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Second edition
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Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content

Concentrés de cuivre, de plomb, de zinc et de nickel — Procédures d'échantillonnage pour la détermination de la teneur en métal et de l'humidité

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12743 was prepared by Technical Committee ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

This second edition cancels and replaces the first edition (ISO 12743:1998), which has been technically revised. The principal change is the extension of the scope to cover the sampling of nickel concentrates.

Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content

WARNING — This International Standard may involve hazardous materials, operations and equipment. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard sets out the basic methods for sampling copper, lead, zinc and nickel concentrates from moving streams and stationary lots, including stopped-belt sampling, to provide samples for chemical analysis, physical testing and determination of moisture content, in accordance with the relevant International Standards. Where the concentrates are susceptible to significant oxidation or decomposition, it is necessary to use a common sample for moisture determination and chemical analysis to eliminate bias (see ISO 10251). In such cases, the common sample must be sufficiently representative, i.e. unbiased and sufficiently precise, for chemical analysis and determination of moisture content. Any large agglomerates (> 10 mm) present in the primary sample should be crushed prior to further sample processing. Sampling of concentrates in slurry form is specifically excluded from this International Standard.

Stopped-belt sampling is the reference method for collecting concentrate samples against which mechanical and manual-sampling procedures may be compared. Sampling from moving streams is the preferred method. Both falling-stream and cross-belt samplers are described.

Sampling from stationary lots is used only where sampling from moving streams is not possible. The procedures described in this International Standard, for sampling from stationary lots, only minimize some of the systematic sampling errors.

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.

ISO 10251, *Copper, lead, zinc and nickel concentrates — Determination of mass loss of bulk material on drying*

ISO 12744, *Copper, lead, zinc and nickel concentrates — Experimental methods for checking the precision of sampling*

ISO 13292, *Copper, lead, zinc and nickel concentrates — Experimental methods for checking the bias of sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
representative sample
quantity of concentrate representing a larger mass of concentrate with both precision and bias within acceptable limits

3.2
lot
quantity of concentrate to be sampled

3.3
lot sample
quantity of concentrate representative of the lot

3.4
sub-lot
subdivided parts of a lot which are processed separately, each of them producing a subsample which is analysed separately, e.g. for moisture determination

3.5
subsample
quantity of concentrate representative of the sub-lot

3.6
sampling
sequence of operations aimed at obtaining a sample representative of a lot

NOTE It comprises a series of sampling stages, each stage usually comprising operations of selection and preparation

3.7
selection
operation by which a smaller quantity of concentrate is taken from a larger quantity of concentrate

3.8
increment
quantity of concentrate selected by a sampling device in one operation

3.9
increment selection
selection process that consists of extracting from the lot, or from an intermediate sample, successive increments which can be combined to constitute a sample

3.10
division
operation of decreasing sample mass, without change of particle size, where a representative part of the sample is retained

3.11
constant-mass division
method of division in which the retained portions from individual increments or subsamples are of uniform mass

3.12
proportional division
method of division in which the retained portions from individual increments or subsamples are a constant proportion of their original mass

3.13
preparation
nonselective operation without division such as sample transfer, drying, comminution or homogenization

3.14
sample processing
whole sequence of selection and preparation operations which transforms a stage i sample into a test sample

3.15
comminution
operation of reducing particle size by crushing, grinding or pulverisation

3.16
stage i sample
sample obtained at the ith stage of the sampling scheme

3.17
moisture sample
representative quantity of concentrate from which test portions are taken for moisture determination

NOTE Alternatively, the whole moisture sample may be dried to determine its moisture content

3.18
laboratory sample
sample that is processed so that it can be sent to the laboratory and used for further processing and selection of one or more test samples for analysis

3.19
common sample
representative quantity of concentrate which is dried to determine its mass loss and subsequently used for further processing and selection of one or more test samples for chemical analysis

3.20
test sample
representative quantity of concentrate obtained from a laboratory sample when additional preparation, such as drying or hygroscopic moisture determination, is needed prior to the selection of one or more test portions

3.21
test portion
representative quantity of concentrate taken from a moisture sample, a laboratory sample or a test sample which is submitted to moisture determination or analysis in its entirety

3.22
systematic sampling
selection of increments in which the concentrate being sampled is divided into equal strata and the first increment is taken at random within the first stratum, the interval between subsequent increments being equal to the stratum size

3.23
stratified random sampling
selection of increments in which the concentrate being sampled is divided into equal strata, each increment being taken at random within each stratum

3.24
homogenisation
preparation operation which reduces the distribution heterogeneity of the concentrate

3.25
agglomerate
cluster of particles that are held together by chemical or physical phenomena

3.26
nominal top size
aperture size of a test sieve that retains 5 % of the mass of concentrate

3.27
moisture determination
quantitative measurement of the mass loss of the moisture test portion under the conditions of drying specified in ISO 10251

3.28
chemical analysis
quantitative determination of the required chemical constituents of the analysis test portion

3.29
error
in any quantitative measurement, the difference between the true value and the value obtained for an individual measurement

3.30
bias
statistically significant difference between the mean of the test results and an accepted reference value

See also ISO 13292.

3.31
precision
closeness of agreement between independent test results obtained under stipulated conditions

See also ISO 12744.

3.32
interleaved samples
samples constituted by placing consecutive primary increments alternately into two separate sample containers

4 Sampling theory

4.1 General

The basic rule for a correct sampling method is that all possible increments from the concentrate stream or stratum shall have the same probability of being selected and appearing in the sample. Any deviation from this basic requirement can result in a bias. An incorrect sampling scheme cannot be relied on to provide representative samples.

Sampling should preferably be carried out on a systematic basis, either on a mass basis (see 7.2) or on a time basis (see 7.3), but only where it can be shown that no systematic error (or bias) could be introduced due to any periodic variation in quality or quantity that may coincide with, or approximate to, any multiples of the proposed sampling interval. In such cases, it is recommended that stratified random sampling within fixed time or mass intervals be carried out (see 7.4).

The methods for sampling, including sample processing, depend on the final choice of the sampling scheme and on the steps necessary to minimize possible systematic errors. The aim is always to reduce the total variance to an acceptable level, while at the same time eliminating any significant biases, e.g. minimizing degradation of samples used for determination of size distribution.

Moisture samples shall be processed as soon as possible and test portions shall be weighed immediately. If this is not possible, samples shall be stored in impervious airtight containers with a minimum of free air space to minimize any change in moisture content, but should be prepared without delay.

4.2 Total variance

The general aim of a sampling scheme is to provide one or several test portions, sufficiently representative of a lot, for determination of the quality characteristics of the lot. The total variance of the final result, denoted by s_T^2, consists of the variance of sampling (including sample processing) plus the variance of analysis (chemical analysis, moisture determination, determination of particle size distribution, etc.) as follows:

$$s_T^2 = s_S^2 + s_A^2 \qquad \ldots(1)$$

where

s_S^2 is the sampling variance (including sample processing);

s_A^2 is the analytical variance.

In Equation 1, the sampling variance includes the variances due to all sampling (and sample processing) steps, except selection of the test portion. The variance due to selection of the test portion is included in the analytical variance, s_A^2, which is determined in accordance with ISO 12744, because it is difficult to determine separately the "true" analytical variance.

Often replicate analyses of quality characteristics are carried out, which reduces the total variance. In this case, if r replicate analyses are made:

$$s_T^2 = s_S^2 + \frac{s_A^2}{r} \qquad \ldots(2)$$

The estimation or measurement of the total variance can be carried out in several ways, depending on the purpose of the exercise. In many respects, the different approaches are complementary.

The first method, which was developed by Gy[3, 4], is to break up the sampling variance into its components for each sampling stage (see Annex A). The total variance is then given by:

$$s_T^2 = s_{S_1}^2 + \ldots. + s_{S_i}^2 + \ldots. + s_{S_{u-1}}^2 + \frac{s_A^2}{r} \qquad \ldots(3)$$

where

$s_{S_1}^2$ is the sampling variance for stage 1, i.e. the primary sampling variance;

$s_{S_i}^2$ is the sampling variance for stage i;

$s_{S_{u-1}}^2$ is the sampling variance for stage $u - 1$, the second last stage;

u is the number of sampling stages, stage u corresponding to selection of the test portion.

This is referred to as the “sampling stage” method (see 4.3) and provides very detailed information on the variance components, which is particularly useful for designing and assessing sampling schemes. However, to obtain maximum benefit, it is necessary to collect data at each sampling stage.

The second method, called the “simplified” method (see 4.4), is to break up the total variance into primary sampling, sample processing and analytical variances only as follows:

$$s_{\mathrm{T}}^2 = s_{\mathrm{S}_1}^2 + s_{\mathrm{P}}^2 + \frac{s_{\mathrm{A}}^2}{r} \qquad \ldots(4)$$

where

$s_{\mathrm{S}_1}^2$ is the primary sampling variance;

s_{P}^2 is the variance due to all subsequent sampling steps, i.e. sample processing, except selection of the test portion;

s_{A}^2 is the analytical variance, including selection of the test portion (at stage u in Equation 3).

The primary sampling variance is identical to the sampling variance for stage 1 in Equation 3, while s_{P}^2 is equal to the total sampling variance for the remaining sampling stages, except for selection of the test portion which is included in the analytical variance. The relative magnitudes of the variance components in Equation 4 indicate where additional effort is required to reduce the total variance. However, it is not possible to separate the variances of the separate sample-processing stages. This method is suitable for estimating the total variance for new sampling schemes based on the same sample-processing procedures, where the numbers of primary increments, sample processings and analyses are varied.

Finally, the total variance s_{T}^2 can be estimated experimentally by collecting interpenetrating duplicate samples (see 4.5). This is called the “interleaved sample” method and gives valuable information on the total variance actually achieved for a given sampling scheme with no extra effort, provided facilities are available for collecting duplicate samples (Merks[5]). It gives no information on variance components, but the total variance can be compared with the analytical variance to ascertain whether the sampling scheme used was optimized or not. It is therefore of limited use for designing sampling schemes, but it can be used to monitor whether a sampling scheme is in control.

4.3 Sampling-stage method of estimating sampling and total variance

The sampling variance for stage i is given by (see Annex A):

$$s_{\mathrm{S}_i}^2 = \frac{s_{\mathrm{b}_i}^2}{n_i} \qquad \ldots(5)$$

where

$s_{\mathrm{b}_i}^2$ is the variance between increments for stage i;

n_i is the number of increments for stage i.

The variance between increments for stage i, $s_{\mathrm{b}_i}^2$, can be estimated using the following equation:

$$s_{b_i}^2 = \frac{\sum_{j=1}^{n}(x_j - \bar{x})^2}{n_i - 1} - s_{PA}^2 \quad \ldots(6)$$

where

x_j is the test result for increment j;

$\bar{x}$ is the mean test result for all increments;

s_{PA}^2 is the variance of subsequent sample processing and analysis.

The variance of subsequent sample processing and analysis of each increment, s_{PA}^2, has been taken into account in Equation 6 to obtain an unbiased estimate of $s_{b_i}^2$.

NOTE Care is needed in subtracting variances. The difference is significant only when the F ratio of the variances being subtracted is statistically significant.

Remembering that the variance due to selection of the test portion is included in the analytical variance s_A^2, the total sampling variance is given by:

$$s_S^2 = \sum_{i=1}^{u-1} \frac{s_{b_i}^2}{n_i} \quad \ldots(7)$$

Combining Equations 2 and 7 gives the total variance s_T^2 as follows:

$$s_T^2 = \sum_{i=1}^{u-1} \frac{s_{b_i}^2}{n_i} + \frac{s_A^2}{r} \quad \ldots(8)$$

For a three-stage sampling scheme (including selection of the test portion), Equation 8 reduces to:

$$s_T^2 = \frac{s_{b_i}^2}{n_1} + \frac{s_{b_2}^2}{n_2} + \frac{s_A^2}{r} \quad \ldots(9)$$

The best way of reducing the value of s_T^2 to an acceptable level is to reduce the largest terms in Equation 8 first. Clearly $s_{b_i}^2 / n_i$ for a given sampling stage can be reduced by increasing the number of increments n_i or reducing $s_{b_i}^2$ by homogenizing the concentrate prior to sampling. The last term can be reduced by reducing the particle size prior to selection of the test portion, or performing replicate analyses. Selecting the optimum number of increments n_i for each sampling stage may require several iterations to obtain the required total variance s_T^2.

EXAMPLE Consider a four-stage sampling scheme for determining the metal content of a copper concentrate containing 31,2 % Cu. Assume that the concentrate is being conveyed at 500 t/h on a conveyor belt, that the lot size is 500 t, and that the following parameters have been determined using Equation 6 where appropriate:

s_{b_1} = 0,3 % Cu

s_{b_2} = 0,2 % Cu

s_{b_3} = 0,1 % Cu

s_A = 0,05 % Cu

NOTE Many measurements may be required to obtain good estimates of s_{b_1}, s_{b_2}, s_{b_3} and s_A.

Stage 1

Assume that the primary cutter takes increments of 12 kg mass at 2 min intervals. Thus:

$n_1 = 30$

Primary sample mass = 360 kg

Equation 5 gives:

$s_{S_1}^2 = (0,3)^2/30 = 0,003\ 0$

Stage 2

The primary increments are collected in a hopper, and then fed to the secondary cutter at a rate of 360 kg/h. Secondary increments of 0,01 kg are taken at 30 s intervals. Thus:

$n_2 = 120$

Divided sample mass = 1,2 kg

$s_{S_2}^2 = (0,2)^2/120 = 0,000\ 333$

Stage 3

The 1,2 kg sample is transported to the sample-processing laboratory and fed through a rotary sample divider with a sample-collection canister divided into 8 equal sectors rotating at 30 rev/min (0,5 s^{-1}). Sample division takes 2 min. Thus:

$n_3 = 60$

Divided sample mass = 150 g

$s_{S_3}^2 = (0,1)^2/60 = 0,000\ 167$

Stage 4

Dry the sample and then pulverize it to 150 µm. Select a 1 g test portion, by taking 10 increments of 0,1 g with a spatula, and conduct a single analysis. Thus:

$s_A = 0,05$ % Cu

Total variance

The total variance is given by:

$$s_T^2 = s_{S_1}^2 + s_{S_2}^2 + s_{S_3}^2 + s_A^2$$

$$= 0,003\ 0 + 0,000\ 333 + 0,000\ 167 + 0,002\ 5$$

$$= 0,006$$

Hence:

s_T = 0,077 % Cu

In this example, the largest components of variance are due to primary sampling and analysis. Consequently, the total variance can be reduced by increasing the number of primary increments and conducting replicate analyses.

An example of the application of the sampling-stage method of estimating total variance to sampling from grabs is given in Annex B.

4.4 Simplified method of estimating sampling and total variance

While it is not possible to partition, i.e. separate, the variances of the individual sample-processing stages, the simplified method is suitable for estimating the total variance for new sampling schemes based on the same sample-processing procedures, where the numbers of primary increments, sample processings and analyses are varied.

Using Equation 5, the primary sampling variance $s^2_{S_1}$ is given by:

$$s^2_{S_1} = \frac{s^2_{b_1}}{n_1} \qquad \ldots(10)$$

where

n_1 is the number of primary increments;

$s^2_{b_1}$ is the variance between primary increments determined using Equation 6.

The primary sampling variance can be reduced by increasing the number of primary increments n_1.

The sample-processing variance s^2_P and analytical variance s^2_A are determined experimentally by duplicate sample processing and determination of quality characteristics in accordance with ISO 12744. The analytical variance s^2_A can also be obtained by carrying out duplicate analyses on test samples.

Multiple sample processings and analyses are often carried out to reduce the total variance. In this case, combining Equations 4 and 10 gives the following.

a) Where a single sample is constituted for the lot and r replicate analyses are carried out on the test sample:

$$s^2_T = \frac{s^2_{b_1}}{n_1} + s^2_P + \frac{s^2_A}{r} \qquad \ldots(11)$$

b) Where the lot is divided into k sub-lots, a subsample is constituted for each sub-lot, and r replicate analyses are carried out on each resultant test sample:

$$s^2_T = \frac{s^2_{b_1}}{n_1} + \frac{s^2_P}{k} + \frac{s^2_A}{rk} \qquad \ldots(12)$$

c) Where sample processing and analysis is carried out on each increment taken from the lot and r replicate analyses are carried out:

$$s_T^2 = \frac{s_{b_1}^2 + s_P^2 + \frac{s_A^2}{r}}{n_1} \qquad \ldots(13)$$

EXAMPLE Assume that 50 primary increments are taken from a zinc concentrate lot that has been divided into two sub-lots. The resultant two subsamples are processed separately and analysed in duplicate. Assume that the primary increment, sample processing and analytical standard deviations have been determined experimentally as follows:

s_{b_1} = 0,3 % Zn

s_{b_2} = 0,1 % Zn

s_{b_3} = 0,05 % Zn

Using Equation 12, the total variance is given by:

$s_T^2 = (0,3)^2/50 + (0,1)^2/2 + (0,05)^2/(2 \times 2)$

$= 0,001\ 8 + 0,005\ 0 + 0,000\ 625$

$= 0,007\ 43$

Hence:

s_T = 0,086 % Zn

In this example, the major component of variance is sample processing. This component could be reduced by dividing the lot into a larger number of sub-lots, and constituting a subsample for each sub-lot.

4.5 Interleaved sample method of measuring total variance

The total variance s_T^2 achieved for a given sampling operation can be estimated experimentally by collecting interleaved duplicate samples as shown in Figure 1. If the number of primary increments for routine sampling is n_1, then $2n_1$ primary increments are taken from each lot and the odd- and even-numbered increments are separately combined to give samples A and B for the lot. Samples A and B are then separately submitted to sample processing and analysis. This procedure is repeated until sampling has been completed. The total variance for a single lot is then given by:

$$s_T^2 = \frac{\pi}{4}\left[\frac{\sum_{i=1}^{N}\left|x_{A_i} - x_{B_i}\right|}{N}\right]^2 \qquad \ldots(14)$$

where

x_{A_i} and x_{B_i} are the analyses for each pair of samples A_i and B_i;

N is the number of lots (in the range 10 to 20);

$\pi/4$ is a statistical factor relating range to variance for a pair of measurements.

Alternatively, if the precision is being checked as part of routine sampling, n primary increments may be taken from each lot and two interleaved samples constituted, each comprising $n/2$ primary increments. However, the overall variance thus obtained is overestimated and the sampling variance component must be divided by 2 to obtain the overall variance for lot samples comprising n primary increments (see ISO 12744).

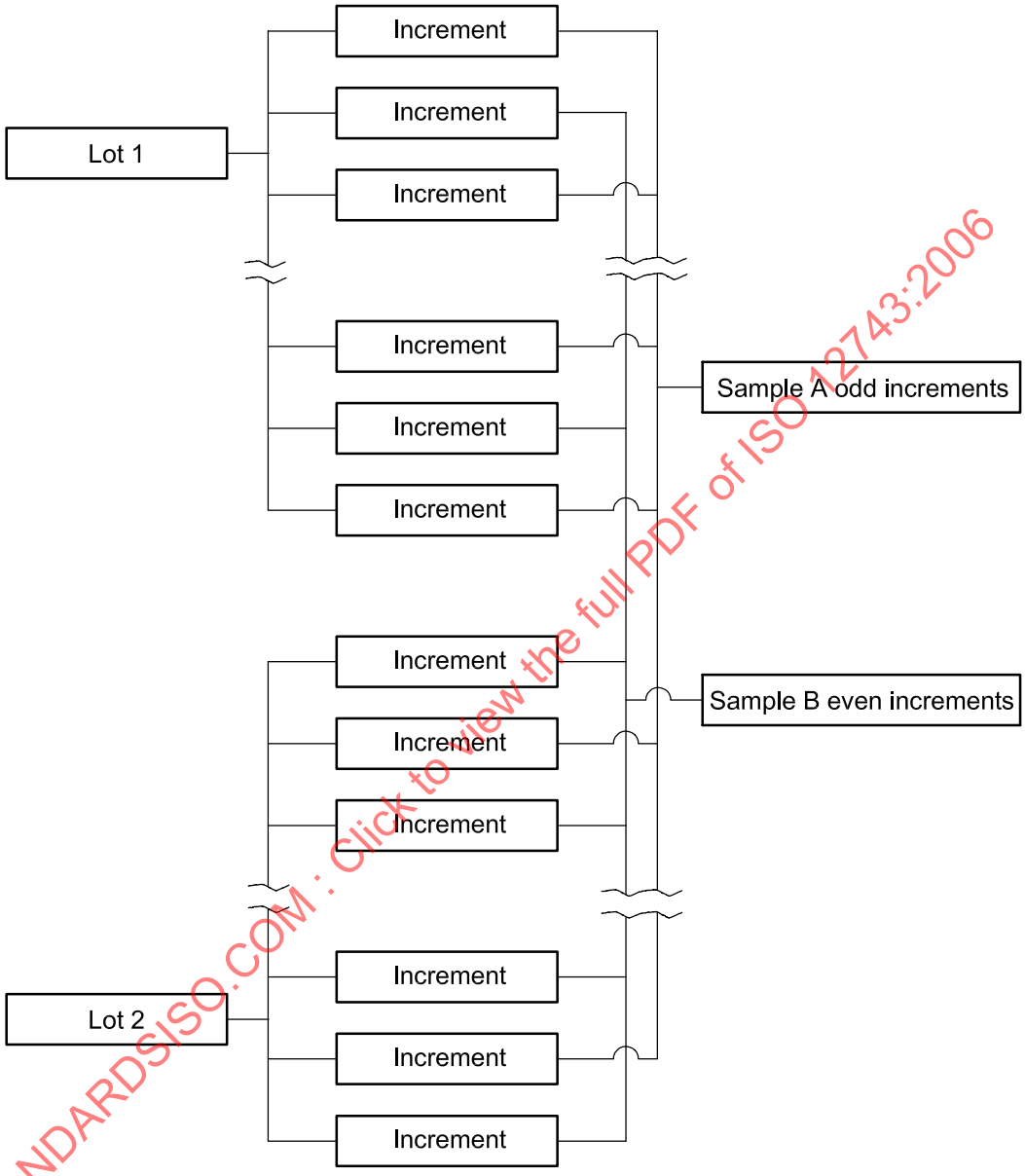


Figure 1 — Example of a plan for interleaved duplicate sampling

EXAMPLE The analyses of interleaved samples taken from 10 lots of copper concentrate are given in Table 1.

Table 1 — Analyses of interleaved samples taken from copper concentrate

Lot	Odd samples (A) Cu % (m/m)	Even samples (B) Cu % (m/m)	Absolute difference Cu % (m/m)
1	30,37	30,34	0,03
2	30,47	30,46	0,01
3	29,99	30,01	0,02
4	29,97	29,98	0,01
5	30,12	30,18	0,06
6	30,02	30,05	0,03
7	30,32	30,35	0,03
8	30,18	30,17	0,01
9	30,31	30,27	0,04
10	30,28	30,25	0,03
Sum of absolute differences			0,27

Equation 14 gives:

$$s_T^2 = (\pi/4)\,(0{,}27/10)^2$$

$$= 0{,}000\,573$$

Hence:

$$s_T = 0{,}024\ \%\ \text{Cu}$$

5 Establishing a sampling scheme

The procedure for establishing a sampling scheme is as follows.

a) Identify the quality characteristics to be measured, and specify the mass of the lot or sub-lot and the desired total variance s_T^2. Typical values of s_T are given in Table 2.

b) Ascertain the nominal top size of the concentrate.

c) Specify the cutter aperture, or the dimensions of the manual-sampling implement, according to the nominal top size of the concentrate.

d) Ascertain the analytical variance s_A^2. Typical values of s_A are given in Table 2.

e) Determine the variance between primary increments $s_{b_1}^2$ and the sample-processing variance s_P^2 if the simplified method is used (see 4.4), or the variance between increments $s_{b_1}^2$ for each proposed stage if the sampling-stage method is used (see 4.3). Table 3 gives typical values of s_{b_1} for the first sampling stage.

f) If the simplified method is used, use Equation 11, 12 or 13 to select the number of primary increments, sample processings and replicate analyses, so that the total variance s_T^2 does not exceed the desired value specified in step a).

   Alternatively, if the sampling-stage method is used, use Equation 8 to select the number of increments n_i required at each sampling stage and the number of replicate analyses so that the total variance s_T^2 does not exceed the value selected in step a).

g) Determine the sampling intervals at each stage, in tonnes for mass-basis systematic sampling (see 7.2) and stratified random sampling within fixed mass intervals (see 7.4), or in minutes for time-basis systematic sampling (see 7.3) and stratified random sampling within fixed time intervals (see 7.4).

h) Take increments at the intervals determined in step g) for each stage during the whole period of handling the lot.

i) Either combine the primary increments into lot samples or subsamples for analysis or analyse each primary increment separately. Examples of suitable sampling schemes are given in Figure 2, but the scheme shown in Figures 2a) and 2c) is not suitable for preparing chemical analysis samples used to analyse for volatile elements, such as mercury.

The lot is often divided into sub-lots from which subsamples are prepared and analysed separately to reduce the total variance, as outlined in 4.4. Subsamples may also be prepared to provide progressive information on the quality of the lot, or to reduce possible changes in the moisture content of samples.

Table 2 — Typical target values of the required total and analytical standard deviations for determination of metal and moisture content

Characteristic	Parameter	Content range/Standard deviation		
Cu	Range	< 30 % (*m*/*m*)	30-50 % (*m*/*m*)	
	s_T	0,05 %	0,1 %	
	s_A	0,03 %	0,03 %	
Pb, Zn	Range	< 30 % (*m*/*m*)	30-50 % (*m*/*m*)	> 50 % (*m*/*m*)
	s_T	0,1 %	0,2 %	0,3 %
	s_A	0,07 %	0,07 %	0,15 %
Ni	Range	< 10 % (*m*/*m*)	10-30 % (*m*/*m*)	
	s_T	0,1 %	0,2 %	
	s_A	0,05 %	0,1 %	
Ag	Range	< 500 g/t	500-100 0 g/t	> 1 000 g/t
	s_T	10 g/t	2 % of concentrate	2 % of concentrate
	s_A	7 g/t	15 g/t	20 g/t
Au	Range	< 5 g/t	5-15 g/t	> 15 g/t
	s_T	0,5 g/t	0,5 g/t	2 % of concentrate
	s_A	0,15 g/t	0,2 g/t	0,4 g/t
Moisture	Range	< 20 % (*m*/*m*)		
	s_T	0,3 %		
	s_A	0,07 %		

NOTE s_T can be reduced if required by performing replicate analyses.

Table 3 — Examples of standard deviations between primary increments

Constituent	Standard deviation between primary increments (s_{b_i})
Cu in copper concentrate	0,1-0,5 % Cu
Pb and Zn in lead and zinc concentrates	0,1-2,0 % Pb/Zn
Ni in nickel concentrates	0,1-0,5 % Ni
Ag in copper, lead and zinc concentrates	1-5 g/t Ag
Au in copper, lead and zinc concentrates	0,5-2,0 g/t Au
Moisture in copper, lead and zinc concentrates	0,2-0,7 %
NOTE The standard deviation between primary increments for mass fraction of silver is applicable to Ag < 500 g/t only.	

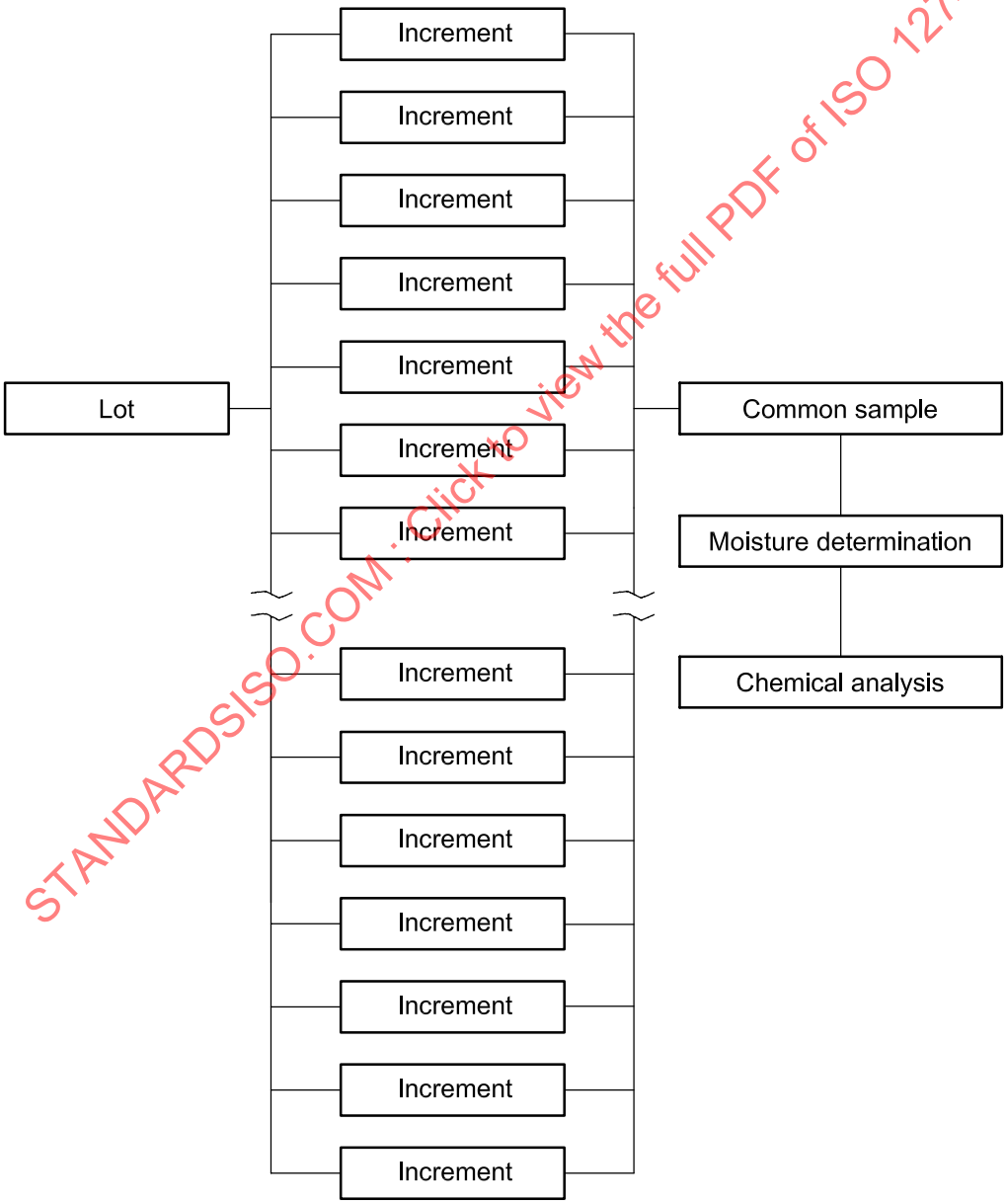
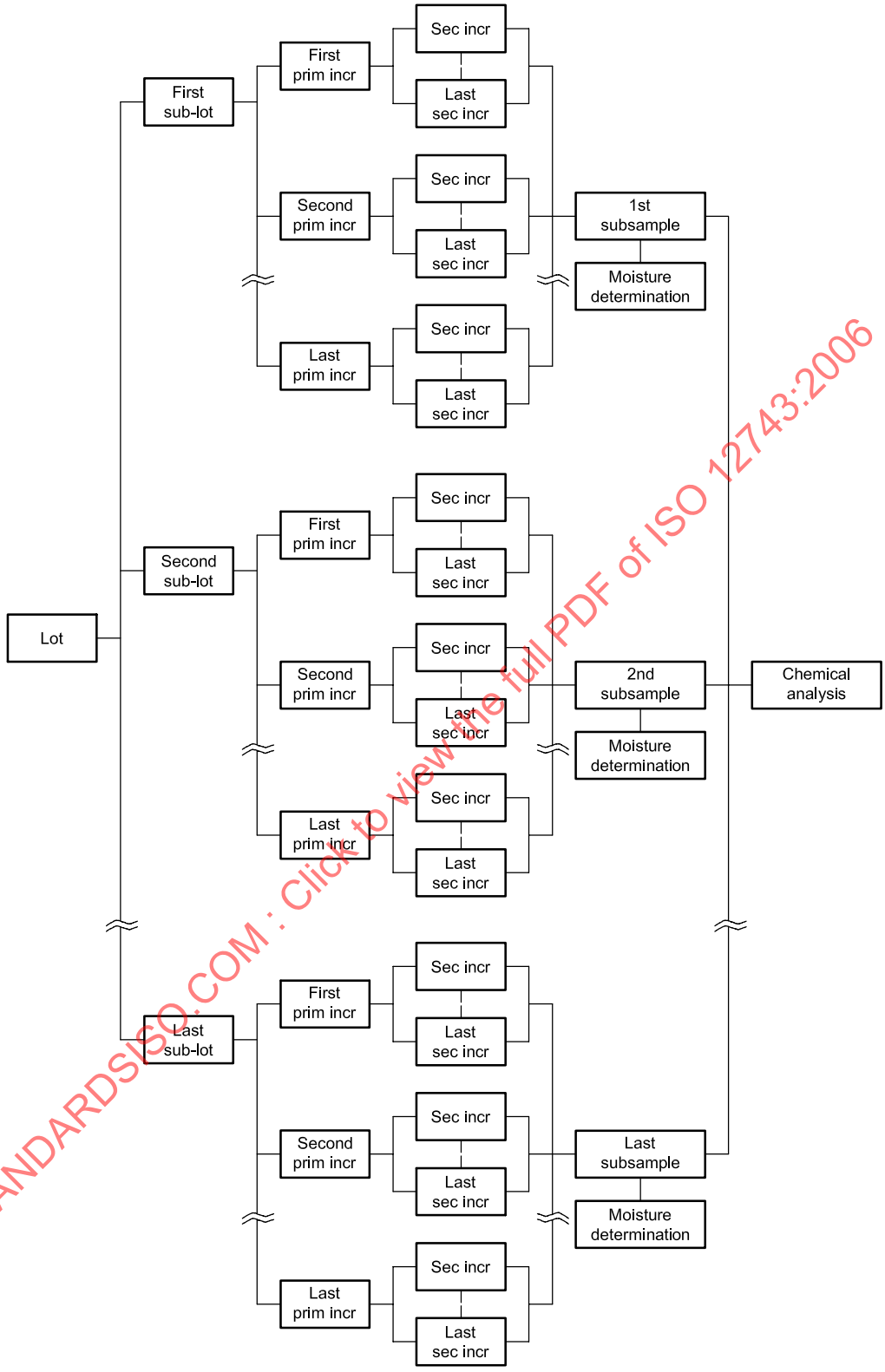


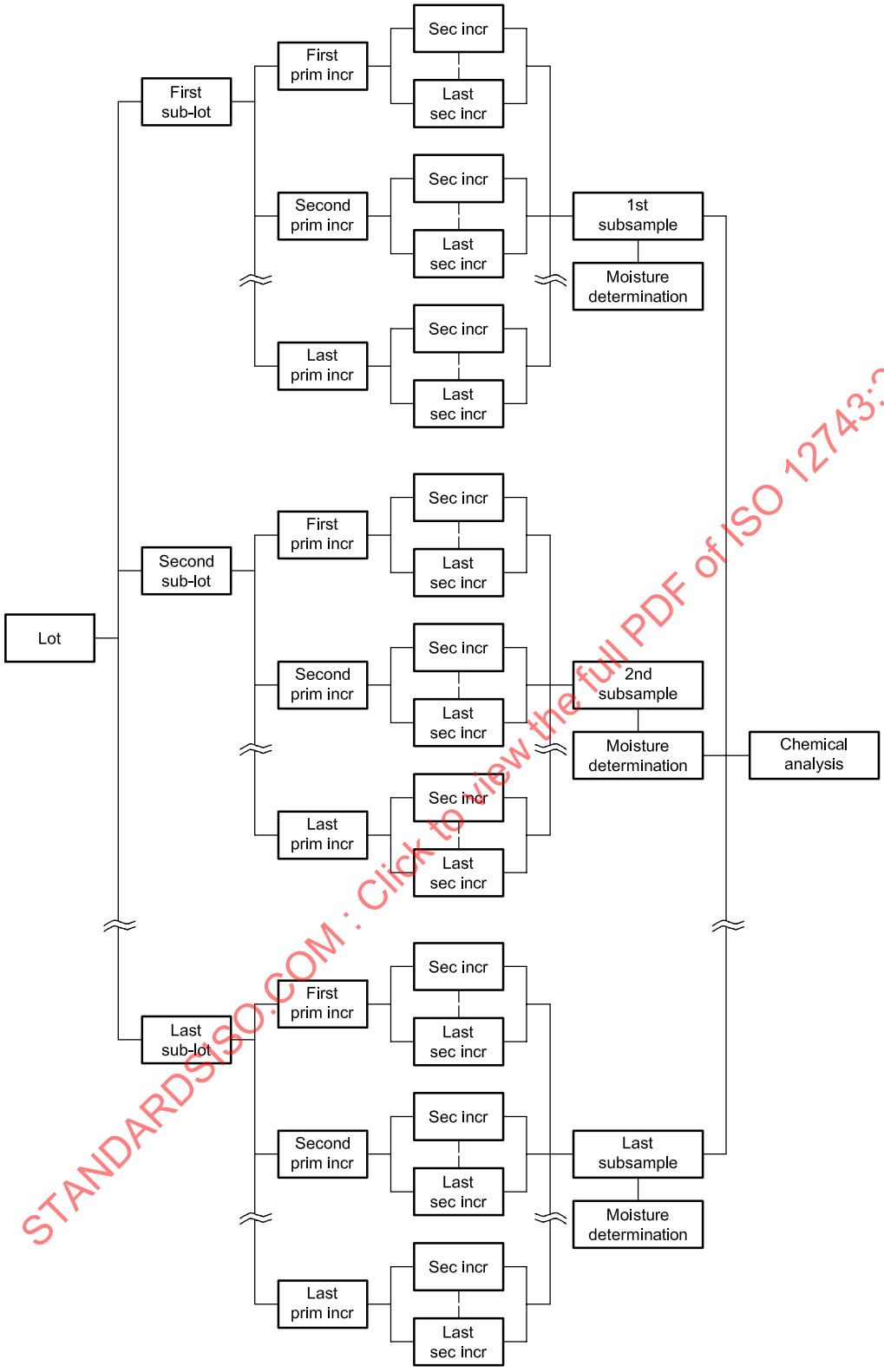
Figure 2a) — Example of a sampling scheme in which a common sample is constituted for the lot for moisture determination and subsequent chemical anaylsis



Key
prim incr: primary increment sec incr: secondary increment

NOTE Mixing, comminution and division steps have been omitted for simplicity. This scheme is not suitable for concentrates that are susceptible to oxidation or decomposition.

Figure 2b) — Example of a sampling scheme in which the lot is divided into sub-lots for moisture determination and a separate lot sample is constituted for chemical analysis



Key
prim incr: primary increment sec incr: secondary increment

NOTE Mixing, comminution and division steps have been omitted for simplicity. This scheme is suitable for concentrates that are susceptible to oxidation or decomposition.

Figure 2c) — Example of a sampling scheme in which the lot is divided into sub-lots for moisture determination, and the dried moisture subsamples are subsequently combined into a single lot sample for chemical analysis

6 Mass of increment

6.1 General

To avoid bias, it is essential that increments are extracted in such a manner that all possible increments from the concentrate stream have the same probability of being selected and becoming part of the final sample for analysis, irrespective of the size, mass or density of individual particles in the stream. This determines the increment mass required to obtain an unbiased sample.

However, there is also a practical minimum increment mass that should always be exceeded to minimize the effect of handling on the characteristics of the increment as it passes through the sampling system. This is particularly important for moisture content, where moisture loss (or gain) must be minimized. The minimum mass needs to be specified for each concentrate type and each sampling device.

6.2 Mass of increment for falling-stream samplers

To avoid bias, the mass of increment at any sampling stage taken by a mechanical or manual cutter type sampler from the concentrate stream at the discharge end of a moving conveyor is determined by the minimum cutter aperture as specified in 8.3.2.2, the maximum cutter speed, as specified in 8.3.3.2, and the flow rate of the concentrate stream as follows:

$$m_I = \frac{GA}{3{,}6\, v_c} \qquad \ldots(15)$$

where

m_I is the mass of increment, in kilograms;

G is the flow rate of the concentrate stream, in tonnes per hour;

A is the cutter aperture, in metres (see 8.3.2.2);

v_c is the cutter speed, in metres per second (see 8.3.3.2).

6.3 Mass of increment for cross-belt samplers

The mass of increment taken by a cross-belt cutter from a moving stream to avoid bias is determined by the minimum cutter aperture as specified in 8.3.2.3, the speed of the concentrate stream (i.e. the belt speed), and the flow rate of the concentrate stream as follows:

$$m_I = \frac{GA}{3{,}6\, v_B} \qquad \ldots(16)$$

where

A is the cutter aperture, in metres (see 8.3.2.3);

v_B is the speed of the concentrate stream, in metres per second.

6.4 Mass of increment for manual sampling from stationary lots

6.4.1 Primary increments

The minimum mass of primary increment taken by a manual-sampling implement from a stationary lot shall be 0,25 kg. However, if the nominal top size exceeds 16 mm due to the presence of agglomerates, the mass shall be increased in accordance with the following equation based on a sampling implement of minimum dimensions $3d \times 3d \times 3d$:

$$m_{\mathrm{I\,min}} = 27\ \rho d^3 \times 10^{-6} \qquad \ldots(17)$$

where

$m_{\mathrm{I\,min}}$ is the minimum mass of increment, in kilograms;

ρ is the bulk density of the concentrate, in tonnes per cubic metre;

d is the nominal top size of the agglomerates, in millimetres.

6.4.2 Mass of secondary and subsequent increments

The minimum mass of secondary and subsequent increments taken by a manual-sampling implement shall be 10 g. However, if the nominal top size is 5 mm or greater due to the presence of agglomerates, the mass shall be increased in accordance with Equation 17.

6.5 Mass of increment for stopped-belt reference sampling

The mass of increment taken from a stopped belt for reference sampling is determined by the minimum length of the sampling frame as specified in Clause 10, the speed of the concentrate stream (i.e. the belt speed) and the flow rate of the concentrate stream as follows:

$$m_I = \frac{GW}{3{,}6 v_{\mathrm{B}}} \qquad \ldots(18)$$

where W is the length of the sampling frame, in metres (see Clause 10).

7 Methods of sampling from concentrate streams

7.1 General

Mechanical and manual sampling of concentrate streams may be carried out on either a mass-basis or a time-basis. This needs to be decided before designing a sampling system and before sampling commences.

7.2 Mass-basis systematic sampling

7.2.1 General

Mass-basis sampling involves the following steps:

a) Distributing the required number of primary increments on a uniform tonnage basis throughout the lot to be sampled.

b) Taking increments of almost uniform mass from each tonnage interval.

NOTE Sample masses are considered to be almost uniform if the coefficient of variation (CV) of the masses is 20 % or less. If increments are not of almost uniform mass, constant-mass division is required, so that the mass of sample reporting to the lot sample or subsample is almost uniform.

7.2.2 Sampling interval

The interval between primary increments for mass-basis sampling shall be determined from the following equation:

$$\Delta m \leqslant \frac{m_L}{n_1} \qquad \ldots(19)$$

where

Δm is the mass interval between primary increments, in tonnes;

m_L is the mass of the lot, in tonnes;

n_1 is the number of primary increments determined in accordance with Clause 5.

The mass interval between primary increments should be rounded down to the nearest tonne, to ensure that the number of primary increments taken will be larger than the minimum number required.

7.2.3 Sample cutter

The following cutters may be used for taking primary increments:

a) A falling-stream cutter whose cutting speed is constant during the course of handling the entire lot.

b) A falling-stream cutter whose cutting speed is constant while cutting the stream but can be regulated, primary increment by primary increment, corresponding to the flow rate of the concentrate on the conveyor belt.

c) A cross-belt cutter.

7.2.4 Taking of primary increments

Each primary increment shall preferably be taken by a single traverse of the sampling device, so that a full cross-section of the concentrate stream is taken. However, in manual-sampling cases where it is not possible to obtain a complete cross-section in one operation, increments may be taken systematically across the concentrate stream so that, when they are combined, they represent the full cross-section of the stream (see 9.7).

The first primary increment shall be taken at a random mass, less than the mass interval Δm determined in 7.2.2. Thereafter, the required number of primary increments shall be taken at fixed mass intervals of Δm, and this interval shall not be changed during the entire course of sampling the lot.

If the planned number of primary increments has been taken and handling has not been completed, additional primary increments shall be taken at the same mass interval until the handling operation is completed.

7.2.5 Constitution of subsamples and lot samples

If the coefficient of variation of primary increment masses is 20 % or less, primary increments may be combined into subsamples or a lot sample, either as taken or after having been processed individually to a particular stage. Subsamples shall preferably comprise equal numbers of consecutive primary increments.

However, if the coefficient of variation of primary increment masses exceeds 20 %, either

a) each primary increment shall be subjected separately to division (according to the rules of division) and determination of its quality characteristics, or

b) primary increments shall be subjected to constant mass division, prior to combining into subsamples or a lot sample.

Primary increments and subsamples should not be combined into a single sample for the lot, unless the composite sample can be adequately mixed (see 15.3).

For the determination of moisture content, it is recommended that a moisture subsample be constituted for each sub-lot. This will not only reduce the total variance, but it will also minimize loss of moisture and hence bias.

7.2.6 Types of division

Two types of division are applicable to mass-basis sampling as follows.

a) Constant-mass division, which is a method of obtaining divided increments, subsamples or lot samples having almost uniform mass, regardless of the variation in the masses to be divided. Cutter-type dividers having variable cutting frequencies can be used for this type of division (see 15.4.6).

b) Proportional division, which is a method of obtaining divided increments, subsamples or lot samples having masses proportional to the varied masses to be divided. Rotary sample dividers can be used for this type of division (see 15.4.5).

NOTE Cutter-type dividers may lead to moisture loss, so are not recommended for division of moisture samples.

7.2.7 Division of increments

Where increments require division and subsamples or a lot sample are constituted from the divided increments, division shall be carried out as follows (see Table 4):

a) If the coefficient of variation of the increment masses is 20 % or less, either constant-mass or proportional division shall be used.

b) If the coefficient of variation of the increment masses is greater than 20 %, division shall be carried out on an increment-by-increment basis using constant-mass division.

7.2.8 Division of subsamples

Where subsamples are divided and a lot sample is constituted from the divided subsamples, division shall be carried out as follows (see Table 4):

a) If the coefficient of variation of the subsample masses is 20 % or less, and the subsamples consist of an equal number of increments, either constant-mass or proportional division shall be used.

b) If the coefficient of variation of the subsample masses is greater than 20 %, and the subsamples consist of an equal number of increments, constant-mass division shall be used.

c) If the subsamples consist of different numbers of increments, proportional division shall be used.

7.2.9 Division of lot samples

When a lot sample is divided, either constant-mass or proportional division shall be used.

Table 4 — Rules for division of increments, subsamples and lot samples for mass-basis and time-basis sampling

Sample for division	Sampling conditions			Type of division	
	Sampling method	Increments per subsample	CV| %	Constant mass	Proportional
Increment	Mass-basis	—	< 20	Yes	Yes
			> 20	Yes	No
	Time-basis	—	—	No	Yes
Subsample	Mass-basis	Equal	< 20	Yes	Yes
	Mass-basis		> 20	Yes	No
		Unequal	—	No	Yes
	Time-basis	Equal or unequal	—	No	Yes
Lot sample	Mass-basis Time-basis	—	—	Yes	Yes

7.3 Time-basis systematic sampling

7.3.1 General

Time-basis sampling involves the following steps:

a) Distributing the required number of primary increments on a uniform time basis throughout the lot to be sampled.

b) For each time interval, taking increments of mass proportional to the concentrate flow rate at the time of taking the increment.

7.3.2 Sampling interval

The interval between primary increments for time-basis sampling shall be determined from the following equation:

$$\Delta t \leqslant \frac{3\,600\ m_{\mathrm{L}}}{G_{\max}\ n_1} \qquad \ldots(20)$$

where

Δt is the time interval between primary increments, in seconds;

$G_{\max}$ is the maximum flow rate, in tonnes per hour.

The time interval between primary increments should be rounded down to the nearest second, to ensure that the number of primary increments taken will be larger than the minimum number required.

7.3.3 Sample cutter

The following cutters may be used for taking primary increments.

a) A falling-stream cutter whose cutting speed is constant during the course of handling the entire lot.

b) A cross-belt cutter.

7.3.4 Taking of primary increments

Each primary increment shall preferably be taken by a single traverse of the sampling device, so that a full cross-section of the concentrate stream is taken. However, in manual-sampling cases where it is not possible to obtain a complete cross-section in one operation, increments may be taken systematically across the concentrate stream so that, when they are combined, they represent the full cross-section of the stream over time (see 9.7).

The first primary increment shall be taken at a random time less than the time interval Δt determined in 7.3.2. Thereafter, the required number of primary increments shall be taken at fixed time intervals of Δt, and this interval shall not be changed during the entire course of sampling the lot.

If the planned number of primary increments has been taken and handling has not been completed, additional primary increments shall be taken at the same time interval, until the handling operation is completed.

7.3.5 Constitution of subsamples and lot samples

Subsamples or lot samples may be constituted in either of the following ways:

a) primary increments, as taken, shall be combined into subsamples or a lot sample, irrespective of the variation of masses of primary increments; or

b) primary increments shall be divided by proportional division; subsamples or lot samples shall then be constituted by combining divided increments.

Where subsamples are analysed to determine the quality characteristics for the lot, the mass of the sub-lot from which the subsample was taken shall be determined, in order to obtain the weighted average of the quality characteristic for the lot.

Primary increments and subsamples should not be combined into a single sample for the lot, unless the composite sample can be adequately mixed (see 15.3).

For the determination of moisture content, it is recommended that a moisture subsample be constituted for each sub-lot. This will not only reduce the total variance, but it will also minimize loss of moisture and hence bias.

7.3.6 Types of division

Both constant-mass and proportional division are applicable to time-basis sampling (see 7.2.6).

7.3.7 Division of increments and subsamples

Increments and subsamples shall be divided by proportional division (see Table 4).

7.3.8 Division of lot samples

When a lot sample is divided, either constant-mass or proportional division shall be used (see Table 4).

7.4 Stratified random sampling

7.4.1 Fixed mass intervals

The procedure shall be as specified in 7.2 except that, when the mass interval has been set, the sample cutter is programmed to take a primary increment at any point at random within this mass interval. This is achieved by using a random number generator, capable of giving a random mass number anywhere within the mass interval (determined in 7.2.2), which activates the sample cutter at the mass corresponding to the mass number generated. The capacity of hoppers in the sampling system shall be sufficient to hold two adjacent increments.

7.4.2 Fixed time intervals

The procedure shall be as specified in 7.3 except that, when the time interval has been set, the sample cutter is programmed to take one primary increment at any point at random within this time interval. This is achieved by using a random number generator, capable of giving a random time number anywhere within the time interval (determined in 7.3.2), which activates the sample cutter at the time corresponding to the time number generated. The capacity of hoppers in the sampling system shall be sufficient to hold two adjacent increments.

NOTE Because the spacing of increments in mass or time is not constant when conducting stratified random sampling, interleaved sampling requires special equipment to direct closely spaced increments to samples A and B.

8 Mechanical sampling of concentrate streams

8.1 General

There are a number of different mechanical sampling devices, and hence it is not possible to specify any particular type which should be used for specific sampling applications. However, the sampling device selected must pass a bias test, i.e. it shall be unbiased. In this respect, special care shall be taken to minimize change in moisture content, e.g. by minimizing vertical drops, eliminating air flows, and avoiding low flow rates. Degradation of the constituent particles shall also be minimized if particle size determination is to be carried out on the sample. Annex C shows typical examples of sample cutters in common use and should be taken as a guide in choosing suitable equipment.

This International Standard deals only with mechanical sampling devices that take a complete cross-section of the concentrate stream. Sampling devices taking only part of the stream shall only be used if it can be shown, using ISO 13292, that there is no significant bias.

8.2 Design of the sampling system

8.2.1 Safety of operators

From the initial stage of design and construction of a sampling system, due consideration shall be given to the safety of operators. Applicable safety codes of the Regulatory Authorities shall be respected.

8.2.2 Location of sample cutters

When choosing the location of sample cutters, the following criteria shall apply.

a) Sample cutters shall be located at a point providing access to the complete concentrate stream.

b) Sampling shall be performed close to the weighing point in space and time.

c) Sampling should be performed at a point in the handling system where there is minimal segregation of the concentrate stream, and where there is minimal risk of errors due to a systematic variation in flow rate or quality.

Basic requirements are to be taken into account from the early stages of design, construction and installation of the system, as well as during the operation and maintenance of the plant. To permit the bias checks specified in 8.2.5, provision should be made for stopped-belt reference sampling adjacent to the sample cutter.

NOTE It is not essential to construct or operate the mechanical sampling system as a whole. Any principal unit or combination of principal units may be operated mechanically and combined at any stage with manual operations.

8.2.3 Provision for interleaved sampling

The sampling system should be capable of generating pairs of interleaved samples for checking and monitoring the sampling and total variances as a function of time (see 4.5).

8.2.4 Provision for stratified random sampling

The sampling system should be designed to handle closely spaced adjacent increments when stratified random sampling is being conducted. In particular, the capacity of hoppers should be sufficient to hold two increments.

8.2.5 Checking precision and bias

When a mechanical sampling system is commissioned, or when the principal parts are modified, checking experiments for precision (see ISO 12744) and bias (see ISO 13292) should be carried out for the system as a whole.

The level of bias shall preferably be verified by comparison with stopped-belt sampling, using the quality characteristics deemed critical in the operation of the sampling system.

8.2.6 Avoiding bias

The sampling system shall be designed to avoid the following.

a) Spillage of the sample.

b) Restriction of the flow of concentrate through the system.

c) Contamination of the sample, e.g. due to cross-contamination between the sample and the concentrate stream, ingress of non-sampled concentrate, or residual concentrate in the sampling system. Thus, when a change is made in the type of concentrate being sampled, the system should be thoroughly cleaned.

8.2.7 Minimizing bias

The sampling system shall be designed to minimize the following.

a) Change in moisture content, e.g. by enclosing the sampling system and minimizing vertical drops.

b) Loss of dust.

c) Degradation of the constituent particles, if the sample is taken for particle size determination.

8.2.8 Configuration of the sampling system

The sampling system should be arranged in such a way that the principal units can be operated individually. In the event of a breakdown in the crushing and dividing parts of the system, provision should be made to enable sampling to be carried out by alternative means. For example, increments taken by the primary cutter may be diverted to a pre-installed facility, e.g. to a short conveyor for taking secondary increments, or to a concrete pad or a receiving truck for manual sample processing.

8.3 Sample cutters

8.3.1 General

Sample cutters may be divided into two types as follows.

a) Falling-stream cutters which collect the increment from the trajectory of the concentrate stream, e.g. at a transfer point from a conveyor or from the output from a bin or hopper.

b) Cross-belt cutters which collect the increment from the concentrate while it is being carried on a conveyor belt.

8.3.2 Design criteria

8.3.2.1 General

To minimize bias in taking increments, the sample cutter shall, in addition to complying with the requirements specified in 8.2.6 and 8.2.7, fulfil the criteria in either 8.3.2.2 for falling-stream cutters or 8.3.2.3 for cross-belt cutters.

8.3.2.2 Falling-stream cutters

The following criteria shall apply.

a) There shall be no impediment to the flow of concentrate into the cutter at the maximum flow rate of the concentrate.

b) The cutter shall be of the self-clearing type, e.g. stainless steel or polythene lined, discharging each increment completely.

c) Discharge chute angles shall be a minimum of 60° to the horizontal.

d) No materials other than the concentrate sample shall be introduced into the cutter, e.g. dust must be prevented from accumulating in the cutter when it is in the parked position.

e) The cutter shall collect a complete cross-section of the concentrate stream, both the leading and trailing edges completely clearing the stream at the two limits of the cutter path.

f) The cutter shall intersect the concentrate stream either in a plane normal to, or along an arc normal to, the mean trajectory of the stream.

g) The cutter shall travel through the concentrate stream at a uniform speed, not deviating by more than 5 % at any point.

h) The geometry of the cutter opening shall be such that the cutting time at each point in the stream is equal, not deviating by more than 5 %, i.e. straight path cutters shall have parallel cutter lips and radial cutters shall have radial cutter lips.

i) The cutting aperture of the cutter shall be not less than 30 mm or, if agglomerates are present, the greater of 30 mm and three times the nominal top size of the concentrate.

NOTE The cutter aperture may need to be increased above 30 mm if blockages occur for wet concentrates.

j) Bucket cutters shall be of sufficient capacity to accommodate the increment mass obtained at the maximum flow rate of the concentrate.

8.3.2.3 Cross-belt cutters

The following criteria shall apply.

a) There shall be no impediment to the flow of concentrate into the cutter at the maximum flow rate of the concentrate.

b) The cutter shall be of the self-clearing type, e.g. stainless steel or polythene lined, discharging each increment completely.

c) Discharge chute angles shall be a minimum of 60° to the horizontal.

d) No materials other than the concentrate sample shall be introduced into the cutter, e.g. dust must be prevented from accumulating in the cutter when it is in the parked position.

e) The cutter shall collect a complete cross-section of the concentrate stream, both the leading and trailing edges completely clearing the stream at the two limits of the cutter path.

f) The cutter shall intersect the concentrate stream in a plane normal to the mean trajectory of the stream.

g) The cutter shall travel through the concentrate stream at a uniform speed, not deviating by more than 5 % at any point.

h) The geometry of the cutter opening shall be such that the cutting time at each point in the stream is equal, not deviating by more than 5 %.

i) The cutting aperture of the cutter shall be not less than 100 mm or, if agglomerates are present, the greater of 100 mm and three times the nominal top size of the concentrate.

j) The cutter bucket shall be of sufficient capacity to accommodate the increment mass obtained at the maximum flow rate of the concentrate.

k) The profile of the conveyor belt shall be adjusted to the curvature of the cutter path, e.g. by using additional multi-roller idlers, to ensure that all fines are collected from the belt.

l) Any flexible blades, brushes or skirts fitted to the cutter shall be regularly adjusted so that they maintain close contact with the surface of the conveyor belt, to ensure that the complete concentrate section in the path of the cutter is collected from the belt.

8.3.3 Cutter speed

8.3.3.1 General

In designing a mechanical sample cutter, one of the most important parameters is the cutter speed. For example, with falling-stream cutters, too high a cutter speed will lead to:

a) biasing of the sample due to deflection of the larger particles;

b) biasing of the sample by dust and rebounding particles caused by excessive turbulence;

c) shock load problems and difficulties in maintaining constant speed while cutting the concentrate stream.

8.3.3.2 Falling-stream cutters

Experimental work undertaken by Gy[3] shows that, for sampling heterogeneous material streams of low belt loading where the particle size distribution is very narrow, significant bias may be introduced when the cutter speed exceeds 0,6 m/s, or the cutter aperture is less than three times the nominal top size of the material being sampled.

On the basis of this evidence, the speed of cutters which have an “effective cutter aperture” w_o (see Figure 3) equal to 30 mm or three times the nominal top size of the concentrate, whichever is the greater, should not exceed 0,6 m/s.

NOTE Cutter speeds above 0,6 m/s may be used only when it can be shown experimentally, in accordance with ISO 13292, that no significant bias is introduced.

8.3.3.3 Cross-belt cutters

When sampling from a moving conveyor belt using a cross-belt cutter, increments shall be taken from the complete width of the belt, either at right angles to the stream axis or at an angle to the stream axis, so that the cutter does not create a “bow wave” in front of it. The peripheral speed shall be such that excessive turbulence is not created, and shall be a minimum of 1,5 times the belt speed.

8.4 Mass of increments

The minimum mass of each increment obtained in one pass of the sample cutter shall comply with 6.2 or 6.3.

8.5 Number of increments

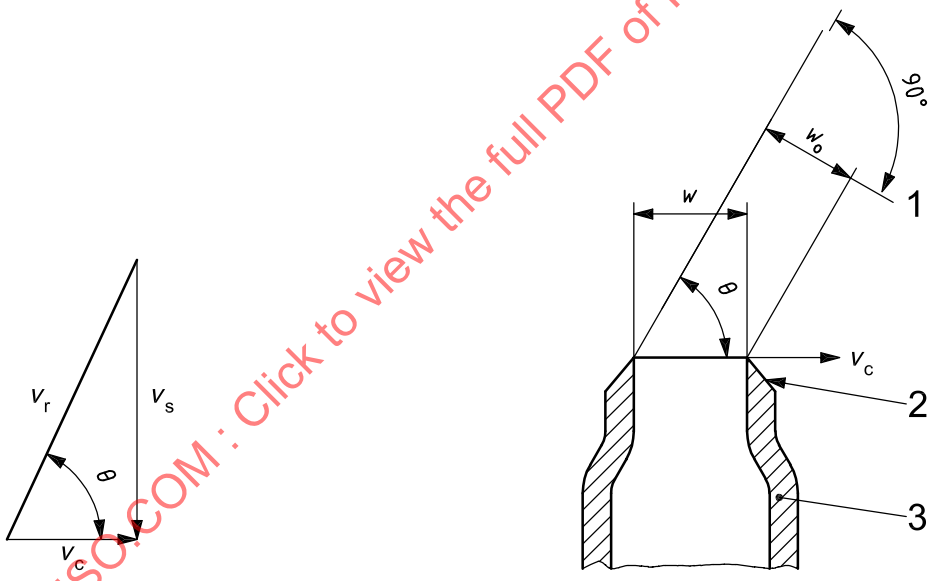
The number of increments shall comply with the requirements of Clause 5.

8.6 Sampling interval

The sampling interval shall comply with the requirements of 7.2, 7.3 or 7.4.

8.7 Routine checking

Maintenance and inspection of the sampling system, particularly cutter apertures, shall be carried out frequently. An example of a checklist is provided in Annex D. A bias-monitoring program, where data are routinely collected for each lot, is a useful exercise. Compliance with this International Standard shall be verified when modifications are made.



a) Vector diagram

b) Effective aperture of cutter

Key

v_s velocity of concentrate stream

v_c velocity of cutter

v_r resultant velocity

θ angle between resultant velocity and direction of cutter path

w cutter aperture

w_o effective cutter aperture

1 effective cutter aperture

2 cutting edges

3 cutter

Figure 3 — Cutter with cutting edges designed to cut normal to the concentrate stream, illustrating the effective cutter aperture

9 Manual sampling of concentrate streams

9.1 General

Manual sampling may be performed on a mass-basis or a time-basis, provided that access is available to the complete concentrate stream and that there is no risk to the safety of the operator. In relation to safety of operators, the applicable safety codes of the Regulatory Authorities shall be respected.

9.2 Choosing the sampling location

The sampling location shall

a) provide complete operator safety,

b) provide access to the complete concentrate stream,

c) provide minimum segregation of the concentrate stream, e.g. in particle size and moisture content, and

d) be close to the weighing point in space and time.

In most handling systems, the only sampling location which satisfies the above requirements is at a transfer point immediately after transfer, where the complete falling stream can be accessed. Partial stream sampling shall be avoided, unless cuts are taken systematically across the full width of the stream and it can be demonstrated experimentally, in accordance with ISO 13292, that there is no significant bias.

Sampling the concentrate from the top of a moving conveyor belt shall also be avoided, unless the surface is a fresh random exposure of the concentrate and cuts are taken systematically across the full width of the stream. It shall also be demonstrated experimentally that no significant bias is introduced.

9.3 Sampling implements

Sampling from falling streams may be performed using a manual cutter of the type specified in Annex E, including mechanically assisted devices.

The manual cutter shall comply with the requirements of 8.3.

Sampling scoops may be used for sampling concentrate from moving conveyor belts (see Annex E), provided it can be shown experimentally, in accordance with ISO 13292, that no significant bias is introduced.

9.4 Mass of increments

The minimum mass of each increment shall comply with the requirements of 6.2.

9.5 Number of increments

The number of increments shall comply with the requirements of Clause 5.

9.6 Sampling interval

The sampling interval shall comply with the requirements of 7.2, 7.3 or 7.4.

9.7 Sampling procedures

9.7.1 General

Sampling shall be carried out by taking a single cut or multiple cuts across the complete concentrate stream with a sampling implement. Care shall be taken to minimize change in moisture content, both during and after sampling.

9.7.2 Full stream cut from a falling stream

Increments shall preferably be taken from a falling stream in a single pass, moving the sampling implement across the full width of the stream at a uniform speed, taking care that the concentrate does not overflow before the sampling implement leaves the stream.

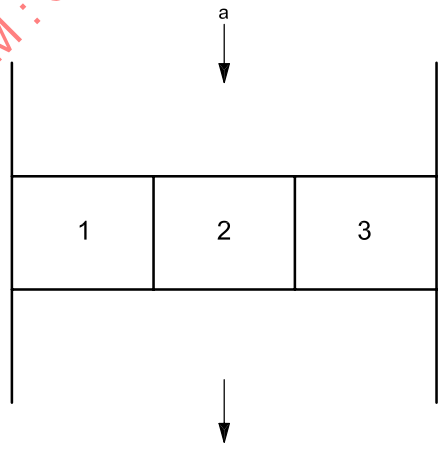
9.7.3 Partial stream cuts from a falling stream

When the concentrate flow rate is too large to take a complete cross-section of the stream in a single pass, the sampling implement may be placed in the stream, to draw increments systematically across the whole concentrate stream in a number of discrete actions, provided it can be shown, in accordance with ISO 13292, on a regular and on-going basis that no significant bias is introduced.

The stream should be divided into separate areas, e.g. at least three areas as illustrated in Figure 4. After taking an increment from the first area, the implement is systematically moved to each remaining area for subsequent increments, so that after three increments the complete cross-section of the concentrate stream has been sampled. Each set of increments may be combined to represent the full cross-section of the concentrate stream.

The sampling implement is inserted upside-down into the stream, inverted and allowed to fill, then withdrawn from the stream. Overfilling shall be avoided, because this may lead to bias in the sample.

Where a subsample or a lot sample is constituted from a number of increments, each subsample or lot sample shall contain increments that represent the complete cross-section of the stream.



[a] Concentrate stream.

Figure 4 — Plan view of a concentrate stream with recommended positions/sequence for manually taking increments across the stream

The actual number of cuts required to obtain an increment from the complete cross-section of a falling stream will depend on the stream geometry, its density, and the dimensions of the sampling implement.

9.7.4 Sampling from moving conveyor belts

When access to a falling stream is severely restricted or unsafe, increments may be taken from concentrate *in situ* on a moving conveyor belt, provided the surface of the concentrate on the conveyor is a fresh random exposure of concentrate, and it can be shown, in accordance with ISO 13292, on a regular and on-going basis that no significant bias is introduced. To satisfy these requirements, increments shall be taken as close as possible to the point at which the concentrate is transferred onto the conveyor belt.

Increments shall be drawn systematically across the whole concentrate stream in a number of discrete actions using a sampling scoop (see Annex E), in a similar manner to that illustrated in Figure 4. Each set of increments taken across the stream may be combined to represent the full cross-section of the concentrate stream. Alternatively, if interleaved samples are being constituted, increments taken systematically across the stream shall be directed alternately to sample A and sample B.

10 Stopped-belt reference sampling

Stopped-belt sampling is the accepted method for obtaining a reference sample against which other sampling procedures may be compared, although it presents operational difficulties even if the handling system is capable of being restarted with a fully loaded belt. The main problems are losses in production tonnage and the difficulty experienced in sequence starting the handling system. During a ship loading or unloading operation, this can cause delays in the turnaround time of the ship. Special care is also required if the samples are taken for moisture determination, because moisture may be lost while the reference sample is being removed from the conveyor.

An alternative reference method, which is also expensive, is to divert the concentrate flow onto a transfer conveyor belt, to produce a concentrate bed identical to that on the main belt where routine sampling is being conducted. Stopped-belt sampling is then carried out on the transfer belt. The transfer conveyor belt should be of sufficient length to allow establishment of a concentrate bed that is not influenced by any longitudinal segregation introduced by the diversion plate. The point of diversion to the transfer belt should be as close as possible to the point where routine sampling is being conducted.

The procedure for sampling from a stopped belt shall be as follows.

a) Determine the parameters for sampling in accordance with Clause 5.

b) Stop the belt at the time or mass intervals determined in accordance with 7.2, 7.3 or 7.4.

c) At each stoppage, place a suitably profiled sampling frame (see Annex F) with minimum internal dimensions of 30 mm or three times the nominal top size of the concentrate, whichever is the greater, across the width of the belt, and insert it through the concentrate so that it is in contact with the belt across its full width, thereby defining the reference increment and its mass (see 6.5).

d) Should any large agglomerates obstruct insertion of the frame, push those at the left-hand edge of the frame into the increment and those at the right-hand edge of the frame out of the increment.

e) Remove the concentrate within the sampling frame in the shortest possible time to prevent loss of moisture, ensuring that all concentrate particles are collected by sweeping the belt clean, and deposit each increment into a suitable container.

f) If the reference increment is to be used for moisture determination, seal the container to prevent any change in moisture content.

   For the determination of moisture content, it is recommended that a moisture subsample be constituted from each sub-lot. This will not only reduce the total variance, but will also minimize loss of moisture and hence bias.

g) If paired comparisons are required on an increment-by-increment basis, keep the increments separate.

If the quality of the lot is required, combine the increments into subsamples or a lot sample in accordance with 7.2.5 or 7.3.5.

h) Store the increments, subsamples or lot samples in labelled containers as specified in Clause 17.

11 Sampling from grabs

11.1 General

Mechanical sampling from moving streams is the preferred method. However, while a vessel is being unloaded, sampling may be carried out by taking increments from the concentrate contained in the grab, particularly if a gantry scale is being used for weighing.

11.2 Mass of primary increments

The minimum mass of primary increments shall comply with the requirements of 6.4. Primary increments shall be of almost uniform mass, i.e. the coefficient of variation of the masses shall be not greater than 20 %.

11.3 Number of primary increments

The number of primary increments shall comply with the requirements of Clause 5. These increments shall be taken from the concentrate in the grab every n_G grabs, where n_G is given by:

$$n_G \leqslant \frac{m_L}{n_1 m_G} \qquad \ldots(21)$$

where m_G is the average mass of concentrate in each grab, in tonnes.

The value of n_G obtained in Equation 21 shall be rounded down to the next lower whole number.

11.4 Method of sampling

One increment shall be taken from the concentrate in each selected grab using a spear sampler (see Figure 5 and 15.4.8) of appropriate dimensions. Bias tests shall be conducted, in accordance with ISO 13292, on a regular and on-going basis to show that no significant bias is introduced.

11.5 Constitution of subsamples and lot samples

The lot sample shall be constituted by combining all increments taken from the lot at an appropriate stage of sample processing. When the lot is divided into sub-lots, subsamples shall be constituted by combining the increments taken from a given sub-lot at an appropriate stage of sample processing, in accordance with 7.2.5, if required.

For the determination of moisture content, it is recommended that a moisture subsample be constituted from each sub-lot. This will not only reduce the total variance, but will also minimize loss of moisture and hence bias.

An example of the design of a sampling scheme, and the estimation of the sampling and total variance for sampling from grabs, is given in Annex B.

12 Sampling from trucks, railway wagons and sampling hoppers

12.1 General

Mechanical sampling from moving streams is the preferred method. However, sampling from trucks, railway wagons and sampling hoppers may be carried out, particularly when the trucks, railway wagons and sampling hoppers are weighed, provided that access to the full depth of the concentrate is available.

12.2 Mass of primary increments

The minimum mass of primary increments shall comply with the requirements of 6.4. Primary increments shall be of almost uniform mass, i.e. the coefficient of variation of the masses shall be not greater than 20 %.

12.3 Number of primary increments

The number of primary increments shall comply with the requirements of Clause 5, and the number to be taken from each truck, railway wagon or sampling hopper constituting the lot is given by the following equation:

$$n_W \geqslant \frac{n_1}{N_T} \qquad \ldots(22)$$

where

n_W is the number of increments to be taken from each truck, railway wagon or sampling hopper;

N_T is the number of trucks, railway wagons or sampling hoppers constituting the lot.

The value of n_W obtained from Equation 22 shall be rounded up to the next higher whole number.

12.4 Method of sampling

The number of increments n_W to be taken from each truck, railway wagon or sampling hopper shall be taken from locations spaced as evenly as possible over the surface of the concentrate in the truck, railway wagon or sampling hopper using a spear sampler (see Figure 5) or a grab sampler (see Figure 6), so that increments represent almost uniform masses of concentrate. The minimum internal dimensions of the spear or grab sampler shall be 30 mm or, if agglomerates are present, three times the nominal top size of the concentrate, whichever is the greater. It is essential that each increment be taken from the full depth of the concentrate in the truck, railway wagon or sampling hopper, and that the full vertical column is extracted for the sample to be representative of the lot, particularly when sampling a lot that has segregated or when the moisture has migrated due to long periods of storage or long transportation distances. Bias tests should be carried out in accordance with ISO 13292 on a regular and on-going basis, e.g. by introducing a portable conveyor into the handling system, to confirm that the procedure is not significantly biased. Increments shall not be taken from the top layers only, because this is likely to introduce serious bias.

NOTE Care needs to be taken when using spear samplers, because internal friction within the probe can prevent the full vertical column from being collected.

12.5 Constitution of subsamples and lot samples

The lot sample shall be constituted by combining all increments taken from the lot at an appropriate stage of sample processing. When the lot is divided into sub-lots, subsamples shall be constituted by combining the increments taken from a given sub-lot at an appropriate stage of sample processing, in accordance with 7.2.5 and 4.5, if required.

For the determination of moisture content, it is recommended that a moisture subsample be constituted from each sub-lot. This will not only reduce the total variance, but will also minimize bias.

An example of a sampling scheme for sampling from trucks is given in Table 5.

Table 5 — Example of a sampling scheme for sampling from trucks

Stage	Selection					Preparation and comments
	Method	Sub-lot (100 t)		Lot (500 t)		
		n_i	Sample mass (kg)	n_i	Sample mass (kg)	
1	Automatic spear sampling (m_l = 3 kg)	12	36	60	180	Two increments per truck
2	Manual spear sampling (m_l = 0,35 kg)	24	8.4	120	42	Subsamples from each sub-lot kept separate
3	Manual increment division (m_l = 0,1 kg)	20 (×2)	2.0 (×2)	200	20	Moisture determination on subsamples, then combine and crush to − 1 mm to form a lot sample for chemical analysis
4	Rotary division (1/8)			Large	2,5	Pulverise to 150 µm
5	Rotary division (1/8)			Large	0,3 (×4)	

Dimensions in millimetres

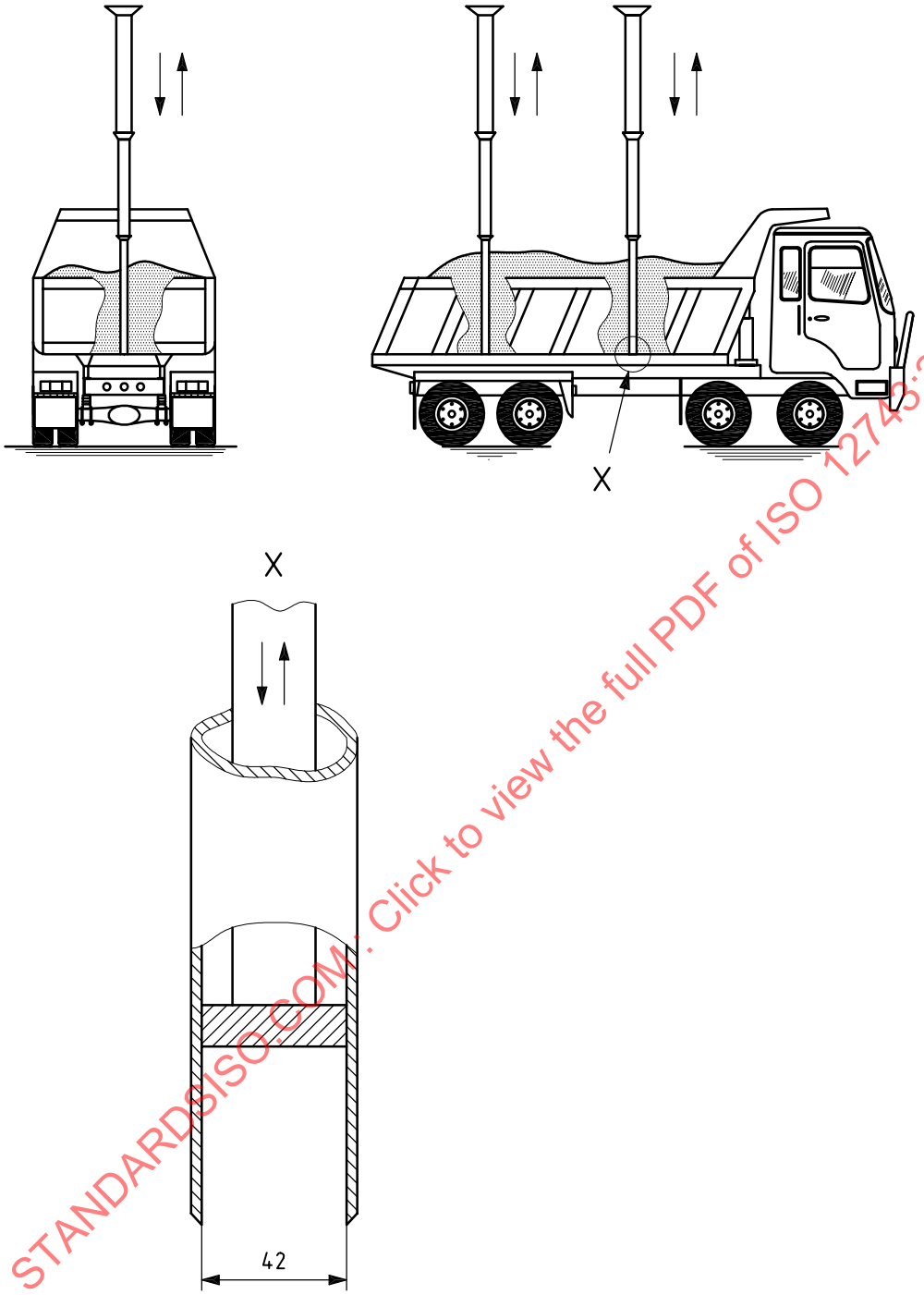


Figure 5 — Example of a spear sampler

Dimensions in meters

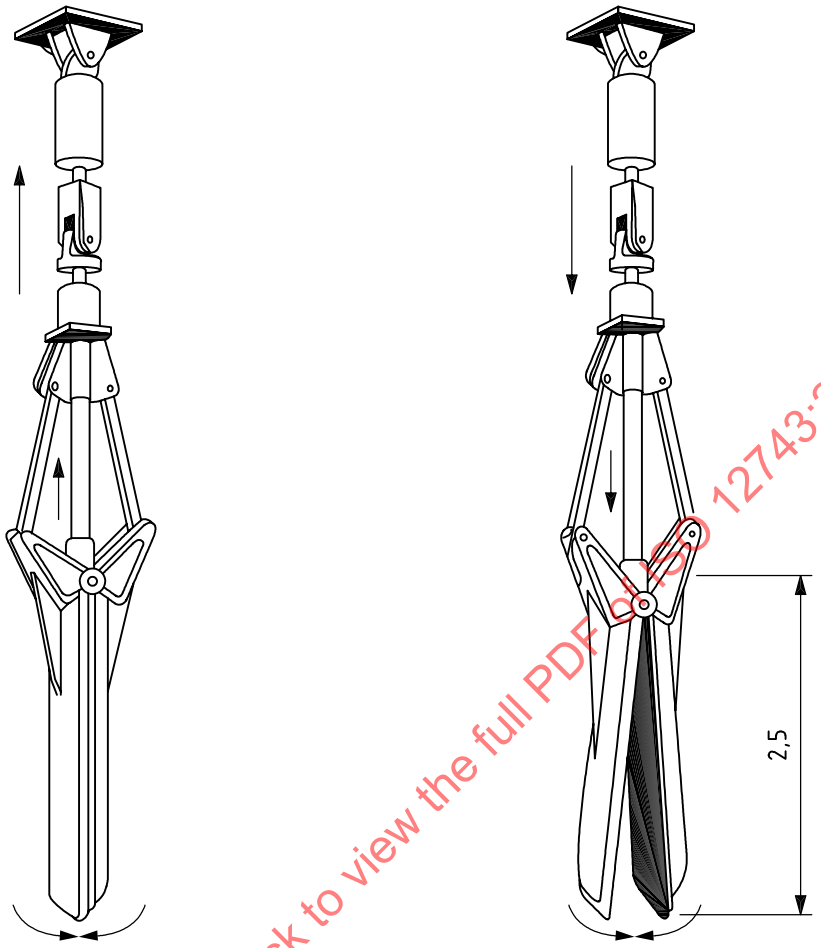


Figure 6 — Example of a grab sampler

13 Sampling of concentrate in bags or drums

13.1 General

Some concentrates are shipped in bags or drums. They should be sampled at the point where weighing takes place.

13.2 Mass of primary increments

The minimum mass of primary increments shall comply with the requirements of 6.4. Primary increments shall be of almost uniform mass, i.e. the coefficient of variation of the masses shall be not greater than 20 %.

13.3 Number of primary increments

The number of primary increments shall comply with the requirements of Clause 5. If this number is greater than the number of bags or drums, the number of increments to be taken from each bag or drum may be calculated as follows:

$$n_b \geqslant \frac{n_1}{N_b} \qquad \ldots(23)$$

where

n_b is the number of increments to be taken from each bag or drum;

N_b is the number of bags or drums constituting the lot.

The value of n_b obtained from Equation 23 shall be rounded up to the next higher whole number.

On the other hand, if the required number of primary increments is less that the number of bags or drums, the increments shall be taken from every n_d bags or drums, where n_d is given by:

$$n_d \leqslant \frac{N_b}{n_1} \quad \ldots(24)$$

The value of n_d obtained in Equation 24 shall be rounded down to the next lower whole number.

13.4 Method of sampling

13.4.1 General

The recommended method of sampling bags and drums is to sample during the filling or emptying of the bag or drum. However, spears (see Figure 5) may also be used to sample the concentrate in the bag or drum provided that the spear penetrates the full depth of the bag or drum and the full column of concentrate is extracted.

13.4.2 Sampling during filling or emptying

If the bags or drums are being filled from a hopper or emptied into a hopper, the falling stream of concentrate can be sampled mechanically using a cutter or a divider in accordance with Clause 8 or manually using a suitable implement in accordance with Clause 9.

13.4.3 Spear sampling

The spear shall have a minimum diameter of 30 mm or, if agglomerates are present, three times the nominal top size of the concentrate, whichever is larger. It shall be long enough to penetrate to the bottom of the bag or drum. The procedure is as follows:

a) Insert the spear into the top of the concentrate in the bag or drum.

b) Push the spear into the concentrate until it reaches the bottom of the bag or drum. Considerable force may be required to achieve this.

c) Withdraw the spear from the concentrate, ensuring that no concentrate is lost from inside the spear.

d) Remove the increment from the spear and place the increment in a container made of material that is impervious to moisture.

e) Place a lid on the container between increments to minimize moisture loss.

13.5 Constitution of subsamples and lot samples

The lot sample shall be constituted by combining all increments taken from the lot at an appropriate stage of sample processing. When the lot is divided into sub-lots, subsamples shall be constituted by combining the increments taken from a given sub-lot at an appropriate stage of sample processing, in accordance with 7.2.5, if required.

For the determination of moisture content, it is recommended that a moisture subsample be constituted from each sub-lot. This will not only reduce the total variance, but will also minimize loss of moisture and hence bias.

14 Sampling of stockpiles

A discussion of issues relevant to the sampling of stockpiles is given in Annex G.

15 Methods of comminution, mixing and division

15.1 General

Each sampling stage consists of a series of comminution, mixing and division operations. When the sample is to be used for chemical analysis, all three operations may be carried out, with drying being conducted at temperatures of up to 105 °C ± 5 °C, if necessary, to facilitate subsequent sample processing. However, where analysis for volatile elements such as mercury is required, drying shall be carried out at temperatures not exceeding 60 °C.

If the sample is to be used for moisture determination, drying is not permitted during processing of the moisture sample and care must be taken to minimize loss of moisture by keeping exposure to the atmosphere to a minimum. Hence, moisture samples shall not be submitted to comminution, and shall not be mixed prior to division, unless mixing is carried out in a closed container, e.g. a plastic bag.

15.2 Comminution

15.2.1 General

Crushers, grinders and pulverizers, called mills in this International Standard, are used to reduce the nominal top size of samples for chemical analysis to a suitable level for subsequent division. They are not applicable to moisture samples, which shall not be crushed or pulverized.

Crushers are usually used if the concentrate is coarse, e.g. due to the presence of agglomerates, whereas pulverizers are used at the final stage of sample processing to reduce the particle size to 150 µm. Grinders are used at the intermediate stages.

The sample shall be fed uniformly into mills in such a way that choking of the mill or changes in mill speed, which may result in variation in the particle size distribution of the product, are avoided.

During preparation of the chemical analysis sample, screening to remove oversize particles for re-crushing and recombination shall not be carried out, unless it can be shown experimentally, in accordance with ISO 13292, for each concentrate being processed that no significant bias is introduced.

Material that is difficult to crush is usually different in composition from the remainder of the sample and cannot be easily mixed back into the sample.

The precision of sample division and analysis is adversely affected by the presence of oversize material. Mill performance should be checked regularly to ensure that at least 95 % of the mill product is below the stated nominal top size.

15.2.2 Mills

Suitable mills include jaw crushers, roll crushers, cone crushers, plate mills and ring mills. They should be easy to clean and shall be cleaned between samples.

Those parts of the mill that come into contact with the concentrate should be of wear-resistant material to minimize contamination. This is particularly important for samples in which trace elements are to be determined, and every effort should be made to use equipment that does not contain any of the elements to be determined.

Certain mills, such as ring mills and plate mills, tend to become heated, and samples shall not be allowed to remain in them long enough to become affected. If a mill is used for a series of samples, it should either be water cooled or allowed to cool between samples.

Factors which influence the choice of mill for any stage of sample processing are:

a) the particle size of the concentrate;

b) the type of crushing action of the mill;

c) the type of testing which is to be carried out on the sample.

Mills that crush mainly by compression, such as jaw crushers and roll crushers, are preferred to those that grind by attrition under pressure, e.g. plate mills. Because of the danger of sample contamination for small gap settings, plate mills are only suitable for intermediate grinding prior to final comminution.

15.3 Mixing

15.3.1 General

The precision of division can be improved by thorough mixing of the sample prior to division. The need for mixing is particularly important where samples from more than one source are combined. Where possible, the sample-processing scheme should be designed so that the need for mixing is minimized.

Mixing of moisture samples may result in moisture loss and hence bias. Consequently, moisture samples shall not be mixed, unless mixing is carried out in a sealed container, e.g. a plastic bag.

15.3.2 Methods of mixing

Mixing can be carried out by one of the following methods.

a) Use of mechanical mixers such as a V-mixer (see Figure 7), a gyratory cylinder or a ploughshare mixer.

b) Use of a pair of mixing trays (see Figure 8), in which the sample is transferred from one tray onto the other a minimum of six times.

c) Strip mixing in which the concentrate is formed into a strip by careful distribution of the concentrate from a shovel. The length/width ratio of the strip shall be not less than 10:1. A complete cross-section of the concentrate strip is taken, commencing at one end, and spread out to form a new strip. Each successive cross-section is spread out on top of the preceding cross-section, layer upon layer, until the old strip has been converted into the new strip. The above process is repeated twice.

d) Pulverization in a ring mill.

e) Passing the sample through a riffle, or preferably a rotary sample divider, three times in succession, recombining the portions after each pass. Dust losses shall be minimized.

NOTE Some methods of hand mixing, for example forming and reforming a conical pile, can have the opposite effect to that intended, and can lead to increased segregation.

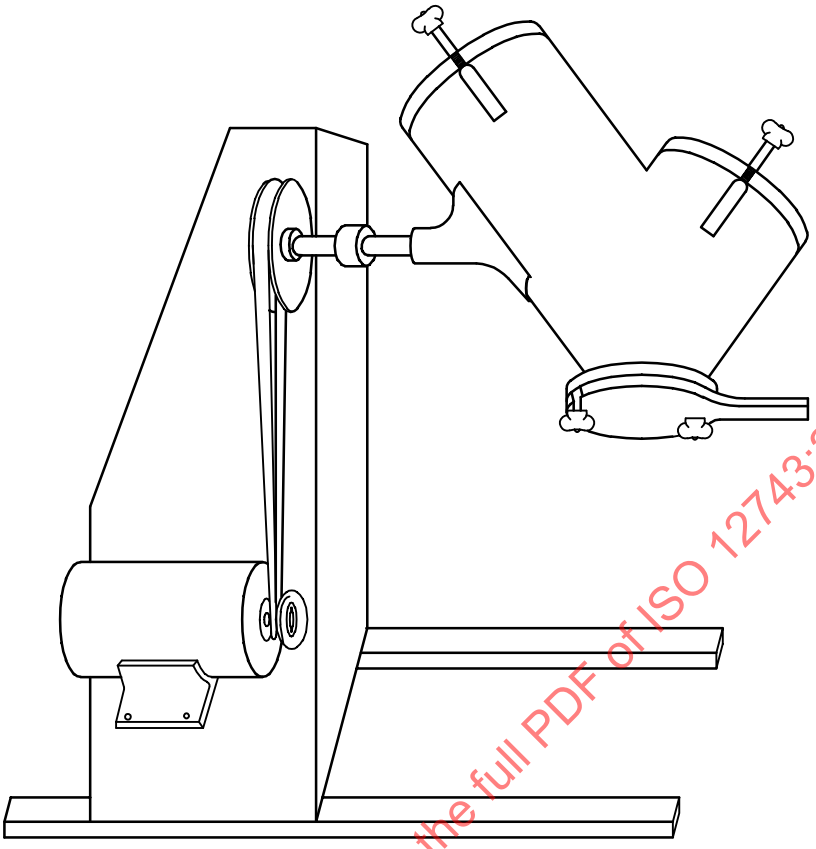


Figure 7 — Example of a V-mixer for sample mixing

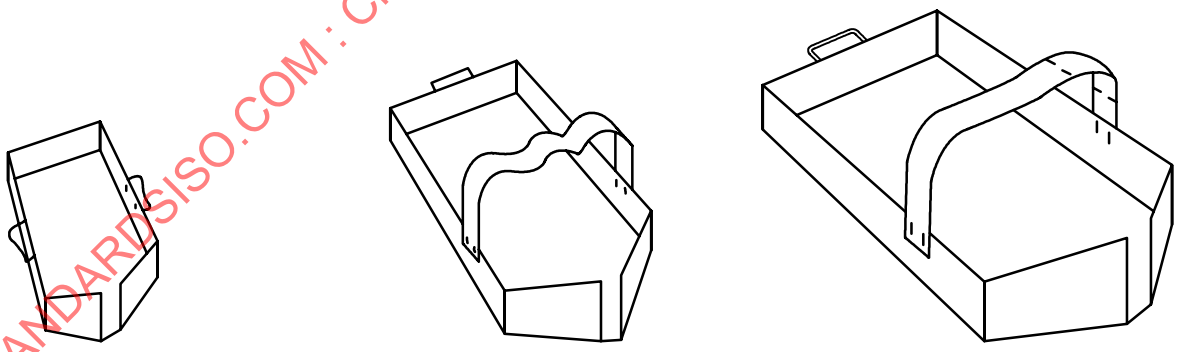


Figure 8 — Mixing trays

15.4 Division

15.4.1 Chemical analysis samples

Any of the following methods may be used, separately or in combination, for division of samples for chemical analysis:

a) rotary sample division;

b) cutter-type division;

c) increment division;

d) spear division;

e) fractional shovelling;

f) ribbon division;

g) stationary riffle division (except for dry concentrates).

Before using any of these methods, they shall be shown to be free of bias in the proposed application. For free-flowing concentrates, rotary sample division and increment division are the preferred methods of division.

NOTE Division by coning and quartering is not recommended.

15.4.2 Moisture samples

The following methods, used separately or in combination, are suitable for division of moisture samples.

a) spear division;

b) increment division.

If necessary, each sample shall first be mixed in its container to re-absorb any moisture that may have condensed on the inside walls of the container. The moisture sample shall then be divided as quickly as possible, minimizing exposure to the atmosphere.

15.4.3 Number of increments for division

The number of increments for division of preceding increments, subsamples and lot samples should be determined experimentally in accordance with 4.3. However, if no information is available on variance between the increments taken, the following numbers of increments may be used as a starting point:

a) for lot samples: a minimum of twenty increments;

b) for subsamples: a minimum of ten increments;

c) for individual increments: a minimum of four subsequent increments.

NOTE The provisions of this subclause do not apply to riffle division.

15.4.4 Minimum mass of divided sample

The minimum mass of divided sample for division of lot samples, subsamples and individual increments is specified in Table 6, subject to an absolute minimum of 200 g.

Table 6 — Minimum mass of divided sample for division of lot samples, subsamples and individual increments

Nominal top size of sample mm	Minimum mass of divided sample kg		
	Lot sample	Subsample	Individual increment
22,4	11,2	5,6	2,2
16	7,5	3,7	1,5
10	4,5	2,3	0,9
5	2,5	1,3	0,5
2,8	1,2	0,6	0,25
1,0	0,5	0,25	0,2
⩽ 250 µm	0,2	0,2	0,2

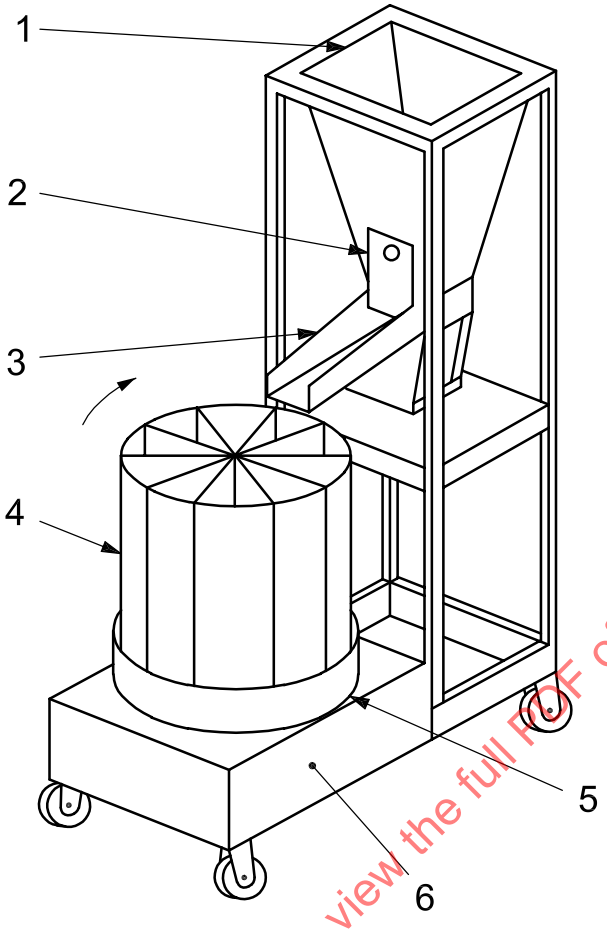
NOTE The minimum mass of divided lot samples, subsamples and individual increments is based on taking 20, 10 and 4 increments respectively using a scoop appropriate for the nominal top size of the sample as specified in Table H.1.

15.4.5 Rotary sample division

Rotary sample division is carried out using a rotary sample divider (see Figure 9) in which the bulk sample discharges from a feed hopper, either directly or over a vibrating or belt feeder into rotating sample containers. Alternatively, the containers are stationary and the sample discharge outlet rotates. The rotational speed shall be uniform and the linear speed where the cutter intercepts the stream shall not exceed 0,6 m/s. Dimensions depend upon parameters such as the mass of the sample and the required division ratio, but the cutter aperture where it intercepts the stream shall not be less than 30 mm.

The number of rotations of the sample carousel or sample discharge outlet shall conform to the requirements of 15.4.3, while the minimum mass of divided samples shall conform to the requirements of 15.4.4.

Rotary sample division is suitable for dividing samples for chemical analysis, but shall not be used for the division of moisture samples.



Key

1 feed hopper
2 slide gate
3 vibratory feeder
4 removable canisters
5 turntable
6 drive (enclosed)

Figure 9 — Example of a rotary sample divider

15.4.6 Cutter-type division

The procedure for cutter-type division is as follows.

a) Discharge the concentrate from a feed hopper onto a belt feeder.

b) Using a suitable falling-stream cutter conforming to the design principles of 8.1, 8.2, 8.3 and 8.4, take as a minimum the number of increments specified in 15.4.3.

c) If constant-mass division is being applied, the interval between increments shall be varied according to the mass of the lot sample, subsample or increment to be divided in accordance with the principles of 7.2.2. The first increment shall be taken at random within the first mass interval.

   If proportional division is being applied, the interval between increments shall be maintained constant, regardless of the mass of the lot sample, subsample or increment to be divided in accordance with the principles of 7.3.2. The first increment shall be taken at random within the first time interval.

The minimum mass of divided samples shall conform to the requirements of 15.4.4.

Cutter-type division is suitable for dividing samples for chemical analysis, but is unsuitable for dividing moisture samples.

15.4.7 Manual increment division

The procedure for manual increment division is as follows.

a) Spread the concentrate on a smooth clean surface in the form of a rectangle of uniform thickness as specified in Annex H, Table H.1.

b) Mark a matrix on the spread sample (see Figure 10), comprising four, ten or twenty parts as specified in 15.4.3.

c) Collect one increment of approximately equal mass from each part of the matrix with a flat-bottomed scoop chosen from Table H.1.

d) Insert a flat bump plate vertically through the spread concentrate until it comes into contact with the mixing surface. Then insert the scoop to the bottom of the spread concentrate, and take the increment by moving the scoop horizontally until its open end comes into contact with the bump plate, ensuring that all concentrate particles are collected off the top of the mixing surface.

e) Lift the scoop and bump plate together, so that the bump plate prevents concentrate from falling from the open end of the scoop.

The minimum mass of divided samples shall conform to the requirements of 15.4.4.

Manual increment division is suitable for division of both moisture and chemical analysis samples.

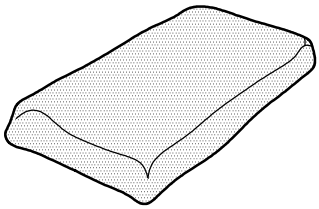
15.4.8 Spear division

Sample division using a spear is an acceptable method of division for obtaining the moisture sample or moisture test portions and for dividing samples for chemical analysis. It is not recommended for division of dry concentrates, because part of the increment is likely to be lost from the spear when it is withdrawn.

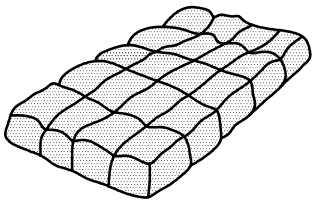
Prior to spear division, the concentrate shall be placed in a suitable container, which minimizes the surface area of concentrate exposed to the atmosphere, to reduce moisture losses. The diameter of the spear shall be 30 mm or, if agglomerates are present, at least three times the nominal top size of the concentrate, whichever is the greater. Care shall be taken to ensure that the full column of the concentrate is taken out and that no particles are lost when the implement is being extracted. Ensure that wet concentrate is not allowed to adhere to the outside of the implement when it is withdrawn, and that wet concentrate is not left adhering to the inside of the implement when removing the increment.

The number of increments for division of lot samples, subsamples and individual increments shall conform to the requirements of 15.4.3. The increments shall be taken from positions spaced as evenly as possible over the surface of the concentrate to be divided, so that the increments represent almost uniform masses of concentrate.

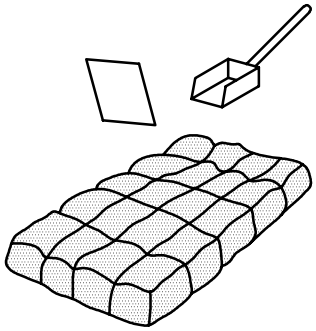
The minimum mass of divided samples shall conform to the requirements of 15.4.4.



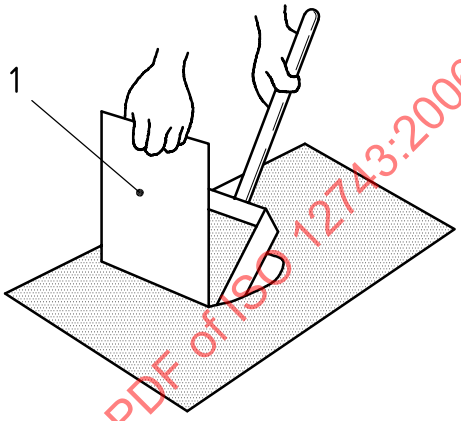
1) Spread the crushed gross sample into a rectangle with a thickness as specified in Table H.1



2) Arrange in 20 equal parts e.g. into 5 equal parts lengthwise and 4 equal parts breadthwise



3) Take a scoopful of sample at random from each of the 20 parts, by inserting the scoop to the bottom of the sample layer, and combine the 20 scoopfuls of sample into a divided sample



Key

1 bump plate

Outline of taking an increment by using a bump plate as shown in 3).

Figure 10 — Manual increment division (20 parts)

15.4.9 Fractional shovelling

The procedure for division by fractional shovelling (see Figure 11) is as follows.

a) Mix the concentrate and form a conical heap on a smooth clean surface.

b) Take successive shovelfuls from the base of the heap, working around the base, until the whole of the conical heap has been redistributed, by placing the shovelful on separate heaps. The number of heaps is determined by the division ratio but shall not exceed 20. For example, if a 1 in 5 division ratio is required, five heaps (N_1, N_2, N_3, N_4 and N_5) are formed as shown in Figure 11. The number of shovelfuls (i.e. increments) placed on each heap shall conform to the requirements of 15.4.3.

c) Select at random the heap to be retained.

The minimum mass of divided samples shall conform to the requirements of 15.4.4.

Fractional shovelling is suitable for dividing samples for chemical analysis, but is unsuitable for division of moisture samples.

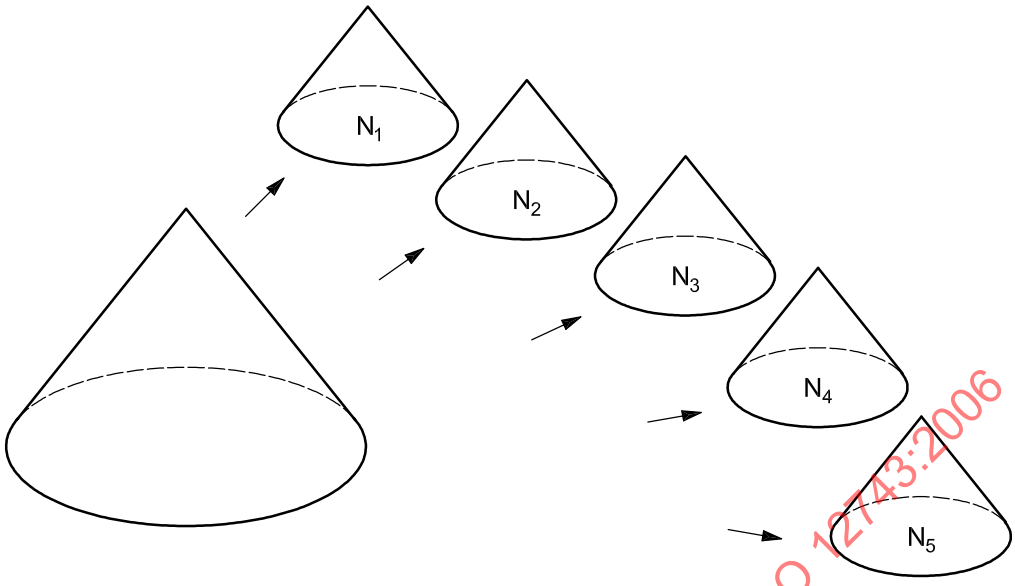


Figure 11 — Division by fractional shovelling

15.4.10 Ribbon division

The procedure for division using the ribbon method is as follows.

a) Spread out the concentrate without preliminary mixing in a linear chute constructed from removable sections (see Figure 12) to form a "ribbon" of length 1 m to 2 m, width 7 cm and approximate thickness 7 cm.

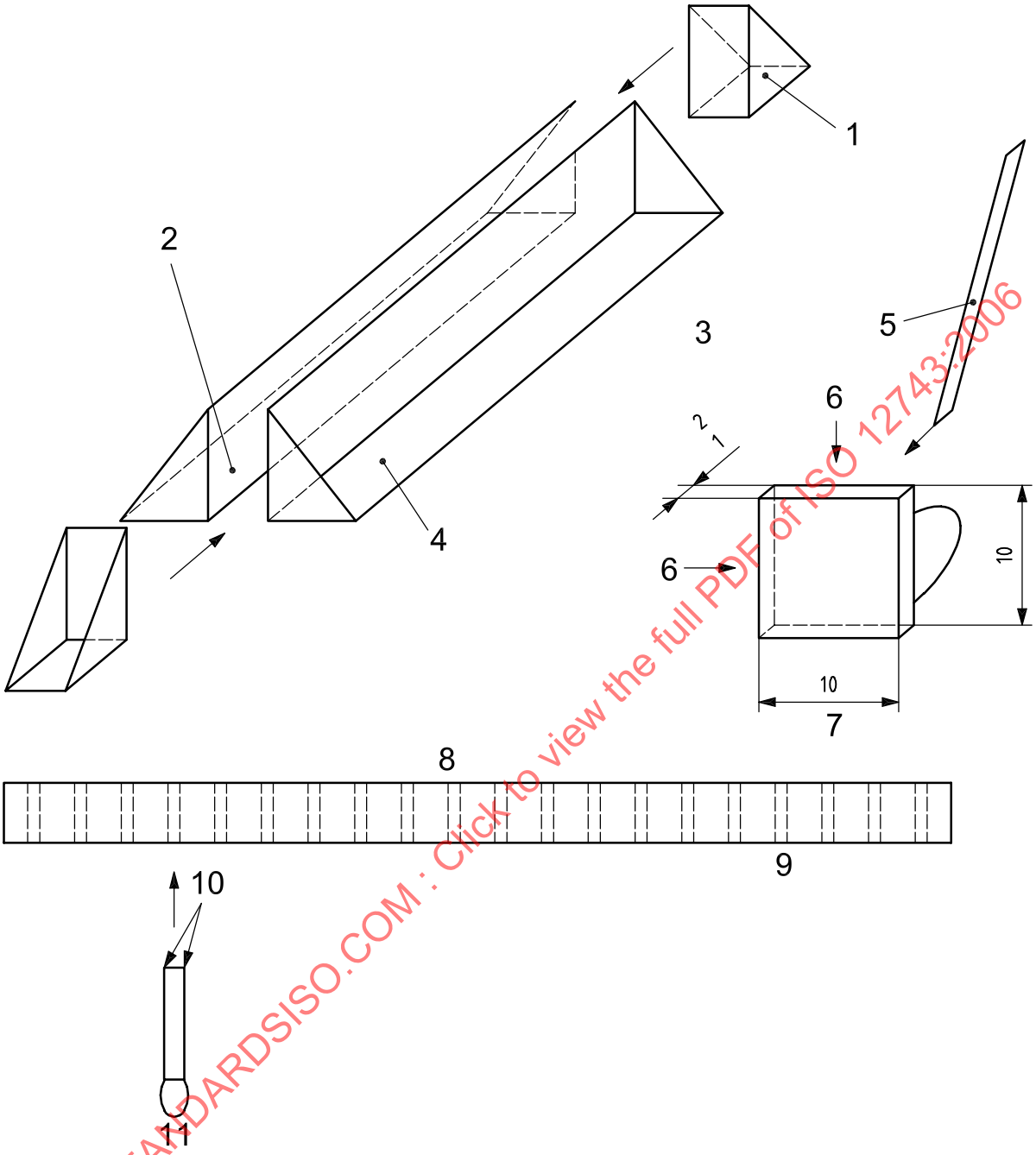
b) Remove the front section to gain access to the concentrate to be divided.

c) Using an increment scoop of 2 cm width, 10 cm depth and 10 cm height (see Figure 12), extract the number of increments specified in 15.4.3, equally spaced along the length of the ribbon. When taking each increment, the full cross-section of the ribbon shall be extracted.

The minimum mass of divided samples shall conform to the requirements of 15.4.4.

Ribbon division is suitable for division of chemical analysis samples, but is unsuitable for division of moisture samples or concentrates containing agglomerates.

Dimensions in centimetres



Key

1 side wedge (PVC)
2 back-corner wedge (PVC)
3 removal of front-corner wedge as soon as ribbon is formed
4 front-corner wedge (PV)
5 rod (stainless steel) shovel, if required
6 open face
7 sampling shovel (stainless steel)
8 ribbon of L = 1 m to 2 m
9 example with ni = 20 increments (view from above)
10 “front” bevelled edges
11 sampling scoop

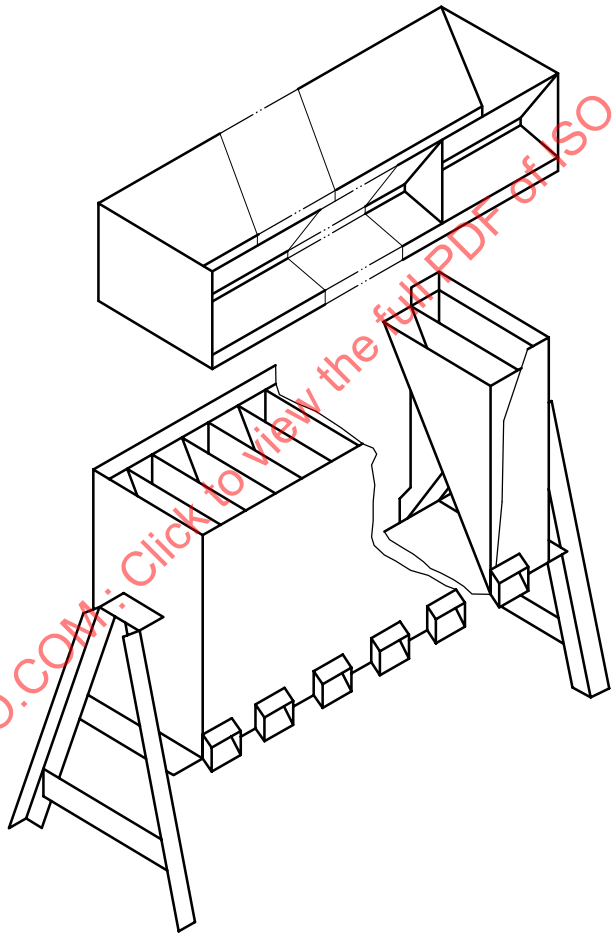
Figure 12 — Ribbon method of division

15.4.11 Riffle division

A riffle (see Figure 13) is a sample divider which is used to divide the concentrate fed into it into halves, one half being retained and the other rejected. It operates by allowing the concentrate to fall through a set of parallel slots of uniform width, adjacent slots feeding opposite containers.

A riffle shall be symmetrical (so that the sample may be taken from either side) and all surfaces on which the concentrate might rest should be inclined at not less than 60° to the horizontal. Receivers that fit closely against the body of the riffle are recommended to minimize dust loss. It is essential that the riffle used be appropriate for the nominal top size of the concentrate to be divided, because serious errors may be introduced if the slots are too small or there are too few.

The slot width shall be at least 10 mm or, if agglomerates are present, three times the nominal top size of the concentrate, whichever is the greater. There shall be at least eight slots for each half of the riffle.



a) Open riffle

Figure 13 — Examples of riffles

Riffling shall be carried out as follows.

a) Mix the concentrate and place it in the feed container.

b) Spread the concentrate in the feed container so that it is spread uniformly along the full length of the riffle.

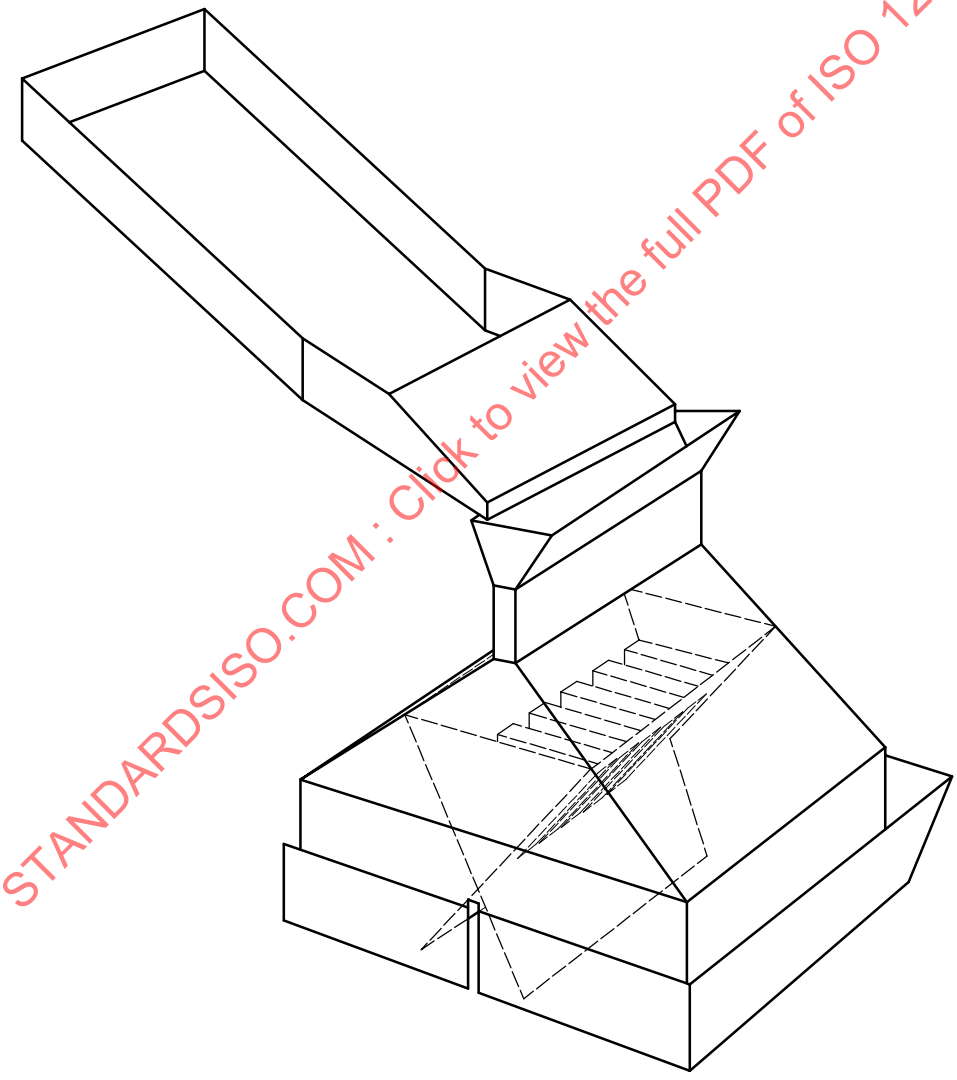
c) Pass the concentrate through the riffle and collect it in two receivers.

d) Retain the sample from one of the receivers, chosen at random.

e) If further subdivision is required, the retained sample may be passed through the riffle again.

The minimum mass of divided samples shall conform to the requirements of 15.4.4.

Riffles are recommended for division of free-flowing concentrates only. They are suitable for dividing samples for chemical analysis, but shall not be used for division of moisture samples.



b) Closed riffle

Figure 13 — Examples of riffles (*continued*)

16 Sample requirements

16.1 Moisture samples

16.1.1 Mass of test portion

The mass of each test portion shall be not less than 1 kg.

If agglomerates are present, it may be necessary to increase the mass of the test portion. For a nominal top size of 11,2 mm, 1 kg is sufficient. For nominal top sizes of 11,2 mm to 45 mm, the mass of the moisture test portion shall be increased to 2,5 kg.

16.1.2 Processing of samples

The number of test portions indicated in Table 7 shall be taken from unscreened subsamples or lot samples by one of the methods of division specified in 15.4 and weighed immediately. If this is not possible, the lot sample or subsample shall be stored in an impervious airtight container with a minimum of free air space to minimize any change of moisture content, but should be processed without delay. Some plastics are not impervious to moisture, so care shall be taken to ensure that suitable containers are used.

Moisture determination shall be carried out in accordance with ISO 10251, as soon as possible after the test portions have been taken. Crushing and/or screening of moisture samples is not acceptable, because it leads to moisture loss.

If separate samples are prepared for determination of moisture and metal content, both samples shall be of approximately the same mass and shall be dried in the same manner.

Table 7 — Minimum number of test portions for moisture determination

Type of sample	Number of test portions	Number of subsamples per lot
Lot sample	4	—
Subsample	2 1	2 - 3 ⩾ 4
Increment	1	—

16.2 Chemical analysis samples

Laboratory samples of typically 200 g for chemical analysis shall be taken from lot samples, subsamples or dried test portions used for moisture determination. These samples shall be heat sealed in plastic-lined aluminium pouches, or in a glass bottle having a tight-fitting lid. Paper bags or plastic bags are not suitable.

The nominal top size of the analysis sample shall be 150 µm. Smaller nominal top sizes may be used to improve precision, provided the additional grinding does not cause oxidation, decomposition, sublimation or smearing of the sample.

Where analysis for volatile elements such as mercury is required, the chemical analysis sample shall not be prepared from dried test portions used for moisture determination. A separate chemical analysis sample shall be prepared, ensuring that drying the sample is carried out at temperatures not exceeding 60 °C.

16.3 Physical test samples

Laboratory samples for physical testing include samples for particle-size determination, transportable-moisture limit and angle of repose. They should be stored in sealed containers. Any further treatment of the samples shall be as specified in the applicable test procedure.

17 Packing and marking of samples

Samples obtained for subsequent sample processing for testing, which is to be carried out remote from the sampling system, shall be placed in impervious containers. The relevant information shall be shown on the label and a card placed in the container. Examples of the information are as follows:

a) type, grade and identification of the lot (name of ship, number of train, etc.);

b) wet mass of the lot or sub-lot;

c) sample number or portion of lot and/or sub-lot the sample represents;

d) place, date and time of sampling;

e) special purpose or test for which the sample was taken.

Annex A
(normative)

Sampling stage method of estimating sampling and total variance

A.1 Components of sampling error and sampling variance

As shown by Gy[3], the total sampling error TSE can be broken up into a number of components corresponding to each sampling stage 1, 2, ..., i, ..., u as follows:

$$TSE_1 + ... + TSE_i + ... + TSE_u \quad \text{…(A.1)}$$

where

TSE_1 is the sampling error for stage 1;

TSE_i is the sampling error for stage i;

TSE_u is the sampling error for stage u, the last stage.

The above break-up is possible, because each component of sampling error is independent. The errors may be random or systematic (i.e. a bias).

Each sampling stage consists of two operations. These are selection (or sampling) and preparation. In this context, preparation is a non-selective operation involving operations such as crushing, drying, etc. Thus:

$$TSE = SE + PE \quad \text{…(A.2)}$$

where

SE is the selection error;

PE is the preparation error.

Typical preparation errors include sample contamination, sample loss, alteration of the chemical or physical composition of the sample and operator mistakes.

The selection error can be further broken up into the integration error CE and the materialization error ME as follows:

$$SE = CE + ME \quad \text{…(A.3)}$$

The integration error arises from the manner in which the sampling points are selected on the time or mass axes. The materialization error arises from the physical manner in which increments are taken, and can be eliminated by correct cutter design and operation.

The integration error also consists of two components caused by variations in quality and flow rate. Thus:

$$CE = QE + WE \quad \text{…(A.4)}$$

where

QE is the quality fluctuation error;

WE is the weighting error.

The quality fluctuation errors are of three types, namely short-range, long-range and periodic. Hence:

$$QE = QE_1 + QE_2 + QE_3 \quad \text{…(A.5)}$$

where

QE_1 is the short-range quality fluctuation error;

QE_2 is the long-range quality fluctuation error;

QE_3 is the periodic quality fluctuation error.

The short-range fluctuations result from two properties related to the particulate nature of the concentrate. These are the composition of the particles (fundamental error) and the manner in which the particles are grouped (segregation/grouping error). Thus:

$$QE_1 = FE + GE \quad \text{…(A.6)}$$

where

FE is the fundamental error;

GE is the segregation and grouping error.

The materialization error can be further broken up into the delimitation error DE and the extraction error EE as follows:

$$ME = DE + EE \quad \text{…(A.7)}$$

The delimitation error is eliminated if all parts of the concentrate stream are intercepted by the sample cutter for the same length of time. The extraction error is eliminated if the increment is completely extracted from the stream without any concentrate rebounding from the cutter.

Combining Equations A.2 to A.7 gives the following equation for the sampling error at each stage:

$$TSE = FE + GE + QE_2 + QE_3 + WE + DE + EE + PE \quad \text{…(A.8)}$$

The last three error components in Equation A.8, i.e. DE, EE and PE, are systematic errors, which introduce bias. They arise from not respecting the correct principles of sampling from the mechanical standpoint. They can be eliminated by using correct sampling practices, which are described in Clauses 8, 9, 10, and 11. Practical experience with concentrates also shows that the weighting error WE is negligible compared to QE_2, even when there are significant variations in flow rate. Likewise, the periodic quality fluctuation error QE_3 is also negligible, except in exceptional cases where production or stockpiling procedures introduce some periodicity. Hence, the equation for the total sampling error reduces to:

$$TSE = FE + GE + QE_2 \quad \text{…(A.9)}$$

or alternatively:

$$TSE = QE_1 + QE_2 \quad \text{…(A.10)}$$

These error components are random errors.

From Equations A.9 and A.10, it is clear that the variance of the total sampling error is given by:

$$s_S^2 = s_{FE}^2 + s_{GE}^2 + s_{QE_2}^2 \quad \text{…(A.11)}$$

where

s_S^2 is the total sampling variance;

s_{FE}^2 is the fundamental variance;

s_{GE}^2 is the segregation and grouping variance;

$s_{QE_2}^2$ is the long-range quality fluctuation variance.

or alternatively:

$$s_S^2 = s_{QE_1}^2 + s_{QE_2}^2 = s_{QE}^2 \quad \text{…(A.12)}$$

where

$s_{QE_1}^2$ is the short-range quality fluctuation variance;

s_{QE}^2 is the quality fluctuation variance.

In Equation A.12, the long-range quality fluctuation variance is often referred to as the distribution variance.

A.2 Estimation of fundamental variance

Gy[3] has shown that the variance of the fundamental error, s_{FE}^2, is given by:

$$s_{FE}^2 = \frac{C d^3 a^2}{m_S} \quad \text{…(A.13)}$$

where

C is the sampling constant for a given concentrate of given particle size and critical constituent;

d is the nominal top size of the concentrate, in centimetres;

m_S is the sample mass at a given sampling stage, in grams;

a is the fractional concentration of the constituent under consideration.

The sampling constant C is given by:

$$C = c\,l\,f\,g \quad \text{…(A.14)}$$

where

c is the mineralogical composition factor calculated in Equation (A.15);

l is the liberation factor;

f is the particle shape factor, which can usually be taken to be 0,5;

g is the size-range factor, usually between 0,25 and 1,0.

$l = \sqrt{(d_l / d)}$ when liberation is incomplete, d_l being the nominal top size at which complete liberation occurs; l = 1 when liberation is complete.

If d_l is unknown, a conservative assumption is to set: $d_l = d$.

The mineralogical composition factor is given by:

$$c = \frac{(1-a)\left[(1-a)\rho_1 + a\,\rho_2\right]}{a} \qquad \text{...(A.15)}$$

where

ρ_1 is the density of the particles of the critical component, in grams per cubic centimetre;

ρ_2 is the density of gangue particles, in grams per cubic centimetre.

The size-range factor g can be estimated from the ratio d / d' of the nominal top size d to the lower size limit d' (about 5 % undersize) as follows:

Large size range ($d / d' > 4$) g = 0,25

Medium size range ($2 \leqslant d / d' \leqslant 4$) g = 0,50

Small size range ($d / d' < 2$) g = 0,75

Uniform size (d / d' = 1) g = 1,00

Equation A.13 can be transposed to give the minimum sample mass required to achieve a given fundamental error variance as follows:

$$m_S = \frac{C\,d^3\,a^2}{s_{FE}^2} \qquad \text{...(A.16)}$$

EXAMPLE A.1 A zinc concentrate, having d = 150 µm (i.e. 0,015 cm), d_l = 50 µm and a large particle size range, is to be sampled. Assume that the mineral is ZnS with a particle density, ρ_1, of 5,0 gcm^{-3}, and that the gangue consists of silicates with a particle density, ρ_2, of 2,6 gcm^{-3}. Also, assume that the ZnS concentration is 50 % (i.e. a = 0,5) and that the fundamental error must not exceed 0,02 % Zn or 0,03 % ZnS (i.e. s_{FE} = 0,000 3).

$$c = \frac{(1-0{,}5)\left[(1-0{,}5)\times 5{,}0 + (0{,}5\times 2{,}6)\right]}{0{,}5} = 3{,}8$$

$$l = \sqrt{(50/150)} = 0{,}58$$

C = 3,8 × 0,58 × 0,5 × 0,25 = 0,28

Using Equation A.16, the minimum sample mass for a fundamental error s_{FE} of 0,000 3 is given by:

m_S = [0,28 × (0,015)^3 × (0,5)^2]/(0,000 3)^2 = 2,6 g

The mass exceeds the mass of test portion usually used for chemical analysis. However, if the sample is pulverized to a nominal top size of 75 µm, m_S is reduced to 0,45 g, thereby enabling test portions of 0,5 g to 1,0 g to be used for the above ZnS concentrate.

EXAMPLE A.2 A copper concentrate having d = 150 µm, d_l = 100 µm and a large particle size range is to be sampled. Assume that the mineral is $CuFeS_2$ with a particle density, ρ_1, of 4,2 gcm^{-3}, and that the gangue consists of silicates with a particle density, ρ_2, of 2,6 gcm^{-3}. Also, assume that the $CuFeS_2$ concentration is 90 % (i.e. a = 0,9) and that the fundamental error must not exceed 0,02 % Cu or 0,06 % $CuFeS_2$ (i.e. s_{FE} = 0,000 6).

$$c = \frac{(1-0,9)\left[(1-0,9)\times 4,2 + (0,9\times 2,6)\right]}{0,9} = 0,31$$

$$l = \sqrt{(100/150)} = 0,82$$

$$C = 0,31 \times 0,82 \times 0,5 \times 0,25 = 0,032$$

In this case, the minimum sample mass is given by:

$$m_S = [0,032 \times (0,015)^3 \times (0,9)^2]/(0,000\,6)^2 = 0,24 \text{ g}$$

It should be noted that round-robin testwork and replicate analyses provide the best estimates of the analytical variance s_A^2, which includes the variance due to selection of the test portion. The above calculations indicate whether the fundamental variance s_{FE}^2 is likely to be a major component of s_A^2 or not.

A fundamental characteristic of s_{FE}^2 is that it diminishes very quickly when d is reduced, and not so quickly when m_S is increased, but it can never be eliminated, no matter what crushing and homogenization procedures are used. However, for the usual fine flotation concentrates, the fundamental variance is negligible when the sample mass exceeds about 100 g.

A.3 Segregation and grouping variance

Gy[3] has shown that the segregation and grouping variance is either smaller or about the same magnitude as the fundamental variance. Consequently, it is always safe to assume that it is equal to the fundamental variance, in which case:

$$s_{QE_1}^2 = 2 s_{FE}^2 \qquad \text{...(A.17)}$$

A.4 Long-range quality fluctuation variance

The long-range quality fluctuation variance $s_{QE_2}^2$ can be estimated by extracting a large number of successive increments (say 30 to 50) at a given sampling stage and analysing them individually. There are two principal methods of analysing the resultant data.

A better method is to calculate the variogram, which examines the differences between increments at increasing intervals (called lags) apart. The variogram approach allows for serial correlation between increments, and enables the separate contributions of the variances $s_{QE_1}^2$ and $s_{QE_2}^2$ to be determined. However, the method is reasonably long and better suited to those wishing to fine tune their sampling scheme.

NOTE The interpenetrating sample method also takes into account the second term of the variogram.

The alternative method, which forms the basis of this International Standard, is a simplified approach involving calculation of the variance between increments s_b^2. However, unlike the variogram approach, the contributions of the variances $s_{QE_1}^2$ and $s_{QE_2}^2$ cannot be separated. Only the sampling variance s_S^2 can be determined.

The variance between increments s_b^2 can be estimated for a given sampling stage using the following equation:

$$s_b^2 = \frac{\sum_{j=1}^{n}\left(x_j - \bar{x}\right)^2}{n-1} - s_{PA}^2 \qquad \ldots(A.18)$$

where

x_j is the test result for increment j;

$\bar{x}$ is the mean test result for all increments;

n is the number of increments;

s_{PA}^2 is the variance of subsequent sample processing and analysis of each increment.

Thus, if n increments are taken for this sampling stage, the sampling variance s_S^2 for the sample obtained by combining all increments is given by:

$$s_S^2 = \frac{s_b^2}{n} \qquad \ldots(A.19)$$

Rearranging Equation A.19 enables the number of increments required to achieve a given sampling variance to be calculated as follows:

$$n = \frac{s_b^2}{s_S^2} \qquad \ldots(A.20)$$

Care must be taken when subtracting variances. The difference is significant only when the F ratio of the variances being subtracted is statistically significant.

A.5 Practical estimation of total variance

Using Equation A.19, the sampling variance $s_{S_i}^2$ for sampling stage "i" is given by:

$$s_{S_i}^2 = \frac{s_{b_i}^2}{n_i} \qquad \ldots(A.21)$$

Consequently, the sampling variance s_S^2 for all sampling stages (1 through to u-1) is given by:

$$s_S^2 = \sum_{i=1}^{u-1} \frac{s_{b_i}^2}{n_i} \qquad \ldots(A.22)$$

Now the total variance s_{T}^2 is given by:

$$s_{\mathrm{T}}^2 = s_{\mathrm{S}}^2 + \frac{s_{\mathrm{A}}^2}{r} \qquad \ldots(\mathrm{A.23})$$

where r is the number of replicate analyses.

Combining Equations A.22 and A.23 gives:

$$s_{\mathrm{T}}^2 = \sum_{i=1}^{u-1} \frac{s_{b_i}^2}{n_i} + \frac{s_A^2}{r} \qquad \ldots(\mathrm{A.24})$$

For a three-stage sampling scheme (including selection of the test portion), Equation A.24 reduces to:

$$s_{\mathrm{T}}^2 = \frac{s_{\mathrm{b}_1}^2}{n_1} + \frac{s_{\mathrm{b}_2}^2}{n_2} + \frac{s_{\mathrm{A}}^2}{r} \qquad \ldots(\mathrm{A.25})$$

The best way of reducing the value of s_{T}^2 to an acceptable level is to reduce the largest terms in Equation A.24 first. Clearly $s_{\mathrm{b}_i}^2 / n_i$ for a given sampling stage can be reduced by increasing the number of increments n_i or reducing $s_{\mathrm{b}_i}^2$ by homogenizing the concentrate prior to sampling. The last term can be reduced either by increasing the mass of the test portion, reducing the particle size prior to selecting the test portion, or performing replicate analyses. Selecting the optimum number of increments n_i for each sampling stage may require several iterations to obtain the required total variance s_{T}^2.

Annex B
(informative)

Estimation of total variance — Barge unloading using a grab

B.1 Definition of example

A barge containing 500 tonnes of zinc concentrate is unloaded using a grab of capacity 2 tonnes. For chemical analysis, primary increments of 0,5 kg are taken after every fifth grab from the fresh concentrate surface exposed in the barge by the grab, resulting in 50 primary increments. For moisture determination, separate increments of 0,5 kg are taken after every tenth grab, resulting in 25 primary increments.

Assume that the concentrate contains 42,87 % Zn, 830 g/t Ag and 6,45 % moisture. A four-stage sampling scheme is used for the chemical analysis sample (including selection of the test portion), and a two-stage scheme is used for the moisture sample. The parameters in Table B.1 were estimated using Equation (A.18) in A.4.

Table B.1 — Summary of increment and analytical standard deviations

Standard deviation	Mass fraction of Zn %	Mass fraction of Ag g/t	Moisture content %
s_{b_1}	0,15	25	0,24
s_{b_2}	0,10	15	—
s_{b_3}	Negligible	Negligible	—
s_A	0,06	8	0,05

NOTE The analytical standard deviations (s_A) were obtained from round-robin testwork, and hence include the sampling error due to selection of the test portion.

B.2 Estimation of total variance

B.2.1 Determination of mass fraction of metal

Stage 1

Primary increments of 0,5 kg mass are taken every 5 grabs.

$n_1 = 50$

Primary sample mass = 25 kg

Equation 5 gives:

a) Mass fraction of zinc

$s^2_{S_1} = 0{,}15^2/50 = 0{,}000\ 45$

b) Mass fraction of silver

$$s_{S_1}^2 = 25^2/50 = 12,5$$

Stage 2

The primary increments are combined and partially dried. After breaking up the agglomerates, the sample is divided to 2,8 kg by fractional shovelling, 20 secondary increments being placed on each of nine heaps.

n_2 = 20

Divided sample mass = 2,8 kg

a) Mass fraction of zinc

$$s_{S_2}^2 = 0,10^2/20 = 0,000\ 50$$

b) Mass fraction of silver

$$s_{S_2}^2 = 15^2/20 = 11,25$$

Stage 3

After drying and crushing to 0,5 mm particle size, the 2,8 kg sample is divided to 230 g using a rotary sample divider (RSD) and a large number of revolutions of the RSD.

n_3 = very large value

Divided sample mass = 230 g

$s_{S_3}^2$ = negligible value for mass fraction of both zinc and silver

Stage 4

The sample is pulverized to 150 µm and a single test portion of 2,5 g taken for analysis. The sampling variance, due to selection of the test portion, is included in the analytical variance.

a) Mass fraction of zinc

$$s_A^2 = 0,06^2 = 0,003\ 6$$

b) Mass fraction of silver

$$s_A^2 = 8^2 = 64$$

Total variance

Assuming that the analyses are carried out in quadruplicate, the total variance is given by:

$$s_T^2 = s_{S_1}^2 + s_{S_2}^2 + s_{S_3}^2 + \frac{s_A^2}{4}$$

a) Mass fraction of zinc

s_T^2 = 0,000 45 + 0,000 50 + negligible value + 0,003 6/4

= 0,001 85

Hence:

s_T = 0,04 % Zn

b) Mass fraction of silver

s_T^2 = 12,5 + 11,25 + negligible value + 64/4

= 39,75

Hence:

s_T = 6,3 g/t Ag

B.2.2 Determination of moisture content

Stage 1

Primary increments of 0,5 kg mass are taken every 10 grabs.

n_1 = 25

Primary sample mass = 12,5 kg

Equation 5 gives:

$s_{S_1}^2 = 0{,}24^2/25 = 0{,}002\ 3$

Stage 2

The primary increments are combined and the agglomerates are manually broken up. The sample is then quickly divided to about 1 kg, using manual increment division (20 secondary increments), to obtain test portions for moisture determination. The sampling variance, due to selection of the test portion, is included in the analytical variance.

$s_A^2 = 0{,}05^2 = 0{,}002\ 5$

Total variance

Assuming that moisture determination is carried out in duplicate, the total variance is given by:

$$s_T^2 = s_{S_1}^2 + \frac{s_A^2}{2}$$

= 0,002 3 + 0,002 5/2

= 0,0035 5

Hence:

s_T = 0,06 % H_2O

B.3 Summary

The estimated variances and standard deviations are summarized in Table B.2. For both zinc and silver determination, the major component of variance is due to analysis. If required, this could be reduced by carrying out more replicate analyses.

On the other hand, for moisture determination, the larger component of variance arises from the first sampling stage. If required, this could be reduced by taking more primary increments.

Table B.2 — Summary of estimated variances and standard deviations for barge unloading using a grab

Content (mass fraction)	**Stage 1** $s_{S_1}^2$	**Stage 2** $s_{S_2}^2$	**Stage 3** $s_{S_3}^2$	s_A^2 / r	s_T^2	s_T
Zinc 42,87 %	0,000 45	0,000 50	Negligible	0,000 90	0,001 85	0,04
Silver 830 g/t	12,5	11,25	Negligible	16	39,75	6,3
Moisture 6,45 %	0,002 3	—	—	0,001 25	0,003 55	0,06