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Standard Test Methods of Sampling and Testing Pulps to be Used in the Manufacture of Electrical Insulation[1]

This standard is issued under the fixed designation D3376; 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.

1. ~~Scope~~ Scope*

1.1 These test methods cover the sampling and testing of cellulosic pulps for use in the manufacture of electrical insulating papers and boards or in the direct application of pulp fibers as insulation to electrical conductors.

NOTE 1—The significance of any one pulp property test method, as set forth herein, should be considered with discretion depending on the product made from the pulp.

1.2 Sections on Reagents, Sampling, and Report are integral parts of each of the individual test methods that follow.

1.3 Each test method is described as being a measure of either a bulk property of the pulp or a property of a handsheet formed from the pulp.

1.3.1 Bulk characteristics determinable by these procedures appear in the following sections:

~~Procedure~~	~~Sections~~	~~ASTM Method Reference~~	~~TAPPI Method Reference~~
Procedure	Sections	ASTM Method Reference	TAPPI Method Reference
~~Aqueous Extract Conductivity~~	~~8 and 9~~	~~D202~~	~~...~~
Aqueous Extract Conductivity	8 and 9	D202	...
~~Aqueous Extract pH~~	~~10 and 11~~	~~D202~~	~~...~~
Aqueous Extract pH	10 and 11	D202	...
~~Aqueous Extractable Acidity—Alkalinity~~	~~12 and 13~~	~~D202~~	~~...~~
Aqueous Extractable Acidity-Alkalinity	12 and 13	D202	...
~~Analysis of Ash for Cations by Atomic Absorption Spectrophotometry~~	~~73—81~~	~~D1193 and D2576~~	~~...~~
Analysis of Ash for Cations by Atomic Absorption Spectrophotometry	70 – 78	D1193 and D2576	...
~~Ash Content~~	~~82—85~~	~~D202~~	~~T 413~~
Ash Content	79 – 82	D202	T 413
~~Dirt in Pulp~~	~~42 and 43~~	~~...~~	~~T 213~~
Dirt in Pulp	40 and 41	...	T 213
~~Fiber Analysis~~	~~24 and 25~~	~~D202 and D1030~~	~~...~~
Fiber Analysis	23 and 24	D202 and D1030	...
~~Fiber Length of Pulp~~	~~44 and 45~~	~~...~~	~~T 232, T 233~~
Fiber Length of Pulp	42 and 43	...	T 232, T 233

[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.

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***A Summary of Changes section appears at the end of this standard**

Procedure	Section	ASTM Method Reference	TAPPI Method Reference
~~Freeness (Canadian Standard — Freeness)~~	~~56 and 57~~	...	~~T 227~~
Freeness (Canadian Standard Freeness)	53 and 54	...	T 227
~~Kappa Number/Permanganate — Number (Substances Oxi — dizable by Permanganate)~~	~~28 and 29~~	...	~~T 236, — UM 251~~
Kappa Number/Permanganate Number (Substances Oxidizable by Permanganate)	27 and 28	...	T 236, UM 251
~~Laboratory Processing of Pulp — (Beater Method)~~	~~54 and 55~~	...	~~T 200~~
Laboratory Processing of Pulp (Beater Method)	51 and 52	...	T 200
~~Moisture in Pulp~~	~~32 – 34~~	...	~~T 210~~
Moisture in Pulp	31 – 33	...	T 210
~~Neutral Aqueous Extractable — Hardness in Pulp~~	~~16 – 23~~	~~D202, D1126, and — D2576~~	...
Neutral Aqueous Extractable Hardness in Pulp	16 – 22	D202, D1126, and D2576	...
~~Pentosan Content of Pulp~~	~~30 and 31~~	...	~~T 223~~
Pentosan Content of Pulp	29 and 30	...	T 223
~~Resistance of Pulp to Disinte — gration (Standard RPG)~~	~~46 – 53~~	...	~~T 239, — UM 252~~
Resistance of Pulp to Disintegration (Standard RPG)	44 – 50	...	T 239, UM 252
~~Shive Count~~	~~35 – 41~~	...	...
Shive Count	34 – 39	...	...
~~Solvent-Soluble Matter in Pulp~~	~~26 and 27~~	~~D202~~	...
Solvent-Soluble Matter in Pulp	25 and 26	D202	...
~~Tensile Properties~~	~~68 – 72~~	~~D202~~	...
Tensile Properties	65 – 69	D202	...
~~Water-Extractable Chlorides~~	~~14 and 15~~	~~D202~~	...
Water-Extractable Chlorides	14 and 15	D202	...

1.3.2 Handsheet characteristics determinable by these procedures appear in the following sections:

Procedure	Section	ASTM Method Reference	TAPPI Method Reference
~~Air Resistance (Porosity)~~	~~60 and 61~~	~~D202~~	~~T 205~~
Air Resistance (Porosity)	57 and 58	D202	T 205
~~Apparent Density~~	~~66 and 67~~	~~D202~~	~~T 205~~
Apparent Density	63 and 64	D202	T 205
~~Bursting Strength~~	~~62 and 63~~	~~D202 and D774/D774M~~	~~T 205~~
Bursting Strength	59 and 60	D202 and D774/D774M	T 205
~~Folding Endurance (M.I.T.)~~	~~54 and 55~~	~~D202 and D2176~~	~~T 205~~
Folding Endurance (M.I.T.)	51 and 52	D202 and D2176	T 205
~~Forming Handsheets for — Physical Tests of Pulp~~	~~58 and 59~~	...	~~T 205~~
Forming Handsheets for Physical Tests of Pulp	55 and 56	...	T 205
~~Tensile Strength~~	~~68 and 69~~	~~D202 and D828~~	~~T 205~~
Tensile Strength	65 and 66	D202 and D828	T 205

NOTE 2—Methods for Ash, Silica, selected cations from Ash, Heat Stability, α, β, and γ Cellulose, Viscosity, Total Chlorine, Tear, and Dissipation Factor

and Relative Permittivity, will be considered for addition as methods are developed.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D202 Test Methods for Sampling and Testing Untreated Paper Used for Electrical Insulation
D774/D774M Test Method for Bursting Strength of Paper (Withdrawn 2010)[3]
D828 Test Method for Tensile Properties of Paper and Paperboard Using Constant-Rate-of-Elongation Apparatus (Withdrawn 2009)[3]
D1030 Test Method for Fiber Analysis of Paper and Paperboard
D1126 Test Method for Hardness in Water
D1193 Specification for Reagent Water
D1711 Terminology Relating to Electrical Insulation
D2176 Test Method for Folding Endurance of Paper by the M.I.T. Tester (Withdrawn 2010)[3]
D2576 Method of Test for Metals in Water and Waste Water by Atomic Absorption Spectrophotometry (Withdrawn 1979)[3]
D3376 Test Methods of Sampling and Testing Pulps to be Used in the Manufacture of Electrical Insulation
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 *TAPPI Standards:*[4]

T 200 Laboratory Processing of Pulp (Beater Method)
T 205 Forming Handsheets for Physical Tests of Pulp
T 210 Weighing, Sampling, and Testing Pulp for Moisture
T 213 Dirt in Pulp
T 221 Drainage Time of Pulp
T 223 Pentosans in Wood and Pulp
T 227 Freeness of Pulp
T 232 Fiber Length of Pulp by Projection
T 233 Fiber Length of Pulp by Classification
T 236 Kappa Number of Pulp
T 413 Ash in Paper and Paperboard
T 445 Identification of Specks and Spots in Paper
T 1002 Drainage Time for Insulating Board
UM 203 Freeness of Pulp (William Tester)
UM 251 Permanganate Number of Pulp
UM 252 Resistance of Pulp and Paper Stock to Disintegration

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods and associated with electrical and electronic insulating materials use Terminology D1711.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aqueous extractable hardness, n*—the amount of calcium and magnesium present in pulp and which may be extracted by hot neutral water under prescribed conditions.

3.2.2 *hardness, n*—a characteristic of water that represents the total concentration of calcium and magnesium in the water, expressed as parts per million (ppm) $CaCO_3$.

3.2.3 *pulp, n*—a fibrous material that is made by chemical or mechanical treatment, or both, of wood, cotton, hemp, or other cellulosic fiber to achieve substantially separate fibers that are suitable for a sheet-forming process.

NOTE 3—Electrical insulation made from pulp may be papers or boards used for capacitors, transformer coils, creped papers, ~~etc.~~ and so forth. It may also be pulp applied directly onto electrical conductors.

3.2.4 *resistance to disintegration, n*—the amount of work (expressed as revolutions per gram of pulp) required under standard conditions to bring a sample of pulp to a state of complete dispersion of single fibers.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.
[3] The last approved version of this historical standard is referenced on www.astm.org.
[4] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

3.2.5 *shive, n*—a particle in pulp or paper that is a bundle of cellulosic fibers bonded together in a parallel arrangement.

NOTE 4—Dark single fibers are not to be counted as shives. Count only bundles of fibers regardless of color.

3.2.6 *shive count, n*—the quantitative expression of the concentration of shives in a quantity of pulp or paper.

3.2.6.1 *Discussion*—

For this method the shive count is restricted to the number of shives that exceed 1.5 mm in length that are present after a specified processing of the pulp to form handsheets for evaluation.

4. Summary of Test Methods

4.1 These test methods describe the specific procedures for testing the properties of pulp, both in its original bulk form and after it has been formed into a handsheet in the testing laboratory.

5. Reagents

5.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] ~~Other grades may be used,~~ It is acceptable to use other grades, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean water conforming to Specification D1193, Type III.

6. Sampling

6.1 Terminology regarding sampling and evaluation terminology shall conform to those in the sampling sections of Test Methods D202.

6.2 Obtain the sample of pulp from the lot to be evaluated in a manner that will maximize the probability that a representative sample is collected. Where practicable, use one of the sampling plans shown in Test Methods D202. Protect the material sample from contamination during handling and transporting to a laboratory for testing. The instructions for preparation of specimens are given in the sections pertaining to the individual property tests. Take the sample for moisture content in accordance with TAPPI T 210.

6.3 Condition samples in a container suitable for preventing moisture variation over the period of testing. When test specimens are drawn, determine the moisture content of the material to allow correction of weights to moisture-free equivalent weight.

7. Report

7.1 At the completion of any or all of the following tests, report the test results (as defined in 6.1) of the pulp properties with identifying units as follows:

7.1.1 Identification of the pulp sampled and tested by lot number, type, grade, ~~etc.,~~and so forth,

7.1.2 Dates of testing,

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

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

7.1.5 Indication of the variance in test measurements (as defined in 6.1) such as high, low, standard deviation, ~~etc.,~~ and so forth, and

7.1.6 Any information particular to the cited procedure.

7.2 Report the test results (as defined in 6.1) 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 limiting value. (See the rounding method of Practice E29.)

AQUEOUS EXTRACT CONDUCTIVITY

8. Significance and Use

8.1 The conductivity of the water extract of electrical grade pulp results from electrolytic impurities in the pulp ~~which may be~~ potentially present as ionizable acids, bases, salts, or a combination of these. The presence of electrolytic impurities in electrical insulation is undesirable as they tend to lower insulation resistance and have corrosion-producing tendencies under conditions of

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

applied potential. When comparing test ~~data it should be noted~~ data, note that the extract conductivity of pulps, especially those of high purity, ~~may change~~ changes with time after ~~manufacturing.~~ manufacturing in some instances. This test is useful for routine acceptance testing, the comparison of different pulps, and research work.[6]

9. Procedure

9.1 Follow Test Methods D202 except use a specimen weight equivalent to 1 g of moisture-free pulp.

AQUEOUS EXTRACT pH

10. Significance and Use

10.1 The extract pH determination measures the degree to which a pulp alters the hydrogen-hydroxyl equilibrium of pure water. The test gives a measure of the active acidity or alkalinity of the pulp extract. ~~The~~ It is possible that the presence of active acidic or alkaline contaminants in a pulp ~~may~~will result in their being incorporated into the electrical insulation made from the pulp, and can lead to a deterioration of the insulation in service. This test is useful for routine acceptance testing, the comparison of different pulps, and research work.[2]

11. Procedure

11.1 Follow Test Methods D202, except use a specimen weight equivalent to 1 g of moisture-free pulp.

AQUEOUS EXTRACTABLE ACIDITY-ALKALINITY

12. Significance and Use

12.1 The extract acidity-alkalinity determination for a pulp measures the quantity of extracted ionizable material, which alters the hydrogen-hydroxyl equilibrium of pure water. ~~The~~ It is possible that the presence of active acidic or alkaline contaminants in a pulp ~~may~~will result in their being incorporated into the electrical insulation made from the pulp, and this can lead to a deterioration of the insulation in service. This test is useful for routine acceptance testing, the comparison of different pulps, and research.[4]

13. Procedure

13.1 Follow Test Methods D202, except use a specimen weight equivalent to 1 g of moisture-free pulp.

WATER-EXTRACTABLE CHLORIDES

14. Significance and Use

14.1 ~~The~~ It is possible that the occurrence of significant amounts of chloride ion in a pulp ~~may~~will lead to the incorporation of the ion in the electrical insulation made from the pulp. ~~The~~ It is possible that the presence of chloride ions ~~may~~will adversely affect the electrical properties and service life of the insulation. This test is useful for routine acceptance testing, the comparison of different pulps, and research testing.

15. Procedure

15.1 Follow Test Methods D202, except use a specimen weight equivalent to 4 g of moisture-free pulp. For pulps with higher levels of chloride (greater than 30 ~~ppm)~~ppm), 10 min of masceration as in the above method for aqueous extract conductivity ~~may be used~~ is an acceptable way to hasten the extraction, followed by 1 h refluxing as in Test Methods D202. When the chloride content is less than 30 ppm, masceration is not permitted. The appropriate extraction time must be determined to give complete extraction of the chloride for each pulp type. Times greater than 1 h ~~may be necessary.~~ will be necessary in some instances.

NEUTRAL AQUEOUS EXTRACTABLE HARDNESS PULP

~~16. Terminology~~

~~16.1 *Definitions of Terms Specific to This Standard:*~~

~~16.1.1 *aqueous extractable hardness, n*—the amount of calcium and magnesium present in pulp and which may be extracted by hot neutral water under prescribed conditions.~~

~~16.1.2 *hardness, n*—a characteristic of water that represents the total concentration of calcium and magnesium in the water expressed as parts per million (ppm) $CaCO_3$.~~

[6] For more detailed information see *Paper and Paperboard—Characteristics,Nomenclature, and Significance of Tests, ASTM STP 60 B*, Am. Soc. Testing Mats., 1963, pp. 59–61.

16. Significance and Use

16.1 Cellulose pulps ~~may~~ contain varying amounts of aqueous extractable hardness as supplied to the purchaser. ~~The~~ It is possible that the dissolved hardness from the pulp ~~may~~will accumulate in process water used in wet-forming methods and ~~may~~will interfere with the action of process additives and affect product quality adversely.

16.2 Method A is the preferred method and shall be used for reference purposes.

METHOD A

17. Procedure

17.1 *Extraction:*

17.1.1 Prepare extracts of the pulp specimens in accordance with the Test Methods D202 method for aqueous extract conductivity, except:

17.1.2 Use a specimen weight equivalent to 2.0 g of moisture-free pulp. Determine the moisture content of the pulp sample on a separate specimen taken at the same time as the test specimen.

17.1.3 The extraction volume shall be 200 mL.

17.1.4 Run a blank determination concurrently with the test specimen determination.

17.1.5 Following extraction and filtration, collect the clear filtrate and adjust the volume to exactly 200 mL.

17.2 Determine the calcium and magnesium concentration of the extract in accordance with Test Method D2576.

18. Calculation

18.1 Calculate the hardness of the extracts as follows:

$$\text{Hardness, ppm} = 100\left[2.497\left(P_{1} - P_{a}\right) + 4.117\left(P_{2} - P_{b}\right)\right] \quad (1)$$

where:

P_1 = ppm calcium in the pulp extract,
P_a = ppm calcium in the blank,
P_2 = ppm magnesium in the pulp extract, and
P_b = ppm magnesium in the blank.

METHOD B

19. Procedure

19.1 Follow the procedure of Method A for the preparation of the extract.

19.2 Take two 100-mL aliquots of the extract and titrate for total hardness, following the "low total hardness" procedure of the nonreferee volumetric method of Test Method D1126.

20. Calculation

20.1 Calculate the hardness of the specimen extract as follows:

$$\text{Hardness, ppm} = 500\left(V_{1} + V_{2} - V_{a} - V_{b}\right) \quad (2)$$

where:

V_1 = standard EDTA solution for titration of first aliquot of extract, mL
V_2 = standard EDTA solution for titration of second aliquot of extract, mL
V_a = standard EDTA solution for titration of first blank aliquot, mL, and
V_b = standard EDTA solution for titration of second blank aliquot, mL.

21. Report

21.1 Report the results as neutral aqueous extractable hardness, ppm, expressed as calcium carbonate according to the appropriate method of Test Methods D3376.

22. Precision and Bias

22.1 The precision of this test has not been determined. No statement can be made about the bias of this test since standard material is not available.

FIBER ANALYSIS

23. Significance and Use

23.1 The fiber composition of a pulp (fiber source and pulping treatment) strongly affects the ultimate product characteristics. Fiber analysis is useful both as a specification and as a control test, and ~~may be used~~ is acceptable in referee testing or research.