Designation: D 202 – 97 (2002)$^{\epsilon1}$

An American National Standard

Standard Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation[1]

This standard is issued under the fixed designation D 202; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

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

$^{\epsilon1}$ NOTE—Editorial changes made to footnotes 13-15, and Section 1 September 2002.

1. Scope

1.1 These test methods cover procedures for sampling and testing untreated paper to be used as an electrical insulator or as a constituent of a composite material used for electrical insulating purposes.

1.1.1 Untreated papers are thin, fibrous sheets normally laid down from a water suspension of pulped fibers (usually cellulosic), which may contain various amounts of nonfibrous ingredients, and which are calendared, if required, to obtain desired thickness and density. Nevertheless, these test methods are applicable, generally although not invariably, to papers formed by other means, to papers modified (during or after formation) by additions, and to papers given subsequent mechanical treatments such as creping.

1.1.2 As an electrical insulating and dielectric material, paper is considered "untreated" until it is subjected to a manufacturing process such as drying, impregnation, or varnish treatment.

1.1.3 The test methods given herein were developed specifically for papers having a thickness of 0.75 mm (0.030 in.) or less. A number of these test methods are also suitable for use on other materials such as pulps or boards. Refer to Test Methods D 3376 or Methods D 3394 to determine which tests are applicable to pulps or electrical insulating boards. In the paper industry, some products in thicknesses of less than 0.75 mm are termed "paperboard". Such products are included within the scope of these methods.

1.1.4 These test methods are applicable to flexible fibrous-mat materials formed from suspensions of fiber in fluids other than water. These mats may have thicknesses approaching 2 mm, and may contain fibers that are natural, synthetic, organic, or inorganic; fillers that are natural, synthetic, organic, or inorganic; and flexible polymeric binder materials.

1.2 The procedures appear in the following sections:

Procedure	Sections	ASTM or TAPPI Reference (Modified)
Absorption (Rise of Water)	78 to 83	...
Acidity-Alkalinity-pH	45 to 54	E 70
Air Resistance	98 to 101	D 726
Aqueous Extract Conductivity	55 to 64	...
Ash Content	40 to 44	D 586
Bursting Strength	102 to 107	D 774
Chlorides (Water-Extractable)	165 to 183	...
Conditioning	15	D 6054
Conducting Paths	138 to 151	...
Density, Apparent	29 to 33	...
Dielectric Strength	152 to 157	D 149
Dimensions of Sheet, Rolls and Cores	16 to 24	D 374
Dissipation Factor and Permittivity	158 to 164	D 150
Edge-Tearing Resistance	126 to 130	D 827
Fiber Analysis	74 to 77	D 1030
Folding Endurance	108 to 110	T 423 and D2176
Grammage	25 to 28	D 646
Permittivity	158 to 164	D 150
Heat Stability in Air	131 to 137	D 827
Impregnation Time	84 to 91	...
Internal-Tearing Resistance	121 to 125	D 689 or T414
Moisture Content	34 to 39	D 644 and D3277
Particulate Copper	193 to 202	...
Particulate Iron	184 to 192	...
Reagents	4	D 1193
Reports	14	E 29
Sampling	6 to 13	D 3636
Silver Tarnishing by Paper and Paperboard	203 to 206	T 444
Solvent-Soluble Matter	65 to 73	...
Surface Friction	92 to 97	D 528 and T455
Tensile Properties	111 to 120	D 76, E4
Thickness (see Dimensions)	16 to 24	D 374

1.3 The tests for Holes and Felt Hair Inclusions and the Stain Test for Fine Pores, have been removed from this compilation of test methods. These test methods were specific to grades of capacitor paper formerly covered by Specification D 1930, which has been withdrawn.

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[1] These test methods are under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials and are the direct responsibility of Subcommittee D09.19 on Dielectric Sheet and Roll Products.

Current edition approved March 10, 1997. Published March 1998. Originally published as D 202 – 24 T. Last previous edition D 202 – 92.

NOTE 1—This compilation of test methods is closely related to IEC Publication 60554-2. Not all of the individual methods included herein are included in IEC 60554-2, nor are all of the methods in IEC 60554-2 included in this standard. The individual procedures as described in the two standards are in general sufficiently close to each other that it is reasonable to expect that test results obtained by most of the procedures specified in either standard will not differ significantly. However, before assuming that a procedure in these test methods is exactly equivalent to an IEC 60554-2 procedure, the written procedures should be compared closely, and if it seems advisable, test results by the two procedures should be compared.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See 43.2.1, 71.1, 143.1, 148.1 and 156.1 for specific hazards.

2. Referenced Documents

2.1 *ASTM Standards:*

D 76 Specification for Tensile Testing Machines for Textiles[2]

D 149 Test Method for Dielectric Breakdown Voltage and Dielectric Strength of Solid Electrical Insulating Materials at Commercial Power Frequencies[3]

D 150 Test Methods for AC Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials[3]

D 374 Test Methods for Thickness of Solid Electrical Insulation[3]

D 528 Test Method for Machine Direction of Paper and Paperboard[4]

D 586 Test Method for Ash in Paper[4]

D 644 Test Method for Moisture Content of Paper and Paperboard by Oven Drying[4]

D 646 Test Method for Grammage of Paper and Paperboard (Weight Per Unit Area)[4]

D 689 Test Method for Internal Tearing Resistance of Paper[4]

D 726 Test Method for Resistance of Nonporous Paper to Passage of Air[4]

D 774 Test Method for Bursting Strength of Paper[4]

D 827 Test Method for Edge-Tearing Resistance of Paper[5]

D 1030 Test Method for Fiber Analysis of Paper and Paperboard[4]

D 1193 Specification for Reagent Water[6]

D 1389 Test Method for Proof-Voltage Testing of Thin Solid Insulating Materials[3]

D 1677 Methods for Sampling and Testing Untreated Mica Paper Used for Electrical Insulation[3]

D 1711 Terminology Relating to Electrical Insulation[3]

D 2176 Test Method for Folding Endurance of Paper by the M.I.T. Tester[4]

D 2413 Test Methods for Preparation and Electrical Testing of Insulating Paper and Board Impregnated with a Liquid Dielectric[3]

D 2753 Specification for Electrolytic Capacitor Paper[3]

D 2865 Practice for Calibration of Standards and Equipment for Electrical Insulating Materials Testing[7]

D 3277 Test Method for Moisture Content of Oil-Impregnated Cellulosic Insulation[8]

D 3376 Test Methods of Sampling and Testing Pulps to be Used in the Manufacture of Electrical Insulation[7]

D 3394 Test Methods for Sampling and Testing Electrical Insulating Board[7]

D 3636 Practice for Sampling and Judging Quality of Solid Electrical Insulating Materials[7]

D 6054 Practice for Conditioning Electrical Insulating Materials for Testing[7]

E 4 Practices for Force Verification of Testing Machines[9]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[10]

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[11]

2.2 *TAPPI Standards:*[12]

T 413 Ash in Paper and Paperboard

T 414 Internal Tearing Resistance of Paper

T 423 Folding Endurance of Paper (Schopper Type Test)

T 444 Silver Tarnishing by Paper and Paperboard

T 455 Identification of Wire Side of Paper

T 470 Edge Tearing Resistance of Paper

2.3 *IEC Standard:*

IEC 60554-2 Specification for cellulosic papers for electrical purposes—Part 2: Methods of test[13]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions pertaining to sampling refer to Terminology D 1711 or to Practice D 3636.

3.1.2 For definitions pertaining to dissipation factor and permittivity refer to Terminology D 1711 or to Test Methods D 150.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *air resistance, of paper*, *n*—a paper property which quantifies impediment to the transverse passage of air through the paper under specific conditions of test, and reported as either time for a specified volume per area of test or volume for a specified time per area of test.

3.2.1.1 *Discussion*—It is expressed in terms of time (seconds) required for passage of a specified volume of air through a known area of paper, or, as the volume of air passing through the paper in a given length of time.

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[2] *Annual Book of ASTM Standards*, Vol 07.01.

[3] *Annual Book of ASTM Standards*, Vol 10.01.

[4] *Annual Book of ASTM Standards*, Vol 15.09.

[5] *Discontinued 1981. See Annual Book of ASTM Standards*, Vol 15.09.

[6] *Annual Book of ASTM Standards*, Vol 11.01.

[7] *Annual Book of ASTM Standards*, Vol 10.02.

[8] *Annual Book of ASTM Standards*, Vol 10.03.

[9] *Annual Book of ASTM Standards*, Vol 03.01.

[10] *Annual Book of ASTM Standards*, Vol 14.02.

[11] *Annual Book of ASTM Standards*, Vol 15.05.

[12] Available from the Technical Association of the Pulp and Paper Industry, 15 Technology Parkway, S., Norcross, GA 30092.

[13] Available from Global Engineering Documents, 15 Inverness Way, East Englewood, CO 80112-5704.

3.2.2 *ash content of paper*, *n*—the solid residue remaining after combustion of the paper under specified conditions, expressed as a percentage of the dry mass of the original specimen.

3.2.3 *basis weight of paper*—see *grammage of paper*.

3.2.4 *bursting strength of paper*, *n*—the hydrostatic pressure required to produce rupture of a circular area of the material under specified test procedures.

3.2.5 *coverage of paper*, *n*—the reciprocal of grammage (or basis weight).

3.2.6 *elongation of paper*, *n*—the maximum tensile strain developed in the test specimen before break in a tension test under prescribed conditions, calculated as the ratio of the increase in length of the test specimen to the original test span, and expressed as a percentage.

3.2.6.1 *Discussion*—It is calculated as the ratio of the increase in length of the test specimen to the original test span, and is expressed as a percentage.

3.2.7 *folding endurance of paper*, *n*—the resistance to fatigue resulting from repeated folding under specified conditions of test, expressed as the number of double folds required to rupture a specimen, or as the logarithm of that number.

3.2.7.1 *Discussion*—The level is expressed as the number of double folds required to rupture a specimen. Sometimes the level is expressed as the logarithm of the number.

3.2.8 *grammage of paper*, *n*—the mass per unit area of paper, expressed as grams per square metre.

3.2.8.1 *Discussion*—Grammage is sometimes called weight or basis weight of paper. These terms are most frequently used when non-metric units are used, and the area is that of the paper in one of the several standard reams of papers defined within the paper industry.

3.2.9 *impregnation time of paper*, *n*—the time in seconds required for a liquid of specified composition and viscosity to penetrate completely from one face of a sheet of paper to the other under certain prescribed conditions.

3.2.10 *internal tearing resistance of paper*, *n*—the force required to continue a previously-initiated tear across a specified distance in a single thickness of paper, expressed as the average force per sheet to tear one or more sheets together.

3.2.10.1 *Discussion*—It is indicated on the specified apparatus and reported as the average force per sheet to tear one or more sheets together across a specified distance.

3.2.11 *loss on ignition of inorganic fiber paper*, *n*—the volatile and combustible fraction of a paper, expressed as a percentage of the original dry mass lost upon ignition, using a specified procedure.

3.2.11.1 *Discussion*—It is expressed as a percentage of the original dry weight lost upon ignition, and is usually used instead of ash content when dealing with papers which are principally composed of inorganic fibers.

3.2.12 *solvent-soluble material in paper*, *n*— the mass of material that can be extracted from a dry specimen by a specified solvent under prescribed conditions, expressed as a percentage of the original dry mass.

3.2.13 *kinetic surface friction of paper*, *n*— the ratio of the force parallel to the surfaces of two pieces of paper in contact with each other to the force normal to the surfaces required to continue previously-initiated movement relative to each other at constant speed.

3.2.13.1 *Discussion*—The test may be made using a paper-covered block on a paper-covered inclined plane, in which case the result is expressed in degrees of angle of inclination of the plane which will cause the block to continue an initiated movement.

3.2.14 *tensile energy absorption of paper (TEA)*, *n*—the work performed when a paper specimen is stressed to break in tension under prescribed conditions, as measured by the integral of the tensile stress over the range of tensile strain from zero to the strain corresponding to maximum stress, expressed as energy (work) per unit of original surface area of the test specimen.

3.2.14.1 *Discussion*—The TEA is expressed as energy (work) per unit of original surface area (length × width) of the test specimen.

3.2.15 *tensile strength of paper*, *n*—the maximum tensile stress developed in a test specimen in a tension test carried to break under prescribed conditions, expressed for thin papers as force per unit original width of the test specimen.

3.2.15.1 *Discussion*—Tensile stress is the force per unit of original cross-sectional area, but in thin materials such as paper it is commonly expressed in terms of force per unit of original width.

3.2.16 *thickness of an electrical insulating material*, *n*—the perpendicular distance between the two surfaces of interest, determined in accordance with a standard method.

3.2.16.1 *Discussion*—The thickness of papers under 0.05 mm (0.002 in.) in thickness, is often defined as one tenth that of a stack of ten sheets in certain paper specifications.

3.2.17 *water extract conductivity of paper*, *n*—the apparent volume conductivity at 60 Hz of a specimen of water that has been used to dissolve water-soluble impurities from a specimen of paper under prescribed conditions.

4. Reagents

4.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[14] Other grades are acceptable, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Except where otherwise indicated, use reagent water, Type III, of Specification D 1193.

5. Precision and Bias

5.1 For individual test methods that follow, where no precision and bias section is included and where the procedure is contained in another standard to which reference is made,

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[14] "Reagent Chemicals, American Chemical Society PO Box 182426, Columbus, OH 43218-2426." For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

refer to that standard for information relative to precision and bias for that test method.

SAMPLING

6. Scope

6.1 This test method covers the procedure for judging lot acceptability of electrical insulating papers. It is designed for the purpose of determining acceptability of all or that portion of a shipment to a customer identified by a manufacturer's lot number. It is not intended to cover internal paper mill quality control plans. This test method is intended for use in conjunction with product specifications for electrical insulating papers.

7. Summary of Test Method

7.1 After Acceptable Quality Levels (AQLs) are agreed upon for each of the various specification properties, sampling plans are selected and the basis for acceptance or rejection of a lot of material is established.

8. Significance and Use

8.1 In the buyer-seller relationship it is necessary that an understanding exists as to the expected nominal characteristics of the product, and the magnitude of permissible departure from the nominal values. Also, it is necessary that an agreement be reached as to how many units of a lot can fall outside of the specification limits without rejection of the lot. It is this latter subject that is addressed by this test method.

9. Establishing AQLs

9.1 AQLs for each critical major and minor property are as mutually agreed upon between the manufacturer and the customer. If needed, establish group AQLs for given groups of properties; these too are mutually agreed upon between the manufacturer and the purchaser.

10. Selection of Samples

10.1 A number of paper properties are listed in Table 1, together with the appropriate number of test specimens and test measurements for each property. Use these values for guidance in determining sample sizes.

10.2 From Table 2 select a sampling plan appropriate to the lot size and the agreed-upon AQL. Alternatively, refer to Practice D 3636 for selection of a sampling plan. Refer to Practice D 3636 for further information relative to the principles and practices of sampling methods.

10.3 Inasmuch as several properties of paper (notably moisture content and aqueous extract conductivity) may change with time, define a reasonable maximum time between receiving a lot of paper and testing it for such properties, either in the material specification or by agreement between the seller and the purchaser.

10.4 For purposes of sampling for lot acceptance or rejection, select the number of units of product from each lot in the shipment in accordance with sampling plans selected from 10.2. Select units of product at random so as to be representative of the lot. Take care to avoid selection of all units of product from the top or bottom, one side or the other, or from any specific location in the lot.

10.5 If more than one lot sample size is used, first determine those properties measured from the smaller sample, after which this sample may be included as part of the larger sample.

10.6 *Selecting Test Unit from Unit of Product*:

10.6.1 For units of product consisting of rolls 380 mm (15 in.) or more in width, take a test unit at least 0.5 m^2 (5 ft^2) in area, cut across the entire width of the roll.

10.6.2 Cut test specimens from this area such that they represent the entire width of the roll.

10.6.3 If the paper is available in rolls less than 380 mm in width, take a test unit at least 1.25 m (4 ft) in length and cut test specimens so as to be representative of the full width of the roll.

10.6.4 When the unit of product is defined as a sheet, take the test unit from the sheet so that the entire width and length of the sheet are represented.

TABLE 1 Number of Test Specimens per Test Unit; Number of Test Measurements per Test Specimen

Property	Unit of Product—Roll, Pad, Bobbin, or Sheet		Unit of Product—Skid, Pallet, Box, Carton, Case, Package, Bundle, or Ream	
	Number of Test Specimens per Test Unit	Minimum Number of Test Measurements per Test Specimen	Number of Test Specimens per Test Unit	Minimum Number of Test Measurements per Test Specimen
Aqueous extract conductivity, acidity-alkalinity-pH, ash, moisture, solvent-soluble matter, chlorides, fiber analysis, surface friction	2	1	2	1
Basis weight, bursting strength, folding endurance, tensile properties, absorption	10	1	10	1
Thickness, dielectric strength^A	1	5^A	5	1
Holes and felt hair inclusions, dissipation factor, density, dry coverage, core dimensions, sheet squareness	1	1	1	1
Conducting paths	5	1	5	1
Tearing strength	5	1	5	1
Air resistance^A	1^A	10^A	10	1
Impregnation time	6	1	6	1
Heat stability:				
If folds or edge tear are used	10	1	10	1
If internal tear is used	5	1	5	1
Roll width, sheet dimensions	1	2	1	2

^{A} Indicates exception to number of specimens and test called for by the test method.

TABLE 2 Sampling Plans

Lot Sample Size	Acceptance Number	Rejection Number	Lot Tolerance Percent Defective (P_t)
AQL = 2.0 % (1.5 to 2.5)^A			
15	1	2	24
20	1	2	18
30	1	2	12.5
35	2	3	14.5
40	2	3	12.8
50	2	3	10.5
AQL = 4.0 % (2.6 to 5.0)^A			
7	1	2	45
10	1	2	34
20	2	3	24
30	3	4	21
35	3	4	18
40	3	4	16
50	3	4	13
AQL = 8.0 % (5.1 to 10.0)^A			
5	1	2	58
10	2	3	45
15	2	3	32
20	3	4	30
25	3	4	25

[A] Refers to the range of AQL's covered.

10.6.5 Where the unit is defined as a skid, pallet, box, carton, case, package, bundle, or ream and contains paper in the following forms:

10.6.5.1 *Sheet Form*—Take the test unit in such a way that each test specimen is cut at random from the sheet and each sheet is taken at random throughout the unit of product in order that the test unit is representative of the unit of product (wherever applicable). Exclude the first 12 mm (½ in.) of paper from the top or bottom (or ends) from the sampling.

10.6.5.2 *Roll, Pad, or Bobbin Form*—Select the test unit at random from the rolls that make up the unit of product. (Do not include the first few turns of each roll as part of the test unit.)

11. Identification of Lot Sample Pieces

11.1 Mark each unit of product of the sample so that it can be identified at any time.

12. Lot Disposition

12.1 If the lot sample fails to meet the requirements for acceptability, the entire lot is subject to rejection.

13. Waiver of Requirements

13.1 The customer may agree to waive requirements with respect to the sampling plans, conducting of tests, applicable property specified limits, or lot rejection.

REPORTS

14. Report

14.1 At the completion of testing, report the test results of the paper properties with identifying units on a report form that includes the following:

14.1.1 Identification of the paper sampled and tested by lot number, type, grade, etc.,

14.1.2 Dates of testing,

14.1.3 Location of the testing laboratory and the person responsible for the testing,

14.1.4 Remarks indicating test method or procedure used and the deviation, if any, from the standard test procedures, and

14.1.5 Indication of the variance in test measurements such as range, standard deviation, σ, etc.

14.2 Report the test results either as calculated or observed values rounded to the nearest unit in the last right-hand place of figures used in the material specification to express the tolerances. (See the rounding Method of Practice E 29).

CONDITIONING

15. Conditioning

15.1 Condition samples in air at 50 ± 2 % relative humidity at a temperature of 23 ± 2°C (73.4 ± 3.6°F). Hold the samples in the conditioned air for not less than 4 h prior to the tests, and support them so as to allow a free circulation around each sample. (See Practice D 6054).

15.2 Make the following physical tests in the conditioned atmosphere: thickness, basis weight, tensile strength, stretch under tension, internal tearing resistance, bursting strength, folding endurance, absorption, air resistance, impregnation time, dimensions, surface friction, and edge-tearing resistance.

15.3 For work of such precision that the hysteresis in the equilibrium moisture content may lead to appreciable error, approach the moisture content equilibrium under standard conditions from a drier state, following the preconditioning provisions in Practice D 6054.

DIMENSIONS OF SHEETS, ROLLS, AND CORES

16. Scope

16.1 These test methods cover procedures for the determination of dimensions of sheets of electrical insulating paper; rolls of electrical insulating paper; and cores upon which rolls of paper are wound.

16.2 The length of any sheet is the dimension measured in the machine direction of the paper, and the width of the sheet is the dimension measured in the cross-machine direction. The thickness of any sheet is as defined in 3.1.18.

17. Summary of Test Method

17.1 *Thickness measurements*:

17.1.1 Three types of micrometers may be used for these measurements; machinist's micrometer with ratchet, deadweight dial micrometer, or motor-operated micrometer.

17.1.2 Measurements are made in prescribed manners, using the micrometer designated for a particular case. The use of a machinist's micrometer is not recommended except for screening or rough measurements to be later confirmed by the more accurate instruments designated herein.

17.2 *Sheet Length and Width, and Roll and Core Dimensions*—Steel scales, vernier calipers, or go-no-go gages are used with conventional techniques to determine the width, length, and squareness of sheets, the width and diameter of rolls, and the inside and outside diameters of cores.

18. Significance and Use

18.1 Accurate determination of thickness is important both for acceptance tests and for design purpose. The number of layers of paper required for a certain overall thickness of insulation depends on this dimension. Since apparent density is a function of weight per unit area and thickness, the latter must be known in order to calculate apparent density. Thickness enters into the calculation of dielectric strength, resistivity, and other electrical properties.

18.2 Essentially all paper is purchased with the other dimensions of the sheet or roll specified, with tolerances on these dimensions. Compliance with these requirements is usually necessary for trouble-free use of the paper in manufacturing operations.

18.3 The dimensions of rolls and of the roll cores determine the weight that must be handled, and if the roll will physically fit on the payoff stand of the equipment on which it will be further processed.

19. Apparatus

19.1 *Thickness*:

19.1.1 Determine the thickness using any one of the following apparatus:

19.1.1.1 *Method A—Machinist's Micrometer* with ratchet or equivalent, as described in the Apparatus Section of Test Methods D 374.

19.1.1.2 *Method C—Dead-Weight Dial Micrometer*, as described in the Apparatus Section of Test Methods D 374. This apparatus is not to be used for papers under 0.05 mm (0.002 in.) in nominal thickness.

19.1.1.3 *Method D—Motor-Operated Micrometer*, conforming to the following requirements. The apparatus shall be a dead-weight(not spring-) actuated, dial-type, motor-operated micrometer. It shall conform to the apparatus described in the Apparatus Section (Method B) of Test Methods D 374, except that the capacity shall exceed 0.8 mm (0.03 in.). Design the motor-operating mechanism that controls the lowering of the presser foot to ensure that the loading on the specimen created by the falling presser foot is below the loading created by a free-falling presser foot dropped from a height of 0.008 mm (0.0003 in.) above the specimen surface.

NOTE 2—For example, any free-falling body dropped from a height of 0.008 mm will attain a maximum theoretical velocity of approximately 12 mm/s (0.5 in./s). A presser foot dropping at a controlled velocity of 0.8 to 1.5 mm/s (0.03 to 0.06 in./s) will create a loading equivalent to the loading produced by a free-falling pressor foot dropped from heights of 0.000028 to 0.000119 mm (0.000001 to 0.000005 in.).

19.1.2 Calibrate micrometers in accordance with the Calibration Section of Test Methods D 374.

19.2 *Other Measurements*:

19.2.1 *Scale*—A machinist's precision steel scale of suitable length graduated to read within the accuracy specified for the sheet or roll size tolerances. A similar scale of suitable length is also required to measure diagonals of sheets.

19.2.2 *Calipers*—A machinist's vernier caliper of suitable size graduated to read within the degree of accuracy specified for the inside diameter tolerances of the core.

19.2.3 The measuring scales and calipers shall be graduated so that half of the specified tolerance can be read directly, that is, if the tolerance is 1.0 mm (or $\frac{1}{32}$in.) then the scale shall be graduated to at least 0.5 mm (or $\frac{1}{64}$ in.)

19.2.4 *Gages*—A set of two gages ("go" and "no-go") for each size core. Each gage in a set shall have a diameter within ±0.005 mm (0.0002 in.) of the specified maximum or minimum diameter.

20. Sampling

20.1 Sample in accordance with Sections 6 to 13.

21. Test Specimens

21.1 Take test specimens of sheets from the original samples, conditioned flat in accordance with Section 15.

21.2 For papers over 0.051 mm (0.002 in.) in nominal thickness use a single sheet as a test specimen for thickness measurements.

21.3 For papers 0.051 mm (0.002 in.) and under in nominal thickness, the specimen for thickness measurements may be a single sheet or a stack of ten sheets as mutually agreed upon between the purchaser and the supplier.

NOTE 3—In selecting the options given in 21.2 and 21.3, several factors are hereby given for consideration: (*1*) Greater reliability of micrometer measurements is achieved when measurements are made on stack specimens. (*2*) The thickness of a ten-sheet stack of paper does not necessarily bear a constant relationship to the thickness of a single sheet. (*3*) Variations in a single-sheet thickness are largely hidden in stack measurements. (*4*) Differences between measurements are greater on single-sheet specimens than on stack specimens.

21.4 Use a single sheet as the test specimen for length, width, and squareness of sheets.

21.5 For paper in roll form, use the entire roll as a specimen. It is not necessary to condition this specimen prior to dimension measurements.

22. Procedure

22.1 *Thickness*:

22.1.1 *Requirements Applicable to all Methods*:

22.1.1.1 The procedure for using any micrometer requires the presser foot and anvil surfaces be clean during measurements, that proper calibration operations are performed, including the construction of a calibration curve if necessary; and that dial-type micrometers be mounted on a solid level surface free of excessive vibration.

22.1.1.2 When the width of the sample permits, make all measurements with edges of the presser foot and the anvil at least 6 mm (0.25 in.) away from the edges of the specimen.

22.1.1.3 Take a specified number of measurements (mutually agreed upon between the purchaser and the supplier) at regular intervals across the entire width of each specimen, preferably in a line that is at right angles to the machine direction of the paper. In all cases make at least five such measurements. Apply the deviations for the parts of the scale corresponding to the paper thickness measured as corrections to the thickness reading.

22.1.1.4 When using multiple-sheet test specimens, do not place the presser foot closer than 20 mm (0.75 in.) from any folded edge of the stack.

22.1.2 *Method A*—Determine the thickness in accordance with the Procedure Section of Test Methods D 374.

22.1.3 *Method C*:

22.1.3.1 Place the specimen between the contact surfaces and lower the presser foot onto the specimen at a location outside of the area to be measured. This will indicate the approximate thickness so that the conditions set forth herein can be maintained.

22.1.3.2 Raise the presser foot, move the specimen to the measurement position, and lower the presser foot to 0.0075 mm (0.0003 in.) above the thickness obtained on the first determination; then let the presser foot drop.

22.1.3.3 For each succeeding measurement raise the presser foot, move the specimen to the next measurement location, and lower the presser foot to 0.0075 mm above the thickness obtained on the first determination before letting the presser foot drop.

22.1.3.4 An alternative technique is to lower the presser foot at some velocity less than 13 mm/s (0.5 in./s) onto the surface of the paper specimen.

22.1.3.5 When making thickness measurements, maintain the presser foot dead weight loading on the test specimen for at least 2 s, but not more than 4 s before taking the reading.

NOTE 4—The procedure described in 22.1.3 minimizes small errors present when the presser foot is lowered slowly onto the specimen.

NOTE 5—When measuring the thickness of noticeably compressible papers, the purchaser and the supplier may wish to fix the exact time, within the above limits, that the pressure is applied to the test specimen.

22.1.4 *Method D*—Using the motor-operated micrometer, follow the procedures described in 22.1.3. Place the specimens between the presser foot and the anvil and obtain thickness readings. When making thickness measurements, maintain the deadweight loading on the test specimen for at least 2 s but not more than 4 s before taking a reading.

NOTE 6—When measuring the thickness of noticeably compressible papers, the purchaser and the supplier may wish to fix the exact time, within the above limits, that the proper pressure is applied to the test specimen as well as the exact velocity of the fall of the presser foot.

22.2 *Length and Width of Sheets*—Measure the length and width of the specimen to the nearest appropriate unit. Make two measurements in each dimension.

22.3 *Squareness of Sheets*—Measure the lengths of both diagonals of the sheet.

22.4 *Roll Dimensions*:

22.4.1 Measure the width of the specimen to the nearest appropriate unit. Make at least two measurements.

22.4.2 Measure the outside diameter of the specimen at least two points on each end of the roll.

22.5 *Core Dimensions*:

22.5.1 Measure the inside core diameter at each end with go-no-go gages to determine whether the core meets the minimum and maximum specified diameters. Measure cores having diameters outside of the specified limits at least two points on each end with an inside feeler gage or the vernier calipers.

22.5.2 Measure the outside core diameter at least two points on each end with the vernier calipers.

23. Calculation and Report

23.1 Report in accordance with Section 14, and include the following information, as applicable:

23.1.1 *Thickness*:

23.1.1.1 Report the average, the minimum, and the maximum of the individual readings for single-sheet specimens.

23.1.1.2 For multiple-sheet test specimens, divide the micrometer readings by the number of sheets in the specimen stack and use the resulting quotient as the individual "single sheet" thickness. In all cases where multiple-sheet stacks are used report the number of sheets in the stack.

23.1.2 *Sheet Size*, reported as the average of the measurements in each dimension.

23.1.3 *Squareness*, reported as the difference in the lengths of the diagonals divided by the shorter length.

23.1.4 *Roll Dimensions*:

23.1.4.1 *Roll Width*, reported as the average of the measurements for each specimen and,

23.1.4.2 *Roll Diameter*, reported as the average of the measurements for each specimen.

23.1.5 *Core Dimensions*:

23.1.5.1 Number of cores that were within the limits of the go-no-go gages and the number that exceeded the limits of the go-no-go gages,

23.1.5.2 Measured inside diameters of cores not within the limits for inside diameter (if specified) and,

23.1.5.3 Average outside diameter of cores (if specified).

24. Precision and Bias

24.1 *Precision*—This test method has been in use for many years, but no statement for precision has been made, and no activity is planned to develop such a statement.

24.2 *Bias*—A statement of bias cannot be made because of the lack of a standard reference material.

GRAMMAGE (WEIGHT PER UNIT AREA) AND DRY COVERAGE

25. Scope

25.1 This test method covers the determination of the weight (or mass) per unit area of paper.

25.2 The weight per unit area is reported in any of several units, such as grams per square metre, pounds per square foot (or per 1000 square feet), or pounds per ream. For most paper products these are the more customary units. Test Method D 646 provides conversion factors for calculating results in these units.

26. Summary of Test Method

26.1 The area of several sheets of paper is determined from linear measurements and the mass (commonly called "weight") is determined by weighing. The grammage is calculated from the ratio of the mass to the area.

27. Significance and Use

27.1 Knowledge of the grammage is useful in the selection of materials for economical design purposes, product specification, and routine area calculations.

28. Procedure

28.1 *Grammage*—Determine the grammage in accordance with Test Method D 646, except sample the material in accordance with Sections 6 to 13 of these test methods. Report the results in grams per square metre, or as otherwise specified.

28.2 *Dry Coverage*—Proceed as in 28.1, cutting the specimen to the prescribed dimensions after conditioning in accordance with Section 15, and then drying the specimens to constant weight using the techniques described in Test Method D 644. Calculate the coverage as square metres per gram of oven-dry weight.

NOTE 7—Commercially, coverage is expressed as square inches per pound of oven-dry weight. Multiply square metres per gram of oven-dry weight by 703×10^{3} to convert to commercially used units.

APPARENT DENSITY

29. Scope

29.1 This test method covers procedures for measuring and calculating the apparent density of paper. Two test methods for calculating and reporting the density are described.

29.2 See also the procedures given in Section 163, relating density to dissipation factor and permittivity.

30. Summary of Test Method

30.1 The volume and the weight of the test specimen are determined and used to calculate the density of the specimen in grams per cubic centimetre. Either the conditioned weight or the oven-dried weight of the specimen is used, as specified.

31. Significance and Use

31.1 The apparent density of untreated paper used for electrical insulating purposes describes the weight-to-volume ratio of the paper, the weight and volume being determined according to certain prescribed conditions. A knowledge of this property is useful in the design of electrical insulating systems and in determining the economic aspects of paper use. Many physical and electrical properties of paper are related to apparent density.

32. Procedure

32.1 *Procedure A, Wet-Wet Density*—Prepare three rectangular test specimens, of such size that the width and length can be measured to within ± 1 %. Condition the specimens in accordance with Section 15. Measure the thickness, length, and width in accordance with Sections 16 to 24. Determine the weight and calculate the density. The average density of the three specimens is the test result.

NOTE 8—If the dimensions are measured in inches, multiply the calculated volume in cubic inches by 16.387 to obtain cubic centimetres.

32.2 *Procedure B, Wet-Dry Density*—The technique for obtaining wet-dry apparent density is identical with that described in 32.1, except obtain the weight of the specimens after drying to a constant weight in an oven at 105 ± 3°C as in Test Method D 644.

33. Precision and Bias

33.1 *Precision*—The precision of this test method has not been determined. However, it is dependent upon the precision with which the four separate measurements entering into the calculations are made, and upon the atmospheric conditions, particularly the relative humidity, in which the specimens are conditioned prior to test.

33.2 *Bias*—A statement of bias is not practicable because of lack of a standard reference material.

MOISTURE CONTENT

34. Scope

34.1 This test method covers two procedures for determining the mass percent of moisture in paper. The oven-drying procedure is used for most applications, and the solvent-extraction procedure is for oil-impregnated samples.

35. Summary of Test Method

35.1 *Oven-Drying Procedure*—Specimens of paper are weighed initially and after oven drying to equilibrium weight. The moisture content is calculated as a percentage of the initial weight.

35.2 *Solvent-Extraction Procedure*—Water is extracted from the specimen using an organic solvent. The water content of the solvent is then determined using the Karl Fischer titration procedure.

36. Significance and Use

36.1 A knowledge of moisture content is necessary to calculate, to a dry basis, analytical results obtained from conditioned specimens. The moisture content affects cost when purchasing papers, and is important in the design of electrical insulating systems, since it affects properties such as shrinkage characteristics. Moisture content has a significant effect on many of the physical, electrical, and thermal aging properties of insulating papers, including runability on processing equipment.

36.2 For applications involving paper in manufacturing, fabricating, and converting operations, use the oven-drying procedure (Procedure A). This includes virtually all specification and quality control, and many research situations.

36.3 For paper impregnated with insulating fluid, or subjected to a drying operation, the solvent-extraction procedure (Procedure B) may be applicable. In most cases this will be in research or trouble-shooting applications.

37. Sampling and Preparation of Test Specimens

37.1 Sample in accordance with Sections 6 to 13 of these test methods.

37.2 Use procedures for sampling and specimen preparation such that exposure of the test material to the open air, and resultant changes in moisture content, are minimized.

37.3 Prepare test specimens as specified in Test Method D 644 or Test Method D 3277, as applicable.

38. Procedures

38.1 *Procedure A*—Determine the moisture content in accordance with Test Method D 644.

38.2 *Procedure B*—Determine the moisture content in accordance with Method D 3277, using Method A or Method B of that method, as applicable.

39. Report

39.1 Report the moisture content as a percentage of the initial weight of the specimen, or, in the case of oil-impregnated materials, in accordance with Test Method D 3277.

39.2 Report in accordance with Section 14, and the report section of Test Method D 644 or Test Method D 3277, as applicable.

ASH CONTENT

40. Scope

40.1 This test method covers two procedures: *Procedure A* for the determination of the noncombustible portion of paper, usually applied to cellulosic papers which have a small amount of residue after combustion; and *Procedure B* for the determination of loss on ignition of papers having high percentages of inorganic material and which do not entirely lose their physical integrity during ignition.

41. Summary of Test Methods

41.1 *Procedure A*—The weighed, oven-dried specimen is ignited at a constant temperature in a covered crucible to constant weight. The weight of the noncombustible residue is determined and expressed as a percentage of the original oven-dried weight.

41.2 *Procedure B*—A gas burner is used to burn off volatile matter from an oven-dried specimen held in the burner flame. The weight loss during this procedure is determined and expressed as a percentage of the original weight.

42. Significance and Use

42.1 The ash determination is a relatively simple and convenient method to detect the presence of inorganic fillers, coatings, pigments, or contaminants in paper. This test method is also useful to prepare specimens for the quantitative determination of inorganic constituents in paper. This procedure is suitable for control testing, research, and referee analysis. The specified ashing temperature is selected to minimize loss of those inorganic constituents that are volatile at higher ashing temperatures.

42.2 The loss-on-ignition procedure is useful as a quality control test for papers having high levels of inorganic materials in their structures. Do not use for papers having volatile or combustible contents greater than 75 %.

43. Procedure

43.1 *Procedure A*—Determine the ash content in accordance with Test Method D 586, except use 575 ± 25°C as the ashing temperature.

43.2 *Procedure B*:

43.2.1 **Precaution**—Perform this test in an exhausted fume hood. Avoid inhaling any of the products of combustion.

43.2.2 Cut a specimen approximately 100 by 150 mm (4 by 6 in.). Oven dry the specimen to constant weight at 100 ± 5°C. Record the oven dry weight (W_{od}) to the nearest milligram.

43.2.3 Burn off the volatile matter over a Meker-type burner in such a manner as to avoid melting the inorganic fibers and thus entrapping volatiles. A properly ignited residue appears white with no trace of carbon residue.

43.2.4 After burning off the volatiles, cool the specimen and immediately weigh it to the nearest milligram. This is the ash weight (W_a).

43.2.5 Calculate the percent loss on ignition:

$$\%\ \text{loss on ignition} = (W_{od} - W_a)/W_{od} \times 100 \tag{1}$$

43.2.6 Report the loss on ignition to the nearest 0.1 %, in accordance with Section 14.

44. Precision and Bias

44.1 For the precision and bias of Procedure A, refer to Test Method D 586.

44.2 The precision of Procedure B has not been determined, and no activity is planned to determine its precision.

44.3 Procedure B has no bias because the loss on ignition is defined in terms of this test method.

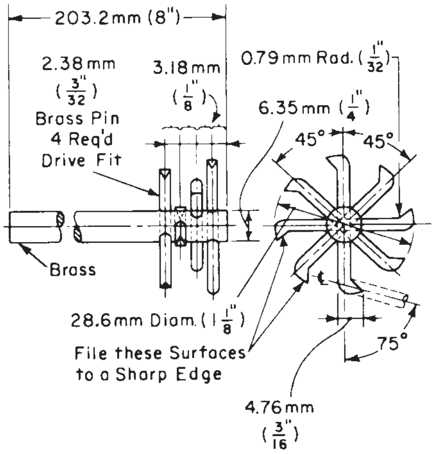

FIG. 1 Stirrer

ACIDITY-ALKALINITY-pH

45. Scope

45.1 This test method is designed to indicate the active and the total acidity or alkalinity of an aqueous extract of electrical insulating papers. Since the aqueous extracts of most untreated papers used for electrical insulation are normally unbuffered and are readily affected by atmospheric conditions, this method embodies features to minimize error from this source.

46. Summary of Test Method

46.1 This test method consists of a hot-water extraction of the specimen followed by a pH measurement or an alkalinity-acidity titration of the extract solution.

47. Significance and Use

47.1 The pH determination measures the extent to which the paper alters the hydrogen-hydroxyl ionic equilibrium of pure water. The acidity-alkalinity determination measures the quantity of extracted ionic material that contributes to that equilibrium change. Such constituents may represent potential shortcomings, either initially, or after prolonged service, of electrical equipment using this paper. These tests are useful for routine acceptance testing, research work or in the evaluation of different materials.[15]

48. Apparatus

48.1 *Bath*—A hot-water bath.

48.2 *Motor and Stirrer*—A motor with a stirrer constructed as shown in Fig. 1. Use an acid- and alkali-resistant stirrer. Chromium-plated brass is a suitable material.

48.3 *Thermometers*—Thermometers having a range from 50 to 100°C and graduated in 1° intervals.

48.4 *Buret*—A 10-mL buret graduated to 0.05 mL.

48.5 *Electric Hot Plate*.

48.6 *Suction Filtering Apparatus*.

48.7 *pH Meter*—A pH meter conforming to the requirements prescribed in Test Method E 70.

49. Reagents

49.1 *Purity of Water*—Use water in this test method that meets the requirements described in 4.2, and is free of carbon dioxide, and with a pH between 6.2 and 7.2 at 25°C.

49.2 *Buffer Solution, Standard (for standardizing the glass electrode)*—Dry about 6 g of potassium acid phthalate for not less than 2 h at 120°C. Cool in a desiccator. Add 5.0905 g of the salt to 500 mL of water at 25°C. The pH of this buffer solution is 4.0 at 25°C.

49.3 *Indicator Solution*—Add approximately 500 mg of neutral red to 300 mL of denatured ethanol. When it is thoroughly dissolved, dilute with ethanol to 500 mL in a volumetric flask. Stopper the flask and allow to age overnight at room temperature. Filter the aged liquid through a fritted glass filter using suction if necessary. Measure the pH of this solution and, if necessary, adjust to pH 7.0 by the addition of 0.10 N NaOH solution.

49.4 *Sodium Hydroxide, Standard Solution* (0.005 *N*)—Dissolve 0.2 g of sodium hydroxide (NaOH) in water and dilute to 1 L in a volumetric flask. To standardize, prepare 250 mL of a 0.005 N potassium acid phthalate solution by dissolving 0.2552 g of the dried salt in water and making up to the mark in a 250-mL volumetric flask at 20°C. (Do not dry the salt at a temperature above 125°C). Pipet 25 mL of this solution into a 250-mL flask. Add 25 mL of water. Immerse the pH electrode or if an indicator is used, add a few drops of indicator solution. Pass nitrogen through the solution for 10 min. Titrate in a closed system with the standard NaOH solution to pH 7 or to an orange shade. If preferred, the potassium acid phthalate solution may be heated to boiling and titrated immediately, taking care that the temperature does not fall below 80°C during the titration. Run three specimens in the above way at each standardization of the NaOH solution. Determine a blank on the same volume of water and indicator and deduct from the titration obtained above. Calculate the normality of the NaOH solution as follows:

$$\text{Normality of NaOH solution} = (25 \times 0.005)/\text{mL} + \text{NaOH solution required} \tag{2}$$

NOTE 9—Good laboratory practice requires use of a freshly prepared and standardized NaOH solution.

49.5 *Sulfuric Acid*, *Standard* (0.005 *N*)—Prepare 0.005 *N* sulfuric acid (H_2SO_4) and determine the alkali equivalent of the acid as follows: Transfer 10 mL of the acid to a 250-mL Erlenmeyer flask and dilute with 100 mL of water. Titrate in a closed system or at the boiling point with the standard NaOH solution as described in 49.4 for the standardization of the NaOH solution. Determine a blank on the same volume of water and indicator solution and deduct from the titration obtained above. Calculate the NaOH equivalent of the acid as follows:

$$E = A/B \tag{3}$$

where:

E = NaOH equivalent (in millilitres) to 1 mL of H_2SO_4,
A = NaOH solution required (corrected), mL, and
B = H_2SO_4 taken, mL.

50. Test Specimen

50.1 From the sample obtained in accordance with Sections 6 to 13, cut a composite test specimen, weighing at least 5 g, into small pieces approximately 0.4 in. (10 mm) square. Thoroughly mix the specimen. During preparation, avoid any contamination by handling.

51. Procedure

51.1 Place a 1-g portion of the composite specimen in a 250-mL wide-mouth Erlenmeyer flask and add 100 mL of boiling water. Clamp the flask in position in a boiling water bath so that at least one half of the flask is immersed in the water bath. During the stirring, maintain the temperature of the contents of the flask at 95°C or above. Mount the stirrer so that the blades are within 10 mm (0.4 in.) of the bottom of the flask. The assembled extraction apparatus is shown in Fig. 2. Drive the stirrer at a speed of 4000 to 5000 r/min for 5 min. At the end of this period the specimen should have been thoroughly pulped. For papers unusually difficult to pulp increase the period of stirring to 10 min.

51.2 Immediately after the specimen has been pulped, filter the contents of the flask rapidly with vacuum through a perforated porcelain disk, refiltering the first portion of the filtrate to permit the formation of a mat. Do not wash the residual pulp.

NOTE 10—It is important to accomplish filtration of the extract as promptly as possible after the disintegration. When the fibers are too short to form a satisfactory mat on the perforated porcelain disk, filter with suction through a fine quantitative filter paper that has been washed twice in a Buchner funnel with 100-mL portions of boiled water.

51.3 Immerse the electrode assembly in the hot (95 to 100°C) extract solution and determine the pH. For acidity or alkalinity determinations add the standardized (see 49.4 and 49.5) alkali or acid, depending on the level of the pH measurement. Carry out the acidity or alkalinity titration to an end point of pH 7.0 as indicated by the pH meter. If a colorimetric indicator is used for end point determination, add a few drops to the extract solution. The color will determine whether or not the extract is acid or alkaline.

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[15] See *Paper and Paperboard—Characteristics, Nomenclature, and Significance of Tests, ASTM STP 60-B*, ASTM, 1963, pp. 59–61.

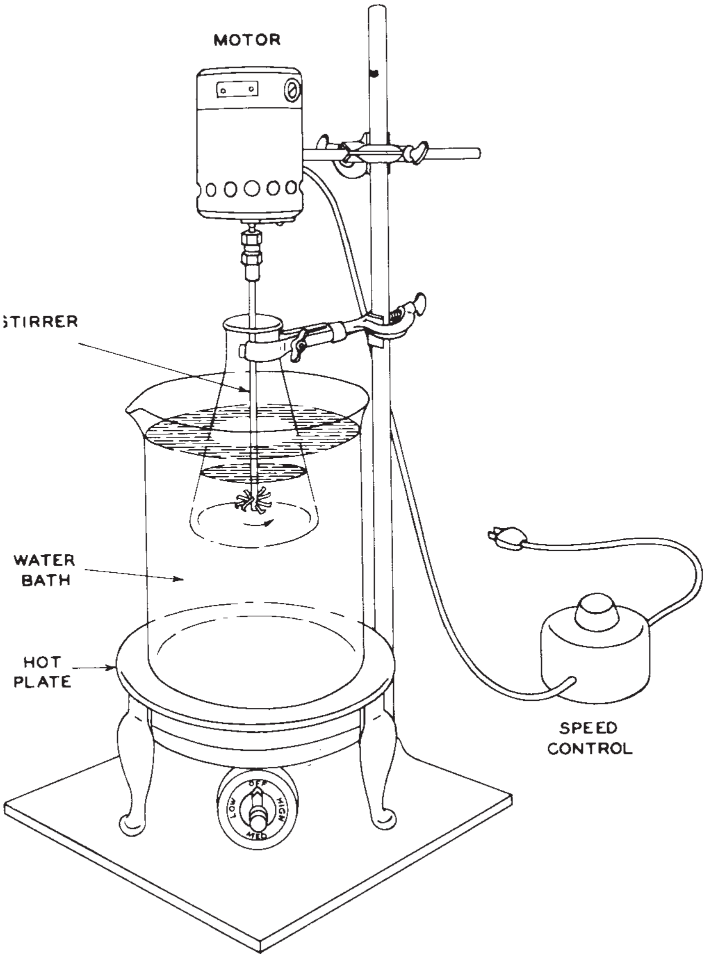

FIG. 2 Assembled Extraction Apparatus

51.4 Neutral red is a deep yellow under alkaline conditions and deep red under acid conditions. Take the end point when the solution is orange. Experience indicates that pH at this point is 6.8 to 7.2.

NOTE 11—For routine control work, 0.01 N H_2SO_4 may be used for titrations.

51.5 *Blank*—Make a blank determination in parallel with the actual determination, using a volume of water equal to that of the extract at the end point, and subjecting it to the same conditions of temperature, agitation, etc., as the extract. Use the values for the blank measurement in the calculations in 52.1.

52. Calculation

52.1 Four combinations of conditions that affect the calculations can exist. These are included in 52.1.1 through 52.1.4, with the appropriate calculation procedure combining extract and blank titration values for each:

52.1.1 *Acid Extract and Acid Blank*:

$$M = [(a - b) \times N]/W \tag{4}$$

52.1.2 *Acid Extract and Alkaline Blank*:

$$M = [(a + (d \times E)) \times N]/W \tag{5}$$

52.1.3 *Alkaline Extract and Acid Blank*:

$$M = [((c \times E) + b) \times N]/W \tag{6}$$

52.1.4 *Alkaline Extract and Alkaline Blank*:

$$M = [(c - d) \times E \times N]/W \tag{7}$$

where:
M = milliequivalents of acid or alkali per gram of specimen,
N = normality of standard NaOH solution
E = NaOH equivalent to 1 mL of H_2SO_4 (see 49.5), mL,
a = NaOH solution to titrate an acid extract, mL,
b = NaOH solution to titrate an acid blank, mL,
c = H_2SO_4 to titrate an alkaline extract, mL,
d = H_2SO_4 to titrate an alkaline blank, mL,
W = mass of paper specimen, g.

52.2 Determine the acidity or alkalinity of the extract and the blank from the reading of the pH meter, or the color of the indicator, before the titration is made, as indicated in 51.4.

52.3 It is assumed that the volume required for the titration of the blank will be less than that required for the titration of the extract. Under this assumption the calculated values for M in 52.1.1 and 52.1.2 are in milliequivalents of acid, and the values for *M* in 52.1.3 and 52.1.4 are in milliequivalents of alkali per gram of paper.

53. Report

53.1 Report in accordance with Section 14.

54. Precision and Bias

54.1 *Precision*—This test method has been in use for many years, but no statement for precision has been made, and no activity is planned to develop such a statement.

54.2 *Bias*—This procedure has no bias because the values for acidity, alkalinity, and pH are defined in terms of this test method.

AQUEOUS EXTRACT CONDUCTIVITY

55. Scope

55.1 This test method determines the electrical conductivity imparted to reagent water by boiling a specimen of paper in the water under carefully defined conditions.

56. Summary of Test Method

56.1 A specimen of paper is boiled while being agitated in reagent water and the electrical conductivity of the water is then determined. A blank determination is also made and the appropriate correction made to obtain the reported result.

57. Significance and Use

57.1 The conductivity of the water extract of insulating paper results from electrolytic impurities in the paper which may be present as ionizable acids, bases, salts, or a combination of these. These impurities are residues from the manufacturing process which have been incompletely removed. The presence of excessive amounts of electrolytic impurities is undesirable, as they tend to lower insulation resistance and have corrosion-producing tendencies under conditions of applied electrical potential. The fact that the conductivity of high-purity kraft papers may increase after manufacture, for as yet undetermined reasons, should be recognized in all comparisons of data. This test method is suitable for routine acceptance tests, control tests, and research tests.

58. Apparatus

58.1 *Conductivity Bridge*—A 60-Hz ac conductivity bridge or resistance indicator capable of measuring resistances up to 1 MΩ with an accuracy of ±5 %. Bridges operating at other frequencies, with equivalent accuracy may be used where specified.

NOTE 12—A convenient way to check the accuracy of the bridge is with precision resistors of ±1 % accuracy.

58.2 *Motor and Stirrer*—A motor with a stirrer constructed as shown in Fig. 1. Use an acid- and alkali-resistant stirrer. Chromium-plated brass is a suitable material.

58.3 *Constant-Temperature Bath*—A water bath maintained at 25 ± 0.5°C.

58.4 *Beakers*—Acid- and alkali-resistant glass 125-mL tall-form beakers, or any beakers of such dimensions that when the dip cell is immersed in 100 mL of liquid contained therein, the electrodes are fully covered.

58.5 *Flasks*—Acid- and alkali-resistant glass, wide-mouth, 250-mL Erlenmeyer flasks.

58.6 *Suction Filtering Apparatus*.

58.7 *Perforated Disk*—A perforated porcelain or fritted glass disk 50 mm in diameter with its edge beveled at an angle of 60°, and having approximately 90 perforations, each approximately 1 mm in diameter.

58.8 *Funnel*—An acid- and alkali-resistant glass funnel having a top diameter of 100 mm and made with an exact 60° angle.

58.9 *Thermometers*—One thermometer having a range from − 10 to + 110°C and graduated in 1°C intervals (for extract solution), and one thermometer having a range from − 5 to + 50°C and graduated in 0.1°C intervals (for constant-temperature bath).

58.10 *Electric Hot Plate*.

58.11 *Conductivity Cell*—Use a dip-type cell with a cell constant of 0.1 cm^{-1} with platinum electrodes securely mounted and adequately protected so that their relative positions will not be affected by handling or moderate jarring. The area of each electrode is to exceed 20 mm^2. Construct the cell so that the electrodes will be completely immersed on dipping the cell into the liquid medium. Platinize the electrodes (see 61.2) to make measurements at low frequency (60 Hz). At a frequency of 1 kHz this precaution is unnecessary.

59. Reagents

59.1 *Reagent Water*—In preparing the extract and KCl solutions, use deionized water having a conductivity not greater than 1.0 µS/cm at 25 ± 0.5°C when boiled and tested in accordance with the procedure described in Section 62 in the absence of a paper sample. Alternatively, prepare reagent water by double distillation, the second distillation being over alkaline permanganate. Use acid- and alkali-resistant glass apparatus for those distillations.

59.2 *Potassium Chloride Solution* (0.01 *M*)—Prepare a 0.01 *M* solution with reagent grade potassium chloride (KCl) which has been dried for 2 h at 110°C. After cooling, dissolve 0.7455 g of the dried salt in reagent water and make up to 1 L in a volumetric flask at 20°C.

60. Test Specimen

60.1 From the samples obtained in accordance with Sections 6 to 13, cut a composite test specimen, weighing at least 5 g into small pieces approximately 0.4 in. (10 mm) square. Thoroughly mix the specimen, and during preparation avoid any contamination by handling.

61. Preparation and Calibration of Conductivity Cell

61.1 If unplatinized, clean a new cell with warm chromic acid solution, wash thoroughly with reagent water, and rinse with alcohol and ether. If the electrodes are already platinized, omit the chromic acid wash.

61.2 To platinize the electrodes, immerse the cell in a solution of 3.0 g of chloroplatinic acid and 0.010 g of lead acetate in 100 mL of reagent water. Electrolyze, using a current density of 30 mA/cm ^{2}, for 8 min, reversing the current every 2 min. Wash the electrodes thoroughly with reagent water (see 59.1). To test for completeness of removal of electrolyte, immerse the cell in 50 mL of reagent water and measure the resistance initially and at the end of 10 min. If a decrease in resistance occurs, repeat the washing. Keep the cell immersed in reagent water when not in use.

61.3 To determine the cell constant, place a beaker containing 0.01 *M* KCl solution (see 59.2) in the constant-temperature bath maintained at 25 ± 0.5°C. After thermal equilibrium is established, measure the resistance of this solution. Calculate the cell constant, *K* as follows:

$$K = C \times R \text{ cm}^{-1} \tag{8}$$

where:

R = resistance measured, Ω, and

C = conductivity of the potassium chloride solution. The value for C, at 25°C is 1.41×10^{-3} S/cm.

62. Procedure

62.1 Place a 1-g portion of the composite specimen in the 250-mL Erlenmeyer flask and add 100 mL of boiling reagent water. Clamp the flask in position in a boiling-water bath so that at least one half of the flask is immersed in the water-bath. During the stirring, maintain the temperature of the contents of the flask at 95°C or above. Mount the stirrer so that the blades are within 10 mm (0.4 in.) of the bottom of the flask. The assembled extraction apparatus is shown in Fig. 2. Drive the stirrer at a speed of 4000 to 5000 r/min for 5 min. At the end of this period the specimen should have been thoroughly pulped. For paper unusually difficult to pulp, increase the period of stirring to 10 min.

62.2 Immediately after pulping the specimen, filter the contents of the flask rapidly with suction through the 50-mm perforated porcelain disk supported in the 100-mm glass funnel. Refilter the first portion of the filtrate after a satisfactory mat has formed on the disk. Do not wash the residual pulp.

NOTE 13—If the fibers are too short, it may be impossible to form a mat on the bare porcelain disk. In this event place a 55-mm quantitative filter paper on the disk. Before using, wash the filter paper twice with 100-mL portions of hot reagent water. A Gooch-type crucible with a fritted-glass disk may also be used if the fibers are too short.

NOTE 14—It is important to accomplish filtration of the extract as promptly as possible after disintegration.

62.3 After the filtration, dilute the extract solution to 100 mL with hot reagent water by bringing it up to the mark in a 100-mL graduated cylinder. Transfer to the tall-form beaker to make the conductance measurement. Stopper the beaker with an aluminum foil- or tin foil-covered rubber stopper and place in the water bath maintained at 25 ± 0.5°C.

62.4 As soon as thermal equilibrium is established, place the dip cell in the extract solution, making certain that the electrodes are completely immersed. Measure the resistance on the most sensitive scale of the bridge. Move the cell up and down in the solution several times and repeat the measurement until successive readings are constant.

62.5 Before each measurement rinse the cell thoroughly in reagent water and gently shake off any water clinging to the surfaces.

62.6 *Blank*—Correct the conductivity of the extract solutions for the blank error. Determine this correction by running a blank in parallel with the actual determination, using the same volume of reagent water.

62.7 Test at least two specimens. If the conductivities on duplicate specimens do not agree within 10 %, repeat the determination.

62.8 For referee purposes, condition the specimen and weigh at the standard test conditions specified in Section 15.

63. Calculation and Report

63.1 *Calculation*—Calculate the conductivity of the extract solution (based on the weight of 1 g of the air-dry sample) as follows:

$$\text{Conductivity} = [(K/R_2) - (K/R_3)] \times 10^6 \ \mu\text{S/cm} \tag{9}$$

where:

K = cell constant ($C \times R$), cm^{-1},

R_2 = resistance of extract solution at 25 ± 0.5°C, Ω, and

R_3 = resistance of water blank at 25 ± 0.5°C, Ω.

63.2 *Report*—Report in accordance with Section 14.

64. Precision and Bias

64.1 This test method has been in use for many years, but no statement for precision has been made and no activity is planned to develop such a statement.

64.2 This procedure has no bias because the value for aqueous extract conductivity is defined in terms of this method.

SOLVENT-SOLUBLE MATTER

65. Scope

65.1 This test method covers a procedure for determining the weight percentage of material removable from a specimen of paper, using a solvent-extraction method.

66. Summary of Test Method

66.1 A Soxhlet extraction apparatus is used, with appropriate volatile solvent to extract soluble material from the specimen. After extraction, the solvent is evaporated, and the nonvolatile residue is weighed and calculated as a percentage of the original weight of the specimen.

67. Significance and Use

67.1 Solvent-extractable materials in electrical insulating paper include various contaminants which may be present in the raw material. If present in sufficient quantity, these materials may lower the quality of the insulation or have deleterious effects on the electrical characteristics of the liquid compounds used in contact with the paper in various types of electrical apparatus. Ethanol-soluble materials in capacitor paper are found to increase the electrical conductivity of some dielectric fluids which are used as impregnants in capacitors.

67.2 This test method, with a specified solvent, may be used for routine acceptance and for research tests.

68. Apparatus

68.1 A medium-size glass Soxhlet extraction apparatus provided with a siphon chamber approximately 35 mm diameter and 90 mm high. Alternatively, use a modified extraction apparatus of similar size equipped with a siphon cup to hold the thimble.

68.2 *Thimble*, Alundum or paper, or some other inert device to prevent fiber being carried over into the flask.

68.3 *Heating Apparatus*—Steam bath or variable temperature hot plates.

68.4 *Evaporation Facilities*.

69. Reagent

69.1 *Solvent*, of specified composition.

70. Test Specimen

70.1 From the sample obtained in accordance with Sections 6 to 13, cut a composite test specimen, weighing at least 25 g, into small pieces approximately 10 mm (0.4 in.) square, and mix thoroughly. Determine the moisture content in accordance with 38.1 on a separate portion of the composite specimen in moisture equilibrium with the portion for analysis.

71. Procedure

71.1 **Warning:** The solvents used are likely to be flammable and may be physiologically hazardous. Take appropriate precautions to prevent ignition and to reduce exposure to the liquids and their vapors to below the maximum safe levels.

71.2 Heat the flask to constant weight in an oven at 105 to 110°C, allow to cool in a desiccator, and weigh to the nearest 1 mg.

71.3 Place not less than 5 g of the specimen (weighed to the nearest 0.01 g) in the fiber-retaining device (thimble), that was previously extracted with the solvent being used or tested to show there is no contribution to the test from this source.

71.4 Place the thimble in the extraction apparatus and add sufficient solvent so that a safe excess will remain in the bottom of the flask when the siphon cup is full.

71.5 Place the assembled apparatus on the hot plate with the heat adjusted so that siphoning occurs no more than once every 6 min. At the end of the extraction, at least 60 times unless otherwise specified, (Note 15), pour the solvent from the siphon cup into the flask.

NOTE 15—When extracting with a solvent with a large heat of vaporization, such as water, the heating should be adjusted so that siphoning occurs at least once every 10 to 12 min. In this case end the extraction at 36 siphonings. For the "medium" Soxhlet apparatus to siphon properly with water, it may be necessary to have the siphon tube replaced with one of larger internal diameter. Alternatively, the "large" (123 by 43-mm thimble) size of Soxhlet extraction apparatus usually is satisfactory.

71.6 Dry the previously tared flask on the heating apparatus, using solvent recovery if desired, and then dry to constant weight in an oven at 105 to 110°C. Allow to cool in a desiccator, and weigh to the nearest 1 mg. (Note that static may be a problem in these weighings, and take steps to ensure the correct weight is obtained.)

71.7 Alternatively, evaporate the solvent from a tared evaporating vessel, taking care to rinse the flask into the dish; in which case, taring of the flask as specified in 71.2 is not required.

71.8 Test at least two specimens.

72. Calculation and Report

72.1 *Calculation*:

72.1.1 Calculate the solvent-extractable content on the basis of the oven dry weight of the specimen as follows:

$$A = 100R/[W(1.0 - 0.01\,M)]\% \tag{10}$$

where:

A = solvent extracted material, %,
R = weight of residue, g,
W = original weight of specimen, g, and
M = moisture in the specimen, %.

72.1.2 The test result is the average of the values calculated in 72.1.1 for all specimens.

72.2 *Report* in accordance with Section 14, and include the following information:

72.2.1 Previous treatment the sample may have had, for example, previous extraction with another solvent,

72.2.2 Solvent used, and

72.2.3 Time of extraction and approximate number of siphonings.

73. Precision and Bias

73.1 This test method has been in use for many years, but no statement for precision has been made and no activity is planned to develop such a statement.

73.2 This procedure has no bias because the value of solvent-soluble matter is defined in terms of this test method.

FIBER ANALYSIS

74. Summary of Test Method

74.1 A fiber analysis consists of identifications of the kinds and types of fiber in a paper and determination of the proportions in which they are present.

74.2 The results are reported as percentages by weight of the total fiber composition to the nearest multiple of 5. When any fiber is found present in amounts less than 2½ % it is reported as a "trace."

75. Significance and Use

75.1 Fiber analysis is useful as a specification and a control test. It may be used in referee or research testing to determine conformance to specification or purchase requirements concerning fiber composition. For accurate results the analyst needs considerable training and experience; needs to make frequent use of standard papers of known composition; needs to make frequent use of known samples of fiber; and to be thoroughly familiar with the reactions of different fibers to exposure to various stains.

76. Procedure

76.1 Determine the fiber analysis in accordance with Test Method D 1030, except sample the material in accordance with Sections 6 to 13 of these test methods.

77. Report

77.1 Report in accordance with Section 14 and with Test Method D 1030.

ABSORPTION (Rise of Water)

78. Significance and Use

78.1 The rise of water, which has a fixed surface tension, reflects a combination of conditions within the paper, including fiber arrangement, fiber size, spacing between fibers, specific surface area of the fibers, and the presence, if any, of chemical treatment of the paper fibers during or subsequent to manufacture of the paper.

78.2 This test method is useful for control purposes as one criterion of uniformity.

78.3 With caution, this test may be used as an indicator of impregnation rate.

79. Apparatus (see Fig. 3)

79.1 *Container*.

79.2 *Support* for test specimens and scale.

79.3 *Linear scale*.

80. Test Specimens

80.1 Cut ten test specimens, 25 mm (1 in.) in width and at least 125 mm (5 in.) in length, from the samples obtained in accordance with Sections 6 to 13, five being cut parallel with the machine direction of the paper and five cut parallel with cross-machine direction of the paper. Condition the specimens as prescribed in Section 15.

81. Procedure

81.1 Suspend the specimens vertically, with one end dipping 3 mm (⅛ in.) in reagent water at room temperature, and after 5 min note the rise of the water in the specimen above the level of the water in the container. Measure the rise by reading the height of absorption directly from the scale. Record the height of absorption to the nearest 3 mm for each direction for each specimen, and calculate the averages as the results.

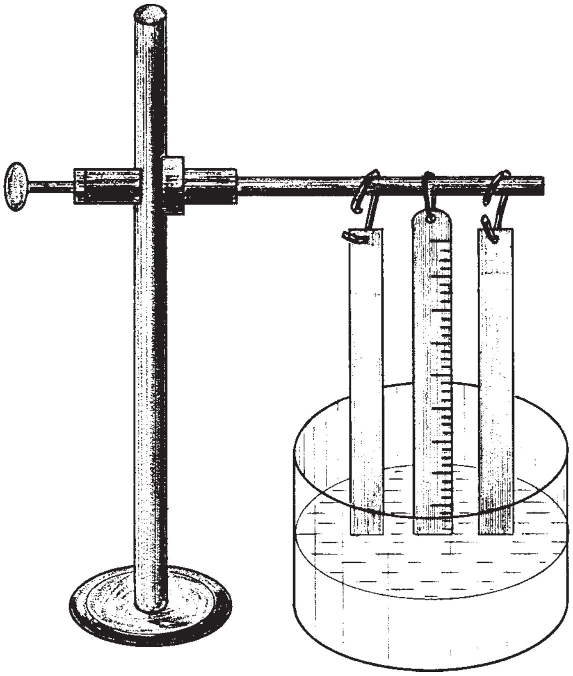

FIG. 3 Apparatus for Absorption Test

82. Report

82.1 Report in accordance with Section 14.

83. Precision and Bias

83.1 This test method has been in use for many years, but no statement for precision has been made and no activity is planned to develop such a statement.

83.2 This test has no bias because the value for absorption is defined in terms of this test method.

IMPREGNATION TIME

84. Scope

84.1 This test method measures the time required for castor oil, or another specified non-aqueous liquid, to penetrate through the thickness of a paper, under specified conditions.

85. Summary of Test Method

85.1 A specimen of paper is clamped over an opening in the top of a container filled with a designated liquid. The container is then tilted so that the liquid is in contact with the bottom surface of the paper specimen. The time required for the liquid to penetrate through the paper is measured, and recorded as the impregnation time. The endpoint is determined by visual observation.

86. Significance and Use

86.1 Impregnation time of a paper, using a standard liquid, may be used to predict the rapidity and degree of impregnation that may be obtained in commercial impregnation of this paper with suitable liquids.

86.2 Where castor oil is used as the testing liquid, the impregnation time has been shown to correlate with the rate of commercial impregnation with phenolic resin varnishes. For phenolic laminates, the impregnation time of the base paper is an important factor affecting the strength, moisture-resistance, and electrical properties of the finished laminated product.

86.3 This test method is also used in selection of cable paper; for such use it is usually desirable to use the commercial impregnant in the instrument.

87. Apparatus

87.1 *Penetration Tester*—A Williams standard tilting-type penetration tester with orifice 60 mm (2.375 in.) in diameter as shown in Fig. 4, or equivalent penetrometer, equipped with a stop clock graduated in seconds.

87.2 *Thermometer*—A thermometer of suitable range.

87.3 *Test Liquid*—Double-pressed castor oil having a viscosity of 700 ± 30 cP (0.7 ± 0.03 Pa·s) at 25°C (77°F), or mineral oils of the same viscosity and comparable wetting characteristics are most commonly used as the test liquid. However, other liquids which are more specific to the intended

FIG. 4 Penetration Tester

application for the paper may be used, if specified. The liquid used must be defined in the test report.

88. Test Specimens

88.1 From samples obtained in accordance with Sections 5 to 13, cut six test specimens 75 mm (3 in.) square. Condition the specimens as prescribed in Section 15.

89. Procedure

89.1 Measure the test specimens for thickness in accordance with Sections 16 to 3.

89.2 Number the test specimens consecutively on the same side of the paper. Test the odd-numbered specimens with their numbered sides up, that is, not in contact with the impregnating liquid. Test the even-numbered specimens with their numbered sides down, that is, in contact with the impregnating liquid.

89.3 Place the liquid container in a horizontal position and fix the thermometer so that its bulb is immersed in the test liquid. Fill the container with the impregnating liquid to within 6 mm (¼ in.) of the upper edge of the container orifice. Maintain the liquid at this level by adding small amounts of the impregnating liquid to replace that absorbed by the test specimens during the testing period. Maintain the temperature of the liquid at 25 ± 1.1°C (77 ± 2°F) during testing by means of the electrical heater and the thermostat in the base of the container.

89.4 Place the specimen over the container orifice and clamp in place with the ring clamp. After setting the stop clock at zero, depress the container handle quickly until the front end of the container touches the base plate and start the clock. Observe the surface of the paper closely and at the instant of complete penetration of the liquid throughout the entire paper area under test, stop the clock by immediately raising the front end of the liquid container.

NOTE 16—It has been found that, for some liquids, particularly mineral oils, the use of ultraviolet light is helpful in determining the "instant of complete penetration of the liquid throughout the entire paper area under test".

89.5 Record the elapsed time as the time of impregnation.

89.6 The average value for the six specimens is the test result.

90. Report

90.1 Report in accordance with Section 14, and include the following information:

90.1.1 Average impregnation time,

90.1.2 Minimum and maximum impregnation times, or other indication of variance,

90.1.3 Description of the test liquid, and

90.1.4 Average, maximum, and minimum thickness before impregnation and the thicknesses of the specimens showing the lowest and highest impregnation time.

91. Precision and Bias

91.1 This test method has been in use for many years, but no statement for precision has been made and no activity is planned to develop such a statement.

91.2 This procedure has no bias because the value of impregnation time is defined in terms of this method.

SURFACE FRICTION

92. Significance and Use

92.1 The coefficient of dynamic friction of paper is a factor in the satisfactory application of paper tapes used to insulate conductors in electrical apparatus.

92.2 The sliding of one surface of paper over another paper surface has been found to correlate with the frictional forces that exist when paper surfaces slide over smooth metal surfaces such as are encountered in high-speed tape winding equipment.

92.3 Paper-taped conductors may be bent during assembly of electrical apparatus and it is desirable that the paper slide upon itself when such bending is encountered.

93. Apparatus

93.1 *Inclined Plane* (See Fig. 5)—constructed with a rigid smooth-surfaced flat material such as wood, plywood, or decorative thermoset laminate, with the dimensions approximately as shown in Fig. 5.

93.2 Mount the plane on a horizontal table.

93.3 Equip the plane with a pointer to indicate the markings of the angle between the level table top and the inclined plane. Mark the scale to read angles to the nearest ¼° with a maximum angle of 30°.

93.4 Equip the plane with a mechanical linkage of gears and offset wheel or rack and pinion to raise the plane very slowly and smoothly during tests.

93.5 Make provisions on the plane to secure temporarily the 200 mm (8 in.) wide test specimen to the plane surface. Bars and wedges are useful for meeting this requirement.

93.6 Use an attached or portable leveling device to indicate levelness of the plane in both dimensions at zero elevation.

93.7 *Sliding Block*—The sliding block (Fig. 6) of hardwood is 25 mm (1 in.) thick, 63 mm (2½ in.) wide, and 75 mm (3 in.) long with a smooth flat bearing surface and rounded edge and with provisions for securing the ends of the paper specimen when it is wrapped around the flat sliding area. The block weighs 235 ± 10 g.

94. Test Specimens

94.1 Samples obtained in accordance with Sections 6 to 13 must have particular attention paid to them. Take them from freshly exposed layers of paper and handle them so that they do not rub against one another or other objects, or become wrinkled or touched in any part of the area to be submitted to the friction test.

94.2 Condition all samples in accordance with Section 15.

94.3 Determine the wire side in accordance with TAPPI T455, and the machine direction in accordance with Test Method D 528. Mark an arrow on one side of each specimen to identify the wire side and the machine direction.

94.4 For each test cut two test specimens 63 mm (2½ in.) wide by 170 mm (7 in.) in the machine direction for use on the sliding block. Cut one specimen 200 mm (8 in.) wide by 250 mm (10 in.) in the machine direction for use on the plane. If it is specified that tests be made in the cross-machine direction change the direction of cutting appropriately.

94.5 The purchase order or specification for the material should indicate whether the test is to be made on the felt or wire side.

95. Procedure

95.1 Apply the test specimens to the inclined plane and sliding block. It is important that the contact surfaces of the paper specimens attached to the apparatus be smooth and free of wrinkles.

95.2 Gently place the block on the right back corner of the level plane, in position to slide. Place the block on the plane so that the rounded edge is on the downhill side of the incline.

95.3 By preliminary tests, determine approximately the expected friction angle. Slowly raise the plane from 2° below that setting and at the same time gently tap the back edge of the block. A pencil is a convenient tapping instrument.

95.4 The tapping should advance the block 3 to 6 mm (⅛ to ¼ in.) down the plane for each tap and be of a frequency that at least one tap is registered for each ½° of rise of the adjustable inclined plane. The intensity of the taps to produce the desired motion of the sliding block will decrease as the sliding block slides down the plane. Record the angle at which the block slides down the plane without further tapping as the preliminary test.

95.5 Take the second 63 by 170 mm specimen and attach it to the block. Place the block on the front half of the strip on the inclined plane and repeat the procedure described in 95.3 and 95.4, except raise the inclined plane to an angle 1° less than the angle obtained in the preliminary test. The angle at which the

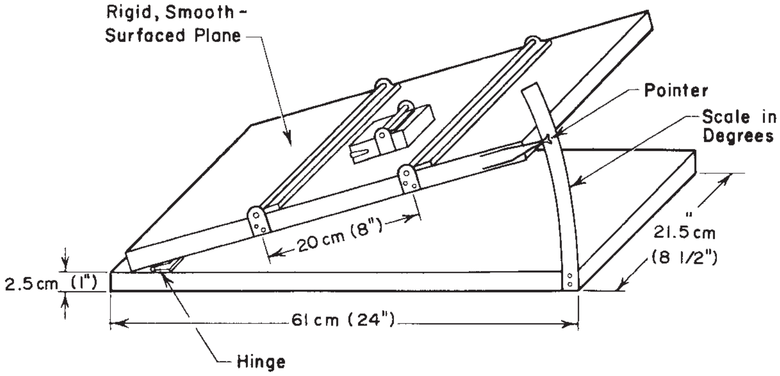

FIG. 5 Included Plane for Surface Friction Test

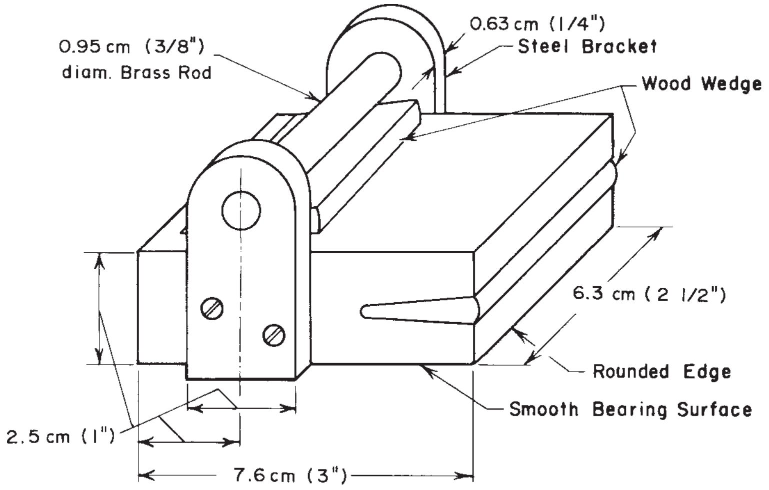

FIG. 6 Sliding Block for Surface Friction Test

block slides down the plane without further tapping is the final surface friction angle of the specimen. Record this angle to the nearest ¼°.

95.6 Make three tests from each sample, unless the material specification requires a different number. The average value of all tests is the test result.

96. Report

96.1 Report in accordance with Section 14. If desired, the test results may be reported as dynamic coefficient of friction by determining the tangent of the surface friction angle.

97. Precision and Bias

97.1 This test method has been in use for many years, but no statement for precision has been made, and no activity is planned to develop such a statement.

97.2 This test method has no bias because the value for surface friction is defined in terms of this method.

AIR RESISTANCE

98. Scope

98.1 These test methods provide three procedures for the determination of the air resistance of paper. While they are similar in principle, the procedures are intended for different ranges of porosities, and the results obtained using one method cannot be compared directly with those obtained by another test method.

99. Significance and Use

99.1 Air resistance is related to dielectric strength and absorbancy, and thus may be used as a criterion in specifications for insulating papers, while recognizing that the values obtained may vary over a considerable range even on paper of quite uniform formation.

99.2 Air resistance reflects the internal structure and surface condition of the paper: differences in the types of fibers, in their degree of hydration, length, orientation, and compaction, in the amount or nature of noncellulosic additives, or in surface treatment.

100. Procedure

100.1 Determine air resistance in accordance with Method A, B, or C of Test Methods D 726, except sample the material in accordance with Sections 6 to 13 herein.

100.2 Select the test method most suitable for use on the paper being tested, as discussed in Test Methods D 726. It is recommended that the test method to be used be given in the specification for the paper being tested.

101. Report

101.1 Report in accordance with Section 14, and also include the following information:

101.1.1 A statement as to which test method was used, and

101.1.2 Information specified in the pertinent report section of Test Methods D 726.

BURSTING STRENGTH

102. Scope

102.1 This test method describes two procedures for measuring the bursting strength of paper. One procedure applies to papers bursting up to 1.4 MPa (200 psi) and the second procedure applies to papers bursting at values above this level.

103. Summary of Test Method

103.1 A conditioned specimen of paper is clamped between two flat surfaces that have circular openings of a specified diameter and edge shape. A rubber diaphragm is pressed against one surface of the specimen with steadily-increasing hydraulic pressure until the specimen ruptures. The hydraulic pressure at the moment of rupture is the test value.

104. Significance and Use

104.1 Bursting strength has considerable use as a mill control test and, to some extent, as a purchase specification requirement.

104.2 It is not easy to identify applications for electrical insulating paper in which forces are normally encountered that duplicate the forces exerted in this test method. For this reason there is controversy among experts regarding the significance of this test when applied to electrical insulating papers.

105. Test Specimen

105.1 Prepare specimens from the sample obtained in accordance with Sections 6 to 13, and conditioned in accordance with Section 15.

105.2 For each test, allow at least a 100 mm (4 in.) square area.

105.3 If practicable, cut sufficient material to permit ten bursting tests on a straight line across the sheet or roll.

106. Procedure

106.1 *Procedure A* (for papers bursting up to 1.4 MPa (200 psi)—Determine the bursting strength in accordance with Test Method D 774, except sample and condition the material in accordance with Sections 6 to 13 of these test methods.

106.2 *Procedure B* (for papers bursting at more than 1.4 MPa);

106.2.1 Apparatus for Procedure B is a testing machine conforming to the following requirements:

106.2.1.1 *Clamps*, similar in all respects to those used on the machine specified in Test Method D 774.

106.2.1.2 *Rubber Diaphragm* made of pure gum rubber, free of mineral loading material, and conforming to dimensions of Fig. 7.

106.2.1.3 *Motor*—The hydrostatic pressure applied to the underside of the rubber diaphragm until the test specimen bursts shall be generated by a motor-driven piston forcing a liquid (usually glycerin or hydraulic brake fluid) into the pressure chamber of the apparatus at the rate of 170 ± 10 mL/min (10.4 ± 0.6 in.^{3}/min).

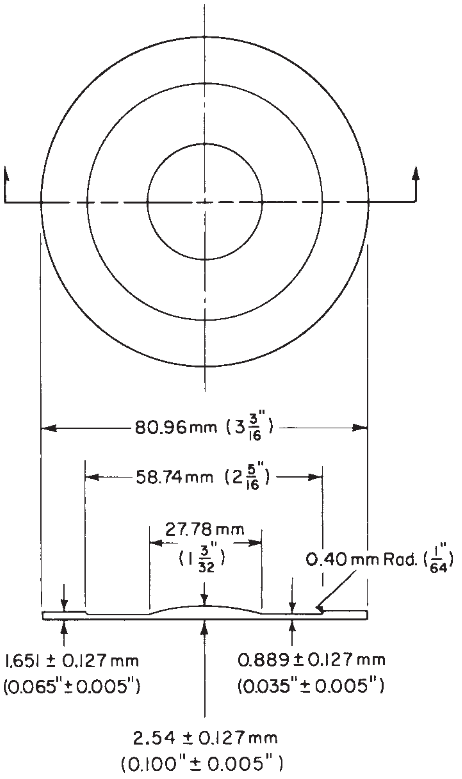

FIG. 7 Rubber Diaphragm for Bursting Strength

106.2.1.4 *Pressure Reading Gages*—The pressure reading gages shall indicate the bursting strength in megapascals (or pounds force per square inch) with the following minimum accuracy:

Pressure		Accuracy	
MPa	psi	MPa	psi
0 to 2.1	0 to 300	0.02	3.0
0 to 4.1	0 to 600	0.04	6.0
0 to 6.9	0 to 1000	0.07	10.0
Above 6.9	Above 1000	0.14	20.0

106.2.2 *Procedure*—Make ten bursts, as in 106.1, using apparatus as described for Procedure A or Procedure B, as applicable, five with one side of the specimen uppermost and five with the other side uppermost. Operate the testing machine at a uniform speed until the specimen bursts.

107. Report

107.1 Report in accordance with Section 14.

FOLDING ENDURANCE

108. Significance and Use

108.1 Folding endurance of paper is a measure of its toughness and brittleness, that is, the tendency for the fibrous structure to pull apart or for the fibers themselves to break in two. Since folding endurance is very sensitive to changes in several of the other physical properties of paper, it is a very useful criterion for determining the extent of changes such as occur in the heat stability test.

108.2 Either of the test methods specified in Section 109 may be used for the determination of the effect of heat aging (Sections 131 to 137), since relative values only are involved. For specification purposes, however, the use of Test Method D 2176 is strongly recommended.

109. Procedure

109.1 Sample in accordance with Sections 6 to 13 and condition in accordance with Section 15 herein.

109.2 Determine the folding endurance in accordance with Test Method D 2176, which is the preferred method; or TAPPI T 423. If TAPPI T 423 is used, it must be so stated in the report.

NOTE 17—The test results obtained using Test Method D 2176 will in general differ greatly from those using TAPPI 423. The difference between the two values will depend upon the applied tensile forces, the grade of paper being tested, and other factors, so that no direct comparison can be made between papers tested by different methods.

110. Report

110.1 Report in accordance with Section 14, and the Report section of Method D 2176 or TAPPI T 423, as applicable, and identify the procedure used.

TENSILE PROPERTIES

111. Scope

111.1 This test method covers the determination of three tensile breaking properties of paper, namely; the energy absorbed per unit of surface area (tensile energy absorption), the force per unit cross-sectional area or per unit width required to break a specimen (tensile strength), and the percentage elongation at break.

111.2 This test method is applicable to all types of paper whose properties are within the working range of the instrument used.

112. Summary of Test Method

112.1 Conditioned test specimens having smooth, straight parallel edges are subjected to tensile stress, uniformly across the width of the specimen, under specified conditions of loading.

112.2 Depending upon which properties are of interest, the stress (load) at rupture, the strain (elongation) at break, or the area (in units of work) under the stress-strain curve are measured for use in calculating the specified properties.

113. Significance and Use

113.1 Tensile strength is an indication of the suitability of papers for applications where, during manufacture or in service, direct tensile stresses are applied to tapes or sheets of the paper.

113.2 The elongation at break is useful in predicting the performance of a paper when applied by machines in the manufacture of coils, capacitors, insulated conductors, etc. Such machinery may subject paper to severe tensile stresses, and the elongation at rupture has been found to correlate with runnability on such machinery. This property is also valuable in evaluating a paper that is required to conform closely to an irregular surface. In the case of creped papers, it is frequently the property of primary interest.

113.3 The tensile energy absorption (TEA) measures the ability of the paper to withstand shock or impact. It is indicative of the suitability of the paper for use on machines where it may be subjected to tensile impact, and for applications where such forces are applied in service.

113.4 All three tensile properties are influenced by:

113.4.1 The kind, quality, and treatment of the paper fibers,

113.4.2 The kind and amount of non-fibrous constituents of the paper,

113.4.3 The formation of the sheet on the paper-making machine,

113.4.4 Subsequent drying, finishing and treatment of the paper,

113.4.5 Exposure of the paper to severe environments, including high temperature, high humidity, or other degrading factors, and

113.4.6 Moisture content at time of testing.

113.5 Values for all of the tensile properties are useful for material specifications, for process control, and for research.

114. Apparatus

114.1 *Tensile Testing Machine*, having the following characteristics:

114.1.1 Capable of applying a tensile load in a predetermined manner, smoothly and reproducibly, using suitable jaws (see 114.2) to clamp the test specimen.

114.1.2 Means for indicating the applied load at break with an accuracy of ±1 % of the load at break, at the loading rate used for the test,

114.1.3 Means for determining elongation of the test specimen at the time of break to an accuracy of ±0.05 % of the initial test span, and

114.1.4 Means for recording the applied load and the elongation of the test specimen, without loss of accuracy, continuously from initial application of load until the time of failure.

NOTE 18—The recorder specified in 114.1.4 may not be needed if TEA is not being measured. Also if only tensile strength is of interest, the load at break will be the only reading of significance.

NOTE 19—Tensile testing machines of both the constant-rate-of-extension (CRE) and pendulum type are commonly used for measuring tensile properties of paper. CRE machines are almost universally used for determining TEA. Results obtained on one type of machine may not conform closely to results obtained on duplicate specimens tested on a machine of the other type.

114.2 *Clamping Jaws*, for gripping the ends of the test specimen.

114.2.1 The clamping surfaces shall be in the same plane, parallel to the direction of motion of the moving jaw. The jaws shall be at least as wide as the test specimen, with the edges of the clamping surfaces perpendicular to the edges of the test specimen.

114.2.2 Creped papers, which tend to slip in the jaws of conventional flat-faced clamps, may be tested using line clamps of the type shown in Fig. 8, whereby the specimen is clamped between a sharp ridge on one jaw and a reinforced rubber mat on the other. Use a cloth-inserted rubber sheet

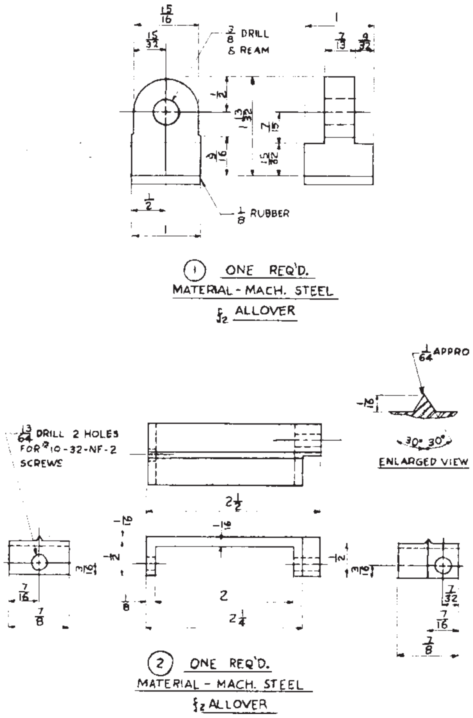

FIG. 8 Modified Line Clamp Jaws for Scott A-1 Clamp

packing 3 mm (⅛in.) thick[16] for covering the flat jaw. When using jaws of this type, the initial separation is the distance between the ridges on the jaws at the time tension is first applied.

114.3 *Planimeter*, for determination of the area under the load-elongation curve, with an accuracy of ±1 %.

114.3.1 Alternatively, the recorder may be equipped with an automatic integrator having the same accuracy.

114.3.2 If neither a planimeter nor an integrator is available, the area under the curve may be determined by cutting the recorder paper along the curve, the elongation axis, and the line representing the elongation at rupture. The weight of this piece of paper and the weight of a rectangular section from the same piece of paper, and having an area of at least two-thirds that of the area under the curve, may then be determined on an analytical balance, and the area under the curve calculated.

114.4 *Specimen Cutter*, for preparing specimens meeting the requirements of 115.3.

114.5 *Scale and Magnifier, or Optical Comparator*, accurate to the nearest 0.1 mm (0.004 in.), for measuring the width of specimens.

115. Test Specimens

115.1 Prepare test specimens from samples taken in accordance with Sections 6 to 13, and conditioned in accordance with Section 15. Store and test prepared specimens in a standard laboratory atmosphere as specified in Section 15.

115.2 Prepare sufficient specimens to obtain ten valid tests in each of the principal directions of interest. Because of the possibility of some test values being rejected for reasons as given in 117.6, it is advisable to prepare a few extra specimens.

115.3 Cut the test specimens with clean straight parallel edges to a width of within 0.1 mm (0.004 in.) of the specified width, and of a length sufficient to permit clamping the specimens in the jaws at the specified initial span, without the necessity for touching the portion of the specimen that will be between the jaws. If the width of the specimens cannot be held within 0.1 mm of the specified width, measure and record the width of each specimen to an accuracy of ±0.1 mm. In all cases, the edges of the specimens must be straight and parallel with the variation in width of any specimen not exceeding 0.1 mm. The specimen width must in any case be within 1.0 mm (0.040 in.) of the specified width.

115.4 The recommended specimen widths and the initial test spans are as follows:

Condition	Width	Span
Measurement of TEA, and simultaneous determination of tensile strength and elongation at break.	25 mm (1 in.)	200 mm (8 in.)
Measurement of tensile strength and elongation at break on uncreped papers	15 mm (0.6 in.)	180 mm (7 in.)
Measurement of tensile strength and elongation at break on creped papers.	25 mm (1 in.)	See Note 20

NOTE 20—For creped papers, the most suitable test span is a function of the expected elongation at break, and the test span to be used should be specified in the document calling for this test method, or otherwise agreed upon between all parties concerned with the tests. Suggested test spans are:

Expected Elongation, %	Test Span, mm (in.)
0 to 50	180 or 200 (7 or 8)
51 to 100	100 (4)
101 to 200	50 (2)
201 to 400	25 (1)

Some pendulum-type testers may not have sufficient travel to permit testing with specimens of these lengths. If such instruments are to be used, it may be necessary to agree to use shorter test spans for some grades of paper.

115.5 In all cases the specimens shall be between 13 mm (0.5 in.) and 50 mm (2.0 in.) in width.

NOTE 21—In general, varying the width of the test specimen within this range has little effect on the test results for the papers normally used for electrical insulation. However, this assumption should be verified by actual tests on the papers under consideration before placing reliance on it.

115.6 Cut the test specimens from areas of the sample which are free of abnormalities, creases, or wrinkles, unless such defects are typical of the material being tested, or unless the purpose of the test is to determine the effect of such abnormalities.

116. Calibration

116.1 Calibrate the testing machine in accordance with the manufacturer's instructions and with Specification D 76 and Practices E 4.

116.2 Calibrate the planimeter or other area measuring equipment in accordance with the manufacturer's instructions.

116.3 Calibrate the scale or comparator in accordance with Practice D 2865.

117. Procedure

117.1 Determine the width of the specimens to within 0.1 mm (0.004 in.). Discard specimens whose width is not uniform within 0.1 mm over their entire length. While making this measurement and in all other steps until the specimen is broken, take care to avoid touching the test specimen with the fingers in the area which will be between the jaws, or otherwise damaging or contaminating the specimen in the test area.

117.2 If the tensile strength is to be reported in stress per unit of cross-sectional area, determine the thickness of each specimen to within 0.01 mm (0.0005 in.).

NOTE 22—Ordinarily, the tensile strength is expressed in stress per unit width for papers up to 0.25 mm (0.010 in.) or even greater in thickness. For thinner papers, a decision to require determination and reporting of tensile strength in units of force per unit cross-sectional area should be made only after considering the effect that errors in measuring thickness will have on the accuracy of the calculated test results.

117.3 Set the span between the jaws to the proper distance, as given in 115.4, or as may be otherwise specified for the material being tested. The test span shall be within 5 % of the specified distance and shall not vary over a range of more than 1 % of the distance specified during the testing of any group of test specimens. The actual span shall be known with an accuracy of ±0.5 % if TEA or elongation at break is to be determined.

117.4 Align a test specimen in the two sets of jaws and clamp it in the upper set of jaws.

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[16] Raybestos-Manhattan 645 Rubber Diaphragm Sheets have been found satisfactory for this method.

117.4.1 Remove slack from the specimen and align it by putting a small tension on it while clamping it in the lower set of jaws.

117.4.2 Use a clamping pressure sufficient to prevent slippage during test without damaging the specimen.

NOTE 23—Except for a referee test, time may be saved, when testing thinner papers, by aligning and clamping the ends of the ten specimens together in the upper jaws, and breaking them one at a time, after clamping the lower end of each in the lower jaws.

NOTE 24—Some test machines operate with the lengthwise center line of the test specimen in a horizontal plane rather than in a vertical plane. For these machines, the phrases "upper jaws" and "lower jaws" in the preceding and subsequent sections shall be appropriately modified to accommodate the peculiarities of these machines.

117.5 Operate the test machine to apply stress to the specimen and break it.

117.5.1 Except as noted below or in the specification for the material under test, operate the tester at a speed such that the average time from initial application of load to break is 5 to 15 s for any set of ten specimens. For referee tests, the average time to break shall be 8 to 12 s.

117.5.2 Most pendulum-type machines operate at a single traverse rate of 300 mm/min (12 in./min), or at a small number of fixed speeds, usually including 300 mm/min. The average time to break and the traverse speed should be noted when this type of machine is used.

117.5.3 When determining TEA on a CRE machine, use a speed of 25 ± 5 mm/min (1.0 ± 0.2 in./min), when using a 200 mm (8 in.) test span. For other test spans, adjust the speed proportionally to give the same straining rate.

117.5.4 In selecting the proper speed, as indicated in 117.5.3 or as specified in the document calling for this test method, the maximum speed that can accurately be used on a given machine is determined in part by the response time of the devices for indicating and recording load and elongation. The rate of increase of load or of elongation must be low enough that errors at any point on the curve due to lag in the indicating or recording devices will be less than 1 % of the values at break. In general full-scale response time should be less than 5 s in either direction. See Specification D 76 for further discussion of this subject.

117.6 Reject readings from individual specimens that slip in the jaws or break in or at the edge of the jaws. Slippage is usually caused by low clamping pressure. Jaw breaks may be caused by excessive clamping pressure, sharp jaw edges, or uneven stresses.

117.7 Record the values for load at break and for elongation at break. The point of break is that of maximum applied load. If possible, select scales for indicating or recording load and elongation such that the values at break are within the upper half of the scale, and, in any case, to give the accuracies specified in 114.1.

117.8 Determine the area enclosed by the load-elongation curve, the elongation axis and the line representing elongation at break, using a planimeter, or alternate equipment as described in 114.3.

118. Calculation

118.1 For each test specimen calculate:

118.1.1 Tensile strength, either in force per unit width of specimen (kN/m or lb/in.), or in force per unit cross-sectional area of specimen (kPa or psi).

118.1.2 Elongation at break, expressed as a percentage of the span at initial application of load.

118.1.3 Multiply the integrator or planimeter value by the appropriate factor for the equipment and settings to obtain the area under the load elongation curve. Calculate the tensile energy absorption according to one of the following equations:

$$TEA = 10^{6}\, A/LW\ J/m^{2} \tag{11}$$

$$TEA = 12\, a/lw\ \text{lbf•ft/ft}^{2} \tag{12}$$

where:

TEA = tensile energy absorption, J/m^2 or lbf·ft/ft^2

A = area under load-elongation curve, J,

L = initial test span, mm,

W = specimen width, mm,

a = area under load-elongation curve, lbf·in.,

l = initial test span, in.,

w = specimen width, in., and 1 J/m^2 = 0.0685 lb·ft/ft ^{2}.

118.2 For each set of test specimens calculate the averages for tensile strength, elongation at break, and TEA; and determine the range, standard deviation, or other indication of variability.

119. Report

119.1 Report in accordance with Section 14, and include the following information:

119.1.1 Average value and indication of variability for tensile strength, elongation at break, and TEA, as calculated in Section 118,

119.1.2 Type of testing machine used,

119.1.3 Rate of loading,

119.1.4 Average time to break,

119.1.5 Width of test specimens,

119.1.6 Initial test span, and

119.1.7 Number of individual tests rejected, and the reasons for rejection.

120. Precision and Bias[17,18]

120.1 *Precision*:

120.1.1 Interlaboratory tests indicate that two test results, each representing an average of ten determinations, from the same or different samples as noted, can be expected to agree within the amounts as follows (95 % probability):

	Tensile Strength	Elongation	TEA
Repeatability (same sample, operator and apparatus),%^{A}	5	9	10 to 16
Comparability (samples markedly different in other properties, but not in designated property, same operator and apparatus),%^{A}	9	16	15 to 16
Reproducibility (same sample, different laboratories), %^{A}	10	25	22 to 36

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[17] Lashof, T. W., "Precision of Methods for Measuring Tensile Strength, Stretch and Tensile Energy Absorption of Paper," TAPPI Vol 46, January 1963, pp. 52–59.

[18] Data in Section 120 were abstracted from TAPPI T-494, Tensile Breaking Properties of Paper and Paperboard.

[A] Percentage of the average of the two test results.

120.1.2 In each case, the coefficient of variation of a test result (average of 10 determinations) is expected to be about 0.36 times the value shown in 120.1.1.

120.1.3 The information in 120.1.1 is based on tests made on papers having elongation of 10 % or less. The validity of these figures for papers of greater elongation has not been determined.

120.2 *Bias*—A statement of bias cannot be made due to the lack of a standard reference material.

INTERNAL-TEARING RESISTANCE

121. Scope

121.1 This test method covers the determination of the average force in grams required to tear a single sheet of paper after the tear has been started.

121.2 This test method is not suitable for determining the cross-directional tearing resistance of papers having tensile strength in the machine direction that is much higher than in the cross-machine direction.

122. Summary of Test Method

122.1 A pendulum is permitted to make one swing and thereby cause the tearing of one or more sheets together through a fixed distance. The work done in tearing is measured by the loss in potential energy of the pendulum. The scale is calibrated to indicate average force (work done divided by tearing distance).

123. Significance and Use

123.1 The results of the test are suitable for the following:

123.1.1 For acceptance; process control; research; or referee testing, and

123.1.2 For measuring serviceability (suitability) of papers which in use are subjected to tear strain.

123.2 Tearing resistance of paper is influenced by:

123.2.1 The kind, quality, and treatment of the paper fibers,

123.2.2 The formation of the sheet on the paper-making machine, and

123.2.3 The treatment of the sheet after formation (including machine wet pressing and calendering).

124. Procedure

124.1 Determine the tearing resistance in accordance with Test Method D 689 or TAPPI T 414, except sample the material in accordance with Sections 6 to 13 of these test methods, and use the minimum number of sheets necessary to bring 80 % of the test measurements above 20, the minimum permissible scale reading.

125. Report

125.1 Report in accordance with Section 14 and with Test Method D 689 or TAPPI T 414.

EDGE-TEARING RESISTANCE

126. Scope

126.1 This test method covers the determination of the edge-tearing resistance of electrical papers. A strip of paper is folded around the edge of a shallow V-notch in a thin beam and pulled to tear the paper at the edges. A standard testing machine is used to apply and measure the force required to cause tearing.

127. Significance and Use

127.1 For certain manufacturing operations such as high-speed wire wrapping, the edge-tearing resistance may correlate well with performance of a given paper.

127.2 When comparing papers whose properties other than edge-tearing resistance differ greatly, it is generally not accurate to predict the relative performances of the papers solely on the basis of their edge-tearing resistances. Other properties, such as tensile strength, elongation, internal tearing resistance, surface friction, and directionality may also affect the performance of a given paper.

128. Procedure

128.1 Determine the edge-tearing resistance in accordance with Test Method D 827 or TAPPI T 470 except sample in accordance with Sections 6 to 13.

129. Report

129.1 Report in accordance with Section 14 and Test Method D 827 or TAPPI T 470.

130. Precision and Bias

130.1 *Precision*—From a round-robin test[19] involving five laboratories each testing five grades of paper on two types of machines, with a common stirrup used by all laboratories in addition to each one's individual stirrups, the following conclusions were drawn:

130.1.1 For papers having edge-tearing resistance between 18 *N* (4 lb) and 200 *N* (45 lb), the between-laboratory difference on specimens cut from the same sample of paper should not exceed 10 %.

130.1.2 For some grades of papers (three of the five tested), the use of two different stirrups, even by the same laboratory, produced values that were, with 95 % confidence, significantly different.

130.1.3 At the 95 % confidence level, there was no significant difference between the results obtained on the pendulum-type and on the constant-rate-of-extension machines.

130.2 This method has no bias because the value for edge-tearing resistance is defined in terms of the method.

HEAT STABILITY IN AIR

131. Scope

131.1 This test method determines the retention of one or more selected properties of a paper sample when test specimens are subjected to a specified period of heating in air at 120°C.

132. Significance and Use

132.1 During the fabrication of electrical equipment in which electrical insulating paper is a component, the paper may be exposed to air at elevated temperature. This heat

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[19] Supporting data are available from ASTM Headquarters. Request RR:D-9-1004.

stability test evaluates the effect of such exposure upon the physical characteristics of the paper. Results of this test method are suitable for production control, for routine acceptance testing, and for research.

132.2 Heat stability in air, as measured by this test method, does not evaluate the "life", "thermal aging", or suitability of a paper for use at elevated temperatures. More complex methods are required to evaluate such characteristics.

133. Apparatus

133.1 *Baking Oven*—An electrically heated oven.

133.2 *Specimen Holding Fixture*—A fixture for holding the specimens while in the oven. The fixture must be of appropriate dimensions for holding the specimens and keeping them separated and protected from damage during exposure to heating in the oven.

133.3 *Testing Equipment*—There must be suitable test equipment for each property by which heat stability is to be evaluated. Depending on the intended use, evaluation may reasonably be in terms of folding endurance (Sections 108 to 110), internal tearing resistance (Sections 121 to 125), edge tearing resistance (Sections 126 to 130), tensile strength (Sections 111 to 120), elongation (Sections 111 to 120), tensile energy absorption (Sections 111 to 120), bursting strength (Sections 102 to 107), or a combination of properties.

134. Test Specimens

134.1 Cut a sufficient number of test specimens in one or both of the principal directions, as may be specified, from samples obtained in accordance with Sections 6 to 13, in dimensions to suit the chosen test or tests to be made. Divide the test specimens into two similar sets, each composed of corresponding adjacent pieces as cut from the samples. One set is for the baking treatment and subsequent tests, the other set for comparative tests of unbaked specimens.

135. Procedure

135.1 Mount the specimens to be heat-treated on the fixture specified in 133.2.

135.2 Place the specimens, in the holding fixture, into the oven. Establish the period of baking such that at least one variety of test specimen shall experience at least 20 % of loss in the test. A 72-h period is usually sufficient for this requirement.

135.3 Remove the baked specimens from the holding fixture, taking care not to damage them, and condition them in accordance with Section 15 before testing. Approach the conditions of testing for both the baked and the unbaked specimens from a humidity lower than 50 % relative humidity to avoid possible hysteresis effects. Test the conditioned unbaked and baked specimens during the same test period.

135.4 Calculate the heat stability as 100 times the ratio of the test result for the aged specimens to the corresponding test result for the unaged specimens.

136. Report

136.1 Report in accordance with Section 14, and also include the following information:

136.1.1 The time of baking, h,

136.1.2 The property by which stability was measured, and the method used to determine that property,

136.1.3 Test results for the baked and unbaked specimens, and

136.1.4 Calculated heat stability, %.

137. Precision and Bias

137.1 The precision of this test method has not been determined, and no activity is planned to determine its precision.

137.2 This test method has no bias because the value for heat stability is defined in terms of the method.

CONDUCTING PATHS

138. Scope

138.1 These test methods provide procedures for testing electrical insulating paper for the presence of localized areas that will conduct an electric current. Method A is limited to papers not over 0.040 mm (0.0015 in.) thick. Method B is limited to papers over 0.04 mm in thickness, since thinner paper might be damaged mechanically by the test. The upper limit of thickness is self-determining at the greatest thickness that can be handled in a continuous web on the equipment.

138.2 There are similarities between the tests described herein and Test Method D 1389. The principal, and critical, difference with D1389 is that D1389 uses alternating voltages for detecting conducting paths.

139. Significance and Use

139.1 Conducting paths seriously reduce the insulating value of paper under electrical stress, and may result from either structural imperfections or inclusions.

139.1.1 The conducting paths count may reflect the relative cleanliness of the base pulp, the process water, the physical manufacturing system, or any combination thereof.

139.2 The conducting paths tests indicate the frequency of occurrence of dielectric defects and facilitate isolation of areas where they are excessive.

139.2.1 Method B permits location of the defects for repair or removal, as may be desired.

139.3 Conducting paths tests may be used for manufacturing control, or for acceptance evaluation.

140. Sampling

140.1 Sample in accordance with Sections 6 to 13, and with consideration of the specimen requirements and capabilities of the test method.

Method A—For Papers 0.04 mm (0.0015 in.) and Under in Thickness

141. Apparatus

141.1 *Electrodes*—Two electrodes, consisting of a smoothly machined cast iron or other metal plate, and a hand-operated metal roller with smoothly machined face and fitted with an insulated handle. The plate shall be of convenient size to accommodate the specimens to be tested. The finished dimensions of the roller shall be as follows:

	mm	in.
Diameter	50	2.0
Width of face	25	1.0

NOTE 25—For papers less than 38 mm (1.5 in.) wide use a roller with a face width of 10 mm (0.37 in.).

141.2 *Detector*—An impulse counter or loud speaker having a resistance of 3000 Ω.

141.3 *Resistor*, 80 000 Ω.

141.4 *Voltage Supply*—A source of 110 V dc, with one terminal grounded.

141.5 *Circuit*—Connect the detector in series between the plate electrode and the grounded terminal of the voltage supply. Connect the resistor in series between the roller electrode and the 110-V terminal of the voltage supply.

142. Test Specimen

142.1 The test specimen, of sufficient size to cover the plate electrode, shall be held in place by weights. Test several such specimens. The paper must contain not more than 8 % moisture when tested.

142.2 It has been found in the testing of thin papers that great care must be taken in handling the specimens. They must not be touched with the hands nor should they be laid upon anything but the plate electrode after removal from the roll.

143. Procedure

143.1 **Warning**— *High Voltage: Lethal voltages may be present during these tests. It is essential that the test apparatus and all associated equipment that may be electrically connected to it be properly designed and installed for safe operation. Solidly ground all metal parts that any person might come into contact with during the test. Thoroughly instruct all operators in the proper way to conduct the tests safely. When making high voltage tests, particularly in compressed gas or in oil, the energy released at breakdown may be sufficient to result in fire, explosion, or rupture of the test chamber. Design of test equipment, test chambers, and test specimens should be such as to minimize the possibility of such occurrences, and to eliminate the possibility of personal injury.*

143.2 Secure the test specimen flat upon the plate electrode by means of weights at the ends. Turn on the voltage supply and, when it is delivering 110 V, pass the roller once over parallel sections of the test specimen, carefully not overlapping, at a speed from 25 to 100 mm/s (1 to 4 in./s). Each impulse recorded by the counter or heard as a click on the loud speaker indicates a conducting path.

144. Calculation

144.1 Calculate the number of conducting paths per unit area as follows:

$$\text{Number of conducting paths per unit area} = B/A \tag{13}$$

where:

B = total number of conducting paths determined on all test specimens, and

A = total area contacted by the roller.

145. Report

145.1 Report in accordance with Section 14, and include the number of conducting paths per unit area and the total area of paper tested.

Method B—For Papers Over 0.04 mm (0.0015 in.) in Thickness

146. Apparatus

146.1 *Electrodes, Type I*—Two electrodes consisting of a smoothly machined cast iron or other metal plate, and a set of smooth solid brass rollers. The plate dimension parallel to the direction over which the test specimen is moved shall be at least 150 mm (6 in.); the other dimension of the plate shall be at least as great as the width of the test specimen.

146.1.1 The surface of the rollers shall be machined and polished to smooth cylinders. The finished dimensions of the brass rollers shall be as follows:

	mm	in.
Diameter	38	1.5
Width of face	25	1.0

146.1.2 The rollers shall be mounted in two parallel rows above the plate electrode, and so arranged that each roller may be raised above or lowered onto the plate surface, automatically aligning the roller surfaces with the plate surface. The rollers and plate shall be adequately insulated from each other. The rollers shall be mounted so that continuity of electrical connection between them and the potential source is assured when they are rotating. The two rows of rollers shall be mounted with their axes 90° to the direction in which the test specimen is moved. The rollers in each row shall be spaced 35 mm (1⅜in.) apart on center lines, and the two rows shall be so placed that the midpoints of the rollers in one row are opposite the midpoints of the spaces in the other row as shown schematically in Fig. 9. The total weight exerted by each roller on the surface of the specimen shall be not more than 315 g nor less than 245 g.

146.2 *Electrodes, Type 2*—Two electrodes consisting of a steel or other suitable metal roller, 150 mm (6 in.) or more in diameter, and at least as wide as the width of the paper web to be tested; and a flexible metal blade or blades. The blades shall be mounted in such a way that they may be pressed tangentially against the roller electrode in a pattern wide enough to cover the entire width of the paper under test, with the exception of such a margin as is needed at the edge to prevent the electrodes from coming in direct contact with one another, or to prevent flashover between the electrodes.

146.2.1 The blade electrode may be a single blade designed to fit a given width of paper. It may also be two blades mounted in overlapping position which can be moved laterally to provide an adjustable width, or it may be a series of narrow blades mounted in a double row so that the midpoints of the blades in one row are aligned with the midpoints of the spaces between the blades in the other row. The individual blades shall be wider than the open spaces between blades so that the entire area of paper moving between the electrodes will be contacted by at least one blade electrode in passage.

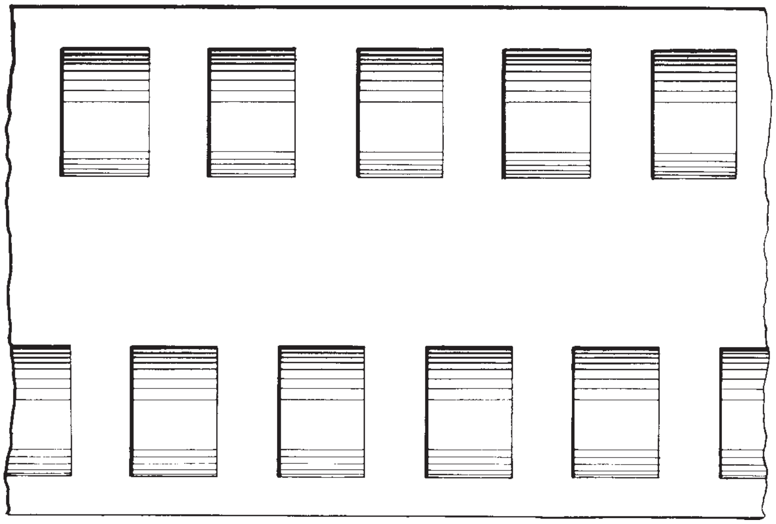

FIG. 9 Arrangement of Brass Rollers Used in Surveying Medium Thick Papers for Conducting Paths (Method B)

146.3 *Backstand*—A stand upon which the roll of paper to be tested may be mounted with suitable tension controlling devices, such as are commonly used to control the unwind of paper.

146.4 *Winder*—A takeup device which can draw the paper from the roll through the electrodes and rewind it into a good roll, suitable for its intended use.

146.5 *Voltage Source*—A source of direct voltage which may be applied to the electrodes and varied to suit the requirements of the paper under test. The voltage supply should be capable of delivering voltage up to the breakdown level of the most resistant paper expected to be tested in the apparatus.

NOTE 26—Breakdown level in this case means the voltage at which almost continuous discharge between the electrodes through the moving web of paper is observed.

146.5.1 The voltage supply and collateral wiring should be capable of handling an unknown but substantial current for a short time when a conducting path passes between the electrodes.

146.6 *Detector*—A signal or counting device that will indicate or record, or both, the momentary flow of current when a conducting path passes between the electrodes. This may be supplemented as desired by devices that will mark the web at the approximate location of the conducting path.

146.7 *Measuring Device*—A measuring wheel that contacts the moving web, attached to a suitable counter that will record the length of paper drawn through the electrodes.

147. Test Specimen

147.1 Prepare the test specimen to whatever width is convenient (usually the width of a commercial jumbo roll of paper) and with a total area not less than 100 m^2 or 1000 ft^2 according to the units used in reporting the results of the test. The specimen may be an entire lot of paper rather than a sample selected to represent the lot.

148. Procedure

148.1 **Warning**—See 143.1 for high-voltage warning.

148.2 Mount the rolled specimen on the backstand, pass the end of it between the electrodes, and fasten it to a core on the winder. Engage the measuring device and the detector. Apply voltage of a predetermined level to the electrodes. Start the winder and draw the specimen through the electrodes at the predetermined speed. Record the number of conducting paths in the paper and the length of paper drawn through the electrodes.

148.3 The standard level of stress shall be direct voltage at 5.5 kV/mm (140 V/mil) of thickness. If any other level of voltage stress is used, it shall be upon agreement between purchaser and seller.

NOTE 27—It has been observed that the number of paths detected increases with increase in voltage up to about 75 % of the breakdown level.

148.4 There is no standard speed specified, as variation in speed has little or no effect on the number of conducting paths detected when using direct voltage.

149. Calculation

149.1 Calculate, from the width of specimen and the recorded length, the area of paper tested. From this area and the recorded number of conducting paths, calculate the number of conducting paths per 100 m^2 or 1000 ft^2, whichever is applicable.

150. Report

150.1 Report in accordance with Section 14, and include the following information:

150.1.1 Number of paths per 100 m^2 or 1000 ft^2, whichever is applicable.

150.1.2 Total area of paper tested,

150.1.3 Voltage applied, and

150.1.4 Running speed.

151. Precision and Bias

151.1 The precision of these test methods has not been determined, and no activity is planned to determine their precisions.

151.2 These test methods have no bias because the values for numbers of conducting paths are defined in terms of these methods.

DIELECTRIC STRENGTH

152. Scope

152.1 The purpose of this test is to determine the dielectric strength of untreated paper samples by the short-time test as described in Test Methods D 149.

152.2 Papers which have been subjected to treatments such as coating or impregnating with varnish may also be tested using this test method. For papers to be tested after impregnation with a dielectric liquid, refer to Test Methods D 2413.

153. Significance and Use

153.1 This test, applied to untreated paper, may be used as a basis for determining uniformity in manufacture. It is suitable for use as a quality control, research, or specification test.

153.2 This test method is suitable for the comparison of only those paper samples of similar thickness and apparent density.

153.3 Refer to the Significance and Use section of Test Methods D 149 for further information on this subject.

154. Apparatus

154.1 The apparatus shall conform to that specified in Test Methods D 149 using the Type 1 electrodes specified in Table 1 for sheet materials except that the upper 51 mm (2 in.) diameter electrode may be thicker than 25 mm (1 in.). This upper electrode shall apply a load of 675 ± 35 g to the specimen. For tapes, or sheet material to be compared with tapes, the electrodes shall be 6 mm (0.25 in.) diameter rods as specified as Type 3 in Table 1 of Test Methods D 149.

155. Test Specimens

155.1 From the sample obtained in accordance with Sections 6 to 13, select specimens of sufficient area to provide for at least five tests. Condition the test specimens as prescribed in Section 15 of these test methods. Make the dielectric tests in the same standard atmosphere.

156. Procedure

156.1 **Warning**—See 143.1 for high-voltage warning.

156.2 Determine the dielectric strength in accordance with the short-time test as described in Test Methods D 149 with the following requirements:

156.2.1 For paper with a total dielectric breakdown voltage less than 2500 V, increase the voltage at a rate of 100 ± 20 V/s.

156.2.2 For paper having a total dielectric breakdown voltage between 2500 and 5000 V, increase the voltage at a rate of 250 ± 50 V/s.

156.2.3 For paper having a total dielectric breakdown voltage over 5000 V, increase the voltage at a rate of 500 ± 100 V/s.

156.2.4 In the case of a series of tests on the same batch of specimens averaging about 2500 V or about 5000 V, make all such series of tests at the slower specified rate of voltage application.

156.2.5 From the average paper thickness for the sample, as determined in Sections 16 to 24, calculate the kilovolts per millimetre or volts per mil for each puncture.

156.3 In the case of papers that are very thin or very low in dielectric strength, use stacks of specimens as called for in the material specification or to effect a total minimum breakdown voltage of at least 600 V.

157. Report

157.1 Report in accordance with Section 14 and the Report section of Test Methods D 149, and include the following information:

157.1.1 Number of thicknesses of paper tested if more than one, and

157.1.2 Size and materials of construction of the electrodes.

60-Hz DISSIPATION FACTOR AND PERMITTIVITY OF DRY INSULATING PAPER

158. Scope

158.1 This test method covers the measurement of dissipation factor and permittivity of dried, unimpregnated electrical insulating paper.

159. Summary of Test Method

159.1 Dissipation factor and capacitance are measured on small specimens of paper held between precisely made electrodes and under prescribed pressures. The specimens thus held are dried under standardized conditions of heat and vacuum, then allowed to cool slowly in dry air while measurements of dissipation factor and capacitance are made at a number of temperatures.

159.1.1 The effective thickness of the specimen during measurement may be determined by use of an auxiliary air-gap capacitance measurement. Dissipation factor and capacitance measurements of specimen and air gap, and calculation of permittivity, are made in accordance with methods described in Test Methods D 150.

159.1.2 The complexity of the mixture of fibers and air comprising a sheet of paper requires that great care be taken in the measurement and interpretation of permittivity and dissipation factor.

160. Significance and Use

160.1 The general significance of dissipation factor and permittivity are stated in Test Methods D 150. With respect to paper, the following also apply:

160.1.1 *Dissipation Factor*—The power loss in the dielectric of ac equipment is important in their design and use. This loss has been found to be principally the result of dielectric losses in the insulating paper of ac equipment such as capacitors and power cables. Knowledge of the dissipation factor and its variation with temperature in insulating paper is important in the design of paper-insulated electrical equipment. The dissipation factor is a very sensitive measure of the purity of raw materials, of chemicals used in the manufacturing process, and of cleanliness in the manufacturing process. The test is thus useful for quality-control purposes, subject to the interpretative caution noted in 159.1.2.

160.1.2 *Permittivity*—A value of permittivity which deviates significantly from that of similar papers measured under comparable conditions suggests nonconformance of the specimen to the electrode surface due to curl, wrinkles, folds, or lumps within the specimen. Permittivity is also directly related to the density of the paper, and may be affected by the addition of nonfibrous components.

161. Apparatus

161.1 *Dissipation Factor and Capacitance Bridge*—A guarded, high-voltage tape Schering bridge as described in Test Methods D 150, or its equivalent, having an accuracy of ±0.0005 dissipation factor or ± 1 % whichever is greater, and a capacitance range from 50 to 1000 pF at 60 Hz.

161.2 *Generator*—A 60-Hz, 300-V or greater, generator. Alternatively, voltage may be supplied from a 60-Hz line, using a transformer and variable-ratio autotransformer.

161.3 *Detector*—A detector having sufficient sensitivity to provide a resolution in the dissipation factor balance of 2×10^{-5}(0.00002) or 1 % of the reading, whichever is higher.

161.4 *Electrodes*—One or more sets of circular electrodes with annular-guard electrodes constructed of a metal not subject to distortion with repeated temperature cycling (See Fig. 10). The electrode area shall be such that the capacitance to be measured shall be within the range of the bridge. The gap between measuring and guard electrodes shall be as small as practicable, preferably no larger than the thickness of specimens to be measured. A dead-weight pressure shall be applied to the specimen by addition of a known weight to the upper electrode. A total of 20 kPa (3.0 psi) is recommended for papers less than 0.04 mm (0.0015 in.) in thickness. The electrode surfaces shall be machined and lapped flat to within 0.00125 mm (0.0005 in.) as determined by an optical flat. The electrode surfaces shall be kept clean and shall be polished periodically with crocus cloth to maintain flatness and smoothness.

161.5 *Vacuum Drying Equipment*:

161.5.1 *Vacuum Chamber*—A vacuum chamber capable of being opened and resealed so as to be capable of evacuation to less than 2.7 Pa (20 µm Hg) under continuous pumping, and of being heated to at least 120°C. Electrical and temperature measurement connections shall be provided for measurements within the chamber. The base plate shall be of suitable metal and thickness (for example, ground cast iron) so that it will not distort on repeated temperature cycling. Connections for evacuation and vacuum measurement, as well as electrical and temperature-measuring leads normally pass through the base plate so that a borosilicate glass bell jar or inverted desiccator provides the sides and top of the chamber.

161.5.2 *Vacuum Pump*—A vacuum pump capable of evacuating the system and maintaining a vacuum of 2.7 Pa (20 µm Hg) pressure or less under continuous pumping.

161.5.3 *Vacuum Gage*.

161.5.4 *Necessary Vacuum Hardware*, for example, valves, tubing, etc.

161.5.5 *Flexible Tubing*, especially rubber or vinyl tubing, should be used only as a means of making flexible joints because of a tendency to leak or outgas.

161.6 *Heating Equipment*—Equipment to provide a means of heating electrodes and specimen within the vacuum chamber to the specified temperature(s). An infrared bulb or a set of incandescent bulbs mounted, outside of the chamber so as to provide as uniform heating of the chamber as possible, and surrounded by a heat reflector, have been found suitable. A heating mantle to fit the desiccator, if used, is also acceptable.

161.7 *Temperature Measurement and Control*—A thermocouple embedded in the guard plate with small diameter leads carried through the base plate will provide a suitable means of recording the temperature, and when used with a pyrometer controller, will provide a means of controlling temperature.

NOTE 28—In any specific test equipment, it is necessary to determine experimentally the relationship between the measured temperature and the actual temperature of the specimen under conditions which match those of the dissipation factor measurement as closely as possible. Temperature calibrations should be performed periodically and whenever changes are made in the system that might affect the relation between observed temperature and true specimen temperature.

161.8 *Punch Die*—Steel circle-punching dies as required to cut specimens. A hardwood block or suitable backing support may be required to hold the paper during punching.

161.9 *Air-Drying Equipment*—Equipment to provide a means of introducing thoroughly dried air into the vacuum chamber (for example, a train including sulfuric acid and phosphorus pentoxide, or activated alumina drying apparatus).

161.10 *Thickness-Measuring Apparatus*— A dead-weight dial micrometer as specified in 19.1.1.2, unless thickness is

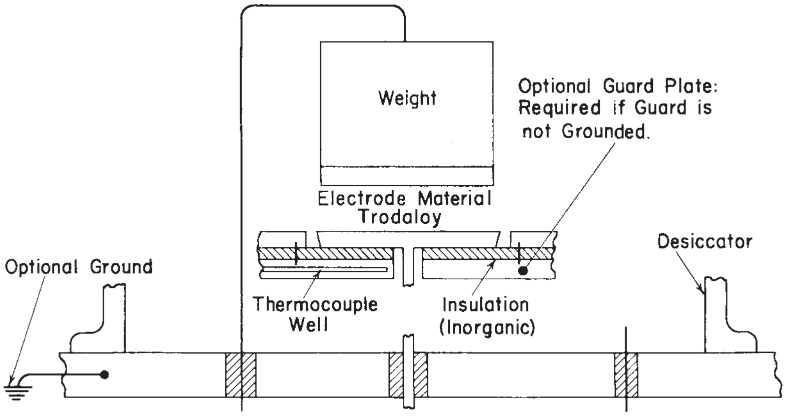

FIG. 10 Electrodes for Dissipation Factor Test

measured by the air-capacitance method, or other micrometer as described in Test Methods D 374.

161.11 *Sample Bottles*—Bottles with ground-glass stoppers for storage and transfer of specimens.

161.12 *Analytical Balance*.

162. Test Specimens

162.1 The test specimens shall consist of one or more punched, circular layers of paper with a diameter at least 3 mm (0.12 in.) larger than that of the guarded electrode.

163. Procedure

163.1 For papers 0.04 mm (0.0015 in.) or less in thickness, stack sufficient sheets to make a pad as close as possible to 0.10 mm (0.004 in.) in thickness, plus two cover sheets. Remove the stack from between the cover sheets with tweezers and mount on the bottom electrode. Never touch the specimen with bare hands.

163.1.1 For papers greater than 0.04 mm and less than 0.25 mm (0.010 in.) in thickness, prepare a stack of one or more specimen sheets plus outside cover sheets. Remove the specimen stack from between the cover sheets with tweezers and mount on the bottom electrode.

163.1.2 Prepare specimens of at least two sheets for thicknesses between 0.4 and 0.25 mm in order to minimize the effect of irregularities of the paper-electrode interfaces which might reduce the effective paper density between electrodes. The electrode area should be such that the capacitance of multisheet specimens of this thickness range will be within the capacitance range of the bridge.

163.1.3 For papers over 0.25 mm and up to 0.75 mm (0.030 in.) in thickness, a single sheet is sufficient. For papers within this thickness range care must be taken to eliminate or prevent any curl of the specimen between the electrodes. Added dead-weight pressure might be required to eliminate curl.

163.1.4 For sheet materials greater than 0.75 mm in thickness, such as paperboard or pressboard, special electrode or cell construction may be required to ensure elimination of air gaps due to curl. A thin metal foil electrode set applied with a minimum of silicone grease is recommended for this purpose, such as described in Test Methods D 150. A guard electrode configuration can be made according to the method described in Test Methods D 150. Accurate concentric alignment of guarded and unguarded electrodes might require an aligning fixture, as indicated in Test Methods D 150. Hold the guarded and unguarded electrode foil disks by an opposing pair of rigid metal electrodes, plus a top dead weight. Make electrical connections by means of the rigid electrode and dead-weight system. The guard electrode should be of diameter sufficient to permit attachment of a clip or soldered lead. Support the guard electrode lead so as not to cause movement of the guard electrode.

163.2 Heat the specimen under continuous vacuum at a temperature not less than 105°C (for cellulose paper) for a period of time sufficient to dry the specimen.

NOTE 29—The specimen is considered dry when the dissipation factor does not decrease with increased time of drying. Usually 16 h at 115°C and 2.7 Pa or less will suffice for cellulose papers. Completeness of drying can be checked by redrying and retesting the undisturbed specimen. The two plots of dissipation factor versus temperature will be identical if the specimen is completely dry. (The curves will also be identical if equal amounts of moisture are accidentally admitted after each drying run.) For papers of materials other than cellulose fibers, modifications of specimen drying might be required.

163.3 Break the vacuum with dry air and allow the specimen to cool. Measure the dissipation factor and capacitance of the specimen at intervals of approximately 10 to 20°C at a voltage stress sufficient to provide the necessary precision, but not exceeding 2 kV/mm (50 V/mil) until the specimen has cooled to 40°C or below.

163.3.1 If the dissipation factor rises rapidly as the system cools to room temperature, it is an indication that moisture is entering the system, either from leakage through a faulty seal or because of inadequate drying of the air drawn into the system through the drying train. The cause of the moisture entry must be determined and corrected before redrying of the specimen and redetermination of the dissipation factor. A positive pressure of dry air swept continuously through the system and across the specimen is recommended.

163.4 Measure the capacitance of the supplementary airgap electrode where used.

NOTE 30—A supplementary guarded electrode, as illustrated in Fig. 11, which has at all times the same separation from the upper electrode as that of the main electrode, may be used to determine thickness by measurement of its capacitance in dry air. The thickness, t, is calculated as follows:

$$t = (K \times A)/C \tag{14}$$

where:
K = 0.885, for t, mm and A, cm^2,
K = 0.2249, for t, in. and A, in.^{2},
t = separation of electrodes,
A = effective area of electrodes (see Test Methods D 150), and
C = capacitance, pF.

It is generally more accurate to measure the actual electrode separation in this way to determine specimen density at the time of the electrical measurement rather than by micrometer measurement of thickness. The dissipation factor and capicitance of the specimen are related directly to the density of the specimen at the time of the electrical measurement rather than to a density determined by a subsequent mechanical measurement.

163.5 Remove the specimen from the cell with tweezers. As quickly as possible punch a smaller diameter piece from the specimen and place it in a stoppered weighing bottle.

163.6 Determine the weight of the specimen and previously tared bottle on an analytical balance, from which specimen weight can be determined.

163.7 Remove the specimen and immediately determine the specimen thickness by a dead-weight dial micrometer in accordance with Sections 18 to 24 for papers of thickness 0.040 mm (0.0015 in.) or less. For papers of thickness greater

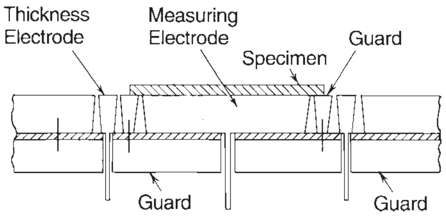

FIG. 11 Guarded Electrode for Determining Thickness

than 0.040 mm, measure the thickness in accordance with Test Methods D 374 by agreement between the seller and the purchaser. Thickness measurement is not required if a measurement of thickness by air-gap electrodes has been made.

163.8 Calculate the apparent density (dry-dry basis) from the weight, the known area, and the thickness of the piece cut in 163.5.

164. Report

164.1 Report in accordance with Section 14, and include the following information:

164.1.1 Curve of dissipation factor as a function of temperature, or a tabulation of dissipation factor at specified temperatures as determined by interpolation of the dissipation factor-temperature curve. Extrapolation is not permitted.

164.1.2 Thickness of the specimen, the number of sheets used, the electrode pressure applied, the applied voltage, whether conforming foil electrodes were used (where applicable), and the method of thickness measurement.

164.1.3 Permittivity of the specimen at a temperature agreed upon between the seller and the purchaser, and calculated in accordance with Test Methods D 150, and

164.1.4 Apparent density of the specimen in grams per cubic centimetre.

WATER-EXTRACTABLE CHLORIDES

165. Scope

165.1 This test method covers the determination of water-extractable chlorides in electrical insulating paper.

165.2 Two methods are described; a potentiometric titration method, and a progressive dilution method.

166. Summary of Test Methods

166.1 Soluble chloride is determined by extracting with water under standard conditions and either titrating the dissolved chloride potentiometrically (Method A), or finding the maximum dilution at which it will produce a visible opalescence with silver nitrate (Method B). For routine control applications an abbreviated extraction procedure may be used with either test method.

167. Significance and Use

167.1 The purity of papers that are used as electrical insulation is of great importance in many applications. The presence of chloride may adversely affect the electrical properties of the paper.

167.2 Method A with short time extraction is suitable as a routine control and acceptance test for all levels of water soluble chloride. Method A with long term extraction is suitable for both routine and referee testing for water-soluble chloride at all concentrations. Method B with either long time or short time extraction is restricted to use for water soluble chloride levels of 16 ppm (parts per million) or greater and where the within-range method of reporting is acceptable.

168. Interferences

168.1 The presence of water soluble halide ions other than chloride will be detected by these test methods, as will the presence of any substance that forms an insoluble silver compound or otherwise interferes with the response of silver to the silver electrode in Method A.

168.2 The amount of chloride ions measured when the test is applied to electrical insulating paper of good quality is very small, from a fraction of a part per million to a few parts per million. For this reason, extreme care must be taken in all phases of testing, from sampling to final addition of reagents. The specimen should not be touched with the hands. All glassware, apparatus, and the atmosphere in contact with the sample or its extract solution must be free of contamination, such as chemical vapors, tobacco smoke, etc. The test method requires titration of a simultaneous blank. The level of chloride indicated by the blank will indicate the amount of interfering contamination of the analysis.

Method A

169. Apparatus

169.1 *Electrometric Titrimeter*[20]—A high-impedance millivoltmeter having a scale with the smallest ruled interval not greater than 5 mV and an ultimate sensitivity of 0.5 mV or less.

169.2 *Standard Glass Electrode.*[21]

169.2.1 Although the glass electrode is preferred, a mercurous sulfate reference electrode may be used.

169.3 *Microburet*, 1.0 mL capacity graduated in 0.01 mL divisions.

169.4 *Magnetic Stirrer*, with glass-covered or PTFE-fluorocarbon-coated stirring bar.

169.5 *Hot Plate*.

169.6 *Tall-form Beaker*.

169.7 *Extraction Glassware*.

169.7.1 *Erlenmeyer Flasks*, 300 mL, standard taper 24/40 joint.

169.7.2 *Condenser*, water-cooled, standard taper 24/40 joint.

169.7.3 *Fluorocarbon Sleeve*, standard taper 24/40. These sleeves eliminate the need for grease, which is a possible contaminant.

169.8 *Balance*, with 0.01 g accuracy.

169.9 *Buchner Funnel*, coarse-fritted glass.

169.10 *Tongs*, stainless steel.

169.11 *Medicine Dropper*.

170. Reagents

170.1 *Water*, conforming to Type III of Specification D 1193, and free of chlorides as detected by this test method.

170.2 *Acetone*, chloride-free, reagent grade. If a blank titration shows the presence of chloride, reflux 1 L of the acetone with 0.1 g silver nitrate for 30 min. Distill the acetone from the silver nitrate, discarding the first 25 mL to flush the apparatus and receiver. Distill approximately 90 % of the acetone and discard the remaining liquid. The chloride ion

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[20] The Radiometer P501, and Leeds & Northrup standard 1199-62 have been found satisfactory for this purpose.

[21] Radiometer K-601, Beckman No. 40455 mercury sulfate sleeve electrode with Beckman No. 8548 saturated potassium sulfate filling solution has been found satisfactory for this purpose.

concentration of the acetone should be equivalent to less than 0.01 mL of 0.0025 *N* $AgNO_3$ solution per 100 mL of the acetone.

170.3 *Nitric Acid* (1 %)—Add 1 mL of concentrated nitric acid (sp gr 1.42) to 80 to 90 mL of water. Then dilute with water to 100 mL.

170.4 *Silver Nitrate, Standard Solution (0.0025 N)*:

170.4.1 Dry the silver nitrate overnight in a desiccator before preparing the solution. Protect both the solid $AgNO_3$ and the solution from light by storage in brown glassware in a dark room or cabinet.

170.4.2 Weigh 424.7 ± 0.1 mg of reagent grade silver nitrate. Transfer it quantitatively to a 1-L volumetric flask and add sufficient water to dissolve the $AgNO_3$. Add 3 mL of concentrated HNO_3 (sp gr 1.42), mix, then add water to the 1 L mark of the volumetric flask. Water and flask should be as close as possible to the standard volumetric temperature indicated on the flask. Standardize against a known chloride standard. Check the titer at least monthly.

171. Sampling

171.1 Sample in accordance with Sections 5 to 13. Take a minimum of 12 g per test unit.

172. Preparation of Apparatus

172.1 Clean all glassware with sulfuric acid-dichromate solution, rinse thoroughly with reagent water, and reflux the glassware extraction assembly for a few minutes with reagent water. Steam other glassware with boiling reagent water. Following cleaning and rinsing, take great care not to handle the glassware directly with the hands or otherwise expose it to potential contamination. Polish the silver billet electrode with No. 4/0 emery paper at least once each day or on dull appearance. Between tests, clean the electrodes by rinsing with water and acetone.

173. Procedure

173.1 *Extraction*:

173.1.1 Cut the specimen into approximately 6-mm squares with clean scissors.

173.1.2 Weigh 4.00 ± 0.01 g of the specimen and introduce it into a 300-mL Erlenmeyer flask without allowing the specimen to contact any contaminating surface.

173.1.3 Add 100.0 ± 0.1 g of water to the flask.

173.1.4 Attach the condenser to the flask and reflux vigorously for 60 min (referee method) or for 10 min (routine method).

173.1.5 Determine the moisture content of the sample on another portion. Weigh this specimen simultaneously with the test specimen.

173.1.6 Remove the apparatus from the hot plate, cover the top of the condenser tightly with a piece of aluminum foil, and allow the entire assembly to cool to room temperature. Cooling may be facilitated by careful immersion of the flask in a container of cold water.

173.2 *Titration*:

173.2.1 Filter or decant the cooled extract solution and weigh 25.0 ± 0.1 g into a tall-form beaker. Add 125 mL of acetone, then 15 drops of 1 % HNO_3.

173.2.2 Place a stirring bar into the beaker, then place the beaker on the magnetic stirrer and adjust the stirrer speed so that the surface of the liquid is not broken.

173.2.3 Immerse the electrodes into the liquid. When the meter is stable, changing less than 3 mV/min, take an initial reading and start the titration.

173.2.4 Add the standard $AgNO_3$ solution in 0.01-mL increments and record the change in millivolts. Normally, small total amounts of solution are required for the titration, thus the use of small increments of titrant. Allow sufficient time between additions to establish a steady and equilibrium emf reading. If three or four 0.01-mL additions result in less than 1 mV/0.01 mL change, slightly larger amounts may be added until such change is observed. Addition of titrant should then be reduced to 0.01 mL per addition for completion of the titration. The end point is the point on the titration curve of millivolt versus millilitre of titrant at which the greatest change of emf per unit volume of titrant is found. Determine and record the volume of titrant required to reach the endpoint.

NOTE 31—Duplicate titrations on a single extract should agree to ±0.01 mL. Duplicate specimens from the same sample should agree to within ± 5 %, except at low chloride levels where percentage differences may be greater. Greater differences indicate errors in procedure.

173.2.5 Titrate a reagent blank consisting of 25 g of water, 125 mL acetone, and 15 drops of 1 % HNO_3.

174. Calculation

174.1 Calculate the chloride content of the specimen as follows:

$$\text{Chloride, ppm} = \frac{(A - B)N \times 35.46}{D/4} \times 10^{3} \qquad (15)$$

where:

A = 0.0025 *N* $AgNO_3$ solution for specimen end point, mL,
B = 0.0025 *N* $AgNO_3$ solution for blank end point, mL,
N = normality of the $AgNO_3$ solution, and
D = weight of specimen, oven-dry basis, g.

175. Report

175.1 Report in accordance with Section 14, except report chlorides in ppm to the next higher integer above the value found.

176. Precision and Bias

176.1 In between laboratory testing where each laboratory makes a given number of tests and reports the average, the 95 % probability limits, expressed as a percentage of the grand average, are as follows, for the referee method only:

Level of Grand Average Water-Soluble Chloride, ppm	95 % Probability Limits of Results Expressed as Percentage of Grand Average				
	Number of Tests per Laboratory				
	2	3	4	5	10
1	100	80	70	60	40
7	30	30	20	20	15
40	10	10	8	8	5

176.2 This test method has no bias because the value for chlorides is defined in terms of the test method.

Method B

177. Apparatus

177.1 Apparatus in accordance with 169.4 through 169.9, except substitute 500-mL Erlenmeyer flasks.

177.2 *Test Tubes*, heat-resistant glass, 25 mm in diameter by 100 mm in length.

177.3 *Comparator*, see Fig. 12.

178. Reagents

178.1 *Nitric Acid*, concentrated reagent, sp gr 1.42.

178.2 *Silver Nitrate Solution* (50 g/L)—Dissolve 50 g reagent grade silver nitrate in 1 L of water.

179. Procedure

179.1 Prepare the specimen in accordance with 173.1.1. Weigh 5.00 ± 0.01 g into a 500-mL Erlenmeyer flask. Determine moisture content in accordance with 173.1.5.

179.2 Add 200.0 ± 0.2 g of water. Attach the condenser and reflux as in 193.1.4, according to short-time or long-time extraction desired.

179.3 Filter or decant the extract and fill two test tubes within 6 mm of the top.

179.4 Add 2 drops of HNO_3 to each tube.

179.5 Place the tubes in the comparator and add 3 drops of $AgNO_3$ solution to one tube. Let stand for 5 min, then compare to detect turbidity using the comparator.

179.6 If the tube containing the $AgNO_3$ solution shows no turbidity, the water soluble chloride content of the specimen is less than 4 ppm.

179.7 If the tube shows turbidity, dilute another portion of the extract with an exactly equal volume of water and repeat 179.4 through 179.6.

179.8 Repeat 179.7, with progressive 1-to-1 dilutions, until turbidity does not appear.

179.9 The water and reagents, when mixed in the amounts and proportions of the test, must show a clear blank. A prepared NaCl solution of 0.1 ppm Cl^- must also show a clear blank test.

180. Calculation

180.1 Chloride content in parts per million when turbidity is last detected is more than

$$4 \times (1/\text{dilution used in test}) \tag{16}$$

180.2 Chloride content in parts per million for no turbidity is less than

$$4 \times (1/\text{dilution used in test}) \tag{17}$$

Example: A test showed perceptible turbidity after three dilutions but none after a fourth. Chloride is therefore more than

$$4 \times 1/(1/2 \times 1/2 \times 1/2) = 2^2 \times 2^3 = 2^5 \tag{18}$$

and less than

$$4 \times 1/(1/2 \times 1/2 \times 1/2 \times 1/2) = 2^2 \times 2^4 = 2^6 \tag{19}$$

181. Report

181.1 Report in accordance with Section 14.

181.1.1 Report chloride content as a range, perceptible to not perceptible. In the above example, the reported value would be 2^5 to 2^6 (32 to 64) ppm chloride.

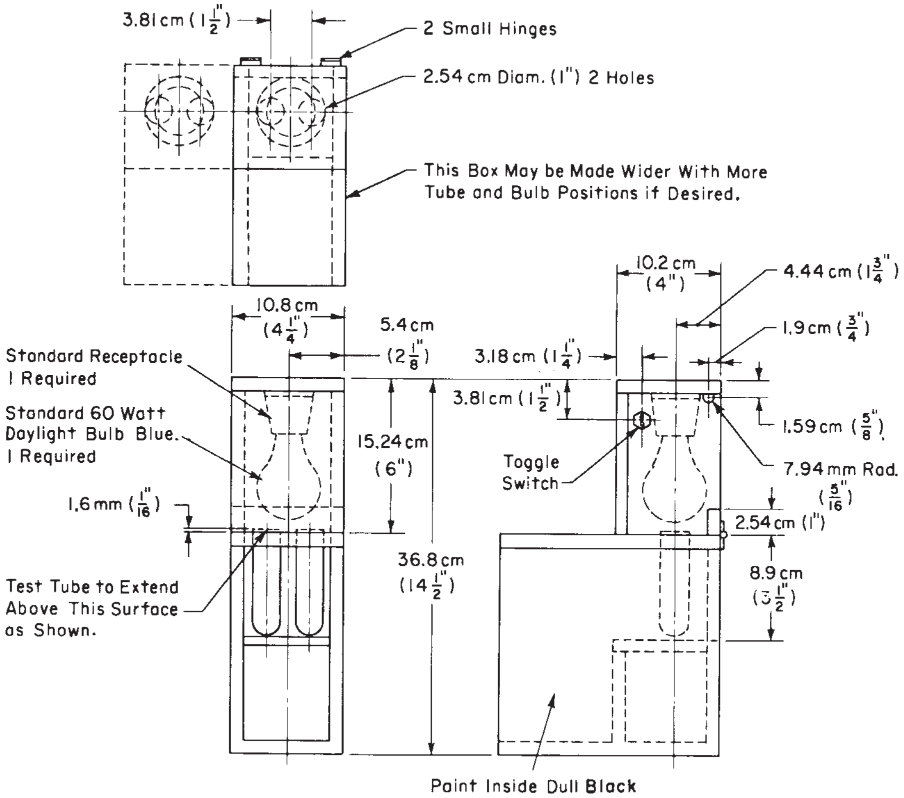

FIG. 12 Light Comparator

182. Precision and Bias

182.1 The reproducibility of this test has not been demonstrated to be sufficiently accurate below 2^4 ppm for acceptance as a standard test. Above this level, there should be no disagreement as to the range when one-to-one dilutions are used.

182.2 This test method has no bias because the value for chlorides is defined in terms of the test method.

PARTICULATE IRON

183. Scope

183.1 This test method covers the determination of the presence or absence of iron particles in electrical insulating paper up to 0.050 mm (0.002 in.) thick. This test method may be useful for papers over 0.050 mm in thickness, but may require a modification in the detection technique.

184. Summary of Test Method

184.1 Electrical insulating paper is immersed in a solution of potassium ferricyanide ($K_3Fe(CN)_6$) solution. The potassium ferricyanide forms Turnbull blue with iron as follows:

$$3\ Fe^{++} + 2\ Fe(CN)_6 \rightarrow Fe_3(Fe(CN)_6)_2\downarrow \qquad (20)$$

The paper is then removed from the solution and placed over an inspection light. Blue spots sufficiently large to indicate the presence of iron particles are then counted and reported.

185. Significance and Use

185.1 The purity of electrical insulating paper is of extreme importance in most applications and the presence of particles of metal would adversely affect the insulating properties of the paper.

186. Apparatus

186.1 *Inspection Light.*[22]

186.2 *Soaking Tray*, glass, with a cover.

187. Reagents

187.1 *Agar*.

187.2 *Potassium Ferricyanide* ($K_3Fe(CN)_6$).

187.3 *Sodium Chloride* (NaCl).

187.4 *Test Solution*—Dissolve 9.75 g of NaCl, 9.75 g of $K_3Fe(CN)_6$ and 0.975 g of agar in water and make up to 1 L. After mixing, it may be stored in a glass bottle until used.

188. Test Specimen

188.1 Take a test specimen 250 by 300 mm (10 by 12 in.) or of equivalent area from the sample obtained in accordance with Sections 5 to 13.

189. Procedure

189.1 Completely immerse the specimen in the ferricyanide solution, but avoid contact of the paper with the bottom of the tray.

189.2 After 5 ± 0.2 min remove the specimen and place it over the inspection light.

189.3 With the light on, examine the specimen for blue spots (Turnbulls blue) of 0.06 mm^2 or larger.

189.4 The presence of blue spots 0.06 mm^2 or larger is evidence of the presence of particulate iron.

189.5 Discard the solution in the try after 8 h, or when it acquires a bluish tinge, whichever occurs first.

190. Report

190.1 Report the presence or absence of particulate iron in accordance with Section 14.

191. Precision and Bias

191.1 No information is presented about either the precision or bias of this test method because the test result is nonquantitative.

PARTICULATE COPPER

192. Scope

192.1 This test method covers the determination of the presence or absence of copper particles in electrical insulation paper up to 0.050 mm (0.002 in.) in thickness. Suitability of the test method for use with thicker papers has not been established.

193. Summary of Test Method

193.1 Electrical insulating paper is immersed in sheet form in a solution of sodium diethyldithiocarbamate. The test solution reacts with any copper particles present to form brown spots. The paper is then removed from the solution and placed over an inspection light. Brown spots due to copper particles are determined and reported.

194. Significance and Use

194.1 See 185.1.

195. Apparatus

195.1 See 186.1 and 186.2.

196. Reagents

196.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

196.2 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

196.3 *Sodium Diethyldithiocarbamate*.

NOTE 32—Both the sodium diethyldithiocarbamate reagent and its solution are subject to deterioration. The reagent should be stored in a tightly sealed bottle, and solution should be prepared from the reagent periodically and tested for activity with a known source of particulate copper.

196.4 *Water*.

197. Test Solution

197.1 Mix the following reagents in the order given, agitating the solution (Note 32) after each addition until the solution is homogeneous:

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[22] I^2R's Glow Box, Model 12-20E-3, manufactured by Instruments for Research and Industry, Cheltenham, PA 19100, has been found satisfactory for this test method.

Water	500 mL
Ammonium hydroxide	43 mL
Nitric acid	9 mL
Sodium diethyldithiocarbamate	2.9 g

198. Test Specimens

198.1 See 188.1.

199. Procedure

199.1 Completely immerse the specimen in the carbamate test solution, but avoid contact of the paper with the bottom of the tray.

199.2 After 10 ± 0.2 min in the test solution, remove the specimen and place it over the inspection light.

199.3 Under illumination examine the specimen for brown spots 0.06 mm^2 or larger.

199.4 The presence of brown spots 0.06 mm^2 or larger is evidence of the presence of particulate copper.

200. Report

200.1 Report the presence or absence of particulate copper in accordance with Section 14.

201. Precision and Bias

201.1 No information is presented about either the precision or bias of this test method because the test result is nonquantitative.

SILVER TARNISHING BY PAPER

202. Scope

202.1 This test method covers the detection of the presence in papers of those contaminating compounds that will react in the presence of heat and moisture to tarnish or stain silver.

203. Significance and Use

203.1 The most common reactive contaminants which will tarnish silver are compounds of sulfur. Many reactive sulfur compounds cause progressive deterioration of the insulation resistance and loss characteristics of dielectric systems. Also, sulfides tend to react with the conductive metals in electrical apparatus and can severely reduce the efficiency of contact areas.

203.2 Insufficiently-washed kraft pulps and sulfur-containing lubricants on tools used to cut insulating shapes are among the sources of reactive sulfur in electrical insulating papers and boards.

204. Procedure

204.1 From the sample obtained in accordance with Sections 6 through 13 take specimens and test them in accordance with TAPPI T-444.

205. Report

205.1 Report in accordance with Section 14 and TAPPI T-444.

206. Keywords

206.1 absorption; acidity; air resistance; alkalinity; aqueous extract conductivity; aqeous extract pH; ash content; basis weight; board; bursting strength; chloride content; conditioning; conducting paths; density; dielectric strength; dimensions; dissipation factor; edge-tearing resistance; fiber analysis; folding endurance; grammage; heat stability; impregnation time; internal-tearing resistance; moisture content; paper; paperboard; particulate copper; particulate iron; permittivity; pH; roll dimensions; sampling; sheet dimensions; silver tarnishing; solvent-soluble content; specific gravity; surface friction; tensile properties; tearing resistance; thickness