



# Standard Test Method for Determination of Moisture in Plastics by Relative Humidity Sensor<sup>1</sup>

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 ( $\epsilon$ ) 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 known ISO equivalent to this standard.

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

[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 \(the Reaction of Iodine with Water\)](#)

[E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

<sup>1</sup> 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.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## 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*<sup>3</sup>, 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:

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.

<sup>3</sup> The sole source of supply of the apparatus known to the committee at this time is Arizona Instrument, 1912 West 4<sup>th</sup> 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,<sup>1</sup> which you may attend.

**TABLE 1 Moisture in Plastics by Relative Humidity Sensor (%)**

Material	Average	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	As % of Avg.	Reproducibility Limit	As % of Avg.
	$\bar{x}$	$S_r$	$S_R$	$r$	$r$	$R$	$R$
Polycarbonate	0.09059	0.00487	0.02240	0.01364	15.05	0.06272	69.23
Polypropylene	0.01025	0.00149	0.00587	0.00418	40.80	0.01642	160.2
Nylon 6	0.42595	0.01702	0.05677	0.04764	11.19	0.15894	37.32
ABS	0.26304	0.00617	0.02333	0.01729	6.57	0.06533	24.84

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 ( $\mu\text{g}$ ) 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,  $\mu\text{g}$ , and

$W$  = sample weight, g.

12.2 If results are displayed as percent of water present and conversion to parts per million (PPM) is desired, calculate as follows:

$$\text{PPM} = \text{Moisture content (\%)} \times 10,000 \quad (2)$$

12.3 No further calculation or interpretation is necessary.

## 13. Report

13.1 Report the following information:

13.1.1 Complete identification of the sample tested, including type of material, source, and manufacturer's code,

13.1.2 Date of test,

13.1.3 Individual specimen size,

13.1.4 Individual specimen moisture, and

13.1.5 Average moisture.

## 14. Precision and Bias

14.1 The precision of this test method is based on an interlaboratory study of D7191 - 05 conducted in 2009. Twelve laboratories tested a total of four different materials. Every *test result* represents an individual determination. Each laboratory reported triplicate test results. Practice E691 was followed for the design and analysis of the data.

14.1.1 *Repeatability limit (r)*—Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the  $r$  value for that material;  $r$  is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.

14.1.1.1 Repeatability limits are listed in Table 1.

14.1.2 *Reproducibility limit (R)*—Two test results shall be judged not equivalent if they differ by more than the  $R$  value for that material;  $R$  is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.

14.1.2.1 Reproducibility limits are listed in Table 1.

14.1.3 The above terms (repeatability limit and reproducibility limit) are used as specified in Practice E177.

14.1.4 Any judgment in accordance with statements 14.1.1 and 14.1.2 would have an approximate 95 % probability of being correct.

## 15. Keywords

15.1 moisture determination; plastics

## APPENDIXES

### (Nonmandatory Information)

#### X1. MOISTURE ANALYZER

X1.1 Suggested test conditions for selected plastics are given in Table X1.1.

NOTE X1.1—These suggested conditions were determined through extensive tests that have been completed on these common plastics. It is

not intended to be inclusive of every grade of every plastic and it will potentially be necessary to contact the analyzer manufacturer for assistance with determining the best test conditions for your specific material.

**TABLE X1.1 Suggested Test Conditions for Selected Plastics**

Material	Test Temp (°C)	Ending Criteria (µg/s)	Sample Size (g)	Vial Purge (s)
ABS	230	Rate = 0.75	1-2	45
ABS + PC	200	Predict	5-7	45
EVOH	215	Rate = 0.75	1-2	45
POM	150	Predict	2-3	45
PA 6	200	Predict	1-2	45
PA 6/6	230	Rate = 0.75	1-2	45
PBT	230	Predict	5-7	45
PC	230	Rate = 0.75	5-7	45
PEI	230	Rate = 0.50	3-5	45
PET	215	Rate = 0.50	5-7	45
PS	250	Rate = 0.75	2-3	45
PVC	200	Rate = 0.50	3-5	45
TPE	200	Predict	3-5	45
TPU	230	Predict	1-2	45

## X2. DETERMINING OPTIMAL TEST CONDITIONS

**TABLE X2.1 Recommended Sample Sizes**

Expected Moisture, %	Recommended Sample Size, g
Less than 0.01	6.9-7.1
0.01-0.03	4.9-5.1
0.03-0.05	2.9-3.1
0.05-0.15	0.9-1.1
Greater than 0.15	0.4-0.6

X2.1 When determining the optimal test conditions for a material, it is useful to have a Karl Fischer apparatus available and test in accordance with Test Method **D6869**. Optimization of test conditions may include adjustment of the sample size, test temperature, ending criteria or vial purge. Use the following guidelines for optimizing test conditions:

X2.1.1 *Sample Size*—Increase or decrease the sample size as needed to produce a result where the total mass of water present is ~1000 µg. See **Table X2.1** for recommended sample sizes keeping in mind that the sample vial must remain less

than half full to prevent the sample coming in contact with the coaxial needle during testing.

X2.1.2 *Test Temperature*—Start at a temperature 20°C below the melting or decomposition temperature of the material, or both. Increase or decrease the temperature in increments of 5°C to achieve the desired results. If no significant change in the test result occurs, proceed to rate adjustment for further optimization.

X2.1.3 *Ending Criteria*—Start at a rate of 0.50 µg/s. Increase or decrease the rate in increments of 0.05 µg/s to achieve the desired result.

X2.1.4 *Vial Purge Time*—Increase or decrease the vial purge time as needed to minimize interferences from atmospheric moisture present in the sample vial or the presence of low boiling volatiles present in the sample. It will also be necessary to decrease the vial purge in some cases to zero when testing materials with high moisture content or where the moisture evolves quickly.

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