Designation: D6403 – 99 (Reapproved 2014)

Standard Test Method for Determining Moisture in Raw and Spent Materials[1]

This standard is issued under the fixed designation D6403; 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 This test method is intended for use in determining the moisture content in raw and spent materials that are extracted for tannin analysis. The moisture content of the sample is operationally defined to be equal to the weight loss experienced as a result of the evaporation which occurs in the drying oven.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D6405 Practice for Extraction of Tannins from Raw and Spent Materials

2.2 *ALCA Methods*

A6 Moisture in Raw and Spent Materials[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *raw material*—any of the various parts of plants that are used as a source of vegetable tannins.

3.1.2 *spent material*—plant tissue by-products from industrial processes which may contain significant quantities of vegetable tannins.

3.1.3 *tannin*—an astringent substance found in the various parts of plants such as bark, wood, leaves, nuts, fruits, roots, etc.

3.1.4 *vegetable tannins*—mixtures of substances (natural products) obtained from plant tissues by water extraction which have the chemical and physical properties necessary to convert animal hides and skins into leather.

4. Summary of Test Method

4.1 A specimen of the material sample prepared for use in Practice D6405 is dried overnight in a forced-air oven. The loss in weight represents the moisture in the specimen.

5. Significance and Use

5.1 This test method is used to determine the moisture content of materials (raw or spent) that are to be extracted for tannin analysis. The value obtained for moisture content by this test method is used to calculate the results of the other analyses on this material to a moisture-free basis.

5.2 The specimens are obtained from the material prepared for extraction in Practice D6405.

5.3 Tanning materials contain moisture in varying amounts, depending both on the nature of the material and on the climatic conditions, therefore sampling must be carried out as quickly as is consistent with thoroughness in order to avoid changes in moisture content.

5.4 Negative errors may occur in the moisture determination because under the conditions of this method there may be retention of moisture by certain components (for example, hydrated salts or water bound to organic structures) of the raw or spent material or because of oxidation of other components (for example, tannins) of the raw or spent material.

5.5 Positive errors may occur in the moisture determination because under the conditions of this test method there may be volatilization of certain components of the raw or spent material other than water.

5.6 It is known that other factors can also affect the quantity of volatile matter (moisture) released by the specimen. These factors include but are not limited to: particle size of the test specimen, quantity of test specimen, oven temperature, exposure time in the oven, shape of the specimen container, and type of oven (for example, gravity or mechanical convection) used.

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[1] This test method is under the jurisdiction of ASTM Committee D31 on Leather and is the direct responsibility of Subcommittee D31.01 on Vegetable Leather. This test method has been adapted from and is a replacement for Method A6 of the Official Methods of the American Leather Chemists Association.

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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] Official Methods of the American Leather Chemists Association. Available from the American Leather Chemists Association, University of Cincinnati, P.O. Box 210014, Cincinnati, OH 45221-0014.

5.7 Because of the possibility of unknown errors in this test method it is essential that the method be followed exactly in order to obtain reproducible results both among specimens within a laboratory and for analyses between laboratories.

6. Apparatus

6.1 *Balance,* analytical balance which will weigh up to 100 g with an accuracy of ± 0.1 mg (± 0.0001 g).

6.2 *Tannin Dish,* crystallizing dish, borosilicate glass, 50 mm tall, 70 mm outside diameter. The bottom corner shall be rounded to a radius of 6 mm, the bottom shall be flat and not cupped in the center, and the top edge shall be rounded and polished.

6.3 *Watch Glass,* a suitable size to be used as a cover for the tannin dishes. These can be used to keep dust and other possible contaminates from settling into the dishes while exposed to the laboratory environment before and after the specimen transfer operation. The watch glass covers must be removed prior to placing the dishes in the drying oven.

6.4 *Drying Oven,* a forced-air convection oven (or mechanical-convection draft oven) capable of maintaining a temperature of 100 ± 2.0°C.

6.5 *Thermometer,* accurate to ± 0.2°C which is used to check and monitor the oven set point.

6.6 *Dessicator,* any convenient form or size, using any normal dessicant.

7. Test Specimen

7.1 The specimen shall consist of approximately 10 g of the material from the sample prepared as described in the Preparation of Sample and Test Specimens sections of Practice D6405.

8. Procedure

8.1 *Preparation of Tannin Dishes:*

8.1.1 Place the clean, empty tannin dish in the oven at 100 ± 2.0°C for at least 1 h.

8.1.2 Remove the dish from the oven and place it in a dessicator until it has reached temperature equilibrium with the laboratory environment.

8.1.3 Label the dish and record the weight as W_1 to the nearest ± 0.0001 g.

8.1.4 Return the dish to the dessicator and store it there until used.

8.2 *Specimen transfer:*

8.2.1 Transfer approximately 10 g of the material from the sample prepared as described in Section 7 and Section 8 of Practice D6405 into the previously tared tannin dish prepared as above.

8.2.2 Record the weight of the dish with the added material as W_2 to the nearest ± 0.0001 g.

NOTE 1—Since the moisture content of many materials varies greatly depending upon the humidity of the surrounding environment, it is essential that all weighings be made rapidly. It is further recommended that no weighings of samples be carried out if the humidity of the atmosphere at the balance varies by more than ± 20 % from the humidity conditions under which the sample was equilibrated.

8.3 *Oven Drying:*

8.3.1 Place the tannin dish containing the test specimen on a shelf in the drying oven at 100 ± 2.0°C for 17 ± 1 h.

8.3.2 Remove the dish from the oven and place it in a dessicator until it has reached temperature equilibrium with the laboratory environment.

8.3.3 Record the weight of the dish with dried material as W_3 to the nearest ± 0.0001 g.

9. Calculation

9.1 Calculate the percent volatile matter (moisture) in the sample material as follows:

$$\text{moisture (volatile matter)},\ \% = [(W_2 - W_3)/(W_2 - W_1)] \times 100 \quad (1)$$

where:

W_1 = weight (grams), tare weight of tannin dish,
W_2 = weight (grams), tannin dish plus original specimen, and
W_3 = weight (grams), tannin dish plus oven-dried specimen.

9.2 Two specimens from each material sample shall be tested. The average (mean) of the results from the two specimens shall be taken as the moisture in the sample, except as described in 9.3.

9.3 When the original material was so wet that it had to receive a preliminary drying before being ground in Practice D6405, the moisture content of the original material shall be determined as follows:

9.3.1 The whole of the original, wet, sample shall be weighed. After drying as described in Practice D6405, the weight of the partially dried sample shall be determined. The moisture loss in this preliminary drying shall be calculated as follows:

$$\text{moisture loss (preliminary drying step)},\ \% = [(W_A - W_B)/(W_A)] \times 100 \quad (2)$$

where:

W_A = weight (grams), the original wet sample, and
W_B = weight (grams), the partially dried sample.

9.3.2 The residual moisture in the partially dried and finely ground sample shall be determined as described in Section 8 and Section 9.

9.3.3 Then the total moisture in the original, wet sample shall be calculated as follows:

$$\text{total moisture (original, wet sample)},\ \% = A + \{B \times [(100 - A)/100]\} \quad (3)$$

where:

A = (%) moisture loss (preliminary drying step), and
B = (%) residual moisture content in the partially dried sample.

10. Report

10.1 Report the moisture as percent volatile matter in the material to the nearest 0.01 %.

11. Precision and Bias

11.1 This test method is adopted from Method A6 of The Official Methods of the ALCA. This test method has long been in use and was approved for publication before the inclusion of precision and bias statements were mandated. The original inter-laboratory test data is no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias (or reproducibility) of this test method is adequate for the contemplated use.

11.2 The moisture content obtained by this test method is operationally defined as the weight loss of the specimen after oven drying. There is no independent measure of the moisture content of a sample. Therefore the bias cannot be related to the true moisture content of the sample.

12. Keywords

12.1 moisture; tannin analysis; vegetable tannin analysis; volatile matter