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Hard coal and coke — Mechanical sampling —

Part 2: Coal — Sampling from moving streams

Houille et coke — Échantillonnage mécanique —

Partie 2: Charbon — Échantillonnage en continu

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

This second edition cancels and replaces the first edition (ISO 13909-2:2001), which has been technically revised.

ISO 13909 consists of the following parts, under the general title *Hard coal and coke — Mechanical sampling*:

— *Part 1: General introduction*

— *Part 2: Coal — Sampling from moving streams*

— *Part 3: Coal — Sampling from stationary lots*

— *Part 4: Coal — Preparation of test samples*

— *Part 5: Coke — Sampling from moving streams*

— *Part 6: Coke — Preparation of test samples*

— *Part 7: Methods for determining the precision of sampling, sample preparation and testing*

— *Part 8: Methods of testing for bias*

Annex B forms a normative part of this part of ISO 13909. Annex A of this part of ISO 13909 is for information only.

Hard coal and coke — Mechanical sampling —

Part 2:
Coal — Sampling from moving streams

1 Scope

This part of ISO 13909 specifies procedures and requirements for the design and establishment of mechanical samplers for the sampling of coal from moving streams and describes the methods of sampling used.

It does not cover mechanical sampling from stationary lots which is dealt with in ISO 13909-3[1].

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 13909-1:2016, *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 13909-7, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*

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

ISO 21398, *Hard coal and coke — Guidance to the inspection of mechanical sampling systems*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13909-1 apply.

4 Establishing a sampling scheme

4.1 General

The general procedure for establishing a sampling scheme is as follows.

a) Define the quality parameters to be determined and the types of samples required.

b) Define the lot.

c) Define or assume the precision required (see 4.3.1).

d) Determine the method of combining the increments into samples and the method of sample preparation (see ISO 13909-4).

e) Determine or assume the variability of the coal (see 4.3.2) and the variance of preparation and testing (see 4.3.3). Methods for determining variability and the variance of preparation and testing are given in ISO 13909-7.

f) Establish the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see 4.3.4).

g) Decide whether to use time-basis or mass-basis sampling (see Clause 5) and define the sampling intervals in minutes for time-basis sampling or in tonnes for mass-basis sampling.

h) Ascertain the nominal top size of coal for the purpose of determining the minimum mass of sample (see 4.4 and Table 1).

NOTE The nominal top size may initially be ascertained by consulting the consignment details, or by visual estimation, and may be verified, if necessary, by preliminary test work.

i) Determine the minimum average increment mass (see 4.5).

4.2 Design of the sampling scheme

4.2.1 Material to be sampled

The first stage in the design of the scheme is to identify the coal to be sampled. Samples may be required for technical evaluation, process control, quality control and for commercial reasons by both the producer and the customer. It is essential to ascertain exactly at what stage in the coal-handling process the sample is required and, as far as practicable, to design the scheme accordingly. In some instances, however, it may prove impracticable to obtain samples at the preferred points and, in such cases, a more practicable alternative is required.

4.2.2 Division of lots

A lot may be sampled as a whole or as a series of sub-lots, e.g. coal dispatched or delivered over a period of time, a ship load, a train load, a wagon load or coal produced in a certain period, e.g. a shift.

It may be necessary to divide a lot into a number of sub-lots in order to improve the precision of the results. For lots sampled over long periods, it may be expedient to divide the lot into a series of sub-lots, obtaining a sample for each.

4.2.3 Basis of sampling

Sampling may be carried out on either a time-basis or a mass-basis. In time-basis sampling, the sampling interval is defined in minutes and seconds and the increment mass is proportional to the flow rate at the time of taking the increment. In mass-basis sampling, the sampling interval is defined in tonnes and the mass of increments constituting the sample is uniform. Of these two alternatives, time-basis sampling is easier to implement and verify, because only a fixed speed cutter and a timing device are required. On the other hand, for mass-basis sampling, a conveyor belt weightometer is required as well as a device that is controlled sufficiently to adjust the primary cutter speed increment by increment to achieve uniform increment mass.

4.2.4 Precision of sampling

After the desired sampling precision has been selected, the number of sub-lots and the minimum number of increments per sub-lot collected shall be determined as described in 4.3.4, and the average mass of the primary increments shall be determined as described in 4.5.

For single lots, the quality variation shall be assumed as the worst case (see 4.3.2). The precision of sampling achieved may be measured using the procedure of replicate sampling (see ISO 13909-7).

At the start of regular sampling of unknown coals, the worst-case quality variation shall be assumed, in accordance with 4.3.2. When sampling is in operation, a check may be carried out to confirm that the desired precision has been achieved, using the procedures described in ISO 13909-7.

If any subsequent change in precision is required, the number of sub-lots and of increments shall be changed as determined in 4.3.4 and the precision attained shall be rechecked. The precision shall also be checked if there is any reason to suppose that the variability of the coal being sampled has increased. The number of increments determined in 4.3.4 applies to the precision of the result when the sampling errors are large relative to the testing errors, e.g. for moisture content.

4.2.5 Bias of sampling

It is of particular importance in sampling to ensure, as far as possible, that the parameter to be measured is not altered by the sampling and sample preparation process or by subsequent storage prior to testing. This may require, in some circumstances, a limit on the minimum mass of primary increment (see 4.5).

When collecting samples for moisture determination from lots over an extended period, it may be necessary to limit the standing time of samples by dividing the lot into a number of sub-lots (see 4.3.4).

Sampling systems shall be tested for bias in accordance with the methods given in ISO 13909-8.

4.3 Precision of results

4.3.1 Precision and total variance

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

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

The desired overall precision for a lot is normally agreed between the parties concerned. In the absence of such agreement, a value of one tenth of the ash content may be assumed up to 10 % ash, subject to a maximum of 1 % absolute for ash contents above 10 %.

The theory of the estimation of precision is discussed in ISO 13909-7. The following formula is derived:

$$P_L = 2\sqrt{\frac{\frac{V_I}{n} + V_{PT}}{m}} \qquad (1)$$

where

P_L is the estimated index of overall precision of sampling, sample preparation and testing for the lot, expressed as a percentage absolute;

V_I is the primary increment variance;

n is the number of increments per sub-lot;

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

V_{PT} is the preparation and testing variance.

If the quality of a coal of a type not previously sampled is required, then in order to devise a sampling scheme, assumptions have to be made about the variability (see 4.3.2). The precision actually achieved for a particular lot by the scheme devised can be measured by the procedures given in ISO 13909-7.

4.3.2 Primary increment variance

The primary increment variance, V_I, depends upon the type and nominal top size of coal, the degree of pre-treatment and mixing, the absolute value of the parameter to be determined and the mass of increment taken.

The number of increments required for the general-analysis sample and the moisture sample shall be calculated separately using the relevant values of increment variance and the desired precision. If a common sample is required, the number of increments required for that sample shall be the greater of the numbers calculated for the general-analysis sample and the moisture sample respectively.

NOTE For many coals, the increment variance for ash is higher than that for moisture and hence, for the same precision, the number of increments required for the general-analysis sample will be adequate for the moisture sample and for the common sample.

The value of the primary increment variance, V_I, required for the calculation of the precision using Formula (1) can be obtained by either

a) direct determination on the coal to be sampled using one of the methods described in ISO 13909-7, or

b) assuming a value determined for a similar coal from a similar coal handling and sampling system.

If neither of these values is available, a value of $V_I = 5$ for the ash content of unwashed and blended coals and $V_I = 3$ for the ash content of washed coals can be assumed initially and checked, after the sampling has been carried out, using one of the methods described in ISO 13909-7.

4.3.3 Preparation and testing variance

The value of the preparation and testing variance, V_{PT}, required for the calculation of the precision using Formula (1) can be obtained by either

a) direct determination on the coal to be sampled using one of the methods described in ISO 13909-7, or

b) assuming a value determined for a similar coal from a similar sample preparation scheme.

If neither of these values is available, a value of 0,20 for ash content can be assumed initially and checked, after the preparation and testing has been carried out, using one of the methods described in ISO 13909-7.

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

4.3.4.1 General

The number of increments taken from a lot in order to achieve a particular precision is a function of the variability of the quality of the coal in the lot, irrespective of the mass of the lot. The lot may be sampled as a whole, resulting in one sample, or divided into a number of sub-lots resulting in a sample from each. Such division may be necessary in order to achieve the required precision, and the necessary number of sub-lots shall be calculated using the procedure given in 4.3.4.2.

Another important reason for dividing the lot is to maintain the integrity of the sample, i.e. to avoid bias after taking the increment, particularly, in order to minimize loss of moisture due to standing. The need to do this is dependent on factors such as the time taken to collect samples, ambient temperature and humidity conditions, the ease of keeping the sample in sealed containers during collection and the particle size of the coal. It is recommended that, if moisture loss is suspected, a bias test be carried out to compare the quality of a reference sample immediately after extraction with the sample after standing for the normal time. If bias is found, the sample standing time should be reduced by collecting samples more frequently, i.e. increasing the number of sub-lots.

There may be other practical reasons for dividing the lot such as the following:

a) for convenience when sampling over a long period;

b) to keep sample masses manageable.

The designer of a sampling scheme should cater for the worst case anticipated and will then tend to use a higher value for V_I than may actually occur when the system is in operation. On implementing a new sampling scheme, a check on the actual precision being achieved should be carried out using the methods described in ISO 13909-7. This may indicate that some changes are required to achieve the required precision, in which case, the number of sub-lots and increments shall be recalculated using the procedures given in 4.3.4.2.

4.3.4.2 Calculation of number of sub-lots and increments

The number of sub-lots and number of increments required per sub-lot are established using the following procedure.

Determine the minimum number of sub-lots required for practical reasons (see 4.3.4.1).

Estimate the number of increments, n, in each sub-lot for a desired precision from the following formula [obtained by transposing Formula (1)]:

$$n = \frac{4V_I}{mP_L^2 - 4V_{PT}} \tag{2}$$

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

a) Choose a number corresponding to a convenient mass, recalculate n from Formula (2) and repeat this process until n is a practicable number.

b) Decide on the maximum practicable number of increments per sub-lot, n_1, and calculate m from Formula (3).

$$m = \frac{4V_I + 4n_1V_{PT}}{n_1P_L^2} \tag{3}$$

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

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

NOTE The formulae given in 4.3.4.2 will generally estimate a higher number for the required number of increments. This is because it is based on the assumption that the quality of coal has no serial correlation; however, serial correlation is always present to some degree. In addition, because a certain amount of preparation and testing is required when measuring the increment variance, the preparation and testing errors are included more than once.

Example 1

The lot is 20 000 t delivered in 5 000 t train loads and the required precision, P_L , is 0,25 % ash. The quality variation is known and the following values have been determined:

primary increment variance, V_I = 0,5;

preparation and testing variance, V_{PT} = 0,05.

a) Initial number of sub-lots

It has been decided that the minimum number of sub-lots shall be four. Therefore, take sub-lots of 5 000 t each (i.e. one sub-lot per train load in this case).

b) Number of increments per sub-lot

$$n = \frac{4 \times 0{,}5}{(4 \times 0{,}25^2) - (4 \times 0{,}05)} = 40 \text{ using Formula} \qquad (2)$$

Therefore, take four sub-lots of 40 increments each, (i.e. 40 increments from each sub-lot, which is a reasonable number).

Example 2

The lot is 100 000 t delivered as 5 000 t/day over two shifts.

Required precision, P_L = 0,25 % ash

Primary increment variance, V_I = 5

Preparation and testing variance, V_{PT}, unknown; initially assumed = 0,20

a) Initial number of sub-lots

Take a daily sample (i.e. m = 20 in order to avoid risk of bias by overnight storage of samples).

b) Number of increments per sub-lot

$$n = \frac{4 \times 5}{(20 \times 0{,}25^2) - (4 \times 0{,}20)} = 45 \text{ using Formula} \qquad (2)$$

If this number of increments is considered to be too large, increase the number of sub-lots to 40, i.e. one per shift.

$$n = \frac{4 \times 5}{(40 \times 0{,}25^2) - (4 \times 0{,}20)} = 12$$

It would then be sensible to take 12 increments per shift, i.e. one every 40 min.

Example 3

The lot is 100 000 t of washed coal delivered at 10 000 t/h via a shiploading conveyor.

Required precision, P_L = 0,2 % ash

Primary increment variance, V_I = 3 (washed coal)

Preparation and testing variance, V_{PT} = 0,05

a) Initial number of sub-lots

Take an hourly sample, i.e. m = 10.

b) Number of increments per sub-lot

$$n = \frac{4 \times 3}{(10 \times 0{,}2^2) - (4 \times 0{,}05)} = 60 \text{ using Formula} \qquad (2)$$

Therefore, divide the lot into 10 sub-lots and take increments at 1 min intervals.

Example 4

The lot is 8 000 t in a single load and the required precision, P_L, is 0,5 % ash. The quality variation is known and the following values have been determined:

primary increment variance, V_I = 5;

preparation and testing variance, V_{PT} = 0,20.

