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

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Hochleistungskeramik - Prüfverfahren keramische Pulver - T eil 5: Bestimmung der Teilchengrößenverteilung

Céramiques techniques avancées - Méthodes d'essai pour les poudres céramiques - Partie 5: Détermination de la distribution granulométrique

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CEN

European Committee for Standardization
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Foreword

This European Standard has been prepared by the Technical Committee CEN/TC 184 "Advanced technical ceramics" of which the secretariat is held by BSI.

EN 725 "Advanced technical ceramics - Methods of test for ceramic powders" consists of 11 Parts:

Part 1 :	Determination of impurities in alumina
Part 2 :	Determination of impurities in barium titanate (ENV)
Part 3 :	Determination of oxygen content of non-oxides by thermal extraction with a carrier gas
Part 4 :	Determination of oxygen content of aluminium nitride by XRF (ENV)
Part 5 :	Determination of particle size distribution
Part 6 :	Determination of the specific surface area
Part 7 :	Determination of absolute density
Part 8 :	Determination of tapped density
Part 9 :	Determination of untamped density
Part 10 :	Determination of compaction properties
Part 11 :	Determination of densification on natural sintering (ENV)

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of an identical text or by endorsement, at the latest by July 1996, and conflicting national standards shall be withdrawn at the latest by July 1996 .

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, 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 is appropriate for measurements either by the sedimentation method, with the detection of X-ray absorption, or the laser light scattering method.

2 Preparation of the suspension

2.1 Selection of liquid

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

For the sedimentation method, the density of the liquid shall be less than that of the powder by at least 0,5 g/cm^3.

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

NOTE : This is generally 633 nm.

The liquid shall have a refractive index which is substantially different from that of the sample. Any specific manufacturer's instructions shall be considered.

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

NOTE : Additional information is given in the references listed in annex B.

The dispersion of powder in the liquid shall be checked by one of the methods given in 2.2.

2.2 Dispersion checking

2.2.1 *Optical microscopic examination*

A drop of the prepared suspension is placed on the glass slide of a microscope and is then carefully covered with a cover slip. The observation of the preparation with a suitable magnification allows one 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 $< 5\ \mu m$.

2.2.2 *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.

2.2.3 *Scanning electron microscope examination (S.E.M.)*

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.

2.2.4 *Quantitative test by sedimentation*

Perform the quantitative tests, while allowing a variation of the various parameters liable to influence the dispersion quality and 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.

3 Calibration

In order to check the apparatus, standard powders shall be analyzed regularly.

Suitable powders are available from the B.C.R. (Community Bureau of Reference). Their particle size distribution is expressed in the form of cumulative curves. If dispersion and test conditions are rigorously identical for the successive analyses of standard powders, the lack of curve deviation gives an indication of the good working order of the apparatus. As the curves of the standard powders are given as equivalent Stokes' diameters, those obtained from the sedimentation method by gravity and detection of X-ray absorption should be comparable.

Four of these reference powders show a particle size distribution in the range of 0,1 μm to 100 μm; their characteristics are summarized in table 1:

Table 1 : Standard reference powders

Reference	Type	Size range
CRM 066	Quartz powder	0,35 μm to 3,50 μm
CRM 067	Quartz powder	2,4 μm to 32 μm
CRM 069	Quartzic sand	14 μm to 90 μm
CRM 070	Quartz powder	1,2 μm to 20 μm
For more information, contact Community Bureau of Reference Directorate General for Science, Research and Education Rue de la Loi, 200 BRUSSELS B 1049		

4 Procedure

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

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.

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

If particles travel through the laser beam inside a liquid film of thickness *e*, the maximum sample quantity is given by:

$$Q_m = \frac{4}{3\alpha^2} \frac{a}{e} \rho V$$

where:

a mean radius of particles, in millimetres;
e liquid film thickness;
ρ sample density, in gramms per cubic centimetres;
V total volume of carrier liquid in cubic centimetres;
α proportionality factor;
Q_m maximum sample quantity in gramms.

4.2 In a 50 ml to 100 ml beaker prepare a first test sample. Mix the suspending liquid and the dispersing agent, add the powder while stirring the suspension, and disperse ultrasonically until free of agglomerates. Continue stirring with a magnetic agitator until the start of the analysis.

4.3 Read the technical instructions of the instrument for general adjustments prior to the test, and perform the test following the manufacturer's recommendations.

Repeat the procedure on a second test sample and plot the particle size distribution curves. If both curves are sufficiently similar, this allows one to ascertain 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 2.2).

5 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.