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Standard Test Method for Hygroscopic Sorption Isotherms of Building Materials¹

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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 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:²

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)

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.
- ¹ 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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- 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.

- 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 ± 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 material³, for example, glass, shall be provided with tight-fitting lids and the volume shall not be less than 15 cm^3 (0.0005 ft³).
- 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 $\pm 2K$ ($\pm 4^{\circ}F$) for temperatures less than 75°C (167°F) and $\pm 4K$ ($\pm 8^{\circ}F$) for temperatures above 75°C (167°F), 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 solutions⁴, (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 $\pm 0.1 \text{K}$ ($\pm 0.2^{\circ}\text{F}$) is recommended. The maximum variation permitted by this standard shall not exceed $\pm 0.5 \text{K}$ ($\pm 1^{\circ}\text{F}$). 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 $\pm 1 \text{K}$ ($\pm 2^{\circ}\text{F}$).
- 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 ± 3 % for the full range of RH.⁵ Temperature shall be maintained within $\pm 0.5 \text{K}$ ($\pm 1^{\circ}\text{F}$).
- 5.5 *Desiccator*, with (a) calcium chloride as desiccant for drying, or (b) with saturated salt solution to generate specific relative humidity level.

6. Test Specimens

 $6.1\,$ A test specimen shall have a mass of at least $10\,$ g ($0.022\,$ lb). The test specimen may be cut into several smaller pieces, but not powdered, to reduce the time to reach equilibrium with the environment.

³ 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.

⁴ 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.

⁵ 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.

6.2 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 (73°F) 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 ($\cong 20-25^{\circ}\text{C}$) 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