Designation: ~~D6980 – 12~~ D6980 – 17

Standard Test Method for Determination of Moisture in Plastics by Loss in Weight[1]

This standard is issued under the fixed designation D6980; 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~~ Scope*

1.1 This test method covers the quantitative determination of moisture by means of loss in weight technology down to 50 mg/kg (50 ppm) as it applies to most plastics.

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

1.3 Specimens tested by this method will be hot, use caution when handling them after testing has been 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.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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)

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E2935 Practice for Conducting Equivalence Testing in Laboratory Applications

3. Terminology

3.1 *Definitions*—The definitions used in this test method are in accordance with Terminologies D883 and D1600.

3.2 *Symbols:*

3.2.1 *lift*—the result of convection currents created during the heating of the specimen raising the sample pan off of its support falsely indicating a moisture loss.

3.2.1.1 *Discussion*—

The effects of lift are compensated for in different ways by different manufacturers.

~~3.2.2 *tempering*—a process that re-defines the molecular structure of a metal to enhance its performance.~~

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[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 ~~Feb. 1, 2012~~June 15, 2017. Published ~~March 2012~~August 2017. Originally approved in 2004. Last previous edition approved in ~~2009~~2012 as ~~D6980 - 09.~~D6980 - 12. DOI: ~~10.1520/D6980-12.~~10.1520/D6980-17.

[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.

***A Summary of Changes section appears at the end of this standard**

4. Summary of Test Method

4.1 The specimen is spread onto a sample pan that is supported on a balance in a heating chamber that has been preheated and equilibrated to the specified idle temperature. It is then heated to vaporize the moisture. The analysis is completed when the indicated weight loss falls below a rate specified in the test conditions. The total loss of weight is integrated and displayed as the percent of moisture. ~~Both the analyzer's balance and heater are calibrated to NIST standards to achieve precise and accurate results.~~

4.2 Through adjustment of the analyzer's parameter settings, a set of conditions is developed to measure moisture.

5. Significance and Use

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

5.2 Moisture will affect the processability of some materials. For these materials, defects will occur if they are processed with a moisture content outside of the recommended range.

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

6. Interferences

6.1 When testing plastic materials for moisture by a loss in weight technique, the possibility exists for volatiles other than moisture to be evolved and cause a biased high result if the material has not been dried to remove excess moisture and low boiling volatiles. It is important to have a working knowledge of the material that is being tested and to remain below any melting or decomposition temperatures that would unnecessarily cause the emission of volatiles which can be harmful.

7. Apparatus

7.1 *Moisture Analyzer,* containing:

7.1.1 The capability of the oven shall be selected based upon the specific material being tested. Suggested test temperatures for specific plastics are shown in Tables ~~A1.1 and~~ A1.1-~~A2.1~~A3.1.

NOTE 2—It will be necessary to contact the analyzer manufacturer for suggested test temperatures for materials not listed in Tables ~~A1.1 and~~ A1.1-~~A2.1~~A3.1.

7.1.2 A balance capable of measuring to 0.0001 g.

7.1.3 An electronic or mechanical means of compensating for lift caused by convection currents created during testing.

7.1.4 A processor that is capable of converting the loss of weight to digital data.

7.1.5 Digital display for presenting the digital data as percent moisture.

7.1.6 *Sample Pans,* made from "0" temper, Aluminum 3003 or other nonreactive material.

8. Test Specimen and Sample

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

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

8.3 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.

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

9. Calibration and Standardization

9.1 To maintain the integrity of the test results the balance and heater shall both be calibrated using ~~NIST-traceable weights and an NIST-traceable temperature calibration interface.~~a traceable standard.

9.2 The calibration is verified using sodium tartrate dihydrate exhibiting a known crystal water content of 15.66 % with an acceptable result range of 15.61 to 15.71 %. Other materials with verifiable theoretical water content are acceptable for validation.

9.3 Prepare the analyzer for use and perform the analysis as described in 10.1.

9.4 If the result is not within the acceptable range, return to 9.3 for re-analysis.

9.5 If results are still not within the acceptable range, first perform a temperature calibration and then a balance calibration to ensure analyzer performance. Retest with sodium tartrate dihydrate. If results still are not within the acceptable range, the cause of the nonconformance must be determined and corrected before proceeding with testing.

10. Procedure

10.1 *Sample Analysis:*

10.1.1 Prepare analyzer as suggested by instrument manufacturer.

10.1.2 Program the analyzer with the suggested test conditions listed in Annex A1, Annex A2, or Annex A3.

NOTE 3—If test conditions for a specific material are not listed in Annex A1, Annex A2, or Annex A3, they will have to be determined experimentally or by contacting the analyzer manufacturer.

10.1.3 Begin the program and follow the prompts for placing the sample on the sample pan.

10.1.4 At the end of the test allow the analyzer to cool (if standby heating is not enabled) and remove the sample pan.

10.1.5 Record the result as displayed in percent moisture.

10.1.6 Place a clean sample pan in the analyzer and allow equilibration prior to beginning subsequent tests.

10.2 *Determination of Optimal Test Conditions:*

NOTE 4—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 or contact the analyzer manufacturer who in some cases will provide this service for you.

10.2.1 Program the analyzer in accordance with the conditions listed in Annex A1, Annex A2~~or~~, or Annex ~~A2~~A3.

10.2.2 To determine the optimum test temperature for a material, run a single test which includes several consecutive programs that have been linked together. Each program is identical in its parameters except the temperature is increased ~~5°.~~5°C.

NOTE 5—When increasing the test temperature, do not exceed a temperature where the potential exists for the emission of harmful fumes.

NOTE 6—Ensure that the program selected to run first is the lowest temperature.

10.2.3 After the tests have completed, plot the result versus temperature to make a curve as in Fig. 1.

10.2.3.1 Most of the moisture is vaporized in temperature range from points 1 to 3.

10.2.3.2 Between points 3 and 5 the moisture result is very low and constant. Choose a temperature in this range as the optimum test temperature.

10.2.3.3 Above point 5 the moisture result begins to increase. This is likely caused by the generation of water due to decomposition or solid phase polymerization of the sample.

NOTE 7—It is not uncommon for the optimal test temperature to be above the melting point of the selected plastic due to the distance between the resistive thermal device and the sample pan.

11. Calculation

11.1 Result is reported in percent moisture to three decimal places so no further calculations are necessary.

11.2 If conversion to mg/kg is desired, calculate as follows:

$$\text{mg/kg} = \text{Moisture content (\%)} \times 10000 \quad (1)$$

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the sample tested, including type of material, source, manufacturer's code, form, and previous history,

12.1.2 Date of test,

12.1.3 Individual specimen size,

12.1.4 Individual specimen moisture, and

12.1.5 Average moisture if multiple tests are run.

13. Precision and Bias[3,4]

13.1 The ~~precision~~repeatability of this test method is based on an interlaboratory study conducted in 2007. Eight laboratories analyzed eight different plastic materials for moisture content. Every "test result" represents an individual determination. The

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1250.

[4] Equivalence testing in ASTM standards are covered by Practice E2935.

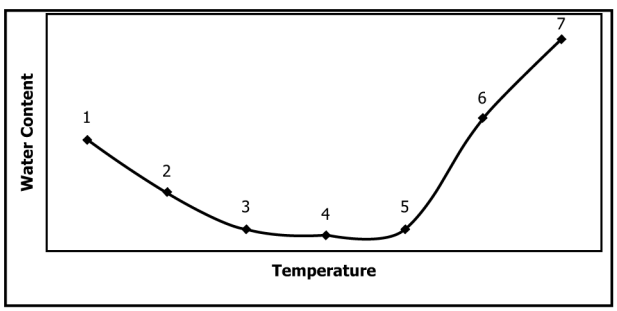

FIG. 1 Optimum Test Temperature Selection

TABLE 1 Moisture (%)

Material	Average[A]	Standard Deviation of Lab Averages	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	~~$\bar{x}$~~	~~$S_{\bar{x}}$~~	~~s_r~~	~~s_R~~	~~r~~	~~R~~
$\bar{x}$	S	s_r	s_R	r	R	
A (Nylon 6/6)	0.4954	0.0131	0.0086	0.0151	0.0242	0.0423
B (PET)	0.0292	0.0084	0.0047	0.0094	0.0133	0.0263
C (TPE)	0.0107	0.0070	0.0026	0.0073	0.0072	0.0204
D (PC)	0.0748	0.0040	0.0061	0.0067	0.0171	0.0187
E (Nylon 6/6)	0.1390	0.0302	0.0241	0.0367	0.0674	0.1027
F (TPE)	0.0406	0.0329	0.0024	0.0330	0.0068	0.0924
G (PC)	0.0181	0.0085	0.0030	0.0088	0.0083	0.0247
H (PET)	0.1053	0.0109	0.0049	0.0117	0.0138	0.0327

[A]The average of the laboratories' calculated averages.

laboratories reported two to four replicate results for each analysis in order to estimate the repeatability and reproducibility limits of the standard. Practice E691 was followed for the design and analysis of the data.

13.1.1 *Repeatability Limit (r)*—~~Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the~~ The r~~value for that material; below which r is the interval representing the critical~~ the absolute difference between two individual test results ~~for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.~~obtained under repeatability conditions may be expected to occur with a probability of approximately 0.95 (95 %).

13.1.1.1 Repeatability limits are listed in Table 1.

13.1.2 *Reproducibility Limit (R)*—~~Two test results shall be judged not equivalent if they differ by more than the~~ The R~~value for that material; below which R is the interval representing the critical~~ the absolute difference between two individual test results ~~for the same material, obtained by different operators using different equipment in different laboratories.~~obtained under reproducibility conditions may be expected to occur with a probability of approximately 0.95 (95 %).

13.1.2.1 Reproducibility limits are listed in Table 1.

~~13.1.3 Any judgment in accordance with statements 13.1.1 and 13.1.2 would have an approximate 95 % probability of being correct.~~

13.2 *Bias*—At the time of the study, the test specimens chosen for analysis were not accepted reference materials suitable for determining the bias for this test method, therefore no statement on bias is being made.

13.3 The ~~precision~~repeatability statement was determined through statistical examination of 162 data points, from eight laboratories, on eight materials. These eight materials were described as the following:

Material A: Nylon 6/6 (not dried)
Material B: PET (dried)
Material C: TPE (dried)
Material D: Polycarbonate (not dried)
Material E: Nylon 6/6 (dried)
Material F: TPE (not dried)
Material G: PC (dried)
Material H: PET (not-dried)

13.4 To judge the equivalency of two test results, it is recommended to choose the material closest in characteristics to the test material.

14. Keywords

14.1 moisture determination; plastics; volatile content

ANNEXES

(Mandatory Information)

A1. MOISTURE ANALYZER WITH ELECTRONIC LIFT COMPENSATION

A1.1 Suggested test conditions for ~~sodium tartrate dihydrate and~~ selected plastics are given in Table A1.1.

A1.2 Use the following guidelines for determining test conditions for a material not listed in Table A1.1, and then perform procedure in 10.2 to determine the optimal test temperature:

	~~Materials with an expected moisture content below 0.10 %~~ Materials with an expected moisture content below 0.10 %	~~Materials with an expected moisture content below 0.10 %~~ Materials with an expected moisture content above 0.10 %
Temperatures	Test—Set to 30°C below melt point Hi Start—25°C Idle—100°C	Test—Set to 30°C below melt point Hi Start—25°C Idle—100°C
Ending Criteria	End on Rate—0.005 %/min	End on Rate—0.010 %/min
Sample Size	30 ± 2 g sample window	20 ± 2 g sample window
Tare Options	Pan Tare—Ultra Low Moisture Sample Tare—8 s	Pan Tare—Ultra Low Moisture Sample Tare—8 s
Lift Compensation	100 %	100 %

TABLE A1.1 Suggested Test Conditions for Selected Plastics

Material	Test Temp. (°C)	Idle Temp.	Rate	Sample Size (grams)	Pan Tare	Sample Tare (seconds)	Lift Compensation
ABS	140	100	0.010	18 to 22	Ultra-Low	8	100
ABS + PC	155	100	0.005	28 to 32	Ultra-Low	8	100
POM	160	100	0.005	18 to 22	Ultra-Low	8	100
Acrylic	150	100	0.007	28 to 32	Ultra-Low	8	100
PA 6	175	100	0.015	18 to 22	Ultra-Low	8	100
PA 6/6	210	100	0.015	18 to 22	Ultra-Low	8	100
PBT	175	100	0.005	28 to 32	Ultra-Low	8	100
PC	170	100	0.005	28 to 32	Ultra-Low	8	100
PEI	170	100	0.005	28 to 32	Ultra-Low	8	100
PET	160	100	0.005	28 to 32	Ultra-Low	8	100
PPS	170	100	0.005	28 to 32	Ultra-Low	8	100
PS	165	100	0.005	28 to 32	Ultra-Low	8	100
PVC	105	100	0.010	18 to 22	Ultra-Low	8	100
TPE	140	100	0.005	28 to 32	Ultra-Low	8	100
TPU	155	100	0.010	18 to 22	Ultra-Low	8	100

NOTE 1—If you notice degradation of material including more than moderate discoloration, melting, or smoke, lower the test temperature by 10°C per test until no discoloration appears and the pellets retain their shape.