to take a separate sample for this and weigh the sample before opening the container.

3. Procedure

3.1 The specimens comprising the test sample shall be so selected as to be representative of the entire lot of paper. One set of specimens shall be taken from one unit out of every twenty in the shipment, except that the minimum number of sets taken from a shipment shall be 5 and the maximum number 20. The same number of specimens shall be taken from each unit. The units shall be rolls, cases, frames, skids, or bundles.

3.2 In the case of rolls, special care shall be taken to select test sheets that are not damaged. It is good practice, where moisture is not important, to discard the first three layers of the roll to be sure of obtaining a unit sample in good representative condition. When the moisture is to be determined, the sample shall not be taken within ½ inch of the outside of the roll. The specimens shall be cut from sheets taken across the full width of several unharmed layers.

3.3 In the case of sheet-cut paper, specimens shall be cut from at least five consecutive sheets taken from a point or points over ½ inch from the top or bottom of each case, frame, skid, or bundle sampled.

3.4 The specimens shall be trimmed with their edges exactly parallel to the machine and cross directions of the paper.

3.5 A sufficient number of specimens from each unit sample shall then be arranged consecutively in rotation to form a representative test sample. A convenient way to do this is to number consecutively the sheets comprising each set and then select sheets bearing consecutive numbers, one or more from each set in rotation.

4. Resampling

4.1 In case of necessity for resampling a lot of paper, the samples shall be taken as described above, except that the sample sheets shall be taken from units different from those previously sampled.

TEARING RESISTANCE

1. Scope

1.1 This method is adapted for determining the force in grams required to tear a single sheet of paper after the tear has been started.

2. Test specimens

2.1 Specimens for the test shall be cut accurately in each principal direction of the paper about 76 millimeters (3 inches) long by exactly 63 millimeters (2.5 inches) wide, with the slit to be cut 20 millimeters long, leaving exactly 43 millimeters (1.69 inches) between the end of the slit and the edge of the specimen.

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3. Apparatus

3.1 The apparatus shall be an Elmendorf type of tester conforming to the following:

3.1.1 The test apparatus shall have: (1) a stationary clamp and a movable clamp carried on a pendulum, preferably formed by a sector of a wheel or circle, free to swing on a ball or other substantially frictionless bearings; (2) a pointer and pointer stop to record the maximum arc of swing of the sector pendulum; (3) a sector release to hold the pendulum in the raised position during the mounting of the sample, and permitting it to fall through the force of gravity; (4) a pendulum carrying a circumferential graduated scale so as to indicate the force used in tearing the specimen; (5) a knife attachment for initial slitting of the specimen.

3.1.2 With the pendulum in the raised position, the movable clamp shall lie in the same plane as the fixed clamp forming as it were an extension to the fixed clamp. This plane shall be perpendicular to the plane of oscillation of the pendulum. The gripping surface of the jaws in each clamp shall be 25 millimeters (0.984 inch) by 16.5 millimeters (0.650 inch). The clamps shall be separated by a distance of 2.5 millimeters (0.098 inch). The knife attachment shall slit the specimen midway between the clamps at right angles to the upper edge of the clamps. The slit shall extend from the bottom edge of the specimen to a point 4 millimeters (0.157 inch) above the top edge of the clamps leaving a distance of 43 millimeters (1.692 inches) of uncut specimen perpendicular to the long dimension of the specimen. The perpendicular from the line formed by the top edge of the clamps to the axis of suspension shall be 104 millimeters (4.09 inches) and shall make an angle of 27.5° with the plane of the specimen.

4. Procedure

4.1 The specimen shall be placed securely in the clamps. By use of the knife blade a slit shall be cut in the specimen starting at the bottom edge and perpendicular thereto, and ending 43 millimeters (1.692 inches) short of the top edge. The accuracy of the readings obtained with the apparatus depends to a great extent on the exactness of this 43 millimeters of uncut specimen. The sector release shall be pressed, causing the sector to fall, thus moving the pendulum jaw away from the fixed jaw so as to tear the specimen. The force required to tear the specimen in the given direction shall be read from the scale. Readings obtained where the tear deviates more than 10 millimeters (3/8 inch) away from the line of the initial slit shall be rejected. Likewise if the side of the specimen above the movable jaw rubs against the sector as the tear is made, the reading shall be rejected.

4.2 The test shall be made on enough sheets so that when torn together, the scale readings are between 20 and 60, and the number of sheets used shall be recorded. Not less than five tests shall be made in each principal direction of the paper.

5. Report

5.1 Report the results as the average force in grams required to tear a single sheet. Since the scale readings are made 1/16 of the actual values, this is calculated by multiplying the average instrument reading by 16 and dividing by the number of sheets torn at one time. Report the results obtained on strips torn in the machine direction as tearing resistance, machine direction, and on strips torn across the machine direction as tearing resistance, cross direction.

TENSILE BREAKING STRENGTH (DRY) AND ELONGATION

1. Scope

1.1 This method is for the measurement of the dry tensile strength of all weights of paper and paperboard.

2. Test specimens

2.1 The specimens for test shall be strips cut accurately and with the sides parallel. The length shall be not less than 140 millimeters (5.5 inches), and the width shall be 15 millimeters (0.5 inch), except that for heavy papers and paperboards, the width may be 25 millimeters (1 inch) or 51 millimeters (2 inches).

3. Apparatus

3.1 The tensile testing instrument shall conform to the following:

3.1.1 Two clamps whose centers shall be in the same plane parallel with the direction of motion of the stressing clamp and so aligned that they will hold the test specimen wholly in one plane.

3.1.2 A pendulum so attached to one clamp as to balance the load applied to the specimen accurately, with a device to indicate on a graduated scale the breaking load of the test specimen.

3.1.3 A means of moving the stressing clamp at a uniform rate.

4. Procedure

4.1 The ratio of the clearance distance between jaws to the width of the specimen shall be not less than 5:1, nor more than 12:1. The test specimen shall be firmly clamped squarely in the jaws of the clamps, and the stressing jaw then operated at a speed of 30.5 centimeters (12 inches) per minute until the specimen breaks. The breaking load shall be recorded to the nearest 2 percent of the total indicated reading. The tester shall be of such capacity that the tensile strength of the paper tested will be not greater than 90 percent nor less than 10 percent of the capacity of the tester. Not less than 10 strips cut in each principal direction of the paper shall be tested. All the readings obtained when the paper breaks at or in the jaws shall be rejected. Elongation (or stretch) shall be measured at the instant of rupture of the specimen, either by means of a recording device attached to the machine, or by measuring between reference marks on the specimen with a scale. Not less than 10 specimens shall be tested. When elongation is to be measured, care shall be taken to apply no more initial tension to the test strip before clamping it than is required to straighten it.

5. Report

5.1 The average of the test results shall be reported for each of the principal directions of the paper in kilograms per 15 millimeters width, or pounds per inch width, to the nearest 2 percent of the total reading. Test results of strips cut in the machine direction of the paper shall be reported as tensile breaking strength, machine direction, and test results of strips cut across the machine direction shall be reported as tensile breaking strength, cross direction. The average of the elongation test results shall be reported as a percentage of the length of the strips between the clamps to the nearest 1.

TENSILE BREAKING STRENGTH, WET

1. Scope

1.1 This method is for the determination of the tensile breaking strength of paper and paperboard after they have been wetted with water or other liquid.

2. Test specimens

2.1 The specimens for test shall be as specified in method 171.

3. Apparatus

3.1 A tensile tester as specified in method 171.

3.2 A Finch wet-strength attachment for the tensile tester. The device consists of a stirrup about 1.5 inches wide and 3 inches long, made of two metal straps which support horizontally a cylindrical rod, 3/16 inch (5 mm.) in diameter and a little more than 1 inch long. Between the straps is a small vertically movable container for holding water or other liquid. The liquid container locks in its uppermost position, so that the horizontal rod is immersed in the liquid to a depth of at least 0.75 inch. A metal tongue at the bottom of the stirrup permits it to be clamped in the lower jaw of a tensile tester.

4. Procedure

4.1 Immerse the test specimens in the liquid for the length of time stated in the detail specification.

4.2 Use the Finch device for wetting tissue papers, paper towels, and other papers that are difficult to handle when wet. Clamp the device in the lower jaw of the tensile tester. Place the liquid container in its lowest position and nearly fill it with the liquid. Loop the dry test specimen under the dry horizontal rod and clamp the two ends of the specimen in the upper jaw of the tensile tester. Raise the liquid container so that it locks in its uppermost position, thereby immersing the looped end of the specimen to a depth of at least 0.75 inch. At the end of the immersion period, proceed with the test with the liquid container in its uppermost position. The indicated strength is divided by 2 to obtain the wet strength of a single strip.

4.3 To wet materials that can be handled without difficulty when wet, and paperboards, wholly immerse each test strip in a shallow dish of liquid. After the predetermined time of wetting, withdraw the strip, lay it flat and straight on a pad of blotting paper, cover with a sheet of blotting paper, and pass a 500-gram cylinder over the cover, taking about 1 second to complete the rolling.

4.4 The test shall be made in accordance with the procedure given in method 171.

5. Report

5.1 The results shall be reported as specified in method 171.

THICKNESS

1. Scope

1.1 This method shall be used for determining the thickness of a single sheet of paper or paperboard as it would measure when placed between plane surfaces.

2. Test specimen

2.1 Whenever possible the test specimen shall be not less than 51 by 51 millimeters (2 by 2 inches). It shall be free from wrinkles and creases.

3. Apparatus

3.1 *Micrometer.*—The micrometer used shall be in accordance with the following requirements:

3.1.1 It shall have two plane faces, the smaller of which is circular and 0.25 to 0.33 square inch (160 to 215 sq. mm.) in area, corresponding to a diameter of 0.56 to 0.65 inch (14.3 to 16.5 mm.). The faces shall be parallel to within 0.0002 inch (0.005 mm.) and constrained to move apart in an axis perpendicular to themselves.

3.1.2 When the specimen is clamped between the faces, it shall be under a steady pressure of 7.0 to 9.0 pounds per square inch (0.40 to 0.63 kg. per sq. cm.).

3.1.3 The distance between the graduations on the dial shall be such as to permit of estimating the thickness to at least 0.0001 inch.

3.1.4 The micrometer shall be such as to repeat its readings to within 0.0001 inch at zero setting or on a steel gage block.

3.1.5 Measurements made on standard steel thickness gages shall be within the following tolerances of permissible deviation of the reading from the actual thickness of the standard steel gage:

Intervals		Tolerance	
Millimeters	*Inch*	*Millimeters*	*Inch*
0 to 0.25	0 to 0.01	0.0025	0.0001
Over 0.25 to 1.02	0.01 to 0.04	0.0051	0.0002
Over 1.02 to 3.05	0.04 to 0.12	0.0102	0.0004

3.2 *Calibration.*

3.2.1 *Parallelism of the faces.*—A hard steel ball about 1/16 inch in diameter, fixed firmly in a thinner flat piece of metal to serve as a handle, shall be placed at different points on the anvil and the readings noted.

3.2.2 *Accuracy of the readings.*—The instrument shall be set to zero and standard gages shall be placed between the plane faces and the corresponding dial readings observed over the range to be used. For thickness measurements, standard steel gage blocks having an accuracy of 0.00001 inch shall be used.

3.2.3 *Pressure between the faces.*—The force required to just prevent the movement of the plunger foot, from a reading corresponding to about the average paper thickness tested to a lower reading, shall be determined with a suitable balance, and the contact pressure determined with this force. For example where the pressure foot projects through the top of the apparatus, a fine copper wire is attached to it and by means of a coarse balance or a calibrated spring, the force necessary to prevent the closing of the foot is measured.

3.2.3.1 Alternatively, a stirrup may be used, made of a flat metal plate having a hole larger than the diameter of the micrometer foot, covered at the bottom of the plate by a thin metal disk of about the average thickness of the paper to be measured. The stirrup is suspended from a suitable balance.

4. Procedure

4.1 Place the specimen between the jaws of the micrometer and lower the pressure foot gently upon the surface of the paper at least, whenever possible, 0.25 inch (6 mm.) from the edge of the paper, and record the reading on the dial. Each of 10 different sheets shall be tested in not less than two different places.

5. Report

5.1 The thickness shall be reported as an average of the test results in decimals of an inch to the nearest 0.0001 inch (0.002 mm.).

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WATER ABSORPTIVENESS

1. Scope

1.1 This method is applicable to paper having a fairly rapid rate of absorption of water such as paper toweling and other tissue papers.

2. Test specimens

2.1 Each test specimen shall consist of a single sheet approximately 4 inches square.

3. Apparatus

3.1 A 1-milliliter measuring pipette graduated in hundredths of a milliliter, or the Carson microburette. (For information on the latter consult National Bureau of Standards Research Paper RP959.)

3.2 A 4-mesh wire screen.

4. Procedure

4.1 Place a specimen on the screen and fill the pipette or microburette with distilled water having a temperature of 23° ± 2°C. (73° ± 3.5°F.). With the pipette or microburette at an angle of about 30° with the horizontal, and with the tip nearly in contact with the paper, allow a measured amount of the water to flow on the specimen near its center. While the water is flowing, keep the tip in the drop of water until delivery of the water is completed. The amount of water delivered shall be as stated in the detail specification. If the time of absorption is more than about 2 minutes, cover the water on the paper with a watch glass. Measure with a stopwatch the rate of absorption in seconds from the start of flow of water until the drop of water is completely absorbed as indicated by no further reflection of light from it when viewed at an angle. An equal number of tests shall be made on each side of the paper and not less than 10 specimens shall be tested.

5. Report

5.1 The report shall state the average of the test results in seconds.

WATER RESISTANCE

1. Scope

1.1 This method, termed the dry-indicator method, is for testing the time of permeation of water through papers and paperboards.

2. Test specimens

2.1 Whenever possible the test specimens shall be about 64 millimeters by 64 millimeters (2.5 inches by 2.5 inches). They shall be free from folds, wrinkles, or other blemishes not commonly inherent in the material.

3. Apparatus

3.1 A vessel of water of sufficient size for floating several specimens simultaneously, immersed in a water bath in which the temperature of the water in the vessel can be maintained at 23° ± 0.5°C. (73° ±1°F.).

4. Indicator

4.1 The dry indicator shall be made of pure cane sugar, pure soluble starch, and methyl violet, as follows: Grind each ingredient separately until it passes through a No. 100 screen, and completely dry it in a desiccator over $CaCl_2$ before making the mixture. When dry, weigh and mix the following proportion by weight:

Sugar	45
Soluble starch	5
Dye	1

Mix the ingredients by screening repeatedly through a No. 60 screen until the mixture is uniform. Keep the indicator in a desiccator when it is not being used.

5. Procedure

5.1 The indicator reagent shall be sprinkled evenly over approximately a 2-inch-square area (1-inch for 2- by 2-inch specimens) in the center of the sample. Care should be taken to apply this reagent as evenly as possible since bunching or piling of the reagent causes different values. A 2-inch watch glass (1-inch for 2- by 2-inch specimens) is sealed over the indicator powder using a mixture of 50 percent beeswax and 50 percent rosin. The edges shall be turned up or dipped in wax to prevent contact of the water with the adhesive and the specimen floated on tap water maintained at 23° ± 0.5°C. (73° ± 1°F.). The time required for a definite development of color to appear in the indicator shall be noted and recorded as the penetration time. Make at least five tests from each side of the material.

6. Report

6.1 The water resistance shall be the average of all test results reported in minutes or hours.

WATER VAPOR PERMEABILITY

1. Scope

1.1 This method is for testing the water vapor permeability of paper, paperboard, and other sheet materials.

2. Test specimens

2.1 Each test specimen shall be a circle of the material and shall be not less than 3 inches in diameter.

3. Apparatus

3.1 An open-mouthed cup or dish of such size and shape that it can be accommodated readily on the pan of an analytical balance. The opening should be as large as practical, an area of at least 50 square centimeters being preferred. (This is equivalent to a circle about 3 inches in diameter.) The test dish shall be of such design that a wax seal can be made which will be impervious to leakage of water vapor and will define clearly the test area.

3.2 A template for use in defining the test area and effecting the wax seal, consisting of a circular metal disk ⅛ to ¼ inch thick, with the edge bevelled to an angle of about 45°. The diameter of the bottom (smaller) face of the template shall not be greater than the diameter of the effective opening of the cell in contact with the specimen.

3.3 An analytical balance of 200-gram capacity, sensitive to 0.001 gram, with pans large enough to hold the test dishes.

3.4 A testing room or cabinet wherein the circulating air can be maintained at either 50 ± 2 percent relative humidity and 23° ± 2°C. (73° ± 3.5°F.), or at 90 ± 2 percent relative humidity and 38° ± 2°C. (100° ± 1°F.). The room or cabinet shall have suitable grills or racks to support the assembled specimen dishes, with means for circulating the conditioned air continuously over the entire exposed area of the test specimens. In testing at the higher humidity and temperature, the design of the room or cabinet shall be such as to avoid condensation in the vicinity of the test specimens, and if it is necessary to remove the test specimen assemblies from the room or cabinet for weighing, tight covers must be provided for the test dishes.

4. Procedure

4.1 Use as a desiccant, anhydrous calcium chloride in the form of small lumps, free from fines, that will pass a No. 30 screen. Place sufficient desiccant inside the dish to cover the sheet area evenly to a depth of about 15 millimeters.

4.2 The following method of affixing the test specimen to the dish is for paper and for paperboards not over ⅛ inch thick: Cut the test specimen so that its diameter is equal to that of the larger diameter of the template. Place the sheet over the aperture of the dish and center it as closely as possible on the supporting ring or flange. With the tip of the finger apply a thin film of petrolatum to the bevelled edge of the template. Wipe off any petrolatum which may have been deposited on the smaller surface of the template. Center the template, with the smaller surface down, exactly over the specimen and dish opening. Flow molten wax into the annular space surrounding the bevelled edge of the template, using a medicine dropper to dispense the molten wax. Remove the template from the sheet surface as soon as the wax has cooled and solidified. The wax shall consist of a mixture of 60 percent amorphous wax and 40 percent crystalline paraffin wax.

4.3 For paperboards over ⅛ inch thick, the method of preparing the test assembly is modified as follows: To prevent edge leakage, rotate the edge of the test specimen in

molten wax of such fluidity that it does not penetrate into the edge more than 1/16 inch, suspend it inside the dish to a distance of about half the thickness of the specimen, and run molten wax around the edge of the specimen to seal it to the dish.

4.4 Weigh the assembly on the analytical balance to 0.001 gram. Place the dish on the rack inside the testing room or cabinet in an inverted position so that the layer of desiccant is in direct contact and evenly distributed over the inner face of the test sheet and so that free access of the conditioned circulating air is provided on the exposed surface of the sheet.

4.5 The atmosphere of the testing room or cabinet shall be maintained during the testing at either 50 ± 2 percent relative humidity and 23° ± 2°C., or 90 ± 2 percent relative humidity and 38° ± 2°C., as specified in the detail specification.

4.6 Make successive weighings of the assembly at suitable intervals until a constant rate of gain of weight is attained. If the dishes are weighed in an atmosphere different from that of the testing atmosphere, they shall have tightly fitting covers during the weighings. For papers that are relatively pervious, the weighings should be frequent enough to complete the test before drops of liquid agglomerate are formed in the desiccant or caking of the desiccant occurs. These conditions will be indicated by a drift from the constant rate of gain. At least four specimens shall be tested, with an equal number of each side exposed to the higher humidity. Plot the gain in weight against time. The slope of the resulting curve will furnish a measure of the water vapor permeability.

5. Report

5.1 The water vapor permeability shall be reported as an average of all test results to two significant figures as grams per square meter per 24 hours. The calculation shall be made for the period of constant rate of gain.

WIRE AND FELT SIDES

1. Scope

1.1 The wire side of paper is the side that was in contact with the wire of the paper machine; the other side is termed the felt side. It is desirable to have means of distinguishing between the two sides because they generally differ in some physical characteristics. It is not always possible to make the distinction, this being particularly true of coated and other surface-treated papers, certain high-grade papers made from well beaten rag stock, and specialties made with variations in the usual paper-machine practice. When one of the following procedures does not give a definite indication, it is advisable to try one or more of the other procedures.

2. Procedures

2.1 Fold over a sheet of the paper and observe the relative smoothness of the two sides. Often the pattern of diamond-shaped impressions of the machine wire can be seen, thus identifying the wire side. In viewing the paper, it should be held in a horizontal position with the light striking it at an angle of about 45° and with the line of vision of the eye also at an angle of 45° to the surface of the paper. Observation with a microscope is helpful.

2.2 Dip the paper in water or in a weak caustic soda solution, drain off the excess liquid, allow to stand a few minutes, and again examine the two sides. This treatment tends to restore to paper the texture it had on the machine wire. View as described in 2.1.

2.3 Hold a sheet of paper in one hand in such a way that the grain (machine) direction is parallel to the line of vision and the sheet surface is approximately horizontal. Holding the sheet in this position with one hand, pull upward with the other to start a tear in the sheet so that the line of tear follows the grain of the paper. As the tear is being made, gradually guide it so that it moves in the cross direction and toward the outer edge of the sheet, producing a tear line following a curved path. Turn the sheet over so that the opposite side is uppermost and make a similar tear in the sheet. Observe the feathering caused by the splitting of the sheet at the edge of the two tears which have been made. One of these tears will show a distinct feather edge as compared to the other, especially in the curved portion where the line of tear departs from the grain or machine direction and moves toward the cross direction. The tear with the more feathered edge is always made when the wire side of the sheet faces upwards.

2.4 Cut specimens approximately 1 inch wide and 2 inches long, with the long dimension at right angles to the machine direction of the paper. Place the specimens in a drying oven at approximately 100°C. or in a desiccator over a suitable drying agent, such as anhydrous calcium chloride. It is essential that both sides be exposed alike to the drying air, and they must be suspended or otherwise held so that they can curl without restraint. The direction of curl under these conditions will furnish a reliable means of distinguishing between the two sides of most papers. On drying, any pronounced curl of the specimens will be toward the wire side with the axis of the curl parallel to the machine direction of the paper.

ACIDITY, pH

1. Definition

1.1 In this method the pH value of a paper refers to its acidity or alkalinity in terms of hydrogen ion concentration of the unfiltered aqueous extract of the cut or ground paper.

2. Test specimen

2.1 The test specimen shall consist of 1 gram of air-dry paper. Ordinary papers such as bond and writing may be tested in the form of small cuttings. Paper more than 0.012 inch thick or very dense paper shall be reduced to a fluffy form by a disintegrator.

3. Apparatus

3.1 The special apparatus required for this test is a circuit including a glass electrode and a saturated KCl-calomel half-cell capable of precision to within 0.1 pH. The complete apparatus is commercially available. The apparatus used shall be standardized frequently against 0.05 *M* potassium acid phthalate solution which has a pH of 4.0 over the entire range of room temperature.

4. Distilled water

4.1 For making the extraction, water containing ordinary amounts of CO_2, but not more than corresponds to a pH of 5.9, may be used to test all papers, including those in the neutral range between pH 6.0 to 7.0. A sample of the water must be tested for alkaline impurities, however, by boiling for a few minutes, cooling, and then measuring its pH. If this is found to be higher than 7.3, the water must be distilled from a solution containing approximately 1 gram of $KMnO_4$, and 4 grams of NaOH per liter. A double still-head is usually necessary.

5. Procedure

5.1 Weigh 1.0 gram of the air-dry cut or ground paper into a 125-milliliter Erlenmeyer flask. Add about 20 milliliters of distilled water and macerate with a flattened stirring rod until the specimen is uniformly wet. Then add 50 milliliters more of the water, stir, and affix to the flask a stopper containing a glass tube about 9 milliliters in diameter and 75 centimeters in length, which serves as a condenser. A soil-digestion flask which has a ground-glass stopper and condensing tube in one piece, or a rubber stopper covered with clean metal foil may be used. Place the flask in a steam bath which will maintain the contents of the flask at 95° to 100°C. without boiling the water. Heat at this temperature for 1 hour with occasional shaking, then cool to 20° to 30° C. (68° to 86° F.), and measure the pH of the unfiltered mixture with the glass electrode. If the glass electrode had just previously been used for measurements in the alkaline region, and if the paper to be tested is in the neutral region, immerse the electrode in 0.05 *M* potassium acid phthalate for 1 minute and rinse thoroughly several times with distilled water before use.

6. Report

6.1 The pH value of the paper shall be the average of duplicate determinations which agree within 0.1 pH. The average shall be expressed to the nearest 0.1 pH.

ALPHA CELLULOSE

1. Definition and scope

1.1 Alpha cellulose is that portion of the cellulose of paper fibers that is insoluble in a strong solution of sodium hydroxide under definitely prescribed conditions. It is more stable than the modified forms of cellulose that are dissolved by sodium hydroxide. It is suitable for all kinds of papers.

2. Test specimen

2.1 A sufficient amount of the paper for the tests shall be reduced to cotton-like form in a disintegrator and then thoroughly mixed. Each test specimen shall weigh 0.3 gram.

3. Apparatus

3.1 The special apparatus required for this test is as follows:

3.1.1 A disintegrator which will completely disintegrate the paper without heating or contaminating it. A Koerner type or its equivalent shall be used.

3.1.2 A water bath which can be maintained at 20° ± 0.1°C. (68° ± 2°F.).

4. Reagents

4.1 *Sodium hydroxide solution, 17.5 percent (5.24 N.).*—Allow a 50 percent solution of C.P. NaOH to stand about 1 week in a stoppered vessel to permit settling of Na_2CO_3. Draw off 2.00 milliliters of the supernatant liquid with a pipette; add about 50 milliliters of distilled water and 1 milliliter of 1.5 *M* $BaCl_2$ solution to lessen the effect of CO_2 on the endpoint; and titrate with standard 1 *N* hydrochloric acid, using phenolphthalein as indicator. Knowing the approximate normality of the concentrated NaOH, dilute it with distilled water to 5.24 ± 0.03 *N*, checking the diluted NaOH by titrating 10.00 milliliters of it as before, and diluting further, if necessary, to obtain the normality specified, which will correspond to a strength of 17.5 ± 0.1 percent. (The final solution should have a density of 1.194 ± 0.001 at 15° C., or 1.192 ± 0.001 at 20°C.)

4.2 *Potassium bichromate solution.*—Dissolve 90.0 grams of C.P. moisture-free (100°-105° C.) $K_2Cr_2O_7$ in hot water (70°-90° C.) and dilute to 1 liter after allowing the solution to cool.

4.3 *Ferrous ammonium sulphate solution.*—Dissolve 195 grams of C.P. $Fe(NH_4)_2(SO_4)_2 6H_2O$ in water containing 10 milliliters of concentrated H_2SO_4 and dilute to 1 liter. If the solution is kept out of contact with oxygen, by a slow, continuous stream of hydrogen, for example, its strength will remain quite constant. The amount of H_2 thus used is about one fifth of a 200-cubic-foot cylinder per year. This is not necessary but reduces the frequency with which the bichromate-ferrous ammonium sulphate ratio must be determined from daily to two or three times monthly.

5. Procedure

5.1 Perform all operations described in 5.2 and keep all liquids, as nearly as possible, at 20.0° ± 0.1°C.

5.2 Weigh 0.3 gram ± 10 milligrams of the specimen in a 100-milliliter beaker. Add 20.0 milliliters of 17.5 percent NaOH solution, macerate until the fibers are uniformly wet and dispersed and let stand 10 minutes from the time of addition of HaOH. Then add 33.0 milliliters of water, stir the mixture thoroughly, and let stand 1 hour longer, stirring once during the interval. After stirring once more, pour about 5 milliliters of the unsettled mixture on an 80-mesh copper or brass wire screen fitted into a Gooch crucible. The crucible and ring are supported by a funnel fitted into the neck of a 100-milliliter volumetric flask with a rubber stopper through which passes a glass tube for suction. Form a mat with gentle suction (pres-

sure differential 10 to 20 mm. of Hg). Avoid excessive packing of the fibers as this retards filtering. It may be necessary to refilter the first filtrate, but loss of small amounts of alpha-cellulose to the filtrate does not affect the results appreciably. Pour the remainder of the mixture on the mat, and, before the last of the liquid has run through, wash the beaker and the mat with 35 milliliters of water.

5.3 Moisten the residue of alpha-cellulose with water and remove it from the crucible. Place the crucible upright in a 400-milliliter beaker, fill it with 25 milliliters of 12 *M* H_2SO_4, at room temperature, and rinse it after a few minutes with 50 milliliters more of the acid. Disintegrate the alpha-cellulose pad in the acid, using a thermometer as a stirring rod. Add to the alpha-cellulose solution, with a pipette, 25.00 milliliters of the bichromate solution and heat at 140° to 150° C. for 10 minutes. Bubble air in a fine stream through the solution to prevent bumping, and keep the beaker covered with a watch glass notched to permit entrance of the thermometer and the bubbling tube.

5.4 After the solution has cooled to 130° C., and 50 milliliters of water, rinse the thermometer, etc., and cool the solution to 60° C. or lower. Titrate the remaining bichromate with the ferrous ammonium sulphate solution.

5.5 Pipet exactly half of the filtrate from the alpha-cellulose, after all fibers present have settled, into a 400-milliliter beaker containing 5.0 milliliters of the bichromate solution. If the paper contains oxidizable fillers such as ZnS pigment or $CaSO_3$, filter the filtrate once through a thick pad of asbestos in a Gooch crucible before taking the portion for analysis. (Such fillers remaining with the alpha-cellulose may cause some error, but this is usually slight.) Cautiously and with constant stirring, pour 50 milliliters of concentrated H_2SO_4 down the side of the beaker containing the portion of the filtrate, then heat and titrate as before.

5.6 The amount of bichromate solution consumed in milliliters is calculated for each fraction as follows:

(a) For alpha fraction, ml.$=25-(V_1 \times r)$

(b) For filtrate, ml.$=2\ (5\ V_2 \times r)$

where V_1 and V_2 represent the volumes of ferrous ammonium sulphate necessary to titrate the bichromate remaining after oxidation in the two cases, respectively, and r is the volume of bichromate solution equivalent to 1 milliliter of ferrous ammonium sulphate solution, determined frequently by titrating 5 milliliters of bichromate solution in 100 milliliters of 1:1 H_2SO_4. The alpha-cellulose percentage of the total cellulose is calculated from the volumetric data by substitution in the following equation:

$$\text{Alpha-cellulose, percent} = \frac{(a) \times 100}{(a) + (b)}$$

6. Report

6.1 Alpha cellulose shall be reported as a percentage of the total cellulose, and shall be the average of duplicate determinations the results of which agree within at least 0.4. The average shall be expressed to the nearest whole percent.

ASH

1. Scope

1.1 This method is for the determination of ash, which is defined as the residue after complete combustion of paper and paperboard.

2. Test specimen

2.1 The test specimen shall consist of small pieces of paper so selected as to be representative of the sample. Its total weight shall not be less than 1 gram.

2.2 The specimen shall be dried to constant weight at 100° to 105° C. and weighed to the nearest 1 milligram. This may be done with sufficient accuracy for the purpose in the ignited and weighed crucible used for the ashing of the paper.

3. Apparatus

3.1 A crucible, such as platinum, alundum, porcelain, or silica, which does not change in weight under the ignition conditions, and having a tightly fitting lid, is used for the ignition.

3.2 An electric muffle furnace with an operating temperature of about 925° C. (1,700° F.) is recommended, but a gas burner yielding a similar temperature is also suitable.

4. Procedure

4.1 Ignite the specimen in the crucible, which, together with the cover, has previously been ignited and weighed to 0.1 milligram. To avoid loss of small particles, the crucible shall be covered during the initial ignition of the paper, which shall be done at low temperature, and the temperature shall then be gradually raised to a maximum of about 925° C. (1,700° F.). After this temperature is reached, the lid of the crucible may be slid to one side until the combustion is complete. Care must be taken at all times to protect the contents of the crucible from air drafts. When the paper is completely burned, as indicated by absence of black particles, remove the covered crucible to a desiccator and let it remain until its temperature is in equilibrium with that of the surrounding atmosphere. Weigh the crucible and contents to the nearest 0.1 milligram. Repeat the ignition and weighing until the weight is constant.

4.2 The percentage of ash shall be computed on the basis of the weight of the specimen when dried to constant weight at 100° to 105° C. and shall be the average of at least two determinations. The results should agree to within 0.1 for papers containing 5 percent ash or less, to within 0.15 for papers containing from 5 to 10 percent ash, and to within 0.2 for papers containing over 10 percent ash.

5. Report

5.1 The percentage of ash shall be reported to the nearest 0.05 for papers containing 5 percent ash or less. For papers containing 5 to 10 percent ash it shall be reported to the nearest 0.1, and for paper containing over 10 percent ash to the nearest 0.2.

COPPER NUMBER

1. Definition and scope

1.1 Copper number of paper designates the amount of copper precipitated as cuprous oxide by the cellulose under definitely prescribed conditions. It is a measure of the amount of unstable modified forms of cellulose present and thus is related to the stability of papers. It is suitable for all papers except those containing zinc sulfide, which is usually a component of zinc pigments.

2. Test specimen

2.1 A sufficient amount of paper for the tests shall be reduced to cotton-like form in a disintegrator and then thoroughly mixed. Each test specimen shall weigh about 1.5 grams.

3. Apparatus

3.1 The special apparatus required for this test is as follows:

3.1.1 A grinder which will completely disintegrate the paper without heating or contaminating it. The grinder shall be a Koerner type or its equivalent.

3.1.2 A steam or oil bath which can be maintained at 100° C.

4. Reagents

4.1 *Copper sulphate solution.*—Dissolve 100 grams of $CuSO_4 5H_2O$ in water and dilute to 1 liter.

4.2 *Carbonate bicarbonate solution.* — Dissolve 350 grams of $Na_2Co_3.10H_2O$ (or 129 grams of anhydrous Na_2CO_3) and 50 grams of $NaHCO_3$ in water and dilute to 1 liter.

4.3 *Molybdophosphoric acid.* — Dissolve 100 grams of sodium molybdate, $Na_2Mo04.2H_2O$, and 75 milliliters of phosphoric acid (83 percent) in a mixture of 275 milliliters of concentrated H_2SO_4 and 1.75 liters of water.

4.4 *Sodium carbonate solution.*—An approximately 5 percent solution of Na_2CO_3 in water.

4.5 *Potassium permanganate solution.*—0.05 *N*: 1.5815 grams per liter.

5. Procedure

5.1 Allow the specimen to come to moisture equilibrium with the atmosphere of the balance. Weigh about 1.5 grams (to nearest 10 mg.) of the ground paper. Weigh at the same time, samples for moisture and ash determinations.

5.2 Immediately before use, add 5.0 milliliters of copper sulphate solution to 95 milliliters of carbonate-bicarbonate solution. Bring the mixture to a boil in 2 minutes, and pour it over 1.5 grams of the ground sample in a 125-milliliter Erlenmeyer flask. Stir well with a glass rod in order to distribute the fibers and to remove air bubbles. Fit the flask with a loosely fitting glass bulb or stopper and submerge completely in a steam bath at atmospheric pressure. Occasionally fibers tend to float to the surface, therefore the should be shaken from time to time to tribute them. Remove the flask from the steam bath at the end of 3 hours. Filter on an ashless filter paper in a 7.5 centimeter Buchner funnel, using suction. Wash by flooding with 100 milliliters of 5 percent Na_2CO_3 solution at about 20° C. and then by flooding with 250 milliliters of hot water (about 95° C.), discarding the filtrates. Transfer the fibers and filter paper to a small beaker, add 25 milliliters of the molybdophosphoric acid solution and macerate well with a flattened glass rod. Transfer to a Büchner funnel again and wash thoroughly with cold water until the blue molybdenum color is removed from the fibers. Dilute the filtrate with water to approximately 700 milliliters and titrate it with 0.05 *N* $KMn0_4$ to a faint pink.

5.3 The copper number is the number of grams of metallic Cu in the Cu_2O resulting from the reduction of the $CuSO_4$ by 100 grams of the paper fibers. This is calculated by the formula:

$$\text{Copper number} = \frac{6.36 \times \text{ml. } KMnO_4 \times N}{W}$$

where N is the normality of the $KMnO_4$, and W is the weight in grams of the test specimen after deduction of the weight of the non-fibrous materials. Correction of the weight of the test specimen shall always be made for moisture and ash. Not less than two determinations shall be made and the average of the results rounded off to the nearest 0.1, shall be reported. Duplicate determinations should agree within 0.2.

6. Report

6.1 The copper number shall be reported to one decimal place on the basis of total fiber content.

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MOISTURE

1. Scope

1.1 This method is for determining the amount of moisture in paper.

2. Test specimens

2.1 When the amount of moisture is determined to compute the results of chemical tests of paper on a moisture-free basis, the test specimens shall weigh not less than 1 gram and preferably not less than 2 grams each, and when weighed shall be in moisture equilibrium with the specimens being analyzed. When moisture is determined to compute the amount of moisture in a delivery of paper, the test specimens shall weigh not less than 50 grams each, and they shall be obtained in accordance with method 160. In sampling a delivery for moisture content, extreme care shall be taken to avoid change in moisture content during sampling. Handle the specimen with clean, dry rubber gloves, transfer it to the container as soon as withdrawn, and close the container immediately. If a delay of over a second or two in transferring the specimen to the container is unavoidable, keep the specimen covered on both sides with several adjacent layers of the paper from which it was withdrawn until ready to place it in the container. Unless the specimen is later to be spread out in the oven, avoid stuffing the container tightly.

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3. Apparatus

3.1 The special apparatus required for this test is an airtight container in which the test specimen is dried and weighed. For the minimum size of specimen designated, a weighing bottle approximately 65 millimeters (2½ inches) in height and 45 millimeters (1¾ inches) in diameter is suitable. For the larger specimens, a proportionately larger, lightweight, airtight metal can should be used, preferably containing a removable lightweight wire-mesh basket.

3.2 The oven used to dry the paper shall be equipped with means for ensuring adequate temperature control and air circulation and preferably equipped with means of drying the air entering the oven.

4. Procedure

4.1 After the specimen has been placed in a weighed container, weigh it in the closed container to obtain the moist gross weight of the sample.

4.2 For large samples, unless the container has a removable basket, take the specimen from the container in which they were weighed, spread them in a basket or tray which will permit free circulation of air around them, and place them, as well as the original containers, in the oven. Heat for about 2 hours, replace the specimens in the original container and close it, doing this if possible without removing the specimens from the oven. Let the closed container and contents cool at room temperature, and weigh.

4.3 For small samples, without removing the specimens from the weighing bottles, remove the stoppers, heat for 1 hour, close the bottles in the oven, cool to room temperature in a desiccator, and weigh. Remove the stopper momentarily just before weighing, to adjust any change in air pressure.

4.4 Dry the specimens in either case at 100° to 105° C. Repeat the periodical drying and weighing until the difference in weight between two successive weighings is not more than 0.1 percent of the weight of the

specimen. For 2-gram samples, all weighings shall be made to an accuracy of 1 milligram; for other samples, at least to an accuracy of 1/2000 of the original weight of the sample.

5. Report

5.1 The moisture shall be reported as the percentage loss in the original weight of the paper to the nearest 0.1.

ROSIN

1. Scope

1.1 This method is for the qualitative and quantitative determination of rosin (colophony) in paper.

2. Qualitative tests

2.1 A positive result obtained by both of the following procedures shall be regarded as conclusive evidence of the presence of rosin or rosin soap added as a sizing material.

2.1.1 *Lieberman-Storch test.* — Place about 1 gram of the paper, cut into small pieces, in a clean, dry test tube. Add 5 milliliters of C.P. acetic anhydride and boil down to about 1 milliliter. (*Caution*—The fumes of the anhydride are very irritating and should be burned as they leave the mouth of the test tube.) Pour the liquid residue into a clean, dry porcelain crucible and cool to room temperature or lower. If any waxy particles separate out, they should be filtered off on a dry filter paper. Add carefully down the side of the crucible, 1 drop of concentrated sulphuric acid. A fugitive, rose-violet coloration formed where the acid meets the anhydride indicates rosin.

2.1.2 *Raspail test.*—Place the paper on a glass or porcelain plate and apply a drop of a nearly saturated solution of sugar. After a few moments remove the excess sugar solution with filter paper. Add a drop of concentrated H_2SO_4 to the sugar on the paper. A raspberry-red coloration indicates the presence of rosin.

3. Quantitative determination

3.1 *Test specimen.*—The test specimen shall consist of strips about 0.25 by 1.6 inches (6 x 40 mm.) weighing 5 to 7 grams.

3.2 *Apparatus.*—A suitable extraction apparatus, such as a Soxhlet or Underwriters', is required.

3.3 *Extracting solvent.*—To 1 liter of 95 percent ethyl alcohol add 4 milliliters of concentrated HCl and mix.

3.4 *Procedure.*—Place the strips lengthwise in the syphon cup of the extractor, taking care to avoid packing them together tightly. For the extraction, use about 2 to 2.5 times the volume of solvent required to fill the syphon cup. Extract at a rate of 15 syphonings per hour (which should yield a volume of about 250 ml. of solvent distilled per hour) for a period of 2 hours.

3.4.1 When the extraction period is completed, evaporate off the solvent in the flask on the steam bath until the odors of alcohol and HCl are no longer noticeable. Place the flask in an oven at 100° to 105°C. for 15 minutes, cool to room temperature and add 20 milliliters of anhydrous ether. The rosin dissolves in from 5 to 30 seconds, but if covered by foreign material it must be uncovered by scraping the mass with a stirring rod. After standing 15 to 20 minutes to further the coagulation and settling out of the foreign matter if necessary, filter the ether solution with suction through a fine-pore filter paper cut to fit a Gooch crucible, directly into a weighed beaker. It is usually necessary to refilter the filtered solution through the same paper. Another 20-milliliter portion of ether is used for rinsing, adding the rinsing to the solution in the weighed container. Evaporate the ether, then dry in an oven at 100° to 105°C. for 15 minutes and weigh to the nearest 1 milligram. Repeat drying and weighing until the weight is constant to plus or minus 1 milligram.

3.5 *Report.*—The result of the rosin determination shall be reported to the nearest 0.1 as a percentage of the paper dried at 100° to 105°C.

FIBER IDENTIFICATION AND QUANTITATIVE DETERMINATION

1. Scope

1.1 This method covers the identification of the kinds of fibers present in paper and paperboard, and their quantitative estimation when the material contains mixtures of different kinds of fibers.

2. Test specimen

2.1 The specimen for test shall consist, when possible, of pieces having a total area of 25 square centimeters (4 sq. in.) cut from different portions of the test sample so as to be representative of it. If sufficient specimen is not available, an area of 3 square centimeters (½ sq. in.) will be recognized for quantitative analysis and 0.9 square centimeter (⅛ sq. in.) for fiber identification.

3. Apparatus and materials

3.1 *Microscope.*—A compound microscope equipped with a mechanical stage, Abbe condenser, eyepiece, and achromatic objective. A magnification of 100 diameters is recommended. This is obtained by using a 16-milimeter objective and a 10-X Huygenian eyepiece. If the microscope has an adjustable draw tube, a 160-millimeter tube length is used; and if an apochromatic objective is employed, it is necessary to use a compensating eyepiece and an achromatic condenser.

3.1.1 *Counting eyepiece.*—The eyepiece shall be equipped with a suitable reference point (cross hairs, pointer, or dot) for counting the fibers passing under it.

3.2 *Dropper.*—A glass tube 6 millimeters in internal diameter and about 10 centimeters long, fitted at one end with a rubber bulb and having the other end carefully smoothed but not constricted. The tube shall be graduated at the lower end so as to deliver 0.5 cubic centimeter.

3.3 *Slides and cover glasses.*—Slides and cover glasses shall be kept in 50 percent alcohol, or washed with it before use. The slides shall be 1 inch x 3 inch, of colorless glass. The cover glass shall be 1 inch square (No. 2).

3.4 *Hot plate.*—The hot plate shall have a solid metal top with an absolutely level surface and be capable of heating to 50° to 60° C. The top of the hot plate may be painted with a dull black paint, well baked-on, and then marked with suitable lines to correspond with the lines on the ends of the slides. This is for convenience in making up samples.

3.5 *Dissecting needles.*

3.6 *Aluminum soap solution.*

3.6.1 To 600 cubic centimeters of distilled water add 15 grams of pure white soap shavings (Ivory soap is satisfactory) and stir until completely dissolved; then add 10 grams of pure aluminum sulphate, $Al_2(SO_4)_3.18\ H_2O$, and stir until the precipitate forms a wax-like mass of Al-stearate which may be lifted out with a stirring rod. Place the precipitate in a desiccator for 48 hours or more. Store in a well stoppered bottle for use as needed.

3.6.2 To 50 cubic centimeters of benzene in a glass-stoppered bottle add 0.7 gram of the Al-stearate and shake well daily until completely dissolved. This usually requires about 10 days.

3.6.3 If, after standing for several weeks, the mixture should appear to have lost some of its water-repelling capacity, a small piece of Al-stearate dropped into the solution will correct this condition within a few hours.

4. Disintegration of test specimen

4.1 Tear the test specimen into small pieces and place in a small beaker. Obtain

the approximate weight of the specimen in order to calculate the proper dilution of the disintegrated specimen. Cover with 1-percent NaOH solution, bring to a boil on a hot plate, decant the liquid, and wash twice with distilled water. Cover with 0.05 *N* HCl, let stand several minutes, decant the acid, and wash several times with distilled water. Drain off the water, roll the pieces of paper into pellets gently between the thumb and fingers, put into a 500-milliliter Erlenmeyer flask, add a little water and shake vigorously until the water is absorbed by the paper. Add more water and shake, and continue this treatment until the paper is thoroughly defibered. Dilute the suspension of fibers by pouring away part of it and adding water to the remainder until the suspension has a consistency of about 0.05 percent fibers. Partially fill a test tube with the mixture.

5. Preparation of slides

5.1 Keep the microscope slides and cover glasses in 50 percent alcohol. After the slides have been dried and polished, draw lines on them 1 inch from each end in the following manner: Arrange six slides or less so that the edges are in contact. Place a 6-inch wooden rule (1 inch wide and having a metal ferrule) on the slides so that the rear edge of the rule is even with their ends. Dip a clean steel pen in an ordinary pen holder into the Al-soap solution, and let the excess flow off by touching the point to the edge of the bottle. Draw a line on the slides along the metal edge of the ruler. Repeat the operation on the opposite ends of the slides. This gives a thin line of Al-stearate across each slide 1 inch from each end, and these lines will keep the fiber suspension within the area of 1 square inch at each end of the slide. It is advisable to wipe the pen point clean after each set of markings. Remove dust and lint from the slides with a small camel-hair brush; then place the slides on the hot plate at about 60° C. The hot plate must be level.

5.2 Thoroughly mix the test-tube suspension of the sample, insert the dropper to the middle of the suspension and withdraw a portion of the mixture. Place 0.5 cubic centimeters of the suspension immediately on each end of the slide. (The test tube must be shaken and sample withdrawn for each end of the slide). Evaporate a portion of the water, then carefully tap the suspension with a dissecting needle to distribute the fibers evenly inside the 1-inch-square areas on the end of each slide. Leave the slides on the hot plate until completely dried. They are then ready for staining as described under the various stains.

NOTE 1.—Care must be taken that the unstained fibers on the slide are not touched by the analyst's fingers. The slide should be allowed to cool before adding the stain, otherwise confusing colors may be obtained.

6. Procedure

6.1 Examine the prepared slides microscopically, using a magnification of about 100 diameters. For a quantitative estimation, place the stained slide on the mechanical stage of the microscope and count the different kinds of fibers across the slide, beginning 2.5 millimeters from one long edge and making five trips across one end of the slide along lines spaced approximately 5 millimeters apart. The distribution of fibers on the slide shall be such that five trips across the slide at 5-millimeter intervals will include between 200 and 300 fibers. Repeat this on the sample on the other end of the slide. If these two results agree within 5 percent, no further counts need be made; otherwise another count shall be made on one end of a second slide and the three results averaged.

6.1.1 As each fiber passes under the reference point in the eyepiece, count it as one regardless of its size. If aggregations of fibers, such as occur in groundwood, are encountered, the number of single fibers in the aggregation shall be estimated and counted as if the fibers were separated completely.

7. Staining and identification

7.1 *Herzberg stain.*

7.1.1 Prepare the following solutions:

(a) An aqueous solution of C.P. zinc chloride (fused sticks in 50-gram, unbroken, sealed bottles) saturated at 20° C.

(b) 0.25 gram of C.P. iodine and 5.25 grams of C.P. potassium iodide dissolved in 12.5 cubic centimeter of distilled water.

Mix 25 cubic centimeters of solution (a), measured at 20° C., with solution (b). Pour into a narrow cylinder and let stand until clear (12 to 24 hours). Decant the supernatant liquid into an amber-colored glass-stoppered bottle and add a small piece of iodine to the solution. Avoid undue exposure to light and air.

7.1.2 For staining, apply 3 or 4 drops of the stain to the dried fibers, cover the whole with a cover glass in such a manner as to avoid air bubbles, let stand for 1 or 2 minutes, and drain off the surplus by tipping the slide edgewise on a blotter.

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7.1.3 The following colors are developed by this stain:

Red—Linen, cotton, bleached manila hemp (abacá).

Blue—Chemically prepared fibers low in lignin, from wood, straw, and esparto.

Yellow—Fibers high in lignin, such as groundwood, (unbleached) jute, and unbleached manila hemp (abacá).

7.2 *Lofton-Merritt stain.*—This stain shall be limited to differentiation between coniferous (softwood) unbleached sulphite and coniferous (softwood) unbleached sulphate.

7.2.1 Prepare the following solutions:

(a) 2 grams of malachite green in 100 cubic centimeters of water.

(b) 1 gram of basic fuchsin in 100 cubic centimeters of water.

Mix these in the proportion of 1 part of solution (a) to 2 parts of solution (b). Add a few drops of the compound stain to the fibers and let stand 2 minutes. Remove excess stain by means of a hard filter paper and add a few drops of 0.1 percent HCl. After about 30 seconds remove the excess acid. Finally add a few drops of water and remove the excess.

7.2.2 As dyes from different sources vary, it is necessary to test them by staining known fibers. Unbleached sulphate fibers are stained blue or blue-green and unbleached sulphite fibers purple or lavender. If any purple fibers appear in unbleached sulphate fibers, this indicates there is too much fuchsin present and more malachite green solution must be added. The opposite is indicated if some unbleached sulphite fibers develop a green or blue color.

7.3 *Modified Bright stain.*—This stain shall be used only to show the presence of groundwood and unbleached pulps. Percentages of unbleached or groundwood pulp found by this method may be lower than the actual amount present, due to the fact that some well-cooked unbleached pulp may fail to give a blue color.

7.3.1 Prepare the following solutions:

(a) 2.7 grams of ferric chloride, $FeCl_3 \cdot 6H_2O$, per 100 cubic centimeters of distilled water.

(b) 3.29 grams of potassium ferricyanide, $K_3Fe(CN)_6$, per 100 cubic centimeters of distilled water.

(c) 0.5 grams of benzopurpurin 4B crude in 100 cubic centimeters of 50 percent ethyl alcohol. The dye used shall be DuPont Purpurin 4B Concentrated, or its equivalent. The solution is warmed until the dye is completely dissolved. (Some of the dye will precipitate on cooling.)

Filter solutions (a) and (b) and keep 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.

7.3.2 Apply 3 drops each of solutions (a) and (b) to the fibers and let stand 1 minute. Remove the excess stain by means

of blotting or filter paper, add a few drops of solution (c), and let stand 2 minutes. Finally remove water, using a glass cover.

7.3.3 Any blue coloration of fibers shows the presence of groundwood and/or unbleached pulps. Bleached fibers or fibers practically free from ligno-cellulose stain red.

8. Calculation of results of quantitative estimation

8.1 Multiply the number of each kind of fiber counted by its factor as given in table I to obtain the weighted count. Add together the weighted counts of each kind of fiber and calculate each to a percentage of the total.

Table I.—*Weight factors for different kinds of fibers*

Kind of fiber	Factor
Groundwood	1.3
Linen and cotton	1.0
Chemical coniferous wood	0.9
Chemical deciduous wood	0.6

9. Report

9.1 The proportions of the various fibers found shall be reported in terms of percentages by weight of the total fiber composition to the nearest multiple of 5. When any fiber is found present in amount less than 2½ percent, it shall be reported as "trace."

9.2 In reporting results by the Herzberg stain the following nomenclature shall be used:

Rag (cotton, linen, hemp).....Esparto
Coniferous chemical wood.....Jute or manila
Deciduous chemical wood.....Straw
Mechanical wood.....Japanese fiber

When stains other than the Herzberg are used, appropriate qualifying terms may be applied, such as bleached, unbleached, sulphate, sulphite, etc.

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Copies of this specification may be purchased for 25 cents.

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a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional

b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional

c. MAILING ADDRESS *(Street, City, State, ZIP Code)* – Optional

8. DATE OF SUBMISSION *(YYMMDD)*

(TO DETACH THIS FORM, CUT ALONG THIS LINE.)

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