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Iron ores — Sampling of slurries

Minerais de fer — Échantillonnage des schlamms

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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).

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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*.

This corrected version of ISO 16742:2014 incorporates the following correction:

— In Table 3, third row, the values in the second column ("Up to") have been correctly aligned with the corresponding values in the first column ("Over").

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

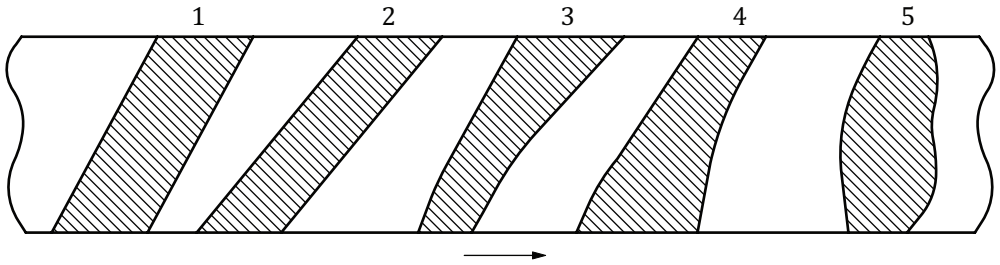
The processes of sampling, sample preparation, and measurement are experimental procedures, and each procedure has its own uncertainty appearing as variations in the final results. When the average of these variations is close to zero, they are called random errors. More serious variations contributing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

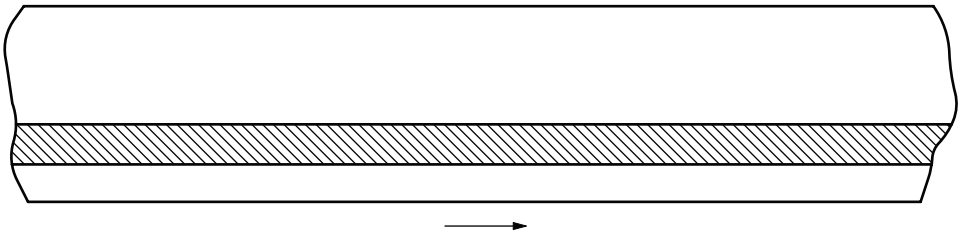
a) taking the whole stream part of the time with a cross-stream cutter as shown in Figure 1a) (based on Reference [4]), usually when the slurry falls from a pipe or over a weir or step. Cuts 1 and 2 show correct sampling with the cutter diverting all parts of the stream for the same length of time. Cuts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

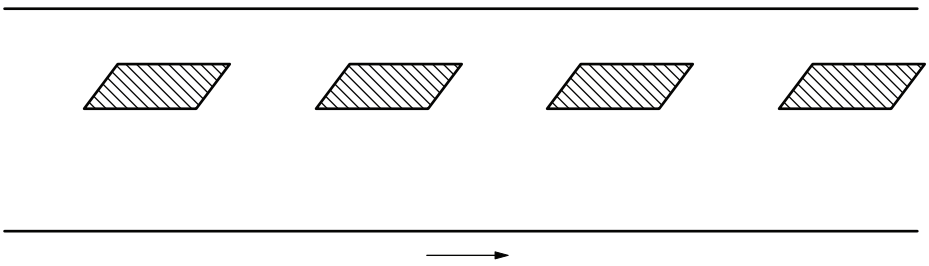
c) taking part of the stream part of the time as shown in Figure 1c) (based on Reference [4]), also with an in-stream point sampler or probe within a pipe or channel, which is always incorrect.



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.*

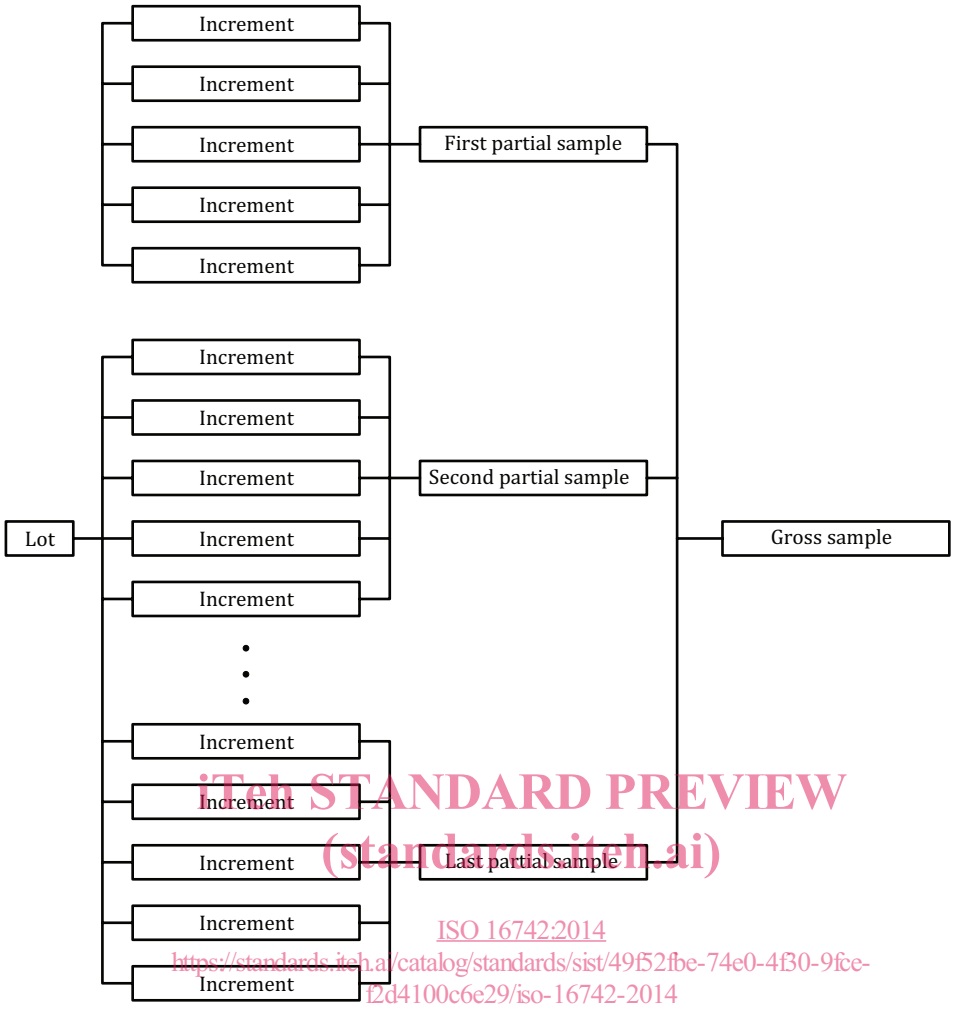


Figure 2 — Example of a sampling plan where a single gross sample is constituted for analysis

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.