

BRITISH STANDARD

Sampling and analysis of glass-making sands –

Part 2: Methods for chemical analysis

ICS 81.040.10



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Summary of pages

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Foreword

Publishing information

This part of BS 2975 is published by BSI and came into effect on 29 August 2008. It was prepared by Technical Committee LBI/36, *Laboratory glassware and related apparatus*. A list of organizations represented on this committee can be obtained on request to its secretary.

Supersession

This part of BS 2975, along with BS 2975-1:2004, supersedes BS 2975:1988, which is withdrawn.

Relationship with other publications

BS 2975 is published in two parts, as follows:

- Part 1: *Methods for sampling and physical testing of glass-making sands*;
- Part 2: *Methods for chemical analysis*.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its methods are expressed as a set of instructions, a description, or in sentences in which the principal auxiliary verb is “shall”.

Contractual and legal considerations

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Compliance with a British Standard cannot confer immunity from legal obligations.

1 Scope

This British Standard describes methods suitable for the chemical analysis of glass-making sands, including instrumental methods using X-ray fluorescence spectrometry (XRF), inductively coupled plasma optical emission spectrometry (ICP-OES), ultraviolet-visible spectrometry (UV-VIS) and atomic absorption spectrometry (AAS).

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.

BS 2975-1, *Sampling and analysis of glass-making sands – Part 1: Methods for sampling and physical testing of glass-making sands*

BS EN ISO 12677:2003, *Chemical analysis of refractory products by XRF – Fused cast bead method*

3 Type of material

The calibration ranges and methods to be used for the chemical analysis of glass-making sands shall be as set out in Table 1.

Table 1 Calibration ranges and methods

Oxide	Method	Range % m/m	
		Min	Max
LOI	Gravimetric	0.10	1.0
Fe ₂ O ₃	XRF / UV-VIS / ICP-OES	0.005	1.0
Al ₂ O ₃	XRF / ICP-OES / AAS	0.04	5.0
Cr ₂ O ₃	ICP-OES / AAS	0.000 1	0.03
K ₂ O	XRF / Flame Photometer / AAS	0.01	3.0
SiO ₂	XRF / Gravimetric	94.0	99.8
TiO ₂	XRF / UV-VIS	0.016	0.10
CaO	XRF / AAS / Titration	0.006	1.0
MgO	XRF / AAS	0.002	1.0
Mn ₃ O ₄	XRF / AAS	0.0	0.03
S=	XRF / ICP-OES / Combustion	0.0	0.05
Ni	ICP-OES	0.0	0.000 6
Co	ICP-OES	0.0	0.000 1
Cu	ICP-OES	0.0	0.000 35
Na ₂ O	XRF / AAS / ICP-OES / Flame Photometer	0.05	2.0
Pb ₃ O ₄	XRF	0.0	0.000 5
Carbon	Combustion	0.0	0.1

NOTE The analytes are expressed in the stable oxidation state; correction should be made if other oxidation states are needed.

4 Sample preparation

4.1 General

Control (extraction) systems shall be put in place to reduce exposure to respirable crystalline silica dust to the lowest practicable level.

The specimen or solution to be presented to the instrument shall be prepared from a dry homogeneous finely powdered sample (the procedure for drying silica sand is given in 4.3.3).

Appropriate bulk sampling methods shall be used to produce a laboratory sample as set out in BS 2975-1. The particle size of the laboratory sample shall be reduced for both wet chemical and XRF techniques.

The accuracy of XRF methods for pressed powder samples is highly dependent on the particle size distribution of the sample; the particle size of material to be used for the preparation of pressed powders shall be less than 75 µm.

NOTE 1 Particle size reduction can be carried out by grinding in a suitable mechanical device or by hand using appropriate grinding media.

NOTE 2 Consistent grain size may be difficult to obtain if the sample is ground by hand. Commercially available mechanical mills for particle size reduction on a laboratory scale include:

- ball mills, either planetary or centrifugal (3 axes);
- disc mills, also called swing mills, ring-and-puck mills or shatterboxes (2 axes);
- mortar mills (essentially mechanized pestle-and-mortars).

The potential for contamination of the sample with elements that are to be determined should be considered when grinding media are chosen. For this reason carbon steel, chrome steel, and zirconia media are not recommended. Suitable media are shown in Table 2.

4.2 Apparatus

4.2.1 *Drying vessel.*

4.2.2 *Drying oven to 110 °C ± 10 °C.*

4.2.3 *Riffle box.*

4.2.4 *Balance, capable of weighing to 0.01 g.*

4.2.5 *Timer.*

4.2.6 *Extraction system.*

4.2.7 *Pestle and mortar or mechanical mill, type dependent on sample size and intended analysis (see Table 2).*

4.2.8 *Desiccator containing dry desiccant.*

4.2.9 *Fine brush.*

Table 2 Suitable grinding media for silica sands

Recommended media	Recommended use	Typical analysis	Possible contamination	Notes
Tungsten carbide (normally cemented with Co, but Fe, etc., may be used)	XRF fused cast bead	WC 94%, Co 6%	W, Co or other cementation elements, e.g. Ni	Check specification with supplier
Agate	All XRF techniques, wet chemistry	SiO ₂ 99.9%	Insignificant	Use with a slow mill speed
Syalon (silicon nitride, silicon aluminium oxynitride)	All XRF techniques, wet chemistry	Si 53.5%, N 35.2%, O 4.8%, Y (or Er) 3.5%, Al 2.5%, Mg 0.5%	Al, Y (or Er)	Can be brittle
Hard porcelain	All XRF techniques, wet chemistry	Al ₂ O ₃ 40%, SiO ₂ 60%	Al	Used in mortar mills

NOTE Samples may be hand ground in any of the above media, to an appropriate particle size. Sample loss should be avoided. Consideration should be given to manufacturer's recommendations regarding appropriate sample size and milling time.

4.3 Procedure

NOTE This procedure should be carried out with appropriate extraction (4.2.6) control measures to reduce exposure to respirable crystalline silica dust.

4.3.1 Select grinding medium, based on sample size, sample hardness and chemical analysis to be performed (see Table 2). Very small samples can be ground by hand using agate or porcelain pestle and mortar (4.2.7). To minimize particle size effects, prepare the sand by grinding to ensure that the fineness is to the tolerance of 10% maximum retained at 75 µm and 0% retained at 150 µm.

4.3.2 If the sample is dry proceed to 4.3.5. If the sample is moist, use a cone and quartering technique to obtain sufficient sample for the capacity of the grinding vessel to be used.

4.3.3 Dry the moist sand for 1 h or to constant mass in a suitable drying vessel (4.2.1) in an oven at 110 °C ± 10 °C (4.2.2).

4.3.4 Remove from the oven (4.2.2) and cool in a desiccator containing dry desiccant (4.2.8).

4.3.5 Riffle dry sample in a riffle box (4.2.3).

4.3.6 Clean the mill (4.2.7), finishing the process with one of the cleaning agents recommended in A.12.

4.3.7 Weigh on a balance (4.2.4) the recommended amount of sample for the chosen milling vessel.

4.3.8 Place weighed sample into the mill (4.2.7), transfer into the mechanical device and grind for the required time using a timer (4.2.5) if one is not fitted to the device.

4.3.9 Empty the mill (4.2.7), under extraction (4.2.6), onto a clean surface removing as much of the sample as possible using a fine brush (4.2.9).

NOTE An appropriate sized sieve and receiver can be used for ease of separating and cleaning spherical grinding media.

4.3.10 Place the ground sample into a clean receptacle or self seal plastic bag and label it appropriately.

4.3.11 Clean the mill (4.2.7) and the brush (4.2.9).

4.4 Validation of grain size

Contamination of the sample by the grinding media shall be quantified in accordance with Table 2. The grinding performance of the mill shall be monitored to ensure the required fineness of the material is achieved. To combat wear caused to the grinding media over time, which can impact on the grain size of the product, pucks, rings and spheres shall be checked, and pots shall be relined as necessary to maintain particle size distribution.

When using spheres, the number and mass per grinding pot shall be kept constant.

5 Loss on ignition (LOI)

COMMENTARY ON CLAUSE 5

LOI is the weight change, expressed as a percentage, caused by heating the sample to a high temperature. It is used to give a measure of the extent of substances that are volatile below the given temperature.

For further details on LOI methodology, see BS EN ISO 21587-1.

5.1 Apparatus

5.1.1 *Platinum boat, or other suitable vessel of sufficient size to contain 2 g of sample at 1 050 °C.*

5.1.2 *Balance, capable of weighing to 0.000 1 g.*

5.1.3 *Desiccator containing dry desiccant, e.g. silica gel.*

5.1.4 *Furnace held at 1 050 °C ± 50 °C.*

5.1.5 *Long handled tongs.*

5.1.6 *Spatula.*

5.1.7 *Drying oven to 110 °C ± 10 °C.*

5.2 Procedure

5.2.1 Dry not less than 10 g of the ground sample in an oven (5.1.7) at 110 °C ± 10 °C or to constant mass (7.8.1.18) in order to evaluate LOI.

5.2.2 Store over a drying agent in a desiccator (5.1.3) whilst cooling.

5.2.3 Weigh boat (5.1.1) and record weight W_1 .

5.2.4 Using a balance (5.1.2) add 2 g ± 0.000 1 g of sample into the boat (5.1.1) and record the weight W_2 .

5.2.5 Using the tongs (5.1.5) place boat (5.1.1) and sample in the furnace (5.1.4) set at 1 050 °C for 60 minutes.

5.2.6 Remove from furnace (5.1.4) with tongs (5.1.5) and place in a desiccator (5.1.3) to cool.

5.2.7 Once cool, re-weigh boat (5.1.1) and sample and record weight W_3 .

Calculate the LOI (%) in accordance with the following equation:

$$\text{LOI} = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

where:

W_1 = weight of boat;

W_2 = weight of boat and sample;

W_3 = weight of ignited boat and sample.

6 X-ray fluorescence (XRF)

6.1 General

The intensities of the fluorescent X-rays from the elements of interest in the sample shall be measured and the chemical composition of the sample shall be analysed by reference to previously determined calibration graphs or equations, applying correction factors for inter-element effects.

NOTE 1 The X-ray intensity might not be directly proportional to the concentration but modified by the concentration of other analytes in the sample (inter-element or matrix effects).

Calibration equations and inter-element corrections shall be established from specimens made from Certified Reference Materials (CRMs) and, where appropriate, synthetic calibrating materials prepared in the same way as the unknowns. Homogeneity, particle size distribution, matrix effects, instrument drift and accuracy of relevant calibration lines shall therefore be taken in to account during specimen measurement.

NOTE 2 Specimens can be prepared for analysis as:

- a) fused cast beads;
- b) pressed powder pellets;
- c) loose powders.

NOTE 3 The use of fused cast beads offers the following advantages and disadvantages:

- a) mineralogical and particulate effects are removed;
- b) calibration can include beads prepared from mixtures of pure oxides or carbonates in addition to CRMs;
- c) dilution with borate flux might significantly affect the sensitivity of analytes present at "trace" level.

NOTE 4 Pressed powder calibration standards should be the same type of matrix as the unknown samples (e.g. same mineralogy). In most cases the mineralogical composition of available reference materials is unknown. Calibration with such materials using pressed pellets is subject to uncertainty.

NOTE 5 The XRF spectrometer software can support a number of models for line overlap and matrix corrections to determine coefficients that describe the shape of the calibration curve. Instrument specific drift correction should be applied if the calibration falls outside acceptable limit values.