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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.

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 13909-2 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 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.

Examples of calculations of the number of sub-lots and number of increments per sub-lot are given in annex A. Requirements for the evaluation of sampling equipment are given in annex B and information on the operation of mechanical samplers is given in annex C.

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 13909-4:2001, *Hard coal and coke — Mechanical sampling — Part 4: Coal — 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 or assume the precision required (see 4.4.1).

d) Decide whether continuous or intermittent sampling is to be used (see 4.2).

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

f) Determine or assume the variability of the coal (see 4.4.2 and, if relevant, 4.4.3) and the variance of preparation and testing (see 4.4.4). Methods for determining variability and the variance of preparation and testing are given in ISO 13909-7.

g) Establish the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see 4.4.5).

h) 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.

i) Ascertain the nominal top size of coal for the purpose of determining the minimum mass of sample (see 4.5).

   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.

j) Determine the minimum average increment mass (see 4.6).

4.2 Continuous and intermittent sampling

4.2.1 Continuous sampling

In continuous sampling, every sub-lot is sampled and the same sampling interval (time or mass) applies to all sub-lots. There are as many sample results for the lot as there are sub-lots. The mean result for the lot should be of the required precision but if it is desired to check that the required precision has been attained, it is possible to do this by using the procedures of replicate sampling described in ISO 13909-7.

4.2.2 Intermittent sampling

If coal of the same type is sampled regularly, it may be satisfactory to collect increments from some of the sub-lots but not from others. This is called intermittent sampling. The same minimum number of increments shall be taken from every sub-lot that is sampled (see 4.4.5.3). The sub-lots to be sampled shall be chosen at random, unless it can be demonstrated that no bias, for example as a result of time-dependent variance, is introduced by choosing sub-lots systematically. Such demonstration shall be repeated from time to time and at random intervals.

There are as many sample results per lot as there are sub-lots sampled, but because some sub-lots are not sampled, it is not possible to say whether the average of these results will have the required precision for the lot unless information about the variation between sub-lots is available. This can be obtained by following the procedure described in ISO 13909-7. If the variation between sub-lots is too large, it may be necessary to introduce continuous sampling to achieve the desired precision.

Use of intermittent sampling shall be agreed between contracting parties and shall be recorded in the sampling report.

4.3 Design of the sampling scheme

4.3.1 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.

4.3.2 Division of lots

A lot may be sampled as a whole or as a series of sub-lots, e.g. coal despatched 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.

4.3.3 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.

4.3.4 Precision of results

After the precision has been decided, the number of sub-lots and the minimum number of increments per sub-lot collected shall be determined as described in 4.4.5, and the average mass of the primary increments shall be determined as described in 4.6.

For single lots, the quality variation shall be assumed as the worst case (see 4.4.2 and 4.4.3). 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 4.4.2, 4.4.3 and 4.4.4. 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 4.4.5 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 4.4.5 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, the testing errors are themselves large. In this case, it may be necessary to form two or more test portions from the sample and use the mean of the determinations to give a better precision.

4.3.5 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. This may require, in some circumstances, a limit on the minimum mass of primary increment (see 4.6).

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 4.4.5).

When a coal-sampling scheme is implemented, it shall be checked for bias in accordance with the methods given in ISO 13909-8.

4.4 Precision of sampling

4.4.1 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.

NOTE The required 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.

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

$$P_{\mathrm{L}} = 2\sqrt{\frac{\frac{V_{\mathrm{I}}}{n} + \left(1 - \frac{u}{m}\right) V_{\mathrm{m}} + V_{\mathrm{PT}}}{u}} \qquad (1)$$

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;

n is the number of increments per sub-lot;

u is the number of sub-lots actually sampled;

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

V_{m} is the sub-lot variance;

V_{PT} is the preparation and testing variance.

For continuous sampling, where $u = m$, equation (1) is simplified as follows

$$P_{\mathrm{L}} = 2\sqrt{\frac{\frac{V_{\mathrm{I}}}{n} + V_{\mathrm{PT}}}{m}} \qquad (2)$$

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 4.4.2 and 4.4.3). The precision actually achieved for a particular lot by the scheme devised can be measured by the procedures given in ISO 13909-7.

4.4.2 Primary increment variance

The primary increment variance, V_{I}, depends upon the type and nominal top size of coal, the degree of pretreatment 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 equation (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 $V_I = 20$ for ash content can be assumed initially and checked, after the sampling has been carried out, using one of the methods described in ISO 13909-7.

4.4.3 Sub-lot variance

The sub-lot variance, V_m, is influenced by the same factors as the primary increment variance but to a lesser degree.

If the sub-lot variance is known from previous experience, this may be used. If conditions permit, the sub-lot variance may be determined by the methods described in ISO 13909-7. In all other circumstances, a sub-lot variance of 5 shall be assumed initially.

4.4.4 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 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 0,2 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.

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

4.4.5.1 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 4.4.5.2 or 4.4.5.3, as appropriate.

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:

a) for convenience when sampling over a long period;

b) to keep sample masses manageable.

Establish the number of sub-lots and number of increments required per sub-lot in accordance with 4.4.5.2 or 4.4.5.3, as appropriate.

NOTE The formulae given in 4.4.5.2 and 4.4.5.3 will generally give an overestimate of the required number of increments. This is because they are 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 or the sub-lot 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 will then tend to use higher values for V_{I} and V_{m} 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 be necessary to achieve the required precision, in which case the number of sub-lots is calculated using the procedures given in 4.4.5.2 and 4.4.5.3.

4.4.5.2 Continuous sampling

Determine the minimum number of sub-lots required for practical reasons (see 4.4.5.1).

Estimate the number of increments, n, in each sub-lot for a desired precision from the following equation [obtained by transposing equation (2)]:

$$n = \frac{4V_{\mathrm{I}}}{mP_{\mathrm{L}}^2 - 4V_{\mathrm{PT}}} \qquad (3)$$

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 equation (3) 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 the following equation:

$$m = \frac{4V_{\mathrm{I}} + 4n_1V_{\mathrm{PT}}}{n_1P_{\mathrm{L}}^2} \qquad (4)$$

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.

Examples of calculations for continuous sampling from moving streams are given in annex A.

4.4.5.3 Intermittent sampling

Initially decide on the number of sub-lots, m, and the minimum number, u, required to be sampled for practical reasons (see 4.4.5.1).

Estimate the number of increments for a desired precision in a lot from the following equation [obtained by transposing equation (1)]:

$$n = \frac{4V_{\mathrm{I}}}{uP_{\mathrm{L}}^2 - 4\left(1 - \frac{u}{m}\right)V_{\mathrm{m}} - 4V_{\mathrm{PT}}} \qquad (5)$$

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 to be sampled by one of the following means.

a) Choose a larger value for u, the number of sub-lots actually sampled, recalculate n and repeat this process until the value of n is a practicable number.

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

$$u = \frac{4m\left(\frac{V_\mathrm{I}}{n_1} + V_\mathrm{m} + V_\mathrm{PT}\right)}{mP_\mathrm{L}^2 + 4V_\mathrm{m}} \quad (6)$$

Adjust m upwards, if necessary, to a convenient number and recalculate n from equation (5).

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

Examples of calculations for intermittent sampling from moving streams are given in annex A.

4.5 Minimum mass of sample

For most parameters, particularly size analysis 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 coal being sampled.

The minimum mass of a sample is dependent on the nominal top size of the coal, 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, because precision is also dependent on the number of increments in the sample and their variability (see 4.4.5).

Values for the minimum mass of samples for general analysis to reduce the variance due to the particulate nature of the coal to 0,01, corresponding to a precision of 0,2 % with regard to ash, are given in column 2 of Table 1 (see CSIRO report [2]). Column 3 of Table 1 gives the corresponding minimum masses of divided samples for total moisture analysis, which are approximately 20 % of the minimum masses for general analysis, subject to an absolute minimum of 0,65 kg.

The minimum mass of sample, m_S, for other desired levels of precision for determination of ash may be calculated from the following equation:

$$m_\mathrm{S} = m_{\mathrm{S},0}\left(\frac{0{,}2}{P_\mathrm{R}}\right)^2 \quad (7)$$

where

$m_{\mathrm{S},0}$ is the minimum mass of sample specified in Table 1 for a given nominal top size;

P_R is the required precision, with regard to ash, due to the particulate nature of the coal.

When a coal 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 may be adjusted accordingly. However, the masses shall not be reduced below the minimum requirements laid down in the relevant analysis standards.

When preparing coal to produce samples for multiple use, account shall also be taken of the individual masses and size distribution of the test samples required for each test.

4.6 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 (8):

$$m_\mathrm{I} = \frac{Cb \times 10^{-3}}{3{,}6 v_\mathrm{C}} \quad (8)$$

where

C is the flow rate, in tonnes per hour;

b is the cutter aperture width, in millimetres;

NOTE The cutter aperture value used for calculating the mass of an increment is the distance between the leading edges of the cutter lips first striking the stream of the material.

v_C is the cutter speed, in metres per second (see 6.8.2).

For a cross-belt sampler, the mass, m_I, in kilograms, of increment can be calculated from equation (9):

$$m_I = \frac{Cb \times 10^{-3}}{3{,}6 v_B} \quad (9)$$

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 (10):

$$m_I' = \frac{m_S}{n} \quad (10)$$

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 4.4.5).

In most mechanical systems, the mass of primary increment collected [see equations (8) and (9)] 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 6.8 or 6.9, the extraction of an increment from the coal 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, then 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 free 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. 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.

