BS ISO 13909-3:2016



BSI Standards Publication

Hard coal and coke — Mechanical sampling

Part 3: Coal — Sampling from stationary lots



National foreword

This British Standard is the UK implementation of ISO 13909-3:2016. It supersedes BS ISO 13909-3:2001 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/16, Solid mineral fuels.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 83176 8

ICS 73.040

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 June 2016.

Amendments/corrigenda issued since publication

Date Text affected

INTERNATIONAL STANDARD

ISO 13909-3:2016 ISO 13909-3

Second edition 2016-07-01

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



BS ISO 13909-3:2016 ISO 13909-3:2016(E)



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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.

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 13909-3:2001), 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.

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- 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 <u>4.3.2</u>) and the variance of preparation and testing (see <u>4.3.3</u>). 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 <u>4.4</u> and <u>Table 1</u>).
 - 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 <u>4.5</u>).

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 <u>4.3.4</u>, and the average mass of the primary increments shall be determined as described in <u>4.5</u>.

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:

$$P_{\rm L} = 2\sqrt{\frac{V_{\rm I}}{n} + V_{\rm PT}} \tag{1}$$

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;

 $V_{\rm I}$ is the primary increment variance;

n is the number of increments per sub-lot;

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

 $V_{\rm PT}$ 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 <u>4.3.2</u>). 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, $V_{\rm I}$, 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, $V_{\rm I}$, 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 $V_{\rm I}$ = 5 for ash content of unwashed and blended coals and $V_{\rm I}$ = 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 $V_{\rm I}$ 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)]:

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

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):

$$m = \frac{4V_{\rm I} + 4n_1 V_{\rm PT}}{n_1 P_{\rm I}^2} \tag{3}$$

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 <u>4.3.4.2</u> 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, $P_{\rm L}$, is 0,25 % ash. The quality variation is known and the following values have been determined:

primary increment variance, $V_{\rm I}$ = 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

$$n = \frac{4 \times 0.5}{4 \times 0.25^2 - 4 \times 0.05} = 40 \text{ using Formula}$$
 (2)

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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, $V_{\rm I}$, 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, $V_{\rm I}$ = 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 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 <u>Table 1</u>, column 2 (see CSIRO report^[1]). <u>Table 1</u>, 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).

$$m_{\rm S} = m_{\rm S,0} \left(\frac{0.2}{P_{\rm R}}\right)^2 \tag{4}$$

where

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

 $P_{\rm R}$ 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.

Table 1 — Minimum mass of sample for general analysis and determination of total moisture content

Nominal top size of coal	General-analysis samples and common samples	Samples for determination of total moisture content	
mm	kg	kg	
300	15 000	3 000	
200	5 400	1 100	
150	2 600	500	
125	1 700	350	
90	750	125	
75	470	95	
63	300	60	
50	170	35	
45	125	25	
38	85	17	
31,5	55	10	
22,4	32	7	
16,0	20	4	
11,2	13	2,50	
10	10	2	
8,0	6	1,50	
5,6	3	1,20	
4,0	1,50	1,00	
2,8	0,65	0,65	
2,0	0,25	0,65	
1,0	0,10	0,65	

NOTE 1 The masses for the general analysis and common samples have been determined to reduce the variance due to the particulate nature of coal to 0,01, corresponding to a precision of 0,2 % ash.

4.5 Mass of primary increment

The mass, $m_{\rm I}$, in kilograms, of an increment taken by a mechanical auger (see <u>Clause 7</u>) from a stationary lot of coal can be calculated from <u>Formula (5)</u>.

$$m_{\rm I} = \frac{\pi d^2 l \rho}{4} \tag{5}$$

where

- d is the diameter of the auger tube, in metres;
- *l* is the depth of penetration of the auger, in metres;
- r is the bulk density of the coal, in kilograms per m^3 .

NOTE 2 Extraction of the total-moisture sample from the common sample is described in ISO 13909-4.

The minimum average mass of primary increment to be collected, $m'_{\rm I}$, is calculated from Formula (6).

$$m_{l}' = \frac{m_{s}}{n} \tag{6}$$

where

 $m_{\rm S}$ is the minimum mass of sample (see <u>Table 1</u>);

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

With most mechanical augers, the mass of primary increment collected [see Formulae (5) and (6)] will greatly exceed that necessary to make up a sample of the required mass. In some cases, the primary increments are therefore divided, either as taken or after reduction, in order to avoid the mass of the sample becoming excessive.

When measuring primary increment variance (see ISO 13909-7:2016, 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.

4.6 Size analysis

Within the scope of this part of ISO 13909, the coals to be sampled will exhibit large differences in size, size range and size distribution. In addition, the parameters to be determined (percentage retained on a particular sieve, mean size, etc.) may differ from case to case. Furthermore, when sample division is applied, division errors shall be taken into account, whereas they are non-existent if sizing is performed without any preceding division.

Take these factors into account when applying the techniques for calculating numbers of increments for a particular precision (see 4.3.1 to 4.3.4). In the absence of any information on increment variance etc., initially take 25 increments per sample.

The precision for the particular parameter required shall then be checked and the number of increments adjusted according to the procedure described in ISO 13909-7.

Minimization of degradation of samples used for determination of size distribution is vital to reduce bias in the measured size distribution. To prevent particle degradation, it is essential to keep free-fall drops to a minimum. In addition, some auger samplers utilize a screw device to transport the sample increment up the column and this may cause degradation of size. Trial tests should be made in accordance with the method given in ISO 13909-8 to determine the degree of degradation.

The minimum masses of sample for size analysis are given in <u>Table 2</u>. The masses have been calculated on the basis of the precision of the determination of oversize, i.e. the coal above the nominal top size. Precision for other size fractions will normally be better than this.

Table 2 — Minimum mass of sample for size analysis

Nominal top size of coal	$\begin{array}{c} \textbf{Minimum mass for a precision of} \\ 1 \ \% \end{array}$	$\begin{array}{c} \textbf{Minimum mass for a precision of} \\ 2\ \% \end{array}$	
mm	kg	kg	
300	54 000	13 500	
200	16 000	4 000	
150	6 750	1 700	
125	4 000	1 000	
90	1 500	400	
75	950	250	
63	500	125	
50	280	70	
45	200	50	
38	130	30	
31,5	65	15	
22,4	25	6	
16,0	8	2	
11,2	3	0,70	
10,0	2	0,50	
8,0	1	0,25	
5,6	0,50	0,25	
4,0	0,25	0,25	
2,8	0,25	0,25	

5 Methods of sampling from wagons, barges and ships

5.1 General

Sampling from stationary lots is carried out on a mass-basis only. The methods described in this clause are applicable to railway wagons, road wagons, shallow barges and ships up to a size such that full-depth sampling can be achieved. A sub-lot can be one or any number of wagons, an entire barge, several barges or one hold of a barge.

5.2 Number of increments and sub-lots

5.2.1 General analysis and moisture samples

The number of sub-lots in the lot and the required number of increments in each sub-lot are calculated by the methods given in 4.3.4.

5.2.2 Common sample

Where a moisture sample is to be extracted from a common sample, the initial number of increments collected shall be that required for general analysis or moisture determination, whichever is the greater. Increase the mass of each increment or the number of increments if there will not be sufficient coal left for the general-analysis sample after the removal of the moisture sample in accordance with ISO 13909-4.

5.3 Taking the increments

Take increments using a mechanical auger or other suitable full-depth mechanical sampler; the use of an auger is preferred (see <u>Clause 7</u>). Ensure that the sampler penetrates the full depth of coal and that a full column of coal is extracted, so that a representative increment is obtained. Large and hard pieces of coal or rock shall not be pushed aside deliberately when an increment is collected. Do not allow wet coal to adhere to the sampling equipment.

5.4 Distribution of increments

5.4.1 Wagons

5.4.1.1 Selection of wagons

If the number of increments required is less than the number of wagons in the sub-lot, take one increment from each of that number of wagons. When the number of increments required is greater than the number of wagons in the sub-lot, the number of increments taken from each wagon shall be determined by dividing the total number of increments by the number of wagons; if after this division there is a remainder of increments, these shall be distributed over the sub-lot. The selection of wagons may either be systematic (e.g. every third wagon) or random (see <u>5.4.4</u>).

5.4.1.2 Position of increments within wagons

Vary the positions of the increments from wagon to wagon so that all parts are represented as much as possible. There will be locations in wagons that cannot be augured due to getting too close to the sides or the bed. Thus, all parts may not be accessible. There are various methods of doing this and different schemes may be preferred for use with different designs or sizes of wagons.

For example, the coal surface in the wagon could be divided into numbered squares, each side about 1 m, the number of squares being dependent on the size of the wagon. If only a single increment is required from each wagon, systematic sampling can be used, i.e. taking increments from the numbered squares in rotation. In all other circumstances, random selection shall be used (see <u>5.4.4</u>).

5.4.2 Barges

Although barges or even their holds are generally larger than wagons, the method of distribution of increments is, in principle, the same. Barges and/or barge-holds shall therefore be sampled using the procedures given in 5.3 and 5.4.1.2.

5.4.3 Ships

Sampling of stationary coal in the holds of large barges and large ships is impracticable because of the difficulty of obtaining a full-depth representative sample. This difficulty increases with the size of the vessel and the normal practice with large vessels is to sample the coal from a moving stream (in accordance with ISO 13909-2) at port facilities during loading or unloading operations. Hence, sampling from the holds of such vessels is excluded from this part of ISO 13909.

5.4.4 Random selection of increments

Identify all the possible sampling areas (wagons, barges, barge-holds or parts thereof) and number them. Select the areas to be sampled by one of the following methods:

a) generate a random number for each increment required from a set corresponding to the total identified;

or

- b) provide a set of numbered discs, one disc corresponding to each sampling area, and then proceed as follows.
 - 1) When selecting wagons, barges or barge-holds, place the discs in a bag and draw sufficient discs from the bag to coincide with the total number to be sampled. Attach the selected discs to a reference board and sample those wagons, barges or holds corresponding to the numbers on the selected discs.
 - 2) When selecting sampling areas within containers (wagons, barges or holds, see Figure 1), place the discs in a bag close to the sampling point and provide a diagram on a fixed board showing the locations of the areas across the surface of the coal. To sample the first selected container, draw sufficient discs from the bag to coincide with the total number of increments to be taken from that container and take an increment from those areas corresponding to the numbers on the selected discs. Place these discs in a second bag after use. For the second container, follow the same procedure by drawing discs from those remaining in the first bag. Continue this process for subsequent containers until all the discs are used up and then swap the bags over so that discs are drawn from the second bag and placed in the first bag.

NOTE This procedure ensures that the order of the sampling areas from which increments are taken is always different.

1	4	7	10	13	16
2	5	8	11	14	17
3	6	9	12	15	18

Figure 1 — Example of sampling areas within a container

6 Methods of sampling from stockpiles

Sample a stockpile when it is laid down or picked up by the method for sampling of moving streams described in ISO 13909-2. If it is not possible to sample the coal in this way, use the following method.

The sampling of a stockpile in situ usually presents problems in obtaining a representative sample and may be used only if it is not possible to sample the coal as a moving stream.

Stockpiles which are to be sampled from the surface shall be sufficiently compacted to safely bear the weight of sampling personnel and equipment.

Determine the number of sub-lots in accordance with <u>4.3.4</u> and calculate the mass of a sub-lot by dividing the mass of the lot by the number of sub-lots. Take increments using a mechanical auger (see <u>Clause 7</u>). Ensure that a full column of coal is extracted, so that a representative increment is obtained, by using equipment that can penetrate to the bottom of the stockpile. Choose the positions of the boreholes so that all parts of the stockpile are represented by one of the following methods:

a) divide the surface of the stockpile on a grid basis and take a core from each section of the grid;

b) carry out an initial ground survey and then take cores across the stockpile on the basis of sampling from equal volumes of coal.

7 Sampling equipment — mechanical auger

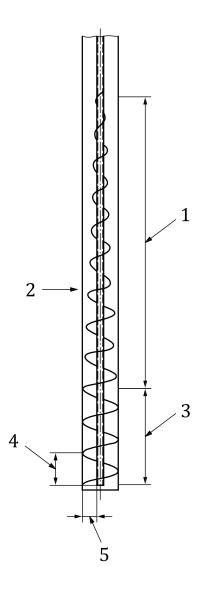
There are few commercially available, power-operated samplers which are suitable for mechanical sampling of coal. It is essential that any mechanical sampler used is capable of taking full-depth samples. One such sampler is the mechanical auger. Using an auger for sampling is preferred, but is impractical in some circumstances, for example, sampling of fuels of large top size.

An auger consists of a cylindrical steel tube containing an Archimedian screw, shown in Figure 2, which is mounted on a structure in such a way that it takes a vertical core from the full depth of the coal. The pitch of the screw and the annular gap (the distance between the shaft and the inside of the tube) shall each be at least three times the nominal top size of the coal.

One particular design of Archimedian screw contains relatively few turns of the flight, leaving a space above the base of the auger which becomes filled with a column of coal during operation.

An alternative design for a mechanical coal sampler is shown in <u>Figure 3</u>, which is a rotary cylinder sampler.

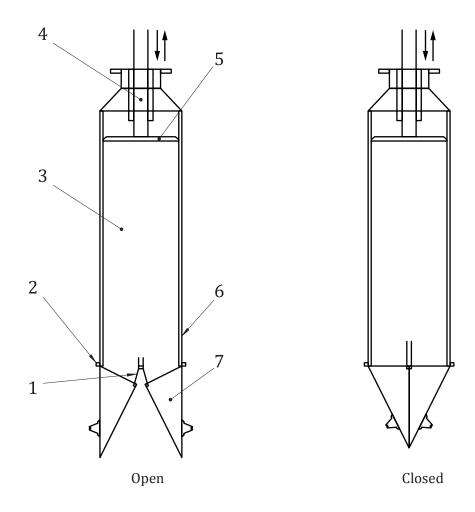
All mechanical sampling equipment shall be checked for bias in accordance with ISO 13909-8, by comparing samples obtained using the equipment with samples taken by the stopped-belt reference method.



Key

- 1 tapered flights
- 2 auger tube
- 3 full flights
- 4 pitch: 3 × top size
- 5 annular gap: 3 × top size

Figure 2 — Example of a mechanical auger



Key

- 1 connecting board
- 2 hinge
- 3 storage cylinder
- 4 piston push rod

- 5 sample discharger (unloading)
- 6 outer cylinder
- 7 claw

Figure 3 — Example of a rotary cylinder sampler

8 Handling and storage of samples

Place the increments or divided increments as quickly as possible in sample containers and take appropriate precautions to minimize moisture losses during sampling. Seal the containers immediately after sampling is completed.

The increments or divided increments from each sub-lot shall be placed in a separate container or set of containers; if duplicate samples are required, a separate container or set of containers shall be provided for each duplicate sample.

If common samples or moisture samples are required, the sample containers shall be impervious to water and vapour and have sufficient mechanical strength to ensure that the integrity of the sample will not be impaired during removal to the sample preparation site.

If general-analysis test samples are required, the sample containers for such samples shall provide adequate protection against contamination and loss of sample material.

Moisture samples and common samples shall be kept in a cool, dry place during any storage, and the moisture content shall be determined as quickly as possible after sample collection.

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The sample in each sample container shall be fully and permanently identifiable. It is recommended that, for this purpose, the container be provided with two waterproof tags, each marked by means of waterproof ink with adequate identifying information, one tag being placed on the outside of the container and one being placed inside the container; if a plastic inner liner is used, the latter tag should be placed inside this liner.

When using a mechanical auger, unusually large increments are collected. Immediately after taking the increments, they should be prepared to manageable size using an on-line or off-line preparation facility. These preparation facilities shall be tested for bias.

9 Sample preparation

Sample preparation shall comply with the requirements of ISO 13909-4.

10 Minimization of bias

10.1 Causes of bias

The test results obtained from samples may be biased for a number of reasons. The causes of bias resulting from operation of the sampling equipment and the actions to be taken to minimize them are given in a) to d) below.

a) Improper operation

Inspection and/or measurement of the operating parameters shall be documented to verify compliance with the sampling plan, as well as the system specifications.

b) Improper maintenance

Maintenance of the sampler components shall be scheduled and documented by hours of use. Special attention shall be given to the maintenance of items that wear and/or need adjustments. For example, seals may wear causing material to be lost or drying to occur.

c) Improper cleaning

The mechanical sampling system shall be cleaned between lots to avoid sample contamination. Access to the interior of the system components is therefore essential.

d) Segregation

Segregation of particle sizes may occur as coal is loaded into wagons, barges, ships and stockpiles. It may be very difficult to capture the size distribution of a lot in these cases, therefore, a bias in size is likely to result in the sample.

10.2 Checking for precision and bias

The precision of sampling shall be checked using the methods described in ISO 13909-7 and, if necessary, adjustments made to the number of increments and/or sub-lots to achieve the specified precision. To this end, the scheme shall be designed so that increments can be processed separately and included alternately in at least two separate samples to produce replicate samples. It is not permitted to prepare duplicate samples from a number of increments already compounded.

The mechanical sampler shall be checked for bias by comparing the analysis of reference samples taken by stopped-belt sampling as the batch of test material is moved and off-line preparation with that taken from the same coal by the mechanical system (see ISO 13909-8). This is of particular importance when moisture content is to be determined on the crushed sample.

If preparation components are added to the sampler, they shall also be checked for bias.

11 Verification

Proper design shall be verified prior to installation and use. After installation, proper design shall be verified by conducting a bias test of the sampling scheme in accordance with ISO 13909-8.

Sampling plants shall be rechecked for bias at predetermined intervals as part of a routine maintenance plan.

NOTE The time intervals between these routine bias tests will depend on the throughput and type of fuel and on any modification/alteration of the system.

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[1] Holmes R.J. 1987), Assessment of Minimum Sample Mass for Coal, Mineral Engineering Communication, MIE/C23, CSIRO Division of Mineral Engineering, May 1987, 22 pp





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