



Designation: D7191 – 05

Standard Test Method for Determination of Moisture in Plastics by Relative Humidity Sensor¹

This standard is issued under the fixed designation D7191; 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.

1. Scope

1.1 This test method covers the quantitative determination of water down to 20 ppm in plastics using a relative humidity sensor.

1.2 Values stated in SI units are to be regarded as standard.

1.3 Specimens tested in this test method can reach or exceed 250°C, use caution when handling them after testing has completed.

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:*²

D1193 Specification for Reagent Water

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D6869 Test Method for Coulometric and Volumetric Determination of Moisture in Plastics Using the Karl Fischer Reaction (the Reaction of Iodine with Water)

3. Terminology

3.1 *Definitions*—The definitions used in this test method are in accordance with Terminology D883.

4. Summary of Test Method

4.1 A sample is loaded into a septum-capped glass vial that is moved into a heater to evolve the volatiles from the sample into the headspace. A coaxial needle, or two needle set, pierces the septum of the vial as it enters the heater. A dry carrier gas then flows into the vial and carries the evolved volatiles in the

headspace into the sensor manifold. In the sensor manifold, the carrier gas is cooled to allow high-boiling volatiles to condense on a hydrophobic filter. The filter's hydrophobic properties allow the moisture in the carrier gas to pass through and then be measured as an increase in potential at the relative humidity sensor. This sensor signal is integrated over time to provide a measurement of the total mass of water in the sample. The total moisture is then divided by sample mass to yield moisture content.

4.2 This test method utilizes a sealed, airtight flow system that prevents contamination of the analyzer from water present in the atmosphere.

5. Significance and Use

5.1 This test method is intended for use as a control, acceptance, and assessment test.

5.2 Moisture can seriously affect the processability of plastics. It is possible that high moisture content will cause surface imperfections (that is, splay or bubbling) or degradation by hydrolysis. Low moisture (with high temperature) has been known to cause solid phase polymerization.

5.3 The physical properties of some plastics are greatly affected by the moisture content.

6. Interferences

6.1 Elevated concentrations of some common solvents such as methanol, ethanol and acetone will give biased high readings due to their polar characteristics and ability to permeate the thermoset polymer layers of the relative humidity sensor.

7. Apparatus

7.1 *Moisture Analyzer*³, an apparatus that consists of:

7.1.1 *Flow Regulator*, capable of maintaining the carrier gas flow rate within the manufacturer's specified conditions.

7.1.2 *Flow Meter*, capable of measuring the carrier gas flow rate in accordance with the manufacturer's specified conditions.

7.1.3 *Manifold*, which provides:

³ The sole source of supply of the apparatus known to the committee at this time is Arizona Instrument, 1912 West 4th Street, Tempe, AZ 85281. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

Current edition approved November 1, 2005. Published November 2005. DOI: 10.1520/D7191-05.

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

7.1.3.1 A thermally stable port for mounting and operation of the relative humidity sensor.

7.1.3.2 Inlet and outlet ports for the carrier gas.

7.1.3.3 A cold trap filter loop which filters out particulates and re-condensed high boiling volatiles.

7.1.3.4 A port for mounting the coaxial needle, or two needle set.

7.1.4 *Coaxial Needle*, a needle which has a dual flow path allowing the carrier gas to flow into the sample vial and then back into the manifold, or a two needle set configured to allow inflow into the sample vial through one needle and outflow from the sample vial through the second needle.

7.1.5 *Relative Humidity (RH) Sensor*, a capacitive sensing element that measures the relative humidity of the carrier gas coming into the manifold.

7.1.6 *Sample Vial Heater*, capable of maintaining the sample vial temperature within 1°C of the programmed temperature between 25°C and 275°C.

7.1.7 *Microcontroller*, which provides:

7.1.7.1 Capability of integrating and converting the RH sensor signal.

7.1.7.2 Capability of controlling the temperature of the sample vial heater and sensor manifold.

7.2 *Balance*, external, with 1-mg readability.

8. Reagents and Materials

8.1 *Capillary Tubes*—NIST-traceable at the desired total mass of water, typically 1000 µg, or other qualified water standard devices.

8.2 *Sample Vials*—70-mm tall and 25-mL capacity.

8.3 *Sample Vial Septa*—polytetrafluoroethylene (PTFE)-coated silicone.

8.4 *Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification **D1193**.

8.5 *Carrier Gas*—Any dry gas with water content less than 3 ppm including but not limited to dry air, nitrogen, helium or argon.

9. Sampling and Test Specs and Units

9.1 Due to the small specimen size, exercise care to ensure that the specimen is representative of the sample.

9.2 Due to the hygroscopic nature of many plastics, samples shall be stored in airtight containers made of glass or other qualified or suitable material.

9.3 Samples that have been heated to remove moisture prior to processing and testing shall be allowed to cool to room temperature in a sealed container prior to determination.

9.4 Test specimens in the form of liquid, powders, pellets, or ground material.

10. Calibration and Standardization

10.1 To maintain the integrity of the test results, the vial heater shall be calibrated using a NIST-traceable temperature calibration interface, and the RH sensor shall be verified and calibrated using NIST-traceable capillary tubes. Alternatively, other suitable instrument calibration methods and standards can be used as specified by the instrument manufacturer.

10.2 Perform the vial heater calibration in accordance with the manufacturer's instructions.

10.3 If the results are not within the acceptable range, contact the analyzer manufacturer.

10.4 Perform the RH sensor verification daily in accordance with the manufacturer's instructions by way of using a capillary tube (see 8.1) filled with water (see 8.4) or other method as specified by the instrument manufacturer.

10.5 If the RH sensor verification result is not within the acceptable range, perform the RH sensor calibration in accordance with the manufacturer's instructions using a capillary tube (see 8.1) filled with water (see 8.4) or other method as specified by the instrument manufacturer.

10.6 Repeat step 10.4 to verify RH sensor calibration.

10.7 If results are not within the acceptable range, contact the analyzer manufacturer.

11. Procedure

11.1 *Sample Analysis*:

11.1.1 Turn on the analyzer.

11.1.2 Establish carrier gas flow in accordance with the manufacturer's specifications.

11.1.3 Program the analyzer with the appropriate test conditions.

NOTE 2—Suggested test conditions are listed in **Appendix X1**. If test conditions for a specific material are not listed in **Appendix X1**, they will have to be determined experimentally as described in **Appendix X2** or by contacting the analyzer manufacturer.

11.1.4 Place a clean vial on the balance and tare it.

11.1.5 Refer to **Table X2.1** and place the appropriate amount of sample in the vial keeping in mind that the vial must remain less than half full.

11.1.6 Place the vial with sample back on the balance and reweigh.

11.1.7 Record the sample weight.

11.1.8 Cap the vial ensuring the PTFE-coated side of the septum is facing the inside of the vial.

11.1.9 With the vial on its side, ensure the sample is evenly distributed in the vial.

11.1.10 Place the vial in the testing position in the analyzer.

11.1.11 Begin the program and follow the prompts for starting the analysis.

11.1.12 At the end of the test, the bottle will be very hot so use caution when handling.

11.1.13 Record the result as displayed.

11.1.14 Repeat steps 11.1.4-11.1.14 for subsequent tests.

12. Calculation or Interpretation of Results

12.1 If results are displayed in total micrograms (µg) of water present, then calculate percent moisture content of the sample as follows:

$$\text{Moisture content, \%} = R \times 10^4 - 4 / W \quad (1)$$

where:

R = total water result for sample, µg, and

W = sample weight, g.