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

Part 2:

Coal — Sampling from moving streams

Houille et coke — Échantillonnage mécanique —

Partie 2: Charbon — Échantillonnage en continu



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Printed in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 13909 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13909-2 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

ISO 13909 cancels and replaces ISO 9411-1:1994, *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal* and ISO 9411-2:1993, *Solid mineral fuels — Mechanical sampling from moving streams — Part 2: Coke*, of which it constitutes a technical revision. It also supersedes the methods of mechanical sampling of coal and coke given in ISO 1988:1975, *Hard coal — Sampling* and ISO 2309:1980, *Coke — Sampling*.

ISO 13909 consists of the following parts, under the general title *Hard coal and coke — Mechanical sampling*:

- *Part 1: General introduction*
- *Part 2: Coal — Sampling from moving streams*
- *Part 3: Coal — Sampling from stationary lots*
- *Part 4: Coal — Preparation of test samples*
- *Part 5: Coke — Sampling from moving streams*
- *Part 6: Coke — Preparation of test samples*
- *Part 7: Methods for determining the precision of sampling, sample preparation and testing*
- *Part 8: Methods of testing for bias*

Annex B forms a normative part of this part of ISO 13909. Annexes A and C of this part of ISO 13909 are for information only.

Hard coal and coke — Mechanical sampling —

Part 2:

Coal — Sampling from moving streams

1 Scope

This part of ISO 13909 specifies procedures and requirements for the design and establishment of mechanical samplers for the sampling of coal from moving streams and describes the methods of sampling used.

It does not cover mechanical sampling from stationary lots which is dealt with in ISO 13909-3.

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13909. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13909 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 13909-1:2001, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*.

ISO 13909-4:2001, *Hard coal and coke — Mechanical sampling — Part 4: Coal — Preparation of test samples*.

ISO 13909-7:2001, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*.

ISO 13909-8:2001, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*.

3 Terms and definitions

For the purposes of this part of ISO 13909, the terms and definitions given in ISO 13909-1 apply.

4 Establishing a sampling scheme

4.1 General

The general procedure for establishing a sampling scheme is as follows.

- a) Define the quality parameters to be determined and the types of samples required.
- b) Define the lot.
- c) Define or assume the precision required (see 4.4.1).
- d) Decide whether continuous or intermittent sampling is to be used (see 4.2).

- e) Determine the method of combining the increments into samples and the method of sample preparation (see ISO 13909-4).
- f) Determine or assume the variability of the coal (see 4.4.2 and, if relevant, 4.4.3) and the variance of preparation and testing (see 4.4.4). Methods for determining variability and the variance of preparation and testing are given in ISO 13909-7.
- g) Establish the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see 4.4.5).
- h) Decide whether to use time-basis or mass-basis sampling (see clause 5) and define the sampling intervals in minutes for time-basis sampling or in tonnes for mass-basis sampling.
- i) Ascertain the nominal top size of coal for the purpose of determining the minimum mass of sample (see 4.5).

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

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

4.2 Continuous and intermittent sampling

4.2.1 Continuous sampling

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

4.2.2 Intermittent sampling

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

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

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

4.3 Design of the sampling scheme

4.3.1 Material to be sampled

The first stage in the design of the scheme is to identify the coal to be sampled. Samples may be required for technical evaluation, process control, quality control and for commercial reasons by both the producer and the customer. It is essential to ascertain exactly at what stage in the coal-handling process the sample is required and, as far as practicable, to design the scheme accordingly. In some instances, however, it may prove impracticable to obtain samples at the preferred points and, in such cases, a more practicable alternative is required.

4.3.2 Division of lots

A lot may be sampled as a whole or as a series of sub-lots, e.g. coal despatched or delivered over a period of time, a ship load, a train load, a wagon load, or coal produced in a certain period, e.g. a shift.

It may be necessary to divide a lot into a number of sub-lots in order to improve the precision of the results.

For lots sampled over long periods, it may be expedient to divide the lot into a series of sub-lots, obtaining a sample for each.

4.3.3 Basis of sampling

Sampling may be carried out on either a time-basis or a mass-basis. In time-basis sampling, the sampling interval is defined in minutes and seconds and the increment mass is proportional to the flow rate at the time of taking the increment. In mass-basis sampling, the sampling interval is defined in tonnes and the mass of increments constituting the sample is uniform.

4.3.4 Precision of results

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

For single lots, the quality variation shall be assumed as the worst case (see 4.4.2 and 4.4.3). The precision of sampling achieved may be measured using the procedure of replicate sampling (see ISO 13909-7).

At the start of regular sampling of unknown coals, the worst-case quality variation shall be assumed, in accordance with 4.4.2, 4.4.3 and 4.4.4. When sampling is in operation, a check may be carried out to confirm that the desired precision has been achieved, using the procedures described in ISO 13909-7.

If any subsequent change in precision is required, the number of sub-lots and of increments shall be changed as determined in 4.4.5 and the precision attained shall be rechecked. The precision shall also be checked if there is any reason to suppose that the variability of the coal being sampled has increased. The number of increments determined in 4.4.5 applies to the precision of the result when the sampling errors are large relative to the testing errors, e.g. moisture. However, in some tests, the testing errors are themselves large. In this case, it may be necessary to form two or more test portions from the sample and use the mean of the determinations to give a better precision.

4.3.5 Bias of results

It is of particular importance in sampling to ensure, as far as possible, that the parameter to be measured is not altered by the sampling and sample preparation process or by subsequent storage prior to testing. This may require, in some circumstances, a limit on the minimum mass of primary increment (see 4.6).

When collecting samples for moisture determination from lots over an extended period, it may be necessary to limit the standing time of samples by dividing the lot into a number of sub-lots (see 4.4.5).

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

4.4 Precision of sampling

4.4.1 Precision and total variance

In all methods of sampling, sample preparation and analysis, errors are incurred and the experimental results obtained from such methods for any given parameter will deviate from the true value of that parameter. While the

absolute deviation of a single result from the "true" value cannot be determined, it is possible to make an estimate of the precision of the experimental results. This is the closeness with which the results of a series of measurements made on the same coal agree among themselves, and the deviation of the mean of the results from an accepted reference value, i.e. the bias of the results (see ISO 13909-8).

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

NOTE The required overall precision for a lot is normally agreed between the parties concerned. In the absence of such agreement, a value of one tenth of the ash content may be assumed.

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

$$P_L = 2\sqrt{\frac{V_I}{n} + \left(1 - \frac{u}{m}\right) V_m + V_{PT}} \quad (1)$$

where

P_L is the estimated overall precision of sampling, sample preparation and testing for the lot at a 95 % confidence level, expressed as percentage absolute;

V_I is the primary increment variance;

n is the number of increments per sub-lot;

u is the number of sub-lots actually sampled;

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

V_m is the sub-lot variance;

V_{PT} is the preparation and testing variance.

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

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

If the quality of a coal of a type not previously sampled is required, then in order to devise a sampling scheme, assumptions have to be made about the variability (see 4.4.2 and 4.4.3). The precision actually achieved for a particular lot by the scheme devised can be measured by the procedures given in ISO 13909-7.

4.4.2 Primary increment variance

The primary increment variance, V_I , depends upon the type and nominal top size of coal, the degree of pretreatment and mixing, the absolute value of the parameter to be determined and the mass of increment taken.

The number of increments required for the general-analysis sample and the moisture sample shall be calculated separately using the relevant values of increment variance and the desired precision. If a common sample is required, the number of increments required for that sample shall be the greater of the numbers calculated for the general-analysis sample and the moisture sample respectively.

NOTE For many coals, the increment variance for ash is higher than that for moisture and hence, for the same precision, the number of increments required for the general-analysis sample will be adequate for the moisture sample and for the common sample.

The value of the primary increment variance, V_I , required for the calculation of the precision using equation (1) can be obtained by either

- a) direct determination on the coal to be sampled using one of the methods described in ISO 13909-7, or
- b) assuming a value determined for a similar coal from a similar coal handling and sampling system.

If neither of these values is available, a value of $V_I = 20$ for ash content can be assumed initially and checked, after the sampling has been carried out, using one of the methods described in ISO 13909-7.

4.4.3 Sub-lot variance

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

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

4.4.4 Preparation and testing variance

The value of the preparation and testing variance, V_{PT} , required for the calculation of the precision using equation (1) can be obtained by either

- a) direct determination on the coal to be sampled using one of the methods described in ISO 13909-7, or
- b) assuming a value determined for a similar coal from a similar sample preparation scheme.

If neither of these values is available, a value of 0,2 for ash content can be assumed initially and checked, after the preparation and testing has been carried out, using one of the methods described in ISO 13909-7.

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

4.4.5.1 General

The number of increments taken from a lot in order to achieve a particular precision is a function of the variability of the quality of the coal in the lot, irrespective of the mass of the lot. The lot may be sampled as a whole, resulting in one sample, or divided into a number of sub-lots resulting in a sample from each. Such division may be necessary in order to achieve the required precision, and the necessary number of sub-lots shall be calculated using the procedure given in 4.4.5.2 or 4.4.5.3, as appropriate.

Another important reason for dividing the lot is to maintain the integrity of the sample, i.e. to avoid bias after taking the increment, particularly in order to minimize loss of moisture due to standing. The need to do this is dependent on factors such as the time taken to collect samples, ambient temperature and humidity conditions, the ease of keeping the sample in sealed containers during collection and the particle size of the coal. It is recommended that, if moisture loss is suspected, a bias test be carried out to compare the quality of a reference sample immediately after extraction with the sample after standing for the normal time. If bias is found, the sample standing time should be reduced by collecting samples more frequently, i.e. increasing the number of sub-lots.

There may be other practical reasons for dividing the lot:

- a) for convenience when sampling over a long period;
- b) to keep sample masses manageable.

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

NOTE The formulae given in 4.4.5.2 and 4.4.5.3 will generally give an overestimate of the required number of increments. This is because they are based on the assumption that the quality of coal has no serial correlation; however, serial correlation is always

present to some degree. In addition, because a certain amount of preparation and testing is required when measuring the increment variance or the sub-lot variance, the preparation and testing errors are included more than once.

The designer of a sampling scheme should cater for the worst case anticipated and will then tend to use higher values for V_l and V_m than may actually occur when the system is in operation. On implementing a new sampling scheme, a check on the actual precision being achieved should be carried out using the methods described in ISO 13909-7. This may be necessary to achieve the required precision, in which case the number of sub-lots is calculated using the procedures given in 4.4.5.2 and 4.4.5.3.

4.4.5.2 Continuous sampling

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

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

$$n = \frac{4V_l}{mP_L^2 - 4V_{PT}} \quad (3)$$

A value of infinity or a negative number indicates that the errors of preparation and testing are such that the required precision cannot be achieved with this number of sub-lots. In such cases, or if n is impracticably large, increase the number of sub-lots by one of the following means.

- Choose a number corresponding to a convenient mass, recalculate n from equation (3) and repeat this process until n is a practicable number.
- Decide on the maximum practicable number of increments per sub-lot, n_1 , and calculate m from the following equation:

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

Adjust m upwards, if necessary, to a convenient number and recalculate n .

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

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

4.4.5.3 Intermittent sampling

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

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

$$n = \frac{4V_l}{uP_L^2 - 4\left(1 - \frac{u}{m}\right)V_m - 4V_{PT}} \quad (5)$$

A value of infinity or a negative number indicates that the errors of preparation and testing are such that the required precision cannot be achieved with this number of sub-lots. In such cases or if n is impracticably large, increase the number of sub-lots to be sampled by one of the following means.

- Choose a larger value for u , the number of sub-lots actually sampled, recalculate n and repeat this process until the value of n is a practicable number.
- Decide on the maximum practicable number of increments per sub-lot, n_1 , and calculate u from the following equation:

$$u = \frac{4m \left(\frac{V_l}{n_1} + V_m + V_{PT} \right)}{mP_L^2 + 4V_m} \quad (6)$$

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

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

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

4.5 Minimum mass of sample

For most parameters, particularly size analysis and those that are particle-size related, the precision of the result is limited by the ability of the sample to represent all the particle sizes in the mass of coal being sampled.

The minimum mass of a sample is dependent on the nominal top size of the coal, the precision required for the parameter concerned and the relationship of that parameter to particle size. Some such relationship applies at all stages of preparation. The attainment of this mass will not, in itself, guarantee the required precision, because precision is also dependent on the number of increments in the sample and their variability (see 4.4.5).

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

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

$$m_S = m_{S,0} \left(\frac{0,2}{P_R} \right)^2 \quad (7)$$

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.

4.6 Mass of primary increment

The mass, m_l , in kilograms, of an increment taken by a mechanical cutter with cutting edges normal to the stream at the discharge of a moving stream can be calculated from equation (8):

$$m_l = \frac{Cb \times 10^{-3}}{3,6v_C} \quad (8)$$

where

C is the flow rate, in tonnes per hour;

b is the cutter aperture width, in millimetres;

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

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

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

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

where

C is the flow rate, in tonnes per hour;

b is the cutter aperture width, in millimetres;

v_B is the belt speed, in metres per second.

The minimum average mass of primary increment to be collected, m'_i , is calculated from equation (10):

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

where

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

n is the minimum number of increments taken from the sub-lot (see 4.4.5).

In most mechanical systems, the mass of primary increment collected [see equations (8) and (9)] will greatly exceed that necessary to make up a sample of the required mass. In some systems, the primary increments are therefore divided, either as taken or after reduction, in order to avoid the mass of the sample becoming excessive.

Providing the design of the cutter complies with the requirements of 6.8 or 6.9, the extraction of an increment from the coal stream will be unbiased whatever the flow rate at the time. Even if flow rates are variable, increments taken at low flow rates, and hence of mass less than the average, will not be subject to extraction bias. Therefore, this part of ISO 13909 does not specify an absolute minimum increment mass.

Under some conditions, e.g. high ambient temperature, increments which are smaller than those corresponding to the design capacity of the system may suffer from disproportionate changes in quality, e.g. loss in moisture, and precautions need to be taken to prevent this. If such losses cannot be prevented and are found to cause relevant bias, then such means as buffer hoppers or a variable-speed cutter (mass-basis sampling) shall be used. Alternatively, increments can themselves be retained temporarily in a buffer hopper until there is sufficient mass to ensure free passage, free from relevant bias, through an on-line preparation system. On no account shall a primary sampler, in a time-basis system or a mass-basis system, be switched off at low flow rates to avoid low mass increments.

When measuring primary increment variance (see ISO 13909-7:2001, clause 6) at preliminary stages in the design of the sampling scheme, use increment masses that are close to those expected to be taken by the system. After implementation of the sampling scheme, the precision of the result can be estimated and adjusted (see ISO 13909-7), by increasing or decreasing the number of increments in the sample, keeping the same increment mass.

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	—
1,0	0,10	—

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.

4.7 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 4.4.1 to 4.4.5). 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 pre-set 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 the following equation:

$$\Delta t \leq \frac{60m_{SL}}{Gn} \quad (11)$$

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 clause 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 equation (10).

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 pre-set 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 equation (12):

$$\Delta m = \frac{m_{SL}}{n} \quad (12)$$

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 4.4.5, 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 B. These criteria may be achieved by either of the following procedures.

- 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.
- Take primary increments using a fixed-speed cutter with subsequent division of the individual increments, if necessary, 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, in seconds or minutes, 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 (see informative annex C). The importance of considering the requirements of the sampling systems from the first stage of main plant design cannot be over stressed. 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 5.2.2), 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 that are free from relevant bias.
- 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 is self-clearing and non-clogging and operates in a manner that will minimize the need for maintenance,
- 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.

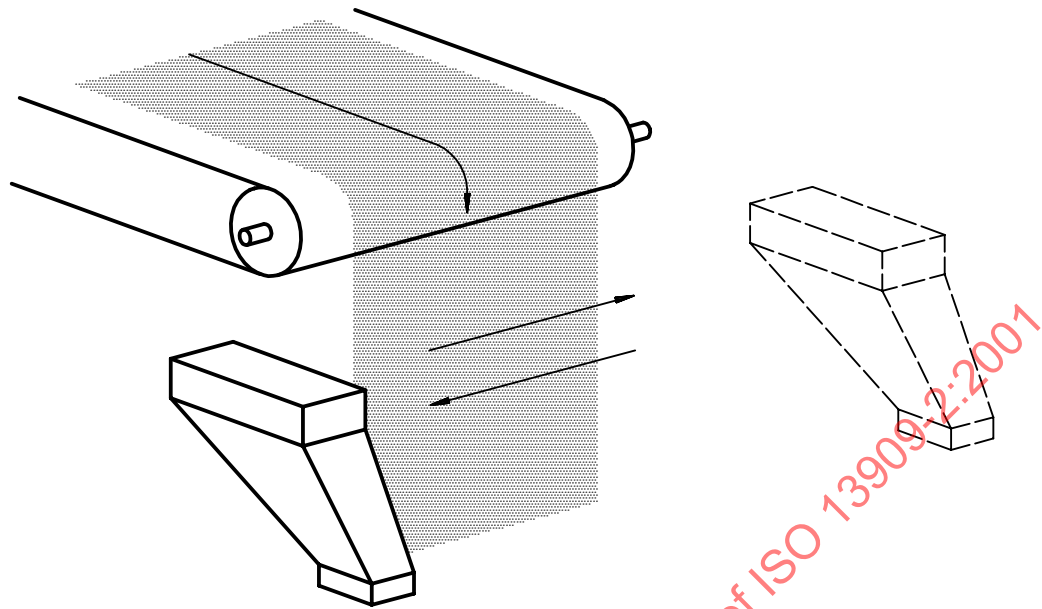
The design objective is to ensure that the mean trajectory of the particles in the stream is as close to normal to the plane of the cutter aperture as possible, to maximize the effective cutter aperture. The cutter velocity is particularly important in this regard, because the particles in the stream 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 aperture.

Examples of different types of falling-stream samplers are shown in Figure 1.

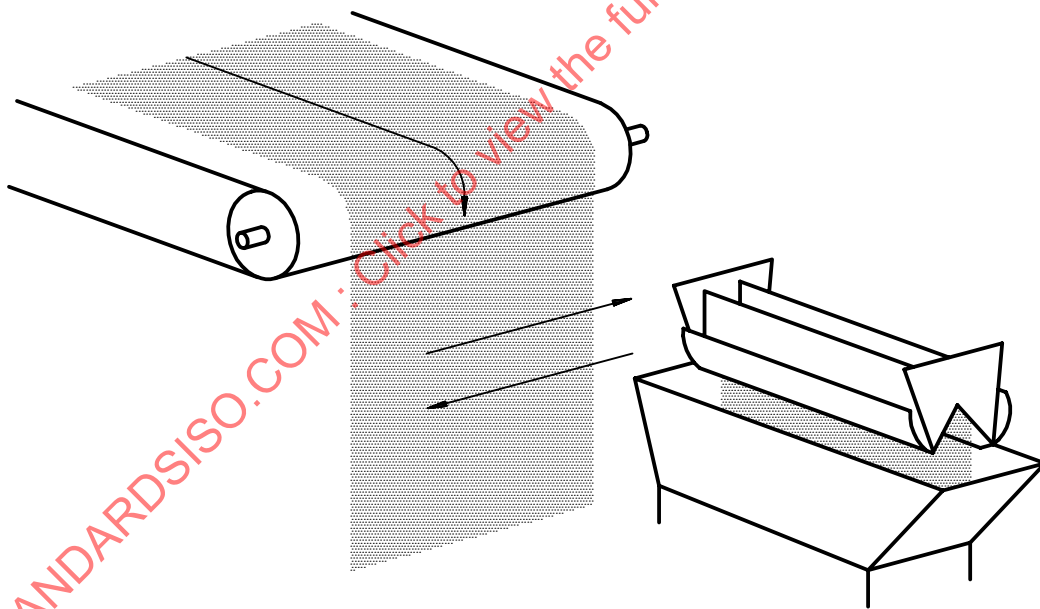
NOTE Other primary sampling devices which conform with the principles laid down in this part of ISO 13909 may be acceptable, providing that they can be shown to have no relevant bias.

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 leading and the trailing cutting edges shall describe either the same plane or the same cylindrical surface. This plane or surface should preferably 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 at any point (see 6.8.2 and 6.9.2).

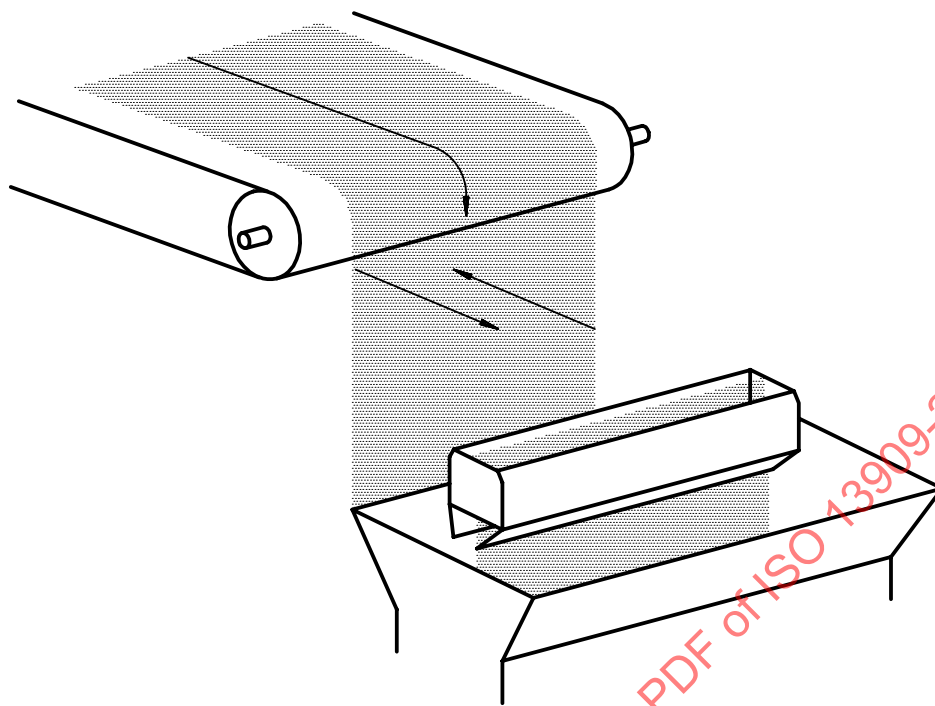


a) Cutter-chute type

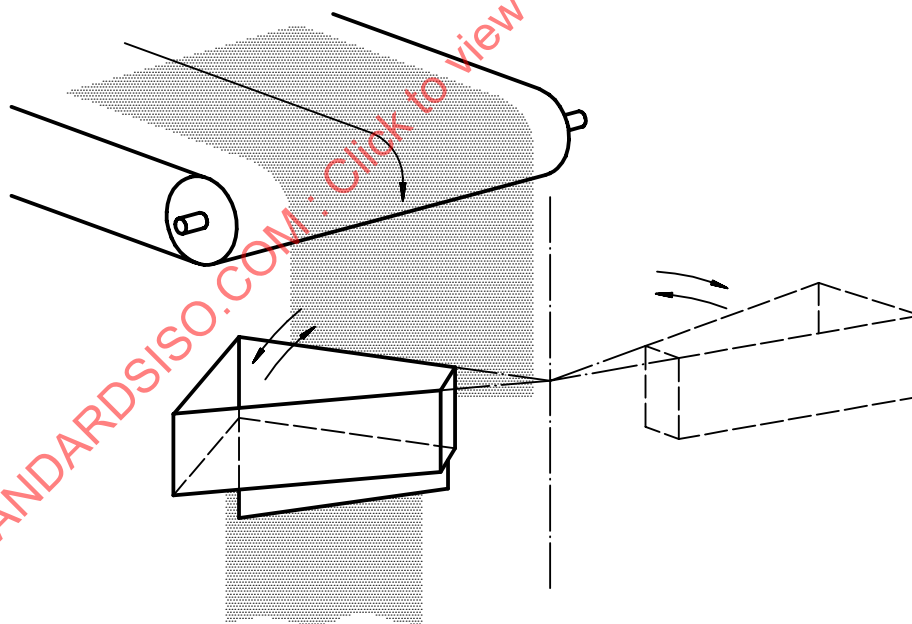


b) Cutter-bucket type 1

Figure 1 — Examples of falling-stream samplers

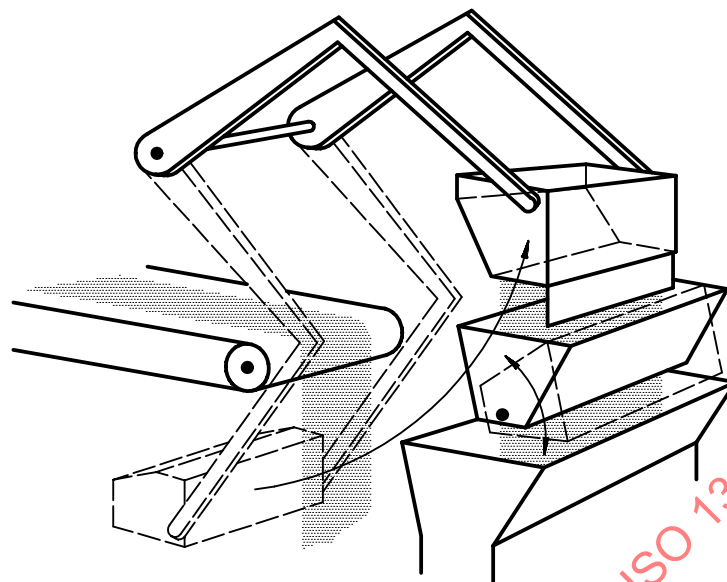


c) Cutter-bucket type 2



d) Swing-arm type 1

Figure 1 — Examples of falling-stream samplers (*continued*)



e) Swing-arm type 2

Figure 1 — Examples of falling-stream samplers (continued)

- 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. For all other cutters see 6.8.2.
- f) The effective capacity of the sampling 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 (Gy^[3]) has shown that, when sampling heterogeneous material streams of low belt loading (low stream density), where the particle-size distribution is very narrow, relevant 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 relevant bias provided that the ratio of cutter aperture to coal top size is a minimum of three.

Irrespective of the cutter speed and aperture, cutters shall be shown to be free from unacceptable bias.

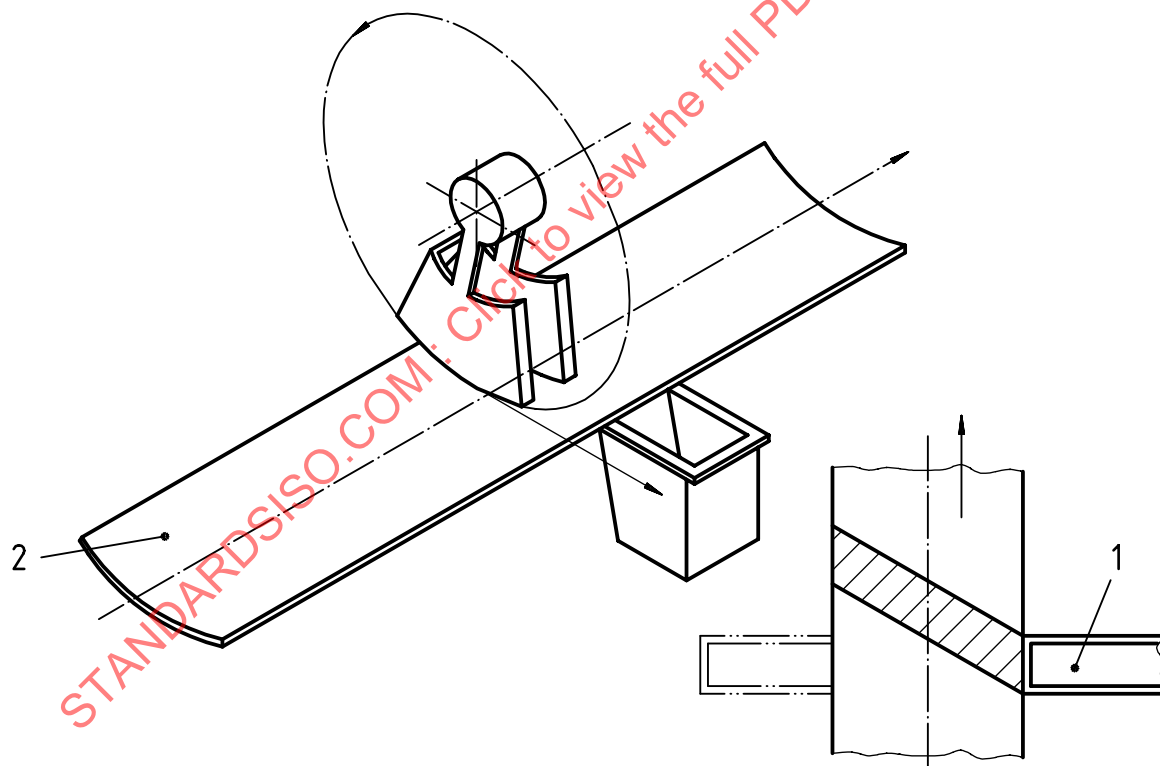
6.9 Cross-belt-type primary samplers

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 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), they are mounted on a trolley or sledge, which during the sampling operation is moved in a direction parallel to the belt and at a velocity equal to the belt velocity. In this way, the influence of belt velocity on the cutter velocity relative to the coal is completely eliminated. However, such ideal sampling conditions are only achieved at the cost of a complicated and somewhat unwieldy sampling device.

For samplers of the type shown in Figure 2 a), the relationship between cutter velocity, belt velocity and cutter velocity relative to the coal is important, since the higher the cutter velocity is in relation to the belt velocity, the larger will be the effective aperture and the more favourable will be the sampling conditions. Furthermore, the higher the cutter velocity, the shorter will be the time during which the cutter, acting as a plough, will hold back the stream of coal.

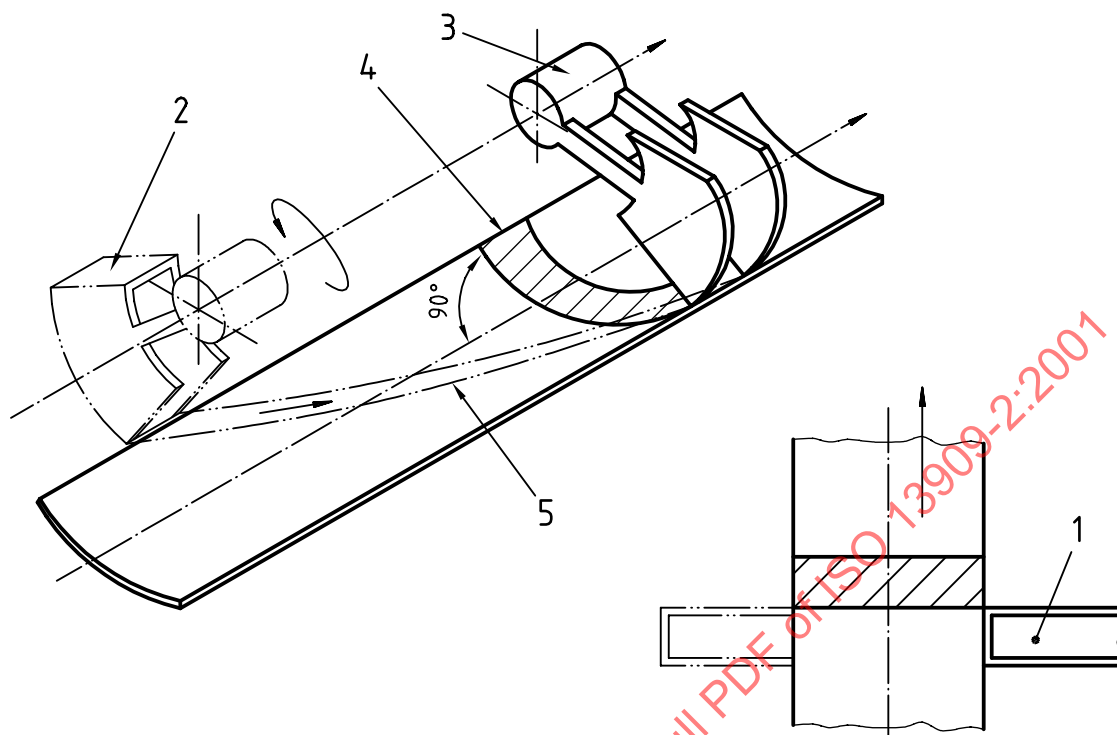


Key

- 1 Cutter
- 2 Belt supported to maintain curvature

a) Normal-sweep (angled cut) type

Figure 2 — Examples of cross-belt samplers

**Key**

- 1 Cutter
- 2 Cutter in park position
- 3 Cutter at end of sampling sweep
- 4 Cross-section of coal collected by cutter (and transferred to receiver)
- 5 Cutter path

b) Angled-sweep (normal cut) type**Figure 2 — Examples of cross-belt samplers**

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. However, the use of high cutter velocities 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.

Irrespective of the cutter speed and aperture, cutters shall be shown to be free from unacceptable bias.

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 shall travel through the stream at a uniform velocity, not deviating by more than 10 % at any point, unless it can be demonstrated that the sampler operates in a bias-free manner.
- d) The cutting aperture of the cutter 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.
- 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.

6.10 Maintenance 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 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.

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 respectively.

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, whereas they need not be impervious to water and vapour.

If physical test samples are required, the sample containers for such samples shall give adequate protection against loss of sample material, but they need not be fully impervious to water and vapour. 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 shall be determined as quickly as possible after sample collection.

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

NOTE 1 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 2 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 clause 8 of ISO 13909-1:2001.

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 relevant 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**

An adequate coal flow rate should be maintained throughout the mechanical sampling system.

9.2 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 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 system (see ISO 13909-8). This is of particular importance when moisture is to be determined on the 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 plants should be rechecked for bias at predetermined intervals as part of a routine maintenance plan, to ensure that they remain free from bias.

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.

Annex A (informative)

Examples of calculations of the number of sub-lots and number of increments per sub-lot (sampling from moving streams)

A.1 Continuous sampling

A.1.1 Example 1

The lot is 80 000 t delivered in 1 000 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 4. Therefore take 4 sub-lots of 20 000 t each.

b) Number of increments per sub-lot

$$n = \frac{4 \times 0,5}{4 \times 0,25^2 - 4 \times 0,05} = 40 \text{ using equation (3)}$$

Therefore take 4 sub-lots of 40 increments each.

A.1.2 Example 2

The lot is 100 000 t delivered as 5 000 t/day over 2 shifts.

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

Primary increment variance, $V_I = 5$

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

$$n = \frac{4 \times 5}{20 \times 0,25^2 - 4 \times 0,2} = 45 \text{ using equation (3)}$$

If this number of increments is considered to be too large, increase the number of sub-lots to 40, i.e. 1 per shift.

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

It would then be sensible to take 12 increments per shift, i.e. 1 every 40 min.

A.1.3 Example 3

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, $V_I = 5$

Preparation and testing variance, $V_{PT} = 0,20$

a) Number of sub-lots

The customer requires a result based on at least 2 samples.

b) Number of increments per sub-lot

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

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 equation (4)

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

This gives a practical sampling method of dividing the lot into 5 sub-lots and taking 50 increments from each.

A.2 Intermittent sampling

A.2.1 Example 1

A lot of 1 000 t is delivered as a train load comprising 50 wagon loads of 20 t each and the required overall precision, P_L , is 0,5 % ash. The wagons are discharged and the coal is converted to a moving stream for sampling.

From previous sampling, the following are known:

$$V_I = 5;$$

$$V_m = 1;$$

$$V_{PT} = 0,10.$$

It has been decided not to sample all the wagons and therefore intermittent sampling is chosen. The maximum number of increments is chosen as 20 per sub lot. The minimum number of wagons to be sampled is calculated from equation (6) as follows:

$$u = \frac{4 \times 50 \left(\frac{5}{20} + 1 + 0,1 \right)}{50 \times 0,5^2 + 4 \times 1} = 16,36$$

Therefore, the coal from 17 of the 50 wagons is sampled from the moving stream.

A.2.2 Example 2

If the required overall precision in the example given in A.2.1 was 0,2 % ash, instead of 0,5 % ash, the calculation of u would be as follows: