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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Refractory products — Sampling of raw materials and unshaped products —

Part 1: Sampling scheme

Produits réfractaires — Échantillonnage des matières premières et des matériaux non façonnés préparés —

Partie 1: Schéma d'échantillonnage

Reference number
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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8656-1 was prepared by Technical Committee ISO/TC 33, *Refractories.*

ISO 8656 consists of the following parts, under the general title *Refractory products — Sampling of raw materials and unshaped products*:

- *Part 1: Sampling scheme*
- *Part 2: Determination of the coefficient of variation*

Annex A forms an integral part of this part of ISO 8656.

Refractory products — Sampling of raw materials and unshaped products —

Part 1: Sampling scheme

1 Scope

This part of ISO 8656 specifies methods and conditions of sampling for raw materials and prepared unshaped refractory products, in order to indicate the mean values of a consignment and the interval of sampling.

It does not apply to products in the form of large static quantities or cargoes from which reliable samples cannot be taken.

The type of sampling equipment, and the preparation and reduction of the samples, which should not alter the properties to be tested, are to be agreed between the interested parties.

NOTE — Difficulties may be encountered when sampling certain types of unshaped products, for example mouldables.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 8656. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 8656 are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3534 : 1977, *Statistics — Vocabulary and symbols.*

ISO 8656-2 : —[1], *Refractory products — Sampling of raw materials and unshaped products — Part 2: Determination of the coefficient of variation.*

3 Definitions

NOTE — See also ISO 3534.

3.1 lot: Defined quantity of a particular material manufactured or produced in conditions which can be considered as being uniform.

3.2 consignment: Quantity of material supplied at one time. A consignment may consist of one or several lots or parts of a lot.

3.3 test lot: Specific quantity subjected to inspection (checking), manufactured normally by a supplier in conditions which can be assumed to be uniform.

3.4 packaged unit: Packaged part of a lot (for example, in a sack or small container).

3.5 sample: Quantity of material taken from a consignment or a lot, intended to supply information, and possibly serve as a basis for a decision concerning the consignment or lot or the process by which it has been produced.

3.6 increment: Quantity of material taken at one time from a larger quantity of material.

3.7 standard deviation of sampling: Standard deviation of the random variations introduced into the measurement of a chosen property by sampling.

3.8 bulk sample: The aggregation of the increments.

3.9 partial sample: Sample obtained by dividing the bulk sample.

3.10 laboratory sample: Sample intended to be used for an inspection or for laboratory tests.

3.11 test sample; final sample, analytical sample: Sample taken from the laboratory sample and prepared in a suitable manner for subjection to particular tests (for example, determination of grain size distribution, humidity, chemical composition, physical or other properties).

3.12 coefficient of variation: Ratio of the standard deviation to the absolute value of the arithmetic mean (this ratio may be expressed as a percentage).

1) To be published.

4 General rules

Because of the diversity of ceramic raw and unshaped materials, the method of delivery and packaging and the conditions and circumstances under which sampling and sample preparation are carried out, rigid rules for sampling cannot be specified in all cases (see annex A), and interested parties should agree on the preferred sampling plan.

4.1 The sampling and sample preparation procedures shall be performed under the supervision of a person having adequate experience of sampling.

4.2 Where required, the consignment may be sub-divided into individual test lots, for example, if it is clear that the consignment consists of various lots or should be treated in separate partial quantities.

4.3 Sampling should preferably be carried out on material in movement during loading or unloading of the consignment or while filling the packaged units.

4.4 In the case of certain prepared unshaped materials (for example, concrete) delivered in sacks which may, at the time of packaging, have been filled with successive layers of unmixed components, or where materials have become separated to some extent during transportation, it is necessary to take the increment(s) after mixing the various components. This sampling method is expensive as it results in large quantities of materials being handled. For this reason the sampling method described in 4.3 shall be preferred.

4.5 The increment shall be taken on the basis of a periodic systematic sampling method with a random starting point and a systematic procedure, on the basis of time, or on the basis of uniform intervals between samplings and/or on the basis of mass.

4.6 The minimum mass of the increment shall be determined, taking account of the maximum grain size of the material in order to avoid systematic errors during sampling (see table 1).

4.7 The number of increments to be taken from a test lot shall be determined taking account of deviations in the properties of the material and the desired precision of sampling (β_1) (see table 2).

4.8 During sampling, sample division, and preparation and storage of the samples, the samples shall be protected against any changes in the properties to be tested.

4.9 The use of a sample to determine several properties is allowed, provided that the result(s) of the test is (are) not changed by the test or the preceding tests.

5 Sampling and sample preparation

5.1 Procedure

5.1.1 Identify the test lot, i.e. of the consignment or part of the consignment to be sampled (nature of product, mass, transport conditions, etc.).

5.1.2 Estimate the maximum grain size of the material.

5.1.3 Determine the mass of the increment in accordance with table 1, taking account of the minimum quantities required for the tests provided for (see note 3 to table 1).

5.1.4 Classify the test lot in a quality variation class (see table 2, 5.3 and 5.4).

5.1.5 Determine the number of increments to be taken (in accordance with table 2).

5.1.6 Calculate the minimum mass of the bulk sample and verify that it is in conformity with the tests provided for (see 5.5).

5.1.7 Determine the sampling point(s) and the methods for sampling and combining increments.

5.1.8 Take the required number of increments in accordance with 5.5.

5.1.9 If necessary, before making up the bulk sample, the increment(s) may be divided in order to have a reserve sample which may be used in case of dispute.

5.1.10 Make up laboratory samples, as agreed between the interested parties, from the increments or bulk sample in accordance with the sampling and sample preparation plan.

NOTE — Care should be exercised not to alter the material when testing properties in the "as-received" condition.

5.1.11 Make up the test samples necessary to carry out the specified programme.

5.2 Mass of the increment

5.2.1 The minimum mass of the increment is determined in accordance with table 1 from the maximum grain size of the material.

Table 1 — Minimum mass of increment depending on the maximum grain size

Maximum grain size mm	Minimum mass of increment
> 100	30 kg
100	15 kg
50	5 kg
20	2 kg
10	500 g
3	200 g
1	50 g

NOTES

1 The masses of the increments relate to a bulk density greater than 1 g/cm^3. For lower bulk densities, the mass of the increment may be determined by multiplying the numerical value in the table by the bulk density of the material.

2 Special agreements should be made in the case of very lumpy products.

In the case of pre-ground or pre-homogenized material, the minimum mass of the increment can be determined, not from the grain size of the coarsest aggregate, but from the maximum size of the grains of the material before aggregating.

3 The actual increment masses should depend on the sampling equipment and the tests to be performed. This is the case for unshaped products if the physico-mechanical properties of specimens taken from these products are to be determined.

5.2.2 When the mass of the increment has been determined, take samples in such a way that all have approximately the same mass.

5.3 Classification of a property according to the range of its variations

5.3.1 The mean value and the standard deviation of a given property, designated respectively by μ and σ, define the coefficient of variation σ/μ of this property.

Expressed in practice as a percentage

$$v = 100\sigma/\mu$$

this is used in this International Standard to assign and define the classes of variation of the property.

5.3.2 The values of the coefficient of variation are divided into three classes:

$0 \leqslant v \leqslant 5\ \%$ small variation, class 1

$5\ \% < v \leqslant 15\ \%$ medium variation, class 2

$15\ \% < v \leqslant 30\ \%$ large variation, class 3

5.3.3 The estimation of the coefficient of variation requires the estimation of the standard deviation of sampling, which is obtained from the appropriate test results, applying the variance method or analysis.

When this is done, a property is immediately assigned to a class of variation. It may be that the most critical coefficient of variation of the property is used for selecting the number n of increments in table 2.

5.3.4 If nothing is known about the coefficient of variation or if it is greater than 30, use the class 3 values (large variations) of table 2 (see examples in 5.6).

5.4 Number of increments and sampling precision

5.4.1 Determine the number n of increments from a property of the material. For example, this may be, depending on the agreements reached, the most important property or the property which is affected by the highest coefficient of variation v.

The coefficient of variation of the determination of a property using the bulk samples and resulting from the sampling is expressed by means of the sampling coefficient of variation between increments v:

$$v_1 = \frac{v}{\sqrt{n}} \qquad \ldots (1)$$

The relative precision of sampling for this property is defined as:

$$\beta_1 = 2v_1 = \frac{2v}{\sqrt{n}} \qquad \ldots (2)$$

5.4.2 The number n of increments shall be such that for the property chosen a specific sampling precision β_1 is obtained:

$$n = \frac{4v^2}{\beta_1^2} \qquad \ldots (3)$$

5.4.3 The number of increments needed to achieve the desired precision of sampling is determined from the class of quality variation of the property and the mass of the test lot as specified in table 2. The value of n given in table 2 is a minimum and generally the actual number should not be less than that specified in the table.

For example:

— if a lower sampling precision is considered sufficient for economic reasons;

or because the relative precisions of the sampling preparation and the test method are too great;

— or if a greater precision is required,

then the number of increments for the required precision can be determined from equation (3) (see also clause 7).

If other properties which are being determined by forming a bulk sample have coefficients of variation different from that of the selected property, their relative sampling precisions β_1 differ from the values given in table 2 and can be calculated from equation (2).

If the test lot is delivered by separate means of transport, for example railway wagons or trucks, at least one increment shall be taken from each of these means of transport.

If the test lot is delivered in smaller packaged units such as bags, sacks, etc., the n increments as specified in table 2 shall be taken from different packaged units. If the number of packaged units in the test lot is less than n, the same number of samples shall be taken from each of the packaged units giving a total at least equal to n. The number of increments per packaged unit shall be reported.

NOTE — The coefficient of variation of the material in any first batch should be determined in accordance with ISO 8656-2.

A number of increments may also be taken per packaged unit to evaluate either the dispersion between the units of a consignment or the dispersion within the packaged unit.

Table 2 — Minimum number of increments and sampling precision

Class of deviations of the property %						Mass of lot m
Class 1		Class 2		Class 3		
$v \leqslant 5$		$5 < v \leqslant 15$		$15 < v \leqslant 30$		
n	β_1	n	β_1	n	β_1	10^3 kg
4	5,00	4	15,00	8	21,21	$m < 1$
4	5,00	6	12,25	12	17,32	$1 \leqslant m < 5$
4	5,00	8	10,61	16	15,00	$5 \leqslant m < 10$
6	4,08	12	8,66	24	12,25	$10 \leqslant m < 50$
8	3,54	16	7,50	32	10,61	$50 \leqslant m < 100$
12	2,89	24	6,12	48	8,66	$100 \leqslant m < 500$
16	2,50	32	5,30	64	7,50	$500 \leqslant m < 1\,000$
20	2,24	40	4,74	80	6,71	$1\,000 \leqslant m$

5.5 Taking of the increments and composition of the bulk sample

5.5.1 The optimum conditions of sampling accuracy are obtained if the increments are taken from the material in movement (see 4.3 and 4.4). However, if the practical delivery conditions do not permit this, sampling may be carried out using the methods described in the annex.

5.5.2 The increments shall be distributed over the complete consignment and shall be taken at regular ponderal or temporal intervals which shall not vary throughout the duration of one sampling operation. In addition, the complete consignment shall be divided according to mass and time into as many intervals as there are increments to be taken. Initial sampling shall be performed at a moment chosen at random within the first interval starting from the taking-over of the material.

NOTE — The term "ponderal" describes fixed intervals of mass.

5.5.3 If the prescribed number of increments has already been taken before the end of the loading and unloading operations, additional samples shall be taken, keeping to the same intervals, until this process is completed.

5.5.4 The bulk sample consists of all the increments.

5.5.5 If the fixed mass for the bulk sample is less than the mass required for the various tests, increase either the mass of the increment or the number of the samples so as to obtain a sufficient amount.

5.6 Examples

5.6.1 Grain density of a magnesite-chrome clinker (< 20 mm)

mass of lot: 20 000 kg

mean value: $\simeq 3{,}51$ g/cm³

standard deviation: $\sigma \simeq 0{,}0176$ g/cm³

$$v = \frac{0{,}0176}{3{,}51} \times 100\,\% = 0{,}5\,\%$$

class: "small variation" (class 1)

minimum mass of sample: 2 kg (see table 1)

number of increments: 6 (see table 2)

5.6.2 Apparent porosity of a single-burned dolomite (< 25 mm)

mass of lot: 2 wagons, 40 000 kg

mean value: $\simeq 11{,}5$ %

standard deviation: $\sigma \simeq 0{,}85$ %

$$v = \frac{0{,}85}{11{,}5} \times 100\,\% = 7{,}4\,\%$$

class: "medium" variation (class 2)

minimum mass of sample: 5 kg (see table 1)

number of increments: 12 (see table 2)

5.6.3 Particle size of a refractory concrete (< 10 mm)

mass of lot: 25 000 kg

a) if only the fraction of the biggest particles is to be determined, in this case 1,7 % > 4 mm, the following is obtained:

mean value: 1,70 %

standard deviation: 0,42 %

$$v = \frac{0{,}42}{1{,}7} \times 100\,\% = 24{,}7\,\%$$

It can be concluded that this is a "large" variation (class 3).

minimum mass of sample: 500 g (see table 1)

number of increments: 24 (see table 2)

The determination of the relatively small fraction of large particles requires quite a large number of increments in order to be sure that the bulk sample adequately represents the lot with regard in particular to the low percentage of particles > 4 mm:

b) but, if the fraction of particles > 2 mm is to be determined, the percentage of which is 32,5, the following is obtained:

$$\left.\begin{array}{l}\text{mean value: } 32{,}5\,\% \\ \text{standard deviation: } 4{,}2\,\%\end{array}\right\} v = \frac{4{,}2}{32{,}5} \times 100\,\% = 12{,}9\,\%$$

It can then be concluded that this is a "medium" variation (class 2).

minimum mass of sample: 500 g (see table 1)

number of increments: 12 (see table 2)

5.6.4 Chemical analysis of sintered magnesia

Table 3 — Variation class depending on the selected oxide

Oxides	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO
$\bar{\mu}$ mean values, %	1,5	0,73	0,03	1,69	95,76
σ standard deviation, %	0,062	0,071	0,019	0,106	0,19
$v = \frac{\sigma}{\bar{\mu}} \times 100$ %	4,13	9,7	63,3	6,3	0,20

Depending on the oxide chosen to determine the class of variation, the range goes from class 1 (MgO, SiO_2) to greater than class 3 (Al_2O_3). By agreement between the interested parties, the characteristic oxide or the oxide with a value closest to the critical value given in the technical specifications shall be selected.

6 Composition and number of laboratory samples and test portions for analysis

The composition and number of laboratory samples from the bulk sample and of test portions for analysis for determining the properties is dependent upon the material and the property itself. This shall be the subject of agreements ensuring that the preparations (division, mixing and, where applicable, crushing) have no effect on the properties to be determined.

The laboratory samples shall be kept in suitable containers.

The following information shall be clearly written on the label and on a card placed inside the container:

a) type of material and designation of consignment or lot subjected to inspection;

b) mass of the consignment;

c) maximum size of grains in the sample;

d) number of increments;

e) date and place of sampling;

f) date and place of preparation of samples;

g) any other details required.

7 Precision of the determination of a property

NOTE — The values given for relative precision (β) in this clause are absolute values, whereas those given in other clauses are expressed as percentages of the mean.

A lot subjected to inspection has undergone n individual sampling operations. All these samples have been collected together to form the bulk sample from which, after intensive homogenization, the test samples are made up for m determinations of the property in question.

Let the value x_i be the result of an individual determination (i = 1, 2 . . ., m). There are three sources of random variation:

a) sampling, with a standard deviation σ_1;

b) preparation of the sample for testing, with a standard deviation σ_2;

c) the method for determining the property in question, with a standard deviation σ_3.

The sampling dispersion, expressed by standard deviation σ_1 and then its coefficient of variation, is the principal basis for the number of increments, specified in table 2.

The σ_1, σ_2 and σ_3 components of variance may be estimated using a test scheme, the principle of which is given in ISO 8656-2.

If m determinations are made of one property, the final result is shown by the mean

$$\bar{x} = \frac{1}{m} \times \sum_{i=1}^{m} x_i$$

As the property sought is thus being estimated from m determinations carried out on the same laboratory sample which has itself been obtained by reducing a bulk sample consisting of n increments, the precision of the mean of m determinations is given as a function of the standard deviations σ_1, σ_2 and σ_3 and the numbers n and m by the equation

$$\sigma_{tot} = \sqrt{\frac{\sigma_1^2}{n} + \sigma_2^2 + \frac{\sigma_3^2}{m}}$$

The overall relative precision β representing the variation in the mean which is only exceeded as a relative value with the probability 0,05 is given by the equation

$$\beta = 2\sigma_{tot} = \sqrt{\beta_1^2 + \beta_2^2 + \beta_3^2}$$

with

$$\beta_1 = \frac{2\sigma_1}{\sqrt{n}}, \qquad \beta_2 = 2\sigma_2, \qquad \beta_3 = \frac{2\sigma_3}{\sqrt{m}}$$

The mean $\bar{x}$ and its precision β permit determination of the confidence interval in which the value sought for the property of the lot under inspection will lie, with a confidence level of 95 %.

$$\bar{x} - 2\sigma_{tot} \leqslant \mu \leqslant \bar{x} + 2\sigma_{tot}$$

that is,

$$\bar{x} - \beta \leqslant \mu \leqslant \bar{x} + \beta$$

8 Example

A 25 tonnes consignment of clay, loaded on a railway wagon is to be sampled in order to determine, for example, the mean content of Al_2O_3. The nominal content is 40 % and the maximum grain size (size of the pieces) is 50 mm. A previous determination of the standard deviation of the content of Al_2O_3 has given a value v = 3 %. According to table 1, the minimum mass of the increment is 5 kg. For a consignment with a mass of 10 tonnes to 50 tonnes and for a coefficient of variation of less than 5, table 2 gives the number of increments n = 6, with a sampling precision of β_1 = 4,08 %, i.e. for each 4 tonnes of clay loaded or unloaded, a 5 kg increment is required (having randomly fixed the start of sampling). The 6 increments obtained according to the sampling plan are gathered together to form a bulk sample from which the laboratory sample will be made up.

NOTE — An example of how this can be done is shown in figure 1.

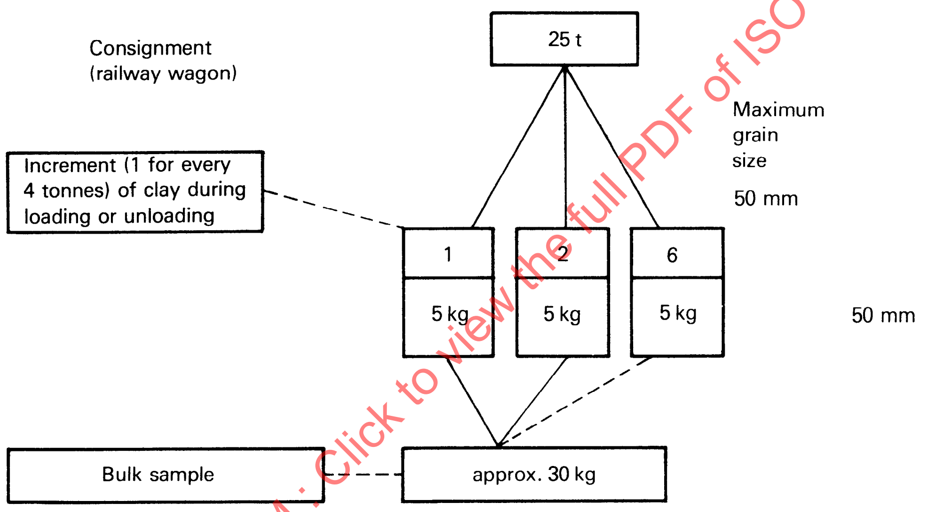

Figure 1 — Example of a sampling scheme (see clause 8)

Annex A
(normative)

Methods for taking increments

A.1 Samples taken from a conveyor belt

If it is possible to stop the belt, two partitions (for example, planks) are fitted at a distance greater than or equal to four times the maximum diameter of the grains or pieces.

Carefully collect all the material between these partitions.

If the plant has an automatic sampling system (for example, sampling from the belt discharge or sweeping of the belt by means of an articulated arm), take care to recover all the mass intercepted.

A.2 Sampling from a wagon, truck or container

A.2.1 Taking an increment

In the case of fine materials, take each sample with a hollow punch; in all other cases, use a scoop (210 mm × 150 mm × 40 mm approximately).

In the case of sample taking using the punch, drive it in to its full height.

For samples taken using a scoop, make a hollow as far as possible down to a depth at least equal to half the depth of the load and take the increment by scraping the walls of the hollow from bottom to top, making sure that the material does not overfill the scoop.

If there is a large grain size dispersion, agreement shall be reached by the interested parties about the sampling requirements, taking into consideration this dispersion, which may mean taking the largest pieces by hand.

A.2.2 Sampling points

Having fixed the number of increments to be taken, arrange the sampling points in a staggered array both for single wagons, trucks or containers or for groups of wagons, trucks or containers (see examples in figure A.1).

If there is considerable dispersion in the grain size of the material, the depths of the sample taking should be alternated, at 1/3 of the depth, or 2/3 of the depth or at the bottom.

A.3 Sampling of packaged materials
(mass limited by packaging to 50 kg)

From the lot, remove for inspection a number of containers (bags or barrels) in accordance with the specifications in table 2, taking them at random from the lot.

Having emptied the total contents of the package(s), divide it up using a riffle divider (or other suitable device) and use one increment, the minimum mass of which is given in table 1.