Tentative Methods of Test for

AMOUNT OF MOISTURE IN TEXTILE MATERIALS[1]

ASTM Designation: D 2654 – 67 T

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia, Pa. 19103.

1. Scope

1.1 These methods cover the determination of the moisture content in textile materials. These methods are applicable to all fibers, natural and man-made, and in all forms from bulk fiber or filaments to finished fabrics, subject to the limitations set forth in 1.1.1 through 1.1.3.

1.1.1 *Option 1*—Oven-drying using ambient air heated to 105 C may be used in any situation in which a simple and convenient method for routine process control or in-plant evaluation is needed. It is not recommended for jute or grease wool, or for acceptance testing in commercial transactions.

1.1.2 *Option 2*—Oven-drying using desiccated air heated to 105 C and other refinements in technique may be used as a basis for commercial transactions for all materials for which it is known that no significant quantity of nonaqueous volatile matter is present on or in the material to be tested.

1.1.3 *Option 3*—Distillation with toluene is the preferred method for jute and grease wool in any circumstance, and is the preferred method for any material where it is known or suspected that a significant quantity of nonaqueous and non-water-miscible volatile matter is present. It also has the best reproducibility of the three options and, therefore, may be preferred in case of dispute.

1.2 Blends of fibers may also be tested by these methods.

1.3 *Options 1 and 2* describe alternative procedures for weighing the dried specimens; a procedure for weighing the specimens in the oven while hot, and a procedure for sealing the specimens in a moisture-tight weighing container and cooling it in a desiccator before weighing it at room temperature.

NOTE 1—Other methods for determining the amount of moisture in textile materials are covered in ASTM Method D 2495, Moisture in Cotton (Oven-Drying Method),[2] ASTM Method D 1576, Moisture in Wool by Oven-Drying,[2] ASTM Method D 2462, Moisture in Wool by

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[1] Under the standardization procedure of the Society, these methods are under the jurisdiction of the ASTM Committee D-13 on Textile Materials, and are the direct responsibility of Subcommittee B-3 on Atmospheric Conditions and Regains. A list of committee members may be found in the ASTM Year Book.

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[2] *1969 Book of ASTM Standards*, Part 25.

Distillation with Toluene,[2] and ASTM Method D 681, Methods of Test for Jute Rove and Plied Yarn for Electrical and Packing Purposes.[2]

2. Definitions

2.1 *Moisture Content, n.*—The amount of moisture in a material determined under prescribed conditions and expressed as a percentage of the mass of the moist material, that is, the original mass comprising the dry substance plus any moisture present.

2.2 *Moisture Regain, n.*—The amount of moisture in a material determined under prescribed conditions and expressed as a percentage of the moisture-free material.

2.3 *Moisture-Free, adj.*—The condition of a material that has been exposed in an atmosphere of desiccated air until there is no progressive significant change in its mass.

NOTE 2—The term "mass" in the above definitions is the correct designation for the property commonly designated as "weight."

2.4 For definitions of other terms used in this method, refer to ASTM Definitions D 123, Terms Relating to Textile Materials.[2,3]

3. Summary of Methods

3.1 These methods include two options based on drying in an oven, and one option based on distillation with an immiscible solvent. More detailed summaries can be found in 5.1, 13.1, and 21.1.

4. Uses and Significance

4.1 The measurement of moisture content or of dry fiber weight (Note 2) is important for several reasons, including the following:

4.1.1 Large quantities of fibers and manufactured products containing some water are bought and sold on the basis of weight. The value of a particular commodity will vary over a significant range with a variation in the amount of water it contains.

4.1.2 Besides the effect of the moisture present when the material is received, the moisture present at the time of testing and subsequent handling and processing can be quite important.

4.1.2.1 Some textile fibers, particularly cellulosic fibers and wool, have physical properties that vary significantly with the amount of moisture present, such as tensile strength, crimp, torsional rigidity, etc.

4.1.2.2 Optimum conditions for processes, such as in carding, include moisture content as an important parameter.

4.1.2.3 Control of blends is sometimes critically dependent on the moisture content of the components.

4.1.2.4 Control of the mass per unit area of fabric and the linear density of yarn depends on control of moisture content.

4.1.2.5 Quantitative analyses of fiber mixtures require information on moisture present.

4.2 In these methods, three options are given, the choice depending primarily on the degree of accuracy required in the result. Oven-drying methods have the virtue of simplicity and economy, but are subject to interferences. For example, when ambient air is passed through the oven and heated to 105 C, a slight amount of residual moisture may be retained because of the relative humidity of the ambient air. When the standard atmosphere for testing textiles is heated to 105 C its relative humidity is 1.25 per cent. Under these conditions, the weight (Note 2) of any oven-dry material is greater than its weight when moisture-free. If the relative humidity of the ambient air is known, the amount of moisture retained by a specimen may be

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[3] Appears in this publication.

estimated from published data.[4] This interference may be circumvented by utilizing desiccated air as specified by the second option included in this method. However, when using either of the oven-drying options, there may be an additional loss in weight, other than water, caused by the evaporation of volatile matter, the amount depending on the characteristics and amount of any added oils or emulsions. The third option, distillation with toluene, is free of both of these types of interference, but should be used with caution if it is suspected that any steam-distillable, water soluble substances such as glycols, are present. Distillation with toluene is known to be a satisfactory method for jute where a decomposition problem exists when jute is exposed to air heated to 105 C and may also be satisfactory for any other bast fibers where a similar problem exists.

4.3 It is sometimes possible, when sampling material for moisture measurement, to select sampling units of a size which coincide with the size required by the testing method for a specimen. In these instances, the weight (Note 2) may be determined immediately, and, if the weight of the oven-dry material is to be determined, no intermediate steps are necessary. However, in other instances, it may be necessary or desirable to perform some intermediate manipulation with the material, such as compositing or sub-sampling. For such instances, in order to circumvent the problem of moisture loss or gain after sampling, provisions are made in these methods to stabilize the material in the working atmosphere, and equations are provided with correction terms to relate the measured moisture content or regain to the amount which existed at the time when the sample was taken. When the toluene distillation option is used, stabilization is necessary because a specimen cannot be loaded into the flask without considerable exposure, with possible change in the moisture present.

OPTION 1—DRYING IN AN OVEN SUPPLIED WITH AMBIENT AIR

5. Summary

5.1 A sample of the material to be tested is exposed to a stream of air, heated to 105 C, until no further loss of mass occurs. The total mass lost in this process is assumed to be moisture and is expressed either as moisture content or moisture regain (see 2.1 and 2.2).

6. Apparatus

6.1 *Oven*, ventilated and thermostatically controlled in the temperature range of 105 ± 2 C throughout the enclosure. The oven may be either the forced draft or convection type.

6.2 *Weighing Containers*, to be used if the specimens are to be weighed in the oven. These may be perforated metal baskets or shallow pans, of a size to fit the particular oven in which they are used. For specimens containing particles of foreign matter that are easily shaken out, use baskets made of or lined with wire screening fine enough to hold the trash.

6.3 *Weighing Containers*, capable of being sealed (such as glass weighing bottles) if the specimen is to be cooled in a desiccator before weighing in the ambient atmosphere.

6.4 *Weighing Containers*, to be used when specimens are to be weighed outside the oven, after cooling in a desiccator. These may be glass weighing bottles or other containers that can be sealed to prevent loss of moisture.

6.5 *Desiccator*, large enough to hold one or more weighing containers (for an alternative in which the specimens are weighed outside the oven).

6.6 *Desiccant*—Any suitable non-caus-

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[4] Toner, R. K., Bower, C. F., and Whitwell, J. C., *Textile Research Journal*, Vol. 17, No. 1, Jan. 1947, p. 7.

tic desiccant may be used provided it is dried or replaced as required for effective desiccation. Anhydrous calcium sulfate is recommended

6.7 *Sampling Containers*, capable of being sealed. Mason jars have been found to be satisfactory where the sample size is not too large. For larger samples, bags of various plastic materials may be suitable if the wall thickness is sufficient to provide a good moisture barrier (at least 4 mils (approximately 0.1 mm) for polyethylene, for example).

6.8 *Balance*, having a capacity adequate for weighing specimens and containers and a sensitivity of 0.005 g. The balance may be an integral part of the drying oven.

7. Sampling

7.1 Sampling should be engineered to accomplish the purpose for which the test is being made, taking due account of the precision required and the variability of the material being sampled.

8. Conditioning

8.1 For Option 1, specimens must not be either conditioned or preconditioned.

9. Procedure

9.1 *Preliminary Set-Up:*

9.1.1 Different materials may have different drying curves depending on the degree of exposure of all parts of a specimen to the drying atmosphere and the level of moisture present in the material. Using relatively moist material, make several preliminary runs, measuring the weight (Note 2) lost versus the time of drying so that a weight-time curve can be plotted. If weighings are performed outside the oven after cooling the specimens, use longer time intervals than would be required if the specimens were being weighed hot in the oven. Use unequal intervals of drying time to avoid achieving a false equilibrium. Plot the curves and find the time, for the slowest specimen, at which at least 98 per cent of the weight loss ultimately achieved has occurred. This will be the normal cycle for this material in this equipment. Twenty per cent of this cycle will be the time interval used for additional drying between successive weighings.

9.1.2 To save the time required to reweigh the empty containers or baskets after each use, adjust them to equal weight within ±0.005 g by grinding or filing. Identify them by numbers and record their weights. The containers must be kept clean and their weights checked regularly.

9.2 Open the sample container and quickly weigh out a specimen of the required size, if specimen-sized portions were not obtained at the time of sampling. If the specimen is adjusted to the specified weight (Note 2) ±0.005 g in not more than 30 sec after the container is opened, and the material is not more than 2 percentage points in moisture content away from the equilibrium value it would attain in the atmosphere in which the weighing is done, no appreciable error is introduced. If these conditions cannot be met, and the resulting error is not tolerable, use the stabilizing procedure described in Option 2.

9.3 *Procedure Using an Oven Balance:*

9.3.1 Place the specimen in a basket in the oven and dry at 105 ± 2 C until the change in weight (Note 2) between successive weighings is less than 0.1 percentage points when spacing the successive weighings according to the instructions given in 9.1.1. Turn off the air current in a forced draft type oven before making a weighing and block off natural air currents present.

9.3.2 Weigh the specimen and basket to the nearest 0.05 g.

9.4 *Procedure Using an Outside Balance:*

9.4.1 Place the specimen and container in the oven, uncover the container, and dry at 105 ± 2 C. If a glass weighing bottle is used, heat the stopper in the oven along with the bottle, otherwise, contraction of the bottle upon cooling may make the stopper too tight to open or may break the bottle. Dry until the change in weight (Note 2) between successive weights is less than 0.1 percentage points when spacing the successive weighings according to the directions given in 9.1.1.

9.4.2 In executing the weighings, close the weighing can or weighing bottle while it is still in the oven. Transfer the closed container to a desiccator and cover the desiccator. While the specimen and container are cooling, uncover the desiccator two or three times, raise the cover of the weighing container slightly for a fraction of a second to equalize the air pressure, and replace the cover on the desiccator (see Note 3). When the container and specimen have cooled to room temperature, weigh them to the nearest 0.005 g. Return the container and specimen to the oven. Uncover and repeat the drying, cooling, and weighing at the intervals required by the directions given in 9.1.1, until the change in weight between two successive weighings is less than 0.1 percentage points. Record the final weight and the weight of the empty container.

NOTE 3—Opening the container is necessary only when using weighing bottles with ground-glass stoppers, or other airtight containers. If it is not done, the partial vacuum created inside the bottle may make it impossible to open the bottle without breakage. A vacuum also imparts buoyancy to the container and decreases the apparent weight. With weighing cans that are not completely sealed by their lids, the air of the desiccator is able to leak in and equalize the pressure.

10. Calculations

10.1 Determine the original weight (Note 2) of the specimen.

10.1.1 If the specimen was weighed as received in the sealed container, calculate the original weight of the specimen by Eq 1 as follows:

$$W_o = W_g - W_t \quad \text{..........}(1)$$

where:

W_o = weight of specimen as received,
W_g = gross weight of specimen and container, and
W_t = weight of empty container.

10.1.2 If the specimen was adjusted to a specified weight in the laboratory, then W_o is the weight thus obtained.

10.2 Calculate the weight of the oven-dry specimen by Eq 2 as follows:

$$W_d = W_b - W_c \quad \text{..........}(2)$$

where:

W_d = weight of oven-dry specimen,
W_b = weight of specimen in basket or weighing bottle, and
W_c = weight of empty weighing container.

10.3 Calculate the moisture content by Eq 3 as follows:

Moisture content, per cent

$$= \frac{W_o - W_d}{W_o} \times 100 \quad ..(3)$$

10.4 Calculate the moisture regain by Eq 4 as follows:

Moisture regain, per cent

$$= \frac{W_o - W_d}{W_d} \times 100 \quad ..(4)$$

10.5 Calculate the moisture content or moisture regain of each specimen to the nearest 0.01 percentage points. When the mean value of several specimens is calculated, compute also the standard error of the mean. Since the true mean is quite likely to lie anywhere within the range of values bounded by the mean ± one standard error, the size of this range determines the decimal position at which variations begin to occur. Carry only one doubtful digit in stating the mean.

Example 1—The following set of measurements, 7.7, 8.1, 8.3, 8.1, 8.6, has a mean of 8.16 and a standard error of the mean of 0.148. The set of values included by ± one standard error is bounded by 8.012 and 8.308. The first place after the decimal point is therefore uncertain, and the mean should be reported as 8.2. (See ASTM Recommended Practice E 29, Indicating Which Places of Figures Are to Be Considered Significant in Specified Limiting Values,[5] for a discussion of rounding-off procedures.)

Example 2—The following set of measurements, 6.1, 12.7, 11.3, 7.6, 9.5, has a mean of 9.44 and a standard error of the mean of 1.19. The set of values included by the mean ± one standard error is bounded by 8.25 and 10.63. The first place before the decimal is therefore uncertain and the mean should be reported as 9.

11. Report

11.1 State that the tests were made as directed in ASTM Method D 2654, Option 1, and report the following information:

11.1.1 The average moisture content or moisture regain to the number of decimal places determined as described in 10.5.

11.1.2 Sample identification, kind of material, and the conditions under which the specimens were taken.

11.1.3 Whether the test was made using an oven balance or an outside balance.

12. Precision and Accuracy

12.1 From an interlaboratory test conducted in 1963 in which four laboratories measured twelve specimens, each of a nominally uniform material, the following components of variance were estimated:

12.1.1 Within-laboratory, 0.04 percentage points squared calculated on the basis of moisture content, and

12.1.2 Between-laboratory, 0.20 percentage points squared calculated on the basis of moisture content.

As a consequence, the within-laboratory precision at a probability of 0.95 is expected to be ±0.39 percentage points for a single determination and ±0.28 percentage points for the average of two determinations. If each of two laboratories measures two specimens of a uniform material, the results should be expected to agree, at a probability level of 0.95 to within ±1.30 percentage points.

12.2 The measurements for this interlaboratory test were performed by laboratories having better than average skill and extensive experience. The predictions made above, therefore, may prove to be optimistic and should be used with caution.

OPTION 2—DRYING IN AN OVEN SUPPLIED WITH DESICCATED AIR

13. Summary

13.1 A sample of the material to be tested is exposed to a stream of air which has been dried so as to contain not more than 0.01 g of water per 1000 liters and heated to 105 C until no further loss in weight (Note 2) occurs. The total weight lost in this process is assumed to be moisture and is expressed as either moisture content or moisture regain (see 2.1 and 2.2).

14. Apparatus

14.1 *Ventilated Drying Oven*, maintained at a temperature of 105 ± 2 C throughout the enclosure. The specimen shall not be subject to direct radiation from the heating elements. The oven shall be supplied with a current of air at a rate sufficient to change the air in the enclosure at least once every 4 min. The

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[5] *1969 Book of ASTM Standards*, Part 30.

current of air shall be pre-dried (less than 0.01 g of water per 1000 liters) by circulating through dehydrating units. The air shall pass freely through the specimens. The oven may be combined with a balance in which case facilities should be provided for shutting off the flow of air during the weighing.

14.2 *Weighing Containers,* to be used if the specimens are weighed in the oven. These may be perforated metal baskets or shallow pans, of a size to fit the particular oven in which they are used. For specimens containing particles of foreign matter that are easily shaken out, use baskets made of or lined with wire screening fine enough to hold the trash.

14.3 *Weighing Containers,* capable of being sealed (such as glass weighing bottles) if the specimen is to be cooled in a desiccator before weighing in the ambient atmosphere.

14.4 *Weighing Containers,* to be used when specimens are to be weighed outside the oven, after cooling in a desiccator. These may be glass weighing bottles or other containers that can be sealed to prevent loss of moisture.

14.5 *Desiccators,* large enough to hold as many weighing containers as will be used at one time (for the alternative in which the specimens are weighed outside the oven).

14.6 *Desiccant*—Any suitable noncaustic desiccant may be used provided it is re-dried or replaced as required for effective desiccation. Anhydrous calcium sulfate is recommended.

14.7 *Sampling Containers,* capable of being sealed. Mason jars have been found to be satisfactory where the sample size is not too large. For larger samples, bags of various plastic materials may be suitable if the wall thickness is sufficient to provide a good moisture barrier (at least 4 mils (approximately 0.1 mm) for polyethylene, for example).

14.8 *Balance,* having a capacity adequate for weighing specimens and containers, and a sensitivity of 0.005 g. The balance may be an integral part of the drying oven.

15. Sampling, Selection and Number of Specimens

15.1 Take a lot sample as directed in the applicable material specification, or as agreed upon by the parties interested in the test results. In the absence of any specification, take a lot sample as directed in ASTM Method D 2525, Devising a Sampling Plan for Various Forms of Wool for the Determination of Moisture Content;[2] ASTM Method D 1441, Sampling Cotton Fibers for Testing[2]; or ASTM Method D 2258, Sampling Yarn for Testing.[3]

15.2 Use extreme care to prevent gain or loss of moisture during the sampling operation and in the transfer of material to the sampling container. Weigh each portion of the sample and its container immediately after sampling. Subtract the tare weight of the container to obtain the net weight at time of sampling, W_o.

15.3 When it is possible to select a sampling unit of a size suitable for use as a specimen, proceed directly to 17. Procedure, otherwise proceed as directed in Method D 2525[2] for the reduction of the lot sample to a laboratory sample or samples, and the selection of specimens. Note that for Option 2, the instructions given in 16. Conditioning, are to be applied to the sample *before* the specimens are selected. The size of the specimen required is determined by the particular oven-drying equipment used.

15.4 Take a minimum of two specimens per laboratory sample.

16. Conditioning

16.1 Condition the previously weighed lot sample (or laboratory sample(s)) by exposure to moving air in the laboratory atmosphere in which sampling and weighing are to be done until moisture equilibrium for testing is achieved.

NOTE 4—Preconditioning is not required. Conditioning at 65 per cent relative humidity as directed in ASTM Method D 1776,[3] Conditioning Textiles and Textile Products for Testing, is acceptable and may be done if desired. An atmosphere of 65 per cent relative humidity is not, however, necessary, since the object of the conditioning for the purpose of this test is merely to stabilize the sample in order that changes in moisture will not occur while the specimens are being prepared and weighed.

16.2 Weigh the conditioned sample(s) to the nearest 0.005 g and record the net weight(s), W (Note 2).

NOTE 5—The weight, W, and the net weight at time of sampling, W_o, will be used to convert the observed moisture present in the conditioned specimen to the moisture present at time of sampling.

17. Procedure

17.1 Place the specimen(s) in the oven in a suitable container (see Note 6) and dry to constant weight (Note 2), defined as a lack of any progressive decrease in weight in excess of 0.1 percentage points as determined by three successive weighings using the procedure in either 17.1.1 or 17.1.2.

NOTE 6—Whether or not the specimen is held by a container during the drying process, all parts of the specimen must be exposed to freely moving air.

17.1.1 If the weighings of the dried specimen are to be performed with the specimen inside the oven, perform the weighings with any forced-air circulation turned off and any natural draft blocked. For time of drying intervals see 9.1.1.

17.1.2 If the weighings of the dried specimen are to be performed outside the oven, provide the specimen with an airtight container having a tight-fitting cover. At the end of the drying period, place the specimen in its container (which is also in the oven), cover the container, and remove it from the oven. Place the covered container in a desiccator, loosen the cover, and cool the specimen and container to approximately room temperature. When cooling is completed, set the cover firmly on the container, remove from the desiccator, and weigh. Replace the specimen and container in the oven and dry for an additional 20 per cent of the normal cyle (see 9.1.1). Repeat the drying, cooling, and weighing until constant weight is attained (Note 3).

18. Calculation of Results

18.1 If the specimens were dried directly from the as-sampled condition, calculate the moisture content or moisture regain of each specimen to the nearest 0.01 per cent by Eq 5 or 6 as follows:

Moisture content, per cent,

$$= \frac{W_o - W_d}{W_o} \times 100 \quad (5)$$

Moisture regain, per cent,

$$= \frac{W_o - W_d}{W_d} \times 100 \quad (6)$$

where:

W_o = weight (Note 2) of specimen as received, and

W_d = oven-dry weight of specimen.

18.2 If stabilizing and sub-sampling have been done, calculate the moisture content or moisture regain of each specimen to the nearest 0.01 per cent by Eq 7 or 8 as follows:

Moisture content, per cent,

$$= \left(1 - \frac{WW_2}{W_oW_1}\right) \times 100 \quad (7)$$

Moisture regain, per cent,

$$= \left(\frac{W_oW_1}{WW_2} - 1\right) \times 100 \quad (8)$$

where:

W_o = net weight (Note 2) of laboratory sample at time of sampling,

W = net weight of laboratory sample at time of measurement,

W_1 = net weight of specimen before drying, and

W_2 = net weight of the oven-dry specimen.

18.3 When the mean value of several specimens is calculated, compute also the standard error of the mean. Since the true mean is quite likely to lie anywhere within the range of values bounded by the mean ± one standard error, the size of this range determines the decimal position at which variations begin to occur. Carry only one doubtful figure in stating the mean (see Examples 1 and 2 under 10.5).

19. Report

19.1 State that the tests were made as directed in ASTM Method D 2654, Option 2, and report the following information:

19.1.1 The average (arithmetic mean) value of the results calculated for a particular lot, stating whether it is a moisture content or moisture regain value.

19.1.2 The number of specimens tested.

19.1.3 The range of the calculated moisture results (difference between the largest and smallest calculated result).

20. Precision

20.1 From an interlaboratory test conducted in 1964 in which each of four laboratories measured six specimens of each of five materials: a polyester, a polyamide, cotton, viscose, and wool, each test material being specially prepared to be as uniform as possible, the following components of variance were estimated.

20.1.1 Within-laboratories, 0.05 percentage points squared when weighed in the oven, calculated on the basis of moisture content.

20.1.2 Within-laboratories, 0.017 percentage points squared when weighed outside the oven, calculated on the basis of moisture content.

20.1.3 Between-laboratories, 0.103 percentage points squared when weighed in the oven, calculated on the basis of moisture content.

20.2 As a consequence, the within-laboratory precision at a probability of 0.95 is expected to be ±0.44 percentage points if weighed in the oven; ±0.25 percentage points if weighed outside the oven. The average of two determinations made in the same laboratory is expected to have a precision of ±0.31 percentage points if weighed in the oven, ±0.18 percentage points if weighed outside the oven, at a probability of 0.95.

If each of two laboratories measures two specimens of a uniform material, at a probability level of 0.95, the averages may be expected to agree to within ±0.99 percentage points squared.

20.3 The measurements for this interlaboratory test were performed by laboratories having better than average skill and extensive experience. The prediction made above, therefore, may prove to be optimistic, and should be used with caution.

OPTION 3—DISTILLATION WITH TOLUENE

21. Summary

21.1 A sample of the material to be tested is immersed in water-saturated toluene, which is heated. The distilled water vapor and solvent vapor condensed are collected in a graduated trap, wherein the water separates from the toluene and settles to the bottom. The volume of the water collected in the trap is converted to its equivalent weight (Note 2). This weight is expressed as moisture content or moisture regain (see 2.1 and 2.2).

22. Apparatus

22.1 *Erlenmeyer Flask,*[6] wide-mouth,

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[6] Corning No. 5100 or equivalent has been found satisfactory for this purpose.

1000-ml capacity (takes a No. 11 stopper).

22.2 *Distilling Receiver,*[7] Dean and Stark, 10-ml capacity, graduated in 0.1 ml.

Note 7—Illustrations of acceptable forms of the glass apparatus required by this method appear in ASTM Specifications E 123, for Apparatus for Determination of Water by Distillation.[5]

22.3 *Condenser,* Liebig, sealed, with 500-mm jacket.

22.4 *Balance,* capacity of at least 500 g with a sensitivity of 0.05 g.

22.5 *Heater,* for distillation apparatus, electrical with variable heat control and arranged so that the surface of the flask above the lowest solution level is not heated by direct radiation.

22.6 *Water Bath,* with thermostatic controls, maintained at a temperature of 23 ± 1 C.

22.7 *Sample Containers,* capable of being sealed. Mason jars have been found to be satisfactory where the sample size is not too large (up to 200 g). For larger samples, bags of various plastic materials are suitable if the wall thickness is sufficient to provide a good moisture barrier. For example, for polyethylene, a wall thickness of at least 4 mils (approximately 0.1 mm) has been found to be satisfactory.

23. Reagents

23.1 *Toluene,* purified, water-saturated, having a boiling range such that the solvent distills completely within a range of 2 C including 110.6 C. Prepare a solution of water-saturated toluene as follows: To each liter of toluene, add 50 to 100 ml of distilled water. Shake for about 5 min and allow to settle. Decant the toluene to a flask and attach a reflux condenser with a calibrated water trap. Reflux for 1 hr or until the water no longer accumulates in the trap. Assume the toluene in the flask to be water-saturated and store in glass-stoppered bottles until used. Prepare only as much water-saturated toluene at one time as will be needed for the samples already submitted.

23.2 *Potassium Dichromate Cleaning Solution*—Prepare this solution by mixing 35 ml of a saturated (at room temperature) solution of potassium dichromate with 1 liter of concentrated sulfuric acid.

24. Safety Precautions

24.1 Toluene is flammable and slightly toxic. It should be used in a well-ventilated area, for example, under a hood, to prevent accumulation of vapors.

25. Sampling, Selection, and Number of Specimens

25.1 Take a lot sample as directed in the applicable material specification, or as agreed upon by the parties interested in the test results. In the absence of any specification, take a lot sample as directed in Method D 2525,[2] Method D 1441,[2] or ASTM Method D 2258.[3]

25.2 Use extreme care to prevent gain or loss of moisture during the sampling operation and in the transfer of material to the sampling container. Weigh each portion of the sample and its container immediately after sampling. Subtract the tare weight (Note 2) of the container to obtain the net weight at time of sampling, W_0.

25.3 Follow the directions given in Method D 2525 for the reduction of the lot sample to a laboratory sample or samples and the selection of specimens. Note that for this method, the instructions in 26. Conditioning, are to be applied to the sample *before* the specimens are selected. Take specimens that weigh at least 50 g but not over 70 g.

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[7] Corning No. 3600 or equivalent has been found satisfactory for this purpose.

25.4 Take a minimum of two specimens per laboratory sample.

26. Conditioning

26.1 Condition the lot sample (or laboratory sample) by exposure to moving air in the laboratory atmosphere in which the sub-sampling and weighing are to be done until moisture equilibrium for testing is achieved (see Note 4).

26.2 Weigh the conditioned sample to the nearest 0.05 g and record the net weight W (see Note 2). This weight and the net weight at time of sampling, W_0, will be used to convert the observed moisture present in the conditioned specimen to the moisture present at time of sampling.

27. Procedure

27.1 Prior to using the equipment for this test, clean the receiver and condenser with potassium dichromate cleaning solution, rinse thoroughly with tap water, then with methyl alcohol, and dry.

NOTE 8—This cleaning operation is not needed between successive determinations in the same equipment.

27.2 Set up, as a control, a flask containing no specimen but containing 700 ml of the water-saturated toluene to which 5.0 ml of tap water have been added using a volumetric pipet. After distillation, the volume of water collected in the trap from this flask should be between 4.95 and 5.05 ml. (This range corresponds to the reading tolerance given in 27.8). If the observed volume does not come within the specified limits, tests made with this toluene supply are not satisfactory. Repeat the tests using a new supply of water-saturated toluene.

27.3 Select a specimen, determine its weight to the nearest 0.05 g, and designate this weight as W_1.

27.4 Transfer the specimen to a flask and add 700 ml of the water-saturated toluene. When the flask with the specimen and the one used as a control are prepared, connect the flasks, receivers, and condensers and place the flasks on electric heaters. Start the tap water flowing through the condensers. Add additional water-saturated toluene through the tops of the condensers until the receiver traps are full of toluene and it begins to run over into the flasks.

27.5 Heat the toluene to the boiling point and adjust the rate of distillation to 2 drops/sec.

27.6 When the water is accumulating at a rate of less than 0.1 ml/15 min, increase the distilling rate to about 4 drops/sec. Wash down the condensers by pouring water-saturated toluene in the tops of the condensers. Brush the condensers down with a nylon brush saturated with toluene, or dislodge any visible drops of water with a copper wire.

27.7 Continue the distillation until no change is observed in the location of the meniscus over a 15 min-period.

27.8 Separate the receivers containing water and toluene from the flasks and condensers. Place the receivers and their contents in the water bath which is maintained at 23 ± 1 C. After approximately 30 min, read the volume of water in the trap, V, to the nearest 0.05 ml.

28. Calculations

28.1 If the specimen weight is the same as the stabilized weight (Note 2) of the laboratory sample, calculate to the nearest 0.01 percentage points the amount of moisture as moisture content or moisture regain by Eq 9 or 10 as follows:

Moisture content, per cent,

$$= \left(1 - \frac{W_1 - V}{W_0}\right) \times 100 \quad (9)$$

Moisture regain, per cent,

$$= \left(\frac{W_0}{W_1 - V} - 1\right) \times 100 \, .. (10)$$

where:

V = volume of the water collected in the trap, in ml,

W_0 = original weight (Note 2) of the sample (at time of sampling), and

W_1 = weight of specimen.

28.2 If the specimen weight, W_1, represents only a portion of the stabilized weight of the laboratory sample, W, calculate the amount of moisture as moisture content or moisture regain by either Eq 11 or 12 as follows:

Moisture content, per cent,

$$= \left(\frac{W}{W_0}\left(\frac{V}{W_1} - 1\right) + 1\right) \times 100 \, .. (11)$$

Moisture regain, per cent,

$$= \left(\frac{W_0}{W\left(1 - \frac{V}{W_1}\right)} - 1\right) \times 100 \, .. (12)$$

where:

V = volume of the water collected in the trap, in ml,

W_1 = weight (Note 2) of the specimen tested,

W_0 = original weight of the sample from which the specimen was taken, and

W = stabilized weight of the sample from which the specimen was taken.

28.3 When the mean value of several specimens is calculated, compute also the standard error of the mean. Since the true mean is quite likely to lie anywhere within the range of values bounded by the mean ± one standard error, the size of the range determines the decimal position at which variations begin to occur. Carry only one doubtful figure in stating the mean. (See Examples 1 and 2 under 10.5.)

29. Report

29.1 State that the tests were made as directed in ASTM Method D 2654, Option 3, and report the following information:

29.1.1 The average (arithmetic mean) value of the results calculated for a particular lot, stating whether it is a moisture content or a moisture regain value,

29.1.2 The number of specimens tested,

29.1.3 The range of the moisture results (difference between the largest and smallest calculated result), and

29.1.4 Volume of water measured for the control (see 27.2).

30. Precision

30.1 An interlaboratory test conducted in 1963 in which each of six laboratories measured at least 12 specimens of each of three different materials at two different levels of moisture content yielded the following components of variance:

30.1.1 Within-laboratories, 0.184 percentage points squared calculated on the basis of moisture content.

30.1.2 Between-laboratories, 0.016 percentage points squared calculated on the basis of moisture content.

30.2 Data collected in 1965 from five different laboratories show an improved estimate of the within-laboratories component to be 0.026 percentage points squared. No additional estimates of between-laboratory variance are available.

30.3 As a consequence it is expected that the within-laboratory precision of the average of the two specimens of a uniform material will be within ± 0.23 percentage points of the "true" moisture content 95 per cent of the time. If two specimens each of a uniform material are measured by two laboratories, it is expected that the laboratory averages will agree to within 0.48 percentage points 95 per cent of the time.