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Hard coal and coke — Mechanical sampling —

Part 5: Coke — Sampling from moving streams

Houille et coke — Échantillonnage mécanique —

Partie 5: Coke — Échantillonnage en continu

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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).

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 13909-5:2001), 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*

Hard coal and coke — Mechanical sampling —

Part 5:
Coke — Sampling from moving streams

1 Scope

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

The diversity of types of equipment for sampling and the conditions under which mechanical sampling is performed make it inappropriate to specify standard designs for samplers which will be applicable to all situations.

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 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — 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 part of ISO 13909, 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 the precision required;

d) determine the method of combining the increments into a sample, or number of sub-lot samples, and the method of sample preparation (see ISO 13909-6);

e) determine or assume the variability of the coke (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) define the sampling interval (see Clause 5);

h) ascertain the nominal top size of the coke 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 cokes 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 coke-handling process the sample is required and, as far as practicable, design the scheme accordingly.

4.2.2 Parameters to be determined on samples

The samples for moisture and physical tests may be collected separately or as one sample, which is then divided. In this part of ISO 13909, a sample which is collected for the determination of moisture (and possibly also for general analysis) is referred to as the moisture sample; a sample which is collected for physical tests only is referred to as the physical sample. If a sample is used for the determination of moisture and for physical tests, it is referred to as a common sample.

In mechanical sampling of coke, the only sample which can, in certain circumstances (see 4.2.6), be processed automatically beyond the divided-increment stage is the moisture sample.

In order to achieve the desired precision, it may be necessary to take different numbers of increments for the moisture and physical samples. Where a common sample is taken, the greater number of increments shall be used.

4.2.3 Division of lots

A lot may be sampled as a whole or as a series of sub-lots, e.g. coke despatched or delivered over a period of time, a ship load, a train load, a wagon load, or coke 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.

4.2.4 Basis of sampling

Sampling may be carried out on either a time-basis or a mass-basis.

In time-basis sampling, increments are taken at fixed time intervals with an increment mass, collected with a fixed speed cutter, which is proportional to the flow rate at the time of extraction.

In mass-basis sampling, increments are taken at fixed mass intervals, using a belt weigher/mass integrator, and fixed mass increments are extracted using a variable speed cutter or sample preparation system which produces a fixed mass divided increment.

The conditions under which mass-basis sampling may seem to offer the advantage of consistent increment mass, for example highly variable flow rates, are those in which it is most difficult to implement in practice.

Time-basis sampling is by far the simplest to implement and is the basis of this part of ISO 13909.

4.2.5 Precision of sampling

The required precision for a lot for each parameter to be measured shall be decided. The number of sub-lots and minimum number of increments per sub-lot collected shall then be determined as described in 4.3.4, and the average mass of 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 cokes, the worst-case quality variation shall be assumed. When sampling is in operation, a check shall be carried out to confirm that the desired precision has been achieved using the procedure of duplicate sampling as described in ISO 13909-7.

If any subsequent change in precision is required, the number of sub-lots and 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 coke 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. moisture content. However, in some tests, e.g. Micum Index, the testing errors are themselves large. In this case, it may be necessary to prepare two or more test portions from the same sample (see 4.3.4.3) and use the mean of the determinations to give a better precision.

4.2.6 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. For example, care shall be taken to avoid breakage of coke intended for physical testing and loss of moisture from the moisture sample during storage. This may require, in some circumstances, a limit on the minimum mass of primary increment (see 4.5 and Clause 8).

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).

The use of on-line crushing and division of the moisture sample for moisture determination should be treated with caution because of the risk of bias caused by loss of moisture in the processing (see 6.2.2). In particular, the crushing of hot coke is not recommended. If the bias is unacceptable, the sample shall be left in the uncrushed state and the sample preparation carried out by manual methods. It should be accepted, however, that some bias is inevitable, whether due to breakage or loss of moisture from hot coke. The object, therefore, shall be to restrict such degradation or moisture loss to a minimum.

When a coke-sampling scheme is implemented, it shall be checked 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 coke 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.

NOTE The required overall precision for a lot is normally agreed between the parties concerned.

The theory of the estimation of precision is given in ISO 13909-7. Formula (1) is derived:

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

where

P_L is the estimated overall precision of sampling, sample preparation and testing for the lot at a 95 % confidence level, expressed as a percentage absolute;

V_I is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of increments to be taken from a sub-lot;

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

If the quality of a coke of a type not previously sampled is required, then, in order to devise a sampling scheme, assumptions should 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.

If the same type of coke is sampled regularly, sampling schemes can be laid down using data derived from previous sampling. The procedures given in ISO 13909-7 can be used to devise the optimum scheme, thus keeping the sampling costs to a minimum.

4.3.2 Primary increment variance

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

The variability for moisture is usually higher than that for ash and hence, for the same precision, the number of increments for moisture will be adequate for ash. If, however, a higher precision is required for ash, the relevant primary increment variance shall be applied for each 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 coke to be sampled using one of the methods described in ISO 13909-7, or

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

If neither of these values is available, a value of 5 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 coke to be sampled using one of the methods described in ISO 13909-7, or

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

If neither of these values is available, a value of 0,2 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 in each 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 coke 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.

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

a) for convenience when sampling over a long period;

b) to keep sample masses manageable;

c) 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 coke. It is recommended that, if moisture loss is suspected, a bias test is 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.

The quality of the lot shall be calculated as the weighted average of the values found for the sub-lots.

As stated in 4.3.1, the precision is determined by the variability of the coke, the number of increments and sub-lots and the preparation and testing variance. By transposing Formula (1), it can be shown that the number of increments per sub-lot for a desired precision for a lot can be estimated from Formula (2):

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

Determine the number of sub-lots required for practical reasons and then estimate the number of increments in each for the desired precision using Formula (2). If n is a practicable number, the initial scheme is established. However, if n is less than 10, take 10 increments per sub-lot.

If n is impracticably large, increase the number of sub-lots using one of the following methods:

a) increase m to a number corresponding to a convenient mass or time, recalculate n 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):

$$m = \frac{4V_I + 4n_1 V_{PT}}{n_1 P_L^2} \quad (3)$$

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

NOTE This method of calculating the number of increments required per sub-lot for a certain precision from the primary increment variance and the preparation and testing variance will generally give an overestimate of the required number. This is because it is based on the assumption that the quality of coke varies in a random manner. 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.

The designer of a sampling scheme should cater for the worst case anticipated and may then use higher values for V_I than may actually occur when the scheme is in operation. When the sampler is commissioned, the precision of the result can be estimated and adjusted (see ISO 13909-7), by increasing or decreasing the number of increments in the sample, keeping the same increment mass so that the required precision can be achieved at minimum cost.

Example 1

The lot is 35 000 t of 40 × 20 mm coke delivered in one day. The primary increment variance and preparation and testing variance for moisture content have been determined as follows.

Primary increment variance for moisture content, $V_I = 5$.

Preparation and testing variance for moisture content, $V_{PT} = 0{,}10$.

The required precision, $P_L = 1{,}0$ % moisture content.

a) Initial number of sub-lots

For convenience and to avoid the sample standing for too long, take three shift samples, (i.e. $m = 3$).

b) Number of increments per sub-lot

$$n = \frac{4 \times 5}{3 \times 1^2 - 4 \times 0{,}10} = 7{,}7 \text{ using Formula (2)}$$

Therefore, split the lot into three sub-lots and take 10 increments from each.

Example 2

The lot is 100 000 t of 100 × 25 mm coke delivered as 5 000 t/day over two 8-hour shifts.

The primary increment variance, V_I, for moisture content is unknown, so initially assume a value of 5.

Required precision $P_L = 0{,}25$ % moisture content.

Preparation and testing variance for moisture content, V_{PT}, from experience assume a value of 0,20.

a) Initial number of sub-lots

For the preliminary calculation, assume a daily sample is constituted, i.e. $m = 20$, in order to avoid the risk of moisture loss by overnight storage of sample increments.

b) Number of increments per sub-lot

$$n = \frac{4 \times 5}{20 \times 0{,}25^2 - 4 \times 0{,}2} = 44{,}4 \text{ using Formula (2)}$$

This number will result in too large a mass to crush as a single moisture sample for each sub-lot [almost 2 tonnes for a typical increment mass of about 45 kg using Formula (4)]. Therefore, increase the number of sub-lots to 40, i.e. one per shift.