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**Fine ceramics (advanced ceramics,
advanced technical ceramics) —
Determination of drying loss of
ceramic granules**

*Céramiques techniques — Détermination de la perte au séchage des
granulés de céramique*



Reference number
ISO 17860:2014(E)

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Foreword

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The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of drying loss of ceramic granules

1 Scope

This International Standard specifies the testing method to determine the drying loss of granulated ceramic powders. The drying loss of granulated ceramic powders means the loss of mass that occurs from drying under specified conditions.

2 Principle

Granulated ceramic powders may contain water, solvent, organic additives, and other volatile matters. Because water, solvent, and, to some extent, also organic additives are removed from granulated powders by drying at high temperature, a mass loss occurs after drying under specified conditions. The mass loss of granulated ceramic powder divided by its initial mass gives its drying loss in percentage by mass.

3 Apparatus

3.1 Drying oven

A drying oven capable of maintaining temperatures up to 110 °C with a precision of ± 5 °C and circulating air in oven. The type of oven shall be stated in the test report, as the design of the oven can influence the test result.

3.1.1 Flat-bottomed dish

A flat-bottomed metallic dish (for example, tin or aluminium), approximately 50 mm in diameter and 30 mm in height. The dimensions of the dish are not critical, but the base should be flat to ensure good thermal contact and to permit the test portion of the powder to be spread to a thin, even layer (the thickness of powder can have a significant influence on the test result).

3.1.2 Balance

A balance capable of weighing at least 10 g with a precision of $\pm 0,001$ g (1 mg).

3.1.3 Desiccator

A desiccator containing a desiccant such as dried calcium chloride impregnated with cobalt chloride.

3.2 Moisture analyser

A moisture analyser with a precision of $\pm 0,001$ g (1 mg) that has a halogen or infrared heating source (instead of the apparatus described in [3.1](#)).

4 Sampling

4.1 The granulated ceramic powder should be tested in the as-received condition.

4.2 The test shall be carried out on three different test portions. For routine in-process-quality-control-measurements, only one test is sufficient.

NOTE Select representative test sample using a sample splitter or equivalent method.

5 Procedure

5.1 Procedure 1: Drying oven

When using a drying oven described in [3.1](#), procedure is as follows.

5.1.1 Dry an empty and clean flat-bottomed dish at $(110 \pm 5)^\circ\text{C}$ for at least 2 h and allow it to cool down to room temperature in a desiccator.

5.1.2 Weigh the empty dish to the nearest 1 mg.

5.1.3 Put a granulated ceramic powder into the dish and spread it evenly over the bottom of the dish by gentle movement of the dish until it gives a layer with a thickness of about 5 mm to 10 mm.

5.1.4 Weigh the as-received powder in the dish to the nearest 1 mg (m_1).

5.1.5 Place the dish with the powder in an oven and dry it at $(110 \pm 5)^\circ\text{C}$ for 2 h. When the drying is completed, transfer the dish to a desiccator and allow it to cool to room temperature.

NOTE If organic additives contained in granulated ceramic powder decompose or evaporate at 110°C , select a lower temperature to dry the dish with the powder considering the nature of the material. The condition chosen shall be shown to be adequate for complete drying using a separate series of tests. Record the temperature in the test report.

5.1.6 Weigh the dried powder in the dish to the nearest 1 mg (m_2).

5.1.7 Compare the mass of dried powder (m_2) with that of as-received powder (m_1). If a mass loss by drying is more than 3 mg, repeat [5.1.5](#) to [5.1.7](#) until it becomes less than 3 mg.

5.1.8 Repeat [5.1.1](#) to [5.1.7](#) with two additional portions and average the results.

5.2 Procedure 2: Moisture analyser

When using a moisture analyser described in [3.2](#), procedure is as follows.

5.2.1 Select proper parameters for sample mass, drying temperature, and abort criterion. Sample mass shall be within the range of 5 g to 10 g. Drying temperature shall be selected under consideration of the constituents of the powder, in particular of the organic additives. Drying shall not result in the decomposition of constituents of the powder. Possible abort criteria are related to duration or to mass change. In case of a duration-related abort criterion, the chosen drying duration shall be sufficient for complete drying. This shall be proved in advance in a measurement series using different drying durations. Use a duration at which the difference of the result compared to that of a shorter duration is less than 0,02 %. In case of a mass change-related abort criterion, it shall be 0,002 g/60 s or smaller.

5.2.2 Perform the measurement according to the operating instructions of the moisture analyser.

5.2.3 Repeat [5.2.2](#) with two additional portions and average the results.

6 Calculation

When using procedure 1, calculate the drying loss of the granulated ceramic powder, L , expressed as percentage by mass, with three significant digits, using Formula (1):

$$L = \frac{m_1 - m_2}{m_1} \times 100 \quad (1)$$

where

- L is the drying loss, in percentage by mass, of the granulated ceramic powder;
- m_1 is the mass, in grams, of the not-dried granulated ceramic powder in the dish;
- m_2 is the mass, in grams, of the dried granulated ceramic powder in the dish.

When using procedure 2, the drying loss of the granulated ceramic powder, L , expressed as percentage by mass, is calculated by the software of the moisture analyser, using Formula (1) and the result is displayed.

7 Test report

The test report shall be in accordance with the reporting provisions of ISO/IEC 17025 and shall contain the following:

- a) the name of the testing establishment;
- b) date of the test, report identification and number, operator, signatory;
- c) temperature and relative humidity in laboratory;
- d) a reference to this International Standard (i.e. ISO 17860:2014);
- e) all details for identification of the granulated ceramic powder (material type, manufacturer, batch or code number);
- f) the type of the dish used (shape, diameter, height, etc.);
- g) the type of oven used;
- h) the type of moisture analyser used (when using a moisture analyser);
- i) the abort criterion used (when using a moisture analyser);
- j) drying temperature;
- k) the result obtained: drying loss (%);
- l) any unusual features noted during the determination;
- m) comments about the test or test results.

