



# Standard Test Method for Hygroscopic Sorption Isotherms of Building Materials<sup>1</sup>

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## 1. Scope

1.1 This test method specifies a laboratory procedure for the determination of hygroscopic sorption isotherms of any construction materials. The method was originally developed for the ASTM Thermal Insulation committee.

1.2 For material characterization, the primary emphasis is on the adsorption isotherm (that is, sorption isotherm that describes the wetting process of the material from the oven-dry condition).

1.3 Determination of desorption isotherm, (that is, sorption isotherm that describes the drying process of a material from the state of absolute saturation with water) is performed when information on drying characteristics of construction materials is required. Typically both adsorption and desorption isotherms are required for the purpose of hygrothermal models.

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

1.5 *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.*

## 2. Referenced Documents

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

**E104** Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solutions

**E337** Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee C16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.33 on Insulation Finishes and Moisture.

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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 of Terms Specific to This Standard:

3.1.1 *adsorption isotherm*—the sorption isotherm measured exclusively during the hygroscopic adsorption process started from the oven-dry condition.

3.1.2 *desorption isotherm*—the sorption isotherm measured exclusively during the hygroscopic desorption process started from the condition of full water saturation of the material.

3.1.3 *hygroscopic adsorption*—fixation of water molecules from ambient air on surfaces of a material until equilibrium is established.

3.1.4 *hygroscopic desorption*—release of adsorbed water molecules from surfaces of a material into the ambient air until equilibrium is established.

3.1.5 *hysteresis*—a physical phenomenon which makes the desorption isotherm different from the adsorption isotherm due to the difference in the energy level of pore water.

3.1.6 *moisture content, by mass*—mass of water retained in the specimen divided by the dry mass of the specimen.

3.1.7 *moisture content, by volume*—volume of water retained in the specimen divided by the volume of the dry specimen.

3.1.8 *sorption isotherm*—relationship between the relative humidity (RH) (see Test Method E337) and the equilibrium moisture content of the material, at a specified temperature.

## 4. Significance and Use

4.1 The purpose of these tests is to obtain, for a specified temperature, by means of a specified laboratory procedure, the values of the equilibrium moisture content at various levels of RH. These values are used either as means to characterize the material or as material characteristics needed as input to appropriate computer models that can simulate wetting or drying potential of individual building materials or material assemblies under specified environmental conditions.

4.2 A specified value of the equilibrium moisture content can also be used for material characterization. If this type of material characterization is called for in a material specification (for example, mineral or cellulose fiber insulation), the equilibrium at  $95 \pm 3\%$  RH shall be used.

4.3 For ease and repeatability of measurements, the measurements for characterization are performed on adsorption isotherms. Though desorption is the reverse of adsorption, most porous materials reach different equilibrium levels during these two processes. Usually, the equilibrium moisture content on the desorption isotherm is higher than that on the adsorption isotherm for the same level of RH.

## 5. Apparatus

5.1 *Weighing Cups*—Weighing cups, made from non-absorbing material<sup>3</sup>, for example, glass, shall be provided with tight-fitting lids and the volume shall not be less than 15 cm<sup>3</sup>.

5.2 *Balance*—An analytical balance capable of weighing the cups within 1 mg shall be used. The accuracy of the balance shall be at least  $\pm 0.1$  percent of the total specimen weight.

5.3 *Drying Oven*—A ventilated drying oven, capable of maintaining the required drying temperature within  $\pm 2$ K for temperatures less than 75°C and  $\pm 4$ K for temperatures above 75°C, and a relative humidity of less than 10 %, shall be used. In warm-humid laboratory environment or at low drying temperatures, it will be necessary to provide a supply of dried air to achieve the less than 10 % relative humidity specification in the drying oven.

5.4 *Environment Chamber*—The specimens shall be exposed to controlled environmental conditions. The precise condition for the test environment shall be maintained in one of the following two ways, (a) with desiccators placed in a room with controlled temperature, or (b) with a climatic chamber.

5.4.1 The test conditions can be generated within the desiccators that contain saturated salt solutions<sup>4</sup>, (see also Practice E104). Since the partial pressure of the vapor above the solution is strongly dependent on the temperature stability, temperature oscillation in the desiccator should be as small as possible. The range  $\pm 0.1$ K is recommended. The maximum variation permitted by this standard shall not exceed  $\pm 0.5$ K. Normally, the desiccators are placed inside a chamber or a room with controlled temperature. In this case, it is recommended that the chamber or room is capable of maintaining the test conditions within  $\pm 1$ K.

5.4.2 If the climatic chamber is used for the determination of the hygroscopic sorption isotherms, the chamber shall be capable of maintaining the test conditions within  $\pm 3$  % for the full range of RH.<sup>5</sup> Temperature shall be maintained within  $\pm 0.5$ K.

5.5 *Desiccator*, with (a) calcium chloride as desiccant for drying, or (b) with saturated salt solution to generate specific relative humidity level.

<sup>3</sup> Normally, the specific area of a porous material is so large that adsorption on surfaces of the weighing cup may be omitted. Yet, when the amount of sorbed water is low and requirements of high precision demand it, weighing of an empty container can be used as the way to improve the precision of sorption measurements.

<sup>4</sup> Greenspan, L., "Humidity Fixed Points of Binary Saturated Aqueous Solutions," *Journal of Research of the National Bureau of Standards—A. Physics and Chemistry*, 1977, Vol 81A, No 1.

<sup>5</sup> This will increase the uncertainty of the test results in comparison with the procedure in 5.4.1. Therefore, when included in materials standards, due consideration shall be given to the intended precision and either 5.4.1 or 5.4.2 shall be specified.

## 6. Test Specimens

6.1 A test specimen shall have a mass of at least 10 g. The test specimen may be cut into several smaller pieces, but not powdered, to reduce the time to reach equilibrium with the environment.

6.2 A minimum of three specimens shall be tested in each environment. The test procedure as specified below, and the precision of weighing in particular, shall be applied to each specimen.

## 7. Procedure

7.1 Unless otherwise specified, the temperature of 23°C shall be used for the test.

7.2 Determine the dry weight of each specimen by placing it in the drying oven (see 5.3) at the required temperature for a minimum of 24 h (see Note 1). Cool the specimen to room temperature ( $\cong 20$ –25°C) either in a desiccator with calcium chloride as desiccant or in a weighing cup with tight-fitting lids and reweigh. Repeat the whole process, until three successive daily weighings agree to within 0.1 % of the specimen weight obtained in the latest weighing. Record the average of these three weights as the dry weight of the specimen.

7.3 *Determination of Adsorption Isotherms*—Prior to testing water adsorption, each test specimen is to be dried to a constant mass. Note 1 provides recommendations on selection of the appropriate temperature. Determination of adsorption isotherms can be performed with either the procedure described in 5.4.1 or with that described in 5.4.2 and the steps as described below.

7.3.1 Place the weighing cup with the dried specimen in the test environment having the lowest RH, typically about 30 %RH. The test environment is achieved either in the desiccator that contains a salt solution and placed in the constant temperature room (5.4.1) or in the climatic chamber (5.4.2). Place the lid beside the weighing cup. Periodically weigh the weighing cup with the specimen until it is in equilibrium with the environment. At each weighing, before the cup with the specimen is removed from the environment to the balance, put the lid on the cup. After weighing, return the cup with the specimen to the test environment, with the lid beside it. Constant mass is reached if in five successive weighings, with 24 h intervals, the change of mass is less than 0.1 % of the specimen mass (see Note 2).

7.3.2 The specimen is placed consecutively in a series of test environments, maintaining a constant temperature and increasing the RH in stages, until the equilibrium is reached in each environment. If determination of the full sorption curve is required, a minimum of five test environments shall be selected. Repeat the whole procedure described in 7.3.1 until the measurement is completed in the test environment with the highest RH. Normally the 98 %RH represents the upper end of the adsorption isotherm.

7.3.3 The equilibrium moisture content at each test condition is calculated from the measured difference between the constant mass in each environment and the dry weight of the specimen.

**7.4 Determination of Desorption Isotherms**—The starting point for this measurement is the material absolute saturation with water (see **Note 3**). Determination of desorption isotherms can be performed with either the procedure described in **5.4.1** or with that described in **5.4.2** and following the steps as described below.

**7.4.1** The fully saturated specimen in a weighing cup is to be placed in the test environment with the highest RH (typically 98 %RH). Place the lid beside the weighing cup with the specimen. Periodically weigh the weighing cup with the specimen until it is in equilibrium with the environment. At each weighing, before the cup with the specimen is removed from the environment to the balance, put the lid on the cup. After weighing, return the cup with the specimen to the test environment, with the lid beside it. Constant mass is reached if in five successive weighings with 24 h intervals the change of mass is less than 0.1 % of the specimen mass (see **Note 2**).

**7.4.2** After achieving the equilibrium, transfer the cup with the specimen and the lid to the test environment with the next lower RH. A minimum of five test environments shall be selected. Repeat the whole procedure as described in **7.4.1** until the measurement is completed in the test environment with the lowest RH.

**7.4.3** Completely dry the specimen at the appropriate temperature to constant mass (see **Note 1**) and weigh the dry specimen.

**7.4.4** The equilibrium moisture content at each test condition is calculated from the measured difference between the constant mass in each environment and the dry weight of the specimen.

**NOTE 1**—Typically, the following temperatures are used for drying the test specimens: *a*) for materials which do not change either structure or dimensions at 105°C, for example, some mineral materials, use 105 ± 4°C, *b*) for materials, in which structural or dimensional changes occur between 70°C and 105°C, for example, some cellular plastics, use 70 ± 2°C, *c*) for materials, in which elevated temperatures bring about chemical or physical changes, for example, crystalline water in gypsum or blowing agent solubility in some cellular plastics, use 40 ± 2°C, and *d*) when drying at the specified aforementioned temperatures adversely affects the building material, dry specimen to moisture free weight (that is dry weight, see **7.2**) in a desiccator at room temperature or inside an airtight chamber flashed with dry air having a dew point less than ≤ − 40°C.

**NOTE 2**—For practical reasons, constant mass means the change in mass is within 0.1 % during three consecutive daily weighings. If the sorption or drying process is slow for example, the uncertainty of the mass determination exceeds 30 % of the change in mass observed in the last three days before the constant mass is assumed, the intervals between successive weighings shall be increased to two or three days.

**NOTE 3**—For practical reasons, the moisture content determined for a specimen either after 3 days of immersion to water exposed to a reduced air pressure (less than 0.4 atm) is acceptable as water saturation for the purpose of testing the desorption isotherm. Alternatively the specimen shall be immersed for 7 days in water with a room temperature, in such a manner that 100-mm water head is acting on its top surface.

## 8. Calculation

**8.1** Calculate the moisture content,  $u$  (kg·kg<sup>−1</sup>), as follows using the mean values of the mass of the test specimens at each test condition:

$$u = \frac{(m - m_0)}{m_0}$$

where:

$m$  = the mean mass of the specimens at equilibrium, and  
 $m_0$  = that of the dry specimens.

**8.2** If both the adsorption and desorption isotherms have been determined, plot the relationships between equilibrium moisture content and the RH for both adsorption and desorption, to express the magnitude of the hysteresis effect.

## 9. Report

**9.1** The test report shall include the following:

**9.1.1** Reference to this ASTM Standard.

**9.1.2** Product identification as:

**9.1.2.1** Name, manufacturer or supplier,

**9.1.2.2** Type, as in manufacturer's specification,

**9.1.2.3** Production code number, if any,

**9.1.2.4** Packaging,

**9.1.2.5** The form in which arrived at the laboratory,

**9.1.2.6** Nominal physical characteristics; for example, bulk density, thickness etc.,

**9.1.3** Test procedure with:

**9.1.3.1** Factors if any, which may have influenced the results,

**9.1.3.2** Date of test, and

**9.1.3.3** Drying temperature, relative humidity and drying procedure.

**9.1.4** Results:

**9.1.4.1** Table of measured values, temperature, RH and moisture content, and

**9.1.4.2** Graph showing the sorption isotherm.

## 10. Precision and Bias

**10.1** The reproducibility precision of this test method is yet to be established. Extensive measurements performed in one laboratory are used to generate the following estimates of repeatability precision.

**10.2** As the worst case scenario, three specimens of stucco, each approximately 10-mm thick and weighing about 30 g and tested over a prolonged period (approximately 6 months) are reported below. With the mass of sorbed water ranging from 5 % to 8 % by weight the standard deviations expressed in percent of the measured value were as follows (see **Table 1**).

**10.3** Two typical cases of hygroscopic materials (oriented strand board and fiberboard) are presented in **Table 2**. Similar size specimens weighing about 10 g (a minimum specified in the standard), were tested over a period of about 1 month. With the moisture content per weight similar to the previous cases (see **10.2**), the standard deviations expressed in percent of the measured value were much smaller.

**TABLE 1 Standard Deviations (%) in Six Consecutive Weighings Used to Determine the Equilibrium During the Adsorption Process on the Stucco Sample**

Equilibrium at	50 %RH	70 %RH	90 %RH
Specimen 1	3.1	3.7	7.1
Specimen 2	9.3	9.3	7.1
Specimen 3	5.7	5.7	7.5



**TABLE 2 Standard Deviations (%) in Six Consecutive Weighings  
Used to Determine the Equilibrium During the Adsorption  
Process on the OSB (Specimens 4-6) and Fiberboard (Specimens  
7-9)**

Equilibrium at	50 %RH	70 %RH	90 %RH
Specimen 4/7	1.9/1.4	0.9/1.1	0.9/2.0
Specimen 5/8	1.8/1.4	0.8/1.2	0.4/2.1
Specimen 6/9	1.4/1.4	0.7/1.2	0.1/2.4

## 11. Keywords

### 11.1 hysteresis; moisture content; water vapor sorption

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