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Fine ceramics (advanced ceramics, advanced technical ceramics) — Sample preparation for the determination of particle size distribution of ceramic powders

Céramiques techniques — Préparation de l'échantillon pour la détermination de la répartition granulométrique des poudres céramiques

Reference number
ISO 14703:2008(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14703 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

This second edition cancels and replaces the first edition (ISO 14703:2000), which has been technically revised.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Sample preparation for the determination of particle size distribution of ceramic powders

1 Scope

This International Standard specifies a general wet-sample preparation technique common to the size analysis of powdered fine ceramic materials. The analyzed size distribution of fine particles is strongly dependent on the sample preparation.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 8213:1986, *Chemical products for industrial use — Sampling techniques — Solid chemical products in the form of particles varying from powders to coarse lumps*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
fine particles
particles of which sizes are less than a few micrometres

3.2
sample preparation
process that includes deagglomeration, dispersion of a test sample and treatment of suspension in stages up to taking the test portion

3.3
test sample
representative part taken from a quantity of material

3.4
test portion
quantity of material taken from the test sample entirely used in the test

3.5
dispersing medium
liquid in which the sample is dispersed and suspended

3.6
Na-HMP
sodium hexametaphosphate

4 Sampling

The sampling method, from a quantity of material to the test sample, shall comply with the requirements specified in ISO 8213, otherwise the methods specified in a national standard, or as mutually agreed upon, shall be complied with.

5 Dispersing media

5.1 Types of dispersing media

5.1.1 Aqueous solution of Na-HMP

A solution of Na-HMP with a mass concentration of 0,01 % to 0,1 % should be used for the samples that are insoluble or can be regarded as insoluble. The dispersing medium shall be used no more than two days after preparation. The water should be equivalent to the water of type 1 defined in ISO 3696.

5.1.2 Ethanol

Ethanol of analytical grade or better should be used for samples soluble in, or reactive with, water.

5.1.3 Other dispersing media

In case sufficient dispersing effects are not obtained with Na-HMP water solution or ethanol, other dispersing media, such as polymer dispersant solution, can be used but the following conditions shall be satisfied.

a) The dispersing medium shall not contain solid or liquid particles.

b) The dispersing medium shall wet the sample material thoroughly.

c) The dispersion medium shall not cause the particulate material to dissolve, swell, shrink or coagulate.

d) The dispersing medium shall not foam during ultrasonic agitation.

e) Analytical grade or better reagents shall be used for the dispersing medium.

f) For wet sedimentation methods, the dispersing medium and the sample material shall have different densities.

g) For the laser diffraction method, the dispersing medium and the sample material shall have different refractive indices.

5.2 Evaluation of dispersed conditions for the selection of dispersing medium

The evaluation of dispersed conditions should be made by one or more of the following techniques, for selection of the dispersant medium or the dispersant concentration.

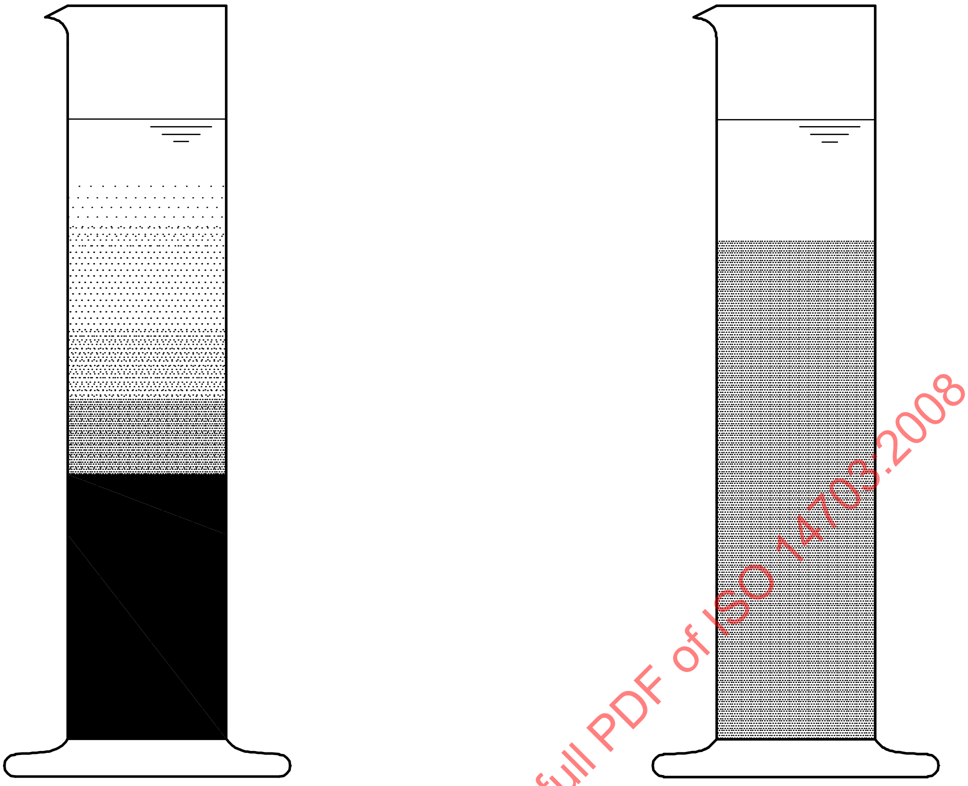
a) Measurement of zeta-potential: the greatest absolute value of zeta-potential, preferably more than 60 mV, is required.

b) Observation of sedimentation process: no clear boundary between the supernatant and the settling suspension shall be detected (see Figure 1).

c) Observation of sediment: the sediment shall be as dense as possible (see Figure 2).

d) Measurement of particle size distribution: the finest particle size distribution curve shall be obtained from the best dispersed particles.

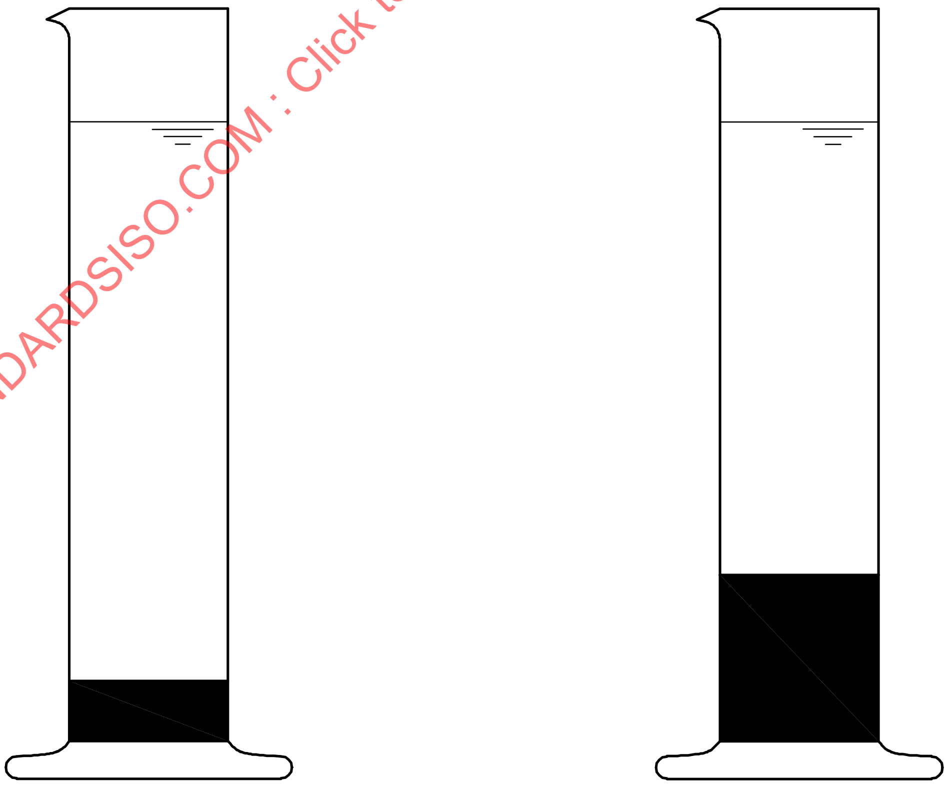
The method of zeta-potential measurement should comply with the methods specified in a national standard or as mutually agreed upon.



a) Well dispersed condition

b) Poorly dispersed condition

Figure 1 — Sedimentation process



a) Well dispersed condition

b) Poorly dispersed condition

Figure 2 — Sediments

6 Deagglomeration of sample

6.1 In order to improve the reproducibility of the size analysis, the test sample should be deagglomerated by means of a mortar and pestle, using the following procedure.

6.2 The weighed sample is placed in a dry mortar.

6.3 The sample is strongly disintegrated with a pestle using the whole inner wall of the mortar until most of the sample adheres to it.

6.4 The sample adhering to the mortar wall is scraped off with a spatula or equivalent and collected at the bottom of the mortar.

6.5 The procedures in 6.3 and 6.4 are repeated five times.

NOTE 1 Where it is known that the sample consists of separated primary particles, deagglomeration is not needed.

NOTE 2 Deagglomeration is not needed to determine the particle size of the sample "as received" or "as is".

7 Suspension and dispersion of sample

7.1 Suspension of sample

The sample shall be suspended in the dispersing medium at a given concentration. The concentration shall be dilute but higher than the proper concentration for the instrumental technique used. As the beaker capacity and the amount of suspension could affect the dispersing conditions, they shall be recorded.

7.2 Ultrasonic agitation of sample

7.2.1 Ultrasonic devices

Dispersion of the sample shall be made with one of the following devices.

a) Ultrasonic bath: the dispersion shall be made with the amount of water in the ultrasonic bath controlled in order to obtain the strongest dispersing effect, and with the beaker set at the position undergoing the strongest ultrasonic intensity.

b) Ultrasonic homogenizer: the suspension shall not be contaminated by foreign particles generated from its tip. Its output, tip size and tip position may influence the dispersing condition and therefore shall be recorded.

NOTE Where foreign particles are magnetic materials, they can be eliminated by mixing with a magnet stirrer.

If the ultrasonic agitation causes a noticeable increase in temperature of more than a few degrees Celsius, the samples should be cooled using water chilled with ice.

7.2.2 Ultrasonic agitation time

The sample suspension shall be agitated until the obtained particle size, for example median diameter, becomes constant or minimum with the agitation time. If the obtained particle size continues to decrease with the agitation time, the agitation can be stopped at a certain time at which the size analysis shall be conducted and recorded.

NOTE Ultrasonic agitation is not needed for size analysis of the sample "as received" or "as is".