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Copper, lead, zinc and nickel concentrates — Sampling of slurries

Concentrés de cuivre, de plomb, de zinc et de nickel — Échantillonnage des schlamms

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
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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 11794 was prepared by Technical Committee ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

Copper, lead, zinc and nickel concentrates — Sampling of slurries

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 particulate material that is mixed with a liquid, usually water, to form a slurry. In industry and in the mining and mineral processing literature, slurry is also referred to as pulp, but this term is not used in this International Standard. At very high ratios of fine particulate solids to liquids where material assumes a soft plastic form, the mixture is correctly termed as a paste. Sampling of pastes is not covered in this International Standard.

The procedures described in this International Standard apply to sampling of particulate materials that are transported in moving streams as slurries, but not pressurized slurries. These streams may fall freely or be confined in pipes, launders, flumes, sluices, spirals or similar channels. Sampling of slurries in stationary situations, such as a settled or even a well-stirred slurry in a holding vessel or dam, is not recommended and is not covered in this International Standard.

This International Standard describes procedures that are designed to provide samples representative of the slurry solids and particle-size distribution of the slurry under examination. After draining the slurry sample of fluid and measuring the fluid volume, damp samples of the contained particulate material in the slurry are available for drying (if required) and measurement of one or more characteristics in an unbiased manner and with a known degree of precision. The characteristics are measured by chemical analysis, physical testing or both.

The sampling methods described are applicable to slurries that require inspection to verify compliance with product specifications, determination of the value of a characteristic as a basis for settlement between trading partners or estimation of a set of average characteristics and variances that describes a system or procedure.

Provided that flow rates are not too high, the reference method against which other sampling procedures are compared is one where the entire stream is diverted into a vessel for a specified time or volume interval. This method corresponds to the stopped-belt method described in ISO 12743.

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 12743, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

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*

ISO 20212, *Copper, lead, zinc and nickel sulfides — Sampling procedures for ores and smelter residues*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 12743, ISO 12744, ISO 13292 and ISO 20212 apply.

4 Principles of sampling slurries

4.1 General

In this International Standard, a slurry is defined as “any fluid mixture of a solid of nominal top size < 1 mm that is mixed with water, which is frequently used as a convenient form to handle solids in bulk”. Slurry flows are found in many mineral processing plants, with the water and entrained solids mixture being transported through the plant circuits by means of pumps and pipelines and under gravity in sluices, flumes and launders. In a number of operations, ore is transported to the mill in slurry form, and in others concentrates are transported long distances in slurry pipelines. Tailings from wet plants are also discharged as slurries through pipelines to the tailings dam. In many of these operations, collection of increments at selected sample points is required for evaluation of the particulate material in the slurry.

A lot sample is constituted from a set of unbiased primary increments from a lot. The sample container is weighed immediately after collection and combination of increments to avoid water loss by evaporation or spillage. Weighing is necessary to determine the percentage of solids by mass in the slurry sample. The sample may then be filtered, dried and weighed. Alternatively, the sample may be sealed in plastic bags after filtering for transport and drying at a later stage. The liquid removed during filtration should be retained if it needs to be analysed.

Test samples are prepared from samples after filtering and drying. Test portions may then be taken from the test sample and analysed using an appropriate and properly calibrated analytical method or test procedure under prescribed conditions.

The objective of the measurement chain is to determine the characteristic of interest in an unbiased manner with an acceptable and affordable degree of precision. The general sampling theory, which is based on the additive property of variances, can be used to determine how the variances of sampling, sample preparation and chemical analysis or physical testing propagate and hence determine the total variance for the measurement chain. This sampling theory can also be used to optimize manual sampling methods and mechanical sampling systems.

If a sampling scheme is to provide representative samples, all parts of the slurry in the lot must have an equal opportunity of being selected and appearing in the sample for testing. Hence, slurries are to be sampled in such a manner that all possible primary increments in the set into which the slurry can be divided have the same probability of being selected. Any deviation from this basic requirement can result in bias. A sampling scheme having incorrect selection techniques, i.e. with non-uniform selection probabilities, cannot be relied upon to provide representative samples.

Sampling of slurries should preferably be carried out by systematic sampling on a time basis (see Clause 9). If the slurry flow rate and the solids concentration vary with time, the slurry volume and the mass of dry solids for each increment will vary accordingly. It needs to be shown that no systematic error (bias) is introduced by periodic variation in quality or quantity, where the proposed sampling interval is approximately equal to a multiple of the period of variation in quantity or quality. Otherwise, stratified random sampling should be used (see Clause 10).

Best practice for sampling slurries is to cut freely falling streams mechanically (see Clause 11), with a complete cross-section of the stream being taken during the traverse of the cutter. Access to freely falling streams can sometimes be engineered at the end of pipes or, alternatively, a full-stream sample by-line can be added to a pipe that diverts the slurry into a holding tank, or weirs can be incorporated in launders, sluices and flumes. If samples are not collected in this manner, non-uniform concentration of solids in the slurry due to segregation and stratification of the solids may lead to bias in the sample that is collected. Slurry flow in pipes can be homogeneous with very fine particles, such as clays, dispersed uniformly in turbulent suspension along the length and across the diameter of the pipe. However, more commonly, the slurry in a pipe will have significant particle concentration gradients across the pipe and there may be particle concentration fluctuations along the length of the pipe. These common conditions are called heterogeneous flow. Examples of such flow are full-pipe flow of a heterogeneous suspension, or partial-pipe flow of a fine particle suspension above a slower moving or even stationary bed of coarser particles in the slurry.

For heterogeneous flow, bias is likely to occur where a tapping is made into the slurry pipe to locate either a flush-fitting sample take-off pipe or a sample tube projecting into the slurry stream for extraction of samples. The bias is caused by non-uniform radial concentration profiles in the pipe and the different trajectories followed by particles of different masses due to their inertia, resulting in larger or denser particles being preferentially rejected from, or included in, the sample.

In slurry channels such as launders, heterogeneous flow is almost always present, and this non-uniformity in particle concentration is usually preserved in the discharge over a weir or step. However, sampling at a weir or step allows complete access to the full width and breadth of the stream, thereby enabling all parts of the slurry stream to be collected with equal probability.

Sampling of slurries in stationary situations, such as a settled or even a well-stirred slurry in a tank, holding vessel or dam, is not recommended, because it is virtually impossible to ensure that all parts of the slurry in the lot have an equal opportunity of being selected and appearing in the lot sample for testing. Instead, sampling should be carried out from moving streams, as the tank, vessel or dam is filled or emptied.

4.2 Sampling errors

4.2.1 General

The processes of sampling, sample preparation and measurement are experimental procedures, and each procedure has its own uncertainty appearing as variations in the final results. Where the average of these variations is close to zero, they are called random errors. More serious variations contributing to the uncertainty of results are systematic errors, which have averages biased away from zero. There are also human errors that introduce variations due to departures from prescribed procedures for which statistical analysis procedures are not applicable.

The characteristics of the solids component of a slurry can be determined by extracting samples from the slurry stream, preparing test samples and measuring the required quality characteristics. The total sampling error TSE can be expressed as the sum of a number of independent components (Gy, 1992; Pitard, 1993). Such a simple additive combination would not be possible if the components were correlated. The sampling error, expressed as a sum of its components, is given by Equation (1):

$$\mathrm{TSE} = \mathrm{QE}_1 + \mathrm{QE}_2 + \mathrm{QE}_3 + \mathrm{WE} + \mathrm{DE} + \mathrm{EE} + \mathrm{PE} \qquad (1)$$

where

QE_1 is the short-range quality-fluctuation error associated with short-range variations in quality of the solids component of the slurry;

QE_2 is the long-range quality-fluctuation error associated with long-range variations in quality of the solids component of the slurry;

QE_3 is the periodic quality-fluctuation error associated with periodic variations in quality of the solids component of the slurry;

WE is the weighting error associated with variations in the slurry flow rate;

DE is the increment delimitation error introduced by incorrect increment delimitation;

EE is the increment extraction error introduced by incorrect increment extraction from the slurry;

PE is the preparation error (also known as accessory error) introduced by departures (usually unintentional) from correct practices, e.g. during constitution of the lot sample, draining and filtering away the water, and transportation and drying of the sample

The short-range quality-fluctuation error consists of two components, as shown by Equation (2):

$$\mathrm{QE}_1 = \mathrm{FE} + \mathrm{GE} \tag{2}$$

where

FE is the fundamental error due to variation in quality between particles;

GE is the segregation and grouping error.

The fundamental error results from the composition heterogeneity of the lot, i.e. the heterogeneity that is inherent to the composition of each particle making up the solids component of the lot. The greater the differences in the compositions of particles, the greater the composition heterogeneity and the higher the fundamental error variance. The fundamental error can never be completely eliminated. It is an inherent error resulting from the variation in composition of the particles in the slurry being sampled.

The segregation and grouping error results from the distribution heterogeneity of the sampled material (Pitard, 1993). The distribution heterogeneity of a lot is the heterogeneity arising from the manner in which particles are distributed in the slurry. It can be reduced by taking a greater number of smaller increments, but it can never be completely eliminated.

A number of the components of the total sampling error, namely DE, EE and PE, can be minimized, or reduced to an acceptable level, by correct design of the sampling procedure.

4.2.2 Preparation error, PE

In this context, the preparation error includes errors associated with non-selective sample-preparation operations that should not change mass, such as sample transfer, draining and filtering, drying, crushing, grinding or mixing. It does not include errors associated with sample division. Preparation errors, also known as accessory errors, include sample contamination, loss of sample material, alteration of the chemical or physical composition of the sample, operator mistakes, fraud or sabotage. These errors can be made negligible by correct design of the sampling system and by staff training. For example, cross-stream slurry cutters should have caps to prevent entry of splashes when the cutter is in the parked position, and care needs to be taken during filtering to avoid loss of fines that are still suspended in the water to be discarded.

4.2.3 Delimitation and extraction errors, DE and EE

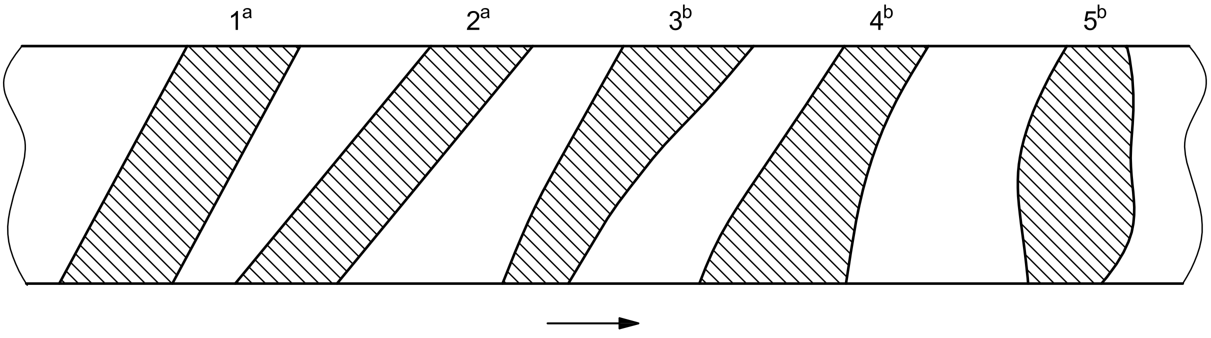
Delimitation and extraction errors arise from incorrect sample cutter design and operation. The increment delimitation error, DE, results from an incorrect shape of the volume delimiting the slurry increment, and this can be due to both design and operation faults. Because of the incorrect shape of the slurry increment volume, sampling with non-uniform selection probabilities results. The average of DE is often non-zero, which makes it a source of sampling bias. The delimitation error can be made negligible if all parts of the stream cross-section are diverted by the sample cutter for the same length of time.

Sampling from moving slurry streams usually involves methods that fall into three broad operational categories as follows (Pitard, 1993).

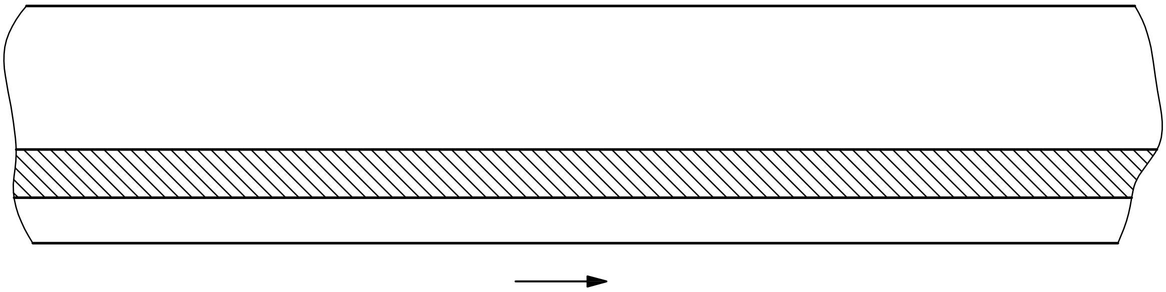
a) Taking the whole stream for part of the time with a cross-stream cutter as shown in Figure 1 a) (after Pitard, 1993), usually where the slurry falls from a pipe or over a weir or step. Cuts 1 and 2 show correct sampling with the cutter diverting all parts of the stream for the same length of time. Cuts 3 to 5 show incorrect sampling where the cutter diverts different parts of the stream for different lengths of time.

b) Taking part of the stream all of the time as shown in Figure 1 b) (after Pitard, 1993) with an on-stream point sampler or probe within a pipe or channel, which is always incorrect.

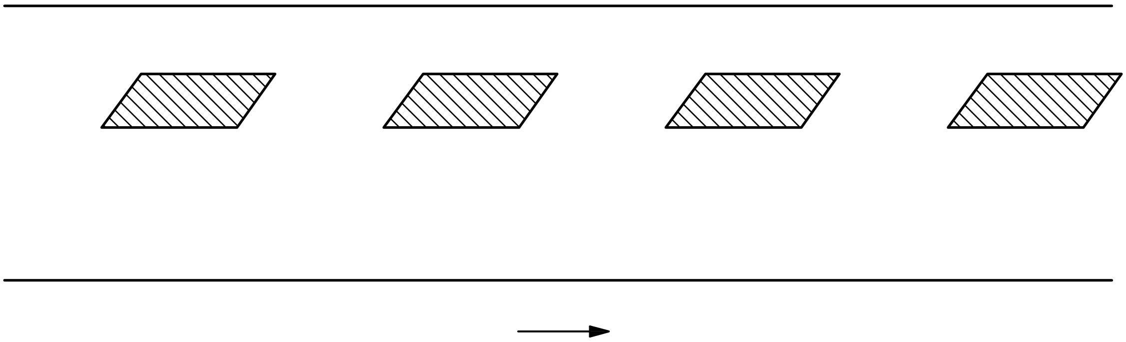
c) Taking part of the stream part of the time as shown in Figure 1 c) (after Pitard, 1993), also with an on-stream point sampler or probe within a pipe or channel, which is always incorrect.



a) Taking all of the stream for part of the time



b) Taking all of the stream for all of the time (always incorrect)



c) Taking all of the stream for part of the time (always incorrect)

Key

[a] Correct.

[b] Incorrect.

Figure 1 — Plan view of volumes diverted by a slurry cutter

The increment extraction error, EE, results from incorrect extraction of the slurry increment. The extraction is said to be correct if, and only if, all particles in the slurry that have their centre of gravity inside the boundaries of the correctly delimited increment are extracted. The average of EE is often non-zero, which makes it a source of sampling bias. The extraction error can be made negligible by ensuring that the slurry increment is completely extracted from the stream without any particulate material being lost from the cutter in splashes or slops. The depth and capacity of the cutter must be sufficient to avoid slurry reflux from the cutter aperture, resulting in loss of part of the extracted slurry increment.

4.2.4 Weighting error, WE

The weighting error is an error component arising from the selection model underlying Equation (1). In the model, the time-dependent flow rate of the solids in the slurry stream is a weighting function applied to the corresponding time-dependent quality characteristic over time, which gives the weighted average quality characteristic of the solids component of the lot. The weighting error results from the application of incorrect weights to the quality characteristics. The best solution to reducing the weighting error is to stabilize the flow rate. As a general rule, the weighting error is negligible for variations in flow rate up to 10 %, and acceptable for variations in flow rate up to 20 %.

4.2.5 Periodic quality-fluctuation error, QE_3

Periodic quality-fluctuation errors result from periodic variations in quality generated by some equipment used for slurry processing and transportation, e.g. grinding and screening circuits, splitters and pumps. In such cases, stratified random sampling should be carried out as discussed in Clause 10. The alternative is to reduce the source of periodic variations in quality significantly, which may require plant redesign.

4.3 Sampling and total variance

4.3.1 Sampling variance

Assume that the weighting error (WE*)*, increment delimitation error (DE), increment extraction error (EE) and preparation error (PE) described in 4.2 have been eliminated or reduced to insignificant values by careful design and sampling practice. In addition, assume that periodic variations in quality have been eliminated and that the flow rate has been regulated. The sampling error in Equation (1) then reduces to the form:

$$\mathrm{TSE} = \mathrm{QE}_1 + \mathrm{QE}_2 \quad (3)$$

Hence, the sampling variance $\left(s_{\mathrm{S}}^2\right)$ is given by:

$$s_{\mathrm{S}}^2 = s_{\mathrm{QE}1}^2 + s_{\mathrm{QE}2}^2 \quad (4)$$

The short-range quality-fluctuation variance, $s_{\mathrm{QE}1}^2$, arises from the different internal composition of increments taken at the shortest possible interval apart. This is a local or random variance due to the particulate nature of the solids in the slurry.

The long-range quality-fluctuation variance, $s_{\mathrm{QE}2}^2$, arises from the continuous trends in quality that occur while sampling a slurry and is usually space- and time-dependent. This component is often the combination of a number of trends generated by diverse causes.

4.3.2 Total variance

Assuming that sources of bias have either been eliminated or minimized, the next objective of a sampling scheme is to provide one or more test portions, sufficiently representative of a lot, for determination of the quality characteristics of the lot with good precision, i.e. low variance. 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, determination of particle-size distribution, etc.) as follows:

$$s_{\mathrm{T}}^2 = s_{\mathrm{S}}^2 + s_{\mathrm{A}}^2 \tag{5}$$

where

s_{S}^2 is the sampling variance (including sample processing);

s_{A}^2 is the analytical variance.

In Equation (5), 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:

$$s_{\mathrm{T}}^2 = s_{\mathrm{S}}^2 + \frac{s_{\mathrm{A}}^2}{r} \tag{6}$$

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, is to break up the sampling variance into its components for each sampling stage (see Annex A). The total variance is then given by:

$$s_{\mathrm{T}}^2 = s_{\mathrm{S}_1}^2 + ... + s_{\mathrm{S}_i}^2 + s_{\mathrm{S}_{u-1}}^2 + \frac{s_{\mathrm{A}}^2}{r} \tag{7}$$

where

$s_{\mathrm{S}_1}^2$ is the sampling variance for stage 1, i.e. the primary sampling variance;

$s_{\mathrm{S}_i}^2$ is the sampling variance for stage i;

$s_{\mathrm{S}_{u-1}}^2$ 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 4.3.3) and provides very detailed information on the variance components that 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 4.3.4), is to break up the total variance into primary sampling, sample processing and analytical variances only as follows:

$$s_{\mathrm{T}}^2 = s_{\mathrm{S}_1}^2 + s_{\mathrm{P}}^2 + \frac{s_{\mathrm{A}}^2}{r} \tag{8}$$

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 (7)].

The primary sampling variance is identical to the sampling variance for stage 1 in Equation (7), 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 (8) 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 interleaved duplicate samples (see 4.3.5). 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, 1986). 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 is 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.

4.3.3 Sampling-stage method of estimating sampling and total variance

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

$$s_{\mathrm{S}_i}^2 = \frac{s_{\mathrm{b}_i}^2}{n_i} \tag{9}$$

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:

$$s_{\mathrm{b}_i}^2 = \frac{\sum_{j=1}^{n}\left(x_j \bar{x}\right)^2}{n_i - 1} - s_{\mathrm{PA}}^2 \tag{10}$$

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 (10) to obtain an unbiased estimate of $s_{\mathrm{b}_i}^2$.

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:

$$s_\mathrm{S}^2 = \sum_{i=1}^{u-1} \frac{s_{\mathrm{b}_i}^2}{n_i} \tag{11}$$

Combining Equations (6) and (11) gives the total variance s_T^2 as follows:

$$s_\mathrm{T}^2 = \sum_{i=1}^{u-1} \frac{s_{\mathrm{b}_i}^2}{n_i} + \frac{s_\mathrm{A}^2}{r} \tag{12}$$

For a three-stage sampling scheme (including selection of the test portion), Equation (12) reduces to:

$$s_\mathrm{T}^2 = \frac{s_{\mathrm{b}_1}^2}{n_1} + \frac{s_{\mathrm{b}_2}^2}{n_2} + \frac{s_\mathrm{A}^2}{r} \tag{13}$$

The best way of reducing the value of s_T^2 to an acceptable level is to reduce the largest terms in Equation (12) 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 slurry 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.

4.3.4 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 (11), the primary sampling variance $s_{\mathrm{S}_1}^2$ is given by:

$$s_{\mathrm{S}_1}^2 = \frac{s_{\mathrm{b}_1}^2}{n_1} \tag{14}$$

where

n_1 is the number of primary increments;

$s_{\mathrm{b}_1}^2$ is the variance between primary increments determined using Equation (10).

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

The sampling processing variance s_P^2 and analytical variance s_A^2 are determined experimentally by duplicate sample processing and determination of quality characteristics in accordance with ISO 12744. The analytical variance s_A^2 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 (8) and (14) gives the following:

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

$$s_{\mathrm{T}}^2 = \frac{s_{\mathrm{b}_1}^2}{n_1} + s_{\mathrm{P}}^2 + \frac{s_{\mathrm{A}}^2}{r} \tag{15}$$

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:

$$s_{\mathrm{T}}^2 = \frac{s_{\mathrm{b}_1}^2}{n_1} + \frac{s_{\mathrm{P}}^2}{k} + \frac{s_{\mathrm{A}}^2}{rk} \tag{16}$$

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

$$s_{\mathrm{T}}^2 = \frac{s_{\mathrm{b}_1}^2 + s_{\mathrm{P}}^2 + \frac{s_{\mathrm{A}}^2}{r}}{n_1} \tag{17}$$

4.3.5 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 2. 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:

$$s_{\mathrm{T}}^2 = \frac{\pi}{4}\left[\frac{\sum_{i=1}^{N}\left|x_{\mathrm{A}_i} - x_{\mathrm{B}_i}\right|}{N}\right]^2 \tag{18}$$

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 pairs (in the range 10 to 20);

π/4 is a statistical factor relating range to variance for a pair of measurements.

The total variance is obtained with a minimum of extra effort, provided faculties are available for collecting interleaved duplicate samples.

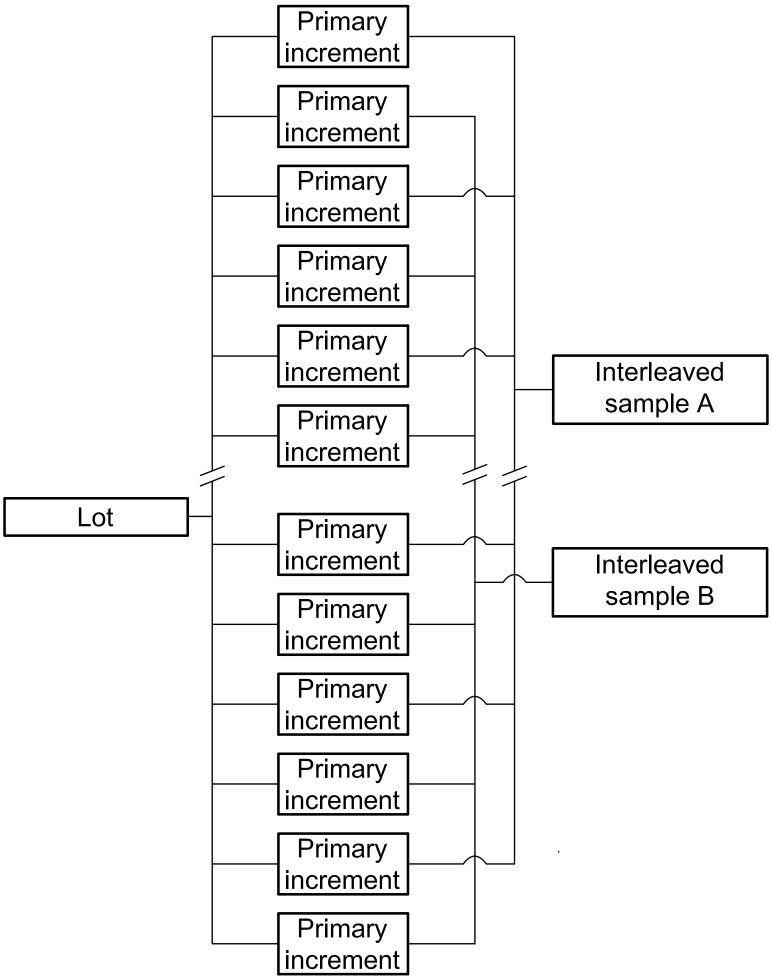


Figure 2 — Example of a plan for interleaved duplicate sampling

5 Establishing a sampling scheme

Most sampling operations are routine and are carried out to determine the average quality characteristics of a lot as well as variations in quality characteristics between sub-lots and lots for monitoring and controlling quality. In establishing a sampling scheme for routine sampling so that the required precision for a lot can be obtained, it is necessary to carry out the following sequence of steps. This sequence includes experimental procedures, such as step f) below, that are non-routine and carried out infrequently, e.g. determining the variance between increments, particularly if a significant change has occurred to the slurry source or to the sampling equipment. The procedure is as follows.

a) Define the purpose for which the samples are being taken. Sampling for commercial transactions is usually the main purpose of sampling standards. However, the procedures described in this International Standard are equally applicable to monitoring plant performance, process control and metallurgical accounting.

b) Define the lot by specifying the duration of slurry flow, e.g. one day of operation.

c) Identify the quality characteristics to be measured and specify the overall precision (combined precision of sampling, sample preparation and measurement) required for each quality characteristic.

d) Ascertain the nominal top size and particle density of the solids in the slurry for determining the minimum mass of solids in the lot sample (see Clause 8).

e) Check that the procedures and equipment for taking slurry increments avoid bias (see Clause 6).

f) Determine the variance between increments of the quality characteristics under consideration and the variance of preparation and measurement (see Clause 4).

g) Determine the number of increments to be taken from the lot and the number of sub-lots required to achieve the desired precision (see Clause 7). If the required precision results in impractical numbers of increments and sub-lots, it may be necessary to adopt a poorer precision.

h) Determine the sampling interval, in minutes, for time-basis systematic sampling (see Clause 9) or stratified random sampling within fixed time intervals (see Clause 10).

i) Take slurry increments at the intervals determined in step h) during the whole period of handling the lot.

During sampling operations, sub-lot samples are usually combined to constitute a single lot sample for analysis (see Figure 3). To improve the overall precision of the measured quality characteristics of the lot, increments taken from sub-lots may be used to constitute sub-lot samples for analysis (see Figure 4). Other reasons for separate preparation and analysis of sub-lot samples are

a) for convenience of materials handling,

b) to provide progressive information on the quality of the lot, or

c) to provide reference or reserve samples after division.

Each increment may also be analysed separately (see Figure 5) to assess the variability of the quality characteristics of the lot. In addition, it is recommended that the precision achieved in practice be checked on an ongoing basis by duplicate sampling where alternate increments are diverted to lot samples A and B (see Figure 2) from which two test samples are prepared and analysed. A substantial number (greater than 15) of data pairs are required to obtain a reliable estimate of precision.

In most situations, the solids in the slurry increment will not need to be crushed or pulverized to allow further division, since most slurries contain only fine particulate material. However, if the particles are coarse and particle-size reduction is required to allow further division, it is necessary to recalculate the minimum sample mass for the lot using the new nominal top size of the crushed solids (see Clause 8).

The initial design of a sampling scheme for a new plant or a slurry having unfamiliar characteristics should, wherever possible, be based on experience with similar handling plants and material types. Alternatively, a substantial number of increments, e.g. 100, can be taken and used to determine the quality variation of the contained solids, but the precision of sampling cannot be determined *a priori.*

Sampling of slurries from stationary situations, such as storage tanks, mixer tanks and tailing dams, is not recommended and is not covered in this International Standard.

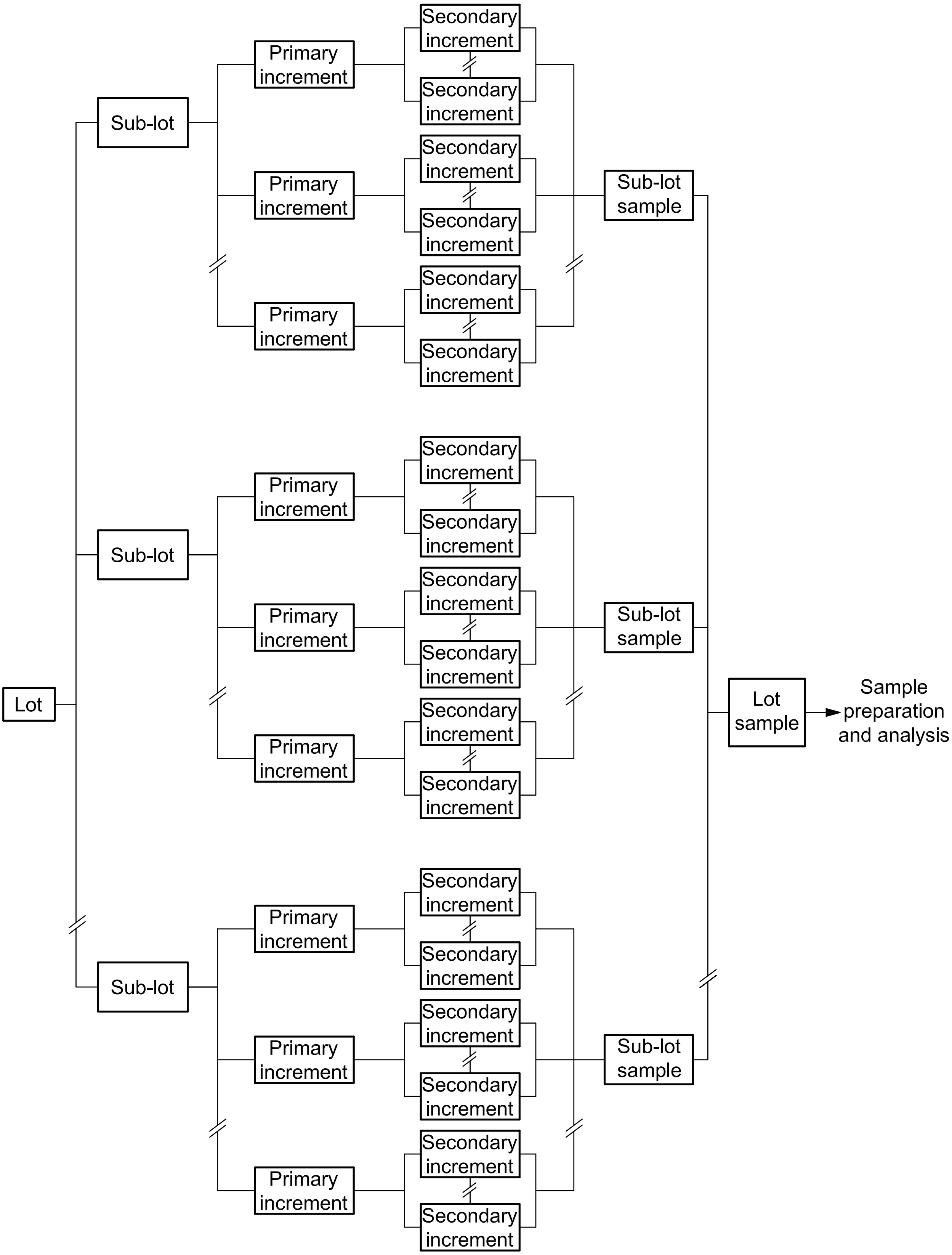


Figure 3 — Example of a sampling plan where a single lot sample is constituted for analysis

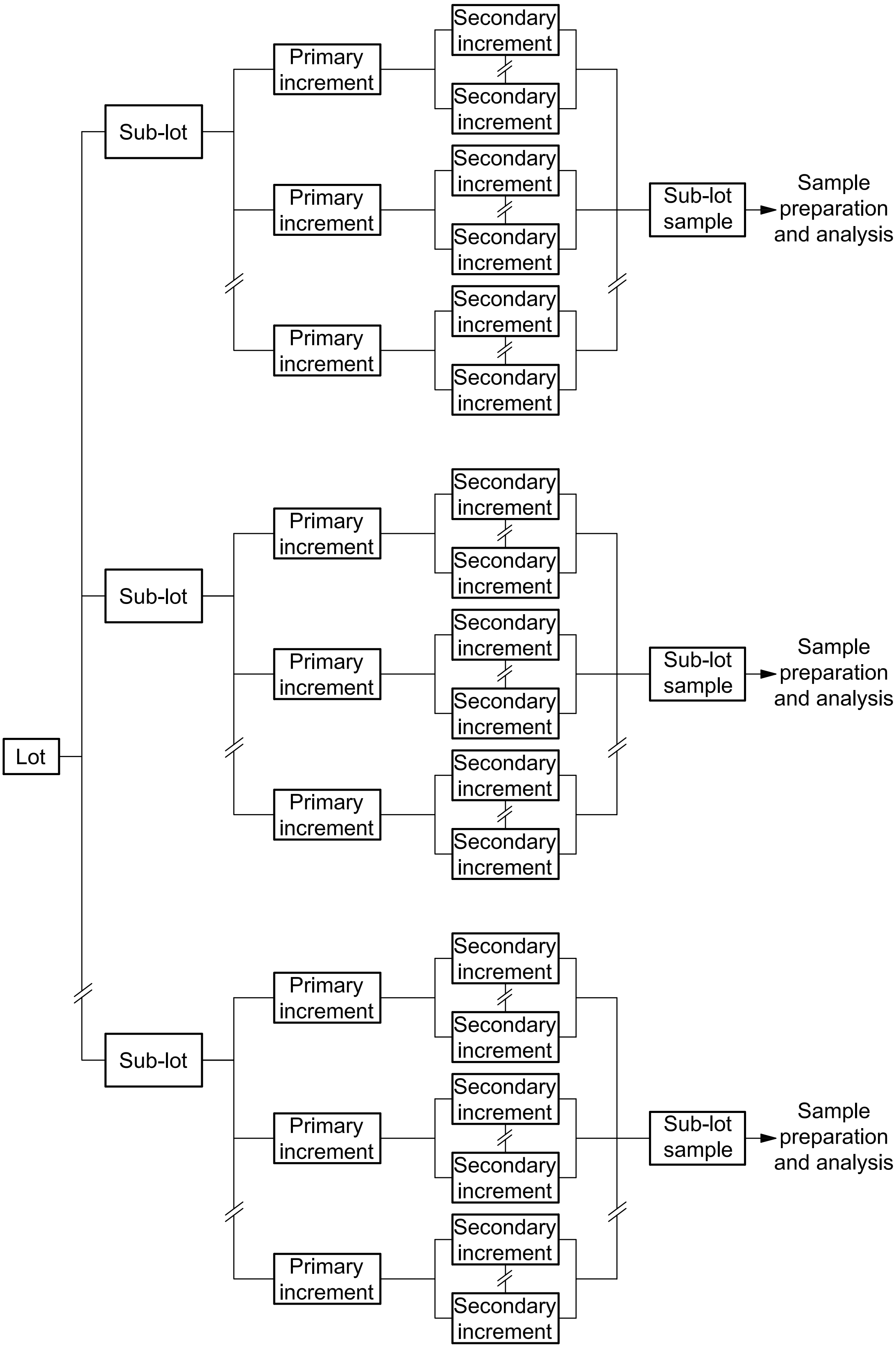


Figure 4 — Example of a sampling plan with each sub-lot sample analysed separately

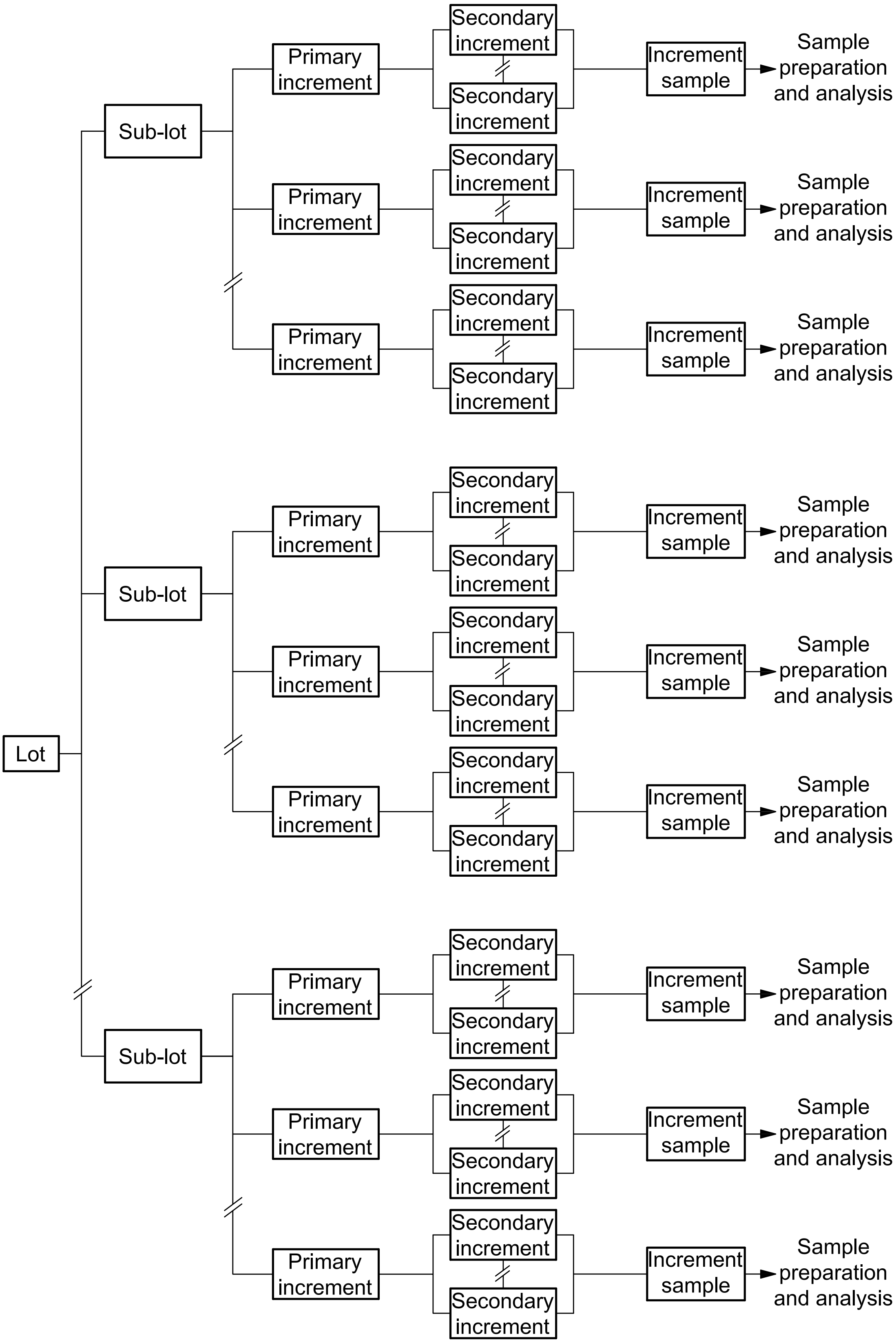


Figure 5 — Example of a sampling plan with each increment analysed separately

6 Minimization of bias and unbiased increment mass

6.1 Minimization of bias

Minimization of bias in sampling and sample preparation is vitally important. Unlike precision, which can be improved by collecting more slurry increments, preparing more test samples or assaying more test portions, bias cannot be reduced by replication. Consequently, sources of bias should be minimized or eliminated at the outset by correct design of the sampling and sample-preparation system. The minimization or elimination of possible bias should be regarded as more important than improvement of precision.

Sources of bias that can be eliminated include sample spillage, sample contamination and incorrect extraction of increments, while a bias source that cannot be fully eliminated is that arising from variable settling rates of particles with different size and density during sample division prior to filtration.

The guiding principle to be followed is that it is essential that increments be extracted from the lot in such a manner that all parts of the particulate bulk material in the slurry have an equal opportunity of being selected and becoming part of the test sample that is used for chemical or physical testing, irrespective of the size, mass or density of individual particles. In practice, this means that a complete cross-section of the slurry must be taken when sampling from a moving stream.

The requirement of equal-selection probabilities should be borne in mind when designing a sampling system. Examples of practical rules that follow from this principle are as follows.

a) A complete cross-section of the slurry stream should be taken when sampling from a moving stream.

b) There should be no sample spillage.

c) The cutter aperture should be at least three times the nominal top size of the particles in the slurry, subject to a minimum of 10 mm.

d) The cutter slot length should be at least 5 cm longer than the maximum depth of the falling slurry stream relative to the direction of cut to intercept the full stream.

e) The cutter lips on straight-path cutters should be parallel, while the cutter lips of Vezin cutters should be radial from the axis of rotation.

f) Cutter lips on straight-path cutters should remain parallel, even after significant wear.

g) Cutters should accelerate from rest while still clear of the stream, traverse the stream at constant velocity, and then decelerate to a stop only after emerging from the stream.

The minimum cutter aperture and maximum cutter speed required to obtain an unbiased sample leads to the smallest acceptable increment volume and associated mass of contained solids consistent with these limiting specifications (see 6.2). However, in some circumstances, using this minimum mass of solids can result in an unacceptably large number of increments to obtain the desired sampling variance. In such cases, the volume of the slurry increment, and hence the mass of contained solids, should be increased above the smallest acceptable value.

Cutters should be designed to accommodate the maximum particle size in the slurry and the maximum slurry flow rate, from which the maximum volume and mass of solids in the increment can be determined for equipment design purposes. In particular, the choice between manual and mechanical sampling should be based on the maximum possible increment volume.

Once a cutter has been installed, there should be regular checks on the average increment mass, which should be compared with the mass predicted from the cutter aperture, cutter speed, slurry flow rate and the percentage of solids by mass in the slurry for falling-stream cutters (see 6.2). If the average mass of solids in the increment is too small compared with the predicted mass of solids for the observed slurry flow rate and solids content, it is likely that large particles are being under-sampled and/or the cutter aperture is partially blocked.

6.2 Volume of increment for falling-stream samplers to avoid bias

At any sampling stage, the minimum volume of each slurry increment taken by a linear cutter-type sampler can be calculated as follows:

$$V = \frac{GA}{v_\mathrm{c}} \tag{19}$$

where

V is the volume of increment, in cubic metres;

G is the slurry flow rate, in cubic metres per second;

A is the cutting aperture of the sampler, in metres;

v_c is the cutter speed of the sampler, in metres per second.

However, there are strict limits on the minimum cutter aperture and the maximum cutter speed to ensure that the cutter takes an unbiased sample (see 11.3.2 and 11.3.3). These limits in turn impose a lower limit on the volume of increment calculated using Equation (19) that needs to be collected to minimize bias.

From the volume of increment calculated using Equation (19), the mass of solids contained in the slurry increment can be calculated using the following equation:

$$m_\mathrm{I} = \frac{V\rho_\mathrm{a}x}{100} \tag{20}$$

where

m_I is the mass of solids contained in the increment, in kilograms;

ρ_a is the slurry density, in kilograms per cubic metre;

x is the percentage of solids by mass in the slurry, in percent.

Hence, after calculating the minimum volume of increment to avoid bias using Equation (19) (corresponding to the minimum cutter aperture and maximum cutter speed), the corresponding minimum mass of solids in the increment can be calculated using Equation (20).

7 Number of increments

7.1 General

The number of increments required to achieve a given sampling variance for a particular lot or sub-lot depends on

a) the variability of the quality characteristic of interest,

b) the duration of the lot, t_L, and

c) the mass, m_I, of the solids contained in each slurry increment.

The variability is quantified by the variance between increments, as described in 4.3.3 and 4.3.4, which need to be determined experimentally for each material type and/or processing plant. With this information, the number of increments required for time-basis sampling can be calculated as outlined below.

7.2 Simplified method

The number of primary increments, n, required to achieve the desired sampling variance for a given sampling stage can be calculated from the following equation:

$$n = \frac{s_{\mathrm{b}_1}^2}{s_{\mathrm{S}_1}^2} \qquad (21)$$

where

$s_{\mathrm{b}_1}^2$ is the variance between primary increments;

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

8 Minimum mass of solids contained in lot and sub-lot samples

8.1 Minimum mass of solids in lot samples

It is essential to ensure that the mass of solids contained in lot samples from the slurry is sufficient to obtain the required sampling variance. Subject to increments being taken in an unbiased manner (see Clause 6), the combination of the average mass of solids contained in a slurry increment and the number of increments determined in Clause 7 will ensure that a slurry sample with a sufficient mass of contained solids is collected at the primary sampling stage. However, during subsequent reduction and division of increments (if required), sub-lot samples and lot samples, it is important to ensure that a sufficient mass of solids is retained at each stage of division to achieve the desired sampling variance. The mass of solids in the sample cannot be reduced below the minimum shown in Table 1, subject to an absolute minimum of 200 g, until the solids are reduced by grinding to a smaller nominal top size.

Table 1 — Minimum mass of solids in divided increments, sub-lot samples and lot samples

Nominal top size of sample	Minimum mass of solids in divided sample kg		
	Individual increment	Sub-lot sample	Lot sample
1,0 mm	0,2	0,25	0,5
⩽ 250 µm	0,2	0,2	0,2

8.2 Minimum mass of solids in sub-lot samples

It is essential that the combined mass of solids contained in all sub-lot samples prepared for the lot be, at each sampling stage, greater than the minimum mass of contained solids in the lot sample defined in 8.1.

8.3 Minimum mass of solids in lot and sub-lot samples after size reduction

Where the solids in lot samples and sub-lot samples are reduced in size by grinding to permit further sample division, the minimum masses need to be redetermined for the nominal top size of the ground material.

9 Time-basis sampling

9.1 General

Sampling of slurry streams is usually carried out on a time basis rather than on a mass basis. Time-basis sampling involves the following steps.

a) Determine the size of the lot, e.g. an hour, a shift or a day's production.

b) Distribute the required number of increments, on a uniform time basis, throughout the total time, t_L, for sampling the lot.

c) Extract slurry increments of volume proportional to the slurry flow rate at the time of taking each increment.

9.2 Sampling interval

The interval between taking increments for time-basis sampling is as follows:

$$\Delta t \leqslant \frac{t_\mathrm{L}}{n} \tag{22}$$

where

Δt is the time interval between taking increments, in minutes;

t_L is the total time allocated to sampling the lot, in minutes;

n is the number of increments determined in Clause 7.

The fixed time interval between increments should be no larger than that calculated using Equation (22), in order to ensure that the number of increments to be taken will be at least the minimum number of primary increments specified.

9.3 Cutters

The following cutters may be used:

a) falling-stream cutters of the hose type (see Figure B.1), of which the cutting speed (i.e. hose trajectory speed) is constant during the cut;

b) falling-stream cutters of the circular path type, i.e. Vezin cutters (see Figure B.2);

c) falling-stream cutters of the diverter type, of which the cutting speed is constant during the cut (see Figure B.3).

9.4 Taking of increments

Each slurry increment is taken by a single traverse of the sampling device. The first increment is taken at a time selected at random within the first time interval. Thereafter, the remaining increments are taken at fixed time intervals according to Equation (22) until the end of the lot.

9.5 Constitution of lot or sub-lot samples

Increments are combined to form lot samples or sub-lot samples in either of the following ways.

a) Increments as taken are combined into sub-lot samples or a lot sample, irrespective of the variation of masses of solids contained in increments.

b) Increments are divided by fixed-rate division. The lot sample or sub-lot sample is then prepared by combining divided increments, provided that the mass of the contained solids in the divided increment is proportional to that of the contained solids in the primary increment, so that the weighted mean of the quality characteristic for the lot is retained.

9.6 Division of increments and sub-lot samples

After time-basis sampling, division of increments and sub-lot samples is carried out by fixed-rate division when the divided samples are to be combined. When samples are not to be combined, fixed-rate division or constant-mass division can be used.

9.7 Division of lot samples

Division of lot samples is carried out by either constant-mass or fixed-rate division.

9.8 Number of cuts for division

The minimum number of cuts and the minimum masses of contained solids for division of increments, sub-lot samples and lot samples should be determined experimentally, as specified in Clauses 7 and 8.

However, as a general guide, the following numbers of cuts may be used.

a) **For lot samples**: a minimum of 20 cuts. The combined mass of the cuts should be greater than the minimum mass of contained solids in the lot sample specified in 8.1.

b) **For sub-lot samples**: a minimum of 10 cuts. The combined mass of the contained solids of the cuts from all sub-lot samples at a given sampling stage should be greater than the minimum mass of contained solids in the lot sample as specified in 8.1.

c) **For individual increments**: a minimum of four cuts. The combined mass of the contained solids of the cuts from all increments at a given sampling stage should be greater than the minimum mass of contained solids in the lot sample as specified in 8.1.

NOTE Since the sampling precision cannot be determined *a priori*, check experiments are recommended to ascertain whether the number of cuts is sufficient.

For fixed-rate division, the interval between taking cuts is constant, regardless of the mass of contained solids in the lot sample, sub-lot sample or increment to be divided in accordance with the principles of 9.2. The first cut is taken at random within the first time interval.

10 Stratified random sampling within fixed time intervals

For stratified random sampling within fixed time intervals, the strata size Δt is determined as follows:

$$\Delta t \leqslant \frac{t_{\mathrm{L}}}{n} \tag{23}$$

When Δt has been established and the total time allocated to sampling (i.e. the duration of the lot) divided up into such time intervals (strata), the sample cutter is programmed to take one increment at a point at random within each of these intervals (strata). This is achieved by using a random number generator, capable of giving a random time value anywhere within the time stratum, which is an input to the programme that controls the cutter time sequence.

11 Mechanical sampling from moving streams

11.1 General

A wide range of different mechanical sample cutters is available, so it is not possible to specify any particular type that should be used for specific sampling applications.

Annex B gives examples from Pitard (1993) of sample cutters for slurry flows in use and should be taken as a guide in the choice of suitable equipment with correct increment extraction and delimitation.

NOTE Annex C gives examples from Pitard (1993) of slurry sampling devices that are in use in industry, but have incorrect increment extraction and delimitation.

Only mechanical cutters that take a complete cross-section of the bulk material stream in one cut may be used (see Annex B). Sampling devices that take only a part of the stream in one operation (see Annex C) do not collect representative samples and hence are not recommended.

11.2 Design of the sampling system

11.2.1 Safety of operators

From the initial stage of design and construction of a sampling system, consideration should be given to the safety of operators. Applicable safety codes of the appropriate regulatory authorities need to be respected.

11.2.2 Location of sample cutters

The location of sample cutters is chosen according to the following criteria.

a) Sample cutters should be located at a point that provides access to the complete slurry stream.

b) Sampling should be performed at a point in the handling system where there is no apparent visual segregation of the material stream and where there is no apparent risk of errors due to a periodic variation in material feed or quality, e.g. away from pulsating slurry pumps.

c) Sampling should be performed as close as possible to the point where the quality characteristics are to be determined.

11.2.3 Provision for duplicate sampling

It is recommended that the system be designed to be capable of aggregating odd- and even-numbered increments separately to constitute duplicate lot and sub-lot samples.

11.2.4 System for checking the precision and bias

When a mechanical sampling system is commissioned or when principal parts are modified, the system should be checked to ensure that correct sampling principles are respected. Check experiments for precision should be carried out for the system as a whole.

With slurries, normal methods for checking the level of bias are severely limited. Methods of verification used to check for bias in “dry” bulk particulate sampling systems, such as “stopped belt” sampling procedures, are not normally available.

11.2.5 Avoiding bias

It is essential that the sampling system be designed to avoid the following.

a) Spillage of the sample or loss of material due to dribbles, run-back on the outside of a discharging slurry pipe or underneath a discharging launder (see Figure 6).

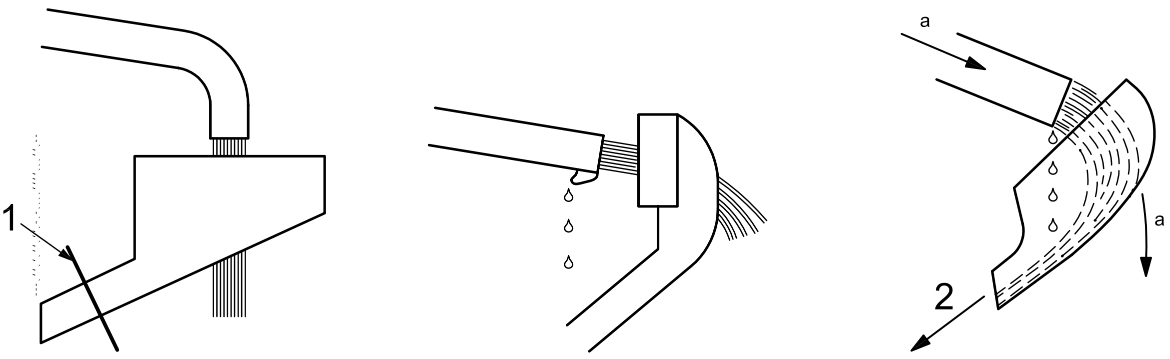
b) Restriction of the flow of the slurry increment through any device causing reflux and overflow. This is particularly important for reverse-spoon cutters where the falling slurry stream is forced to change flow direction as it strikes the inside surface of the spoon.

c) Retention of residual material in the sample cutter between cuts.

d) Contamination of the sample.

Routine inspections of sampling systems should be conducted in accordance with ISO 11790.

When a change is made in the type of solids in the slurry being sampled, the system should be thoroughly cleaned, or a quantity of material taken from the lot to be sampled should be passed through the entire system to remove any contaminants.



a) Correct **b) Incorrect** **c) Correct**

Key

1 splash guard

2 increment

[a] Stream.

Figure 6 — Examples of correct and incorrect designs for cross-stream slurry cutters

11.3 Slurry sample cutters

11.3.1 General

The only satisfactory cutter for sampling a moving stream of slurry is a falling-stream cutter that collects the increment from the stream trajectory of the slurry, e.g. at a transfer point, or discharge into or from a storage tank. Falling-stream cutters can also be used to sample slurry at a step or transfer point in an open flume or launder, provided the cutter can access the full depth and width of the slurry stream during its traverse.

Sampling of moving slurry streams using probes, spears or by-line samplers is not recommended, because they do not intercept the full cross-section of the slurry stream.

11.3.2 Falling-stream cutters

In designing falling-stream cutters, the following criteria apply.

a) The cutter lips should be sharp and the sample cutter streamlined to minimize turbulence as it cuts the slurry stream.

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

c) No slurry other than the sample should be introduced into the cutter, e.g. splashes entering the cutter in the parked position should be prevented.

d) The cutter should collect a complete cross-section of the slurry stream, both the leading and trailing edges clearing the stream in the same path.

e) The cutter should cut the slurry stream in a plane normal to, or along an arc normal to, the main trajectory path of the stream.

f) The cutter should travel through the slurry stream at near-uniform speed, i.e. the speed should not deviate by more than 5 % from the average speed.

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

h) The cutting aperture of the cutter should be at least three times the nominal top size of the particles in the slurry stream, subject to an absolute minimum of 10 mm.

i) The cutter should be of sufficient capacity to accommodate the entire increment at the maximum flow rate of the stream without any slurry loss due to reflux from the cutter aperture.

11.3.3 Cutter velocities

In designing a mechanical sample cutter, one of the most important design parameters is the cutter velocity. Too high a cutter velocity will lead to the following:

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

b) excessive turbulence, which should be avoided to minimize the risk of rebounding slurry causing a bias of the sample;

c) shock-load problems and difficulties in maintaining constant velocity while cutting the slurry stream.

For falling-stream cutters, significant bias may be introduced if the cutter speed exceeds 0,6 m/s.

11.4 Mass of solids in increments

The mass of solids contained in each increment obtained in one pass of the sample cutter is specified in 6.2.

11.5 Number of primary increments

The number of primary increments to be taken is specified in 7.2.

11.6 Routine checking

Maintenance and inspection of the installation, particularly cutter apertures, should be carried out at frequent and regular intervals. Verification of correct cutter design should be carried out when any modifications are made or a change is suspected.

12 Manual sampling from moving streams

12.1 General

Mechanical sampling from moving slurry streams is the recommended method, because it provides more reliable data than manual sampling. However, where mechanical sample cutters are not available, manual sampling may be performed, provided that access is available to the complete slurry stream and that there is no risk to the safety of the operator. In relation to the safety of operators, the safety codes of the appropriate regulatory authorities need to be respected.

Manual sampling from moving streams should not be used for sampling slurries above a maximum flow rate that takes into account the mass of each increment (typically 100 t/h).

12.2 Choosing the sampling location

The sampling location should

a) provide complete operator safety,

b) provide access to the complete slurry stream,

c) allow no apparent visual segregation of the slurry stream, and

d) be as close as possible to the point where the quality characteristics are to be determined.

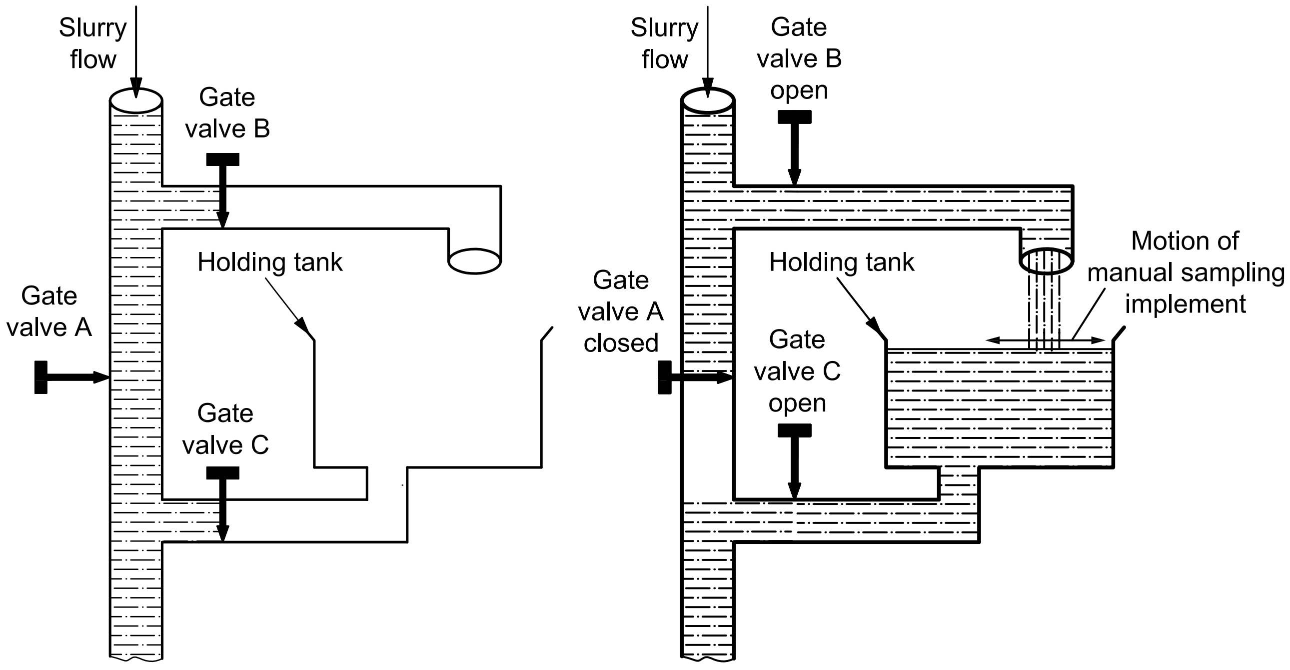
In most cases, the only sampling location that satisfies the above criteria is a transfer point. If a suitable transfer point does not exist, it is possible to construct a sample by-line system as shown in Figure 7, where a gate valve can be used to divert the full slurry flow through a pipe into a holding tank. The full stream can then be manually sampled as it falls into the holding tank, as follows:

a) open gate valves B and C;

b) close gate valve A;

c) take an increment with a manual sample cutter after the flow through the diverter pipe and holding tank has stabilized;

d) open gate valve A and then close gate valves B and C.



a) By-pass position

b) Sampling position

Figure 7 — Sample by-line for manual sampling of slurry in a pipe

12.3 Sampling implements

Manual sampling from moving streams is carried out using manual sample cutters or ladles. Examples of suitable implements are given in Annex D.

12.4 Mass of solids in increments

The mass of solids contained in each increment obtained in one pass of the sample cutter is specified in 6.2.

12.5 Number of primary increments

The number of primary increments to be taken is specified in 7.2.

12.6 Sampling procedures

The following criteria apply for manual sampling from moving streams:

a) the increment should be taken in a single operation, moving the implement across the full width of the slurry stream with both the leading and trailing edges clearing the stream in the same path;

b) the sampling implement should be moved through the stream by the operator as near as possible to constant speed, which should not exceed the maximum speed limitation on mechanical cutters (see 11.3.3);

c) the minimum cutter aperture of the sampling implement should satisfy the same width limit as for mechanical cutters (see 11.3.2);

d) the cutting aperture should be perpendicular to the slurry stream;

e) alternate increments should be taken by traversing the stream in opposite directions;

f) the dimensions of the sampling implement should match the slurry flow rate and cutting speed to prevent slurry reflux and overflow;

g) the combined mass of the sampling implement and the increment at the completion of the cut should take into account occupational health and safety guidelines.

13 Sampling of stationary slurries

Because the particles in stationary slurries settle out and stratify, sampling of stationary slurries is not recommended and hence is not covered in this International Standard.

14 Sample preparation

14.1 General

Lot and sub-lot samples should preferably be dewatered using a vacuum filtration device and then dried in an oven at the temperature specified in the relevant standard prior to further sample preparation. However, if lot or sub-lot samples are excessively large, then they may be divided using a rotary sample divider prior to filtration and drying, provided the samples are completely re-pulped prior to division and the solids content of the divided samples conform to the minimum mass requirements of 6.2.

If the liquid fraction of the slurry is to be analysed as well as the contained solids, then the liquid removed during filtration should be retained and placed in a suitable container.

14.2 Sample division

Division of dried lot and sub-lot samples should be carried out in accordance with the requirements of ISO 12743 and the minimum mass requirements of 6.2.

14.3 Sample grinding

If required, dried lot and sub-lot samples shall be ground down to 150 µm using a suitable pulverizer prior to extraction of chemical analysis samples. Certain pulverizers, such as ring mills and plate mills, tend to become heated, and samples should not be allowed to remain in them long enough to become affected. If a pulverizer is used for a series of samples, it should either be water cooled or allowed to cool between samples.

Screening to remove oversized particles for re-grinding shall not be carried out unless it can be shown experimentally, in accordance with ISO 13292 for each concentrate type being processed, that no significant bias is introduced. Material that is difficult to grind is usually different in composition from the remainder of the sample and cannot be easily mixed back into the sample.

14.4 Chemical analysis samples

Laboratory samples of typically 200 g for chemical analysis are extracted from lot and sub-lot samples. These samples should be placed in glass bottles having tight-fitting lids or may be heat sealed in plastic-lined aluminium pouches.

The nominal top size of the analysis sample is typically 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 should not be prepared from the normal dried lot or sub-lot sample. Instead, a separate chemical analysis sample should be prepared, ensuring that drying of the sample is carried out at temperatures not exceeding 60 °C.

14.5 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 should be as specified in the applicable test procedure.

15 Packing and marking of samples

Samples obtained for subsequent sample preparation for testing, which may be carried out remote from the sampling system, should be placed in airtight containers, with relevant information shown on the label and on a card placed in the container. Examples of the information are as follows:

a) type, grade and nominal top size of the solids content and the name of the lot, e.g. shift;

b) identification of the sampler;

c) time duration of the lot or sub-lot;

d) sample number or portion of the lot or sub-lot the sample represents;

e) place and date of sampling;

f) moisture content;

g) method of sampling, e.g. mechanical or manual;

h) estimated sampling precision, if available;

i) any special purpose or test for which the sample is taken.

Annex A
(normative)

Sampling-stage method for estimating sampling and total variance

A.1 Components of sampling error and sampling variance

Gy (1992) has shown that 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:

$$\mathrm{TSE} = \mathrm{TSE}_1 + ... + \mathrm{TSE}_i + ... + \mathrm{TSE}_u \qquad (A.1)$$

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:

$$\mathrm{TSE} = \mathrm{SE} + \mathrm{PE} \qquad (A.2)$$

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:

$$\mathrm{SE} = \mathrm{CE} + \mathrm{ME} \qquad (A.3)$$

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 as follows:

$$\mathrm{CE} = \mathrm{QE} + \mathrm{WE} \qquad (A.4)$$

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:

$$\mathrm{QE} = \mathrm{QE}_1 + \mathrm{QE}_2 + \mathrm{QE}_3 \tag{A.5}$$

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:

$$\mathrm{QE}_1 = \mathrm{FE} + \mathrm{GE} \tag{A.6}$$

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:

$$\mathrm{ME} = \mathrm{DE} + \mathrm{EE} \tag{A.7}$$

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:

$$\mathrm{TSE} = \mathrm{FE} + \mathrm{GE} + \mathrm{QE}_2 + \mathrm{QE}_3 + \mathrm{WE} + \mathrm{DE} + \mathrm{EE} + \mathrm{PE} \tag{A.8}$$

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:

$$\mathrm{TSE} = \mathrm{FE} + \mathrm{GE} + \mathrm{QE}_2 \tag{A.9}$$

or alternatively:

$$\mathrm{TSE} = \mathrm{QE}_1 + \mathrm{QE}_2 \tag{A.10}$$

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:

$$s_{\mathrm{S}}^2 = s_{\mathrm{FE}}^2 + s_{\mathrm{GE}}^2 + s_{\mathrm{QE}_2}^2 \tag{A.11}$$

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;

$s_{\mathrm{QE}_2}^2$ is the long-range quality-fluctuation variance.

or alternatively:

$$s_{\mathrm{S}}^2 = s_{\mathrm{QE}_1}^2 + s_{\mathrm{QE}_2}^2 = s_{\mathrm{QE}}^2 \tag{A.12}$$

where

$s_{\mathrm{QE}_1}^2$ 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 (1992) has shown that the variance of the fundamental error, s_{FE}^2, is given by:

$$s_{\mathrm{FE}}^2 = \frac{Cd^3a^2}{m_{\mathrm{S}}} \tag{A.13}$$

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:

$$C = clfg \tag{A.14}$$

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;

$l = \sqrt{(d_{\mathrm{l}} / d)}$ 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 $d_{\mathrm{l}} = d$.

The mineralogical composition factor is given by:

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

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:

$$m_S = \frac{Cd^3a^2}{s_{FE}^2} \tag{A.16}$$

EXAMPLE A porphyry copper ore having $d = 0{,}1$ cm, $d_l = 200$ 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 grade is 0,35 % Cu, i.e. 1 % $CuFeS_2$ ($a = 0{,}01$) and that the fundamental error must not exceed 0,02 % Cu or 0,06 % CuFeS2 (i.e. $s_{FE} = 0{,}000\,6$).

$$c = \frac{(1-00{,}1)\left[(1-0{,}01)\times 4{,}2 + 0{,}01\times 2{,}6\right]}{0{,}01} = 414{,}2$$

$$l = \sqrt{\left(\frac{0{,}02}{0{,}1}\right)} = 0{,}447$$

$$C = 414{,}2 \times 0{,}447 \times 0{,}5 \times 0{,}25 = 23{,}14$$

Using Equation (A.16), the minimum sample mass is given by:

$$m_S = \frac{23{,}14 \times (0{,}1)^3 \times (0{,}01)^2}{(0{,}000\,6)^2} = 6{,}4\,\text{g}$$

Thus, the minimum sample mass for this ore for a nominal top size of 0,1 cm and a fundamental error of 0,02 % Cu is 6,4 g. The sample must be ground to a smaller nominal top size before the sample mass can be reduced any further. For example, if the 6,4 g sample is crushed to a nominal top size of 200 µm, the sample mass can then be safely reduced to about 0,1 g, if required.

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 comminution 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 (1992) 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:

$$s_{\mathrm{QE}_1}^2 = 2 s_{\mathrm{FE}}^2 \tag{A.17}$$

A.4 Long-range quality-fluctuation variance

The long-range quality-fluctuation variance $s_{\mathrm{QE}_2}^2$ 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.

The 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 $s_{\mathrm{QE}_1}^2$ and $s_{\mathrm{QE}_2}^2$ to be determined. However, the method is reasonably long and better suited to those wishing to fine-tune their sampling scheme.

NOTE The interleaved 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 $s_{\mathrm{QE}_1}^2$ and $s_{\mathrm{QE}_2}^2$ 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:

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

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:

$$s_{\mathrm{s}}^2 = \frac{s_{\mathrm{b}}^2}{n} \tag{A.19}$$

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

$$n = \frac{s_{\mathrm{b}}^2}{s_{\mathrm{s}}^2} \tag{A.20}$$

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 $s_{\mathrm{s}_i}^2$ for sampling stage 'i' is given by:

$$s_{\mathrm{s}_i}^2 = \frac{s_{\mathrm{b}_i}^2}{n_i} \tag{A.21}$$

Consequently, the sampling variance s_{s}^2 for all sampling stages (1 through to $u - 1$) is given by:

$$s_{\mathrm{s}}^2 = \sum_{i=1}^{u-1} \frac{s_{\mathrm{b}_i}^2}{n_i} \tag{A.22}$$

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

$$s_{\mathrm{T}}^2 = s_{\mathrm{s}}^2 + \frac{s_{\mathrm{A}}^2}{r} \tag{A.23}$$

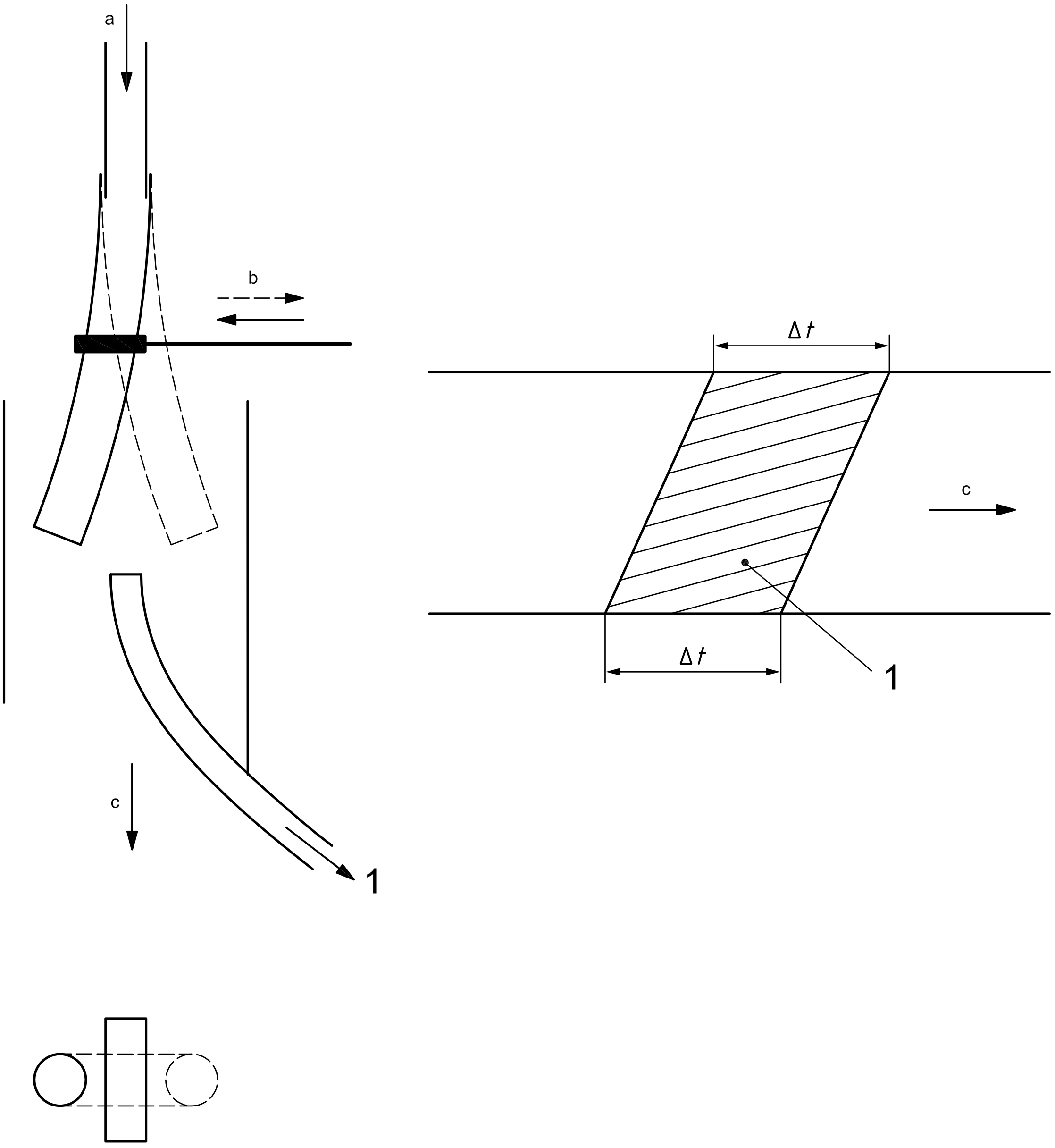
where r is the number of replicate analyses.

Combining Equations (A.22) and (A.23) gives:

$$s_{\mathrm{T}}^2 = \sum_{i-1}^{u-1} \frac{s_{\mathrm{b}_i}^2}{n_i} + \frac{s_{\mathrm{A}}^2}{r} \tag{A.24}$$

Annex B
(informative)

Examples of correct slurry sampling devices



Key

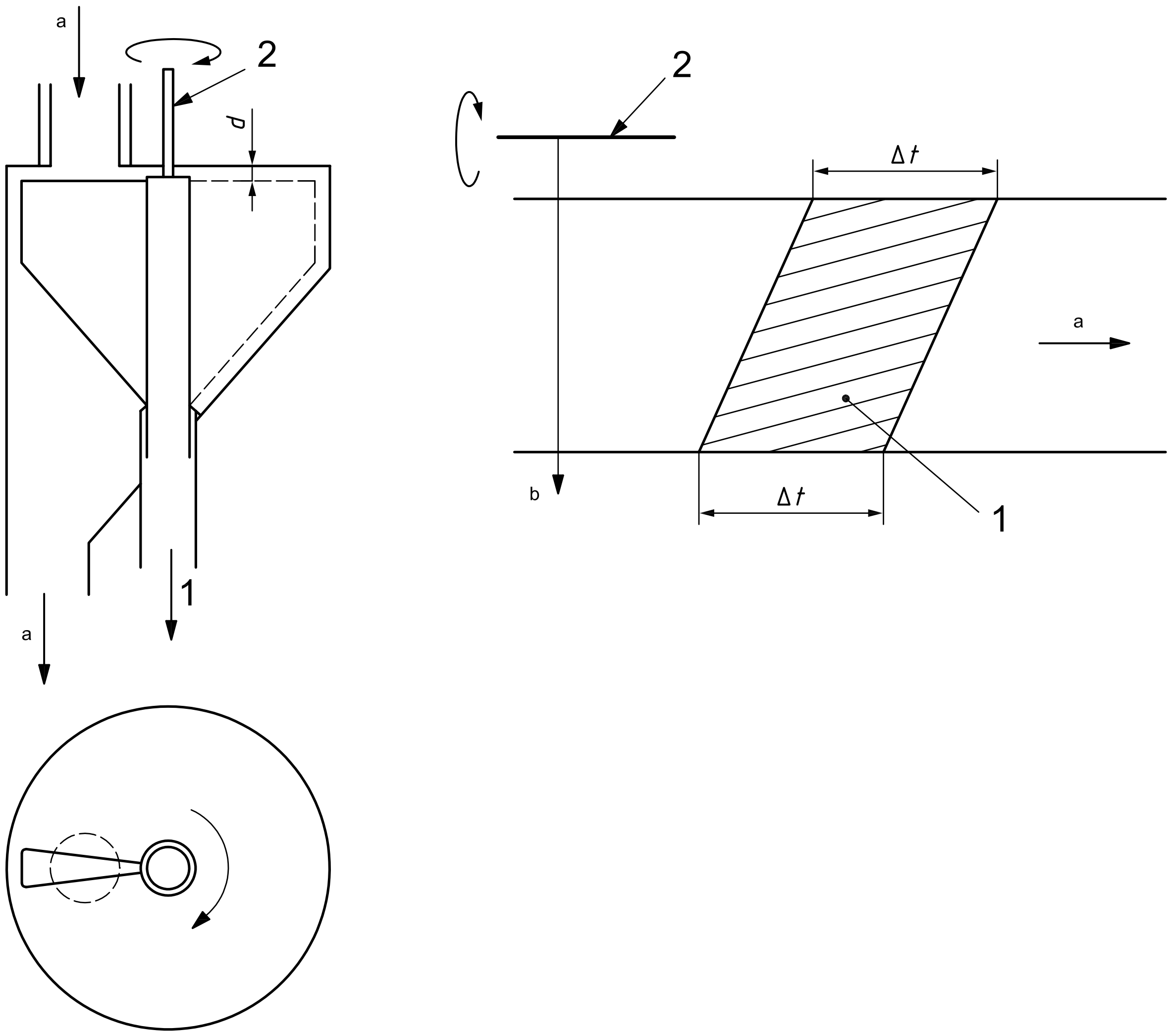
1 increment

^{a} Falling stream.

^{b} Hose trajectory.

^{c} Stream.

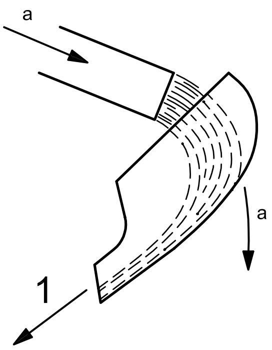
Figure B.1 — Illustration of a correctly designed hose-type slurry cutter (Pitard, 1993)



Key

1	increment
2	rotating axis
a	Stream.
b	Cutter trajectory.

Figure B.2 — Correct layout of a circular path falling-stream cutter, i.e. a Vezin cutter (Pitard, 1993)



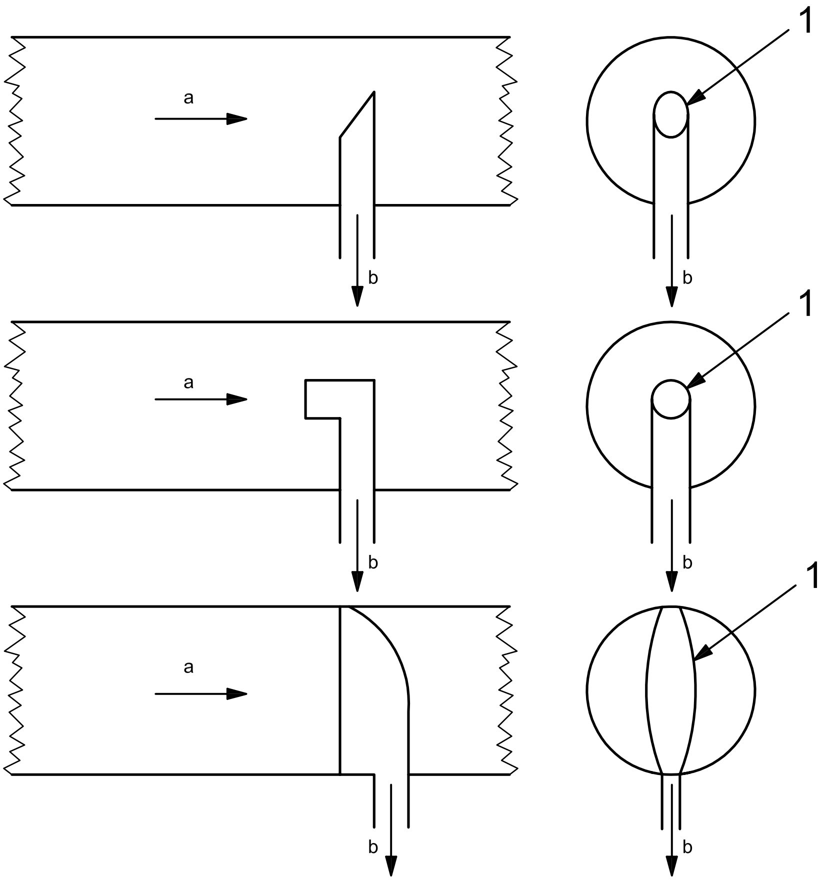
Key

1	increment
[a]	Stream.

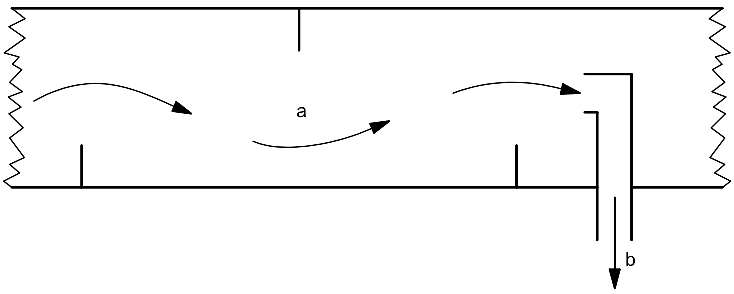
Figure B.3 — Illustration of a correctly designed falling-stream slurry cutter (after Pitard, 1993)

Annex C
(informative)

Examples of incorrect slurry sampling devices



a) Three examples of tubular probes which will always introduce a delimitation error



b) Homogenization of the stream with baffles positioned prior to a sampling probe (their effectiveness is questionable)

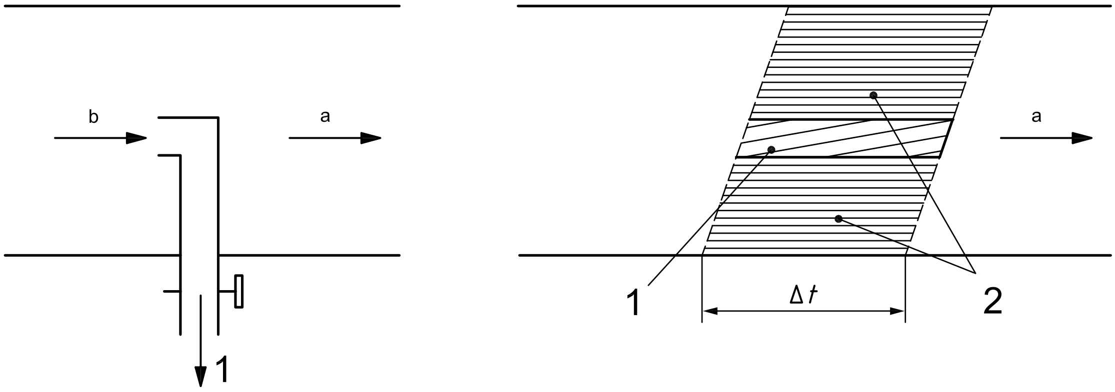
Key

1 slot

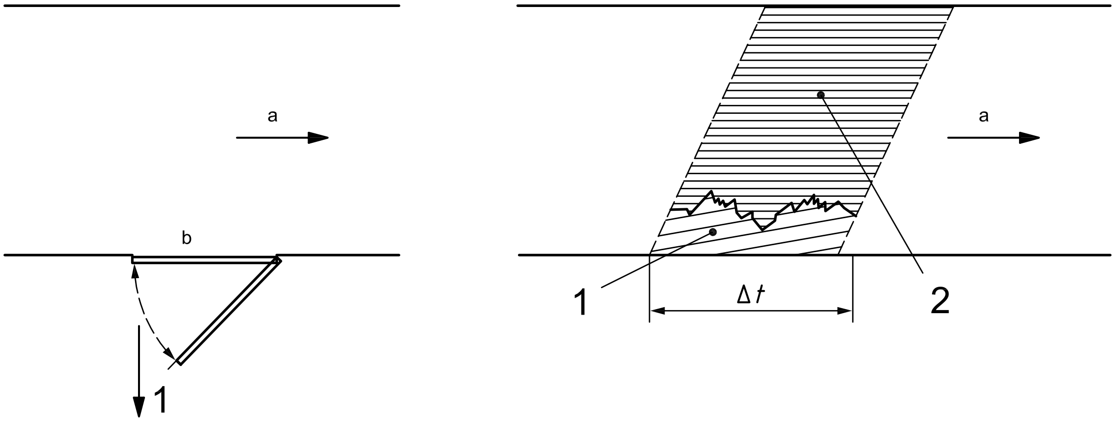
[a] Stream.

[b] Sample.

Figure C.1 — Examples of incorrect in-stream sampling probes (Pitard, 1993)



a) In-stream point sampling (always incorrect)



b) By-line slurry sampling (always incorrect)

Key

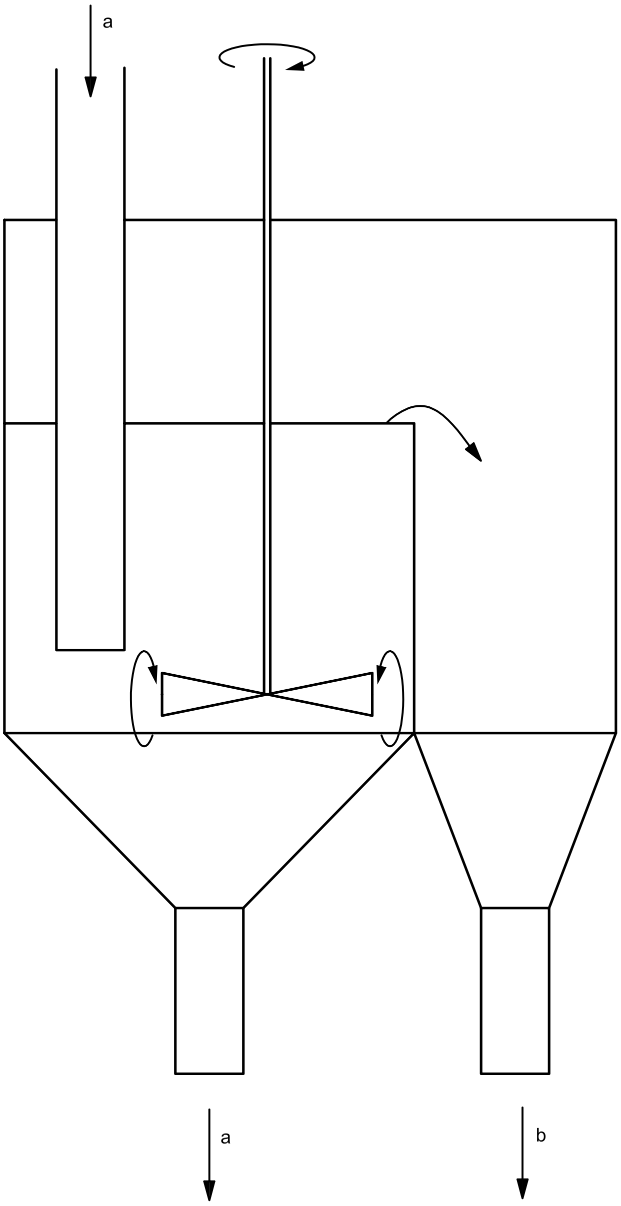
1 increment

2 missing portion of the increment

a Stream.

b Sample point.

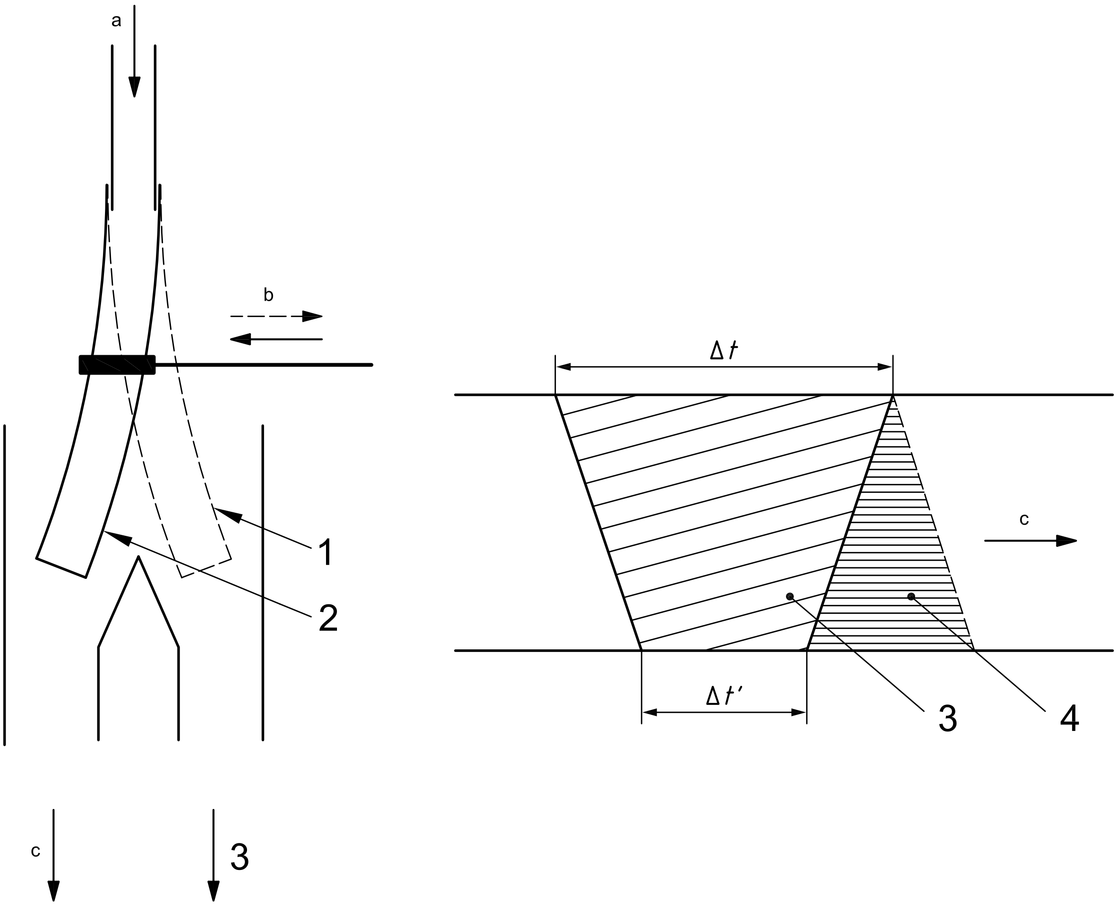
Figure C.2 — Incorrect sample delimitation using an in-stream probe and by-line sampling (Pitard, 1993)



Key

- a Stream.
- b Sample.

Figure C.3 — Illustration of an incorrectly designed sampling system using a header tank (Pitard, 1993)



Key

1 on-stream idle position

2 increment idle position

3 increment

4 missing portion of the increment

[a] Falling stream.

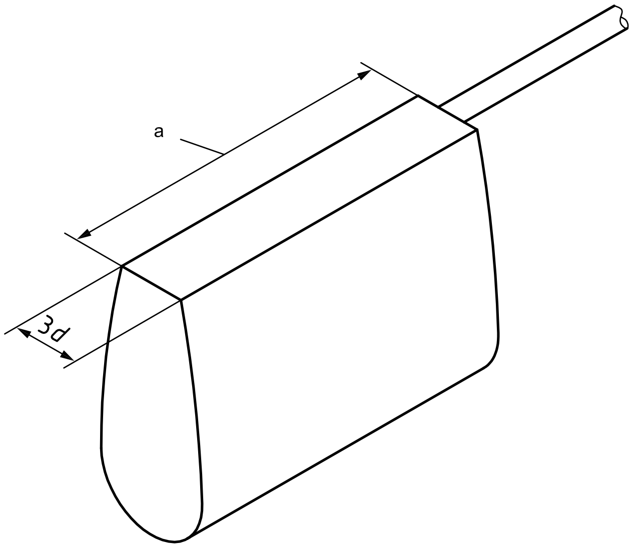
[b] Hose trajectory.

[c] Stream.

Figure C.4 — Incorrectly designed flexible hose slurry sampler (Pitard, 1993)

Annex D
(normative)

Manual sampling implements



Key

d nominal top size of the concentrate, in centimetres

[a] To exceed the depth of the falling stream.

Figure D.1 — Example of a manual sample cutter. The minimum cutter aperture shall be 10 mm

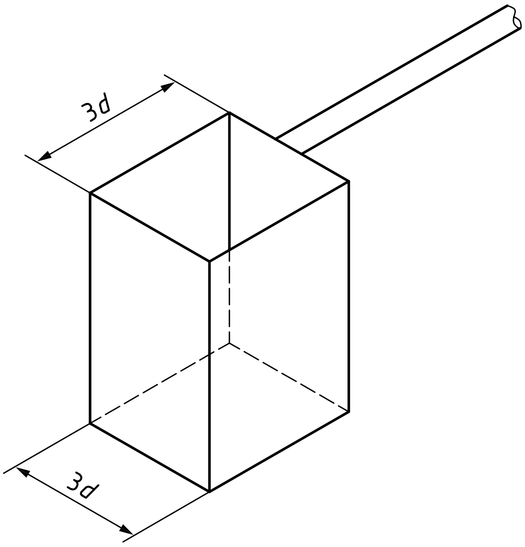


Figure D.2 — Example of a ladle. The minimum cutter aperture shall be 10 mm

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