
Hard coal — Sampling of slurries

Houille — Échantillonnage des schlamms

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Published in Switzerland

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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 20904 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

Hard coal — Sampling of slurries

1 Scope

This International Standard sets out the basic methods for sampling fine coal, coal rejects or tailings of nominal top size < 4 mm that is mixed with water to form a slurry. At very high ratios of fine solids to water when the material assumes a soft plastic form, the mixture is correctly termed a paste. Sampling of pastes is not covered in this International Standard.

The procedures described in this International Standard primarily apply to sampling of coal that is transported in moving streams as a slurry. These streams can fall freely or be confined in pipes, launders, chutes, spirals or similar channels. 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 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 solids 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 or 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 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 13909-2.

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 1213-1, *Solid mineral fuels — Vocabulary — Part 1: Terms relating to coal preparation*

ISO 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

ISO 13909-1, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

ISO 13909-4, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*

ISO 13909-8, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*

3 Definitions

For the purpose of this document, the definitions given in ISO 13909-1, ISO 1213-1 and ISO 1213-2 apply.

4 Principles of sampling slurries

4.1 General

For the purposes of this International Standard, a slurry is defined as fine coal, coal rejects or tailings of nominal top size < 4 mm that is mixed with water, which is frequently used as a convenient form to transport coal, rejects or tailings through plant circuits by means of pumps and pipelines and under gravity in launders or chutes or through long distances in slurry pipelines. Tailings from wet plants are also discharged as a slurry through pipelines to the tailings dam. In many of these operations, collection of increments at selected sample points is required for evaluation of the coal or rejects in the slurry.

A lot or sub-lot sample is constituted from a set of unbiased primary increments from a lot or sub-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 mass percentage of solids in the lot or sub-lot sample. The lot or sub-lot sample can then be filtered, dried and weighed. Alternatively, the lot or sub-lot sample may be sealed in plastic bags after filtering for transport and drying at a later stage.

Except for samples for which their characteristics are determined directly on the slurry, test samples are prepared from lot or sub-lot 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 mechanical sampling systems and manual sampling methods.

If a sampling scheme is to provide representative samples, it is necessary that all parts of the slurry in the lot have an equal opportunity of being selected and appearing in the lot sample for testing. Any deviation from this basic requirement can result in an unacceptable loss of accuracy. 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 10). If the slurry flow rate and the coal-solids concentration vary with time, the slurry volume and the dry solids mass for each increment will vary accordingly. It is necessary to show 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 11).

Best practice for sampling slurries is to mechanically cut freely falling streams (see Clause 12), 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 by incorporating steps or weirs in launders and chutes. If samples are not collected in this manner, non-uniform concentration of coal solids in the slurry due to segregation and stratification of the solids can lead to bias in the sample that is collected. Slurry flow in pipes can be homogenous with very fine particles dispersed uniformly in turbulent suspension along the length and across the diameter of the pipe. However, more commonly, the slurry in a pipe has significant particle-concentration gradients across the pipe and there can be 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 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 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. When 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, E_T , can be expressed as the sum of a number of independent components (Gy, 1982^[5]; Pitard, 1993^[6]). Such a simple additive combination is not possible if the components are correlated. The total sampling error, E_T , expressed as a sum of its components, is given by Equation (1):

$$E_T = E_{Q1} + E_{Q2} + E_{Q3} + E_W + E_D + E_E + E_P \quad (1)$$

where

- E_{Q1} is short-range quality fluctuation error associated with short-range variations in quality of the solids component of the slurry;
- E_{Q2} is long-range quality fluctuation error associated with long-range variations in quality of the solids component of the slurry;
- E_{Q3} is periodic quality fluctuation error associated with periodic variations in quality of the solids component of the slurry;
- E_W is weighting error associated with variations in slurry flow rate;
- E_D is increment delimitation error introduced by incorrect increment delimitation;
- E_E is increment extraction error introduced by incorrect increment extraction from the slurry;
- E_P is the preparation 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):

$$E_{\text{QI}} = E_{\text{F}} + E_{\text{G}} \quad (2)$$

where

E_{F} is the fundamental error due to variation in quality between particles;

E_{G} 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^[6]). 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 more increments, but it can never be completely eliminated.

A number of the components of the total sampling error, namely E_{D} , E_{E} and E_{P} , can be minimized or reduced to an acceptable level by correct design of the sampling procedure.

4.2.2 Preparation error

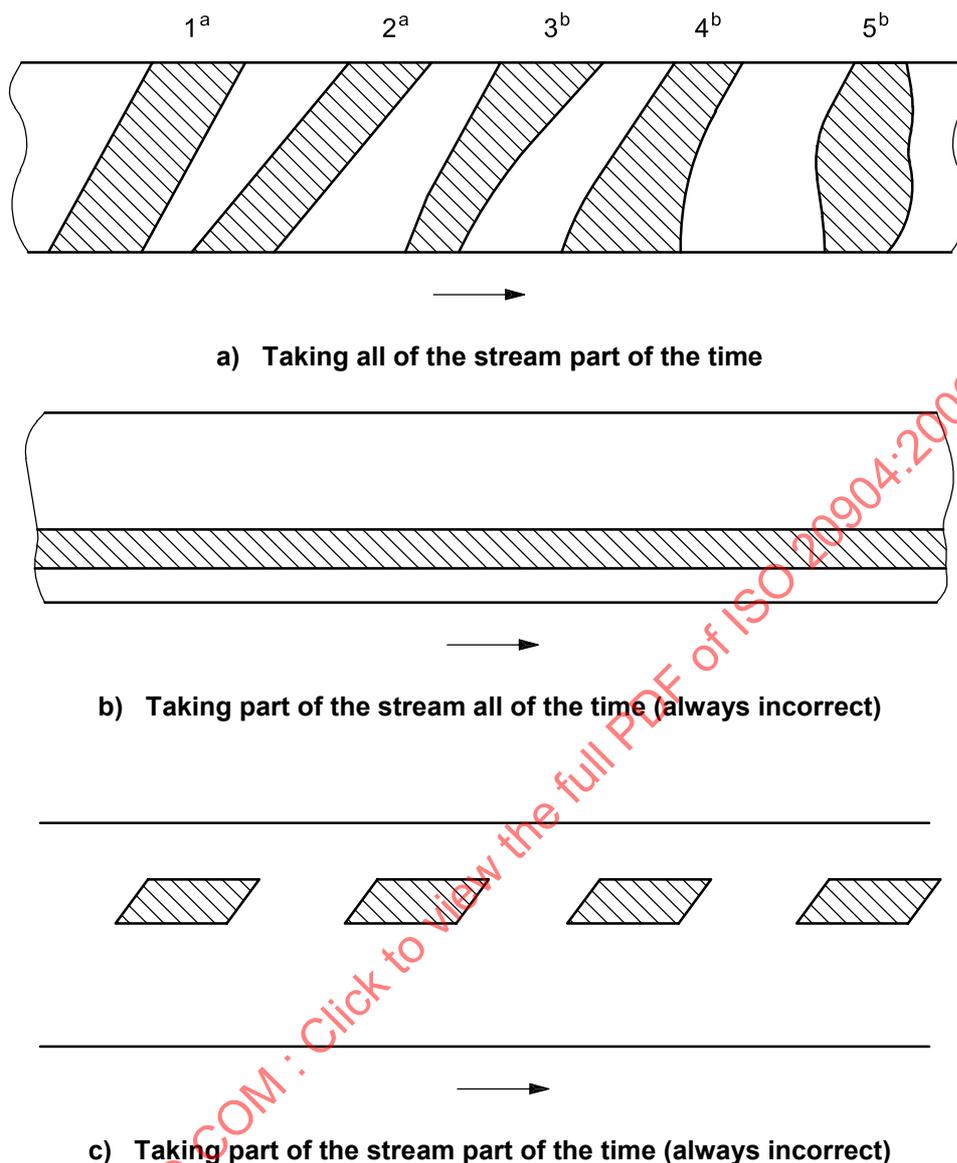
In this context, the preparation error, E_{P} , includes errors associated with non-selective sample preparation operations that should not change mass, such as sample transfer, flocculation, draining and filtering, drying, crushing, grinding or mixing. It does not include errors associated with sample division. Preparation 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 sample plant 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 it is necessary to take care during filtering to avoid loss of fines that are still suspended in the water to be discarded.

4.2.3 Delimitation and extraction errors

Delimitation and extraction errors arise from incorrect sample cutter design and operation. The increment delimitation error, E_{D} , results from an incorrect geometry of the volume delimiting the slurry increment (see Figure 1), 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 E_{D} 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:

- a) taking the whole stream part of the time with a cross-stream cutter as shown in Figure 1 a) (after Pitard, 1993^[6]), usually when 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, 4 and 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^[6]) with an in-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^[6]), also with an in-stream point sampler or probe within a pipe or channel, which is always incorrect.



- a Correct.
b Incorrect.

Figure 1 — Plan view of slurry volumes diverted by sample cutters

The increment extraction error, E_E , 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 E_E 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 due to splashes. It is necessary that the depth and capacity of the cutter 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, E_W

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 of up to 10 % relative and acceptable for variations in flow rate up to 20 % relative.

4.2.5 Periodic quality fluctuation error, E_{Q3}

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. The presence of periodic variations can be detected by determining the variogram (see ISO 13909-7). While in most cases variogram values can be fitted with a simple linear or quadratic function, if periodic behaviour (characterized by regularly spaced maxima and minima) is observed, the fitting function can include a sine-wave term with a period and amplitude to be determined as parameters of the fit (Gy, 1982^[5]). In such cases, stratified random sampling should be carried out as discussed in Clause 11. The alternative is to significantly reduce the source of periodic variations in quality, which can require plant redesign.

4.3 Sampling and overall variance

4.3.1 Sampling variance

Assume that the weighting (E_W), increment delimitation (E_D), increment extraction (E_E) and preparation errors (E_P) described in 4.2.2, 4.2.3 and 4.2.4 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 of Equation (3):

$$E_T = E_{Q1} + E_{Q2} \tag{3}$$

Hence, the sampling variance (V_S) is given by Equation (4):

$$V_S = V_{EQ1} + V_{EQ2} \tag{4}$$

The short-range quality fluctuation variance, V_{EQ1} , 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, V_{EQ2} , 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 Overall variance

The experimental estimate of overall variance is denoted by V_{SPT} . It is comprised of three components, namely the estimated variance of sampling, the estimated variance of sample preparation and the estimated variance of testing, as given in Equation (5):

$$V_{SPT} = V_S + V_P + V_T \tag{5}$$

where

V_S is estimated sampling variance;

V_P is estimated sample preparation variance;

V_T is estimated measurement variance.

Methods for obtaining estimates V_S , V_P , V_T and V_{SPT} can be found in ISO 13909-7.

NOTE The distinction between “sampling” and “sample preparation” is not always clear. For the purposes of this International Standard, “sampling” stages denote those stages of sampling and sample division that take place within the sampling plant where slurry increments are extracted and where drainage of clear water is carried out after the contained solids of the sample settle. On the other hand, “sample preparation” stages denote those stages that take place away from the sampling plant, typically in the plant laboratory.

Sample preparation stages may include additional drainage, filtering and drying of samples before size reduction, sample division and preparation of test samples for measurement. The principles of sampling given in 4.2 apply to sample preparation stages as well as to the sampling stages.

Where a very precise result is required and the sampling variance has been minimized, consideration has to be given to increasing the number of sample preparations and measurements to reduce these components of the overall variance. This is achieved by the following:

- a) carrying out multiple determinations on the contained solids in the lot sample;
- b) analysing the contained solids in individual increments;
- c) dividing the slurry lot into a number of sub-lots or part-lots and analysing the contained solids in a sample from each sub-lot.

The overall variance in each case is then given by one of the following equations:

- where a single lot sample is constituted from a lot and r replicate determinations on the contained solids are carried out on the lot sample, by Equation (6):

$$V_{\text{SPT}} = V_{\text{S}} + V_{\text{P}} + \frac{V_{\text{T}}}{r} \quad (6)$$

- where m sub-lot samples are prepared, each constituted from the contained solids of an equal number of increments, and r replicate determinations are carried out on each sub-lot sample, by Equation (7):

$$V_{\text{SPT}} = V_{\text{S}} + \frac{V_{\text{P}} + \frac{V_{\text{T}}}{r}}{m} \quad (7)$$

- where all n increments are prepared and a single determination is carried out on the contained solids of each increment, by Equation (8):

$$V_{\text{SPT}} = V_{\text{S}} + \frac{V_{\text{P}}}{n} + \frac{V_{\text{T}}}{n} \quad (8)$$

5 Sampling schemes

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 that are non-routine and carried out infrequently, e.g. determining increment variance in step e), particularly when a significant change has occurred to the slurry source or to the sampling equipment. The procedure is as given in the following steps a) through i).

- a) Define the purpose for which the samples are being taken. Sampling for commercial transactions is usually the main purpose of International Standards for sampling. 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. If the required precision results in impractical numbers of increments and sub-lots, it can be necessary to adopt a poorer precision.
- d) Ascertain the nominal top size and particle density of the solids in the slurry for determining the minimum volume of slurry increment and the minimum mass of the solids in the lot sample (see Clauses 6 and 9).
- e) Check the procedures and equipment for taking slurry increments to minimize bias (see Clause 6).
- f) Determine the variability of the coal and the variance of preparation and testing for the quality characteristics under consideration (see Clause 7).
- g) Determine the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see Clause 8).
- h) Determine the sampling interval in minutes for time-basis systematic sampling (see Clause 10) or stratified random sampling within fixed time intervals (see Clause 11).
- 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 may be combined to constitute a single lot sample for analysis (see Figure 2). Alternatively, increments taken from sub-lots may be used to constitute sub-lot samples for analysis, which also improves the overall precision of the measured quality characteristics of the lot (see Figure 3). Other reasons for separate preparation and analysis of sub-lot samples are

- for convenience of materials handling;
- to provide progressive information on the quality of the lot;
- to provide reference or reserve samples after division.

Each increment may also be analysed separately (see Figure 4) to determine the increment variance of quality characteristics of the lot. In addition, it is recommended that the precision achieved in practice should be checked on an ongoing basis by duplicate sampling where alternate increments are diverted to lot samples A and B (see Figure 5) from which two test samples are prepared and analysed. A substantial number of sample pairs (preferably at least 20) is required to obtain a reliable estimate of precision.

In most situations, it is not necessary to crush or pulverise the solids in the slurry increment to allow further division, since most slurries contain only fine particles. However, if the particles are coarse and particle-size reduction is required to allow further division, it is necessary to re-determine the minimum sample mass for the lot using the new nominal top size of the crushed solids (see Clause 9).

The initial design of a sampling scheme for a new plant or a slurry with 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 in stationary situations, such as a settled or even a well-stirred slurry in a tank, holding vessel or dam, is not recommended and is not covered in this International Standard.

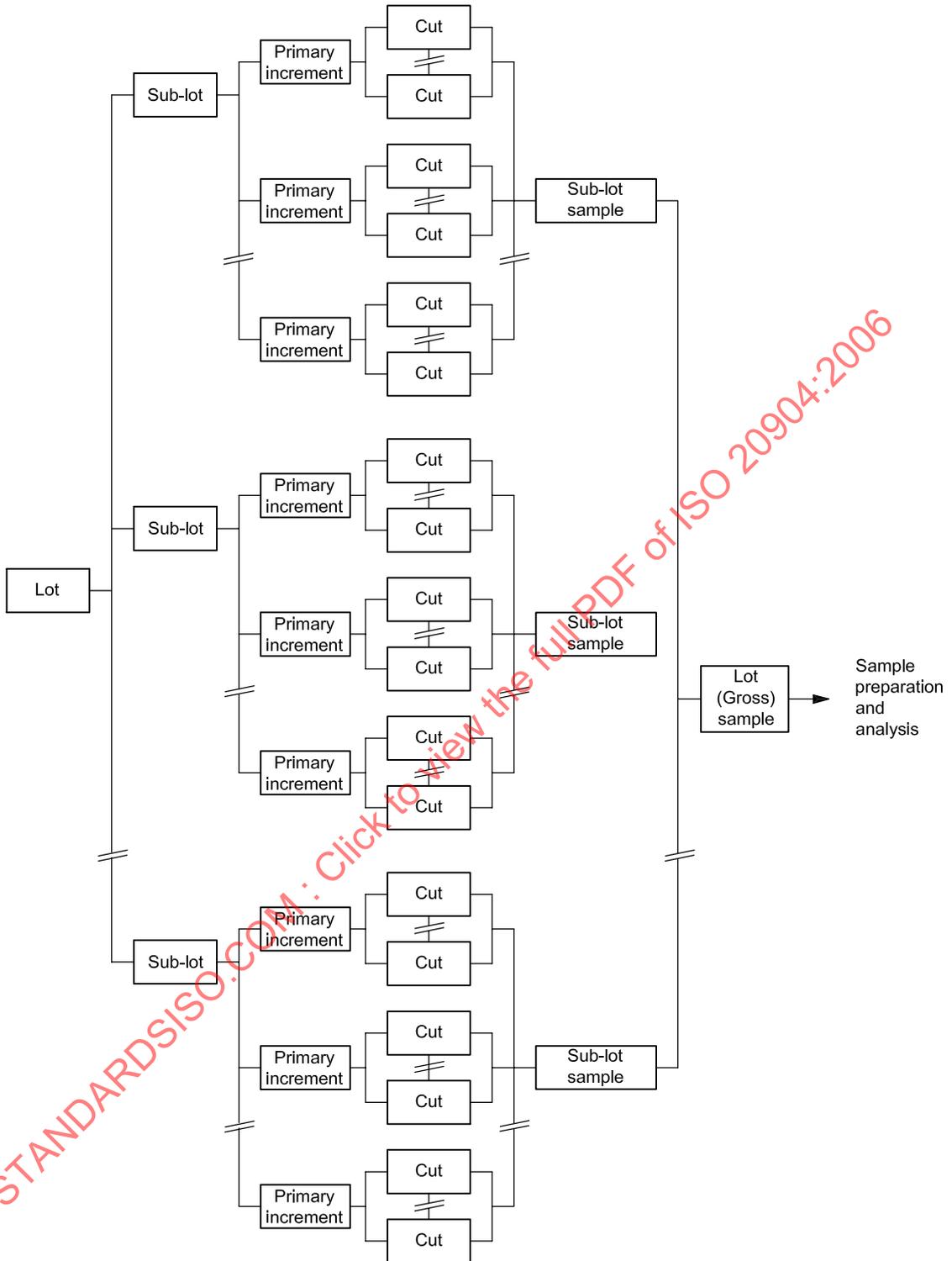


Figure 2 — Example of a sampling plan where a single lot sample is constituted for analysis

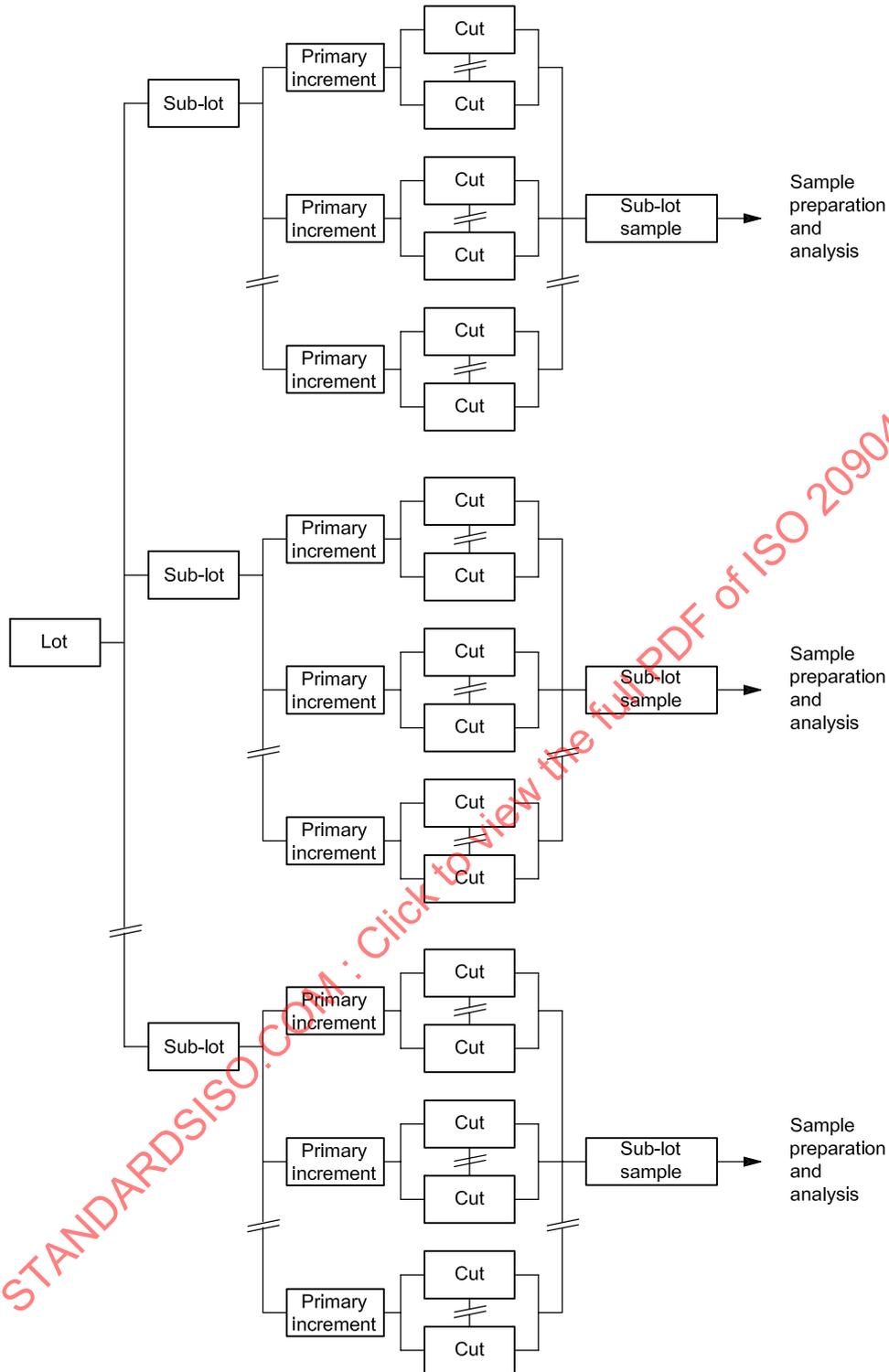


Figure 3 — Example of a sampling plan with each sub-lot sample analysed separately

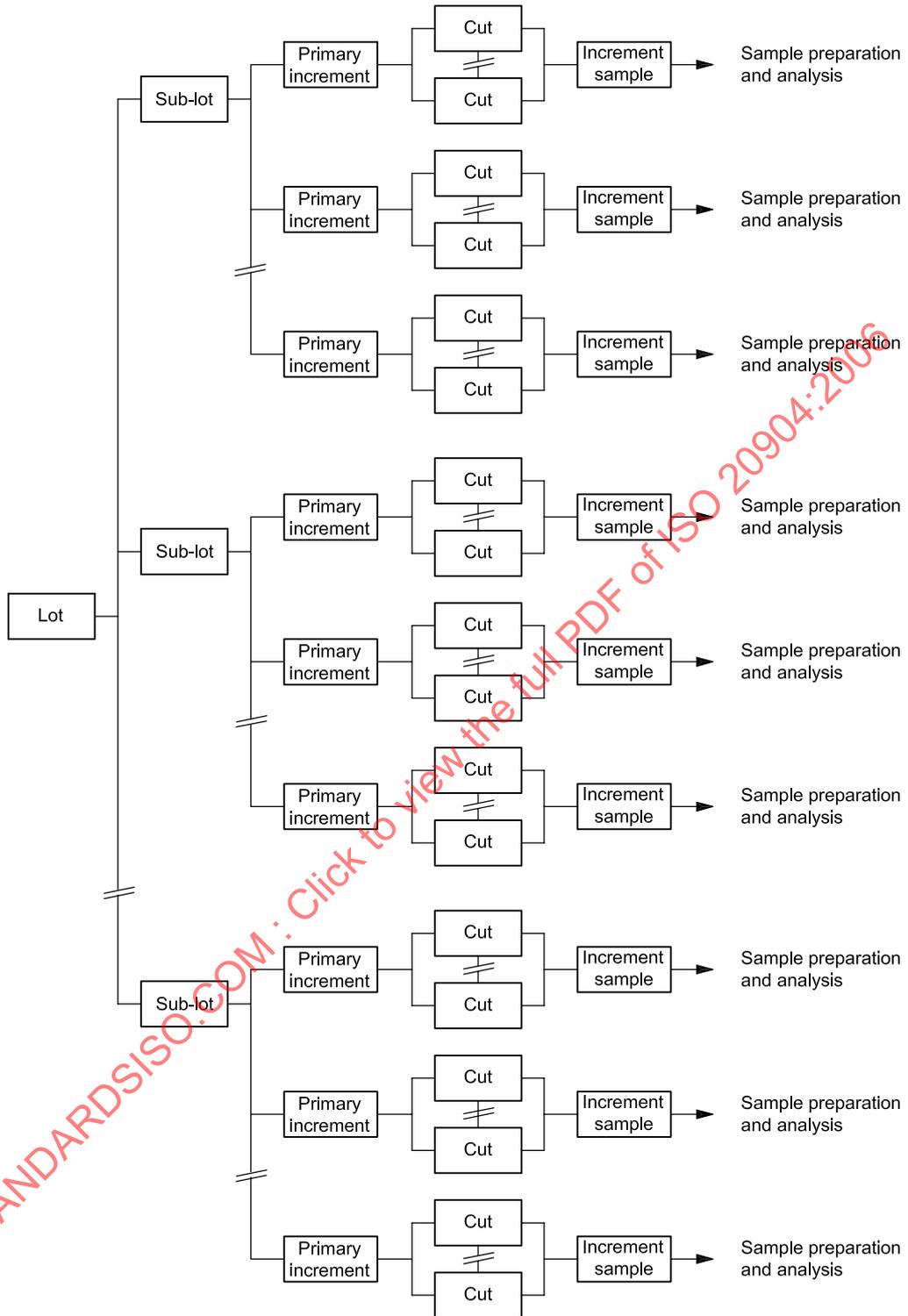


Figure 4 — Example of a sampling plan with each increment analysed separately

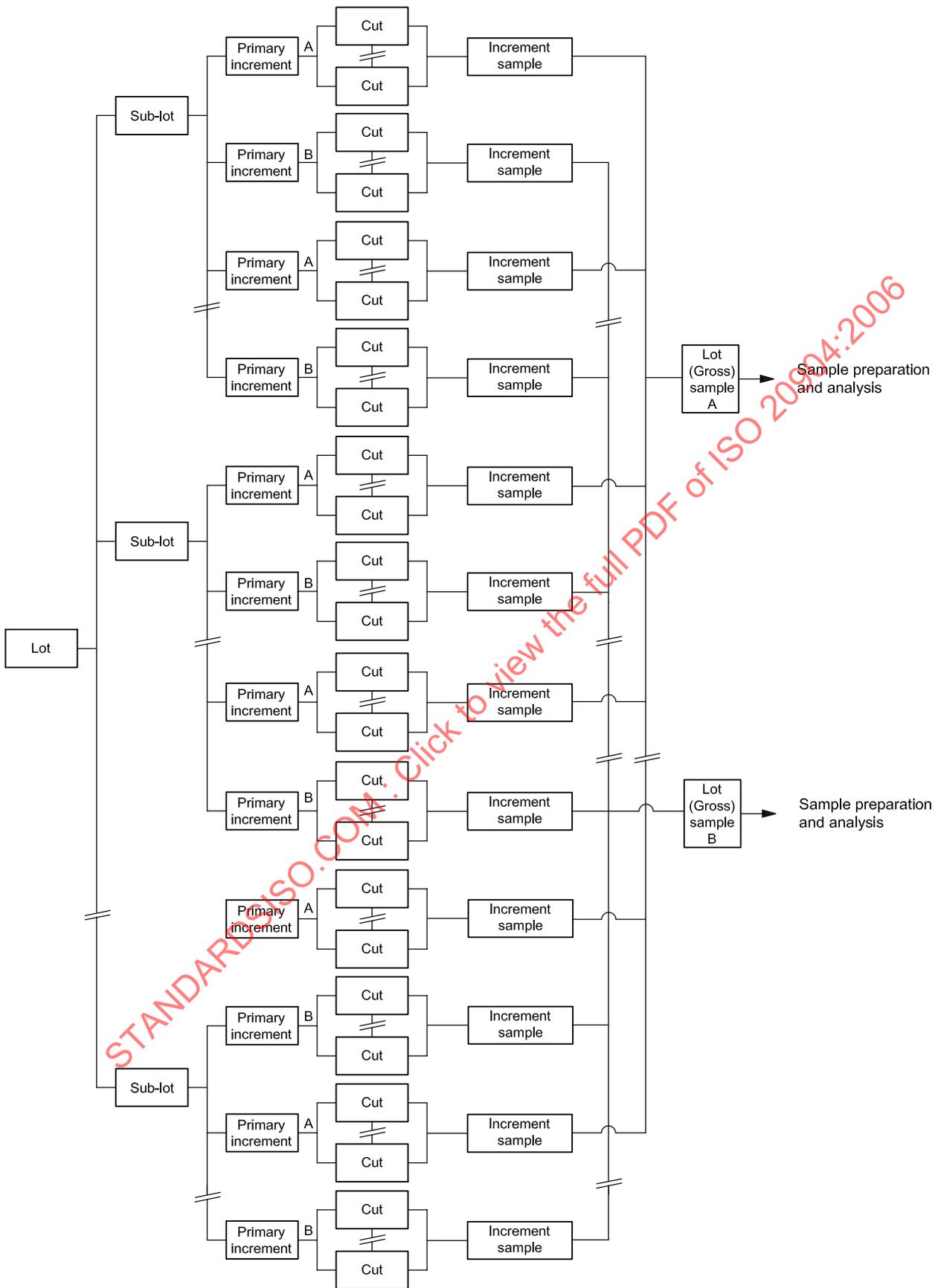


Figure 5 — Example of a duplicate sampling plan for routine analysis or determination of overall precision

6 Minimization of bias and unbiased increment mass

6.1 Minimizing 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 increments are extracted from the lot in such a manner that all parts of 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 the slurry. In practice, this means that it is necessary to take a complete cross-section of the slurry when sampling from a moving stream.

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

- a) A complete cross-section of the slurry stream shall be taken when sampling from a moving stream.
- b) There shall be no loss or spillage of the slurry sample.
- c) The cutter aperture shall 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 shall be substantially 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 shall be parallel, while the cutter lips of Vezin cutters shall be radial from the axis of rotation.
- f) Cutter lips on straight-path cutters shall remain parallel, even after significant wear.
- g) Cutters shall 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 lead 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, shall be increased above the smallest acceptable value.

Cutters shall be designed to accommodate the maximum size of the particles 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. The choice between mechanical and manual sampling shall be based on the maximum possible increment volume and the consequential safety considerations.

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 cutter aperture, cutter speed and the slurry flow rate 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, it is likely that large particles are being under-sampled and/or the cutter aperture is partially blocked.

When new coal-sampling equipment has been installed and/or a new coal-slurry sampling scheme is implemented, a bias check shall be conducted in accordance with the methods given in ISO 13909-8.

6.2 Volume of increment for falling stream samplers to avoid bias

At any sampling stage, the minimum volume of a slurry increment taken by a cutter-type sampler can be calculated from Equation (9):

$$G_1 = \frac{qW}{S} \quad (9)$$

where

- G_1 is the volume of increment, expressed in cubic metres;
- q is the slurry flow rate, expressed in cubic metres per second;
- W is the cutting aperture of the sampler, expressed in metres;
- S is the cutter speed, expressed in metres per second.

However, there are strict limits on the minimum cutter aperture and the maximum cutter speed to ensure the cutter takes an unbiased sample (see 12.3.2 and 12.3.3). These limits in turn impose a lower limit on the volume of increment calculated using Equation (9) that it is necessary to collect to minimize bias.

From the volume of increment calculated using Equation (9), the mass of solids contained in the slurry increment can be calculated using Equation (10):

$$m_1 = \frac{G_1 \rho x}{100} \quad (10)$$

where

- m_1 is the mass of solids contained in the increment, expressed in kilograms;
- ρ is the slurry density, expressed in kilograms per cubic metre;
- x is the percentage solids by mass in the slurry.

Hence, after calculating the minimum volume of increment to avoid bias using Equation (9) (corresponding to the minimum cutter aperture and maximum cutter speed), the corresponding minimum mass of solids in the increment can be calculated using Equation (10).

6.3 Volume of increment for manual sampling to avoid bias

Under favourable conditions (for example, small and accessible slurry flows), manual cross-stream cuts through free-falling streams can be used to extract increments without bias provided

- a) the full stream is cut in one action;
- b) the sampling implement is 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;
- c) the minimum cutter aperture of the sampling implement satisfies the same width limit as for mechanical cutters;
- d) the combined weight of the sampling implement and the increment at the completion of the cut takes into account occupational health and safety guidelines;

- e) the dimensions of the sampling implement match the slurry flow rate and cutting speed to prevent slurry reflux and overflow.

7 Precision of sampling and determination of increment variance

7.1 Overall precision

In all methods of sampling, sample preparation and analysis, errors are incurred and the experimental results obtained from such methods for any given parameter deviate from the true value of that parameter. While the absolute deviation of a single result from the "true" value cannot be determined, it is possible to make an estimate of the precision of the experimental results. This is the closeness with which the results of a series of measurements made on the same coal slurry agree amongst themselves, and the deviation of the mean of the results from an accepted reference value, i.e. the bias of the results (see ISO 13909-8).

It is possible to design a sampling scheme by which, in principle, an arbitrary level of precision can be achieved.

The required overall precision for a lot should be agreed between the parties concerned. In the absence of such agreement, a value of one tenth of the ash content may be assumed.

The theory of methods for estimation of overall precision is discussed in ISO 13909-7, where the Equation (11) is derived for continuous sampling:

$$P_L = 2\sqrt{\frac{V_1 + V_{PT}}{n}} \quad (11)$$

where

P_L is the estimated overall precision of sampling, sample preparation and testing for the lot at 95 % confidence level, expressed as % absolute;

V_1 is the primary increment variance;

n is the number of increments per sub-lot;

m is the number of sub-lots in the lot;

V_{PT} is the preparation and testing variance.

If the quality of a slurry of a type not previously sampled is required, then in order to devise a sampling scheme, assumptions have to be made about the variability. The precision actually achieved for a particular lot by the scheme devised can be measured by the procedures given in ISO 13909-7.

NOTE The primary increment variance, V_1 , in Equation (11) is related to the estimated sampling variance V_S in Equation (5) via the relationship $V_S = \frac{V_1}{n}$.

7.2 Primary increment variance

The primary increment variance, V_1 , in Equation (11) depends upon the type and nominal top size of the solids in the slurry, the degree of pre-treatment and mixing, the absolute value of the parameter to be determined and the mass of increment taken. It can be obtained by either

- direct determination on the slurry to be sampled using one of the methods described in ISO 13909-7, or
- assuming a value determined for a similar slurry from a similar handling and sampling system.

If neither of these values is available, a value of $V_1 = 20$ for ash content can be assumed initially and checked, after the sampling has been carried out, using one of the methods described in ISO 13909-7.

7.3 Preparation and testing variance

The value of the preparation and testing variance, V_{PT} , required for the calculation of the precision using Equation (11) can be obtained by either

- a) direct determination on the slurry to be sampled using one of the methods described in ISO 13909-7, or
- b) assuming a value determined for a similar slurry from a similar sample preparation scheme.

If neither of these values is available, a value of 0,2 for ash content can be assumed initially and checked, after the preparation and testing has been carried out, using one of the methods described in ISO 13909-7.

8 Number of sub-lots and number of increments per sub-lot

The number of increments taken from a lot in order to achieve a particular precision is a function of the variability of the quality of the slurry in the lot irrespective of the mass of the lot. The lot may be sampled as a whole, resulting in one sample, or divided into a number of sub-lots resulting in a sample from each. Such division can be necessary in order to achieve the required precision.

There may be other practical reasons for dividing the lot:

- a) for convenience when sampling over a long period;
- b) to keep sample masses manageable.

After determining the minimum number of sub-lots required for practical reasons, estimate the number of increments in each sub-lot for a desired precision from Equation (12), obtained by transposing Equation (11):

$$n = \frac{4V_1}{mP_L^2 - 4V_{PT}} \quad (12)$$

A value of infinity or a negative number indicates that the preparation and testing variances are such that the required precision cannot be achieved with this number of sub-lots. In such cases, or if n is impracticably large, increase the number of sub-lots by one of the following means.

- a) Choose a number corresponding to a convenient mass, recalculate n from Equation (12) and repeat this process until n is a practicable number.
- b) Decide on the maximum practicable number of increments per sub-lot, n_1 , and calculate m from Equation (13):

$$m = \frac{4V_1 + n_1V_{PT}}{n_1P_L^2} \quad (13)$$

Adjust m upwards if necessary to a convenient number and recalculate n .

Take n as 10 if the final calculated value is less than 10.

NOTE Equation 12 generally gives an overestimate of the required number of increments. This is because it is based on the assumption that the quality of solids in the slurry has no serial correlation; however, serial correlation is always present to some degree. In addition, because a certain amount of preparation and testing is required when measuring the increment variance or the sub-lot variance, the preparation and testing errors are included more than once.

The designer of a sampling scheme should cater for the worst case anticipated and then tend to use higher values for V_I and V_{PT} than can actually occur when the system is in operation. On implementing a new sampling scheme, a check on the actual precision being achieved should be carried out using the methods described in ISO 13909-7. This can be necessary to achieve the required precision, in which case the number of sub-lots is calculated using Equation (13).

9 Minimum mass of solids in lot and sub-lot samples

9.1 General

It is essential to ensure that the mass of solids contained in lot samples collected from the slurry is sufficient to obtain the required sampling precision. 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 8 ensures that a slurry sample with 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 sufficient mass of solids is retained at each stage of division to achieve the desired sampling variance.

9.2 Minimum mass of solids in lot samples

The minimum mass of solids in a sample is dependent on the nominal top size of the coal, the precision required for the parameter concerned and the relationship of that parameter to particle size. Such a relationship applies at all stages of preparation. The attainment of this mass does not, of itself, guarantee the required precision, because precision is also dependent on the number of increments in the sample and their variability.

Values for the minimum mass of solids for general analysis for a precision of 0,2 % with regard to ash are given in Table 1. When a slurry is sampled regularly under the same circumstances, the precision obtained for all the required quality parameters shall be checked (see ISO 13909-7) and the masses may be adjusted accordingly. However, the masses shall not be reduced below the minimum requirements laid down in the relevant analysis International Standards.

When preparing samples for multiple use, account shall also be taken of the individual masses and size distribution of the test samples required for each test.

Table 1 — Minimum mass of solids in lot sample for general analysis

Nominal top size of solids in slurry mm	General analysis samples and common samples kg
4,0	1,6
2,8	0,65
2,0	0,25
1,0	0,10

9.3 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 is, at each sampling stage, greater than the minimum mass of contained solids in the lot sample defined in 9.2.

9.4 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 crushing and/or grinding to permit further sample division, the minimum masses need to be re-determined using Table 1.

10 Time-basis sampling

10.1 General

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

- a) Determine the size of the lot and sub-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 each sub-lot.
- c) Extract slurry increments of volume proportional to the slurry flow rate at the time of taking each increment.

10.2 Sampling interval

The interval between taking increments for time-basis sampling is as given in Equation (14):

$$\Delta t = \frac{t_L}{n} \quad (14)$$

where

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

t_L total time allocated to sampling each sub-lot, expressed in minutes;

n number of increments determined in Clause 8.

10.3 Cutters

The following cutters may be used:

- a) falling-stream cutters of the hose type (see Annex A, Figure A.1), whose cutting speed (i.e. hose-trajectory speed) is constant throughout the sampling of the entire lot;
- b) falling-stream cutters of the circular path type, i.e. Vezin cutters (see Annex A, Figure A.2);
- c) falling-stream cutters of the diverter type, whose cutting speed is constant throughout the sampling of the entire lot (see Annex A, Figure A.3).

If individual increments are analysed, then it is necessary to record the mass represented by each increment to weight the analyses accordingly.

10.4 Taking of increments

Each slurry increment shall be taken by a single traverse of the sampling device. The first increment shall be taken at a time selected at random within the first time interval. Thereafter, the remaining increments shall be taken at fixed time intervals according to Equation (14) until the end of the lot.

The fixed time interval between increments should be no longer than that calculated using Equation (14) to ensure that the number of increments taken will be at least the minimum number of primary increments specified.

10.5 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.

10.6 Division of increments and sub-lot samples

After time-basis sampling, division of increments and sub-lot samples shall be 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.

10.7 Division of lot samples

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

10.8 Number of cuts for division

As a general guide, the following numbers of cuts may be used:

- a) For lot samples — a minimum of 60 cuts. The combined mass of the cuts shall be greater than the minimum mass of contained solids in the lot sample specified in Table 1.

If, during preparation, the sample is thoroughly mixed and it can be established that the required precision can be achieved, the number of cuts may be reduced to 20. If the mass is too low, an alternative manual method of division should be used.

- 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 shall be greater than the minimum mass of contained solids in the lot sample specified in Table 1.
- c) For individual increments — a minimum of four cuts. For subsequent division of individual increments, a minimum of one cut shall be taken from each cut from the preceding division. The combined mass of the contained solids of the cuts from all increments at a given sampling stage shall be greater than the minimum mass of contained solids in the lot sample specified in Table 1.

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

11 Stratified random sampling within fixed time intervals

For stratified random sampling within fixed time intervals, the strata size Δt is determined using Equation (14). When Δt has been established and the total time allocated to sampling each sub-lot divided up into such time intervals (strata), the sample cutter shall be programmed to take one increment at any point at random within each of these intervals (strata). This is achieved by use of a random number generator, capable of giving a random time value anywhere within the time stratum, which is an input to the program that controls the cutter time sequence.

12 Mechanical sampling from moving streams

12.1 General

A wide range of different mechanical sample cutters are available, so it is not possible to specify any particular type that should be used for specific sampling applications; however, they shall fall within the set of falling-stream cutter types listed in 10.3.

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

Annex A gives examples from Pitard (1993^[6]) of sample cutters for slurry flows that are in use and should be taken as a guide in the choice of suitable equipment with correct increment extraction and delimitation.

Annex B gives examples from Pitard (1993^[6]) of slurry sampling devices that are in use in industry but which have incorrect increment extraction and delimitation and should not be used.

12.2 Design of the sampling system

12.2.1 Safety of operators

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

12.2.2 Location of sample cutters

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

- a) Sample cutters shall be located at a point that affords access to the complete slurry stream.
- b) Sampling shall 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 shall be performed as close as possible to the point where the quality characteristics are to be determined.

12.2.3 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 sub-lot samples.

12.2.4 System for checking the precision and bias

When a mechanical sampling system is commissioned or when principal parts are modified, the system shall 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 somewhat limited. Methods of verification used to check for bias in "dry" bulk sampling systems, such as "stopped belt" sampling procedures, are not suitable. However, it is possible to take a reference sample by diverting a slurry pipe into a suitable container for a period of time.

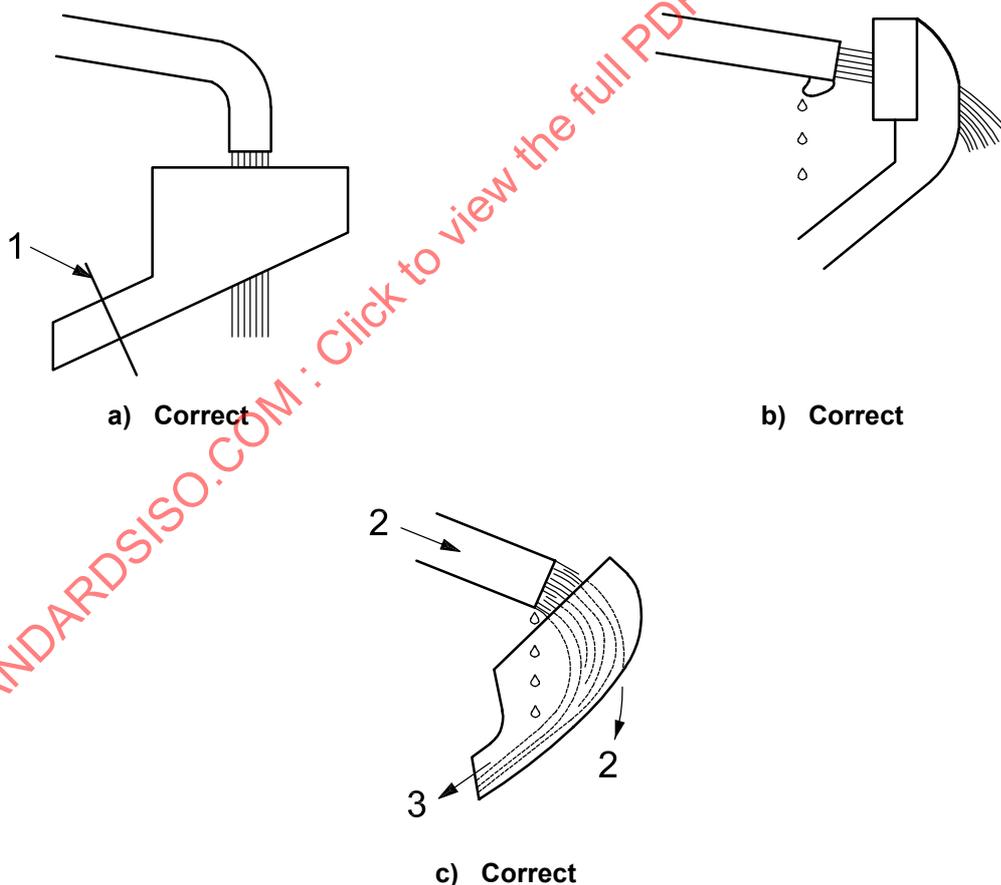
12.2.5 Minimizing bias

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

- spillage of the sample or loss of material due to dribbles or run-back on the outside of a discharging slurry pipe or underneath a discharging launder (see Figure 6);
- 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;
- retention of residual material in the sample cutter between cuts;
- contamination of the sample.

Routine inspections of sampling systems shall be conducted to check for possible non-conformance with the above requirements.

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.



Key

- splash guard
- stream
- increment

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

12.3 Slurry sample cutters

12.3.1 General

The only satisfactory cutter for sampling a moving stream of slurry is a falling-stream cutter, which collects the increment from the stream trajectory of the slurry, e.g. at a transfer point or the 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.

12.3.2 Falling-stream cutters

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

- a) The sample cutter shall be of the self-clearing type, e.g. stainless-steel- or polyurethane-lined, discharging each increment completely.
- b) No slurry other than the sample shall be introduced into the cutter, e.g. splashes entering the cutter in the parked position should be prevented.
- c) The cutter shall collect a complete cross-section of the slurry stream, both the leading and trailing edges clearing the stream in the same path.
- d) The cutter shall cut the slurry stream in a plane normal to, or along an arc normal to, the main trajectory path of the stream.
- e) The cutter shall travel through the slurry stream at near-uniform speed, i.e. the speed shall not deviate by more than 10% from the average speed.
- f) The geometry of the cutter opening shall be such that the cutting time at each point in the stream is nearly equal, not deviating by more than 10 %.
- g) The cutting aperture of the cutter shall be at least three times the nominal top size of the particles in the slurry stream, subject to an absolute minimum of 10 mm.
- h) The cutter shall 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.

12.3.3 Cutter velocities

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

- a) biasing of the sample due to deflection of the larger particles;
- b) excessive turbulence, which needs to be avoided to minimize the risk of rebounding slurry and splashes 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 can be introduced if the cutter speed exceeds 0,6 m/s.

Irrespective of the cutter speed and aperture, cutters shall be shown to be free from unacceptable bias.

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 Clause 8.

12.6 Routine checking

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

13 Manual sampling from moving streams

13.1 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, it is necessary that the safety codes of the appropriate regulatory authorities be respected.

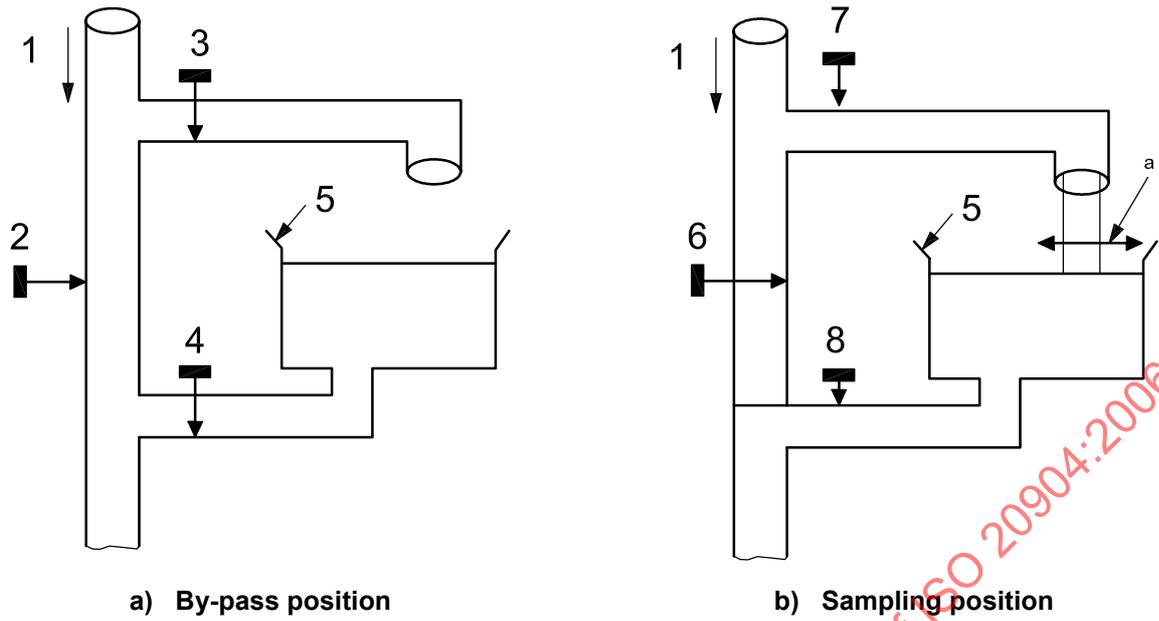
Manual sampling from moving streams should not be used for sampling slurries above a maximum flow rate that takes into account the mass of each increment (typically 100 tph).

13.2 Choosing the sampling location

The sampling location shall

- a) afford complete operator safety;
- b) afford access to the complete slurry stream;
- c) allow no apparent visual segregation of the slurry stream;
- d) be as close as possible to the point where the quality characteristics are 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 surge tank. The full stream can then be manually sampled as it flows into the surge tank.



Key

- 1 slurry flow
- 2 gate valve A
- 3 gate valve B closed
- 4 gate valve C
- 5 surge tank
- 6 gate valve A closed
- 7 gate valve B open
- 8 gate valve C open
- a Motion of manual sampling implement.

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

13.3 Sampling implements

Manual sampling from moving streams shall be carried out using ladles or manual sample cutters. The design criteria for mechanical sample cutters apply (see 12.3.2). Examples of suitable implements are given in Annex C.

13.4 Mass of solids in increments

The mass of solids contained in each increment obtained in one pass of the sample cutter is specified in 6.3.

13.5 Number of primary increments

The number of primary increments to be taken is specified in Clause 8.

13.6 Sampling procedures

The increment shall be taken in a single operation, moving the implement across the full width of the slurry stream at a uniform speed, avoiding overflow of the implement before it leaves the slurry stream. The cutting aperture of the implement shall be perpendicular to the slurry stream. The implement shall cut a complete cross-section of the slurry stream, with both the leading and trailing edges clearing the stream in the same path. Alternate increments shall be taken by traversing the stream in opposite directions.

14 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.

15 Sample preparation procedures

15.1 General

Lot and sub-lot samples shall be dewatered using a vacuum filtration device and then dried in an oven at the temperature specified in the relevant International 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 (see ISO 13909-4) prior to filtration and drying, provided the samples are completely re-pulped prior to division and the solids contents of the divided samples conform to the minimum sample mass requirements of Clause 9.

Samples may be prepared for the following purposes:

- a) general analysis;
- b) size analysis;
- c) other tests, e.g. solids content and relative density.

15.2 Reduction mills

When the solids contained in the slurry are still relatively coarse, e.g. a nominal top size of 4 mm, the nominal top size of the chemical analysis sample shall be reduced to 212 μm before division using an appropriate mill as specified in ISO 13909-4. The mill used shall be designed such that the product particle size can be achieved without using extreme settings. Loss of sample or retention of material from previous samples that can contaminate succeeding samples shall be minimized. Heating of the sample and air-stream effects shall also be minimized.

There shall be no contact between the metal surfaces to avoid local heating of the sample. Totally closed, high-speed (> 20 Hz) ball mills shall not be used. The particle size of the output is influenced by the hardness of the coal, but the effect depends on the speed range.

15.3 Sample division

After drying and particle size reduction (if necessary), division of lot and sub-lot samples shall be carried out in accordance with the requirements of ISO 13909-4 and the minimum sample mass requirements of Clause 9. Suitable dividers include rotary sample dividers and riffle dividers.

15.4 Chemical analysis samples

Laboratory samples of typically 60 g to 300 g at 212 μm nominal top size are extracted from lot and sub-lot samples for chemical analysis depending on the analysis requirements.

15.5 Physical test samples

Laboratory samples for physical testing include samples for determination of size distribution and relative density. The samples should be prepared as specified in the applicable test procedure.

16 Packing and marking of samples

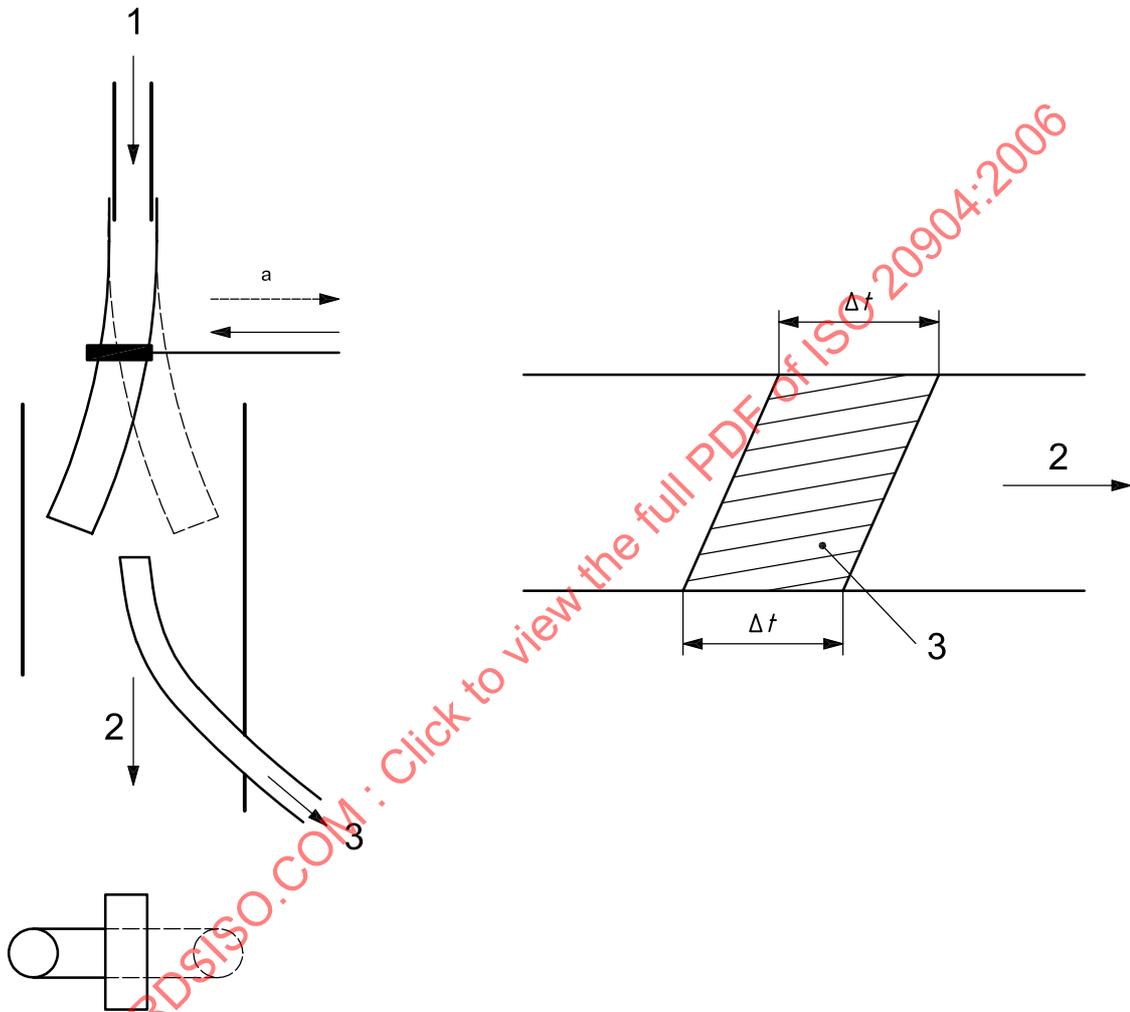
Samples for further preparation and/or analysis should be placed in airtight containers, with relevant information shown on the label and on a card placed in the container. The following are examples of such information:

- a) identification of the lot, e.g. shift;
- b) identification of sampler;
- c) type, quality and nominal top size of the solids content of the slurry;
- d) time duration of the lot or sub-lot;
- e) sample number or portion of the lot or sub-lot the sample represents;
- f) place and date of sampling;
- g) method of sampling, e.g. mechanical or manual;
- h) any special purpose or test for which the sample is taken.

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Annex A
(informative)

Examples of correct slurry devices



Key

- 1 falling stream
- 2 stream
- 3 increment

^a Hose trajectory.

Figure A.1 — Illustration of a correctly designed hose-type slurry cutter^[6]