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Standard Test Methods for
Moisture Content of Oil-Impregnated Cellulosic Insulation [1]

This standard is issued under the fixed designation D 3277; 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.

ϵ^1 NOTE—Editorial changes were made in April 2001.

1. Scope

1.1 These test methods cover the determination of the weight percent of water in new or aged, oil-impregnated electrical insulation. These test methods depend on solvent extraction of the water at room temperature. The range from 0.1 to 7.0 % water has been explored.

1.2 There are four test methods, A, B, C, and D. Methods A and B for thin paper and dense materials, respectively, are manual methods for solvent extraction of water from the specimens. Titration is used to determine the amount of water. Method C uses automatic titration to determine the amount of water. Method D is a direct automated method for extraction and detection of the water.

1.3 *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:*

D 1533 Test Methods for Water in Insulating Liquids (Karl Fischer Reaction Method) [2]

3. Summary of Test Methods

3.1 These test methods depend on solvent extraction of the moisture at room temperature and Karl Fischer titration (see Test Methods D 1533). For paper insulation 0.010 in. (0.25 mm) thick and less, extraction is accomplished by stirring the solvent with small pieces of insulation. In the special case of dense, thick sections, such as pressboard, the extraction rate is increased by delaminating thick sections and pulping the sample in a blender.

4. Significance and Use

4.1 Moisture has an adverse effect on the dielectric strength, dielectric loss, dc resistivity, and aging characteristics of oil-impregnated cellulosic insulating materials.

4.2 When cellulosic insulation such as paper and pressboard are impregnated with and immersed in oil, there is an interchange of moisture between the cellulose and oil until they attain equilibrium with respect to their relative saturations with moisture.

4.3 Considerable care should be taken in using these test methods to measure the water content of dry (<0.5 %) paper and board. Contamination of material by water from the surroundings during sampling and handling may be both rapid and significant in the case of dry test specimens. This is an even greater concern with cellulose insulation prior to oil impregnation.

5. Apparatus

5.1 *Karl Fischer Electrometric Titration Apparatus*.

5.2 *Magnetic Stirrer and TFE-Fluorocarbon Coated Stirring Bars*.

5.3 *Erlenmeyer Flasks*, glass-stoppered, 250-mL.

5.4 *Graduate*, glass, 100-mL.

5.5 *Büchner Funnel*, small porcelain.

5.6 *Micro-Syringe*, total capacity 0.2 mL, 0.01-mL divisions.

5.7 *Blender*, industrial type.

5.8 *Syringe*, 10 mL, ground glass.

5.9 *Drying Oven*, 110± 5°C.

5.10 *Laboratory Desiccator*.

5.11 *Analytical Balance*.

6. Reagents

6.1 *Karl Fischer Reagent*—Commercially available stabilized solution, diluted from approximately 5 mg of water per 1 mL to 2.5 to 3.0 mg of water per 1 mL by adding absolute acetone-free methanol (Methods A and B). For Methods C and D, prepare commercially available solutions for use in automatic titrators in accordance with the manufacturer's instructions.

6.2 *Titration Solvent*—Mix 2 volumes of acetone-free methanol and 1 volume of dry chloroform. Keep the solution tightly capped to prevent moisture absorption from the atmosphere.

6.3 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the Committee on Analytical Reagents of the American Chemical Society, where such specifications are

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[1] These test methods are under the jurisdiction of ASTM Committee D27 on Electrical Insulating Liquids and Gasesand are the direct responsibility of Subcommittee D27.06on Chemical Test.

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

available. Other grades may be used, provided that it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.4 *Methanol*, acetone-free with no more than 0.001 % acetone.

7. Sampling

7.1 The sampling procedure is not defined since it would be different for different apparatus, whether the samples were new or aged. Sample storage may also vary depending upon expected water content and time between sampling and testing. The assumption is made that the sample is representative of the condition the analyst is attempting to assess.

7.1.1 Dried cellulose absorbs moisture at an extremely fast rate. Precautions such as using polyethylene gloves for handling sample materials, and flushing the sample bottles with nitrogen during sampling have been found to be good practices, especially when testing for low levels of moisture in cellulose. Drying the sample bottle may cause a slight negative bias, but this would be expected to be much smaller than the positive bias that would occur if moist air were left in a sample container. Other practices, such as placing the paper immediately into a bottle containing the oil from the apparatus from which it was taken, leaving little or no air space and a high solid to liquid weight ratio, have been found to preserve the water content. The time element during which the paper sample is exposed to ambient conditions must be minimized and kept under several minutes when low (<1 %) water contents are expected. Another method of preservation that has been used is to wrap large sections of paper in plastic wrap followed by a layer of foil wrap.

8. Standardization of Reagent by Distilled Water (Test Methods A and B)

8.1 Add about 100 mL of solvent (chloroform-methanol) to the clean titration flask. Turn on the indicating circuit and magnetic stirrer and adjust the potentiometer to maximum deflection. (Check the battery every day.) Then add the Karl Fischer reagent in large amounts to neutralize the water present in the solvent. At first the needle will deflect due to local concentrations of the unreacted reagent about the electrodes, but will fall back to near the original position. As the end point is reached, the needle will fall back more and more slowly after each addition of Karl Fischer reagent. The end point is reached when, after addition of a single drop of reagent, the needle remains at rest near the position of maximum deflection for 30 s.

8.2 Partially fill a small-volume syringe (such as a Gilmont micrometer syringe of 0.2-mL capacity) with distilled water.

8.3 Weigh the syringe and its contents to the nearest 0.1 mg on an analytical balance.

8.4 Deliver from 0.03 to 0.05 mL of water into the neutralized solvent.

8.5 Reweigh the syringe to determine the exact weight of water added to the neutralized titration solvent.

8.6 Reneutralize the solution by titrating with the Karl Fischer reagent.

8.7 Calculate the water equivalent of the Karl Fischer reagent (grams of water per millilitre of Karl Fischer reagent) by dividing the weight of water added, in grams, by the volume in millilitres of Karl Fischer reagent required for titration of the added water.

9. Procedure

9.1 *Test Method A (0.010 in. (0.254 mm) or Thinner Paper)*:

9.1.1 Proceed in accordance with 8.1.

9.1.2 Dry the solvent containing more than 50 ppm of water by weight, which may be achieved by numerous techniques. One technique is to pass the solvent through a 30 by 1½-in. (76 by 4-cm) column containing 3A molecular sieve dried at 250°C under vacuum for at least 4 h. Purging with dry nitrogen instead of using a vacuum and the use of other temperatures can also achieve properly prepared molecular sieve material. Regardless of the test method used, test the dried solvent using a Karl Fischer apparatus to verify that the water content is less than 50 ppm by weight. Store the dried solvent under nitrogen.

9.1.3 Add 100 mL of titration solvent to each of two Erlenmeyer flasks which have previously been pre-dried for a minimum of 4 h at 125°C. Place a dry TFE-fluorocarbon coated stirring bar (about 1½ in. (38 m) long by 5⁄16 in. (8 mm) in diameter) in each flask.

9.1.4 To one flask, add 2 to 3 g of paper sample to be analyzed, cut into strips ½ by 1½ in. (13 by 38 mm) or smaller.

9.1.5 Insert a stopper in each flask and agitate contents for 40 to 50 min.

9.1.6 Decant the solvent from the flask containing the paper strips into the titration chamber of the Karl Fischer apparatus and titrate.

9.1.7 Place the paper strips in a weighing dish, dry for at least 4 h at 110°C, cool to room temperature in a desiccator, and weigh. This weight, minus the tare, is the specimen weight.

9.1.8 Repeat 9.1.6 for the flask that did not contain any paper. This is the blank.

9.1.9 Determine percent weight of water in the paper as follows:

$$\text{Water, wt \%} = [([1.03\ V_2 - V_1]F) \times 100]/W \tag{1}$$

where:

1.03 = correction factor for the loss of solvent during this analytical procedure due to adsorption of solvent into the paper,

V_2 = Karl Fischer reagent required for titration of the solvent from the flask containing the paper strips, mL,

V_1 = Karl Fischer reagent required for titration of the solvent from the flask containing no paper, mL,

F = water equivalent of the Karl Fischer reagent (grams of water per millilitre of Karl Fischer reagent), and

W = specimen weight, g.

9.1.10 Standardize the Karl Fischer reagent daily in accordance with Section 8.

9.2 *Test Method B (Dense Cellulosic Materials Thicker than 0.010 in. (0.254 mm))*:

9.2.1 Proceed in accordance with 8.1.

9.2.2 Dry the solvent containing more than 50 ppm of water by weight, which may be achieved by numerous techniques. One technique is to pass the solvent through a 30 by 1½-in. (76 by 4-cm) column containing 3A molecular sieve dried at 250°C under vacuum for at least 4 h. Purging with dry nitrogen instead of using vacuum and the use of other temperatures can also achieve properly prepared molecular sieve material. Regardless of the test method used, test the dried solvent using a Karl Fischer apparatus to verify that the water content is less than 50 ppm by weight. Store the dried solvent under nitrogen.

9.2.3 Add 100 mL of titration solvent to a pre-dried stainless steel blender cup of about 350-mL capacity.

9.2.4 Delaminate thick samples to 30-mil (0.76-mm) maximum thickness and cut into ½ by ½-in. (13 by 13-mm) pieces and place in the blender cup. Use approximately 2 to 3 g of sample.

9.2.5 Cover and pulp at a slow speed for 5 min.

9.2.6 Filter off 50 mL of solvent through a pre-dried Büchner funnel into a pre-dried graduated cylinder. (No filter aid is necessary.) If using an automatic titrator, proceed to 9.2.12.

9.2.7 Pour the solvent into the titration chamber of the Karl Fischer apparatus and titrate.

9.2.8 Filter the remainder of the pulped sample, wash with chloroform, oven-dry for at least 4 h at 110°C, then cool to room temperature in a desiccator, and weigh. Verify the oven drying time by determining the time to achieve constant weight for a given material. This weight, minus the tare, is the specimen weight.

9.2.9 Repeat 9.2.3 and 9.2.5 with no sample in the test cup.

9.2.10 Repeat 9.2.6 and 9.2.7 using the entire 100 mL of solvent. This is the blank.

9.2.11 Determine the percent weight of water in the cellulosic insulation as follows:

$$\text{Water, weight \%} = ([(2V_2 - V_1)F] \times 100)/W \quad (2)$$

where:

V_2 = volume of Karl Fisher reagent required for titration of the sample, mL,
V_1 = volume of Karl Fisher reagent required for titration of the blank, mL, and
W = dry weight of pulped cellulose, g

9.2.12 Using a 10-mL ground glass syringe, transfer 10 mL of the solvent to a Karl Fisher automatic titration apparatus, which is set to read micrograms and has an extraction time of 0.5 to 1 min. Titrate the sample in accordance with Test Methods D 1533. Repeat this step to obtain a second reading, and use the average of the two in subsequent calculations.

9.2.13 Proceed as in 9.2.8.

9.2.14 Repeat 9.2.3 and 9.2.5 with no sample in the test cup. Test as directed in 9.2.12. This is the blank.

9.2.15 Determine the percent weight of water in the paper as follows:

$$\text{water, weight \%} = \frac{10\ (W_S - W_B)}{W \times 10^{4}} \quad (3)$$

where:

W_S = micrograms of water titrated in the sample,
W_B = micrograms of water titrated in the blank, and
W = dry weight of pulped cellulose, g.

9.3 *Test Method C (Automatic Titration of Extraction Solvent; 0.01 in. or Thinner Paper)*:

9.3.1 Add a measured amount between 50 to 100 mL of dried extraction solvent (2:1 methanol:chloroform) to each of three 250-mL Erlenmeyer flasks dried for at least 4 h at 125°C. Record the exact amount of solvent added to each flask.

9.3.1.1 Dry the solvent containing more than 50 ppm of water by weight, which may be achieved by numerous techniques. One technique is to pass the solvent through a 30 by 1½-in. (76 by 4-cm) column containing 3A molecular sieve dried at 250°C under vacuum for at least 4 h. Purging with dry nitrogen instead of using vacuum and the use of other temperatures can also achieve properly prepared molecular sieve material. Regardless of the test method used, test the dried solvent using a Karl Fischer apparatus to verify that the water content is less than 50 ppm by weight. Store the dried solvent under nitrogen.

NOTE 1—It may be desired when measuring small concentrations of water in cellulose to add the solvent to the sample bottles immediately after sampling. The extraction can then be performed in the sample container which has been dried for at least 4 h at 125°C. Blank runs should then be performed on similar containers.

9.3.2 Place a dry TFE-fluorocarbon coated (2 in.) stir bar in each flask.

9.3.3 Add 0.2 to 0.5 g of the paper sample cut into small strips to two of the flasks labeled as #1 and #2. Label flask 3 as "Blank". Large sample size to solvent ratios should be used for small concentrations in cellulose samples.

9.3.4 Stopper and stir the samples in the three flasks for 45 min. Remove flasks from stirrer.

9.3.5 Using a 10-mL ground-glass syringe, transfer 5 mL of extract from each flask to a Karl Fisher automatic water titration apparatus, which is set to read micrograms and has an extraction time of 1 min. Titrate the sample following the procedures given in Test Methods D 1533. Repeat this step to obtain a second value and use the average of the two results in the subsequent calculation. Other volumes of extract can be titrated, provided that values obtained are sufficiently above background levels and that the calculation is modified by using the appropriate value for V_E.

9.3.6 Filter the paper samples from the flasks through tared, dried (4 h at 125°C) filter crucibles. Dry the paper samples for 4 h at 110°C. Cool to room temperature in a desiccator and weigh. This weight less the tare weight is the paper sample weight.

9.3.7 Determine the percent weight of water in the paper as follows:

$$\text{Water, weight \%} = \frac{V_E(W_S - W_B)}{5\ W \times 10^{4}} \quad (4)$$

where:

V_E = total volume of extraction solvent,
W_S = micrograms of water titrated in the sample,
W_B = micrograms water titrated in the blank, and
W = dry weight of the cellulose, g.

9.4 *Test Method D (Direct Extraction in Automatic Titration Apparatus of 0.01 in. or Thinner Paper)*:

NOTE 2—This test method may not work for all automatic titration solutions.

9.4.1 Add a single strip of the paper sample to the automatic titration vessel (set to read micrograms) and stir (extract) and titrate for an experimentally determined time period; normally 10 to 15 min is adequate for about 0.2 to 0.5-g sample sizes. To determine the extraction interval, the extreme range of cellulose weight and water contents expected should be tested to find the time required to achieve a titration end point. There should be good comparison between Test Methods C and D if adequate extraction time is provided. It is recommended that Test Method C be used to verify the extraction efficiency of the direct extraction method.

NOTE 3—The size of the paper sample strip is determined by the size of the titration vessel and its sample port diameter. The strip should be as large as possible and still fit easily through the sample port and be completely submerged in vessel solvent. The size of the paper will also affect the time to completely extract the water.

9.4.2 Stop stirring the solution in the titration vessel and remove the sample with clean, dry forceps. Rinse the sample with dry methanol/chloroform solution (2:1 by volume methanol chloroform) prepared in accordance with 9.3.1.

9.4.3 Place the cellulose strip on a glass dish and dry for 4 h at 100°C. Cool to room temperature in a desiccator and weigh.

9.4.4 Determine background moisture (blank) by titrating for the same amount of time as performed for samples.

9.4.5 Determine the percent weight of water in the paper as follows:

$$\text{Water, weight } \% = \frac{(W_S - W_B)}{W \times 10^{4}} \quad (5)$$

where:

W = dry weight of the cellulose, g
W_S = micrograms of water titrated in the sample, and
W_B = micrograms of water titrated in the blank.

NOTE 4—If desired, the water may be extracted from the cellulose by the previous Test Methods A or B, and then the water content of the extract determined by Test Methods D 1533, Method A, B, or C, with the appropriate adjustments being made in the calculations.

NOTE 5—Some automatic Karl Fischer titrators are capable of calculating percent or parts per million and subtracting blank values with subsequent calculations carried out automatically. Consult the instrument manual for specifics.

10. Report

10.1 Report the following information:

10.1.1 ASTM designation D3277,

10.1.2 Test Method used (A, B, C, or D),

10.1.3 Description of test specimen,

10.1.4 Specimen weight, and

10.1.5 Percent water by weight.

11. Precision and Bias

11.1 Due to the difficulty in obtaining a standard sample of moist cellulose insulation, no precision or bias statement can be made about these test methods. Typical data from several laboratories is being developed for use in a repeatability statement.

12. Keywords

12.1 cellulose; insulating oil; moisture; oil; paper; water