BS ISO 13909-5:2016

BSI Standards Publication

Hard coal and coke — Mechanical sampling

Part 5: Coke — Sampling from moving streams

National foreword

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Second edition
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Hard coal and coke — Mechanical sampling —

Part 5: Coke — Sampling from moving streams

Houille et coke — Échantillonnage mécanique —

Partie 5: Coke — Échantillonnage en continu

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Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

This second edition cancels and replaces the first edition (ISO 13909-5:2001), 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*

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INTERNATIONAL STANDARD ISO 13909-5:2016(E)

Hard coal and coke — Mechanical sampling —

Part 5:
Coke — Sampling from moving streams

1 Scope

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

The diversity of types of equipment for sampling and the conditions under which mechanical sampling is performed make it inappropriate to specify standard designs for samplers which will be applicable to all situations.

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 13909-6, *Hard coal and coke — Mechanical sampling — Part 6: Coke — 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 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 the precision required;

d) determine the method of combining the increments into a sample, or number of sub-lot samples, and the method of sample preparation (see ISO 13909-6);

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e) determine or assume the variability of the coke (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) define the sampling interval (see Clause 5);

h) ascertain the nominal top size of the coke 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 cokes 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 coke-handling process the sample is required and, as far as practicable, design the scheme accordingly.

4.2.2 Parameters to be determined on samples

The samples for moisture and physical tests may be collected separately or as one sample, which is then divided. In this part of ISO 13909, a sample which is collected for the determination of moisture (and possibly also for general analysis) is referred to as the moisture sample; a sample which is collected for physical tests only is referred to as the physical sample. If a sample is used for the determination of moisture and for physical tests, it is referred to as a common sample.

In mechanical sampling of coke, the only sample which can, in certain circumstances (see 4.2.6), be processed automatically beyond the divided-increment stage is the moisture sample.

In order to achieve the desired precision, it may be necessary to take different numbers of increments for the moisture and physical samples. Where a common sample is taken, the greater number of increments shall be used.

4.2.3 Division of lots

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

4.2.4 Basis of sampling

Sampling may be carried out on either a time-basis or a mass-basis.

In time-basis sampling, increments are taken at fixed time intervals with an increment mass, collected with a fixed speed cutter, which is proportional to the flow rate at the time of extraction.

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In mass-basis sampling, increments are taken at fixed mass intervals, using a belt weigher/mass integrator, and fixed mass increments are extracted using a variable speed cutter or sample preparation system which produces a fixed mass divided increment.

The conditions under which mass-basis sampling may seem to offer the advantage of consistent increment mass, for example highly variable flow rates, are those in which it is most difficult to implement in practice.

Time-basis sampling is by far the simplest to implement and is the basis of this part of ISO 13909.

4.2.5 Precision of sampling

The required precision for a lot for each parameter to be measured shall be decided. The number of sub-lots and minimum number of increments per sub-lot collected shall then be determined as described in 4.3.4, and the average mass of 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 cokes, the worst-case quality variation shall be assumed. When sampling is in operation, a check shall be carried out to confirm that the desired precision has been achieved using the procedure of duplicate sampling as described in ISO 13909-7.

If any subsequent change in precision is required, the number of sub-lots and 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 coke 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. moisture content. However, in some tests, e.g. Micum Index, the testing errors are themselves large. In this case, it may be necessary to prepare two or more test portions from the same sample (see 4.3.4.3) and use the mean of the determinations to give a better precision.

4.2.6 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. For example, care shall be taken to avoid breakage of coke intended for physical testing and loss of moisture from the moisture sample during storage. This may require, in some circumstances, a limit on the minimum mass of primary increment (see 4.5 and Clause 8).

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).

The use of on-line crushing and division of the moisture sample for moisture determination should be treated with caution because of the risk of bias caused by loss of moisture in the processing (see 6.2.2). In particular, the crushing of hot coke is not recommended. If the bias is unacceptable, the sample shall be left in the uncrushed state and the sample preparation carried out by manual methods. It should be accepted, however, that some bias is inevitable, whether due to breakage or loss of moisture from hot coke. The object, therefore, shall be to restrict such degradation or moisture loss to a minimum.

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

4.3 Precision of results

4.3.1 Precision and total variance

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

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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 coke 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.

The theory of the estimation of precision is given in ISO 13909-7. Formula (1) is derived:

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

where

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

V_I is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of increments to be taken from a sub-lot;

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

If the quality of a coke of a type not previously sampled is required, then, in order to devise a sampling scheme, assumptions should 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.

If the same type of coke is sampled regularly, sampling schemes can be laid down using data derived from previous sampling. The procedures given in ISO 13909-7 can be used to devise the optimum scheme, thus keeping the sampling costs to a minimum.

4.3.2 Primary increment variance

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

The variability for moisture is usually higher than that for ash and hence, for the same precision, the number of increments for moisture will be adequate for ash. If, however, a higher precision is required for ash, the relevant primary increment variance shall be applied for each 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 coke to be sampled using one of the methods described in ISO 13909-7, or

b) assuming a value determined for a similar coke from a similar coke handling and sampling system.

If neither of these values is available, a value of 5 can be assumed initially and checked, after the sampling has been carried out, using one of the methods described in ISO 13909-7.

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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 coke to be sampled using one of the methods described in ISO 13909-7, or

b) assuming a value determined for a similar coke from a similar sample-preparation scheme.

If neither of these values is available, a value of 0,2 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 in each 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 coke 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.

There may be other practical reasons for dividing the lot, such as:

a) for convenience when sampling over a long period;

b) to keep sample masses manageable;

c) 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 coke. It is recommended that, if moisture loss is suspected, a bias test is 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.

The quality of the lot shall be calculated as the weighted average of the values found for the sub-lots.

As stated in 4.3.1, the precision is determined by the variability of the coke, the number of increments and sub-lots and the preparation and testing variance. By transposing Formula (1), it can be shown that the number of increments per sub-lot for a desired precision for a lot can be estimated from Formula (2):

$$n = \frac{4V_I}{mP_L^2 - 4V_{PT}} \tag{2}$$

Determine the number of sub-lots required for practical reasons and then estimate the number of increments in each for the desired precision using Formula (2). If n is a practicable number, the initial scheme is established. However, if n is less than 10, take 10 increments per sub-lot.

If n is impracticably large, increase the number of sub-lots using one of the following methods:

a) increase m to a number corresponding to a convenient mass or time, recalculate n 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):

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$$m = \frac{4V_I + 4n_1 V_{PT}}{n_1 P_L^2} \quad (3)$$

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

NOTE This method of calculating the number of increments required per sub-lot for a certain precision from the primary increment variance and the preparation and testing variance will generally give an overestimate of the required number. This is because it is based on the assumption that the quality of coke varies in a random manner. 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.

The designer of a sampling scheme should cater for the worst case anticipated and may then use higher values for V_I than may actually occur when the scheme is in operation. When the sampler is commissioned, 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 so that the required precision can be achieved at minimum cost.

Example 1

The lot is 35 000 t of 40 × 20 mm coke delivered in one day. The primary increment variance and preparation and testing variance for moisture content have been determined as follows.

Primary increment variance for moisture content, $V_I = 5$.

Preparation and testing variance for moisture content, $V_{PT} = 0{,}10$.

The required precision, $P_L = 1{,}0$ % moisture content.

a) Initial number of sub-lots

For convenience and to avoid the sample standing for too long, take three shift samples, (i.e. $m = 3$).

b) Number of increments per sub-lot

$$n = \frac{4 \times 5}{3 \times 1^2 - 4 \times 0{,}10} = 7{,}7 \text{ using Formula (2)}$$

Therefore, split the lot into three sub-lots and take 10 increments from each.

Example 2

The lot is 100 000 t of 100 × 25 mm coke delivered as 5 000 t/day over two 8-hour shifts.

The primary increment variance, V_I, for moisture content is unknown, so initially assume a value of 5.

Required precision $P_L = 0{,}25$ % moisture content.

Preparation and testing variance for moisture content, V_{PT}, from experience assume a value of 0,20.

a) Initial number of sub-lots

For the preliminary calculation, assume a daily sample is constituted, i.e. $m = 20$, in order to avoid the risk of moisture loss by overnight storage of sample increments.

b) Number of increments per sub-lot

$$n = \frac{4 \times 5}{20 \times 0{,}25^2 - 4 \times 0{,}2} = 44{,}4 \text{ using Formula (2)}$$

This number will result in too large a mass to crush as a single moisture sample for each sub-lot [almost 2 tonnes for a typical increment mass of about 45 kg using Formula (4)]. Therefore, increase the number of sub-lots to 40, i.e. one per shift.

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$$n = \frac{4 \times 5}{40 \times 0,25^2 - 4 \times 0,2} = 11,7 \approx 12$$

Hence, take 12 increments per shift, i.e. one every 40 min, resulting in a moisture sample of about 540 kg for each sub-lot. However, if the equipment available for crushing the sample is not robust enough for this sample mass, further increase the number of sub-lots.

Example 3

The lot is 9 000 *t* of 40 × 25 mm coke.

The primary increment variance for moisture content $V_I = 5$.

The preparation and testing variance for moisture content, V_{PT}, from experience is assumed to be 0,20.

The desired precision $P_L = 0,5$ % moisture content.

a) Number of sub-lots

For convenience, split the lot into 2 sub-lots, i.e. $m = 2$.

b) Number of increments per lot

$$n = \frac{4 \times 5}{2 \times (0,5)^2 - 4 \times 0,2} = \frac{20}{-0,3} \approx -66.7$$

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 40 increments is the maximum practicable number in a sub-lot and from Formula (3).

$$m = \frac{4 \times 5 + 4 \times 40 \times 0,2}{40 \times 0,5^2} = 5,2 \approx 6$$

This gives a practical sampling method of dividing the lot into six sub-lots of 1 500 tonnes each, taking 40 increments from each.

4.3.4.2 Moisture sample

The sampling variance for moisture content may vary in the range 0,2 to 5 depending on the absolute value of the moisture content, the size range of the coke and the extent of cutting, screening and mixing it has undergone prior to sampling. For example, a closely graded, highly cut small-sized industrial coke sampled on delivery to the customer would have a much lower variance than an uncut coke sampled at the wharf or a very large coke on despatch from the producer's works. It may be known from experience what level of variance is to be expected.

It is recommended that the number of increments initially required be sufficient to give a mass of sample greater than the mass given in Table 1, subject to a minimum of 10 increments.

The variance for ash and other chemical properties is usually less than for moisture content. However, it is often desired to obtain a higher precision for the ash result and hence the number of increments should be calculated for each and the greater number taken for the moisture sample.

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Table 1 — Minimum mass of sample

Nominal top size m	Minimum mass kg
> 125	2 000
125	1 000
90	500
63	250
45	125
31,5	60
22,4	30
16,0	15
11,2	8
10,0	6
8,0	4
5,6	2
4	1

4.3.4.3 Physical sample

The cokes to be sampled within the scope of this part of ISO 13909 will exhibit large differences in physical strength, size, size range, and size distribution. In addition, many different parameters, e.g. Micum test, porosity, percentage retained on a particular sieve, mean size, etc., can be determined on the samples. Sample preparation errors may be zero when the test is done on the whole sample or large when division of the sample takes place.

Furthermore, it is usually not possible to determine the individual increment variances for tests such as the Micum test, because the increment mass is too small.

It will be found with many physical tests that the only way to achieve the required precision will be either

a) to divide the lot into sub-lots, or

b) to prepare two or more test portions from the sample, taking the mean of the test results for the sample.

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

4.4 Minimum mass of sample

For most parameters, particularly size grading 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 coke being sampled.

The minimum mass of sample is dependent on the nominal top size of the coke, 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. This is also dependent on the number of increments in the sample and their variability (see 4.3.4).

The masses specified in Table 1 are for guidance on the minimum mass for unknown or heterogeneous cokes. While they can usually be reduced for the moisture sample, they may be inadequate for the determination of, for example, oversize to 1 % precision of sampling and division, particularly on very large cokes.

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When a coke 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 adjusted accordingly. However, the masses shall not be reduced below the minimum requirements laid down in the relevant analysis standards.

Account shall also be taken of the uses to which the sample is to be put and the numbers, masses and size distribution of the test samples required.

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 (4):

$$m_I = \frac{Cb}{3,6v_c} \times 10^{-3} \tag{4}$$

where

C is the flow rate, in tonnes per hour;

b is the cutter aperture width, in millimetres;

v_C is the cutter speed, in metres per second.

For a cross-belt sampler, the mass, m_I, in kilograms, of increment can be calculated from Formula (5):

$$m_I = \frac{Cb}{3,6v_B} \times 10^{-3} \tag{5}$$

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 (6):

$$m_I^{'} = \frac{m_S}{n} \tag{6}$$

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).

In most mechanical systems, the mass of primary increment collected [see Formulae (4) and (5)] 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 6.5 or 6.6, the extraction of an increment from the coke 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.

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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 bias, such means as buffer hoppers shall be used. Alternatively, increments can themselves be retained temporarily in a buffer hopper until there is sufficient mass to ensure passage, without introducing bias into the system, through an on-line preparation system. On no account shall a primary sampler be switched off at low flow rates to avoid low mass increments.

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, based on similar coke from similar sampling systems. 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 coke products to be sampled will exhibit differences in size and amount of fines. 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.

5 Methods of sampling

5.1 General

Sampling shall be carried out by systematic sampling on a time-basis. The procedures of sample preparation vary in accordance with the type of sampling employed (see ISO 13909-6).

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.

It is essential that the increment does not completely fill or overflow the sampling device. With mechanized sampling devices, the increment mass may be considerably larger than that necessary to produce the calculated minimum sample mass. Hence, a system of primary increment division (see ISO 13909-6) 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 primary samplers. Such periodicity may arise from the cycle of operations or feeder systems in use. If necessary modify the sampling interval, or employ stratified random sampling, to remove the possibility of bias.

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5.2 Time-basis sampling

5.2.1 Method of taking primary increments

In order that the increment mass is proportional to the coke flow rate in mechanical sampling, the speed of the cutter shall be constant throughout the sampling of the entire sub-lot (see 6.5.1).

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 Formula (7):

$$\Delta t \leq \frac{60\, m_{SL}}{G\, n} \tag{7}$$

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).

The designer of mechanical systems should ensure that, for all cokes using the system, the time taken for the sampling or processing of an increment shall be less than the time between increments under maximum flow rate.

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 coke stream shall be not less than the minimum average increment mass calculated from Formula (6).

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

5.3 Stratified random sampling

5.3.1 General

Cyclical variations in coke quality may occur during systematic sampling. Every effort shall be made to eliminate coincidence of the cycle with the taking of increments in systematic sampling. 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 interval, the actual taking of the increment is displaced by a random amount of time, 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 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.

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5.3.2 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 coke (see 5.2.3).

5.4 Reference sampling

Reference samples shall be taken by the stopped-belt method to enable checking for bias (see ISO 13909-8).

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 Sampling System

6.2.1 General

It is important that the coke-handling plant be designed and engineered to provide adequate space and satisfactory sampling and operating conditions for the sampling system. Ideally the mechanical sampler should be designed at the same time as the coke-handling plant. If the sampler is added to an existing plant, it is essential that engineering expediency is not allowed to cause any condition which would make the sampler biased and 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 design of the mechanical sampler shall be related to the types of coke to be handled, the quality characteristics to be determined and the maximum number, mass and frequency of increments anticipated as discussed in Clause 4.

The procedure adopted shall be such that bias is kept to a minimum during the taking of the increments.

The methods of taking primary increments are given in Clause 5. The mass of the increment obtained from the mechanical sampler, calculated from Formulae (4) or (5) shall be compared with the minimum average increment mass [see Formula (6)]. If the mass is much greater than this, then a division stage (see ISO 13909-6) may be included to reduce the mass of the sample to a more manageable amount.

From the number of increments required from a sub-lot, as determined from Clause 4, and the length of time taken to handle the sub-lot, a calculation can be made of the sampling interval. The scheme shall then be designed so that sample processing can be carried out in less than the smallest anticipated sampling interval.

From the mass of increment and the number of increments given in the sampling scheme, the total mass of coke that will be collected can be calculated. This should be used to design the sample containers and sample-handling system.

If it is decided, having considered the risk of bias (see 4.2.6), to use on-line sample preparation for the moisture sample (see ISO 13909-6), then a crusher and sample divider shall be added to the sampler. The mass of each crushed and divided increment shall be greater than 1 kg at the average flow rate.

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6.2.2 Checking for precision and bias

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 coke 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.

The precision of the 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 two samples to produce duplicate samples. It is not permitted to prepare duplicate samples from a number of increments already compounded.

6.2.3 Operation of sampler

A fully automatic mechanical sampler shall be capable of operating unattended and all adjustable controls shall be accessible to authorized persons only. The system shall be readily accessible throughout to facilitate inspection, thorough cleaning, repairs or check experiments, e.g. tests for bias.

It is recommended, for maintaining good control, that a counter showing the number of operating cycles of the primary sampler and a remote indication that the sampler is running, stopped or shut down under fault conditions, should be provided.

When modifications are made or change is suspected, the sampler shall be observed in operation, corrections made if required and a check experiment carried out as necessary (6.2.2).

6.3 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, consideration should 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.

6.4 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 unbiased increments.

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 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 coke being sampled,

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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 coke (for example, due to excessive air flow through the equipment) are minimized.

6.5 Design of falling-stream-type samplers

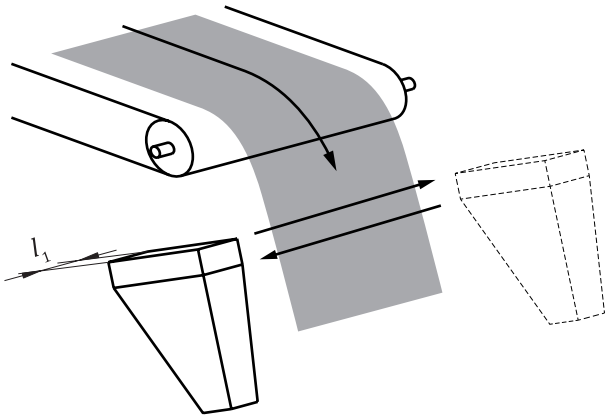
6.5.1 General

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

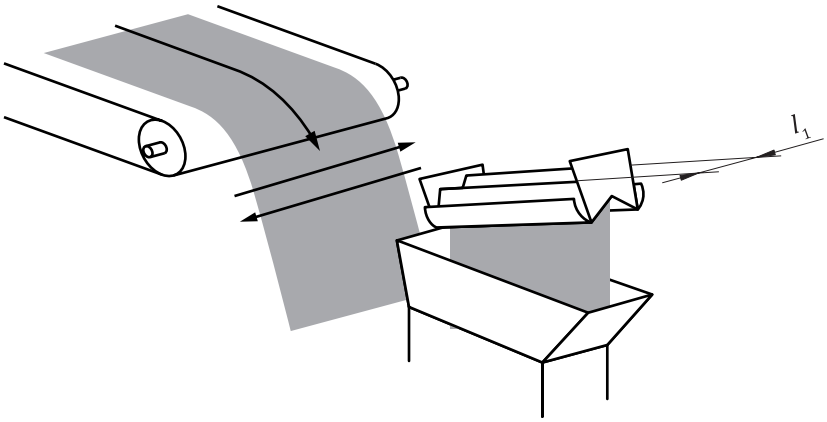
The design objective is to ensure that the mean trajectory of the particles in the stream is as close to normal to the plane of the cutter aperture as possible to maximize the effective cutter aperture. The cutter velocity is particularly important in this regard, because the particles in the stream 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 to the principles laid down in this part of ISO 13909 may be acceptable, providing that they are capable of collecting unbiased increments.



a) Cutter-chute type



b) Cutter bucket type (i)

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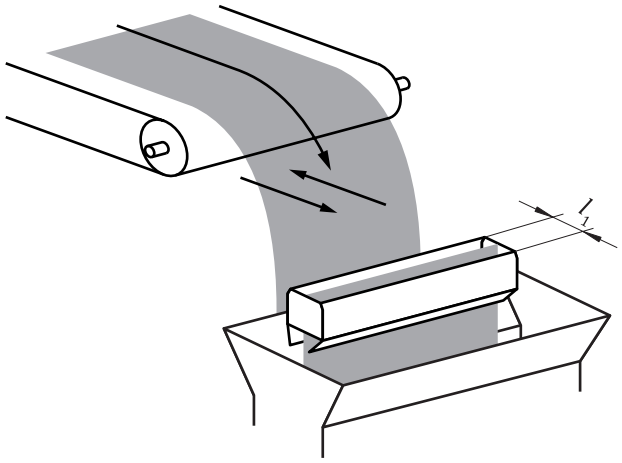
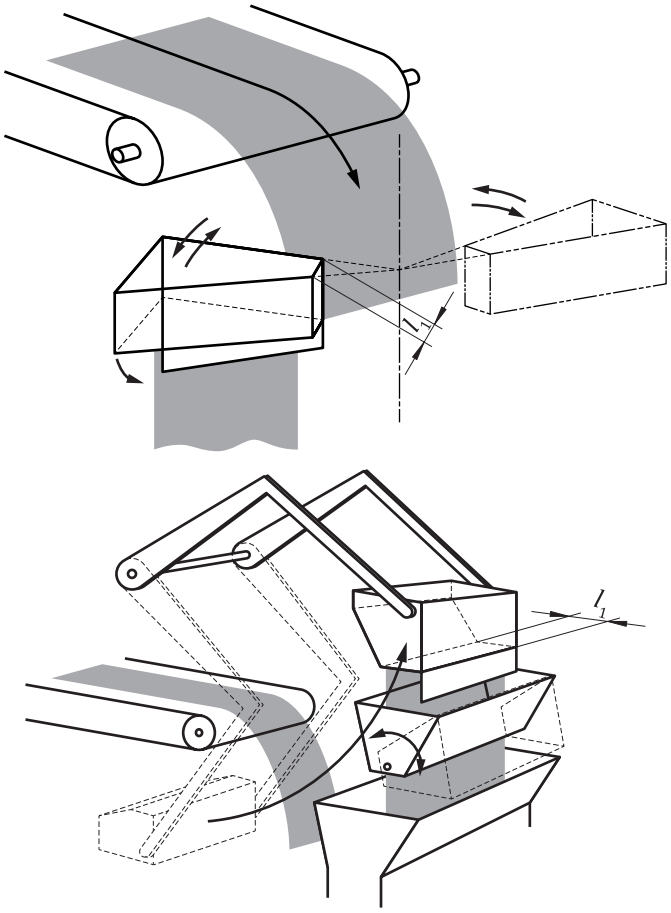


c) Cutter bucket type (ii)



d) Swing arm types

Figure 1 — Examples of falling-stream samplers

A cutter intended for sampling from a falling stream of coke 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.

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c) The cutter shall travel through the coke stream at a uniform velocity; i.e. the velocity shall not deviate by more than ± 5 % from the preselected reference velocity at any point.

d) The design of the cutter aperture shall be such that all parts in 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 coke 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 narrow end.

f) The effective capacity of the sampling cutter shall be determined on the basis of the expected maximum flow rate of the coke 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.

6.5.2 Cutter velocity

Primary cutters are used to sample streams of large capacity where there is a relatively high flow density and wide size distribution.

For falling-stream cutters, experimental work on ores[1] has shown that, when sampling a heterogeneous material stream 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 the cutter width to the 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 coke-handling systems have cutters which sample coke 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 unbiased provided that the ratio of the cutter aperture to coke nominal top size is a minimum of three.

Irrespective of the cutter speed and aperture, cutters shall be shown to be capable of collecting unbiased increments.

6.6 Cross-belt-type primary samplers

6.6.1 Operation

The principle of operation of a cross-belt sampler is shown in Figure 2. The sample cutter pivots on an axis parallel to the centre-line of the belt, and 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.

For cross-belt samplers, the relationship between cutter velocity, belt velocity and cutter velocity relative to the coke 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 coke.

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. On the other hand, the use of high cutter velocities may result in an unacceptable degree of breakage of the coke. In such circumstances, it may be better to use the cross-belt sampler 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 capable of collecting unbiased increments.

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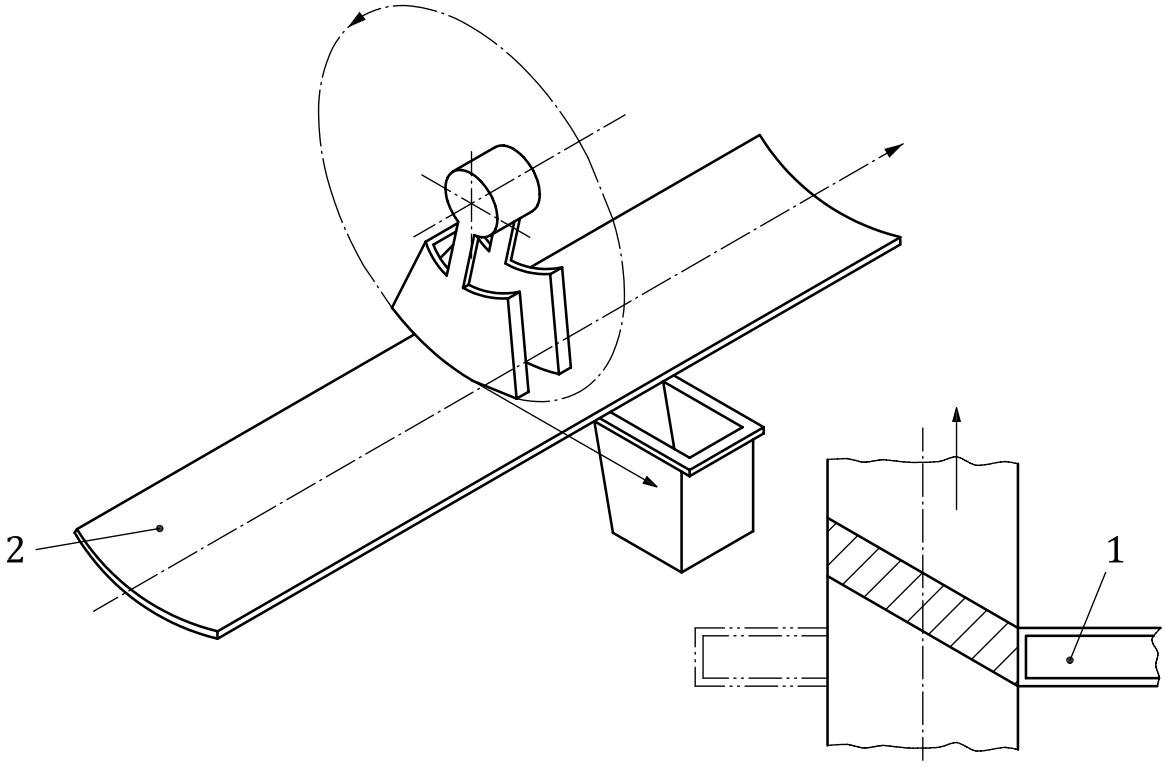

Key

1 cutter

2 belt supported to maintain curvature

Figure 2 — Example of a cross-belt sampler

6.6.2 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.

d) The cutting aperture of the cutter shall be at least three times the nominal top size of the coke 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 coke 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 the 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 coke.

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 coke cross-section in the path of the cutter is collected from the belt.

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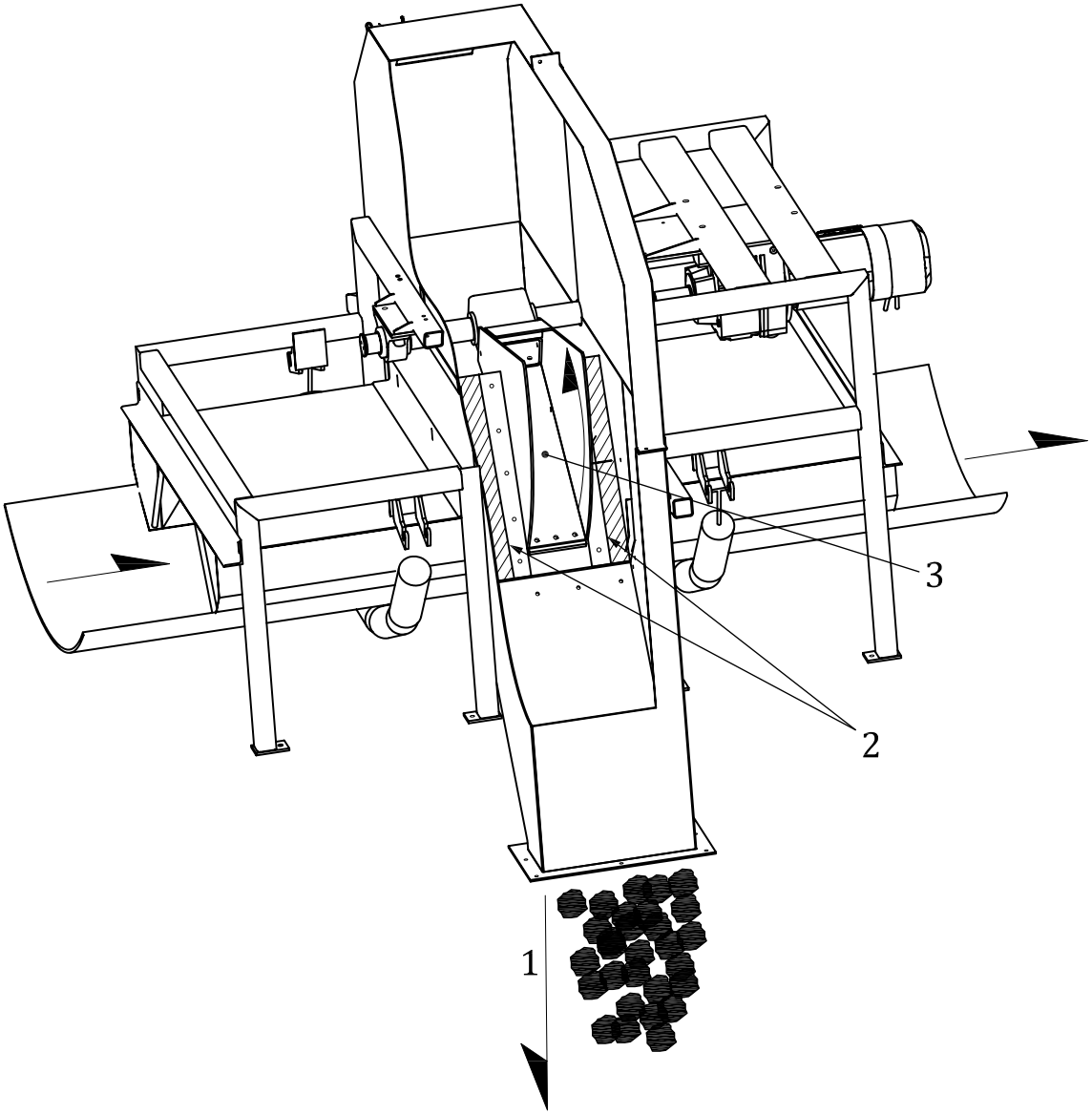
h) Reinforced skirting shall be fitted to the sample chute receiving cross-belt increments to prevent material from outside the sample cutter from falling into the sample collection chute and contaminating the increment as shown in Figure 3.



Key

1 sample
2 throat skirting
3 sample cutter

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

6.6.3 Maintenance of sampling equipment

Sampling systems shall be designed to allow safe inspection of the main components in accordance with ISO 21398. 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.

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All mechanical systems are subject to wear. Such wear may eventually cause a sampler, 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 check list of items to be noted. This check list shall include at least the following items:

a) observation of the taking of an increment;

b) excessive spillage in the area of the mechanical samplers which may cause contamination of the sample;

c) any mechanical changes made to the mechanical sampler or to the coke-handling system immediately upstream of the sampler;

d) for cross-belt samplers, the wear on the brushes and/or resilient skirts of the cutter;

e) for cross-stream samplers, cutter speeds should be checked.

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 completed.

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

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

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

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.

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 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.

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8 Sample preparation

Sample preparation shall comply with the requirements of ISO 13909-6.

9 Minimization of bias

9.1 General

The results obtained from samples taken mechanically may be biased for many reasons, but these may be grouped as follows:

a) the primary increment is not representative of the coke in the stream (similar causes of bias arise during sample division, which is a special case of sampling);

b) changes are brought about to the sample during its handling and storage.

Testing for bias is described in ISO 13909-8.

9.2 Spacing of increments

If there is a relationship between the sampling interval and cyclical plant operations which cannot be avoided, stratified random sampling shall be used (see 5.4)

In order to avoid the bias caused by the non-random sampling of front runnings or tailings, both for primary increment samplers and sample dividers, the starting time of the first increment or cut shall be independent of the starting time of the flow of coke.

9.3 Incorrectly extracted increments

This derives from incorrectly designed and installed cutters and inadequate maintenance. The requirements applying to their design (see 6.5 and 6.6) shall therefore be followed meticulously.

9.4 Preservation of integrity of sample

9.4.1 General

Special problems arise with coke samples for the following reasons.

a) Coke is a friable substance and virtually any handling, including mechanical sampling, produces size degradation. This degradation may have a significant effect, not only on the results of coke size measurements, but also on physical tests such as the Micum test. As such, the location of sampling, and the number of drops and drop distances, prior to sampling, should be noted on the sampling report.

b) Coke is often despatched hot from the producer's works and, therefore, continuously loses moisture. Samples shall be taken before despatch and, under such circumstances, the prevention of further moisture loss from the sample, whether crushed or uncrushed, is practically impossible to achieve.

c) Contaminants such as phosphorus have metallurgical significance even in very low concentrations. Contamination shall therefore be prevented.

The bulk of the material itself will almost inevitably undergo changes, both in size and moisture, before use. Such changes may well be the subject of sampling/testing exercises. Therefore, the existence of avoidable bias at any given sampling position should not be tolerated, despite the possibility that the bulk of the coke will itself undergo subsequent change. Every practicable step shall be taken to ensure that the sample retains the quality that it had at the time it was taken.

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9.4.2 Precautions to reduce bias

The following precautions shall be taken.

a) For physical test samples, in order to minimize breakage, the design shall be such as to require the minimum practicable drops for the coke, to use shock-absorbent material to line chutes/containers and to break falls.

b) For moisture samples, sample containers shall be constructed of impermeable non-absorbent materials and sealed as far as practicable to prevent loss of moisture by evaporation. The sample shall be stored for the minimum time before the moisture is determined and, in any case, not more than 24 h. Where coke is sampled hot, individual increments should not be crushed. The sample shall be stored in a sealed container to cool.

c) Sample containers and chutes shall be designed in such a way that material other than the increments cannot enter them. Thus coke spillage, airborne dust, water sprays, etc. shall not affect the quality of the sample.

d) The materials used for construction of components shall be such that no significant contamination of the sample can be caused by abrasion.

As far as practicable, the system shall be self-cleaning to avoid the risk of contamination by preceding samples. This is particularly important where single lots are to be sampled or changes in the type of coke from one consignment to the next are envisaged. If complete self-cleaning cannot be achieved, an increment of coke of the same type as to be sampled shall be passed through the entire system and discarded in order to remove any contamination.

10 Verification

Proper design shall be verified prior to installation and use (see 6.5 and 6.6). After installation, proper design shall be verified by conducting a bias test of the sampling system in accordance with ISO 13909-8.

Sampling systems should be rechecked for bias and cutter wear 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.

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