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Standard Test Methods for Moisture in Cellulose[1]

This standard is issued under the fixed designation D1348; 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 U.S. Department of Defense.

1. Scope

1.1 These test methods cover the determination of moisture in cellulose using two oven-drying procedures and one Karl Fischer procedure.

1.2 The test procedures appear in the following order:

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

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.*

2. Significance and Use

2.1 These test methods determine the amount of moisture contained in a cellulose sample which determines the amount of bone dry cellulose present in a sample. The actual amount of cellulose in a sample is an essential entity when using cellulose as a starting material for the production of cellulose derivatives.

3. Sampling

3.1 Cellulose in a variety of forms is sampled for moisture, and no single set of directions can be given that is applicable to all types of cellulose material. The following general considerations should be borne in mind.

3.1.1 Cellulose, either in compact form, such as wood, sheeted pulp or paper, baled cotton or baled staple rayon, or in loose form such as sawdust or chips, may have an appreciably different moisture content in sections lying relatively close together. In order to secure representative samples, therefore, a bulk sample should be made up of small portions taken from various parts of the lot and having the proper proportion of edge and center material.

3.1.2 Except for those samples taken in an atmosphere with which the sample is in equilibrium, the moisture content of the sample will begin to change immediately after it is removed from its original surroundings. This change can be reduced by taking extra layers of sheeted material and discarding a few layers from the top and bottom before weighing, folding, or rolling the sample to reduce the exposed area, and by placing small samples in cans or bottles and protecting larger samples by wrapping in rubber sheets, moistureproof cellophane, or other protective wrappings. These means do not provide continuous protection, and the test samples should be weighed as soon as possible.

3.1.3 When possible, bulk samples should be taken. These samples should weigh from 100 to 300 g (3.5 to 10.5 oz), the larger samples being taken when the moisture content is low or variable. Following the initial weighing, the bulk samples should be cut up or torn into small pieces and then mixed and allowed to stand overnight or longer in a sealed container to obtain moisture equilibrium before weighing out test specimens; or the sample may be allowed to come to approximate equilibrium with the laboratory air and reweighed to determine moisture change before weighing test specimens. The latter procedure is recommended since it permits routine weighing of samples without the use of special weighing bottles or boxes, and a series of accumulated samples can be weighed simultaneously. Predrying is very desirable on samples with a high moisture content (more than 5 % above equilibrium value). When samples have been predried, calculate the moisture lost, as follows:

$$R = [M - A/A] \times 100 \quad (1)$$

where:

R = moisture, air-dry sample basis, %,
M = original mass of the sample, g, and

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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A = air-dry mass of sample, g.

3.1.4 The apparent moisture subsequently observed when testing a predried sample must be calculated on the basis of the original sample mass in order to get the original moisture content. Calculate the original mass of the air-dried sample as follows:

$$\text{Original sample mass, g} = [A \times (100 + R)]/100 \quad (2)$$

NOTE 1—*Example*—If 1000 g (35 oz) of bulk (wet) sample on exposure to air lose 200 g (7 oz) of water, the apparent moisture content is 20 % and equivalent regain is 25 %. If an 8-g (0.28-oz) specimen of the air-dry material is taken for drying in the oven, the original mass is 8 × 1.25 = 10 g (0.35 oz).

TEST METHOD A—SPECIMEN WEIGHED IN OVEN

4. Scope

4.1 This test method for moisture determination is applicable to a variety of cellulose types and can be used in most cases where a sample does not contain nonaqueous material volatile at 105°C. The test method can be used for samples having either high or low moisture content.

5. Summary of Test Method

5.1 The specimen is heated to constant mass at 105°C in a ventilated gravity-convection oven, in a current of dry air, for a period of 2 h. If no dry air is used, the specimen is heated for 4 h.

6. Apparatus

6.1 *Oven with Built-In Weighing Equipment*—Such an oven employs a system of tared containers mounted on a table or track that can be rotated to bring specimens to a specific point in the oven, where the specimen and container can be placed on a hook or a tray connected to an outside balance. The oven must be capable of maintaining a constant temperature of 105 ± 3°C, with an average temperature of 105°C; continuous operation below 105°C is not satisfactory.

NOTE 2—In ovens having heating elements at the bottom only, and no cross circulation, the temperature of the lower shelves should be checked before they are used.

6.2 *Dry Air Stream*—Air, dried by passing through silica gel, aluminum oxide, concentrated sulfuric acid, or other suitable drying agent, should be passed through the oven at a rate sufficient to effect a complete change once every 2 min. If the air is dried by means of concentrated sulfuric acid, adequate traps must be provided. The use of sulfuric acid followed by perchloric drying agents should be avoided.

6.3 *Shallow Glass Weighing Bottles*—Bottles with ground glass stoppers, measuring 30 mm high, and having a capacity of 30 mL.

6.4 *Seamless Metal Weighing Boxes,* having a wall height, when open, preferably not over 25.4 mm (1 in.).

7. Procedure

7.1 Remove the basket, shallow pan, or other container supplied for use with the oven (Note 3). Weigh the container and place in it a specimen of 10 to 50 g (0.35 to 1.75 oz) weighed to the nearest 0.005 g. Designate this mass as M. Place the specimen in a tared container in the oven in such a manner that it can be reweighed without removal from the oven.

NOTE 3—Experience has shown that when yarn and fiber specimens are left in comparatively deep weighing bottles in the oven, relatively discordant results are obtained and the drying period is unnecessarily long. To avoid these difficulties specimens should be dried in containers that give the cellulose free access to the air. For fibrous or bulky materials to be weighed in an oven, use containers such as open wire grills or baskets. For small specimens to be weighed out of the oven, use wire screen baskets that will fit in weighing bottles, but if the specimen is powdery or tends to shed lint or fine short fibers, use a small weighing bottle or metal boxes.

7.2 Dry the specimen for 2 h at 105 ± 3°C, passing a current of dry air into the bottom of the oven during the drying period. (If dry air is not forced through the oven, dry for 4 h.)

NOTE 4—Reproducible but less accurate values (under unfavorable conditions the absolute error may be as high as 1 %) will be obtained if the current of predried air is omitted. Since the error will be identical for all similar samples in the oven at one time, the results obtained in these cases will be comparable. The magnitude of error will vary directly with the relative humidity of the air entering the oven and with the equilibrium moisture content of samples at low relative humidity. Thus, the error will be higher on regenerated cellulose than on wood pulp or cotton, but it still would not usually be greater than 0.2 %, absolute.

7.3 At the end of the specified period, cut off the flow of air and weigh the specimen without removing it from the oven. Continue drying for ½ h longer, with the normal air flow, and reweigh the specimen. Repeat the drying and weighing until the mass loss between successive weighings is not more than 0.005 g, or until the specimen shows a gain in mass. Designate this mass as D.

7.4 Additional specimens should not be placed in the oven until the first specimens have attained constant mass.

8. Calculation

8.1 Calculate moisture, as measured in the oven, as follows:

$$\text{Moisture content, \%} = [(M - D)/M] \times 100 \quad (3)$$

$$\text{Moisture, dry basis (regain), \%} = [(M - D)/D] \times 100 \quad (4)$$

where:

M = original mass of specimen, and
D = mass of oven-dry specimen.

9. Report

9.1 Report the moisture in the cellulose on either or both of the following bases:

9.1.1 On the basis of the original sample, when it is termed "moisture content," "moisture as received," or "moisture as is" basis.

9.1.2 On the basis of the oven-dry cellulose, when it is termed "moisture, dry basis" or "moisture regain."

9.2 In order to avoid confusion always use the appropriate term.

10. Precision and Bias

10.1 *Precision:*

10.1.1 Statistical analysis of intralaboratory (repeatability) test results on samples containing 5 to 15 % moisture indicates a precision of ±0.14 % at the 95 % confidence level.

10.1.2 Statistical analysis of interlaboratory (reproducibility) test results on samples containing 5 to 15 % moisture indicates a precision of ±0.2 % at the 95 % confidence level.

10.2 *Bias*—No justifiable statement can be made on the bias of the procedure for measuring moisture in cellulose because no suitable reference material exists.

TEST METHOD B—SPECIMEN WEIGHED OUTSIDE OF OVEN

11. Scope

11.1 The scope and application of this test method are the same as those of Test Method A (see Section 4).

12. Summary of Test Method

12.1 See Test Method A (Section 5).

13. Apparatus

13.1 *Oven Without Built-In Weighing Equipment*—Any ventilated oven capable of maintaining a constant temperature of 105 ± 3°C, with an average temperature of 105°C; continuous operation below 105°C is not satisfactory.

NOTE 5—In ovens having heating elements at the bottom only, and no cross circulation, the temperature of the lower shelves should be checked before they are used.

13.2 *Dry Air Stream*—See 6.2.

13.3 *Shallow Glass Weighing Bottles*—See 6.3.

13.4 *Seamless Metal Weighing Boxes*—See 6.4.

13.5 *Weighing Bottles,* glass with ground-glass stoppers, approximately 40 mm wide and 80 mm high.

13.6 *Wire Baskets*—Fitted baskets for weighing bottles made from approximately 15-mesh stainless steel, Monel, or other suitable screen. The height and depth of the basket will be determined by the weighing bottle used; the basket must slide into and out of the bottle without binding. The basket should have a solid bottom, but no top is required.

14. Procedure

14.1 If the sample is free of lint, dust, or short fibers, place approximately 10 g (0.35 oz) of the sample into a previously dried and desiccated wire basket (Note 2) contained in a dry weighing bottle. Stopper the weighing bottle, and weigh to the nearest 0.001 g. Designate this mass as *M*. Remove the basket containing the specimen from the weighing bottle and place the basket, weighing bottle, and stopper in the oven. If the specimen includes powder-like material, transfer the specimen directly into a small weighing bottle or can. Stopper the bottle and weigh to the nearest 0.001 g (*M*). Remove the stopper from the bottle and place the bottle containing the specimen and the stopper in the oven.

14.2 Dry for 2 h at 105 ± 3°C, passing a current of dry air into the bottom of the oven during the drying period. (If dry air is not forced through the oven, dry for 4 h.)

14.3 At the end of the specified period, quickly place the basket and specimen in the weighing bottle again and stopper it (or stopper directly). Remove the weighing bottle from the oven and place it in a desiccator containing an efficient desiccant, such as anhydrous calcium sulfate. Allow it to cool for 1 h, momentarily open the weighing bottle to equalize the pressure, and weigh to the nearest 0.001 g.

14.4 Return the specimen to the oven, exposing it as directed above, and dry for at least 1 h more. Place it in a desiccator to cool and weigh in accordance with 14.3. Repeat the drying and weighing until the mass loss between two successive weighings is not more than 0.005 g (or until the specimen shows a gain in mass). Designate the lowest observed mass as *D*.

14.5 When constant mass has been obtained, discard the specimen and weigh the weighing bottle (plus basket) or can. Designate this mass as *T*.

14.6 Do not place additional specimens in the oven until the first specimens have attained constant mass.

15. Calculation

15.1 Calculate moisture, as measured outside of the oven, as follows:

$$\text{Moisture content, \%} \quad [(M - D)/(M - T)] \times 100 \tag{5}$$

$$\text{Moisture, dry basis, (regain), \%} = [(M - D)/(D - T)] \times 100 \tag{6}$$

where:

M = original mass of the specimen (plus basket) and weighing bottle,
D = oven-dry mass of the specimen (plus basket), and
T = mass of the empty weighing bottle (plus basket).

16. Report

16.1 Report the moisture in the cellulose as directed in Section 9.

17. Precision and Bias

17.1 See Section 10.

TEST METHOD C—KARL FISCHER METHOD

18. Scope

18.1 This test method covers the determination of moisture in cellulose by titration with Karl Fischer reagent. The test method is applicable to all types and forms of cellulose. It is especially useful with samples containing nonaqueous material volatile at 110°C, since such substances interfere in the oven-drying methods. Anhydrides, alkalies, and large amounts of aldehydes and ketones interfere.

18.2 The Karl Fischer titration method is especially valuable where only small amounts of samples are available. The procedure lends itself to multiple determinations.

18.3 It is essential that the Karl Fischer reagent, standard water solution, and anhydrous methanol be protected from atmospheric moisture at all times. During the titration a stream of dry air or nitrogen will protect the contents of the titration flask from atmospheric moisture pick-up.

19. Summary of Test Method

19.1 An excess of Karl Fischer reagent is added to the specimen suspended in anhydrous methanol. After shaking for 15 to 20 min to extract the moisture, the excess is back-titrated with standard water solution. It is also permissible to titrate directly to the end point with Karl Fischer reagent. The end point is best detected electrometrically, but, with practice, it may be satisfactorily determined visually.

NOTE 6—To determine the moisture content of certain plastic materials the following solvents or solvent combinations may be used: chloroform, *m*-cresol, pyridine, *o*-dichlorobenzene-methanol, dioxane-methanol; dioxane-pyridine, methylene chloride-methanol (1 + 1), pyridine-methanol, and toluene-methanol.

20. Apparatus

20.1 *Buret,* automatic, 25 or 50 mL (0.85 or 1.7 oz), attached to an amber glass reservoir so that any air entering the system must pass through an efficient absorber containing anhydrous calcium sulfate (Drierite) and soda-asbestos (Ascarite).

20.2 *Pipets,* automatic or transfer, 50- and 100-mL (1.70- and 3.40-oz).

20.3 *Flasks,* or bottles, glass-stoppered, 125-mL or 250-mL (4.25- or 8.50-oz), for visual end point, or suitable enclosed reaction vessel for electrometrically determined end point.

20.4 *Bottle,* dropping, or equivalent, for weighing water for standardization of reagent.

20.5 *Electrode and Meter System,* with provision for adequate stirring, and flasks or containers adapted to the assembly if the end point is to be determined electrometrically.

20.6 *Filter,* sintered glass, coarse porosity, 30-mL (1.02-oz).

20.7 *Bottle,* weighing, low-form, 45 by 65 mm (1.8 by 2.6 in.) for drying wet pulp.

21. Reagents

21.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[2] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

21.2 *Air or Nitrogen,* under a pressure of 152.4 to 304.8 mm (6 to 12 in.) of water (1.5 to 3.0 kPa) and dried by passing through concentrated sulfuric acid (H_2SO_4, sp gr 1.84) followed by indicating grade anhydrous calcium sulfate or other suitable indicating desiccant. If the titration assembly provides adequate protection from atmospheric moisture, this will not be required.

21.3 *Karl Fischer Reagent*—Suitable reagent may be purchased from laboratory supply houses or prepared as follows:

21.3.1 Place 530 ± 5 mL (18.02 ± 0.17 oz) of dry methanol (CH_3OH) in a clean, dry, 9-L (2.34-gal) chemical-resistant glass carboy. Add 2025 ± 25 mL (68.85 ± 0.85 oz) of pyridine. Add 1270 ± 1 g (43.18 ± 0.034 oz) of iodine crystals and shake the carboy until the iodine is completely dissolved. Place the carboy in a cracked ice bath and allow to stand about 1 h.

21.3.2 Weigh on a platform scale to 25 g (0.05 lb) a cylinder of sulfur dioxide (SO_2) with chemical-resistant glass tubing attached to the outlet with polyethylene tubing. Tilt the cylinder with the outlet down.

21.3.3 Open the cylinder valve and allow the sulfur dioxide to run into the solution in the carboy. Use the chemical-resistant glass tubing, through which the SO_2 flows, to stir the solution. When 955 ± 5 g (2.1 ± 0.05 lb), as determined by difference in scale mass reading, have run in, stopper the bottle tightly.

21.3.4 Add about 4300 mL (146.2 oz) of methylene chloride (CH_2Cl_2) and mix well. If desired, the methylene chloride may be omitted and a total of 6375 mL (216.75 oz) of pyridine used. Allow the solution to stand 24 h before use. Protect from contamination by atmospheric moisture by storing in an all-glass apparatus. Standardize daily as described in Section 22.

21.4 *Methanol,* anhydrous, water content less than 0.05 %.

21.5 *Water Solution,* for use if back-titration is desired. Prepare by adding 1.0 mL (0.034 oz) of water to 1 L (0.26 gal) of anhydrous methanol. Mix well and store in an all-glass assembly protected from contamination by atmospheric moisture. Standardize as described in Section 22.

22. Standardization of Reagents

22.1 *Back-Titration*—Standardize the Karl Fischer reagent and water solution daily. It is important that the same technique and end point be used both in the standardization and in subsequent analyses. The following directions apply to the visual detection of the end point, but with obvious modification, depending upon the particular apparatus used, they may be followed for instrumental end point detection.

22.1.1 Pass a stream of dry air or nitrogen through the flasks during all additions and titrations unless the titration assembly provides protection from atmospheric moisture.

22.1.2 Add Karl Fischer reagent to 50 mL (1.7 oz) of anhydrous methanol contained in a dry 250-mL (8.5-oz) Erlenmeyer flask until a dark brown color has been reached (A_F). Back-titrate with water solution until the end point described in Section 23 has been reached (A_W). Reserve this solution, refill burets, and accurately measure approximately 15 mL (0.51 oz) of Karl Fischer reagent into this same flask (W_F). Back-titrate with water solution as described above (W_W).

22.1.3 Weigh to the nearest 0.0001 g (0.0028 oz), by difference (conveniently from a Sattler weighing pipet), from 0.15 to 0.20 g (4.2 to 5.6 oz) of water (G) (Note 6) into a dry 250-mL Erlenmeyer flask containing 50 mL of anhydrous methanol. Titrate with Fischer reagent to a dark brown color

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

(S_F), indicating an excess of 2 to 5 mL (0.068 to 0.17 oz) of the reagent. Back-titrate with water solution to the end point (S_W). If the end point should be passed, more Fischer reagent may be added and the titration with water solution continued until the exact end point has been reached.

NOTE 7—If desired, standard Rochelle salts or other material with known water of hydration, may be used instead of weighing the water directly.

22.1.4 Calculate the water equivalent, T, of the Karl Fischer reagent as follows:

$$A = W_F/W_W \quad (7)$$

where:

A = millilitres of Karl Fischer reagent equivalent to 1 mL of water solution,
W_F = millilitres of Karl Fischer reagent measured into the titrated methanol, and
W_W = millilitres of water solution required to titrate the excess Karl Fischer reagent.

$$B = A_F - (A \times A_W) \quad (8)$$

where:

B = millilitres of Karl Fischer reagent required by 50 mL of methanol,
A_F = millilitres of Karl Fischer reagent added to 50 mL of methanol, and
A_W = millilitres of water solution required to titrate the excess Karl Fischer reagent in 50 mL of methanol.

$$T = G/[S_F - (S_W \times A) - B] \quad (9)$$

where:

T = water equivalent of the Karl Fischer reagent, g/mL,
G = grams of water added (21.1.3),
S_F = millilitres of Karl Fischer reagent added to the weighed water, and
S_W = millilitres of water solution required to titrate the excess Karl Fischer reagent.

22.2 *Direct Titration:*

22.2.1 Alternatively, add from an automatic pipet or from an oven-dried pipet, 50 mL of methanol to each of four oven-dried titrating flasks or bottles. Weigh by difference to the nearest 0.1 mg, 0.10 to 0.25 g (0.035 to 0.00875 oz) of distilled water from a weighing bottle fitted with a dropper, into each of two of the flasks. Titrate the solvent-water standards directly to the end point with Karl Fischer reagent (T_1). Titrate the solvent blanks to the same end point as for the standards (T_2).

22.2.2 Calculate the water equivalent of the Karl Fischer reagent as follows:

$$T = A/(T_1 - T_2) \quad (10)$$

where:

T = water equivalent of the Karl Fischer reagent, g/mL,
A = grams of water added (22.2.1),
T_2 = millilitres of Karl Fischer reagent required for titration of solvent blank, and
T_1 = millilitres of Karl Fischer reagent required for titration of solvent and water.

22.2.3 Average the duplicate results and round off the average to three significant figures. Duplicate values of T should agree within 0.0001.

23. Procedure

23.1 Weigh to the nearest 0.0001 g an amount of sample estimated to contain from 0.10 to 0.15 g (0.0035 to 0.00525 oz) of water. For samples containing from 4 to 6 % water, a test specimen weighing from 2.5 to 3.0 g (0.0875 to 0.105 oz) is satisfactory. Transfer the weighed specimen to a dry, 250-mL Erlenmeyer flask with minimum exposure to the atmosphere and add 100 mL (3.4 oz) of anhydrous methanol. Stopper the flask and shake slowly for 15 to 20 min, or allow the stoppered flask to stand for 1 h with occasional shaking.

23.2 With an oven-dried pipet, transfer 50 mL (1.7 oz) of the supernatant liquid to a 125-mL (4.25-oz) oven-dried, glass-stoppered bottle. Exposure to the atmosphere may be minimized by covering all flask openings with rubber sheeting during transfer and titration operations. Pipets and buret tips may be inserted through a small slit in the rubber sheeting.

23.3 Titrate with Karl Fischer reagent to the same end point as in the standardization. If back-titration is to be used, rapidly titrate with Karl Fischer reagent until the solution has a dark brown color. Stopper the flask and allow to stand for 15 min. If the brown color should fade during the standing period, add more Karl Fischer reagent. Record the amount of reagent added.

23.4 Pass a stream of dry air or nitrogen through the flask and titrate with water solution until the end point has been reached, as shown by an instrument or indicated visually. In the latter case, the titrated solution will have a yellow color with just a trace of brown at this point. If the end point should be overtitrated, add more Karl Fischer reagent dropwise to the first appearance of the brown color.

23.5 Alternatively, titrate directly to the end point with Karl Fischer reagent. The visual end point is a color change from light brown to dark reddish-brown.

NOTE 8—An excellent visual standard of comparison is an 0.016 N iodine solution (prepared by mixing 15 mL (0.51 oz) of 0.1 N iodine solution and 75 mL (2.55 oz) of water). The end point is best observed by examining the solution by transmitted light from a 15-W fluorescent tube. It is essential to titrate to the same end point in the standardization and the same titration.

23.6 Run a blank on 50 mL (1.7 oz) of the methanol by titrating as described in 23.3 and 23.4 or 23.5. The titrated alcohol may be stoppered and used as an end point color standard when the end point is detected visually.

23.7 If calculation on the dry mass basis is desired, transfer the cellulose specimen to a coarse, fritted-glass crucible previously washed with methanol or with alcohol conforming to Formula 30 of the U. S. Bureau of Internal Revenue, dried at 120°C, and weighed to the nearest 1 mg. Wash, using suction, with about 15 mL of methanol or Formula 30 alcohol. Suck as dry as possible to remove all alcohol vapors.

23.8 Place the crucible on a hot plate or steam bath (be sure the surface is clean) for 3 to 5 min to flash off any remaining alcohol, and then place it in an oven at 120°C to dry for 30 min.

Cool the crucible containing the cellulose in a desiccator and weigh to the nearest 1 mg. Determine the mass of the dry pulp by difference.

24. Calculation

24.1 Calculate the percent moisture as follows:

24.1.1 *For Back-Titration Method:*

$$P = \left(\left[(C - DA - B) \times T \times R\right] / W\right) \times 100 \quad (11)$$

24.1.2 *For Direct Titration Method:*

$$P = \left(\left[(C - B) \times T \times R\right] / W\right) \times 100 \quad (12)$$

and,

$$\text{Moisture, dry basis, (regain), \%} = \left[P / (100 - P)\right] \times 100 \quad (13)$$

$$= \left[(W - Z)/Z\right] \times 100$$

where:

P = moisture content, %,
C = millilitres of Karl Fischer reagent required for titration of the sample,
D = millilitres of water solution added (23.4),
A = millilitres of Karl Fischer reagent equivalent to 1 mL of the water solution,
B = millilitres of Karl Fischer reagent required for titration of 50 mL of methanol (23.6),
T = grams of water equivalent to 1 mL of the Karl Fischer reagent,
W = grams of sample used,
R = aliquot factor (Note 8), and
Z = grams of dried cellulose.

NOTE 9—The aliquot factor is 2 when 100 mL of methanol is taken to extract the water from the cellulose and 50 mL is titrated.

25. Precision and Bias

25.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results indicates a precision of ±0.82 % at the 95 % confidence level.

25.2 *Bias*—No justifiable statement can be made on the bias of the procedure for measuring moisture in cellulose because no suitable reference material exists.

26. Keywords

26.1 electrochemical detection; Karl Fisher; moisture; oven; predrying; rayon