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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

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 13909 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13909-5 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

ISO 13909 cancels and replaces ISO 9411-1:1994, *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal* and ISO 9411-2:1993, *Solid mineral fuels — Mechanical sampling from moving streams — Part 2: Coke*, of which it constitutes a technical revision. It also supersedes the methods of mechanical sampling of coal and coke given in ISO 1988:1975, *Hard coal — Sampling* and ISO 2309:1980, *Coke — Sampling*.

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. Annexes A and C of this part of ISO 13909 are for information only.

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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13909. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13909 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 13909-1:2001, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*.

ISO 13909-6:2001, *Hard coal and coke — Mechanical sampling — Part 6: Coke — Preparation of test samples*.

ISO 13909-7:2001, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*.

ISO 13909-8:2001, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*.

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 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) 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 coke for the purpose of determining the minimum mass of sample (see 4.4).

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. 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   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, 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 added to the sample is uniform.

4.2.5 Precision of results

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. 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 results

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 has to 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 sampling

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. The following equation is derived:

$$P_L = 2\sqrt{\dfrac{\dfrac{V_I}{n} + V_{PT}}{m}} \tag{1}$$

where

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

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 pretreatment 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 equation (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 25 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 equation (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 0,5 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:

c) for convenience when sampling over a long period;

d) to keep sample masses manageable;

e) 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 equation (1), it can be shown that the number of increments per sub-lot for a desired precision for a lot can be estimated from the following equation:

$$n = \frac{4V_\text{I}}{mP_\text{L}^2 - 4V_\text{PT}} \tag{2}$$

Determine the number of sub-lots required for practical reasons. Estimate the number of increments in each for the desired precision from equation (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:

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; or

b) decide on the maximum practicable number of increments per sub-lot, n_1 and calculate m from the equation:

$$m = \frac{4V_\text{I} + 4n_1V_\text{PT}}{n_1P_\text{L}^2} \tag{3}$$

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.

Examples of calculations are given in annex A.

4.3.4.2 Moisture sample

The sampling variance for moisture may vary in the range 0,2 to 25 depending on the absolute value of the moisture content, the size range of the coke and the extent of cutting, screening and mixing it has undergone prior to sampling. For example, a closely graded, highly cut small-sized industrial coke sampled on delivery to the customer would have a much lower variance than an uncut coke sampled at the wharf or a very large coke on despatch from the producer's works. It may be known from experience what level of variance is to be expected.

It is recommended that the number of increments initially required be sufficient to give a mass of sample greater than the mass given in Table 1, subject to a minimum of 10 increments.

The variance for ash and other chemical properties is usually less than for moisture. However, it is often desired to obtain a higher precision for the ash result and hence the number of increments should be calculated for each and the greater number taken for the moisture sample.

Table 1 — Minimum mass of sample

Nominal top size	Minimum mass
mm	kg
> 125	2 000
125	1 000
90	500
63	250
45	125
31,5	60
22,4	30
16,0	15
11,2	8
10,0	6
8,0	4
5,6	2
4	1

4.3.4.3 Physical sample

The cokes to be sampled within the scope of this part of ISO 13909 will exhibit large differences in physical strength, size, size range and size distribution. In addition, many different parameters e.g. Micum test, porosity, percentage retained on a particular sieve, mean size, etc. can be determined on the samples. Sample preparation errors may be zero when the test is done on the whole sample, or large when division of the sample takes place.

Furthermore, it is usually not possible to determine the individual increment variances for tests such as the Micum test because the increment mass is too small.

It will be found with many physical tests that the only way to achieve the required precision will be either

a) to divide the lot into sub-lots, or

b) to prepare two or more test portions from the sample, taking the mean of the test results for the sample. The precision for the particular parameter required shall then be checked and the number of increments adjusted according to the procedure specified in ISO 13909-7.

4.4 Minimum mass of sample

For most parameters, particularly size grading and those that are particle-size related, the precision of the result is limited by the ability of the sample to represent all the particle sizes in the mass of coke being sampled.

The minimum mass of sample is dependent on the nominal top size of the coke, the precision required for the parameter concerned and the relationship of that parameter to particle size. Some such relationship applies at all stages of preparation. The attainment of this mass will not, in itself, guarantee the required precision. This is also dependent on the number of increments in the sample and their variability (see 4.3.4).

The masses specified in Table 1 are for guidance on the minimum mass for unknown or heterogeneous cokes. Whilst they can usually be reduced for the moisture sample, they may be inadequate for the determination of, for example, oversize to 1 % precision of sampling and division, particularly on very large cokes.

When a coke is regularly sampled under the same circumstances, the precision obtained for all the required quality parameters shall be checked in accordance with ISO 13909-7 and the masses adjusted accordingly. However, the masses shall not be reduced below the minimum requirements laid down in the relevant analysis standards.

Account shall also be taken of the uses to which the sample is to be put and the numbers, masses and size distribution of the test samples required.

4.5   Mass of primary increment

The mass, m_I, in kilograms, of an increment taken by a mechanical cutter with cutting edges normal to the stream at the discharge of a moving stream can be calculated from equation (4):

$$m_I = \frac{Cb}{3,6v_C} \times 10^{-3} \tag{4}$$

where

C is the flow rate, in tonnes per hour;

b is the cutter aperture width, in millimetres;

v_C is the cutter speed, in metres per second.

For a cross-belt sampler, the mass, m_I, in kilograms, of increment can be calculated from equation (5):

$$m_I = \frac{Cb}{3,6v_B} \times 10^{-3} \tag{5}$$

where

C is the flow rate, in tonnes per hour;

b is the cutter aperture width, in millimetres;

v_B is the belt speed, in metres per second.

The minimum average mass of primary increment to be collected, m_I' is calculated from equation (6):

$$m_I' = \frac{m_S}{n} \tag{6}$$

where

m_S is the minimum mass of sample (see Table 1);

n is the minimum number of increments taken from the sub-lot (see 4.3.4).

In most mechanical systems, the mass of primary increment collected [see equations (4) and (5)] will greatly exceed that necessary to make up a sample of the required mass. In some systems, the primary increments are therefore divided, either as taken or after reduction, in order to avoid the mass of the sample becoming excessive.

Providing the design of the cutter complies with the requirements of 6.5 or 6.6, the extraction of an increment from the coke stream will be unbiased whatever the flow rate at the time. Even if flow rates are variable, increments taken at low flow rates, and hence of mass less than the average, will not be subject to extraction bias. Therefore, this part of ISO 13909 does not specify an absolute minimum increment mass.

Under some conditions, e.g. high ambient temperature, increments which are smaller than those corresponding to the design capacity of the system may suffer from disproportionate changes in quality, e.g. loss in moisture, and precautions need to be taken to prevent this. If such losses cannot be prevented and are found to cause relevant bias, such means as buffer hoppers or a variable-speed cutter (mass-basis sampling) shall be used. Alternatively, increments can themselves be retained temporarily in a buffer hopper until there is sufficient mass to ensure passage, free from relevant bias, through an on-line preparation system. On no account shall a primary sampler, in a time-basis system or a mass-basis system, be switched off at low flow rates to avoid low mass increments.

When measuring primary increment variance (see ISO 13909-7:2001, clause 6) at preliminary stages in the design of the sampling scheme, use increment masses that are close to those expected to be taken by the system, based on similar coke from similar sampling systems. After implementation of the sampling scheme, 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.

5 Methods of sampling

5.1 General

Sampling shall be carried out by systematic sampling either on a time-basis or on a mass-basis, or by stratified random sampling. The procedures of sample preparation vary in accordance with the type of sampling employed (see ISO 13909-6).

It is essential that each increment taken from a stream represent the full width and depth of the stream.

The consistency of loading of the belt should be controlled, as far as possible, so that sampling is as efficient as possible. The flow should be made reasonably uniform over the whole cross-section of the stream at all times, by means of controlled loading or suitable devices such as feed hoppers, ploughs, etc.

Whichever method of primary increment collection is used, it is essential that the increment does not completely fill or overflow the sampling device. With mechanized sampling devices, the increment mass may be considerably larger than that necessary to produce the calculated minimum sample mass. Hence, a system of primary increment division (see ISO 13909-6) may be necessary to divide the increment to a manageable mass.

All processes and operations upstream of the sampling location shall be examined for characteristics which could produce periodic variations in belt loading or quality and which may coincide with the operation of the primary samplers. Such periodicity may arise from the cycle of operations or feeder systems in use. If it is not possible to eliminate coincidence between the plant operation cycle and the sampling cycle, stratified random sampling within fixed mass or time intervals shall be adopted.

5.2 Time-basis sampling

5.2.1 Method of taking primary increments

In order that the increment mass is proportional to the coke flow rate in mechanical sampling, the speed of the cutter shall be constant throughout the sampling of the entire sub-lot (see 6.5.1).