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Aluminium ores — Sampling procedures

Minerais alumineux — Procédés d'échantillonnage

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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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 8685 was prepared by Technical Committee ISO/TC 129, *Aluminium ores*, Sub-Committee SC 1, *Sampling*.

Annexes A, B, C and D of this International Standard are for information only.

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Aluminium ores — Sampling procedures

1 Scope

This International Standard sets out requirements for the sampling of aluminium ores from moving streams and stationary situations, including stopped-belt sampling, to provide gross samples for sample preparation. Stopped-belt sampling is the reference method for collecting ore samples against which other sampling procedures may be compared. Sampling from moving streams is the preferred method. Sampling from stationary situations should only be considered when sampling from moving streams is not possible. The procedures described in this International Standard for sampling from stationary situations merely minimize some of the sampling errors.

Although this International Standard is intended to cover all aluminium ore sampling from moving streams, the procedures recommended may not be applicable in cases of extreme segregation, for example very wet ore due to its sticky nature, or very dry ore due to generation of dust. In such cases it may be necessary to revert to stopped-belt sampling.

2 Normative references

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

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

1) To be published.

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

ISO 6138:1991, *Aluminium ores — Experimental determination of the heterogeneity of constitution*.

ISO 6139:—¹⁾, *Aluminium ores — Experimental determination of the heterogeneity of distribution of a lot*.

ISO 6140:1991, *Aluminium ores — Preparation of samples*.

ISO 9033:1989, *Aluminium ores — Determination of the moisture content of bulk material*.

ISO 10226:1991, *Aluminium ores — Experimental methods for checking the bias of sampling*.

ISO 10277:—¹⁾, *Aluminium ores — Experimental methods for checking the precision of sampling*.

3 Definitions

For the purposes this International Standard, the definitions given in ISO 3534 (including the terms “precision” and “accuracy”) and the following, apply.

3.1 bias: The tendency to obtain a value which is persistently higher or persistently lower than the true value. Alternatively, the difference between the true value and the average result obtained from a large number of determinations using a biased method.

3.2 constant mass division: The method of sample division in which the retained portion from individual increments is of uniform mass.

3.3 out: A single pass of the sampling device through the ore stream.

3.4 divided increment: The quantity of ore obtained by division of the increment in order to decrease its mass.

3.5 division: The process of decreasing the sample mass (without modification of the particle size of the constituent pieces) where a representative part of the sample is retained while rejecting the remainder.

3.6 fixed rate division: The method of sample division in which the retained portion from individual increments is a constant proportion of the original mass.

3.7 duplicate sampling: A particular case of replicate sampling (with only two replicate samples), for the purpose of estimating the average precision of sampling from a number of lots or sampling units.

3.8 gross sample: A sample formed when all the primary increments or subsamples, either as taken or after having been prepared individually to a particular stage of sample preparation, are combined in the correct proportions for preparation of a laboratory sample.

3.9 increment: The quantity of material extracted from the lot in a single operation of the sampling device.

3.10 lot: A quantity of ore delivered at one time for which the quality characteristics are to be determined.

NOTE 1 The lot may be composed of one or more sampling units.

3.11 isolated lot: A lot that is to be sampled without knowledge of its sampling characteristics.

3.12 manual sampling: The operation of sampling when the increments forming subsamples and gross samples are taken by human effort using a hand-held implement.

3.13 mass-basis sampling: The method of taking increments at uniform mass intervals throughout the lot or sampling unit.

3.14 mechanical sampling: The operation of sampling when the increments forming subsamples and gross samples are taken by a sampling machine.

3.15 nominal top size: The size of aperture of the finest sieve (complying with ISO 565) through which 95 % of the mass of the ore passes.

3.16 random stratified sampling: The taking of increments at irregular intervals within constant intervals of time, mass or space.

3.17 reduction (in particle size): The decrease in dimension of the pieces constituting the sample without modification of the mass or composition.

3.18 replicate sampling: The taking of increments from the lot at equal intervals of time, mass or space.

NOTE 2 The increments are placed in rotation in different containers to give several replicate samples of approximately equal mass.

3.19 sampling unit: The discrete units (e.g. trains, sections of belt, daily production) which comprise the lot.

3.20 strata: Approximately equal parts of a lot or sampling unit, based on intervals of time, mass or space.

3.21 subsample: A quantity of ore consisting of a number of increments taken from a part of the lot; also a composite of a number of increments each having been individually prepared as necessary.

3.22 systematic stratified sampling: The taking of increments at regular intervals within constant intervals of time, mass or space.

3.23 time-basis sampling: The method of taking of increments at uniform time intervals throughout the lot or sampling unit.

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4 Establishing a sampling scheme

4.1 General

The basic requirement of a correct sampling scheme is that all particles in the stream have an equal opportunity of being selected and appearing in the final gross sample for analysis. Any deviation from this basic requirement can result in an unacceptable loss of accuracy and precision. No incorrect sampling scheme can be relied upon to provide representative samples.

Sampling should be carried out by systematic sampling, either on a mass basis (see clause 8) or on a time basis (see clause 9), but only when it can be shown that no systematic error could be introduced due to any periodic variation in quality or quantity which may coincide with, or approximate to, any multiples of the proposed sampling intervals.

As an example, a primary cutter may be cutting a stream of ore which is being reclaimed from a stockpile by a bucket wheel reclaimer. At both limits of the bucket wheel traverse across the ore face on the stockpile, the ore may have different properties from that of the middle of the stockpile (due to segregation). It is quite possible that every time the primary cutter makes a cut, the cut coincides with ore being delivered from the limit of a traverse of the

bucket wheel reclaimer and a systematic error could thus arise.

This same provision applies to secondary and subsequent stages of division where it is felt that a systematic error could arise, due to the manner in which the ore is handled and presented to division apparatus.

In such cases, it is strongly recommended that stratified random sampling within fixed mass or time intervals be carried out (see clause 10).

The methods for subsampling and sample preparation depend on the final choice of sampling scheme and on the steps necessary to minimize possible systematic errors arising during subsequent division steps.

4.2 Safety of operators

Due consideration shall be given to the safety of operators when employing any method of collecting samples from stationary situations. The applicable safety codes shall be respected.

4.3 General procedure for sampling

The general procedure for sampling is as follows:

- a) decide for what purpose the samples are being taken, e.g. monitoring plant performance, use in commercial transactions;
- b) identify the quality characteristics to be measured and specify the overall precision and sampling precision;
- c) identify the lot or part of the lot to be sampled;
- d) ascertain the nominal top size and particle density of the ore for the purpose of determining the minimum gross sample mass, primary increment mass and cutter opening in the case where a mechanical sampler is used, or the size of ladle in the case where manual sampling is employed;
- e) determine the increment variance, V_i , or the parameters of the variogram if the variogram method is used for the quality characteristic(s) under consideration (see ISO 6139);
- f) determine the coefficient of variation between particles, C_v , of the quality characteristic(s) under consideration (see ISO 6138);
- g) determine the minimum number of primary increments, n , and sampling units, k , required to achieve the required sampling precision (see clause 5);

- h) determine the minimum gross sample mass to achieve the required sampling variance (see clause 6);
- i) determine the minimum primary increment mass (see clause 7);
- j) determine the sampling intervals in tonnes for mass-basis systematic sampling (see clause 8) and stratified random sampling within fixed mass intervals (see clause 10), or in minutes for time-basis systematic sampling (see clause 9) and stratified random sampling within fixed time intervals (see clause 10);
- k) take primary increments at the intervals determined in step j) during the whole period of handling the lot;
- l) combine the increments (see 8.5 or 9.5) into subsamples or a gross sample (an example is given in figure 1);
- m) subsamples are usually prepared and analysed separately to improve overall precision; they may also be prepared
 - 1) for convenience of materials handling,
 - 2) to provide progressive information on the quality of the lot,
 - 3) to provide, after division, reference or reserve samples,
 - 4) to reduce any bias in the test result for the moisture content of a large lot caused by moisture loss (or gain), due to climatic conditions.

It is permissible to divide increments at step l) before constituting a gross sample or subsample, provided that the mass of the divided increment exceeds the minimum mass determined in step i). If the whole of the primary increment or divided primary increment is crushed, to enable further division it is necessary to recalculate the minimum mass of the gross sample and the divided primary increment using the nominal top size of the crushed ore.

NOTE 3 When sampling an isolated lot in which the increment variance (or the variogram) and coefficient of variation between particles, C_v , of the quality characteristics under consideration are not known, it is not possible to design a sampling scheme which guarantees that the specified sampling precision will be obtained. In this situation, the number of increments to be taken and their masses should be agreed between the parties concerned. When sampling of the isolated lot has been completed, it is possible, however, to determine the overall precision obtained using the appropriate method specified in ISO 10277.

4.4 Overall variance

The overall variance, denoted by σ_{SPM}^2 , for measuring the mean value of each quality characteristic is comprised of three components, namely the variance of sampling, the variance of sample preparation and the variance of analysis. The relationship is as follows:

$$\sigma_{SPM}^2 = \sigma_S^2 + \sigma_P^2 + \sigma_M^2$$

where

- σ_S^2 is the variance of sampling;
- σ_P^2 is the variance of sample preparation;
- σ_M^2 is the variance of analysis (measurement).

The method for determining σ_S , σ_P and σ_M may be found in ISO 10226.

The sampling variance consists of two components, namely the short range quality fluctuation variance σ_{QE1}^2 and the long range quality fluctuation variance σ_{QE2}^2 .

The relationship is as follows:

$$\sigma_S^2 = \sigma_{QE1}^2 + \sigma_{QE2}^2$$

The short range quality fluctuation variance also consists of two components as follows:

$$\sigma_{QE1}^2 = \sigma_{FE}^2 + \sigma_{GE}^2$$

where

- σ_{FE}^2 is the fundamental error variance;
- σ_{GE}^2 is the segregation error variance.

Thus

$$\sigma_S^2 = \sigma_{FE}^2 + \sigma_{GE}^2 + \sigma_{QE2}^2$$

The fundamental error variance depends on the gross sample mass while the other two components depend on the distribution heterogeneity of the ore and the number of increments. In this International Standard, the minimum gross sample mass (see 6.1) is chosen so that

$$\sigma_{FE}^2 \leq \frac{\sigma_S^2}{2}$$

In the equation for σ_{SPM}^2 above, the major part of the overall variance is often due to sampling errors. However, when a very precise result is required and the sampling errors have been minimized, consideration shall be given to increasing the number of sample preparations and/or analyses performed in order to reduce these components of the overall variance. This is achieved by carrying out multiple determinations on the gross sample or preferably by dividing the lot into a number of sampling units and preparing and analysing a subsample from each sampling unit (see figure 1).

The overall variance is then given as follows.

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- a) when a single gross sample is constituted for a lot and r replicate determinations are carried out on the gross sample,

$$\sigma_{SPM}^2 = \sigma_S^2 + \sigma_P^2 + \frac{\sigma_M^2}{r}$$

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- b) when k subsamples are prepared and analysed, each constituted from an equal number of increments, and r replicate determinations are carried out on each subsample,

$$\sigma_{SPM}^2 = \sigma_S^2 + \frac{\sigma_P^2 + \frac{\sigma_M^2}{r}}{k}$$

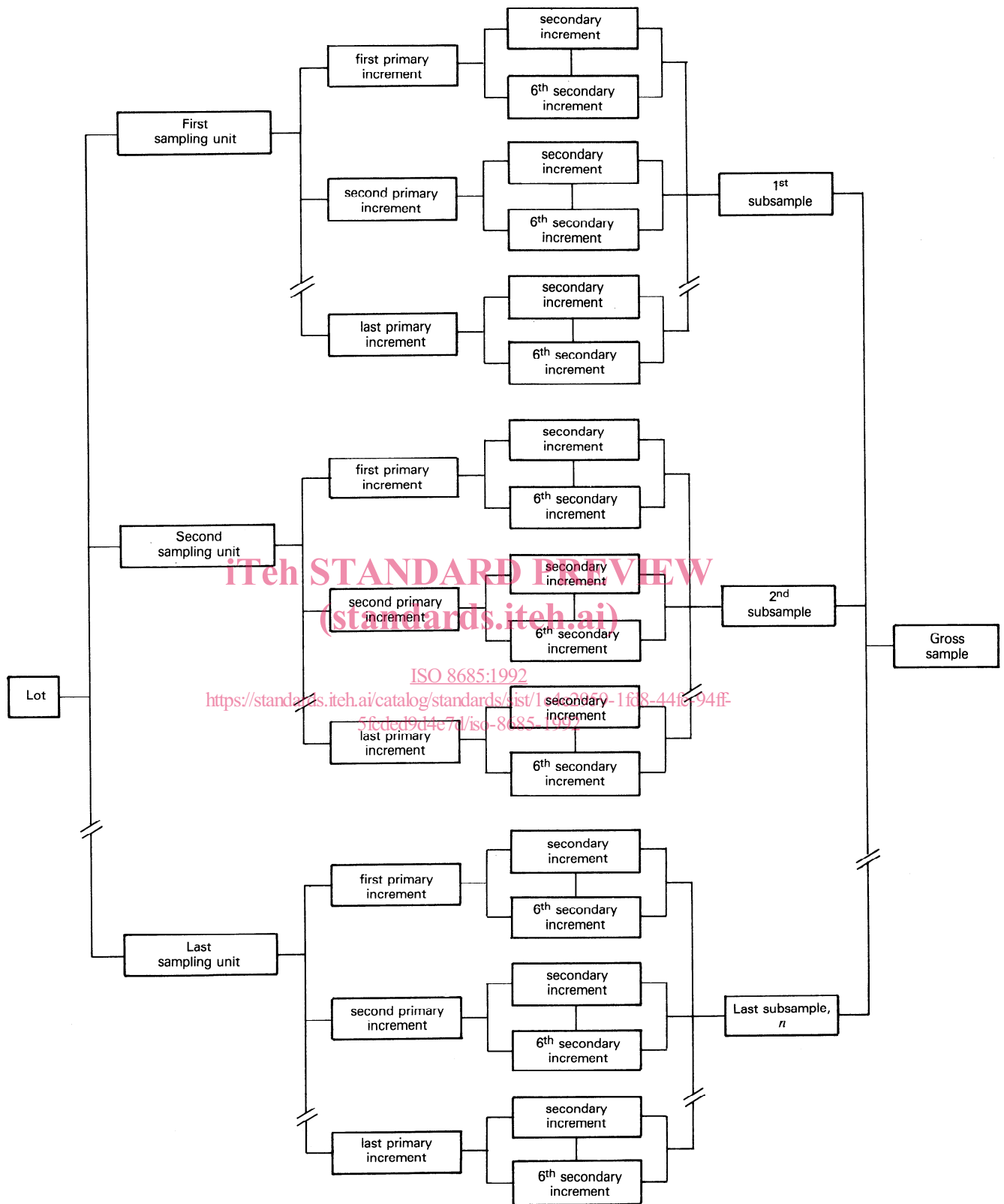


Figure 1 — Example of a sampling plan including six secondary increments
 (Mixing, reduction and division steps have been omitted for simplicity.)

5 Number of primary increments and sampling units

5.1 General

The number of primary increments to be taken from a lot or sampling unit in order to attain the required sampling variance is a function of the variability of the characteristics to be determined. This variability depends on the amount of segregation present in the ore, the particle size range of the ore and the mass of the lot or sampling unit. It is determined experimentally for each type of ore and expressed in terms of either the increment variance V_1 or the intercept A and slope B of the variogram in accordance with ISO 6139.

CAUTION — The determination of moisture requires special consideration due to the fact that it is extremely difficult, if not impossible, to retain the integrity of the sample over extended periods of sample collection. In such cases a bias may occur which can only be overcome by collecting moisture samples at more frequent intervals than may be dictated by a simple calculation of increment numbers of sampling units based on a certain precision. It is therefore recommended that moisture tests be carried out on a number of subsamples and the weighed mean of the test results recorded. This will reduce any bias in the test result caused by moisture loss (or gain) due to climatic conditions. It will also result in better precision.

5.2 Calculation of the number of primary increments

When the variability of the ore has been determined, the number of primary increments to be taken from the lot can be calculated from the following formula at the desired sampling precision.

5.2.1 Increment variance method

$$n = \frac{V_1}{\sigma_s^2}$$

where

- n is the number of primary increments;
- V_1 is the increment variance;
- σ_s^2 is the desired sampling variance.

EXAMPLE

Minimum number of primary increments for different values of V_1 and σ_s

Table 1 — Minimum number of primary increments required

V_1	Sampling standard deviation, σ_s						
	0,1	0,2	0,5	0,75	1	2	3
0,25	25	7	1	1	1	1	1
1	100	25	4	2	1	1	1
4	400 ¹⁾	100	16	8	4	1	1
9	900 ¹⁾	225	36	16	9	2	1
25	2 500 ¹⁾	625 ¹⁾	100	50	25	6	3
100	10 000 ¹⁾	2 500 ¹⁾	400 ¹⁾	200	100	25	11

1) Values indicate that the specified precision may not be practically achievable. In this case, it will be necessary to adopt a poorer sampling precision than that specified in 4.3 b).

5.2.2 Variogram method

a) Systematic sampling

$$n = \frac{A + \sqrt{A^2 + \frac{2}{3} B m_L \sigma_s^2}}{2 \sigma_s^2}$$

where

A is the intercept of the corrected variogram;

n is the number of primary increments;

B is the gradient (slope) of the variogram;

m_L is the mass of the lot;

σ_s^2 is the desired sampling variance.

σ_s^2 is the desired sampling variance.

b) Stratified random sampling

$$n = \frac{A + \sqrt{A^2 + \frac{4}{3} B m_L \sigma_s^2}}{2 \sigma_s^2}$$

EXAMPLE

Assume that systematic sampling is being used and that the parameters of the variogram for Al_2O_3 content, determined in accordance with ISO 6139, are as follows:

$$A = 0,3$$

$$B = 0,000 1$$

$$m_L = 30 000 \text{ t}$$

$$\sigma_s = 0,1 \% (m/m) Al_2O_3$$

Thus

$$n = \frac{0,3 + \sqrt{0,09 + \frac{2}{3} \times 0,000\ 1 \times 30\ 000 \times (0,1)^2}}{2 \times 0,01}$$

$$= \frac{0,3 \sqrt{0,09 + 0,02}}{0,02}$$

$$= \frac{0,63}{0,02}$$

$$= 32$$

5.3 Calculation of the number of subsamples

When the variances of sample preparation and measurement are known, the number of subsamples, k , can be calculated from the following formula:

$$k = \frac{\sigma_p^2 + \frac{\sigma_M^2}{r}}{\sigma_{SPM}^2 - \sigma_S^2}$$

where σ_{SPM} , σ_S , σ_p , σ_M and r are as previously defined.

Several iterations may be required to find the correct combination of σ_S and k .

6 Mass of gross samples and subsamples

6.1 Minimum mass of gross sample

It is essential to ensure that the mass of the gross sample is adequate to achieve the required sampling variance. If the gross sample mass is too small, the desired sampling variance will not be achieved, even though sufficient increments, as calculated in 5.2 may have been taken.

The minimum gross sample mass is given by the following empirical formula (see annex A):

$$m_G = \left(\frac{C_v}{\sigma_S} \right)^2 \rho g D^3 \times 10^{-6}$$

where

m_G is the minimum gross sample mass, in kilograms;

C_v is the coefficient of variation between particles of the quality characteristic under investigation, (according to ISO 6138);

σ_S is the required relative sampling error (standard deviation);

ρ is the density, in tonnes per cubic metre, of the ore particles (not bulk density);

g is the size range factor given in table 2;

D is the nominal top size, in millimetres, of the ore in the lot.

Table 2 — Size range factors

Size range	Value for g
Large size range ($D/D' > 4$)	0,25
Medium size range ($4 \geq D/D' \geq 2$)	0,50
Small size range ($D/D' < 2$)	0,75
Uniform size ($D/D' = 1$)	1,00

D is the nominal top size of the ore;
 D' is the sieve size retaining 95 % of the ore.

The variation of minimum gross sample mass with nominal top size for various values of C_v/σ_S is shown in figures 2 and 3 (assuming $\rho = 2,5 \text{ t/m}^3$).

EXAMPLE

Take the case of sampling an aluminium ore of nominal top size 22,4 mm and of particle density $2,5 \text{ t/m}^3$. Assume the size range is large, the coefficient of variation is 20 % and a sampling error of 0,5 % is required. The minimum mass of the gross sample is given by the following formula:

$$m_G = \left(\frac{20}{0,5} \right)^2 \times 2,5 \times 0,25 \times (22,4)^3 \times 10^{-6}$$

$$= 11,2 \text{ kg}$$

6.2 Minimum mass of subsamples

The minimum mass of individual subsamples shall be as follows:

- when the individual subsamples are prepared and analysed, the minimum mass of the subsample shall be not less than that of the minimum gross sample calculated in 6.1;
- when individual subsamples are combined to form a gross sample, the minimum mass of the subsample, m_s , shall be m_G/k where k is the number of subsamples as defined in 4.3 b).

6.3 Minimum mass of crushed gross samples and subsamples

If gross samples or subsamples are crushed to permit further division, the minimum masses may be calculated using the formula in 6.1 and 6.2, by inserting the nominal top size of the crushed ore.

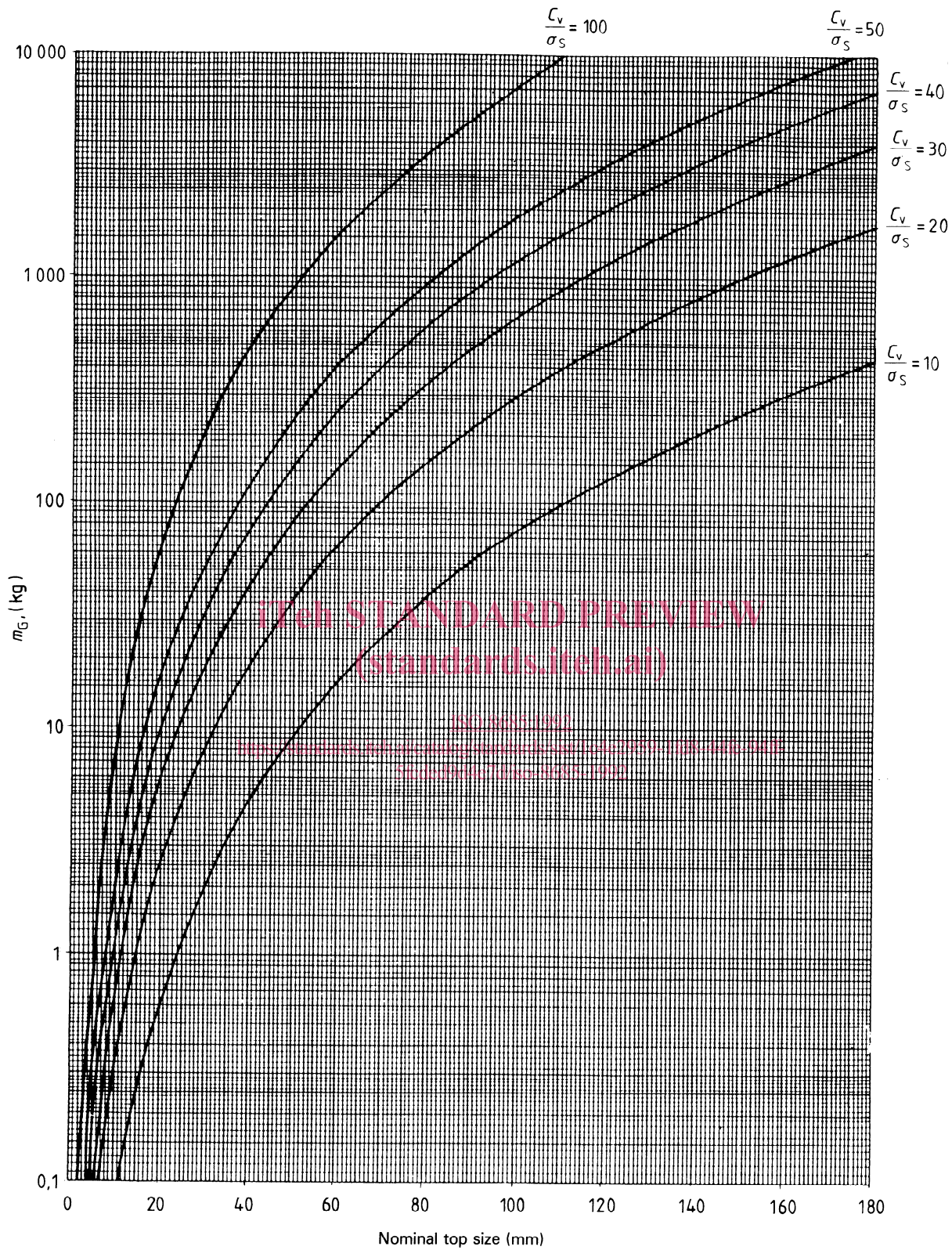


Figure 2 — Minimum gross sample mass as a function of C_v/σ_s and of nominal top size
 (for a nominal density of 2,5 t/m³ and $g = 0,25$)