



Designation: F3606 – 22

Standard Guide for Additive Manufacturing — Feedstock Materials — Testing Moisture Content in Powder Feedstock¹

This standard is issued under the fixed designation F3606; 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 standard provides guidelines for measuring moisture in powder feedstock used in additive manufacturing (AM). It applies to metallic, ceramic, and polymer AM powder feedstocks.

1.2 This guide provides a description of test methods commonly used to measure moisture and references to their associated standards.

1.3 This guide provides best practice guidance on how to apply the test methods to make them suitable for AM powder characterization.

1.4 This guide is suitable for measuring moisture in AM powder feedstock over the range of 10 $\mu\text{g/g}$ to 10 000 $\mu\text{g/g}$.

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

1.6 *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.*

1.7 *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

[B215 Practices for Sampling Metal Powders](#)

[B243 Terminology of Powder Metallurgy](#)

[D6869 Test Method for Coulometric and Volumetric Determination of Moisture in Plastics Using the Karl Fischer Reaction \(the Reaction of Iodine with Water\)](#)

[D7191 Test Method for Determination of Moisture in Plastics by Relative Humidity Sensor](#)

[E473 Terminology Relating to Thermal Analysis and Rheology](#)

[E1131 Test Method for Compositional Analysis by Thermogravimetry](#)

[E1142 Terminology Relating to Thermophysical Properties](#)

[E1409 Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion](#)

[E1868 Test Methods for Loss-On-Drying by Thermogravimetry](#)

2.2 *ISO/ASTM Standard:*²

[ISO/ASTM 52900 Additive manufacturing — General principles — Fundamentals and vocabulary](#)

2.3 *ISO Standards:*³

[ISO 17034 General requirements for the competence of reference material producers](#)

[ISO 3954 Powders for Powder Metallurgy Purposes — Sampling](#)

3. Terminology

3.1 *Definitions*—Powder metallurgy terms are defined in Terminology [B243](#). Additive manufacturing (AM) terms can be found in [ISO/ASTM 52900](#). Technical terms in relation with thermogravimetry (TGA) and loss on drying (LOD) are defined in Terminologies [E473](#) and [E1142](#), respectively. Relative humidity sensor terminology is in accordance with Terminology [D7191](#), while terms related to the Karl Fischer method are described in Test Method [D6869](#).

4. Summary of Guide

4.1 Different methods can be used to measure moisture in AM powders. For all methods of interest, the sample of powders are heated in the instrument to vaporize the water. The amount of water is then evaluated using different approaches

¹ This guide is under the jurisdiction of ASTM Committee [F42](#) on Additive Manufacturing Technologies and is the direct responsibility of Subcommittee [F42.01](#) on Test Methods.

Current edition approved Nov. 1, 2022. Published December 2022. DOI: 10.1520/F3606-22.

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 Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <https://www.iso.org>.

and procedures, depending on the method, as defined in the referenced standards. In coulometric titration methods (for example, Karl Fisher as described in Test Method [D6869](#)) and relative humidity sensor methods (as described in Test Method [D7191](#)), a dry gas carries the volatiles (evaporated when the samples are heated up) into a titration cell (Karl Fischer) or humidity sensor (relative humidity sensor) to measure the amount of water. For gravimetric or loss-on-drying (LOD) methods (as described in Test Methods [D6869](#), [D7191](#), [E1131](#), and [E1868](#)), the weight of the samples is measured during the test and the weight variation is converted into a water content assuming that water is the only compound evaporating from the sample during the test and there is no decomposition or modification of the sample during the test. Presence of other volatiles and the decomposition or reaction of the sample with water or atmosphere may all influence the results.

4.2 In addition to the methodology proposed in standards on coulometric titration, relative humidity sensors and LOD (see Referenced Documents, Section 2), it is recommended to follow the guidelines and recommendations of this guide if measuring moisture in AM powder feedstocks.

5. Significance and Use

5.1 This guide will help manufacturers and users of AM powder feedstocks to identify suitable methods for measuring moisture in the feedstocks.

5.2 This guide will aid control of powder quality and allow powder producers and users of AM machines to assess moisture content of virgin and reused powders.

5.3 This guide is intended to support acceptance and control tests.

5.4 Moisture levels are usually relatively low in metallic powder feedstocks (typically lower than 250 µg/g) but could be significantly more important in polymer and ceramic (typically lower than 10 000 µg/g).

5.5 Moisture may affect powder processability (powder supply and feeding, layer creation) and influence the process and properties of the printed components. As different processes and machines use powders with different characteristics (that is, particle size distribution and shape) and AM machines store and handle powders in different ways, the amount of moisture and its impact may vary significantly depending on the feedstock, process, and machine.

5.6 A proportion of the water is physisorbed on the surface and can be easily adsorbed and desorbed.

5.7 A fraction of the water can be strongly bonded to the surface of the powder (that is, chemisorbed) and can be difficult to extract even at temperatures significantly higher than 100 °C. Thus, the water may not all be recovered during the moisture analysis and some water may remain in the samples. Consequently, the values obtained during the tests may be underestimated. As water bonds differently to different materials, the evaporation of water as a function of temperature may vary from one material to another.

5.8 Because of the reactive nature of powders, water may react with the surface of the powder and form oxides and

hydroxides. Thus, the amount of moisture may change with time even if the powder is stored in a tightly sealed container. Reaction of the powder with water may also happen during the analysis as the powder is heated up. This reaction will reduce the amount of water available at the surface of the powder and may impact the results (that is, underestimate the amount of water measured). If such reactions are expected to occur, their impact on the measurements should be evaluated. This can be done using oxygen analysis to evaluate the amount of oxide formed with time or during a test.

5.9 The amount of water adsorbed on the surface of a powder depends on temperature and relative humidity and is determined by moisture sorption isotherm (water content in equilibrium on a material surface at a given temperature and moisture content). Depending on the temperature and humidity content of the atmosphere, water can adsorb and desorb from the surface of the material to reach an equilibrium with its environment.

5.10 In consideration of [5.6 – 5.9](#), the amount of moisture in powders may change progressively and be affected by the storage, handling, and conditions of utilization. Thus, moisture content should only be measured at the time of interest (for example, shipping, reception, and usage). If not, evaluating how moisture and oxygen content evolve with time is recommended. This can be done by exposing the powder to humidity and evaluating how the moisture and oxygen content (using inert gas fusion method such as described in Test Method [E1409](#)) change with time. The effect of handling can be evaluated by measuring the moisture in test samples before and after a selected operation (for example, sieving, splitting). The stability of the moisture content depends on the nature and specific surface area of the powder and shall be evaluated for every material to be tested.

5.11 The optimum test temperature depends on the material and equipment used. Test conditions should be selected to recover the maximum amount of water while the AM feedstock is not modified or deteriorated. For most equipment and AM powders, the maximum temperature of the equipment is not high enough to recover all the water from the samples and the amount of water is usually underestimated.

5.12 To determine the most suitable test temperature for a specific material, tests can be performed at different temperatures from 50 °C up to the maximum temperature of the equipment. The suitable temperature can be chosen to evaporate the maximum amount of water while avoiding a modification (for example, oxidation or degradation) of the samples during the test. For some materials, it may not be possible to recover all water before the onset of the modification of the material. Consequently, the selection of the test temperature shall be selected case by case.

5.13 Results can only be compared if the tests are conducted under similar conditions (temperature, time, heating rate, gas flow rate, and end criteria) and test methods have been validated and compared with reference materials (see Section 8).

5.14 Depending on the design of the equipment, evaporation conditions (effective temperature seen by the samples, gas