Designation: E2551 – 18

Standard Test Methods for Humidity Calibration (or Conformation) of Humidity Generators for Use with Thermogravimetric Analyzers[1]

This standard is issued under the fixed designation E2551; 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 These test methods describe the humidity calibration (or conformance) of humidity generators for use with thermogravimetric analyzers and other thermal analysis apparatus. The humidity range covered is 5 % relative humidity (% RH) to 95 % RH and the temperature range is 0 °C to 80 °C.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

D1193 Specification for Reagent Water

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E473 Terminology Relating to Thermal Analysis and Rheology

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

E1142 Terminology Relating to Thermophysical Properties

E1582 Test Method for Temperature Calibration of Thermogravimetric Analyzers

3. Terminology

3.1 Specific technical terms used in this standard are defined in Terminologies E473 and E1142. These terms include *first-deviation-from-baseline, isohume, relative humidity, thermal curve,* and *thermogravimetric analysis*.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *water activity, n*—the ratio of actual partial pressure of water to the saturated water vapor pressure at the same temperature, expressed as a decimal fraction.

3.2.1.1 *Discussion*—Water activity is also known as relative pressure in some applications areas.

4. Summary of Test Method

4.1 Humidity generators are devices aimed at producing a specific level of humidity in the purge gas used by thermogravimetric analyzers or other thermal analysis apparatus. The requested humidity levels may be held constant (isohume) or increased or decreased in a continuous or stepped fashion.

4.2 The humidified purge gas is submitted to a thermogravimetric analyzer in which the weight of a hygroscopic material is observed. The relative humidity (or activity) of the moisture in the purge gas is stepped or scanned through a humidity range. At a fixed humidity of the purge gas, the test specimen deliquesces and gains weight. In Test Method A, the humidity of the onset of this weight gain is taken as the humidity calibration point. In Test Methods B and C, the rate of weight change is zero at the humidity calibration point.

5. Significance and Use

5.1 These test methods calibrate or demonstrate conformity of the humidity level in a purge gas generated by a humidity generator at a fixed temperature. Such calibration or demonstration of conformity may be required by quality initiatives.

5.2 Conformance demonstrates that the humidified purge gas is within some established limits.

5.3 Calibration provides an offset and or slope value that may be used for establishing the relative humidity scale of the apparatus.

[1] These test methods are under the jurisdiction of ASTM Committee E37 on Thermal Measurements and are the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

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[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.

6. Interferences

6.1 Temperature regulation of any solution-head space environment to within ±0.1 °C is essential for realizing generated relative humidity values stable to within ±1 % RH (expected).

7. Apparatus

7.1 The humidity generator that is the focus of this standard may be an accessory providing a humidified purge gas to some other thermal analysis apparatus (typically a thermogravimetric analyzer) or it may be part of a self-contained instrument that includes both the humidity generator and the thermal analysis apparatus. In the former case, some of the components described below may be redundant.

7.2 *Humidity Generator*—The essential instrumentation required to provide the minimum humidity generator capability for these test methods includes:

7.2.1 *Temperature Sensor,* to provide an indication of the purge gas temperature readable to within ±0.1 °C.

7.2.2 *Temperature Controller,* capable of executing a specific temperature program by operating heaters or coolers between selected temperature limits at a rate of temperature change of 0.5 °C/min constant to ±0.1 °C/min or at an isothermal temperature constant to within ±0.1 °C.

7.2.3 *Humidity Sensor,* capable of indicating the humidity of the purge gas over the range of 5 % RH to 95 % RH readable to within ±0.1 % RH.

7.2.4 *Humidity Controller,* capable of executing a specific humidity program by operating purge gas humidifiers between selected humidity limits at a rate of humidity change of 0.5 % RH/min constant to within ±0.1 % RH or at an isohume relative humidity to within ±0.1 % RH.

7.2.5 *Purge Gas Flow Sensor,* capable of measuring purge gas flow readable to within ±0.1 mL/min.

7.2.6 *Purge Gas Flow Controller,* capable of controlling purge gas flow readable to within ±0.1 mL/min.

7.2.7 *Humidifier Element,* capable of generating purge gases with relative humidity continuously over the range of 5 % RH to 95 % RH.

7.3 *Thermogravimetric Analyzer (TGA)*—The essential instrumentation required to provide the minimum thermogravimetric analytical capability for this method includes:

7.3.1 A *furnace* to provide uniform controlled heating or cooling of a specimen to a constant temperature or at a constant rate within the applicable temperature range of this method.

7.3.2 A *temperature sensor* to provide an indication of the specimen or furnace temperature to within ±0.1 °C.

7.3.3 A continuously recording balance to measure the specimen weight with a minimum capacity of 100 mg and sensitivity of ±10 µg.

7.3.4 A means of maintaining the specimen/container under atmospheric control at a purge rate of 10 mL/min to 200 mL/min ± 10 mL/min or 1 %, whichever is greater.

7.3.5 A *temperature controller* capable of executing a specific temperature program by operating the furnace between selected temperature limits at a rate of temperature change of 0.5 °C/min constant to within ±0.1 °C/min or to an isothermal temperature that is maintained constant to within ±0.1 °C for a minimum of 100 h.

7.3.6 *Containers* (pans, crucibles, etc.) that are inert to the specimen and that will remain gravimetrically stable within the temperature limits of this method.

7.3.7 *Data storage* capable of storage of the weight and relative humidity signals.

7.3.8 A *display* capable of plotting a thermal curve with weight on the ordinate (Y-axis) and relative humidity (or activity) on the abscissa (X-axis) with a sensitivity of 10 µg for weight and 0.1 % RH, respectively.

8. Reagents and Materials

8.1 One or more inorganic salts taken from Table 1 selected to provide the humidity range of interest.

8.2 *Purity of Reagents*—Reagent grade chemicals (or better) shall be used for preparation of all standard solutions.

8.3 *Purity of Water*—Reagent water produced by distillation or by ion exchange, or reverse osmosis followed by distillation shall be used (see Specification D1193).

TABLE 1 Humidity Fixed Points

NOTE 1—Greenspan, L., "Humidity Fixed Points of Binary Saturated Aqueous Solutions," *Journal of Research of the National Bureau of Standards — A. Physics and Chemistry*, Vol 81A, No. 1, 1977, pp. 89–96.

Temperature (°C)	Lithium Chloride	Potassium Acetate	Magnesium Chloride	Potassium Carbonate	Magnesium Nitrate	Sodium Bromide	Strontium Chloride	Sodium Chloride	Potassium Chloride
10	11.3	23.7	33.5	43.1	57.4	62.2	75.66	75.7	86.8
15	11.3	23.4	33.3	43.2	55.9	60.7	74.13	75.6	85.9
20	11.3	23.1	33.1	43.2	54.4	59.1	72.52	75.5	85.1
25	11.3	22.5	32.8	43.2	52.9	57.6	70.85	75.3	84.3
30	11.3	21.6	32.4	43.2	51.4	56.0	69.12	75.1	83.6
35	11.3	...	32.1	...	49.9	54.6	...	74.9	83.0
40	11.2	...	31.6	...	48.4	53.2	...	74.7	82.3
45	11.2	...	31.1	...	46.9	52.0	...	74.5	81.7
50	11.1	...	30.5	...	45.4	50.9	...	74.4	81.2
55	11.0	...	29.9	...	...	50.2	...	74.4	80.7
60	11.0	...	29.3	...	...	49.7	...	74.5	80.3
65	10.9	...	28.5	...	...	49.5	...	74.7	79.9
70	10.8	...	27.8	...	...	49.7	...	75.1	79.5
75	10.6	...	26.9	...	...	50.3	...	75.6	79.2
80	10.5	...	26.1	...	...	51.4	...	76.3	78.9

9. Hazards

9.1 Salt solutions are extremely corrosive to apparatus if spilled. Care shall be taken in their preparation and handling to prevent contact with apparatus.

10. Preparation of Apparatus

10.1 Perform any setup or calibration procedures recommend by the apparatus manufacturer in the operations manual.

10.2 *Positioning of the Temperature Sensor*—If the system employs a temperature sensor that is movable, it shall be located as close to the specimen as possible without touching it or the balance pan. In addition, it must be located in exactly the same position during calibrations as used during analytical determinations.

11. Calibration and Standardization

11.1 Calibrate the temperature display of the apparatus according to Test Method E1582 using a heating rate of 0.5 °C/min ± 0.2 °C/min.

12. Procedure

12.1 Close the system, adjust the atmospheric flow rate of the purge gas to the selected rate, and zero (tare) the balance.

12.2 Open the system and place 5 mg to 10 mg of the reference material in the specimen container in the same position as would be placed for a test specimen. Close the system.

12.3 Measure the weight of the reference material and report its value.

12.4 *Test Method A:*

12.4.1 Equilibrate the humidity for 60 min at a value that is 5 % RH below the anticipated deliquescence point described in Table 1.

NOTE 1—Other humidity starting points may be used but shall be reported.

12.4.2 Initiate either a humidity program with either 0.2 % RH steps and 12 min soak times or 1 % RH/hr linear increase to an ending humidity value that is 5 % RH higher than the anticipated deliquescence point in Table 1.

NOTE 2—Other humidity ending points and rate of humidity change may be used but shall be reported.

12.4.3 Plot the results as weight of the reference material on the ordinate (Y-axis) and the relative humidity on the abscissa (X-axis) of a thermal curve.

12.4.4 Determine the first-deviation-from-baseline as the deliquescence point of the reference material (see Fig. 1). Report the corresponding humidity as *Mi*.

NOTE 3—The rate of weight gain will increase with relative humidity above the humidity at which the initial weight gain is observed. There is no inflection point. The use of extrapolate onset values shall not be used.

12.5 *Test Method B:*

12.5.1 Equilibrate the humidity for 60 min at 5 % RH above the anticipated deliquescence point described in Table 1.

NOTE 4—Other humidity starting points and equilibrium times may be used but shall be reported.

12.5.2 Initiate decreasing humidity program with either 0.2 % RH steps and 12 min soak times or a linearly decreasing

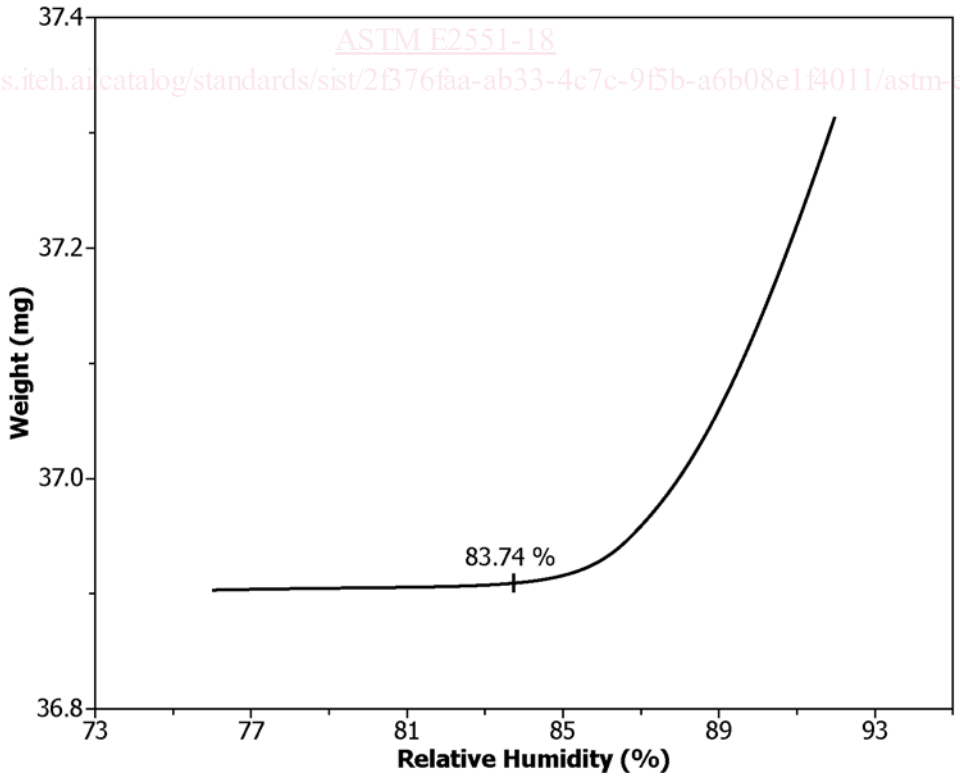

FIG. 1 First-Deviation-From-Baseline as the Deliquescence Point of the Reference Material