



Standard Test Method for Moisture in Plastics by Coulometric Regeneration of Phosphorus Pentoxide¹

This standard is issued under the fixed designation D 4019; 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 Department of Defense.

1. Scope

1.1 This test method is used for the determination of moisture in most plastics. Plastics containing volatile components, such as monomers and plasticizers, may contaminate the electrolytic cell, used in the test, causing incorrect results.

1.2 This test method is suitable for measuring moisture over the range from 0.005 % to almost 100 %.

1.3 The values stated in SI units are to be regarded as the standard.

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.*

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

- D 1193 Specification for Reagent Water²
- D 1898 Practice for Sampling of Plastics³

3. Summary of Test Method

3.1 The sample is weighed and placed in an oven. The oven heat vaporizes the moisture, which is transported to the electrolytic cell by the nitrogen carrier gas. The electrolytic cell has a thin layer of phosphorus pentoxide deposited between two helically-wound electrodes which reacts with the moisture to form phosphoric acid. The phosphoric acid is electrolyzed by a current flowing between the electrodes. The electrolysis changes the phosphoric acid to hydrogen and oxygen, which gases are vented with the carrier gas, coulometrically regener-

ating the phosphorus pentoxide. The current required to completely regenerate the P_2O_5 is integrated and displayed as the total micrograms of water present.

4. Significance and Use

4.1 Moisture can seriously effect the processability of plastics. High moisture content may cause surface imperfections (that is, splay or bubbling) or degradation by hydrolysis. Low moisture (with high temperature) may cause solid phase polymerization.

4.2 The physical properties of plastics may be greatly affected by the moisture content.

4.3 Polymers, such as 6 nylon, that contain high-boiling materials such as plasticizers or high levels of monomer may contaminate the electrolytic cell. This will cause the cell to become unresponsive, requiring it to be cleaned and recoated.

5. Apparatus

5.1 *Moisture Analyzer*⁴, consisting of:

5.1.1 An oven capable of heating the sample to at least 250°C.

5.1.2 A bypass valve system for the saturation prevention of electrolytic cell, needed on one type of instrument.

5.1.3 An electrolytic cell for trapping and electrolyzing the moisture.

5.1.4 Electronics for converting the electrolysis current to digital data.

5.1.5 Digital display for presenting the digital data as micrograms of water.

5.2 *Sample Holders*, made from nickel, 316 stainless steel, glass, or other nonreactive material.

6. Reagents

6.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.01).

In 1994, the title was changed to reflect the scope, the instrument reference in Footnote 4 was updated, and keywords were added.

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 08.01.

⁴ Model 903 Moisture Evolution Analyzer, available from TA Instruments, Inc., 109 Lukens Drive, New Castle, DE 19720, and the Model W/Type LBY Moisture Analyzer, available from MEECO, Inc., 250 Titus Ave., Warrington, PA 18976, have been found satisfactory for this purpose.

all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type III reagent water, as defined in Specification D 1193.

6.3 *Acetone* ($\{CH_3\}_2CO$).

6.4 *Nitrogen* (N_2), less than 5 $\mu\text{g/g}$ (5 ppm) water.

NOTE 2—The nitrogen carrier gas should be supplied to the instrument through metallic, fluorocarbon, or other moisture-impermeable tubing. Use of other tubing may not permit the instrument to be properly zeroed because of atmospheric moisture permeating into the instruments.

6.5 *Phosphoric Acid* (H_3PO_4), 85 %.

6.6 *Sodium Tartrate* ($\{Na_2C_4O_6 \cdot 2H_2O\}$).⁶

6.7 *Potassium Tartrate* ($\{K_2C_4O_6 \cdot 1/2H_2O\}$).⁶

6.8 *Sodium Tungstate* ($\{Na_2WO_4 \cdot 2H_2O\}$).⁶

6.9 *Nitric Acid* (HNO_3).

7. Sampling

7.1 Unless otherwise agreed upon between interested parties or described in a specification, the material shall be sampled in accordance with the General and Specific Sampling Procedures Sections of Practice D 1898.

8. Test Specimen and Sample

8.1 Samples may be in many forms, such as molding powder, fabricated shapes, or molded items.

8.2 Specimens may be cut from fabricated shapes or molded items.

8.3 Since the specimen size is small, 10 g or less (see 10.4.2), care should be exercised to ensure that the specimen is representative of the sample.

8.4 Hygroscopic resin samples must be protected from the atmosphere.

9. Calibration and Standardization

9.1 The calibration factor is determined by using materials with known water content.

9.2 Four methods are described for use with the calibration materials: (1) water, (2) sodium tartrate dihydrate, (3) sodium tungstate dihydrate, and (4) potassium tartrate. The recommended calibration material is sodium tungstate dihydrate, which is stable (in the anhydrous form) to temperatures above 1000°C. The MEECO recommended calibration material is potassium tartrate hemihydrate which is stable to temperatures up to 200°C. Tartrates can decompose above 220°C and can cause deterioration of the detector.

9.3 *Method for Use with Water*—A glass capillary (micropipet) is used to measure a known mass of water, typically 2 mg.

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 The reagent is used for standardization purposes only.

9.3.1 Prepare analyzer for use as described in Section 10.

9.3.2 Fill the micropipet by holding it at midpoint with a pair of tweezers and dipping lightly in distilled water.

9.3.3 Remove the moisture from the sides of the pipet with absorbent tissue. Take care not to touch the end of the pipet with the tissue because it will absorb the moisture from the capillary.

9.3.4 After filling the pipet, place it in the sample holder and set the holder in the oven.

9.3.5 Run the analysis and record the water counter reading as standard reading, *R_{std}*. Use an analysis time of 20 min and a temperature of 150°C.

9.4 *Method for Use with Sodium Tartrate Dihydrate*—This method uses sodium tartrate dihydrate with a theoretical water content of 15.66 % as the basis of standardization.

9.4.1 Prepare the analyzer for use as described in Section 10.

9.4.2 Weigh to the nearest 0.1 mg, 10 to 13 mg of sodium tartrate dihydrate and place in the sample holder.

9.4.3 Place sample holder and contents in the oven.

9.4.4 Same as 9.3.5.

9.5 *Method for Use with Sodium Tungstate Dihydrate*—This method uses sodium tungstate dihydrate with a theoretical water content of 10.92 % as the basis of standardization.

9.5.1 Same as 9.4.1.

9.5.2 Same as 9.4.2 except use 15 to 19 mg.

9.5.3 Same as 9.4.3.

9.5.4 Same as 9.4.4.

9.6 *Method for Use with Potassium Tartrate Hemihydrate*—This method uses potassium tartrate hemihydrate with a theoretical water content of 3.83 % as the basis of standardization.

9.6.1 Same as 9.4.1.

9.6.2 Same as 9.4.2 except use 25 to 30 mg.

9.6.3 Same as 9.4.3.

9.6.4 Same as 9.4.4.

9.7 Calculate the calibration factor, *K*, in micrograms per microgram reading, as follows:

$$K = \frac{F \times W_{std} \cdot 1000}{(R_{std} - R_B)}$$

where:

K = calibration factor in micrograms per microgram ($\mu\text{g}/\mu\text{g}$),

F = fraction of standard material attributed to water:

water method *F* = 1.00

sodium tartrate method *F* = 0.1566

sodium tungstate method *F* = 0.1092 ,

W_{std} = weight of standard material, mg,

R_{std} = reading for standard material, μg , and

R_B = reading for blank, μg .

9.8 The calibration factor should be 1.00 ± 0.10 . A factor outside these limits would indicate a problem such as a contaminated or deteriorated detector.

10. Procedure

10.1 *Preparation of Sample Holders*:

10.1.1 If boats are used, dry as many boats as convenient at