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Advanced technical ceramics - Methods of test for ceramic powders - Part 5: Determination of particle size distribution

Hochleistungskeramik - Prüfverfahren für keramische Pulver - Teil 5: Bestimmung der Teilchengrößenverteilung

Céramiques techniques avancées - Méthodes d'essais pour poudre céramiques - Partie 5: Détermination de la distribution granulométrique

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Advanced technical ceramics - Methods of test for ceramic powders - Part 5: Determination of particle size distribution

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Foreword

This document (EN 725-5:2007) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2007, and conflicting national standards shall be withdrawn at the latest by July 2007.

This document supersedes EN 725-5:1996.

EN 725 *Advanced technical ceramics — Methods of test for ceramic powders* was prepared in Parts as follows:

Part 1: *Determination of impurities in alumina*

Part 2: *Determination of impurities in barium titanate*

Part 3: *Determination of the oxygen content of non-oxides by thermal extraction with a carrier gas*

Part 4: *Determination of oxygen content in aluminium nitride by XRF analysis*

Part 5: *Determination of particle size distribution*

Part 6: *Determination of the specific surface area* [withdrawn]

Part 7: *Determination of the absolute density* [withdrawn]

Part 8: *Determination of tapped bulk density*

Part 9: *Determination of un-tapped bulk density*

Part 10: *Determination of compaction properties*

Part 11: *Determination of densification on natural sintering*

Part 12: *Chemical analysis of zirconia*

Parts 6 and 7 of the series were superseded in 2005 by EN ISO 18757 and EN ISO 18753 respectively.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This Part of EN 725 describes the preparation of suspensions and calibration of apparatus, prior to the measurement of particle size distribution of powders used for advanced technical ceramics.

The preparation described is appropriate for measurements either by the sedimentation method or the laser light scattering method.

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.

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:2005)*

3 Apparatus

3.1 ***Glass microscope slides and cover slips***.

3.2 ***Optical microscope***.

3.3 ***Beaker,*** 50 ml to 100 ml.

3.4 ***Ultrasonicator***.

3.5 ***Magnetic stirrer***.

3.6 ***Particle size analyser***.

4 Preparation of the suspension

4.1 Selection of liquid

The dispersing liquid for the suspension shall not react with or dissolve the powder.

For the sedimentation method of analysis, the density of the liquid shall be less than that of the powder by at least 0,5 g/cm³.

For the laser light scattering method of analysis, the liquid shall be optically transparent for the wavelength used.

NOTE 1 This is generally 633 nm.

The liquid shall have a refractive index which is substantially different from that of the sample.

NOTE 2 Any specific manufacturer's instructions regarding the refractive index of the dispersing liquid should be taken into account.

The liquid for the suspension shall be selected, together with any dispersing agent, from those given in Annex A.

NOTE 3 Additional information is given in the bibliographical references listed.

The dispersion of powder in the liquid shall be checked by one of the methods given in 4.2.

4.2 Dispersion checking

4.2.1 Optical microscopic examination

Place a drop of the prepared suspension on the glass slide (**3.1**) of a microscope (**3.2**) and carefully cover with a cover slip (**3.1**). Observe the preparation under a suitable magnification, to determine if particles are completely separated and well dispersed, or if they are gathered together in chains or clusters.

NOTE This method is not suitable for powders with particle diameters of less than 5 µm.

4.2.2 Qualitative test by sedimentation

Allow the suspension to stand. A correctly dispersed suspension settles less rapidly than a suspension which flocculates, and shows no clear border line between the liquid which becomes clear and the layer which is still turbid as sedimentation proceeds. The sediment obtained is compact and of a minimal volume.

4.2.3 Scanning electron microscope (S.E.M.) examination

Check the correlation of the particle size distribution obtained with the mean size of the ultimate particles observed by S.E.M. If the suspension is not sufficiently dispersed, particles are partially aggregated and particle size distribution measurements thus give values much higher than the mean size of the ultimate particles observed.

4.2.4 Quantitative test by sedimentation

Perform the quantitative tests, while allowing a variation of the various parameters liable to influence the dispersion quality. Choose the dispersion procedure which gives the smallest mean particle diameter. An examination of the shape of the distribution can give an indication of the presence of agglomerates.

5 Calibration

Check the working order of the apparatus, by analysing standard powders regularly.

NOTE Information on available standard reference powders is provided in Annex B.

6 Procedure

6.1 Determine the quantity of powder, the type and quantity of suspending liquid and the dispersing agent to be used.

NOTE In principle, and within the limits imposed by the instrument, the sample mass to be dispersed has no influence on the results. However, it is preferable to use dilute suspensions.

6.2 For the sedimentation method, ensure that the volume concentration is less than or equal to 0,2 %.

6.3 For the laser light scattering method, for all determinations to be accurate, ensure that all particles present in the pencil rays are separate and diffract independently from each other.

NOTE This condition should be fulfilled when each particle with a radius *a* is in the centre of a circle with a radius *R* = *a*, when there is no secant circle and when no particle casts a shadow on the others.

6.4 In a 50 ml to 100 ml beaker (**3.3**) prepare a first test sample. Mix the suspending liquid and the dispersing agent, add the powder while stirring the suspension, and disperse using the ultrasonicator (**3.4**) until free of agglomerates. Continue stirring with a magnetic stirrer (**3.5**) until the start of the analysis.

6.5 Read the technical instructions of the instrument for general adjustments prior to the test and follow the manufacturer's recommendations for using the instrument when performing the test.

6.6 Repeat the procedure on a second test sample and plot the particle size distribution curves. If the curves are similar, it may be deduced that the dispersion is stable and that no mistake has been made during the test. If the curves are not similar, check the dispersion again (see 4.2).

7 Expression of results

Record the suspension and dispersion conditions in a table similar to the example given in Annex C.

Present the results either in graphic form as in Annex D, with the cumulative particle size distribution curve obtained automatically on the measurement sheet of the instrument, specifying test conditions in the appropriate part of the sheet, or as a table of results: a recommended layout example is given in Annex E.

8 Test report

The test report shall be in accordance with the reporting provisions of EN ISO/IEC 17025 and shall include at least the following information:

a) name and address of the testing establishment;

b) date of the test;

c) on each page, a unique report identification and page number;

d) customer name and address;

e) reference to this standard, i.e. determined in accordance with EN 725-5;

f) authorising signature;

g) any deviation from the method described, with appropriate validation, i.e. demonstrated to be acceptable to the parties involved;

h) description of the test material (manufacturer, type, batch or code number, date of receipt) including any treatment before testing;

i) suspension and dispersion conditions in the form of a table similar to the example given in Annex C;

j) reference powder used to calibrate the instrument (see clause 5) and the results obtained on a given date;

k) for the sedimentation method only, the apparent density of the powder, apparent density and viscosity of the suspending liquid, displacement rate of the cell, temperature of the suspension and the initial diameter;

l) comments about the test or test results.

Annex A
(informative)

Suspending liquids and dispersing agents

The following alphabetical list in Table A.1 gives examples of suspending liquids and dispersing agents most commonly used for the main technical ceramic powders.

Surface characteristics, and consequently dispersion behaviour, depend on the powder type, but also on its manufacturing process. Therefore, the suspending liquid and dispersing agent may vary among powders of the same type.

Among the dispersing agents listed in the third column of Table A.1, 9 are designated by a number (d.a No. 1 to No 9). Their chemical composition is as follows:

d.a. No 1 Dioctylsulfosuccinates;

d.a. No 2 Trimethylcetyl ammonium bromide;

d.a. No 3 Polyoxyethylene nonylphenol;

d.a. No 4 Linear polyethoxy derivates;

d.a. No 5 Sodium alkylnaphthalene sulfonate;

d.a. No 6 Sorbitol monolaurate;

d.a. No 7 Polyoxyethylene alkylphenol;

d.a. No 8 Sodium alkylsulfonate;

d.a. No 9 Polyoxyethylene octylphenol.