BRITISH STANDARD

BS EN 725-5 : 1996

Advanced technical ceramics —
Methods of test for ceramic powders

Part 5. Determination of the particle size distribution

The European Standard EN 725-5 : 1996 has the status of a British Standard

ICS 81.060.10

Committees responsible for this British Standard

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AEA Technology
Aluminium Federation
British Ceramic Research Ltd.
British Industrial Ceramic Manufacturers' Association
Department of Trade and Industry (National Physical Laboratory)
Flat Glass Manufacturers' Association
GAMBICA (BEAMA Ltd.)
Institute of Refractories Engineers
Ministry of Defence
Refractories Association of Great Britain
Society of British Aerospace Companies Ltd.
University of Manchester

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Contents

National foreword

This British Standard has been prepared by the Technical Committee RPI/13 and is the English language version of EN 725-5 : 1996 *Advanced technical ceramics — Methods of test for ceramic powders* — Part 5: *Determination of the particle size distribution*, published by the European Committee for Standardization (CEN).

EN 725-5 : 1996 was produced as a result of international discussions in which the United Kingdom took an active part.

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EUROPEAN STANDARD
NORME EUROPÉENNE
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EN 725-5

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Descriptors: Ceramics, powdery materials, grain size analysis, particle size, analysis methods, sedimentation, radiation tests, laser radiation, diffraction

English version

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

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

Hochleistungskeramik — Prüfverfahren keramischer Pulver — Teil 5: Bestimmung der Teilchengrößenverteilung

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN
European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

This European Standard has been prepared by Technical Committee CEN/TC184, 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 Common 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.

Contents

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 µm.

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 analysed 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:

$$Q_\mathrm{m} = \frac{4}{3\,\alpha^2} \cdot \frac{a}{e} \cdot \rho \cdot V$$

where

a = mean radius of particles (mm);

e = liquid film thickness (mm);

ρ = sample density (g/cm^3);

V = total volume of carrier liquid (cm^3);

α = proportionality factor;

Q_m = maximum sample quantity (g).

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.

6 Test report

The test report shall contain the following information:

a) the name of the testing establishment;

b) date of the test, unique identification of report and of each page, customer name and address and signatory;

c) a reference to this European Standard, i.e. determined in accordance with EN 725-5;

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

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

f) the reference powder used to calibrate the instrument (see clause **3**) and the results obtained on a given date;

g) 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;

h) 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;

Table A.1 Suspending liquids and dispersing agents

Material	**Suspending liquid**	**Dispersing agent** (0,5 g/l to 1,0 g/l, unless otherwise stated)
Alumina	see aluminium (oxide)	
Aluminium (oxide)	water	Sodium pyrophosphate
	water	Sodium pyrophosphate
	water	Sodium tartrate
	water	d.a. No. 3 or No. 4 or No. 5 or No. 9
	water	hydrochloric acid (pH 3)
	n-butanol	
	n-butylamine	
	linseed oil/xylene	
	iso-octane	d.a. No. 6
Barium carbonate	cyclohexanol, methanol	
Barium titanate	water	d.a No. 8
Boron (amorphous)	n-butanol	
	n-butanol/ethanol (various mixtures)	
Boron carbide	water	sodium pyrophosphate
	n-butanol/ethanol (various mixtures)	
Boron nitride	n-butanol	
	n-butanol/ethanol (various mixtures)	
Boron oxide	water	sodium pyrophosphate
Chromium oxide	water	sodium phosphate
	cyclohexanol/isoamyl alcohol (9 : 1) (*v/v*)	(0,1 g/l to 0,3 g/l)
Corundum	see aluminium oxide	sodium pyrophosphate
Diamond (powder)	olive oil	
	gelatine in water (1 g/l to 2 g/l)	sodium carbonate (pH 9)
	ethanol	
	water	trisodium orthophospate
Graphite	water	0,5 g/l tannic acid
	water	0,5 g/l d.a. No. 1 or No. 8
	water	5 g/l sodium linoleate (0,88 ml/l to 3,5 ml/l)
	water/ammonia	
	ethanol	
	n-butanol	

Table A.1 Suspending liquids and dispersing agents (*concluded*)

Material	**Suspending liquid**	**Dispersing agent** (0,5 g/l to 1,0 g/l, unless otherwise stated)
Magnesium oxide (magnesia)	water	sodium polymetaphosphate
Quartz	see silica	
Rutile	see titanium (dioxide)	
Silica	water	
	water	sodium polymetaphosphate
	water	d.a. No. 3
	water	sodium pyrophosphate
	water	sodium pyrophosphate (1 g/l) + d.a. No. 7 (0,2 g/l)
	ethanol	trisodium orthophosphate
	xylene	
	water/ethanol (1:1) (*v/v*)	
Silicon carbide	water	sodium pyrophosphate
	water	trisodium orthophosphate
	water/glycol ethylene	sodium pyrophosphate
	water	sodium polymetaphosphate
	n-butanol	
Titanium dioxide (rutile)	water	sodium pyrophosphate
	ethanol/water mixture	
	ethanol	
	methanol	
	xylene	
	cyclohexanone	
	linseed oil	
	cyclohexanol/isoamyl alcohol (9:1) (*v/v*)	
	water	sodium polymetaphosphate d.a. No. 2 or No. 3
	water	
	xylene/linseed oil	
Tungsten carbide	glycolethylene	sodium pyrophosphate
	vegetable oil	
Zircon ($ZrSiO_4$)	water	
	water	sodium pyrophosphate
	water/ethanol (1:1) (*v/v*)	
Zirconium dioxide	water	sodium polymetaphosphate
	water	oleic acid
	water	sodium pyrophosphate
	aqueous glycerol	

Annex B (informative)

Bibliography

List indicating suspending liquids and dispersing agents for technical ceramic powders

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NELSON, R. D (1988). Dispersing powders in liquids — Handbook of powder technology, Vol 7, Williams, J.C. and Allen, T. Editors — Elsevier.

PARFITT, G. D (1986). *Dispersion of powders in liquids* third edition— Elsevier Applied Science Publishers.

Annex C (informative)

Example of table recording suspension and dispersion

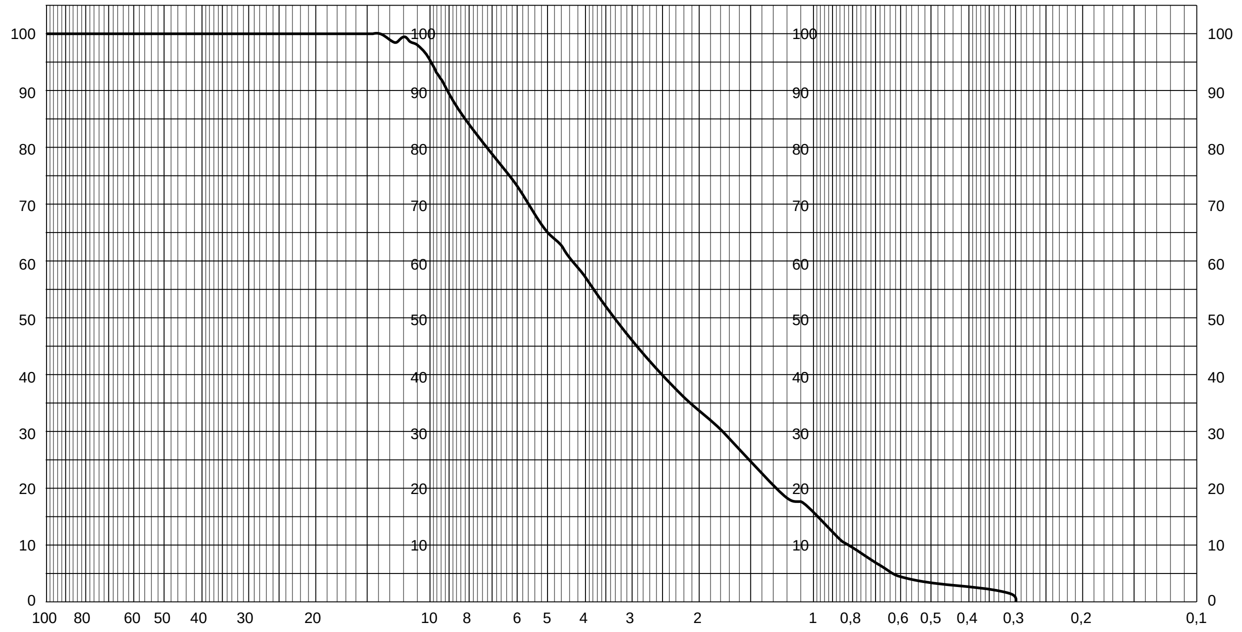
Table C.1 Example of recording suspension and dispersion

Sample type		**Zirconium dioxide**
Laboratory reference		4358
Density of the powder (in g/cm^3)		5,77
Test sample (in g)		2,00
Suspending liquid	type	deionized water
	volume	108 ml
Type of suspending agent		HCl
Weight concentration of dispersing agent with respect to the suspending liquid		0,35 %
Beaker dimension height × diameter		9 cm × 6,5 cm
Stirring before standing	type	magnetic
	duration	6 min
Standing time		24 h
Stirring after standing	type	ultrasonic cell
	duration	30 min
Ultrasonic stirring condition	cell dimension	W = 90 mm, L = 190 mm, H = 60 mm
	frequency	35 kHz
	powder	30 W to 60 W

Annex D (informative)

Example of particle size distribution

Instrument:	Sedigraph 5000	Date:	87-12-10
Sample:	4358	Density (g/cm^3):	5,774
Liquid:	water	Density (g/cm^3):	0,9951
		Viscosity (mPa.s):	0,7679
Operator:	XXX		
Preparation:	US 30 min, pH 8 Temperature (°C): 32		
Rate:	568		
Start diameter:	40 Microns		



Equivalent sphere diameter, in microns

Annex E (informative)

Example of representation of particle size distribution results as a table

Sample reference	4358
Date of the test	87.12.10
Operator	XXX
Powder density (g/cm^3)	5,774
Compartment temperature (°C)	32
Rate	568
Suspending liquid density (g/cm^3)	0,9951
Viscosity (m Pa.s)	0,7679
Equivalent mean diameter (µm)	3,3
Weight fraction inferior to 1 µm (%)	15
Weight fraction superior to 10 µm (%)	5
Difference between the equivalent diameter corresponding to 80 % of the distribution and that corresponding to 20 % of the distribution : d 80 % − d 20 % (µm)	5,5

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