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Hard coal and coke — Mechanical sampling —

Part 3: Coal — Sampling from stationary lots

Houille et coke — Échantillonnage mécanique —

Partie 3: Charbon — Échantillonnage sur lots statiques

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Contents

Page

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).

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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 13909-3:2001), 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 3:
Coal — Sampling from stationary lots

1 Scope

This part of ISO 13909 specifies procedures for the mechanical sampling of coal from stationary lots, for example, from wagons, barges, ships and stockpiles. These procedures are to be used when it is not possible to sample the lots during loading or discharge according to ISO 13909-2. Procedures for sample preparation are given in ISO 13909-4.

This part of ISO 13909 is applicable to mechanical sampling from stationary coal lots, to obtain samples from which test samples for the determination of moisture, and for general analysis including physical and chemical tests, can be prepared in accordance with the requirements and recommendations set out in ISO 13909-4.

In this part of ISO 13909, the principles and procedures for designing a sampling scheme are given, together with typical examples of applications; in addition, practices for the execution of sampling in different sampling situations are described. The methods described are limited to those on which it is possible to conduct a test for bias.

2 Normative references

The following referenced 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, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

ISO 13909-2, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*

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*

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 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 upon the sampling interval, in tonnes.

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

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 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.

4.2.3 Precision of sampling

After the desired sample 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 and 4.3.3). 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 4.3.2, 4.3.3 and 4.3.4. 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 4.3.4 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.

4.2.4 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 4.3.4.1).

When a coal 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 coal agree among themselves.

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

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 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:

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

where

P_L is the estimated index of 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;

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 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 0,2 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.

4.3.4 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:

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 scheme 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 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 is 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 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 the value of 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 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.

EXAMPLE 1 The lot is 20 000 t of washed coal 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 four 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

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

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 of unwashed coal delivered as 5 000 t/day over two shifts.

Required precision, P_L = 0,25 % ash.

Primary increment variance, V_I, unknown; initially assumed = 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

$$n = \frac{4 \times 5}{20 \times 0{,}25^2 - 4 \times 0{,}20} = 44$$

EXAMPLE 3 The lot is 8 000 t of blended coal in a single load and the required overall 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.

a) Number of sub-lots

The customer requires a result based on at least two samples.

b) Number of increments per sub-lot

$$n = \frac{4 \times 5}{2 \times 0{,}5^2 - 4 \times 0{,}20} = \frac{20}{-0{,}3} = -66{,}7 \text{ using Formula} \quad (2)$$

This negative number indicates that the errors of preparation and testing are such that the required overall precision cannot be achieved with this number of sub-lots.

It could be decided that 50 increments is the maximum practicable number in a sub-lot and from Formula (3).

$$m = \frac{4 \times 5 + 4 \times 50 \times 0{,}2}{50 \times 0{,}5^2} = 4{,}8$$

This gives a practical sampling method of dividing the lot into five sub-lots and taking 50 increments from each.

4.4 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 similar 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 4.3.4).

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 Table 1, column 2 (see CSIRO report[1]). Table 1, column 3 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 Formula (4).

$$m_S = m_{S,0}\left(\frac{0,2}{P_R}\right)^2 \tag{4}$$

where

$m_{S,0}$ 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 (see 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.