Designation: D3376 − 14

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

1. 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
Aqueous Extract Conductivity	8 and 9	D202	...
Aqueous Extract pH	10 and 11	D202	...
Aqueous Extractable Acidity-Alkalinity	12 and 13	D202	...
Analysis of Ash for Cations by Atomic Absorption Spectrophotometry	70 – 78	D1193 and D2576	...
Ash Content	79 – 82	D202	T 413
Dirt in Pulp	40 and 41	...	T 213
Fiber Analysis	23 and 24	D202 and D1030	...
Fiber Length of Pulp	42 and 43	...	T 232, T 233
Freeness (Canadian Standard Freeness)	53 and 54	...	T 227
Kappa Number/Permanganate Number (Substances Oxidizable by Permanganate)	27 and 28	...	T 236, UM 251
Laboratory Processing of Pulp (Beater Method)	51 and 52	...	T 200
Moisture in Pulp	31 – 33	...	T 210
Neutral Aqueous Extractable Hardness in Pulp	16 – 22	D202, D1126, and D2576	...
Pentosan Content of Pulp	29 and 30	...	T 223
Resistance of Pulp to Disintegration (Standard RPG)	44 – 50	...	T 239, UM 252
Shive Count	34 – 39	...	...
Solvent-Soluble Matter in Pulp	25 and 26	D202	...
Tensile Properties	65 – 69	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)	57 and 58	D202	T 205
Apparent Density	63 and 64	D202	T 205
Bursting Strength	59 and 60	D202 and D774/D774M	T 205
Folding Endurance (M.I.T.)	51 and 52	D202 and D2176	T 205
Forming Handsheets for Physical Tests of Pulp	55 and 56	...	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

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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.

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

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

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, 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.

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] 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.

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[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.

[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.

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, 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, 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 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 applied potential. When comparing test data, note that the extract conductivity of pulps, especially those of high purity, changes with time after 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. It is possible that the presence of active acidic or alkaline contaminants in a pulp 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. It is possible that the presence of active acidic or alkaline contaminants in a pulp 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 It is possible that the occurrence of significant amounts of chloride ion in a pulp will lead to the incorporation of the ion in the electrical insulation made from the pulp. It is possible that the presence of chloride ions 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), 10 min of masceration as in the above method for aqueous extract conductivity 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 will be necessary in some instances.

NEUTRAL AQUEOUS EXTRACTABLE HARDNESS PULP

16. Significance and Use

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

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[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.

16.2 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 is acceptable in referee testing or research.

NOTE 5—For accurate results the analyst should have considerable training and experience. The analyst should make frequent use of standard fiber sources of known composition, or of authentic fiber samples, and be thoroughly familiar with different fibers and their behavior when treated with the various stains.

24. Procedure

24.1 Follow Test Methods D202 and D1030 using a specimen weight of 0.2 g of pulp drawn from a composite sample of pulp, equivalent to 30 g of moisture-free pulp disintegrated in 2 L of water, as in Test Method D1030 and the section on fiber analysis in Test Methods D202.

SOLVENT-SOLUBLE MATTER IN PULP

25. Significance and Use

25.1 Since pulping processes usually remove most water-soluble and volatile compounds that are also soluble in organic solvents, consider the solvent extractives in pulp to consist primarily of resin and fatty acids, their esters, waxes, and unsaponifiable substances. No single organic solvent is capable of removing all these substances, and different solvents remove different combinations. The mixture of one part (by volume) of 95 % ethanol and two parts of benzene appears to provide the most complete removal of all solvent-extractable substances in pulp.

25.2 Solvent-extractable materials, if present in sufficient quantity in electrical insulation, will lower the quality of the insulation or have a deleterious effect on the liquid compounds used in contact with insulation in various types of apparatus. Ethanol-soluble materials in capacitor paper have been found to increase the conductivity of chlorinated organic compounds, which are used as impregnants in capacitors. A combination of ether and alcohol-benzene extractives is reported to represent the quantity of pitch in wood pulp. Pitch in pulp will, in some instances, cause operating problems in the paper mill, such as the plugging of felts, wires, and so forth.

25.3 This method, with solvent or solvents specified, is acceptable for routine acceptance testing, comparing different pulps, and research tests.

26. Procedure

26.1 Test for solvent-soluble matter in accordance with Test Methods D202.

KAPPA NUMBER/PERMANGANATE NUMBER (SUBSTANCES OXIDIZABLE BY PERMANGANATE)

27. Significance and Use

27.1 The permanganate consumption of a pulp is a measure of its lignin content and is acceptable as a measure of lot-to-lot uniformity. For a given species of wood or other fibers, the permanganate consumption is a measure of the degree of cooking to which the fiber has been subjected, and can relate to the ease of refining of the pulp produced from the cooked fiber. This test is useful for control purposes, specifications, and the comparison of different pulps.

28. Procedure

28.1 *Kappa Number*—Follow TAPPI T 236.

28.2 *Permanganate Number*—Follow TAPPI UM 251.

PENTOSAN CONTENT OF PULP

29. Significance and Use

29.1 The pentosan content of a pulp strongly affects the dissipation factor-temperature relationship of electrical insulation made from it. It is also a potential indicator of lot-to-lot uniformity and is one of several factors related to the bonding power of a pulp, and the amount of energy required to refine the pulp.

30. Procedure

30.1 Follow TAPPI T 223.

MOISTURE IN PULP

31. Significance and Use

31.1 Pulp is purchased on the basis of moisture content. In addition, the moisture content is acceptable for consistency control. It is possible that the moisture content will affect the energy of repulping and the biological degradation of the pulp. This test is useful for control purposes and specifications.

32. Procedure

32.1 Follow TAPPI T 210.

33. Report

33.1 This method gives the percentage moisture content of the pulp. Report percent water and percent moisture-free fiber (equal to 100 minus percent water).

SHIVE COUNT

34. Significance and Use

34.1 Several grades of electrical insulating paper are most effectively manufactured using pulps having a low shive count. Shives in wood pulp to be used for direct application to electrical conductors can be detrimental to the insulating characteristics and strength of the insulating wall. This test is useful for control purposes, specifications, and the comparison of different pulps.

35. Apparatus

35.1 *Disintegrator, Sheet Machine, Press, Blotters, and so forth,* in accordance with TAPPI T 205.

35.2 *Steel Rule,* graduated in 0.5 mm.

35.3 *Balance,* to weigh up to 100 g with 0.1-g accuracy.

35.4 *Specimen Viewer* with white opaque glass and a fluorescent or incandescent light source.

35.5 *Transparent Cylinder,* 0.6 L.

36. Procedure

36.1 From the sample obtained as specified in Section 6, take a quantity of pulp equivalent to 30 g of moisture-free pulp. Soak this pulp in 0.5 L of water for 4 h at a temperature of 20 to 30°C.

36.2 Tear the pulp into smaller pieces (approximately 1 in. or 25 mm square) and dilute to 2.0 L. **Warning—***Tear*, do not cut the pulp.

36.3 Using the TAPPI disintegrator, disintegrate for 10 minute minimum. This time is likely to be sufficient to disperse the pulp completely. A technique for checking the dispersion is as follows:

36.3.1 Take a small sample of the slurry from the disintegrator (about 2 or 3 mL) and dilute to 0.6 L in a clear cylinder. Stopper the cylinder and mix the suspension by rotating the cylinder end over end. Observe the suspension by looking through it toward a strong light source. The suspension shall be free of clumps or agglomerates of fibers, but is allowed to contain shives. If the suspension contains clumps or agglomerates, subject the pulp to additional 5-min periods of disintegration until it is free of clumps and agglomerates.

36.4 Dilute the disintegrated pulp with water to result in a consistency of 0.3 % or 3 g/L.

36.5 Clean the sheet machine thoroughly.

36.6 Form handsheets in accordance with TAPPI T 205 and couch but do not dry the sheets.

36.7 Make at least five handsheets for viewing.

36.8 On each of five handsheets, mark out six viewing areas. Each viewing area shall be 625 mm^2. This step can be facilitated by having previously made a transparent plastic overlay grid with these areas cut out.

36.9 With the viewer and the steel rule, count the shives that exceed 1.5 mm in length that are viewed within each of the six areas. Record the total number of shives in each handsheet.

37. Calculation

37.1 Add the total shives found in all five handsheets. This sum, multiplied by 53, yields the shive count expressed as shives per square metre.

37.2 Calculate the shive count as shives per kilogram if the handsheet area is 0.02 m^2. Shives per kilogram is the product of total shives counted times 888. This multiplier is valid only for the standard handsheets in accordance with TAPPI T 205, with a grammage of 60 ± 1.2 g/m^2 and an area of 0.02 m^2.

38. Report

38.1 Report the disintegration time if more than 10 min.

38.2 Report the shive count as shives per square metre or as shives per kilogram as agreed upon between the supplier and the purchaser.

39. Precision and Bias

39.1 *Precision*—From a round-robin test involving three laboratories, a coefficient of variation between laboratories in the order of 300 was obtained, at a level of 5000 to 8000 shives/m^2.

39.2 *Bias*—No statement of bias can be made because of the unavailability of standard reference material.

DIRT IN PULP

40. Significance and Use

40.1 Dirt content is one indication of the quality of the pulp. This measure gives only a visual indication of contamination. The nature of foreign particles is very significant in determining whether the contamination is detrimental to the end-product use. TAPPI T 445 is likely to be appropriate for identification of the particulate contamination. This test is useful for control purposes and the comparison of different pulps.

41. Procedure

41.1 Follow TAPPI T 213 .

FIBER LENGTH OF PULP

42. Significance and Use

42.1 The fiber length is a means of comparing pulps. It is possible that the fiber length distribution will affect the forming characteristics, which in turn influence the physical characteristics of the end-product. This method is useful for control purposes, the comparison of different pulps, and research.

43. Procedure

43.1 Follow TAPPI T 232, T 233.

RESISTANCE OF PULP TO DISINTEGRATION (STANDARD RPG)

NOTE 6—The method described herein is essentially an adaptation of TAPPI UM 252, with several significant changes. The method is complete and no reference to UM 252 is required in its use.

44. Significance and Use

44.1 Resistance to disintegration is important in that it is a measure of repulpability. The method is useful for control purposes and the comparison of different pulps.

45. Apparatus

45.1 *Disintegrator*—TAPPI standard disintegrator in accordance with T 205; equipped with a 2-L standard vessel, timer/timer controlled power source, 1-s maximum time division.

45.2 *Handsheet Machine*—British Standard sheet machine in accordance with T 205, with couch roll, couch plate, 8 by 8-in. (200 by 200-mm) blotters, and a hot plate.

45.3 *Beakers*—Set of twelve numbered 50-mL beakers and set of twelve numbered 600-mL beakers.

45.4 *Graduated Cylinder,* 500-mL, transparent.

45.5 *Vessel,* to contain 1500 mL or more pulp, calibrated at 1500 mL.

45.6 *Sample Cup,* to hold 50 mL of pulp slurry.

45.7 *Oven,* drying, controlled at 105 ± 3°C.

45.8 *Balance,* to weigh up to 100 g with 0.1-g accuracy.

45.9 *Weighing Bottle,* to hold 10 g of pulp.

45.10 *Light Box*—Source of uniform illumination (incandescent or fluorescent) with surface sufficiently large to hold two handsheets (160-mm diameter disks).

46. Test Specimens

46.1 From the sample obtained as specified in Section 6, weigh out three specimens each equivalent to 60 g of moisture-free pulp. Determine the moisture content in accordance with Sections 31 – 33. Tear, do not cut, the test specimen from the test unit.

47. Procedure

47.1 Connect the disintegrator to the timed power outlet, and fill the disintegrator vessel with 2 L of water (25 ± 5°C). Select a disintegration period appropriate to the resistance of the pulp to disintegration.

NOTE 7—The times shown below may be used as a guide for selecting the appropriate disintegration period.

Resistance of Pulp to Dispersion	Disintegration Period
slight	15
moderate	30
high	45

47.1.1 Take one specimen and briefly immerse it in the water in the disintegrator vessel to wet it thoroughly (for example, for 15 s) in water at 25 ± 5°C. Tear the specimen into pieces about 20 mm square. If the pulp is flash dried, it is acceptable to split it so that the specimen pieces are about 2.5 mm thick.

47.1.2 Quickly put the torn specimen into the disintegrator vessel and start the disintegration/sampling sequence. Disintegrate the specimen for one period, stop the disintegrator, remove 50 mL of the pulp slurry, and put it into beaker No. 1 (50 mL). The sampling operation shall be performed in 1 min or less to minimize any soaking effects that might arise during longer time periods.

NOTE 8—On occasion, lumps of pulp will tend to remain stationary, caught in the flutings on the disintegrator vessel wall. It is necessary to ensure that any such lumps are mixed back into the bulk of the pulp slurry to ensure uniform disintegration response.

47.1.3 Repeat this sequence eleven more times, yielding a total of twelve slurry specimens.

47.2 Make up the first slurry specimen to 1.5 L with tap water (25 ± 5°C), and pour 500 mL into the transparent cylinder. Cover the cylinder with one hand, and turn end-over-end twice to mix the stock. Inspect the contents of the cylinder by viewing the slurry against a light source. Note the appearance of the slurry relative to these stages of disintegration:

(a) gross lumps of pulp
(b) few lumps/many fiber bundles
(c) few fiber bundles
(d) no fiber bundles

47.2.1 Make a handsheet from 500 mL of the diluted stock, couch it, and dry it on the hot plate (between the blotters), and mark the handsheet with the slurry specimen number. It is not necessary to press, or air-dry the handsheet, as it will only be used for visual inspection.

47.2.2 Repeat the stock dilution, inspection, and sheet marking for the eleven remaining slurry specimens.

47.2.3 Subject the remaining stock to a further disintegration of 20 min; then sample and prepare a handsheet in accordance with 47.2 and 47.2.1. This further 20-min disintegration will yield stock and handsheet specimens that are considered to be fully disintegrated reference specimens.

47.3 Place the No. 1 sheet (first slurry sample) on the light box, and inspect it. Note the appearance of the sheet as in 47.2. Compare the sheet with the reference sheet made in accordance with 47.2.3, which is considered to be fully disintegrated.

47.3.1 Compare the remaining 11 sheets, noting at which point complete disintegration is obtained, and at which no change is observed in later specimen handsheets.

47.4 Repeat 47.1.1 – 47.3.1, except 47.2.3, using disintegration period such that complete disintegration will be achieved in not less than four but not more than ten disintegration periods.

47.4.1 Test two specimen lots to conform to the above requirement.

48. Calculation

48.1 Calculate the resistance to disintegration as follows:

$$\text{Resistance to disintegration expressed in revolutions per gram} \quad (3)$$

$$(\text{RPG}) = n\ t\ S/[60 - 0.75\ (n - 1)]$$

where:

n = number of periods to complete disintegration,
t = disintegration period in minutes, and
S = speed of disintegrator (usually 3000 rpm)

The [60 – 0.75 (n – 1)] term corrects for the withdrawal of the specimen masses from the slurry.

NOTE 9—As the slurry specimens are withdrawn, the mass of pulp remaining is progressively reduced. The “first” slurry specimen experiences a RPG of:

$$1\ \ t \times 3000/60 \qquad (4)$$

The second experiences the same as the first plus:

$$1\ t \times 3000/(60 - 1.5) \qquad (5)$$

Averaging such a sum for the n^{th} slurry specimen, it will have undergone an RPG of:

$$n\,t \times 3000/60 - [(1/2)\ (n - 1)\ (1.5)], \text{ or} \qquad (6)$$

$$n\ \ t \times 3000/60 - 0.75\ (n - 1) \qquad (7)$$

49. Report

49.1 Report the mean of two resistance to disintegration determinations, giving the mean, and range to two significant figures.

49.1.1 Report if the disintegrator was run at any speed other than 3000 rpm.

50. Precision and Bias

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

LABORATORY PROCESSING OF PULP (BEATER METHOD)

51. Significance and Use

51.1 The beater method is used to evaluate the relative refining behavior of pulp. The refining behavior is characterized by the change with time of freeness (or slowness), and the development of physical sheet properties (see sections below). This test is useful for control tests, the comparison of different pulps, and research.

52. Procedure

52.1 Follow TAPPI T 200.

FREENESS (CANADIAN STANDARD FREENESS)

53. Significance and Use

53.1 Freeness (or slowness) is a measure of the drainage rate of a pulp slurry. Drainage rate is commonly used to indicate the degree of refining of a pulp.

NOTE 10—Other drainage rate measures used in the paper industry include:

(1) Schopper Riegler Freeness (Slowness) SCAN MS:65 Drainability of Pulp by Schopper Riegler Method.

(2) Drainage Time of Pulp. TAPPI Standard Method T 221.

(3) Drainage Time for Insulating Board. TAPPI Standard Method T 1002.

(4) Freeness of Pulp (Williams Tester). TAPPI Useful Method UM 203.

54. Procedure

54.1 Follow TAPPI T 227.

FORMING HANDSHEETS FOR PHYSICAL TESTS OF PULP

55. Significance and Use

55.1 The forming of handsheets permits the comparison of pulps with respect to their physical properties at different degrees of refining.

56. Procedure

56.1 Follow TAPPI T 205.

AIR RESISTANCE (POROSITY)

57. Significance and Use

57.1 Air resistance measurements on handsheets allow the comparison of pulps with respect to the development of air resistance with degree of refining. Air resistance is related to the dielectric strength and absorbency of electrical insulations.

58. Procedure

58.1 Make pulp handsheets for testing in accordance with Section 56.

58.2 Test for air resistance in accordance with Test Methods D202.

BURSTING STRENGTH

59. Significance and Use

59.1 Bursting strength has considerable use as a control test to indicate general physical strength. This method is useful for comparing the potential bursting strength of different pulps at specified degrees of refining.

60. Procedure

60.1 Make pulp handsheets in accordance with Section 56.

60.2 Test for bursting strength in accordance with Test Methods D202 and D774/D774M.

FOLDING ENDURANCE (M.I.T.)

61. Significance and Use

61.1 Folding endurance of paper is a measure of its toughness and brittleness. It is sensitive to changes in the paper, and is acceptable as a measure of thermal aging. This method is useful for comparing the potential folding strength of different pulps at specified degrees of refining.

62. Procedure

62.1 Make pulp handsheets in accordance with Section 56.

62.2 Test for folding endurance in accordance with Test Methods D202 and D2176.

APPARENT DENSITY

63. Significance and Use

63.1 Many physical and electrical properties of paper are related to the apparent density. The ease with which a sheet of desired density can be obtained is an important measure of the usefulness of a pulp. This method is useful for comparing the potential density of insulations made from different pulps of specified degrees of refining.

64. Procedure

64.1 Make pulp handsheets in accordance with Section 56.

64.2 Determine density in accordance with Test Methods D202, Method B.

TENSILE PROPERTIES

65. Scope

65.1 This test method includes procedures for determination of tensile strength, elongation, and tensile energy absorption (TEA) on pulp handsheets. It is acceptable to use the method to determine any or all of the three properties, as desired.

66. Significance and Use

66.1 This test is useful in determining the potential tensile properties of products which will be made from the pulp being studied; and the effects of refining operations on the potential tensile properties.

66.2 See the applicable sections of Test Methods D202 for further information on significance of the individual properties.

67. Procedure

67.1 Prepare pulp handsheets in accordance with Section 56.

67.2 Determine the specified tensile properties of the handsheets in accordance with the method for Tensile Properties as given in Test Methods D202, except use an initial test span of 51 ± 3 mm (2.0 ± 0.1 in.) and a speed of 6 mm ⁄min (0.25 in. ⁄min) when determining TEA.

68. Report

68.1 Report in accordance with Section 7 and the applicable sections of Test Methods D202.

69. Precision and Bias

69.1 Refer to the applicable sections of Test Methods D202.

ANALYSIS OF ASH FOR CATIONS BYATOMIC ABSORPTION SPECTROMETRY

70. Significance and Use

70.1 In some instances, the ash of pulp for electrical insulation will contain cations such as sodium, potassium, calcium, and magnesium. The presence of these cations has the potential to affect the dielectric characteristics and service performance of electrical paper made from the pulp. Cations react with some process constituents in the paper-making process, forming undesirable precipitates.

70.2 This test determines sodium, potassium, calcium, and magnesium from one dissolved ash specimen.

71. Interferences

71.1 The analysis of calcium, magnesium, potassium, and sodium in unbleached wood pulp is unlikely to be subject to interferences. Therefore, the addition of an interference suppressant for analysis is unnecessary.

72. Apparatus and Materials

72.1 Use apparatus and chemicals as specified in Test Method D2576.

73. Reagents

73.1 *Water*—Use reagent water conforming to the requirements in Specification D1193, for Reagent Water Type I, whenever water is specified in this method.

73.2 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

73.3 *Hydrochloric Acid (1 + 99)*—Add one volume of HCl (sp gr 1.19) to 99 volumes of water.

73.4 *Stock Solutions*—Purchased solutions will have the expiration date on the label. Solutions prepared in the testing laboratory are likely to be stable for one year, if kept in well-stoppered polyethylene bottles.

73.4.1 *Calcium (1 mL = 1.0 mg Ca, equivalent to 1000 mg/L Ca)*—Weigh 2.497 g of calcium carbonate ($CaCO_3$) and transfer it to a 500-mL Erlenmeyer flask. Add 10 mL of water. Pour 10 mL of HCl (sp gr 1.19) slowly down the side of the flask. Add an additional 200 mL of water, and heat until solution is complete. Cool and dilute to 1 L.

73.4.2 *Magnesium (1 mL = 0.1 mg Mg, equivalent to 100 mg/L Mg)*—Dissolve 1.0135 g of magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$) in 200 mL of water, and dilute to 1 L.

73.4.3 *Potassium (1 mL = 1 mg K, equivalent to 1000 mg/L K)*—Dissolve 1.907 g of potassium chloride (KCl) in 1 L of water.

73.4.4 *Sodium (1 mL = 1 mg Na, equivalent to 1000 mg/L Na)*—Dissolve 2.542 g of sodium chloride (NaCl) in 1 L of water.

73.5 *Standard Solutions*—Dilute the stock solutions with HCl (1 + 99) to prepare the standards to be used for calibration. Make these solutions immediately before use, since they have limited stability.

74. Calibration

74.1 Prepare at least three standard solutions containing known concentrations of each of the metal ions to be determined, by diluting the stock solutions as described in 73.5. Prepare the standards so that they bracket the expected values for the diluted unknown. The standards and the diluted unknown must be made up to fall within the linear concentration range of the element.

74.2 Atomize the standards through the instrument, following the instruction manual for the instrument, and using the following wavelength settings:

Metal	Wavelength, nm
Calcium	422.7
Magnesium	285.2
Potassium	766.5
Sodium	589.0 or 589.6

74.3 If the instrument is equipped with a direct concentration readout, adjust the readout to give the concentration of the standards.

74.4 If the instrument does not have a direct concentration readout, prepare a calibration curve by plotting on linear graph paper the absorbance against standard concentration for each standard.

75. Procedure

75.1 Ash the pulp specimen as specified in Sections 79 – 82.

75.2 Add 5 mL of HCl (1 + 1) [add 1 volume of HCl sp gr 1.19 to 1 volume of water] to the ash. Swirl the mixture to dissolve the ash.

75.3 Wash a slow, very retentive ashless filter paper with HCl (1 + 1).

75.4 Filter the ash solution through the ashless filter paper directly into a 100-mL volumetric flask. Wash the filter paper with several small aliquots of HCl (1 + 1). Dilute to volume with water.

75.5 Prepare a blank solution in accordance with 75.1 – 75.4, omitting the ash specimen.

75.6 Determine the concentrations of calcium, magnesium, potassium, and sodium in each solution by atomizing the solutions through the atomic absorption spectrophotometer. This shall be done immediately after the instrument is calibrated with the standards. The linear concentration ranges are as follows:

Metal	Linear Concentration Range, mg/L
Calcium	0.3 to 15.0
Magnesium	0.05 to 3.5
Potassium	0.04 to 4.0
Sodium	0.02 to 2.0

75.7 The measuring range can be increased by dilution of the specimen solution. In unbleached sulfate wood pulps, it is often found that dilution is unnecessary for analysis of potassium, but that for analysis of calcium, magnesium or sodium, a dilution of 1 + 19 with water is required before analysis. The lower limits of measurement are largely dependent on the equipment, as described in Method D2576.

76. Calculation

76.1 Calculate the cation contents of the pulp specimen as follows:

$$\text{Cation content, } \mu g/g = (A - B) \times D \times 100/C \tag{8}$$

where:

A = cation in unknown solution, mg/L,
B = cation in blank solution, mg/L,
C = weight of moisture-free pulp specimen, g, and
D = dilution factor.

77. Report

77.1 Report in accordance with Section 7, and include the results in micrograms per gram of moisture-free pulp for the cations that were determined.

78. Precision and Bias

78.1 The cation content of intralaboratory duplicate samples needs to agree within 10 %.

78.2 Interlaboratory results show differences of up to 30 % for magnesium, and up to 300 % for potassium, sodium, and calcium.

78.3 Bias is unknown.

ASH CONTENT

79. Significance and Use

79.1 The ash content of pulp depends upon the type of fiber, treatment at the pulp mill, and the washing of the pulp. It is a general indicator of quality.

79.2 The ash content has the potential to affect the electrical properties and the heat stability of the paper made from the pulp. This test is useful for routine acceptance testing, comparison of different pulps, and research.

80. Procedure

80.1 Determine the ash content as specified in Test Methods D202, and Method A (TAPPI T 413).

81. Report

81.1 Report in accordance with Section 7 and Test Methods D202 (TAPPI T 413).

82. Precision and Bias

82.1 The results of duplicate ash determinations are suspect if they differ by more than the following:

Weight of Ash, mg	Maximum Acceptable Difference, mg
Over 50	3
Over 20 to 50	2
Up to 20	1

82.2 Precision between laboratories is 0.13 % ash content.

82.3 Precision estimates are based on a round-robin test among five laboratories.

82.4 Bias is unknown.

83. Keywords

83.1 air resistance ash analysis; ash content; apparent density; bulk characteristics; bursting strength; cellulose; cellulosic fiber; conductivity; dirt; disintegration; extractable acidity-alkalinity; extractable chlorides; fiber analysis; fiber length of pulp; folding endurance; freeness (Canadian Standard Freeness); handsheet characteristics; handsheet machine; Kappa/Permanganate number; moisture; pentosan content; pH; pulps; shive count; solvent soluble matter; tensile strength; water

SUMMARY OF CHANGES

Committee D09 has identified the location of selected changes to this standard since the last issue (D3376 – 88 (2009)) that may impact the use of this standard.

(1) Revised 5.1 – 82.1 to eliminate non-mandatory language.
(2) Moved terms from (former) Sections 16, 35, and 46 to Section 3.

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