BS ISO 16742:2014

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Iron ores — Sampling of slurries

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National foreword

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Iron ores — Sampling of slurries

Minerais de fer — Échantillonnage des schlamms

Reference number
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Contents

Page

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 1, *Sampling*.

INTERNATIONAL STANDARD ISO 16742:2014(E)

Iron ores — Sampling of slurries

WARNING — This International Standard may involve hazardous materials, operations, and equipment, and does not purport to address all the safety issues associated with its use. 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 fine iron ore of nominal top size <1 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 (about 80 % solids depending on the particle size distribution of the solids), the mixture is correctly termed a paste. Sampling of pastes is not covered in this International Standard.

The procedures described in this International Standard apply to sampling of iron ore 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 pressurized pipes is not covered in this International Standard. The slurry stream can only be sampled satisfactorily at a transfer point prior to the pressurized pipe at the end of the pipe when the slurry is no longer under pressure. In addition, 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 filtration of the slurry sample, 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, 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 describe 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, ensuring that all parts of the stream are diverted into the vessel for the same period of time. This International Standard corresponds to the stopped-belt method described in ISO 3082. Reference increments have to be taken as close as possible to increments taken using the sampling procedure under evaluation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3082, *Iron ores — Sampling and sample preparation procedures*

ISO 3084, *Iron ores — Experimental methods for evaluation of quality variation*

ISO 3085, *Iron ores — Experimental methods for checking the precision of sampling, sample preparation and measurement*

ISO 3087, *Iron ores — Determination of the moisture content of a lot*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

4 General considerations for sampling slurries

4.1 Basic requirements

In this International Standard, a slurry is defined as iron ore of nominal top size <1 mm that is mixed with water, which is frequently used as a convenient form to transport iron ore 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 tailings dams. In many of these operations, collection of increments at selected sample points is required for evaluation of the iron ore in the slurry.

A gross or partial sample is constituted from a set of unbiased primary increments from a lot. The sample containers and their contained combined increments are weighed immediately after collection to avoid water loss by evaporation or spillage. Weighing is necessary to determine the percentage of solids mass fraction in the gross sample. The gross or partial sample may then be filtered, dried, and weighed. Alternatively, the gross or partial sample can be sealed in plastic bags after filtering for transport and drying at a later stage.

Test samples are prepared from gross or partial samples after filtering and drying, after breaking up any lumps that have formed during drying using a lump breaker, or forcing the sample through a sieve of appropriate aperture. Test portions may then be taken from the test sample and analysed using an appropriate 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, all parts of the slurry in the lot must have an equal opportunity of being selected and appearing in the gross sample for testing. Any deviation from this basic requirement can result in an unacceptable loss of trueness. 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 7). If the slurry flow rate and the solids concentration vary with time, the slurry volume and the dry solids mass 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 8).

Best practice for sampling slurries is to mechanically cut free-falling streams (see Clause 9), with a complete cross section of the stream being taken during the traverse of the cutter. Access to free-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 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 will have significant particle concentration gradients across the pipe and there may 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 and is not covered in this International Standard, 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 gross 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

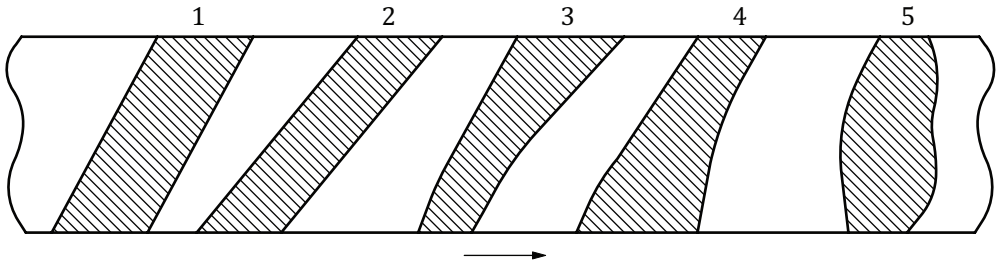
The processes of sampling, sample preparation, and measurement are experimental procedures, and each procedure has its own uncertainty appearing as variations in the final results. When the average of these variations is close to zero, they are called random errors. More serious variations contributing 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.

Sampling from moving slurry streams usually involves methods that fall into three broad operational categories as follows:

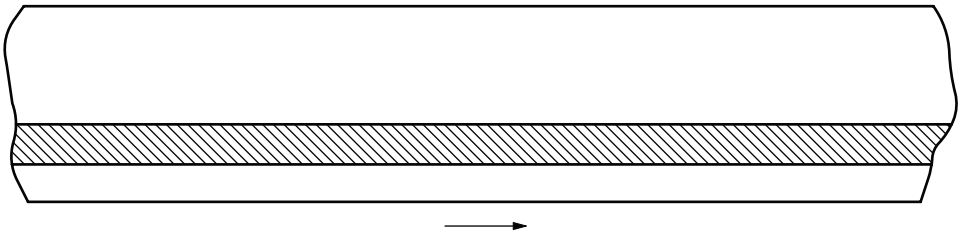
a) taking the whole stream part of the time with a cross-stream cutter as shown in Figure 1a) (based on Reference [4]), usually when the slurry falls from a pipe or over a weir or step. Cuts 1 and 2 show correct sampling with the cutter diverting all parts of the stream for the same length of time. Cuts 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 1b) (based on Reference [4]) with an in-stream point sampler or probe within a pipe or channel, which is always incorrect;

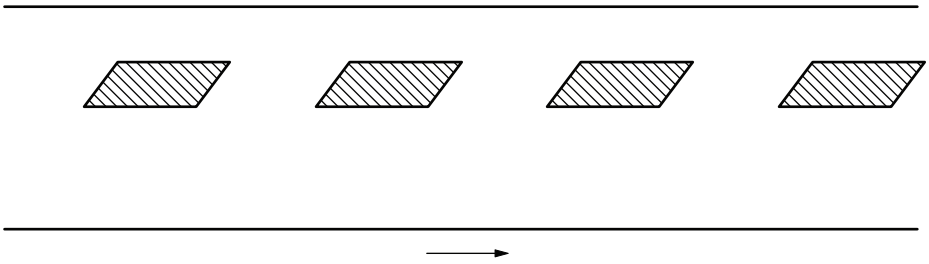
c) taking part of the stream part of the time as shown in Figure 1c) (based on Reference [4]), also with an in-stream point sampler or probe within a pipe or channel, which is always incorrect.



a) Taking all of the stream part of the time



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



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

Key

1 correct
2 correct
3 incorrect
4 incorrect
5 incorrect

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

4.3 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 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 quality variation in step (d), particularly when 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 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/or partial samples, it can be necessary to adopt a poorer precision.

d) Determine the quality variation of the contained solids in the slurry and the precision of preparation and measurement for the quality characteristics under consideration (see 5.5).

e) Determine the number of increments required to attain the desired precision (see 5.6).

f) Ascertain the apparent density of the solids in the slurry and the percentage solids mass fraction in the slurry for determining the mass of the solids in each slurry increment (see 5.2).

g) Check that the procedures and equipment for taking slurry increments minimize bias (see 5.1).

h) Determine the sampling interval in minutes for time-basis systematic sampling (see Clause 7) or stratified random sampling within fixed time intervals (see Clause 8).

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

During sampling operations, partial samples can be combined to constitute a single gross sample for analysis (see Figure 2). Alternatively, increments can be used to constitute partial samples for analysis, which will also improve the overall precision of the measured quality characteristics of the lot. Other reasons for separate preparation and analysis of partial samples are

— for convenience of materials handling,

— to provide progressive information on the quality of the lot, or

— to provide reference or reserve samples after division.

Each increment may also be analysed separately to determine the increment variance of quality characteristics of the lot. In addition, assuming there is no correlation between adjacent increments, 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 partial or gross samples A and B from which two test samples are prepared and analysed. A substantial number of sample pairs is required (preferably at least 20) to obtain a reliable estimate of precision (see ISO 3085).

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 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 6.2).

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.*

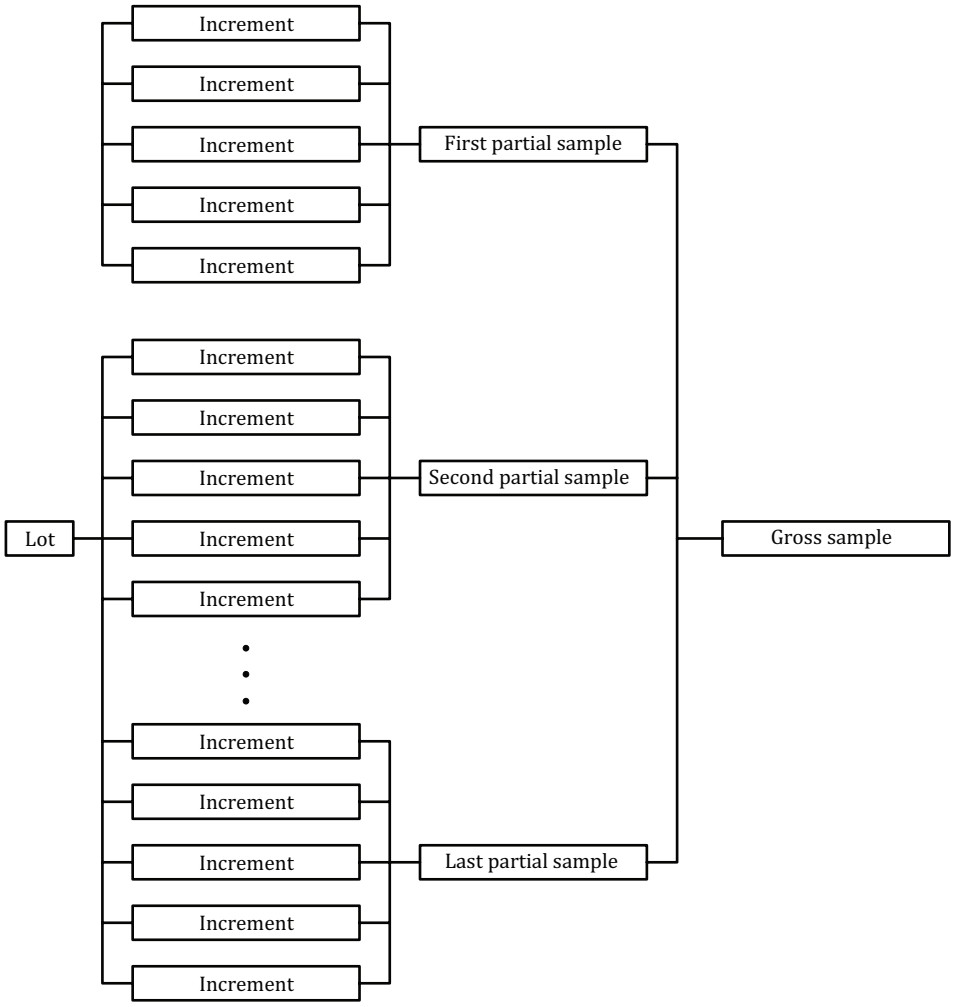


Figure 2 — Example of a sampling plan where a single gross sample is constituted for analysis

5 Fundamentals of sampling and sample preparation

5.1 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 apparent 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 which is used for chemical or physical testing, irrespective of the size, mass, shape, or apparent density of individual particles in the slurry. In practice, this means that a complete cross-section of the slurry must be taken when sampling from a moving stream, otherwise bias is easily introduced.

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 rotary cutters shall be radial from the axis of rotation, and these conditions shall be maintained as the cutter lips wear.

f) The sample cutter shall travel through the slurry stream at uniform speed, not deviating by more than ± 5 % at any point.

g) The angle of cutter chutes, sample chutes, and sample pipes shall be a minimum of 70° to the horizontal.

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 5.2). 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 the cutter aperture, cutter speed, and slurry flow rate for falling-stream cutters (see 5.2). 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 the cutter aperture is partially blocked and the sampling system should be investigated.

5.2 Volume of increment for falling stream samplers to avoid bias

5.2.1 Linear cross-stream cutter

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

$$G_{\mathrm{l}} = \frac{q l_1}{v_{\mathrm{c}}} \tag{1}$$

where

G_{l} is the volume of increment, in cubic metres;

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

l_1 is the cutting aperture of the sampler, in metres;

v_{c} is the cutter speed, 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 9.3.2 and 9.3.3). These limits in turn impose a lower limit on the volume of increment calculated using Formula (1) that needs to be collected to minimize bias.

From the volume of increment calculated using Formula (1), the mass of solids contained in the slurry increment can then be calculated using Formula (2):

$$m_{\mathrm{I}} = \frac{G_{\mathrm{I}}\,\rho_{\mathrm{S}}\,x}{100} \tag{2}$$

where

m_{I} is the mass of solids contained in the increment, in kilograms;

ρ_{S} is the slurry density, in kilograms per cubic metre;

x is the percentage solids mass fraction in the slurry.

5.2.2 Vezin cutter

At any sampling stage, the volume of each slurry increment taken by a Vezin cutter can be calculated as follows:

$$G_{\mathrm{I}} = \frac{q\theta}{6R} \tag{3}$$

where

G_{I} is the volume of increment, in cubic metres;

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

θ is the cutter aperture opening, in degrees;

R is the rotating speed of the cutter, in revolutions per minute.

Once again, there are strict limits on the minimum cutter aperture and the maximum cutter speed to ensure the cutter takes an unbiased sample (see 9.3.2 and 9.3.3). These limits, in turn, impose a lower limit on the volume of increment calculated using Formula (3) that needs to be collected to minimize bias.

From the volume of increment calculated using Formula (3), the mass of solids contained in the slurry increment can then be calculated using Formula (2).

5.3 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.

5.4 Overall precision

This International Standard is designed to attain the overall precision, β_{SPM}, at a probability level of 95 %, given in Table 1 for the total iron, silica, aluminia, phosphorus contents and the percent size fraction of the lot. The precision shall be determined in accordance with ISO 3085.

Table 1 — Overall precision, β_{SPM}

Quality characteristics		Approximate overall precision, β_{SPM} mass fraction, %					
		Mass of solids in the lot t					
		Over 100 000	70 000 to 100 000	45 000 to 70 000	30 000 to 45 000	15 000 to 30 000	Less than 15 000
Iron content		0,38	0,40	0,42	0,45	0,49	0,55
Silica content		0,38	0,40	0,42	0,45	0,49	0,55
Aluminia content		0,13	0,14	0,15	0,16	0,18	0,20
Phosphorus content		0,003 7	0,003 8	0,004 0	0,004 2	0,004 5	0,004 8
Size – Pellet feed	−45 µm fraction, mean 70 %	1,85	1,95	2,0	2,1	2,2	2,5
NOTE The values of β_{SPM} for iron, silica, aluminia, and phosphorus content, as well as sizing, are indicative and subject to confirmation through international test work.							

NOTE The overall precision for other physical characteristics and metallurgical properties is not specified in this International Standard, because they are used to qualitatively compare the behaviour of iron ore slurries during handling and reduction processes.

The overall precision, β_{SPM}, is a measure of the combined precision of sampling, sample preparation, and measurement, and is twice the standard deviation of sampling, sample preparation, and measurement, σ_{SPM}, expressed as an absolute percentage, i.e.

$$\sigma_{SPM} = \sqrt{\sigma_S^2 + \sigma_P^2 + \sigma_M^2} \tag{4}$$

$$\beta_{SPM} = 2\sigma_{SPM} = 2\sqrt{\sigma_S^2 + \sigma_P^2 + \sigma_M^2} \tag{5}$$

$$\sigma_S = \frac{\sigma_W}{\sqrt{n_1}} \tag{6}$$

where

σ_S is the sampling standard deviation;

σ_P is the sample preparation standard deviation;

σ_M is the measurement standard deviation;

σ_W is the quality variation of the slurry;

n_1 is the number of primary increments.

Formulae (4), (5), and (6) are based on the theory of stratified sampling. The number of primary increments to be taken for a lot is dependent on the sampling precision required and on the quality variation of the slurry to be sampled. Thus, before the number of primary increments can be determined, it is necessary to define:

a) the sampling precision, β_S, to be attained;

b) the quality variation, σ_W, of the slurry to be sampled.

When online sample preparation takes place within the sample plant away from the preparation laboratory, the distinction between the terms sampling and sample preparation becomes unclear. The precision of online sample preparation can be included in either the sampling precision or in the sample preparation precision. The choice depends on how easy it is to separate the precision of secondary and tertiary sampling from that of primary sampling. In any event, sample preparation also constitutes a sampling operation, because a representative part of the sample is selected for subsequent processing.

The most rigorous approach is to break up the sampling standard deviation into its components for each sampling stage, in which case Formula (4) becomes:

$$\sigma_{SPM} = \sqrt{\sigma_{S1}^2 + \sigma_{S2}^2 + \sigma_{S3}^2 + \sigma_P^2 + \sigma_M^2} \tag{7}$$

where

σ_{S1} is the sampling standard deviation for primary sampling;

σ_{S2} is the sampling standard deviation for secondary sampling;

σ_{S3} is the sampling standard deviation for tertiary sampling.

Using this approach, the precision of each sampling stage can be separately determined and optimized, resulting in a fully optimized sampling and sample preparation regime.

5.5 Quality variation

The quality variation, σ_W, is a measure of the heterogeneity of the lot and is the standard deviation of the quality characteristics of increments within strata. The characteristics to be selected for determining quality variation include the iron, silica, aluminia, and phosphorus contents and the percentage of a given size fraction.

The value of σ_W shall be measured experimentally for each type or brand of iron ore slurry and for each handling plant under normal operating conditions in accordance with ISO 3084 which assumes there is no serial correlation between adjacent increments. The quality variation of the iron ore slurry can then be classified into three categories according to its magnitude as specified in Table 2.

Table 2 — Classification of quality variation, σ_W

Quality characteristics		**Classification of quality variation,** σ_W mass fraction, %		
		Large	Medium	Small
Iron content		$\sigma_W \geq 2,0$	$2,0 > \sigma_W \geq 1,5$	$\sigma_W < 1,5$
Silica content		$\sigma_W \geq 2,0$	$2,0 > \sigma_W \geq 1,5$	$\sigma_W < 1,5$
Aluminia content		$\sigma_W \geq 0,6$	$0,6 > \sigma_W \geq 0,4$	$\sigma_W < 0,4$
Phosphorus content		$\sigma_W \geq 0,015$	$0,015 > \sigma_W \geq 0,011$	$\sigma_W < 0,011$
Size of pellet feed	−45 µm fraction mean 70 %	$\sigma_W \geq 3$	$3 > \sigma_W \geq 2,25$	$\sigma_W < 2,25$

For iron ore slurries whose quality variation is unknown, measurements shall be conducted at the earliest opportunity in accordance with ISO 3084 to determine the quality variation. Prior to the determination of quality variation, the classification adopted shall be as follows:

a) when no prior information exists on the quality variation of the slurry or similar slurries, the slurry shall be considered to have "large" quality variation;

b) when prior information exists on the quality variation of a similar slurry, the quality variation classification of that slurry shall be adopted as the starting point.

When separate samples are taken for the determination of chemical composition, moisture content and size distribution, the quality variation for the individual characteristics shall be adopted. When separate samples are taken for the determination of other physical characteristics or metallurgical properties not specified in Table 2, large quality variation should be used. When the sample is used for the determination of more than one quality characteristic, the largest classification category for quality variation in Table 2 shall be adopted.

For small lots, it cannot be possible to take the number of increments specified by Formula 8 for large quality variation. In this case, the maximum number of increments possible shall be taken, but in an attempt to compensate for the poorer sampling precision, the precision of sample preparation shall be improved to achieve the required overall precision β_{SPM}, e.g. by preparing and analysing more partial samples. The procedure adopted should be recorded in the sampling report.

5.6 Sampling precision and number of primary increments

When the value of σ_W is known, the number of primary increments, n_1, can be calculated for the desired sampling precision, β_S, as follows:

$$n_1 = \left(\frac{2\sigma_w}{\beta_S}\right)^2 \tag{8}$$

This is the preferable method of determining the number of primary increments. However, when the value of σ_W is classified in terms of large, medium, or small quality, variation in accordance with Table 2 and Table 3 can be used to determine the minimum number of primary increments required for the sampling precision, β_S, specified. In Table 3, the levels of sampling precision have been increased slightly for smaller lot sizes as a trade-off between sampling cost and the uncertainty in the value of the lot.

Table 3 — Example of minimum number of increments required, n_1, for desired sampling precision, β_S

Mass of lot (1 000 t)		**Sampling precision** β_S				**Number of primary increments** n_1		
Over	Up to	Fe or SiO_2 content	Al_2O_3 content	P content	Pellet feed, −45 µm fraction	Quality variation Large (L), medium (M), or small (S)		
						L	M	S
100	100	0,35	0,10	0,002 6	0,53	200	100	50
70	70	0,37	0,11	0,002 7	0,56	180	90	45
45	45	0,39	0,11	0,002 9	0,59	160	80	40
30	30	0,42	0,12	0,003 1	0,63	140	70	35
15	15	0,45	0,13	0,003 4	0,68	120	60	30
0		0,50	0,14	0,003 7	0,75	100	50	25

NOTE The values of n_1 can be increased or decreased to alter the sampling precision, e.g. if the number of increments is $2n_1$, then β_S will be improved by a factor of $1/\sqrt{2} = 0{,}71$; and if it is $n_1/2$, then β_S will be worsened by a factor of $\sqrt{2} = 1{,}4$.

5.7 Precision of sample preparation and overall precision

The precision of sample preparation depends on the choice of the sample preparation scheme. It can be improved if sample preparation is carried out first on individual increments or partial samples at an appropriate stage of sample preparation and then the divided increments or partial samples are combined into a gross sample.

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

— carrying out multiple determinations on the contained solids in the lot sample, i.e. replicate analyses,

— analysing the contained solids in individual increments, or

— preparing a number of partial samples and analysing the contained solids in each partial sample.

The overall variance in each case is then given by one of the following formulae.

a) Where a single lot sample is constituted from a lot and n_2 replicate determinations on the contained solids are carried out on the lot sample,

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \sigma_{\mathrm{P}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_2} \tag{9}$$

b) Where n_3 partial samples are prepared, each constituted from the contained solids of an equal number of increments, and n_2 replicate determinations are carried out on each partial sample,

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \frac{\sigma_{\mathrm{P}}^2 + \frac{\sigma_{\mathrm{M}}^2}{n_2}}{n_3} \tag{10}$$

c) Where all n_1 primary increments are prepared and a single determination is carried out on the contained solids of each increment,

$$\sigma_{\mathrm{SPM}}^2 = \sigma_{\mathrm{S}}^2 + \frac{\sigma_{\mathrm{P}}^2}{n_1} + \frac{\sigma_{\mathrm{M}}^2}{n_1} \tag{11}$$

6 Minimum mass of solids in gross and partial samples

6.1 General

It is essential to ensure that the mass of solids contained in gross samples collected from the slurry is sufficient to obtain the required sampling precision. Subject to increments being taken in an unbiased manner (see 5.1, 5.2, and 5.3), the combination of the average mass of solids contained in a slurry increment and the number of increments determined in 5.6 will ensure 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), partial samples and gross samples, it is important to ensure that sufficient mass of solids is retained at each stage of division to achieve the desired sampling variance.

6.2 Minimum mass of solids in gross samples

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

However, given that the nominal top size of particles in a slurry are <1 mm (see Clause 1), in practice the minimum mass of solids in the gross sample is determined by the required mass of the chemical analysis sample (100 g) and the required number of samples, i.e. one for the seller, one for the buyer, one for the arbitrator, and one in reserve. Taking this into account, the minimum mass of solids in the gross sample shall be 0,5 kg.

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.

6.3 Minimum mass of solids in partial samples

It is essential that the combined mass of solids contained in all partial samples prepared for the lot is, at each sampling stage, greater than the minimum mass of contained solids in the gross sample defined in 6.2.

7 Time-basis sampling

7.1 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) 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 each lot.

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

7.2 Sampling interval

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

$$\Delta t \leq \frac{t_L}{n_1} \tag{12}$$

where

Δt is time interval between taking increments, in minutes;

t_L is the production time allocated to each lot, in minutes;

n_1 is the number of primary increments determined in 5.6.

7.3 Cutters

The following cutters may be used:

a) falling-stream cutters of the hose type (see 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 Figure A.2), whose cutting speed is constant throughout the sampling of the entire lot;

c) falling stream cutters of the diverter type (see Figure A.3), whose cutting speed is constant throughout the sampling of the entire lot.

NOTE If individual increments are analysed, the mass represented by each increment needs to be recorded to weight the analyses accordingly.

7.4 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 Formula (12) until the end of the lot.

The fixed time interval between increments should be no larger than that calculated using Formula (12) to ensure that the number of increments taken will be at least the minimum number of primary increments specified.

7.5 Constitution of gross or partial samples

Increments are combined to form gross samples or partial samples in either of the following ways.

a) Increments as taken are combined into partial samples or a gross sample irrespective of the variation of masses of solids contained in increments.

b) Increments are divided by proportional division. The gross sample or partial 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.

7.6 Division of increments and partial samples

After time-basis sampling, division of increments and partial samples shall be carried out by proportional division when the divided samples are to be combined. When samples are not to be combined, proportional division or constant-mass division can be used.

7.7 Division of gross samples

Division of gross samples shall be carried out by either constant-mass or proportional division.

7.8 Number of cuts for division

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

a) **For gross samples**: a minimum of 20 cuts. The combined mass of the cuts shall be greater than the minimum mass of contained solids in the gross sample specified in 6.2.

b) **For partial samples**: a minimum of 12 cuts. The combined mass of the contained solids of the cuts from all partial samples at a given sampling stage shall be greater than the minimum mass of contained solids in the gross sample specified in 6.2.

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 shall be greater than the minimum mass of contained solids in the gross sample specified in 6.2.

NOTE Since the sampling precision cannot be determined a *priori*, check experiments are recommended to ascertain whether the number of cuts is sufficient. As a general rule, as many cuts as possible should be taken, preferably using a rotary sample divider.

8 Stratified random sampling within fixed time intervals

For stratified random sampling within fixed time intervals, the strata size Δt is determined using Formula (12). 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, but allowing for the fact that a finite time is required to take an increment. The random number is then used as an input to the program that controls the cutter time sequence.

9 Mechanical sampling from moving streams

9.1 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, although they shall fall within the set of falling-stream cutter types listed in 7.3.

Only mechanical cutters that take a complete cross-section of the 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.

NOTE 1 Annex A gives examples from Reference [4] of correctly designed sample cutters for slurry flows and should be taken as a guide in the choice of suitable equipment with correct increment extraction and delimitation.

NOTE 2 Annex B gives examples from Reference [4] of slurry sampling devices which have incorrect increment extraction and delimitation and should not be used.

9.2 Design of the sampling system

9.2.1 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.

9.2.2 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 which affords access to the complete slurry stream;

b) Sampling shall be performed at a point in the handling system 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.

9.2.3 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 gross or partial samples.

9.2.4 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 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.

9.2.5 Minimizing bias

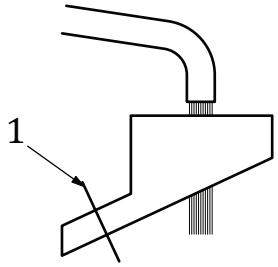
It is essential that the sampling system be designed to avoid the following:

a) Spillage of the sample or loss of material due to dribbles or run-back on the outside of a discharging slurry pipe or underneath a discharging launder (see Figure 3);

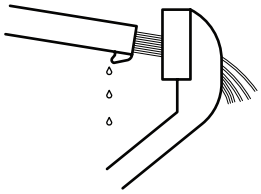
b) Restriction of the flow of the slurry increment through any device causing reflux and overflow. This is particularly important for reverse spoon cutters where the falling slurry stream is forced to change flow direction as it strikes the inside surface of the spoon;

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

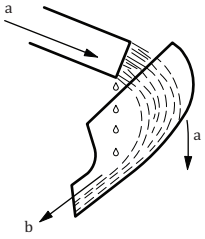
d) Contamination of the sample.



a) Correct



b) Incorrect



c) Correct

Key

1 splash guard

a Stream.

b Increment.

Figure 3 — Examples of correct and incorrect designs for cross-stream slurry cutters

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

NOTE 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.

9.3 Slurry sample cutters

9.3.1 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.

9.3.2 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 5 % 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 5 %.

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 or overflow from the cutter aperture.

9.3.3 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

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, and

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.

9.4 Mass of solids in increments

The mass of solids contained in each increment obtained in one pass of the sample cutter is specified in 5.2.

9.5 Number of primary increments

The number of primary increments to be taken is specified in 5.6.

9.6 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.

10 Manual sampling from moving streams

10.1 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 can 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.

NOTE 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).

10.2 Choosing the sampling location

The sampling location shall

a) afford complete operator safety,

b) afford access to the complete slurry stream, and

c) 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-mentioned 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 4, 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 using a correctly designed sampling implement as the slurry flows into the surge tank as follows:

— open gate valves B and C in Figure 4a);

— close gate valve A in Figure 4a);

— after the slurry flow through the diverter pipe and surge tank in Figure 4b) has stabilized, take an increment from the feed stream to the surge tank using a manual sample cutter and submit the increment for analysis, and finally;

— open gate valve A and then close gate valves B and C.

If necessary, the surge tank should be fitted with a suitable evacuation mechanism to remove residual slurry after taking an increment.

10.3 Sampling implements

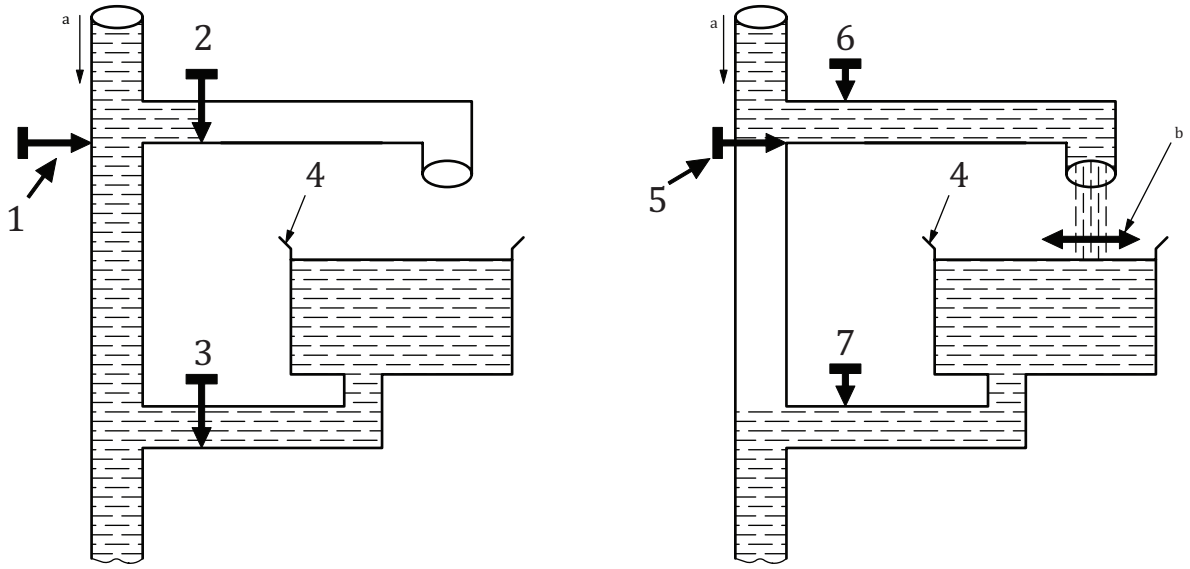
Manual sampling from moving streams shall be carried out using ladles or manual sample cutters. The design criteria for mechanical sample cutters apply (see 9.3.2). Examples of suitable implements are given in Annex C.

10.4 Volume of increments

The volume of each increment obtained in one pass of the sample cutter is specified in 5.2.

10.5 Number of primary increments

The number of primary increments to be taken is specified in 5.6.



a) By-pass position **b) Sampling position**

Key

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

Figure 4 — Sample by-line for manual sampling of slurry in a pipe

10.6 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.

11 Sampling of stationary slurries

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 gross sample for testing. Due to this, sampling of slurries in stationary situations is not covered in this International Standard.

12 Sample preparation procedures

12.1 General

Gross and partial samples shall be dewatered using a vacuum filtration device and then dried in an oven at the temperature specified in ISO 3087 prior to further sample preparation. However, if gross or partial samples are excessively large, then they can be divided using a suitable divider, e.g. a Vezin divider or a riffle, 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 6.

Samples can be prepared for the following purposes:

a) chemical analysis;

b) size analysis;

c) other tests, e.g. solids content and relative density.

12.2 Grinding mills

When the solids contained in the slurry are still relatively coarse, e.g. a nominal top size of 1 mm, the chemical analysis sample shall be ground to –100 µm before division using an appropriate grinder as specified in ISO 3082.

12.3 Sample division

After drying and particle size reduction (if necessary), division of gross and partial samples shall be carried out in accordance with the requirements of ISO 3082 and the minimum sample mass requirements of Clause 6. Suitable dividers include rotary sample dividers and riffle dividers.

12.4 Chemical analysis samples

Laboratory samples of typically 100 g at –100 µm particle size are extracted from gross and partial samples for chemical analysis depending on the analysis requirements. However, for ores containing more than 2,5 % combined moisture and/or oxidizable compounds, where excessive grinding would affect the result, the test sample for chemical analysis shall be –160 µm in particle size and 100 g minimum mass.

12.5 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.

13 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. Examples of the information are as follows.

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.

e) Sample number or portion of the lot the sample represents.

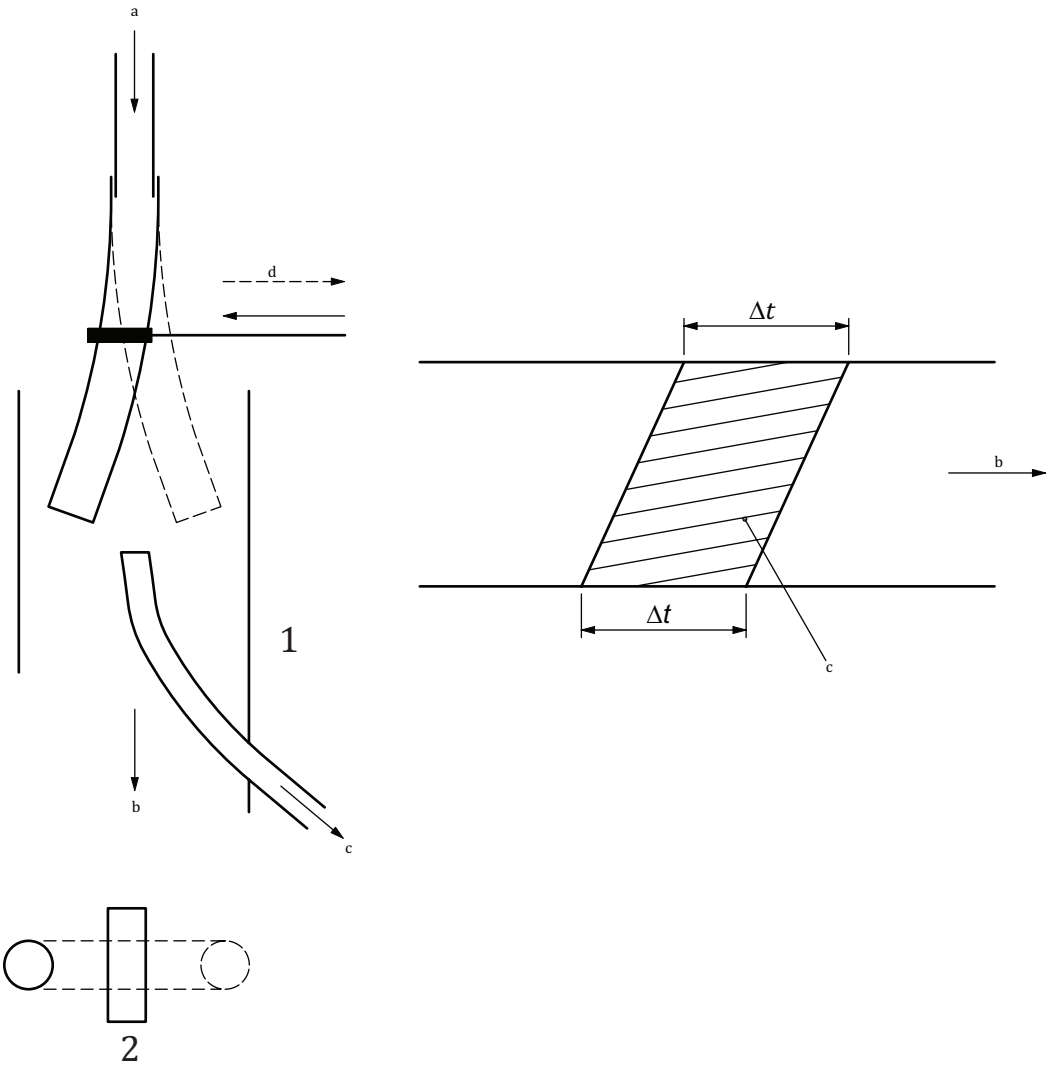
f) Place and date of sampling.

g) Method of sampling, e.g. mechanical or manual.

h) Any special purpose or test for which the sample is taken.

Annex A
(informative)

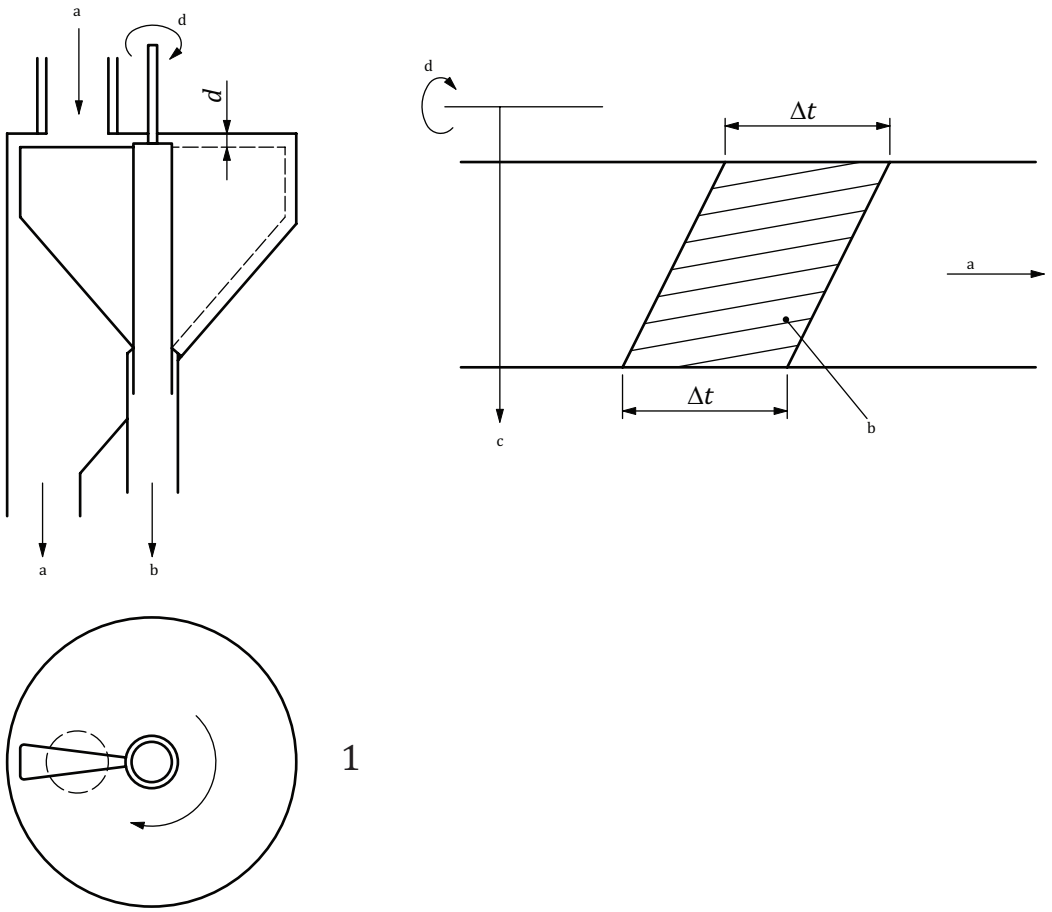
Examples of correct slurry sampling devices



Key

1 side view of the cutter
2 top view of the cutter
a Falling stream.
b Stream.
c Increment.
d Hose trajectory.

Figure A.1 — Illustration of a correctly designed hose-type slurry cutter (Reference [4])



Key

1 top view

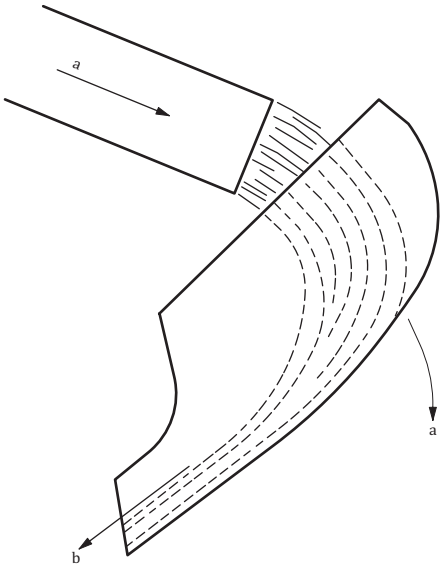
a Stream.

b Increment.

c Cutter trajectory.

d Rotating axis.

Figure A.2 — Correct layout of a circular path falling stream cutter, i.e. a Vezin cutter (Reference [4])



Key

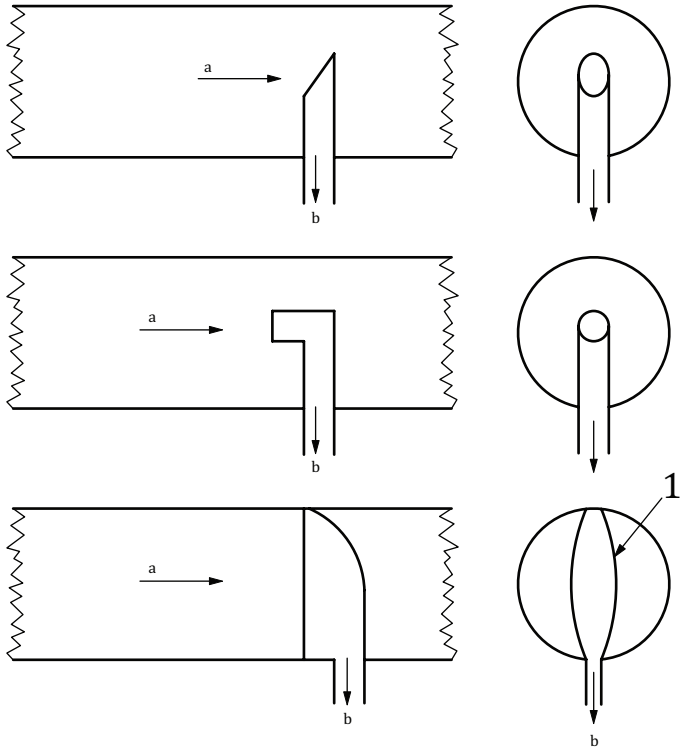
a Stream.

b Increment.

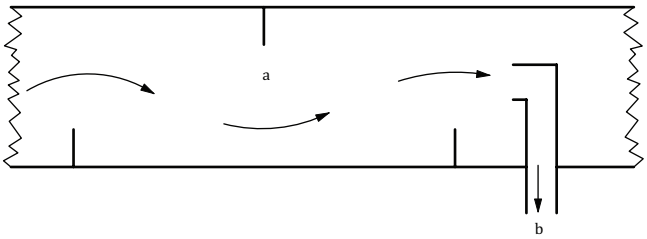
Figure A.3 — Illustration of a correctly designed falling stream slurry cutter (after Reference [4])

Annex B
(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 position prior to a sampling probe – their effectives is questionable

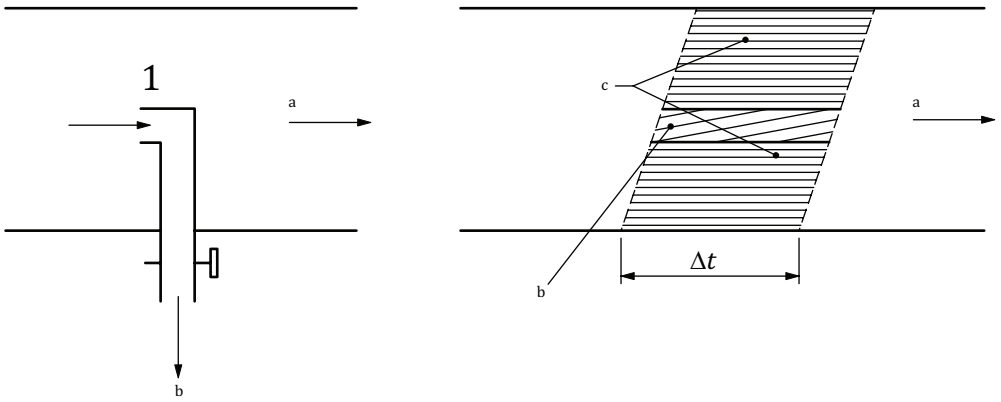
Key

1 slot

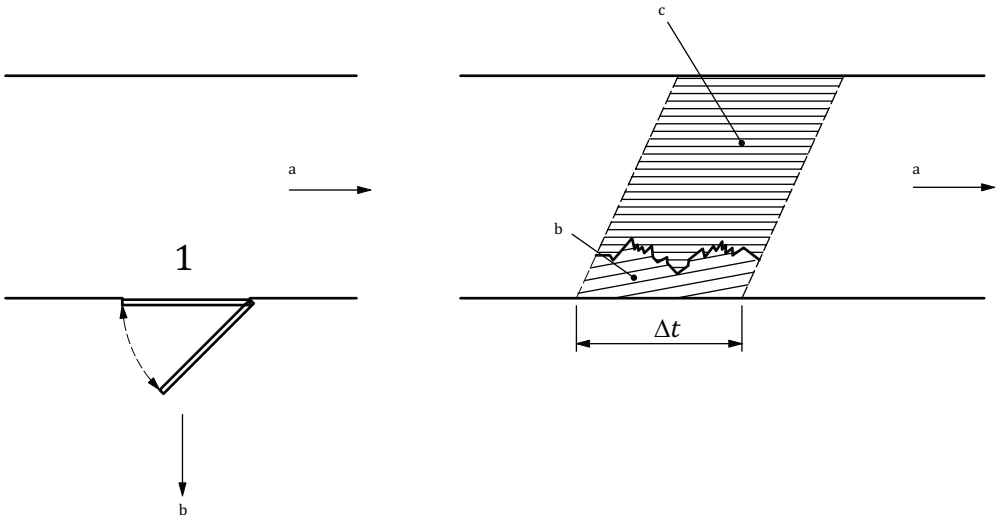
a Stream.

b Sample.

Figure B.1 — In-stream probes (Reference [4])



a) In-stream point sampling – always incorrect



b) By-line slurry sampling – always incorrect

Key

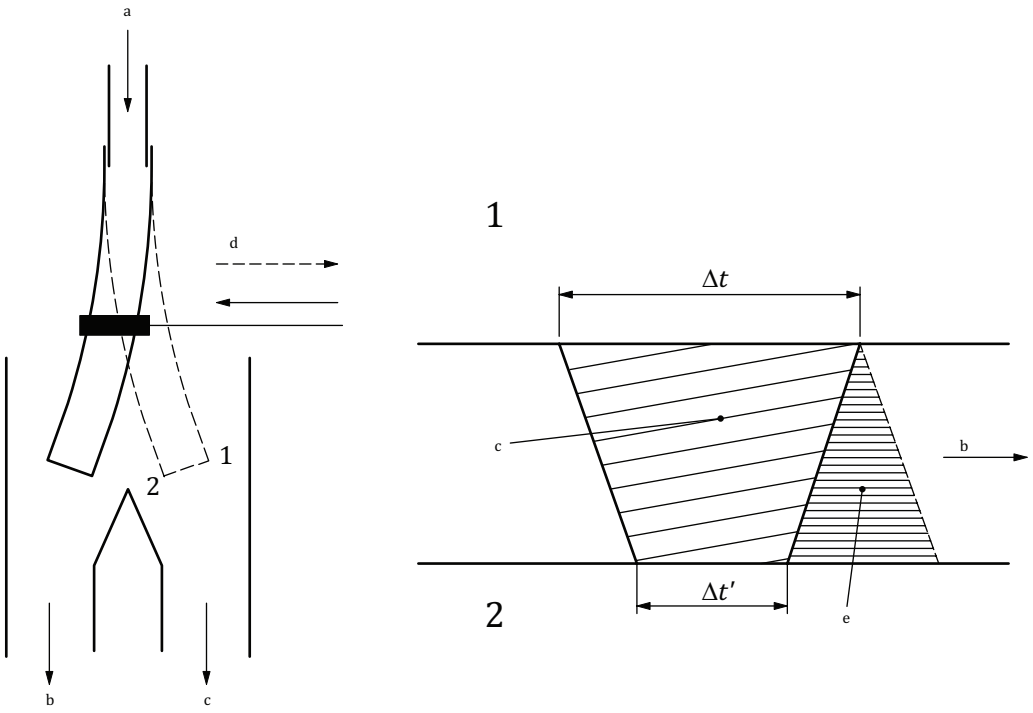
1 sample point

a Stream.

b Increment.

c Missing portion of the increment.

Figure B.2 — Incorrect sample delimitation using an in-stream probe and by-line sampling (Reference [4])



Key

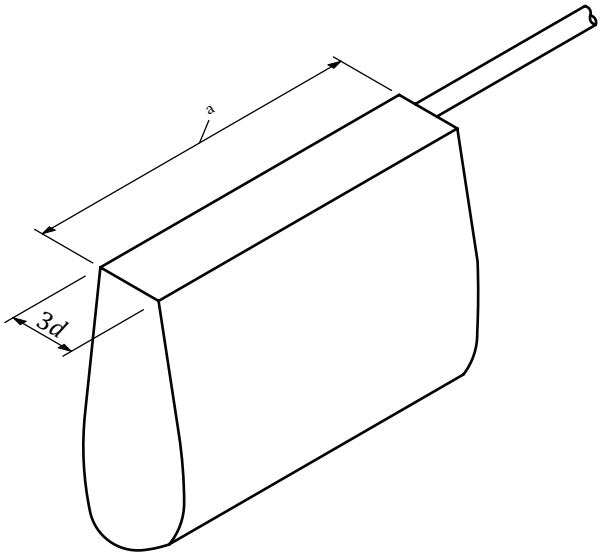
1 on stream idle position
2 increment idle position
a Falling stream.
b Stream.
c Increment.
d Hose trajectory.
e Missing portion of the increment

NOTE This particular design is always incorrect.

Figure B.3 — Flexible hose slurry sampler (Reference [4])

Annex C
(normative)

Manual sampling implements



Key

a To exceed the depth of the falling stream.

Figure C.1 — Example of a manual sample cutter

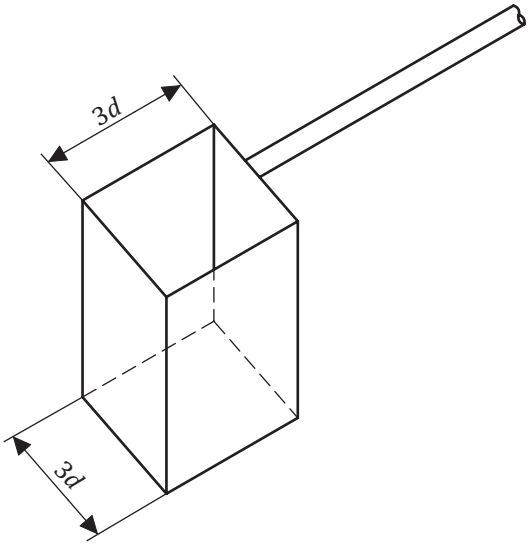


Figure C.2 — Example of a ladle

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[2] ISO 3086, *Iron ores — Experimental methods for checking the bias of sampling*

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