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Hard coal — Sampling of slurries

Houille — Échantillonnage des schlamms

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

Hard coal — Sampling of slurries

1 Scope

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

The procedures described in this International Standard primarily apply to sampling of coal that is transported in moving streams as a slurry. These streams can fall freely or be confined in pipes, launders, chutes, spirals or similar channels. Sampling of slurries in stationary situations, such as a settled or even a well-stirred slurry in a tank, holding vessel or dam, is not recommended and is not covered in this International Standard.

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

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

Provided flow rates are not too high, the reference method against which other sampling procedures are compared is one where the entire stream is diverted into a vessel for a specified time or volume interval. This method corresponds to the stopped-belt method described in ISO 13909-2.

2 Normative references

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

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

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

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

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

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

3 Definitions

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

4 Principles of sampling slurries

4.1 General

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

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

Except for samples for which their characteristics are determined directly on the slurry, test samples are prepared from lot or sub-lot samples after filtering and drying. Test portions may then be taken from the test sample and analysed using an appropriate and properly calibrated analytical method or test procedure under prescribed conditions.

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

If a sampling scheme is to provide representative samples, it is necessary that all parts of the slurry in the lot have an equal opportunity of being selected and appearing in the lot sample for testing. Any deviation from this basic requirement can result in an unacceptable loss of accuracy. A sampling scheme having incorrect selection techniques, i.e. with non-uniform selection probabilities, cannot be relied upon to provide representative samples.

Sampling of slurries should preferably be carried out by systematic sampling on a time basis (see Clause 10). If the slurry flow rate and the coal-solids concentration vary with time, the slurry volume and the dry solids mass for each increment will vary accordingly. It is necessary to show that no systematic error (bias) is introduced by periodic variation in quality or quantity where the proposed sampling interval is approximately equal to a multiple of the period of variation in quantity or quality. Otherwise, stratified random sampling should be used (see Clause 11).

Best practice for sampling slurries is to mechanically cut freely falling streams (see Clause 12), with a complete cross-section of the stream being taken during the traverse of the cutter. Access to freely falling streams can sometimes be engineered at the end of pipes or by incorporating steps or weirs in launders and chutes. If samples are not collected in this manner, non-uniform concentration of coal solids in the slurry due to segregation and stratification of the solids can lead to bias in the sample that is collected. Slurry flow in pipes can be homogenous with very fine particles dispersed uniformly in turbulent suspension along the length and across the diameter of the pipe. However, more commonly, the slurry in a pipe has significant particle-concentration gradients across the pipe and there can be concentration fluctuations along the length of the pipe. These common conditions are called heterogeneous flow. Examples of such flow are full-pipe flow of a heterogeneous suspension or partial-pipe flow of a fine suspension above a slower moving or even stationary bed of coarser particles in the slurry.

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

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

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

4.2 Sampling errors

4.2.1 General

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

The characteristics of the solids component of a slurry can be determined by extracting samples from the slurry stream, preparing test samples and measuring the required quality characteristics. The total sampling error, E_T, can be expressed as the sum of a number of independent components (Gy, 1982[5]; Pitard, 1993[6]). Such a simple additive combination is not possible if the components are correlated. The total sampling error, E_T, expressed as a sum of its components, is given by Equation (1):

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

where

E_{Q1} is short-range quality fluctuation error associated with short-range variations in quality of the solids component of the slurry;

E_{Q2} is long-range quality fluctuation error associated with long-range variations in quality of the solids component of the slurry;

E_{Q3} is periodic quality fluctuation error associated with periodic variations in quality of the solids component of the slurry;

E_W is weighting error associated with variations in slurry flow rate;

E_D is increment delimitation error introduced by incorrect increment delimitation;

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

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

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

$$E_{QI} = E_F + E_G \tag{2}$$

where

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

E_G is the segregation and grouping error.

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

The segregation and grouping error results from the distribution heterogeneity of the sampled material (Pitard, 1993[6]). The distribution heterogeneity of a lot is the heterogeneity arising from the manner in which particles are distributed in the slurry. It can be reduced by taking more increments, but it can never be completely eliminated.

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

4.2.2 Preparation error

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

4.2.3 Delimitation and extraction errors

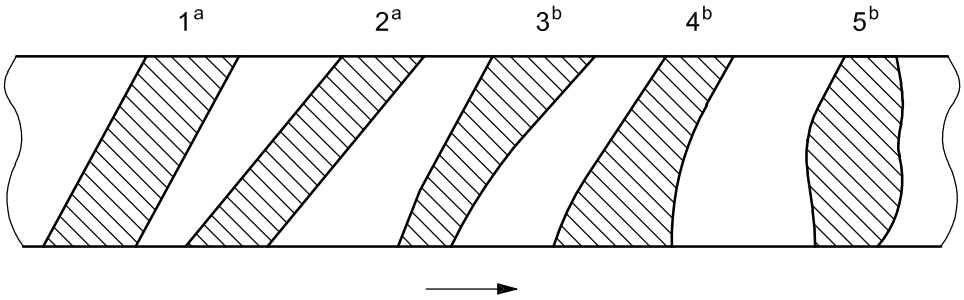
Delimitation and extraction errors arise from incorrect sample cutter design and operation. The increment delimitation error, E_D, results from an incorrect geometry of the volume delimiting the slurry increment (see Figure 1), and this can be due to both design and operation faults. Because of the incorrect shape of the slurry increment volume, sampling with non-uniform selection probabilities results. The average of E_D is often non-zero, which makes it a source of sampling bias. The delimitation error can be made negligible if all parts of the stream cross-section are diverted by the sample cutter for the same length of time.

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

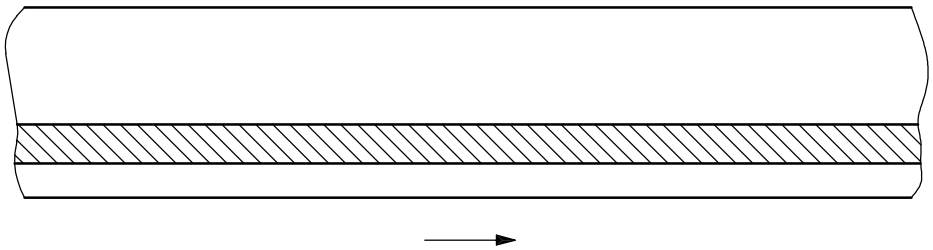
a) taking the whole stream part of the time with a cross-stream cutter as shown in Figure 1 a) (after Pitard, 1993[6]), usually when the slurry falls from a pipe or over a weir or step. Cuts 1 and 2 show correct sampling with the cutter diverting all parts of the stream for the same length of time. Cuts 3, 4 and 5 show incorrect sampling where the cutter diverts different parts of the stream for different lengths of time;

b) taking part of the stream all of the time as shown in Figure 1 b) (after Pitard, 1993[6]) with an in-stream point sampler or probe within a pipe or channel, which is always incorrect;

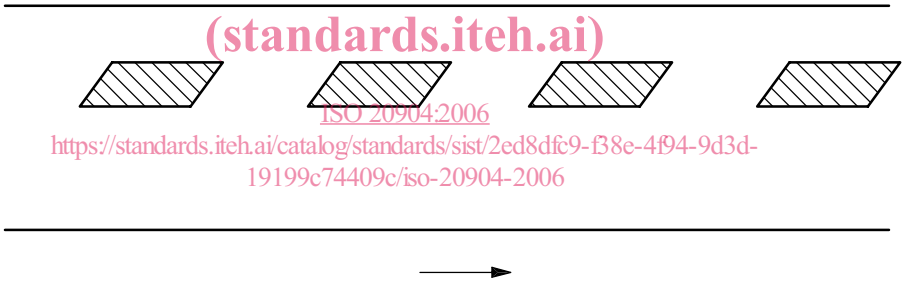
c) taking part of the stream part of the time as shown in Figure 1 c) (after Pitard, 1993[6]), also with an in-stream point sampler or probe within a pipe or channel, which is always incorrect.



a) 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)

[a] Correct.

[b] Incorrect.

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

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

4.2.4 Weighting error, E_W

The weighting error is an error component arising from the selection model underlying Equation (1). In the model, the time-dependent flow rate of the solids in the slurry stream is a weighting function applied to the corresponding time-dependent quality characteristic over time, which gives the weighted-average quality characteristic of the solids component of the lot. The weighting error results from the application of incorrect

weights to the quality characteristics. The best solution to reducing the weighting error is to stabilize the flow rate. As a general rule, the weighting error is negligible for variations in flow rate of up to 10 % relative and acceptable for variations in flow rate up to 20 % relative.

4.2.5 Periodic quality fluctuation error, E_{Q3}

Periodic quality fluctuation errors result from periodic variations in quality generated by some equipment used for slurry processing and transportation, e.g. grinding and screening circuits, splitters and pumps. The presence of periodic variations can be detected by determining the variogram (see ISO 13909-7). While in most cases variogram values can be fitted with a simple linear or quadratic function, if periodic behaviour (characterized by regularly spaced maxima and minima) is observed, the fitting function can include a sine-wave term with a period and amplitude to be determined as parameters of the fit (Gy, 1982[5]). In such cases, stratified random sampling should be carried out as discussed in Clause 11. The alternative is to significantly reduce the source of periodic variations in quality, which can require plant redesign.

4.3 Sampling and overall variance

4.3.1 Sampling variance

Assume that the weighting (E_W), increment delimitation (E_D), increment extraction (E_E) and preparation errors (E_P) described in 4.2.2, 4.2.3 and 4.2.4 have been eliminated or reduced to insignificant values by careful design and sampling practice. In addition, assume that periodic variations in quality have been eliminated and that the flow rate has been regulated. The sampling error in Equation (1) then reduces to the form of Equation (3):

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

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

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

The short-range quality fluctuation variance, V_{EQ1}, arises from the different internal composition of increments taken at the shortest possible interval apart. This is a local or random variance due to the particulate nature of the solids in the slurry.

The long-range quality fluctuation variance, V_{EQ2}, arises from the continuous trends in quality that occur while sampling a slurry and is usually space- and time-dependent. This component is often the combination of a number of trends generated by diverse causes.

4.3.2 Overall variance

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

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

where

V_S is estimated sampling variance;

V_P is estimated sample preparation variance;

V_T is estimated measurement variance.

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

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

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

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

a) carrying out multiple determinations on the contained solids in the lot sample;

b) analysing the contained solids in individual increments;

c) dividing the slurry lot into a number of sub-lots or part-lots and analysing the contained solids in a sample from each sub-lot.

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

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

$$V_{SPT} = V_S + V_P + \frac{V_T}{r} \tag{6}$$

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

$$V_{SPT} = V_S + \frac{V_P + \frac{V_T}{r}}{m} \tag{7}$$

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

$$V_{SPT} = V_S + \frac{V_P}{n} + \frac{V_T}{n} \tag{8}$$

5 Sampling schemes

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

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