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Coal and coke — Mechanical sampling —

Part 3:
Sampling of coal from stationary lots

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Foreword

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This document was prepared by Technical Committee ISO/TC 27, *Coal and coke*, Subcommittee SC 4, *Sampling*.

This third edition cancels and replaces the second edition (ISO 13909-3:2016), which has been technically revised.

The main changes are as follows:

— the title was updated to align with the ISO 13909 series;

— the scope was updated;

— several aspects of the sampling were updated to state of the art.

A list of all parts in the ISO 13909 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Coal and coke — Mechanical sampling —

Part 3:
Sampling of coal from stationary lots

1 Scope

This document 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 document 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 document, 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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, *Coal and coke — Mechanical sampling — Part 1: General introduction*

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

ISO 13909-4, *Coal and coke — Mechanical sampling — Part 4: Preparation of test samples of coal*

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

ISO 13909-8, *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.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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 can 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 can 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 can 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 can 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 may be assumed up to 10 % ash, subject to a maximum of 1 % absolute for ash above 10 %.

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

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

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 taken 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 is 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 of unwashed and blended coals and $V_I = 3$ for the ash 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 with regard to ash 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 division 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 shall be reduced by collecting samples more frequently, i.e. increasing the number of sub-lots.

There can 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 shall 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 shall 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)]:

$$n = \frac{4V_I}{mP_L^2 - 4V_{PT}} \qquad (2)$$

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 for m corresponding to a convenient sublot 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):

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

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 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 generally gives a higher number for the required number than needed. This is because the method is based on the assumption that the quality of coal varies in a random manner and has no serial correlation; however, serial correlation is always present to some degree. In addition, because a certain amount of preparation and testing is required when measuring the increment variance, the preparation and testing errors are included more than once.

EXAMPLE 1 The lot is 20 000 t of washed coal delivered in 5 000 t train loads and the required precision, P_L, is 0,25 % with regard to 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

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

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 % with regard to 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 % with regard to 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

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

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 does 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).