

INTERNATIONAL
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2016-07-01

**Hard coal and coke — Mechanical
sampling —**

Part 2:

Coal — Sampling from moving streams

Houille et coke — Échantillonnage mécanique —

Partie 2: Charbon — Échantillonnage en continu



Reference number
ISO 13909-2:2016(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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 13909-2:2001), 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*

Annex B forms a normative part of this part of ISO 13909. Annex A of this part of ISO 13909 is 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[1].

2 Normative references

The following 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 13909-1:2016, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*

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*

ISO 21398, *Hard coal and coke — Guidance to the inspection of mechanical sampling systems*

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 the variance of preparation and testing are given in ISO 13909-7.

- f) Establish the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see 4.3.4).
- g) Decide whether to use time-basis or mass-basis sampling (see Clause 5) and define the sampling intervals in minutes for time-basis sampling or in tonnes for mass-basis sampling.
- h) Ascertain the nominal top size of coal for the purpose of determining the minimum mass of sample (see 4.4 and Table 1).

NOTE The nominal top size may initially be ascertained by consulting the consignment details, or by visual estimation, and may be verified, if necessary, by preliminary test work.

- i) Determine the minimum average increment mass (see 4.5).

4.2 Design of the sampling scheme

4.2.1 Material to be sampled

The first stage in the design of the scheme is to identify the 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 dispatched 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 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. Of these two alternatives, time-basis sampling is easier to implement and verify, because only a fixed speed cutter and a timing device are required. On the other hand, for mass-basis sampling, a conveyor belt weightometer is required as well as a device that is controlled sufficiently to adjust the primary cutter speed increment by increment to achieve uniform increment mass.

4.2.4 Precision of sampling

After the desired sampling 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). 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. 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.

4.2.5 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).

Sampling systems shall be tested 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, 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.

The desired 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:

$$P_L = 2\sqrt{\frac{V_1 + V_{PT}}{n}} \quad (1)$$

where

P_L is the estimated index of overall precision of sampling, sample preparation and testing for the lot, expressed as a percentage absolute;

V_1 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 the 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 0,20 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 the following:

- 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 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 indicate that some changes are required 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 are 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, n , in each sub-lot for a desired precision from the following formula [obtained by transposing Formula (1)]:

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

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 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).

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

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 it is 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, the preparation and testing errors are included more than once.

Example 1

The lot is 20 000 t 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 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

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

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 delivered as 5 000 t/day over two shifts.

Required precision, $P_L = 0,25$ % ash

Primary increment variance, $V_1 = 5$

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

$$n = \frac{4 \times 5}{(20 \times 0,25^2) - (4 \times 0,20)} = 45 \text{ using Formula} \quad (2)$$

If this number of increments is considered to be too large, increase the number of sub-lots to 40, i.e. one per shift.

$$n = \frac{4 \times 5}{(40 \times 0,25^2) - (4 \times 0,20)} = 12$$

It would then be sensible to take 12 increments per shift, i.e. one every 40 min.

Example 3

The lot is 100 000 t of washed coal delivered at 10 000 t/h via a shiploading conveyor.

Required precision, $P_L = 0,2$ % ash

Primary increment variance, $V_1 = 3$ (washed coal)

Preparation and testing variance, $V_{PT} = 0,05$

a) Initial number of sub-lots

Take an hourly sample, i.e. $m = 10$.

b) Number of increments per sub-lot

$$n = \frac{4 \times 3}{(10 \times 0,2^2) - (4 \times 0,05)} = 60 \text{ using Formula} \quad (2)$$

Therefore, divide the lot into 10 sub-lots and take increments at 1 min intervals.

Example 4

The lot is 8 000 t in a single load and the required precision, P_L , is 0,5 % ash. The quality variation is known and the following values have been determined:

primary increment variance, $V_1 = 5$;

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 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 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 4.3.4).

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 column 2 of Table 1 (see Reference [3]). 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.

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

Nominal top size of coal mm	General-analysis samples and common samples kg	Samples for determination of total moisture content 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.

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

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 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.

4.5 Mass of primary increment

The mass, m_I , in kilograms, of an increment taken by a mechanical cutter with cutting edges normal to the stream at the discharge of a moving stream can be calculated from [Formula \(5\)](#).

$$m_I = \frac{Cb \times 10^{-3}}{3,6 v_C} \quad (5)$$

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 [Formula \(6\)](#).

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

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 [Formula \(7\)](#).

$$m_I' = \frac{m_S}{n} \quad (7)$$

where

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

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

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. 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 Table 2. 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 mm	Minimum mass for a precision of 1 % kg	Minimum mass for a precision of 2 % 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

5.1 General

Sampling shall be carried out by systematic sampling, either on a time-basis or on a mass-basis, or by stratified random sampling. The procedures of sample preparation vary in accordance with the type of sampling employed (see ISO 13909-4).

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

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

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

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

5.2 Time-basis sampling

5.2.1 Method of taking primary increments

In order that the increment mass is proportional to the coal flow rate in mechanical sampling, the speed of the cutter shall be constant throughout the sampling of the entire sub-lot (see 6.8.1).

Primary increments shall be taken at preset equal time intervals throughout the lot or sub-lot. If the calculated number of increments has been taken before the handling has been completed, additional increments shall be taken at the same interval until the handling operation is completed.

5.2.2 Sampling interval

The time interval, Δt , in minutes, between taking primary increments by time-basis sampling is determined by Formula (8).

$$\Delta t \leq \frac{60 m_{\text{SL}}}{G n} \quad (8)$$

where

m_{SL} is the mass of the sub-lot, in tonnes;

G is the maximum flow rate on the conveyor belt, in tonnes per hour;

n is the number of primary increments in the sample (see 4.3.4).

In order to minimize the possibility of any bias being introduced, a random start within the first sampling interval is recommended.

5.2.3 Mass of increment

The mass of the primary increment corresponding to the average flow rate (total mass/operating time) of the coal stream shall be not less than the minimum average increment mass calculated from Formula (7).

The mass of the increment shall be proportional to the flow rate of the coal stream at the time it is taken.

5.3 Mass-basis sampling

5.3.1 Method of taking primary increments

For mechanical sampling, either a fixed- or a variable-speed cutter may be used.

The required number of increments shall be taken by sampling at a preset mass interval. This interval shall not be changed during the sampling of the sub-lot.

If the calculated number of increments has been taken before the handling has been completed, additional increments shall be taken at the same interval until the handling operation is completed.

5.3.2 Sampling interval

The increments shall be spread uniformly on a tonnage basis throughout the mass of the lot or sub-lot.

The mass interval, Δm , in tonnes, between taking increments by mass-basis sampling is determined from [Formula \(9\)](#).

$$\Delta m = \frac{m_{SL}}{n} \quad (9)$$

where

m_{SL} is the mass of the sub-lot, in tonnes;

n is the number of primary increments in the sample.

The mass interval between increments shall be equal to or smaller than that calculated from the number of increments specified in [4.3.4](#), in order to ensure that the number of increments will be at least the minimum number specified.

In order to minimize the possibility of introduction of bias, a random start within the first sampling interval is recommended.

5.3.3 Mass of increment

The masses of the individual increments constituting the sample shall be almost constant, i.e. the coefficient of variation shall be less than 20 %, and there shall be no correlation between the flow rate at the time of taking the increment and the mass of the increment constituting the sample. The method for determining whether or not these criteria have been met is specified in [Annex A](#). These criteria may be achieved by either of the following procedures:

- a) take primary increments of almost constant mass using a cutter with variable speed, whose cutter speed is constant while cutting the stream but can be regulated, increment by increment, in proportion to the flow rate of the coal at the point of sampling;
- b) take primary increments using a fixed-speed cutter with subsequent division of the individual increments to almost constant mass at a practical stage prior to the constitution of the sample.

NOTE Procedure a) is preferred for falling-stream samplers; only procedure b) can be used for cross-belt samplers.

5.4 Stratified random sampling

5.4.1 General

Cyclical variations in coal quality may occur during sampling. Every effort shall be made to eliminate coincidence of the cycle with the taking of increments in systematic sampling, be it time- or mass-basis. If this cannot be done, a bias will invariably be introduced that may be of unacceptable proportions. In such circumstances, stratified random sampling may be adopted in which, for each time or mass interval, the actual taking of the increment is displaced by a random amount of time or mass respectively, subject to the limitation that it shall be taken before that interval has expired.

During stratified random sampling, it is possible that two increments will be collected very close together even though they are collected in different mass or time intervals. It is therefore necessary

that the discharge bin of the primary sampler be of sufficient size to accept a minimum of two primary increments at the maximum flow rate.

5.4.2 Time-basis stratified random sampling

The sampling interval shall be determined as in 5.2.2 and the increment mass as in 5.2.3. Prior to the start of each sampling interval, a random number between zero and the sampling interval shall be generated. The increments shall then be taken after the time indicated by the random number. The mass of the increment shall be proportional to the flow rate of the coal (see 5.2.3).

5.4.3 Mass-basis stratified random sampling

The sampling interval shall be determined as in 5.3.2 and the increment mass as in 5.3.3. Prior to the start of each sampling interval, a random number between zero and the mass of the sampling interval (tonnes) shall be generated. The increment shall be taken after the passage of the mass of coal indicated by the random number. The mass of the increment shall be independent of the flow rate of the coal (see 5.3.3).

5.5 Reference sampling

Reference samples for bias testing of a sampling system shall be taken by the stopped-belt method given in ISO 13909-8 to enable checking for bias.

6 Design of mechanical samplers

6.1 Safety

From the initial stages of design and construction of a system, it is essential that due consideration be given to the safety of the operators. All safety codes applicable at the site where the equipment is to be installed shall be respected.

6.2 Information

It is essential that relevant information concerning the sampling scheme(s) (Clause 4), the method of sampling (Clause 5) and sample preparation (see ISO 13909-4), as well as information about the design and operation of the coal handling plant are available to the designer.

6.3 Basic requirements

It is important that the coal-handling plant be designed and engineered to provide adequate space and satisfactory operating and sampling conditions for the sampling system. The importance of considering the requirements of the sampling systems from the first stage of main plant design cannot be overstressed. It is necessary to ensure that any subsequent changes do not affect the overall performance and reliability of the sampling system. Designers shall take heed of the checks that need to be carried out during operation. Facilities for taking replicate samples and stopped-belt sampling shall be included at the design stage.

The system shall be readily accessible throughout to facilitate inspection, thorough cleaning, repairs or checking experiments, e.g. tests for bias.

NOTE For a mass-basis sampling system, it is useful for provision to be made for conversion from mass-basis to time-basis sampling in the event that the mass-monitoring device breaks down.

6.4 Location of sampling equipment

The location of the sampling equipment shall be chosen according to the following criteria:

- a) the sampling system shall be located at a position which allows access to the whole lot, at the stage in the process where the measurement of quality and quantity is required;
- b) if variable flow rates result in increment masses which are unacceptable for the projected system (see 5.2.3), consideration shall be given to providing suitable holding facilities upstream of the sampling system in order to obtain a more uniform flow, e.g. surge hoppers with adjustable gates.

6.5 Provision for checking precision

Samplers shall be capable of allowing the checking of precision by one of the procedures given in ISO 13909-7.

6.6 Provision for testing for bias

To allow for bias tests to be carried out in accordance with ISO 13909-8, provision shall be made for stopped-belt sampling.

6.7 General requirements for designing mechanical samplers

The principal requirements when designing and constructing a mechanical sampler are as follows.

- a) It shall be capable of collecting increments so that bias is minimized.
- b) It shall maintain this capability under all such conditions of sampling that are stipulated in the relevant specifications and without necessitating that sampling be interrupted for cleaning or maintenance.

In order to meet these requirements, the sampler shall be designed so that

- a) the sampling device is sufficiently robust to withstand the most adverse operating conditions expected,
- b) the sampling device has sufficient capacity to retain completely, or to pass entirely, the increment without loss or spillage,
- c) the sampling device, and the system as a whole, including dividers, chutes, hoppers, feeders, crushers and other equipment shall operate in a manner that facilitates material flow and minimizes the need for cleaning to prevent and clear blockages,
- d) any contamination of the sample is avoided, (e.g. material entering cutters which are in the parked position or when a change is made in the type of coal being sampled),
- e) degradation of the constituent particles is minimized if a sample is taken for particle-size determination, and
- f) any changes in moisture, chemical or physical properties or loss of fine coal (for example, due to excessive air flow through the equipment) are minimized.

6.8 Design of falling-stream-type samplers

6.8.1 General

When designing a sampling device, the cutter velocity, cutter aperture and the angle of presentation of the cutter to the coal stream are important design criteria. These criteria shall be considered jointly because the presentation of the cutter to the stream and cutter velocities affect the "effective" cutter aperture presented to the particles in the stream.

The design objective is to ensure that the mean trajectory of the particles in the stream is as close to normal to the plane of the cutter aperture as possible, to maximize the effective cutter aperture. The cutter velocity is particularly important in this regard because the particles in the stream intercept the cutter aperture at increasingly oblique angles as the cutter velocity increases, thereby, reducing the effective cutter aperture. This places an upper limit on the acceptable cutter velocity.

Examples of different types of falling-stream samplers are shown in [Figure 1](#).

NOTE Other primary sampling devices which conform to the principles laid down in this part of ISO 13909 may be acceptable, providing that they can be shown to minimize bias.

A cutter intended for sampling from a falling stream of coal shall be designed in accordance with the following requirements:

- a) the cutter shall take a complete cross-section of the stream;
- b) the plane or cylindrical surface described by the leading and the trailing cutting edges shall be normal to the mean trajectory of the stream;
- c) the cutter shall travel through the coal stream at a uniform velocity, i.e. the velocity shall not deviate by more than 5 % from the preselected reference velocity while traversing the coal stream (see [6.8.2](#));
- d) the design of the cutter aperture shall be such that all parts of the stream are exposed to the aperture for the same length of time;
- e) the width of the cutter aperture shall be at least three times the nominal top size of the coal to be sampled. The cutter aperture of a primary cutter shall not be less than 30 mm. If the cutter aperture is tapered, as is the case with some swing-arm-type samplers, e.g. the type shown in [Figure 1 d](#)), the minimum width requirement given above shall apply to the width at the narrowest point where the cutter intercepts the coal stream;
- f) the effective capacity of the sample cutter shall be determined on the basis of the expected maximum flow rate of the coal stream. Under these conditions, the sample cutter shall completely retain or entirely pass the increment without loss or spillage and without any part of the cutter aperture ever being blocked up or restricted by material already collected.

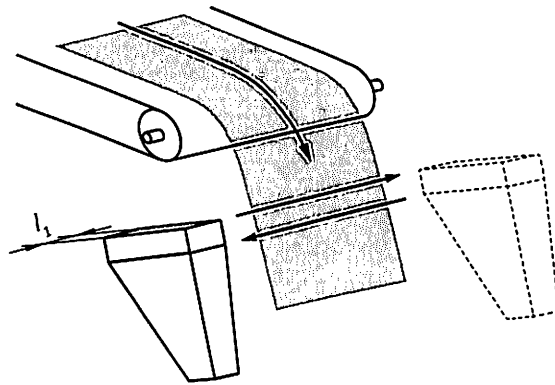
6.8.2 Cutter velocity

The width of the cutter aperture and the cutter velocity are important parameters to be considered when designing a sample cutter. Taken jointly with the velocity of the coal stream, these parameters will determine the effective width of the cutter aperture, i.e. the width of that part of the aperture into which the stream of coal can flow unimpeded.

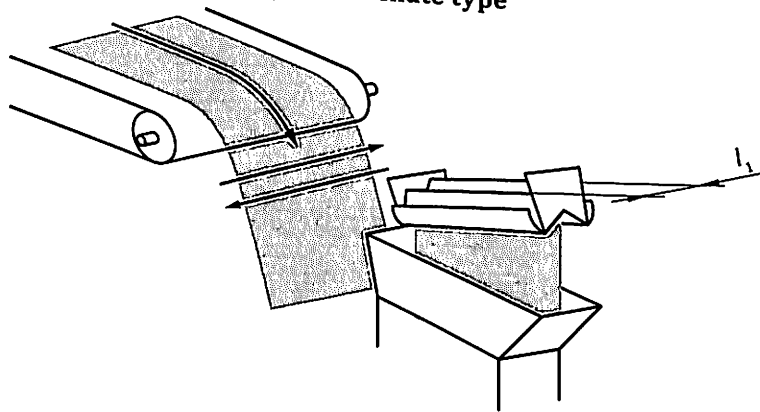
For falling-stream cutters, experimental work on ores (see Reference [2]) has shown that, when sampling heterogeneous material streams of low belt loading (low stream density), where the particle-size distribution is very narrow, bias may be introduced when the cutter speed exceeds 0,6 m/s and/or the cutter aperture is less than three times the nominal top size of the material. The ratio of cutter width to nominal top size of the material will decisively influence the capability of the cutter to take unbiased increments, since the greater this ratio, the less will be the tendency to selectively reject the larger particles.

Modern commercial coal-handling systems have cutters which sample coal streams of large capacity where there is a relatively high stream density and wide particle-size distribution. In such circumstances, cutters, operating at speeds up to 1,5 m/s have been shown to be free from bias provided that the ratio of cutter aperture to coal nominal top size is a minimum of three.

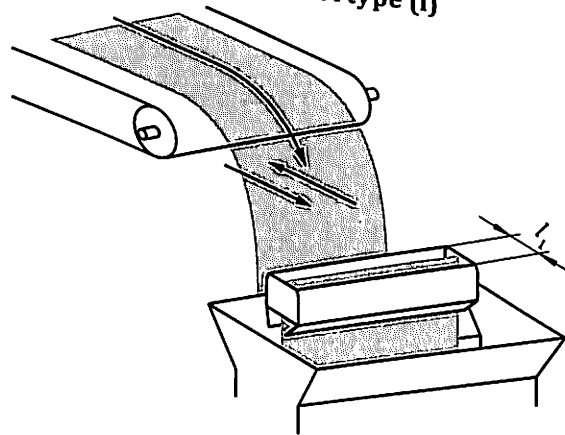
Irrespective of the cutter speed and aperture, cutters shall be shown to minimize bias.



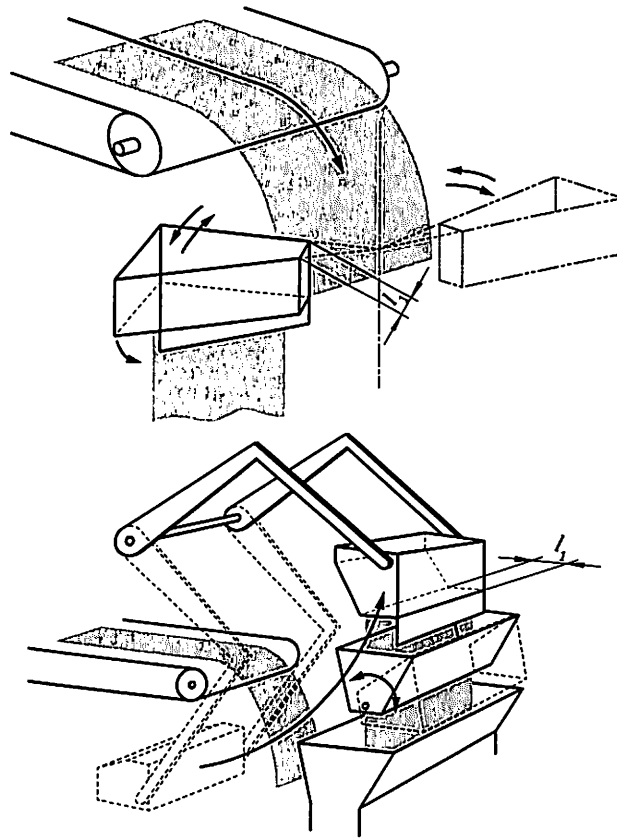
a) Cutter-chute type



b) Cutter bucket type (i)



c) Cutter bucket type (ii)



d) Swing arm types

Figure 1 — Examples of falling-stream samplers

6.9 Cross-belt-type primary samplers

6.9.1 Operation

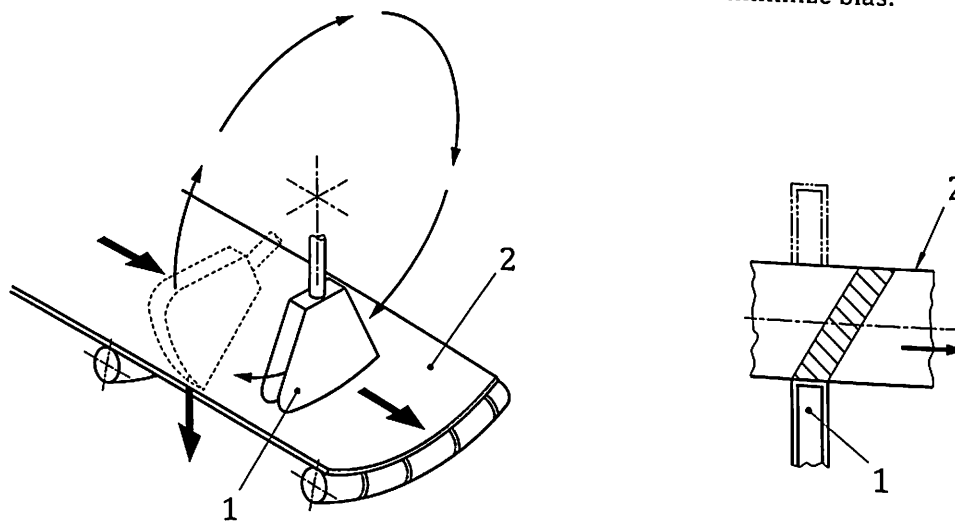
The principles of operation of cross-belt samplers are shown in Figure 2, which illustrates two different examples of such samplers. In both cases, the sampling cutter pivots on an axis parallel to the centre-line of the belt. As the cutter traverses the full width of the belt in a rotary motion, the leading edges of the side plates cut out the increment and the back plate pushes it off the belt.

The two samplers, however, differ considerably as regards the movement of the cutter relative to the coal on the belt. For the sampler shown in Figure 2 a), the bearings, in which the cutter shaft is fitted, are fixed in space. In the case of the sampler shown in Figure 2 b), the bearings are also fixed in space, but the cutter aperture is angled as shown so that during the sampling operation, the influence of belt velocity on the cutter velocity relative to the coal is completely eliminated.

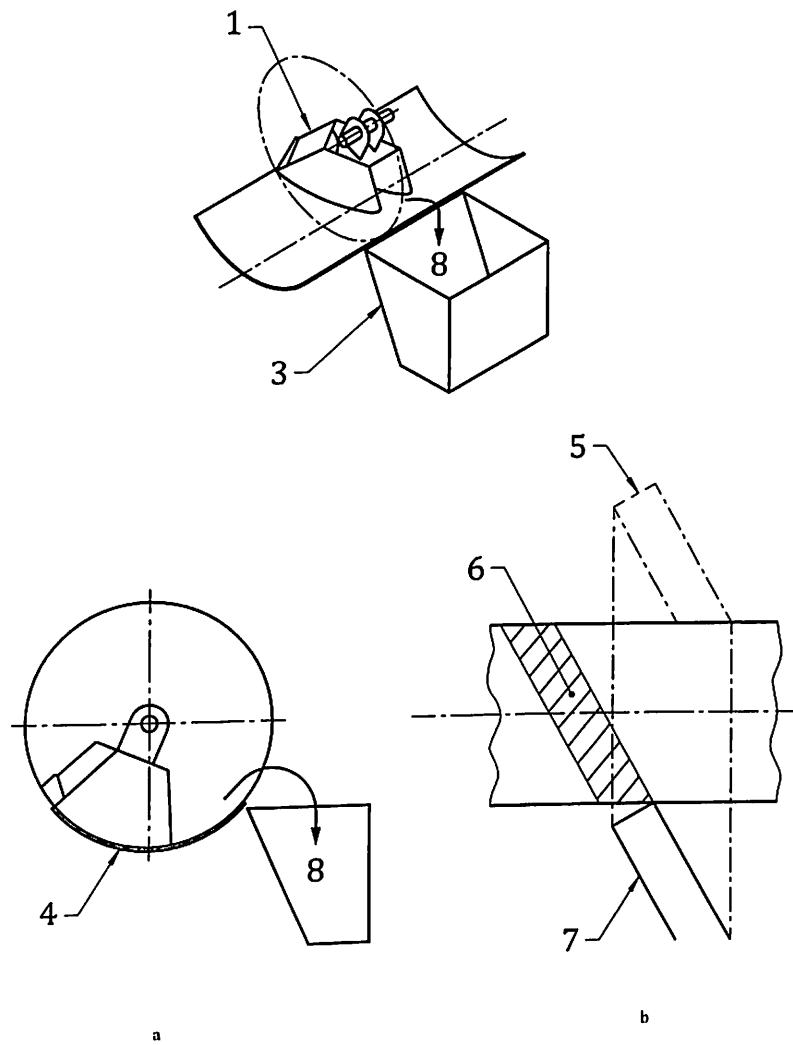
For cross-belt samplers, the relationship between cutter velocity, belt velocity and cutter velocity relative to the coal is important, because the higher the cutter velocity relative to the belt velocity, the larger the effective cutter aperture. Consequently, the sampling conditions are more favourable at higher cutter velocities. Furthermore, the higher the cutter velocity, the shorter the time during which the cutter acts as a plough and holds back the coal stream. For these reasons, the minimum cutter velocity shall be 1,5 times the belt velocity. However, the use of cutter velocities that are too high may result in an unacceptable degree of breakage of sized coal. In such circumstances, the cross-belt sampler may be used at a slower speed with the belt stopped, i.e. using it as a mechanical stopped-belt sampler.

For these reasons, and also because the density of the material to be sampled is considerably higher than in cases of sampling from falling streams, it is undesirable to impose such strict limitations on cutter velocities as those applying to falling-stream samplers.

Irrespective of the cutter speed and aperture, cutters shall be shown to minimize bias.



a) Normal sweep (angled cut) type



b) Angled sweep (angled cut) type

Key

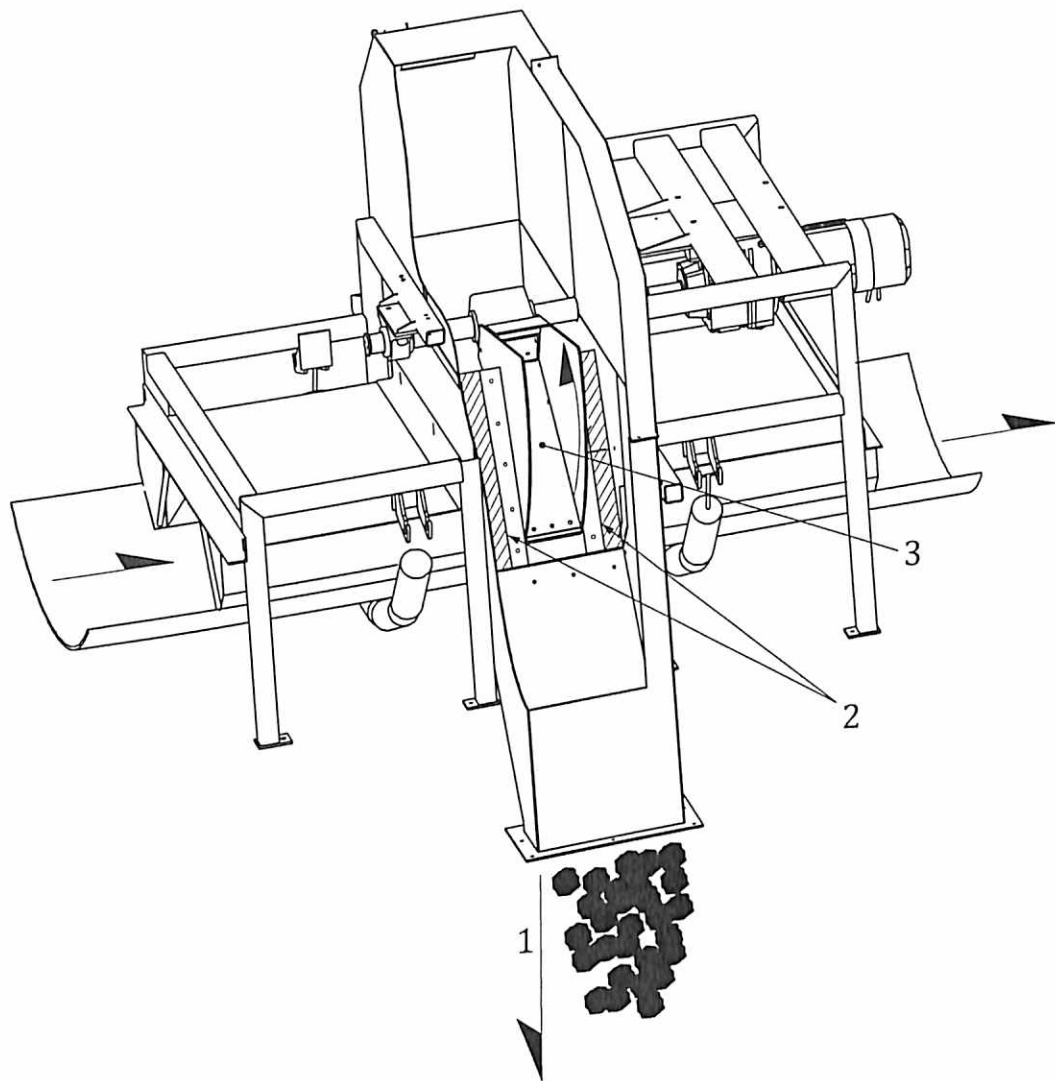
- 1 cutter
- 2 belt supported to maintain curvature
- 3 sample receiver
- 4 belt supported to form curvature
- 5 cutter entering belt
- 6 path of sample cut on belt
- 7 cutter exiting belt
- 8 sample
- a View on end of belt.
- b View on top of belt.

Figure 2 — Examples of cross-belt samplers

6.9.2 Design of cross-belt samplers

Cross-belt samplers shall be designed in accordance with the following criteria:

- a) the cutter lips shall be parallel and shall cut the stream in a plane normal to the centre-line of the conveyor;
- b) the cutter shall take a complete cross-section of the stream, either normal or angled;
- c) the cutter aperture shall be at least three times the nominal top size of the coal being sampled. The minimum cutter aperture of any cutter shall be 30 mm;
- d) the minimum cutter velocity shall be 1,5 times the belt velocity to avoid excessive reduction of the effective cutter aperture while cutting the coal stream;
- e) the cutter shall be of sufficient capacity to accommodate the increment mass obtained at the maximum flow rate of the material;
- f) since fines will tend to be segregated to the bottom of the coal on the belt, in order to avoid selective sampling, the belt curvature shall be profiled to form an arc which is matched by the cutter side plates, and the gap between belt and side plates and/or back plates shall be adjusted to the minimum required to safeguard against direct contact and consequential damage to the belt. In addition, the back plate shall be fitted with brushes and/or resilient skirts to sweep off the bottom layer of coal;
- g) any flexible blades, brushes or skirts fitted to the cutter shall be regularly adjusted so that they maintain close contact with the surface of the moving conveyor belt to ensure that the complete coal cross-section in the path of the cutter is collected from the belt;
- h) reinforced skirting shall be fitted to the sample chute receiving cross-belt increments as shown in Figure 3 to prevent material from outside the sample cutter from falling into the sample collection chute and contaminating the increment.



Key

- 1 sample
- 2 throat skirting
- 3 sample cutter

Figure 3 — Example of a cross-belt sampler showing the location of the reinforced skirting

6.10 Maintenance and checking of sampling equipment

The equipment shall be readily accessible throughout to facilitate inspection, thorough cleaning, repairs or check experiments. Safety codes shall be respected in the design of access points. Inspection and maintenance of the equipment shall be carried out at the intervals recommended by the manufacturer to ensure reliable operation.

All mechanical systems are subject to wear. Such wear may eventually cause a system, which had originally been checked satisfactorily for bias, to produce biased samples. It is essential, therefore, that mechanical sampling systems be the subject of planned maintenance schemes and be inspected frequently in accordance with ISO 21398 to ensure that components have not undergone undue wear or are broken.

The person carrying out the inspection shall be provided with a checklist of items to be noted. This checklist shall include at least the following items:

- a) observation of the taking of an increment;
- b) where a flow-smoothing device is installed, uniformity or otherwise of flow in the stream to be sampled;
- c) excessive spillage in the area of the mechanical samplers which may cause contamination of the sample;
- d) any mechanical changes made to the sampling system or to the coal-handling system immediately upstream of the sampler;
- e) for cross-belt samplers, the wear on the brushes and/or resilient skirts of the cutter;
- f) for cross-stream samplers, check for wear on critical parts;
- g) for cross-stream samplers, cutter speeds should be checked.

Further details on procedures for checking mechanical sampling systems are provided in ISO 21398.

7 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 complete.

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.

If physical test samples are required, the sample containers for such samples shall give adequate protection against loss of sample material. Such samples should be carefully handled at all stages and under all circumstances to prevent breakage and/or degradation.

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.

The sample in each sample container shall be fully and permanently identifiable.

It is useful 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.

NOTE There are circumstances where it is useful that the sample containers be properly and identifiably security-sealed, e.g. with wax, lead or tape.

The label and/or accompanying documents shall give the information detailed in ISO 13909-1:2016, Clause 8.

Referee samples shall be kept available under good custody under conditions which minimize degradation for as long as required.

8 Sample preparation

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

9 Bias

9.1 Minimization of bias

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

a) Improper design

Sampling systems shall be designed to minimize moisture losses incurred throughout the system by tightly enclosing all components, and keeping the flow time of the sample, from collection of primary increment to collection of the final sample, to a minimum.

Requirements for the design of sampling systems are given in 6.7.

b) Improper operation

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

c) Periodicity

The collection of increments at any stage that is synchronized with the cycles of the material flowing to that device, causing increments to be collected that coincide with some cyclical belt loading, or other phenomenon that results in cyclic peaks or valleys of some characteristic of the material being sampled, can result in bias.

In order to avoid bias caused by such cyclical relationships, stratified random sampling shall be used.

In order to avoid the bias caused by the non-random sampling of front runnings or tailings at any stage, the starting time of the first increment shall be independent of the flow of the coal to that sampler.

d) 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. Crusher components may wear, causing an incorrect size material to be introduced to the next stage.

e) Non-adherence to basis of sampling (time-basis or mass-basis)

The sampling operation shall be checked to ensure that the increment masses are strictly flow proportional for time-basis sampling and uniform for mass-basis sampling.

f) 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. If complete cleaning cannot be ensured, it is good practice to purge the system by allowing one or more increments to pass through the system without collecting them in the sample container.

g) Coal flow rate

A consistent coal sample flow rate should be maintained throughout the mechanical sampling system. For example, this can be achieved by using holding hoppers to ensure the full increment, or

batch of increments, pass through a crushing cycle and are completely collected in a hopper before proceeding to the next cutter.

9.2 Checking for precision and bias

The precision of sampling shall be checked at predetermined intervals 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 a sample taken by stopped-belt sampling and off-line preparation with that taken from the same coal by the mechanical crushed sample.

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

10 Verification

Proper design shall be verified prior to installation and use (see 6.8 and 6.9). After installation, proper design shall be verified by conducting a bias test of the sampling system in accordance with ISO 13909-8.

Sampling systems 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.

Sampling systems should also be routinely inspected in accordance with ISO 21398 to ensure that they are operating correctly and conform to the requirements of this part of ISO 13909.

Annex A (normative)

Evaluation of sampling equipment for mass-basis sampling

A.1 General

When using mass-basis sampling, it is essential that the following two requirements are satisfied:

- a) the coefficient of variation of the masses of the individual increments added to the sample shall be less than 20 %;
- b) there shall be no correlation between flow rate and mass of increment.

To check the fulfilment or otherwise of these requirements, proceed as follows.

Take at least 20 increments at flow rates covering the range expected to be encountered. Record the masses, y , of the individual increments at the stage when it is planned to add them to the sample. Also, record for each increment the flow rate, x , at the time of taking it. An example of such data is given in [Table A.1](#).

Table A.1 — Data from checking a mass-basis sampler

Increment number	Flow rate	x^2	Increment mass	y^2	xy
	t/h		kg		
	x		y		
1	1 060	1 123 600	100	10 000	106 000
2	1 050	1 102 500	104	10 816	109 200
3	970	940 900	96	9 216	93 120
4	1 010	1 020 100	105	11 025	106 050
5	950	902 500	94	8 836	8 930
6	860	739 600	86	7 396	73 960
7	720	518 400	68	4 624	48 960
8	840	705 600	75	5 625	63 000
9	890	792 100	82	6 724	72 980
10	970	940 900	104	10 816	100 880
11	1 020	1 040 400	103	10 609	105 060
12	960	921 600	103	10 609	98 880
13	950	902 500	98	9 604	93 100
14	970	940 900	101	10 201	97 970
15	910	828 100	83	6 889	75 530
16	880	774 400	92	8 464	80 960
17	920	846 400	100	10 000	92 000
18	970	940 900	95	9 025	92 150
19	990	980 100	96	9 216	95 040
20	1 020	1 040 400	103	10 609	105 060
Totals	18 910	18 001 900	1 888	180 304	1 799 200
	$\sum x$	$\sum x^2$	$\sum y$	$\sum y^2$	$\sum xy$

A.2 Coefficient of variation of increment masses

Using the data in Table A.1, calculate the coefficient of variation, CV, for the increment masses using Formulae (A.1) to (A.4).

The mean, \bar{y} , is calculated from Formula (A.1).

$$\bar{y} = \frac{\sum y}{n}$$

where

(A.1)

$\sum y$ is an abbreviation for $\sum_{i=1}^n y_i$, i.e. the sum of all the observations;

n is the number of observations.

Hence, $\bar{y} = \frac{1888}{20} = 94,4$ kg [using Formula (A.1)]

The variance, V , is calculated from Formula (A.2).

$$V = \frac{\sum y^2 - \frac{(\sum y)^2}{n}}{n-1} \quad (\text{A.2})$$

where $\sum y^2$ is an abbreviation for $\sum_{i=1}^n y_i^2$, i.e. the sum of the squared observations.

Hence:

$$V = \left(\frac{180\,304 - \frac{1888^2}{20}}{19} \right) = 109,31$$

The standard deviation, s , is calculated from Formula (A.3).

$$s = \sqrt{V} \quad (\text{A.3})$$

Hence:

$$s = \sqrt{109,31} = 10,45$$

The coefficient of variation, CV , is the standard deviation expressed as a percentage of the mean and is calculated from Formula (A.4).

$$CV = \frac{s \times 100}{y} \quad (\text{A.4})$$

Therefore:

$$CV = \frac{10,45 \times 100}{94,4} = 11,07\%$$

If CV is greater than 20 %, calculate the χ^2 critical value for the confidence interval defining the CV as follows.

$$\chi^2 = \left[\frac{(n-1)CV^2}{20^2} \right] \quad (\text{A.5})$$

If χ^2 is greater than or equal to the value read from Table A.2 with $f = n-1$ degrees of freedom, conclude that the requirement for mass basis sampling is not met.

For example, if $n = 25$ and $CV = 26\%$

$$\chi^2 = \frac{(25-1)26^2}{20^2} = 40,56$$

The table value for 24 degrees of freedom is 36,4. Since the calculated statistic, χ^2 , is greater than the table value, the coefficient of variation is significantly greater than 20 % and the requirement for mass-basis sampling is not met.

Table A.2 — Excerpt from a table of the fractiles of chi-squared, using the one-sided distribution case and a 95 % confidence coefficient

<i>f</i>	5	6	7	8	9	10	11	12	13	14	15	16	17
χ^2	11,1	12,6	14,1	15,5	16,9	18,3	19,7	21,0	22,4	23,7	25,0	26,3	27,6
<i>f</i>	18	19	20	21	22	23	24	25	26	27	28	29	30
χ^2	28,9	30,1	31,4	32,7	33,9	35,2	36,4	37,7	38,9	40,1	41,3	42,6	43,8

A.3 Correlation between increment mass and flow rate

Calculate the correlation coefficient between the flow rate and increment mass. The correlation coefficient, *r*, which is a measure of possible relationship between the two sets, can be calculated from Formula (A.6).

$$r = \frac{\sum xy - \frac{\sum x \sum y}{n}}{\sqrt{\sum x^2 - \frac{(\sum x)^2}{n}} \sqrt{\sum y^2 - \frac{(\sum y)^2}{n}}} \quad (\text{A.6})$$

where

x and *y* represent the members of sets of paired data;

$\sum xy$ is the sum of the products of the paired data;

n is the number of pairs.

Hence, for the data in Table A.1:

$$r = \frac{\left(1\,799\,200 - \frac{18\,910 \times 1\,888}{20}\right)}{\sqrt{\left(1\,800\,1900 - \frac{18\,910^2}{20}\right)} \times \sqrt{\left(180\,304 - \frac{1\,888^2}{20}\right)}} = 0,884$$

A statistic, t_c , is calculated from Formula (A.7).

$$t_c = r \frac{\sqrt{n-2}}{\sqrt{1-r^2}}$$

Hence:

$$t_c = 0,884 \frac{\sqrt{20-2}}{\sqrt{1-0,884^2}} = 8,023 \quad (\text{A.7})$$

The value of t_c is compared with the value of Students' *t* in Table A.3 at (20 - 2) degrees of freedom. If the value of t_c is greater than the value of *t* found in this table at (*n* - 2) degrees of freedom, then correlation is present. Since t_c is greater than *t* (18) = 2,101, it is concluded that there is correlation between flow rate and increment mass, and the sampler shall therefore be corrected and retested before use for mass-basis sampling.

Table A.3 — Value of Student's t at 95 % confidence level for two-tailed distributions

Degrees of freedom	Two-tail, t_{α}
5	2,571
6	2,447
7	2,365
8	2,306
9	2,262
10	2,228
11	2,201
12	2,179
13	2,160
14	2,145
15	2,131
16	2,120
17	2,110
18	2,101
19	2,093
20	2,086
21	2,080
22	2,074
23	2,069
24	2,064
25	2,060
26	2,056
27	2,052
28	2,048
29	2,045
30	2,042

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