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

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

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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 voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the 2010 edition (ISO 11794:2010), of which it constitutes a minor revision. The main changes are the deletion of reference ISO 20212, which has not yet been published, and the replacement of “should” with “shall” where the criteria and/or requirements are mandatory.

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

1 Scope

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

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

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

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

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

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

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Principles of sampling slurries

4.1 General

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

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

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

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

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

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

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

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

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

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

4.2 Sampling errors

4.2.1 General

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

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

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

where

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

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

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

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

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

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

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

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

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

where

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

GE is the segregation and grouping error.

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

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

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

4.2.2 Preparation error, PE

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

4.2.3 Delimitation and extraction errors, DE and EE

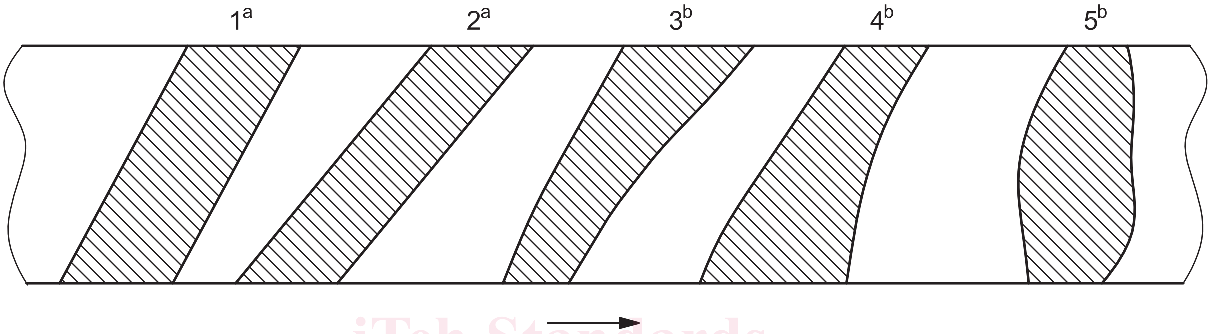
Delimitation and extraction errors arise from incorrect sample cutter design and operation. The increment delimitation error, DE, results from an incorrect shape of the volume delimiting the slurry increment, and this can be due to both design and operation faults. Because of the incorrect shape of the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

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

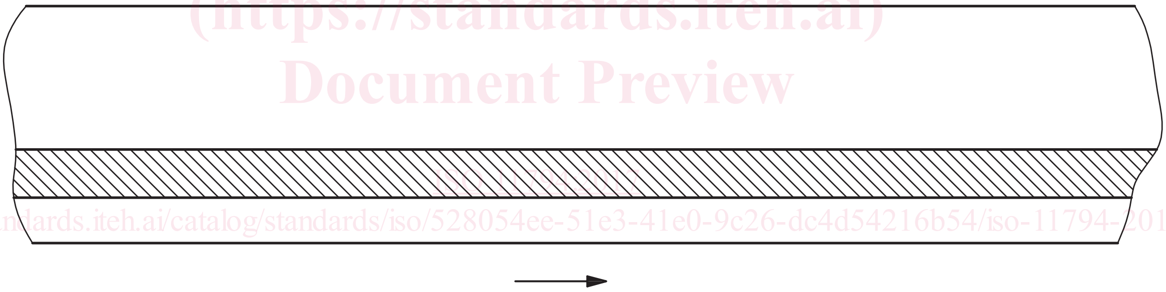
a) Taking the whole stream for part of the time with a cross-stream cutter as shown in Figure 1 a) (after Pitard, 1993), usually where the slurry falls from a pipe or over a weir or step. Cuts 1 and 2 show correct sampling with the cutter diverting all parts of the stream for the same length of time.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

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

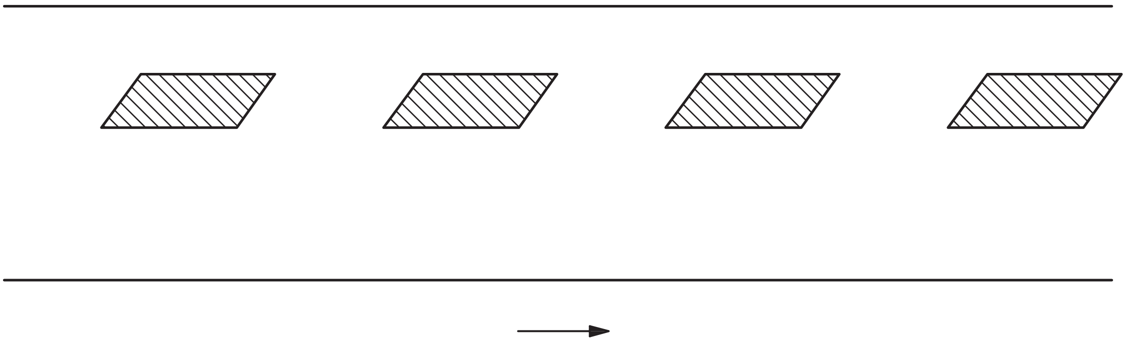
c) Taking part of the stream part of the time as shown in Figure 1 c) (after Pitard, 1993), also with an on-stream point sampler or probe within a pipe or channel, which is always incorrect.



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



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



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

Key

a Correct.

b Incorrect.

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

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

4.2.4 Weighting error, WE

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

4.2.5 Periodic quality-fluctuation error, QE_3

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

4.3 Sampling and total variance

4.3.1 Sampling variance

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

$$\mathrm{TSE} = \mathrm{QE}_1 + \mathrm{QE}_2 \tag{3}$$

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

$$s_{\mathrm{S}}^2 = s_{\mathrm{QE1}}^2 + s_{\mathrm{QE2}}^2 \tag{4}$$

The short-range quality-fluctuation variance, s_{QE1}^2, arises from the different internal composition of increments taken at the shortest possible interval apart. This is a local or random variance due to the particulate nature of the solids in the slurry.

The long-range quality-fluctuation variance, s_{QE2}^2, arises from the continuous trends in quality that occur while sampling a slurry and is usually space- and time-dependent. This component is often the combination of a number of trends generated by diverse causes.

4.3.2 Total variance

Assuming that sources of bias have either been eliminated or minimized, the next objective of a sampling scheme is to provide one or more test portions, sufficiently representative of a lot, for determination of the quality characteristics of the lot with good precision, i.e. low variance. The total variance of the final result, denoted by s_{T}^2, consists of the variance of sampling (including sample processing) plus the variance of analysis (chemical analysis, determination of particle-size distribution, etc.) as follows:

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

where

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

s_{A}^2 is the analytical variance.

In Formula (5), the sampling variance includes the variances due to all sampling (and sample processing) steps except selection of the test portion. The variance due to selection of the test portion is included in the analytical variance, s_{A}^2, which is determined in accordance with ISO 12744, because it is difficult to determine separately the "true" analytical variance.

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

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

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

The first method, which was developed by Gy, is to break up the sampling variance into its components for each sampling stage, as specified in Annex A. The total variance is then given by:

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

where

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

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

$s_{\mathrm{S}_{u-1}}^2$ is the sampling variance for stage *u*–1, the second-last stage;

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

This is referred to as the "sampling-stage" method (see 4.3.3) and provides very detailed information on the variance components that is particularly useful for designing and assessing sampling schemes. However, to obtain maximum benefit, it is necessary to collect data at each sampling stage.

The second method, called the "simplified" method (see 4.3.4), is to break up the total variance into primary sampling, sample processing and analytical variances only as follows:

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

where

$s_{S_1}^2$ is the primary sampling variance;

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

s_A^2 is the analytical variance, including selection of the test portion [at stage *u* in Formula (7)].

The primary sampling variance is identical to the sampling variance for stage 1 in Formula (7), while s_P^2 is equal to the total sampling variance for the remaining sampling stages, except for selection of the test portion which is included in the analytical variance. The relative magnitudes of the variance components in Formula (8) indicate where additional effort is required to reduce the total variance. However, it is not possible to separate the variances of the separate sample-processing stages. This method is suitable for estimating the total variance for new sampling schemes based on the same sample-processing procedures, where the numbers of primary increments, sample processings and analyses are varied.

Finally, the total variance s_T^2 can be estimated experimentally by collecting interleaved duplicate samples (see 4.3.5). This is called the “interleaved sample” method and gives valuable information on the total variance actually achieved for a given sampling scheme with no extra effort, provided facilities are available for collecting duplicate samples (Merks, 1986). It gives no information on variance components, but the total variance can be compared with the analytical variance to ascertain whether the sampling scheme used is optimized or not. It is therefore of limited use for designing sampling schemes, but it can be used to monitor whether a sampling scheme is in control.

4.3.3 Sampling-stage method of estimating sampling and total variance

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

$$s_{S_i}^2 = \frac{s_{b_i}^2}{n_i} \tag{9}$$

where

$s_{b_i}^2$ is the variance between increments for stage *i*;

n_i is the number of increments for stage *i*.

The variance between increments for stage *i*, $s_{b_i}^2$, can be estimated using the following equation:

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

where

x_j is the test result for increment *j*;

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

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