INTERNATIONAL STANDARD

ISO 18283

Second edition
2022-03

Coal and coke — Manual sampling

Houille et coke — Échantillonnage manuel

Reference number
ISO 18283:2022(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword

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

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

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

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

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 18283:2006), which has been technically revised. It also incorporates the Technical Corrigendum ISO 18283:2006/Cor.1:2006.

The main changes are as follows:

— Removal of any reference to intermittent sampling. Only continuous sampling is permitted.

— Discussion of the need to eliminate bias prior to discussing precision.

— Deletion of the separate tables on calculated numbers of increments.

— Deletion of the table on reference increment mass.

— Separation of tables for minimum sample masses for coal and coke.

— Removal of the table for reduced minimum sample mass for large sizes of coal and coke.

— Inclusion of manual sampling from a moving conveyor, provided a risk assessment is conducted at the outset and that this type of sampling is only permitted on a slow-moving belt or at low flow rates. Furthermore, at higher flow rates, mechanical assistance is necessary to ensure that primary increments can be collected safely.

— Restriction of the type of probes that can be used.

— Deletion of augers for manual sampling.

— Inclusion of a photograph of a gated riffle.

— Exclusion of sampling of large fuels in excess of the nominal top sizes in Tables 1, 2 and 4, because it is not practical.

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

Introduction

Mechanical sampling from moving streams is the preferred method for sampling coal and coke. However, often mechanical facilities are not available. Moreover, for sized coal or coke, mechanical sampling may be a problem because of (size) degradation by the sampling system.

The fundamental requirements of sampling are that all particles of the coal or coke in the lot are accessible to the sampling instrument and thus have a non-zero chance of being selected, and that each individual particle of equal mass has an equal probability of being selected and included in the sample.

When sampling manually, conditions are often far from ideal. The methods described in this document are intended to obtain the most representative sample that can be safely achieved. Manual sampling should only be applied if no possibility for mechanical sampling exists.

The purpose of taking and preparing a sample of coal or coke is to provide a test sample that, when analysed, provides test results representative of the lot or sub-lot sampled.

The first stage of sampling, known as primary sampling, is the taking from positions distributed over the entire lot of an adequate number of coal or coke portions known as primary increments. The primary increments are then combined into a sample. From this sample, the required number and types of test samples are prepared by a series of processes jointly known as sample preparation.

In devising a sampling procedure, it is also essential to guard against bias in the taking of increments. Bias can arise from:

a) incorrect location/timing of increments,

b) incorrect delimitation and extraction of increments,

c) particle size segregation at the point of sampling,

d) loss of integrity of increments after extraction.

Methods for measuring bias are described in ISO 13909-8.

Coal and coke — Manual sampling

WARNING — This document can involve hazardous materials, operations and equipment, and does not purport to address all the safety issues associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices.

1 Scope

This document provides the basic terms used in manual sampling of coal and coke and describes the general principles of sampling. It provides procedures and requirements for establishing a manual sampling scheme, methods of manual sampling, sampling equipment, handling and storage of samples, sample preparation and a sampling report, and applies to manual sampling during the transfer of coal or coke. Guidelines for manual sampling in stationary situations are given in Annex B, but this method of sampling does not provide a representative test sample and the sampling report shall state this.

This document covers sampling of brown coals and lignites, but does not include sampling from coal seams, for which guidance is given in ISO 14180. Mechanical sampling of coal and coke is covered in ISO 13909.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 579, *Coke — Determination of total moisture*

ISO 589, *Hard coal — Determination of total moisture*

ISO 687, *Solid mineral fuels — Coke — Determination of moisture in the general analysis test sample*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
air-drying
process of bringing the moisture content of the sample near to equilibrium with the atmosphere in the area in which further reduction and division of the sample are to take place

Note 1 to entry: Air-drying to equilibrium with the atmosphere applies to coal. Drying of coke is generally to facilitate sample preparation.

3.2
bias
systematic error that leads to the average value of a series of results being persistently higher or persistently lower than those that are obtained using a reference sampling method

3.3
common sample
sample collected for more than one intended use

3.4
continuous sampling
taking of a sample from every consecutive *sub-lot* (3.30) so that *increments* (3.10) are taken at uniform intervals over the entire *lot* (3.11) being handled

3.5
cut
see *increment* (3.10)

3.6
divided increment
part obtained from the division of the *increment* (3.10) in order to decrease its mass

Note 1 to entry: Such division can be done with or without prior size reduction.

3.7
fixed-mass division
method of sample division in which the mass retained is predetermined and independent of the mass of the feed

3.8
fixed-ratio division
method of sample division in which the division ratio is predetermined, i.e. the mass of sample retained is a fixed proportion of the mass of the feed

3.9
general analysis test sample
sample prepared to pass a sieve of nominal size of openings of 212 µm used for the determination of most chemical and some physical characteristics

3.10
increment
portion of coal or coke extracted in a single operation of the sampling device

Note 1 to entry: Cut is an equivalent term.

3.11
lot
defined quantity of coal or coke for which the quality is to be determined

Note 1 to entry: A lot can be divided into sub-lots.

3.12
manual sampling
extraction of *increments* (3.10) by human effort

3.13
mass-basis sampling
taking of *increments* (3.10) whereby the position of each *increment* (3.10) to be extracted from the stream of coal or coke is measured by a mass interval of stream flow and the *increment* (3.10) mass is fixed

3.14
mechanical sampling
extraction of *increments* (3.10) by mechanical means

3.15
moisture sample
sample taken specifically for the purpose of determining total moisture

Note 1 to entry: For coke, this sample can also be used for general analysis.

3.16
nominal top size
aperture size of the smallest sieve in the range included in the R 20 Series on which not more than 5 % of the mass of the sample is retained

Note 1 to entry: See ISO 565, square hole.

3.17
physical sample
sample taken specifically for the determination of physical characteristics, e.g. physical strength indices or size distribution

3.18
precision
closeness of agreement between independent test results obtained under stipulated conditions

Note 1 to entry: This is often defined using an index of precision, such as 2 standard deviations.

Note 2 to entry: A determination might be made with great precision and the standard deviation of a number of determinations on the same sub-lot might, therefore, be low; but such results are accurate only if they are free from bias.

3.19
primary increment
increment (3.10) extracted at the first stage of sampling, prior to any sample division and/or sample reduction

3.20
random sampling
extracting of *increments* (3.10) at random mass or time intervals

3.21
replicate sampling
extracting, at intervals, of *increments* (3.10) that are combined in rotation into different containers to give two or more samples of approximately equal mass

3.22
representative sample
sample collected in such a manner that the analyses, size distribution and moisture content represent that of the *lot* (3.11)

3.23
sample
quantity of coal or coke, representative of a larger mass for which the quality is to be determined

3.24
sample division
process in sample preparation whereby the sample is divided into representative, separate portions

3.25
sample preparation
process of bringing samples to the condition required for analysis or testing

Note 1 to entry: Sample preparation covers mixing, particle size reduction, sample division and sometimes *air-drying* (3.1) of the sample and may be performed in several stages

3.26
sample reduction
process in sample preparation whereby the particle size of the sample is reduced by crushing or grinding

3.27
size analysis sample
sample taken specifically for particle size analysis

3.28
standard deviation
square root of the variance

3.29
stratified random sampling
extracting of an increment at random within the mass interval or time interval determined for mass-basis sampling or time-basis sampling respectively

3.30
sub-lot
part of a *lot* (3.11) for which a test result is required

3.31
systematic sampling
extracting of *increments* (3.11) at uniform time intervals according to a predetermined plan

3.32
test sample
sample which is prepared to meet the requirements of a specific test

3.33
time-basis sampling
extracting of increments whereby the position of each increment to be collected from the stream of coal or coke is measured by a time interval and the increment mass is proportional to the flow rate at the time the increment is taken

3.34
variance
measure of dispersion, which is the sum of the squared deviations of observations from their average divided by one less than the number of observations

4 Establishing a sampling scheme

4.1 General

4.1.1 Sampling

Mechanical sampling of coal and coke in accordance with ISO 13909-2, ISO 13909-3 and ISO 13909-5 is the preferred method. However, where this is not possible, manual sampling may be conducted. The recommended method for manual sampling of coal and coke is while it is being transferred, e.g. loading or unloading of ships, barges, wagons, rail cars and trucks, transferring coal or coke using fixed/moveable conveyors and/or stackers, or during the formation of or reclaiming from stockpiles. For safety and practical reasons, manual sampling of coal or coke being transferred is sometimes not possible.

NOTE Manual sampling in stationary situations (see Annex B) refers to static lots, where no formation of or reclaiming from piles/heaps takes place.

Increments should be collected by trained sampling personnel. Instructions should be as complete and as simple as possible, in particular, the position of sampling and the times at which increments are taken should be specified and not left to the personal judgement of the sampling personnel. These instructions, which should preferably be set out in writing, should be prepared by the sampling supervisor from the information given in this document.

4.1.2 Sampling scheme

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) select or assume the required overall precision for the lot (see 4.3.2);

d) determine or assume the variability of the coal or coke (see 4.3.3 and, if relevant, 4.3.4) and the variance of preparation and testing (see 4.3.5);

e) ascertain the nominal top size of the coal or coke for the purpose of determining the mass of increment and sample (see 4.3.6.3 and 4.3.7);

f) the nominal top size should initially be ascertained by consulting the consignment details or by visual estimation and should be verified by preliminary test work;

g) select the sampling device (see Clause 6);

h) establish the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see 4.3.6);

i) determine the method of combining the increments into samples and the method of sample preparation (see Clause 8);

j) define the sampling interval in terms of time or mass (see Clause 5);

k) determine where to collect the increments (see Clause 5).

4.1.3 Parameters

In order to ensure that the result obtained is to the required precision, the following parameters are considered:

a) variability of the coal or coke;

b) number of increments to be taken from the lot;

c) number of sub-lot samples to be constituted for the lot;

d) number of increments comprising each sub-lot sample;

e) mass of sample relative to nominal top size.

4.1.4 Sampling methods

In this document, only continuous sampling methods are considered.

4.2 Design of the sampling scheme

4.2.1 General

The basic first step in the design of a sampling scheme is a review of the requirements for operations in order to draw up instructions for the sampling operator(s). The instructions should cover all sampling problems likely to be encountered.

It is important that the sampling operator receive instructions that are simple, easily understood and capable of only one interpretation. These instructions, which should be set out in writing, should be prepared by the sampling supervisor after inspecting the sampling site and referring to the information given in this document. The following items in the following list and described in 4.2.2 to 4.2.6 should be considered by the supervisor when compiling instructions:

a) coal or coke to be sampled and considerations for sampling;

b) lot size and number of sub-lots;

c) method of sampling;

d) requirements for test samples;

e) number of increments per lot or sub-lot;

f) mass of sample;

g) precision of results;

h) bias of results.

4.2.2 Coal or coke to be sampled and considerations for sampling

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

The following identifications are indispensable for the design of a manual sampling scheme:

a) coal or coke properties, e.g. fines, lump and, more specifically, the nominal top size; furthermore, whether dry, wet or free flowing;

b) location and the handling system;

c) transport means/carriers;

d) where to sample in the handling process, taking into account contract terms and the practicability for sampling;

e) human safety risks.

4.2.3 Division of lots

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. The size of each sub-lot should be selected for convenience of sampling, e.g. coal or coke despatched or delivered over a period of time, a train load, a wagon load, or coal or coke produced during a certain period, e.g. a shift. For large lots, such as ocean-going vessels, it is recommended to sample in multiple sub-lots for the reasons below. It is common industry practice to limit sub-lot sizes to a maximum of 10 000 t. However, when the loading or discharge rate is less than

1 000 t/h, it is recommended that sub-lot sizes be limited to 5 000 t to avoid moisture bias due to long periods of sample collection.

Such division into a number of sub-lots may be necessary to achieve the following:

a) the required precision (calculated by the procedure in 4.5);

b) maintain the integrity of the sample, e.g. avoiding bias that can result from changes of moisture due to standing or oxidation;

c) create convenience when sampling lots over a long period, e.g. on a shift basis;

d) keep sample masses manageable, taking into account the maximum lifting capacity of operators;

e) distinguish different components of a mixture of coal or coke, e.g. different coal types within one lot.

4.2.4 Precision of results

After the overall precision of the lot has been decided, the number of sub-lots and the number of increments per sub-lot collected shall then be determined as described in 4.3.6 and the mass of the primary increments shall be determined as described in 4.3.6.3.

For single lots, the quality variation shall be assumed as the worst case (see 4.3.3). The precision of sampling achieved may be measured using the procedure of replicate sampling (see 4.5).

At the start of regular sampling of unknown coal or coke, the worst-case quality variation shall be assumed in accordance with 4.3.3 and 4.3.4.

If any subsequent change in precision is required, the number of sub-lots and of increments shall be changed as determined in 4.3.6 and the precision attained rechecked. The precision shall also be checked if there is any reason to suppose that the variability of the coal or coke being sampled has increased. The number of increments determined in 4.3.6 applies to the precision of the result when the sampling errors are large relative to the sample preparation and testing errors, e.g. moisture. However, in some tests, the testing errors are themselves large. In this case, it can be necessary to prepare two or more test portions from the sample and use the mean of the determinations to give a better precision.

4.2.5 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 can require, in some circumstances, a limit on the mass of the primary increment, the divided sample and the test sample to maintain integrity (see 4.3.6.3 and 4.3.7).

It may be necessary, when collecting samples for moisture determination from lots over an extended period, to limit the standing time of samples by dividing the lot into a number of sub-lots. For establishing the loss of integrity of the sample, a bias test can be carried out to compare a series of reference samples immediately after extraction with samples after standing for the normal time to establish moisture or calorific value loss due to standing (see ISO 13909-8).

Bias testing for manual sampling can be performed according to the same principles as for mechanical sampling using a reference method to judge a manual sampling practice (see ISO 13909-8).

4.2.6 Requirements for test samples

In the sampling scheme and in the scheme of preparation of samples, attention shall be paid to requirements on the samples for testing.

A number of tests are carried out on crushed or pulverized samples of prepared top sizes as mentioned in the relevant testing standards, e.g. ash on a − 0,212 mm sample. However, some tests require

samples either in the original state or prepared to a particle size somewhere between original state and 0,212 mm.

Examples of physical tests on samples in their original state are size-distribution tests, float and sink tests, coking tests, etc.

Examples of tests on partly crushed and prepared samples are total moisture, Hardgrove grindability index and dilatation.

In view of the above, consideration of the sampling and preparation schemes should foresee either whether all required samples can be taken and prepared from a common sample or whether it is necessary to take a number of separate samples. In all cases, the masses of the common sample and the required test samples should be maintained in accordance with the minimum masses as prescribed in this document and in the standard specifying the test method. In case of differences between standards, the greater mass should be maintained.

In case the mass of the sample as calculated in accordance with this document is insufficient for the masses of the required test samples, the number of increments taken from each lot or sub-lot shall be increased by increasing the sampling frequency to provide the required mass

4.3 Precision of sampling

4.3.1 General

In all methods of sampling, sampling preparation and analysis, errors are incurred, and the experimental results obtained from such methods for any given parameter deviate from the true value of that parameter. As the true value cannot be known exactly, it is not possible to assess the accuracy of the experimental results, i.e. the closeness with which they agree with the true value. However, it is possible to make an estimate of the precision of the experimental results, i.e. the closeness with which the results of a series of experiments made on the same coal or coke agree among themselves.

It is possible to design a sampling scheme that, in principle, can achieve a desired level of precision, which needs to be specified prior to designing the sampling scheme.

The required overall precision on a lot should be agreed between the parties concerned. In the absence of such agreement, a value of one tenth of the ash, expressed as a percentage mass fraction, may be assumed up to 10 % ash (dry basis), subject to a maximum of 1 % absolute for ash above 10 % mass fraction.

4.3.2 Precision and total variance

Precision is the closeness of agreement between the results obtained by applying the experimental procedure several times under specified conditions, and is a characteristic of the sampling scheme used and the variability of the coal or coke being sampled. The smaller the random errors of the scheme, the more precise is the scheme. A commonly accepted index of precision is two times the sample estimate of the population standard deviation, and this index of precision is used throughout this document.

If a number of replicate samples (minimum 10) are taken from a lot of coal or coke, prepared and analysed separately, the precision, P, of a single observation is given by Formula (1):

$$P = 2s = 2\sqrt{V_{SPT}} \tag{1}$$

where

s is the sample estimate of the population standard deviation;

V_{SPT} is the total variance of the results for replicate samples.

The total variance in Formula (1) is a function of the primary increment variance, the number of increments, and the errors associated with sample preparation and testing.

For a single sample, this relationship is expressed by Formula (2):

$$V_{SPT} = \frac{V_I}{n} + V_{PT} \tag{2}$$

where

V_I is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of primary increments in the sample.

Where the result of Formula (2) is the arithmetic mean of a number of sample values, resulting from dividing the lot into a series of sub-lots and taking a sample from each, V_{SPT} is given by Formula (3):

$$V_{SPT} = \frac{V_I}{mn} + \frac{V_{PT}}{m} \tag{3}$$

where

n is the number of primary increments comprising each sample, i.e., the number of increments per sub-lot;

m is the number of sample results, i.e. sub-lots, used to obtain the mean.

Since a sample is equivalent to one member of a set of replicate samples, combining Formula (1) and Formula (3) for continuous sampling results in Formula (4) and Formula (5):

$$P_L = \frac{P_{SL}}{\sqrt{m}} = 2\sqrt{\frac{V_I}{mn} + \frac{V_{PT}}{m}} \tag{4}$$

$$P_{SL} = P_L \sqrt{m} \tag{5}$$

where

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

P_{SL} is the overall precision for the sub-lot at 95 % confidence level, expressed as % absolute;

n is the number of increments per sub-lot;

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

If the quality of a coal or coke of a type not previously sampled is required, then in order to devise a sampling scheme, assumptions have to be made about the variability (see 4.3.3).

4.3.3 Primary increment variance

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

For some coal or coke, the increment variance for ash is higher than that for moisture and, hence, for the same precision, the number of increments required for the general analysis sample is adequate for the moisture sample and the common sample.

The value of the primary increment variance, V_I, required for the precision using Formula (4) can be obtained by either:

a) assuming a value determined for a similar coal or coke from a similar handling and sampling operation, or

b) determining it directly on the coal or coke to be sampled by taking at least 50 increments spread over an entire lot or over several lots of the same type of coal or coke and analysing each increment separately for the required parameters, preferably ash (dry basis) and total moisture.

For calculating the variance, Formula (6) can be used:

$$V_I = \frac{1}{n-1}\left[\sum x_i^2 - \frac{(\sum x_i)^2}{n}\right] - V_{PT} \tag{6}$$

where

n is the number of increments taken;

x_i is the value of the analysed parameter.

If neither of the values for V_I obtained from a) or b) above is available, a value of V_I = 5 for the ash of unwashed and blended coals, as well as for cokes, and V_I = 3 for the ash of washed coals can be assumed initially and checked after sampling has been carried out using one of the methods described in ISO 13909-7.

4.3.4 Sub-lot variance

In some cases (e.g. see 4.3.2), the sub-lot variance, V_{SL}, can be calculated, because, just like the primary increment variance, this value gives an indication of the homogeneity of the coal or coke. For calculation of V_{SL} Formula (7) can be used:

$$V_{SL} = \frac{1}{N_{SL}-1}\left[\sum x_{SL}{}^2 - \frac{(\sum x_{SL})^2}{N_{SL}}\right] - V_{PT} \tag{7}$$

where

N_{SL} is the number of sub-lots;

V_{SL} is the sub-lot variance;

x_{SL} is the value of the analysed parameter from the sub-lot.

If the variance of different lots/sub-lots or different cargoes of the same coal or coke varies considerably, the primary increment variance found for any lot or cargo cannot be used for calculation of the number of increments for the next lot or cargo.

4.3.5 Preparation and testing variance

The value of the preparation and testing variance, V_{PT}, required for the calculation of the precision using Formula (4) can be obtained by either:

a) assuming a value determined for a similar coal or coke using a similar sample preparation scheme, or

b) determining it directly on the coal or coke to be sampled by constituting at least 20 sub-samples spread over the entire lot or over several lots of the same type of coal or coke. Each sub-sample is divided into two parts and prepared so that split portions of each sub-sample are taken at the first division stage. Each portion shall be prepared and tested for the parameters of interest, preferably ash (dry basis) and total moisture. The same analysing methods are applied as are used in routine operations. The difference between the two results shall be calculated for each pair and the preparation and testing variance, V_{PT}, can be calculated using Formula (8) as follows:

$$V_{PT} = \frac{\sum d_i^2}{2n_p} \tag{8}$$

where

d_i is the difference between individual pair members;

n_p is the number of pairs.

Alternately, split one or more sub-lot samples into a minimum of 20 test samples. Prepare and analyse each test sample for the parameters of interest, preferably ash (dry basis) and total moisture. The preparation and testing variance can be calculated using Formula (9) as follows:

$$V_{PT} = \frac{1}{N-1}\left[\sum x_i^2 - \frac{(\sum x_i)^2}{N}\right] \tag{9}$$

where

N is the number of test samples;

x_i is the value of the analysed parameter.

If neither of the values from Formula (8) or Formula (9) is available, a value of V_{PT} = 0,2 for ash can be assumed initially and checked if necessary, after preparation and testing has been carried out.

If high overall precision, P_L, is required, then lower V_{PT} values of 0,1 or 0,05 for ash are required to obtain the required overall precision using a practical number of primary increments and sub-lots (see 4.3.6).

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

4.3.6.1 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 or 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 can be necessary in order to achieve the required precision and the necessary number of sub-lots shall be calculated using the procedure given in 4.3.6.2 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 requirement to do this is dependent on factors such as the time taken to collect the samples, ambient temperature and humidity conditions, the ease of keeping the sample in sealed containers during collection and the particle size of the coal or coke. It is recommended that, if moisture loss is suspected, steps be taken to minimize moisture loss by reducing the sample standing time and collecting samples more frequently, i.e. by increasing the number of sub-lots. See also 4.2.3

Establish the number of sub-lots and number of increments required per sub-lot in accordance with 4.3.6.2 as appropriate.

NOTE The formulae given in 4.3.6.2 generally give an overestimation of the required number of increments. This is because they assume that the quality of coal or coke 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 make provisions for the worst case anticipated and then tend to use higher values for V_I and V_{SL} than may actually occur when the scheme is in operation. On implementing a new sampling scheme, a check on the actual precision being achieved should be carried out using the methods described in ISO 13909-7. This check may be necessary to achieve the required precision, in which case the number of sub-lots is calculated using the procedures given in 4.3.6.2.

4.3.6.2 Number of increments

Determine the number of sub-lots, m, required for practical reasons (see 4.3.6.1) and then estimate the number of increments, n, for a desired precision from Formula (10), obtained by rearranging Formula (4):

$$n = \frac{4V_I}{mP_L^2 - 4V_{PT}} \tag{10}$$

A value for n 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, reduce the errors of sample preparation and testing or increase the number of sub-lots by one of the following means:

a) Choose a new number of sub-lots corresponding to a convenient sub-lot mass, recalculate n from Formula (10) 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 (11);

$$m = \frac{4(V_I + n_1 V_{PT})}{n_1 P_L^2} \tag{11}$$

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.

A worked example of a calculation of overall precision, mass of increments, number of sub-lots and number of increments per sub-lot is given in Annex A.

4.3.6.3 Mass of increments

The minimum mass of increment is determined by the correct dimensions of the manual sampling implement to ensure that the increments are representative.

4.3.7 Minimum mass of samples for general analysis and determination of total moisture content

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 adequately represent all the particle sizes in the mass of coal or coke being sampled

The minimum mass of a sample (primary as well as after division) is dependent on the nominal top size of the coal or 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 does not, of itself, guarantee the required precision. This is also dependent on the number of increments in the sample and their variability (see 4.5).

Values for the minimum mass of coal samples for general analysis to reduce the variance due to the particulate nature of the coal to 0,01, corresponding to a precision of 0,2 % with regard to ash, are given in column 2 of Table 1 (see Reference [3]). Column 3 of Table 1 gives the corresponding minimum masses of divided coal 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 mass of coke samples given Table 2 are for guidance on the minimum mass for unknown or heterogeneous cokes, which can usually be reduced for moisture samples.

The minimum mass of a divided increment shall be such that the combined masses of all the divided increments in the sub-lot (or lot if sub-lots are not established) shall, at each stage, be greater than the mass given in Table 1 for coal or Table 2 for coke corresponding to the purpose for which the sample has been taken and the nominal top size. If the increment masses are too low to satisfy this requirement, the divided increment shall be crushed prior to further division.

Note that in each case, the overall division precision is determined by the sum of the division variances for each division stage.

The minimum sample mass, m_S, for coal for other desired levels of division precision may be calculated from Formula (12):

$$m_S = m_{0,S}\left(\frac{P_D}{P_R}\right)^2 \tag{12}$$

where

$m_{0,S}$ is the mass given in Table 1;

m_S is the resulting mass for the sample to be taken;

P_D is the default division precision value (e.g. 0,2 % ash, due to the particulate nature of the coal);

P_R is the desired division precision for the particular sampling stage.

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

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

NOTE 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.

Table 1 *(continued)*

Nominal top size of coal mm	General-analysis samples and common samples kg	Samples for determination of total moisture content kg
10	10	2
8,0	6	1,50
5,6	3	1,20
4,0	1,50	1,00
2,8	0,65	0,65
2,0	0,25	0,65
1,0	0,10	0,65

NOTE 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.

Table 2 — Minimum mass of coke samples for general analysis, determination of total moisture content and sizing

Nominal top size of coke mm	Minimum mass kg
>125	2 000
125	1 000
100	650
90	500
75	320
63	250
50	200
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

When coal or coke is regularly sampled under the same circumstances, the overall precision obtained for all the required parameters shall be checked (see 4.4) and the masses may be adjusted accordingly. However, the masses shall not be reduced below the minimum requirements laid down in the relevant standard specifying the test method.

Where samples for special tests (see 4.3.6) are to be extracted from a common sample, the initial number of increments collected shall be that required for general analysis or total moisture, whichever is the greater. If there is not sufficient mass of coal or coke left for the general sample after the extraction, the mass of sample given by this number of increments shall be increased by increasing the sampling frequency and taking extra increments. Extraction of a special test sample from the common sample is described in 8.7.

When preparing coal or coke 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.

NOTE Manual sampling of coal or coke with top sizes >90 mm and >125 mm respectively, is not considered feasible.

4.4 Checking the overall precision for the lot by calculation and selection of sampling scheme

Manual sampling offers the flexibility and convenience of selecting the most convenient combination of number of sub-lots and number of increments as needed. In order to check the sampling precision, it is therefore recommended to calculate a variety of combinations and then to decide on the sampling scheme.

A worked example of a calculation for overall precision, mass of increments, number of sub-lots and number of increments per sub-lot is given in Annex A.

4.5 Determination of acquired precision by replicate sampling

4.5.1 General

By using the procedure of replicate sampling, it is possible to test the precision for a specific lot obtained by a particular sampling scheme. With this procedure the same number of increments as usual is collected, but successive increments are placed into a number of different sample containers to give a number of replicate sub-samples. From each of these a separate laboratory sub-sample is prepared and a test is carried out on each so that eventually there are a number of different sub-sample values for ash or any other characteristic tested. It is noted that each replicate sub-sample is composed of a smaller number of increments than normal.

4.5.2 Method and calculation

Establish the parameter to be analysed, e.g. ash (dry basis), and establish the sampling scheme for the required precision in accordance with 4.3.

Instead of forming a sample from each sub-lot, combine the total number of increments, n_T, as replicate samples. The number of replicate samples, N_{RS}, shall be not less than the number of sub-lots, N_{SL}, used in the calculations and not less than 10. If there are 10 such samples and the sample containers are labelled A, B, C, D, E, F, G, H, I, J, then successive increments go into the containers as follows: A, B, C, D, E, F, G, H, I, J, A, B, C, D, ...

A typical calculation for coal or coke using the results in Table 3 is given below.

Table 3 — Results of single-lot sampling for % ash, dry basis

Sample No.	Sample value, x_i %	x_i^2
A	15,30	234,09
B	17,10	292,41
C	16,50	272,25
D	17,20	295,84
E	15,80	249,64
F	16,40	268,96
G	15,70	246,49
H	16,30	265,69
I	18,00	324,00

Table 3 *(continued)*

Sample No.	Sample value, x_i %	x_i^2
J	16,70	278,89
Total	165,00	2 728,26

The number, N_{RS}, of replicate samples is 10.

The mean result is 165/10 = 16,5 %.

The sample estimate of the population standard deviation, *s*, is:

$$s = \sqrt{V} \tag{13}$$

Consequently,

$$s = \sqrt{\frac{\left[\sum x_i^2 - \frac{(\sum x_i)^2}{n}\right]}{(n-1)}} \tag{14}$$

$$= \sqrt{\frac{2728{,}26 - \frac{165^2}{10}}{9}} = 0{,}800$$

The best estimate for the precision, *P*, achieved for the lot is given by Formula (15):

$$P = \frac{2s}{\sqrt{N_{RS}}} \tag{15}$$

i.e.

$$P = \frac{2 \times 0{,}8}{\sqrt{10}} = 0{,}506$$

4.5.3 Precision obtained using normal sampling scheme

If it is desired to design a regular sampling scheme based on the results of this procedure, the estimate of precision obtained using ISO 13909-7, the number of increments per sample and the number of replicate samples (instead of the number of sub-lots) can be substituted into Formula (4) and the value for the increment variance estimated. The procedures given in 4.3 can then be followed to design the regular sampling scheme.

4.6 Size analysis

Within the scope of this document, the coals and cokes to be sampled exhibit wide differences in size, size range and size distribution. In addition, the parameters determined (percentage retained on a particular sieve, mean size, etc.) can 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.

These factors should be taken into account when applying the techniques for calculating the numbers of increments for a particular precision (see 4.3.2).

The minimum mass of the size analysis sample of coal is given in ISO 1953. Manual sampling of coal with top sizes >90 mm is not feasible. 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 is normally better than this. The minimum masses of sample for size analysis of coke are given in Table 2 above.

The precision for the particular parameter required shall then be checked and the number of increments adjusted according to the procedure described in 4.5.

Minimization of degradation of samples used for determination of the size distribution is vital to reduce bias in the measured size distribution. To prevent particle degradation, free-fall drops shall be kept to a minimum.

5 Methods of sampling

5.1 General

The fundamental requirements of sampling are that all parts of the coal or coke in the lot shall be accessible to the sampling instrument. Sampling shall be carried out by systematic sampling, either on a time or mass basis, or by stratified random sampling.

Biased samples are obtained if part of the coal or coke is excluded. Extra care should be taken when sampling particularly heterogeneous or layered coal or coke or mixtures of coal or coke. Cyclical variations in coal or coke quality can occur during 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 is invariably introduced that can be of unacceptable proportions. In such circumstances, stratified random sampling shall be adopted.

The methods described in Clause 5 are applicable to coal or coke being transferred as follows:

a) stopped belt;

b) falling stream;

c) moving belt;

d) stockpiles (building/reclaiming);

e) grabs/front-end loaders;

f) barges/trucks/railcars (loading/unloading).

Annex B presents informative methods for sampling coal or coke in stationary lots. Sampling from lots consisting of different coals or cokes is described in 5.8, while the use of random selection of increments is described in 5.9.

5.2 Sampling by time interval (time-basis sampling)

Primary increments shall be taken at equal pre-set 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 time interval until the handling operation is completed.

The time interval, Δt, expressed in minutes, between taking primary increments is determined from Formula (16):

$$\Delta t \leq \frac{60 m_{SL}}{Gn} \tag{16}$$

where

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

G is the maximum flow rate of the coal or coke, in tonnes per hour;

n is the number of primary increments in the sample (see 4.3.6.2).

5.3 Sampling by mass interval (mass-basis sampling)

Primary increments shall be taken at a pre-set mass interval throughout the mass of the lot or sub-lot. This interval shall not be changed during the sampling operation. If the calculated number of increments has been taken before the handling has been completed, additional increments shall continue to be taken at the same interval until the handling operation is completed.

The mass interval, Δm, expressed in tonnes, between taking increments is determined from Formula (17):

$$\Delta m \leq \frac{m_{SL}}{n} \tag{17}$$

where

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

n is the number of primary increments in the sample.

The mass interval between increments shall be equal to or smaller than that calculated from the number of increments specified in 4.3.6.2 in order to ensure that the number of increments is at least the minimum number specified.

5.4 Stratified random sampling

5.4.1 General

Stratified random sampling means that, 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 are collected very close together even though they are collected in different mass or time intervals.

5.4.2 Stratified random sampling by time interval

The sampling interval shall be determined as in 5.2 and the increment mass as in 4.3.6.3. 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.

5.4.3 Stratified random sampling by mass interval

The sampling interval shall be determined as in 5.3 and the increment mass as in 4.3.6.3. 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.

5.5 Extracting the increment

Suitably trained and experienced sampling personnel shall carry out extraction of the increments using appropriate equipment.

Increments should be extracted in a single operation, without overflowing or spillage from the sampling device.

The aperture of the sampling device shall be at least three times the nominal top size of the coal or coke, with a minimum dimension of 30 mm (see Clause 6). Larger apertures may be necessary, to ensure that the larger particles are not excluded from the increment and/or that wet sticky coal does not cause blinding of the aperture.

Large and hard pieces of coal, coke or rock shall not be pushed aside deliberately when an increment is extracted. Do not allow wet coal or coke to adhere to the sampling equipment.

5.6 Coal or coke in motion

5.6.1 General

Coal or coke in motion can be divided into two categories, i.e. coal and coke in continuous motion where it is being transferred from one point to another by a moving conveyor belt, while coal or coke in incremental motion is where it is being transferred from one point to another in small batches or loads such as by trucks, railway wagons, payloaders, grabs, etc

It is important when manually sampling coal or coke in motion to maximize the number of particles that the sampling implement can access and at the same time minimize the number of particles in the coal and coke with a zero chance of being selected for inclusion in the sample collected.

When selecting a sampling method from the alternatives listed below, the safety of sampling personnel is paramount. Coal or coke in motion involves multiple hazards such as moving conveyor components to mobile machines moving the coal or coke. The risks are increased when ambient conditions are poor, including limited vision (night-time operation), dust and inclement weather. As such, selecting an appropriate method should be based on an assessment of risk, including the mass of the sample to be handled at any given moment by sampling personnel. The sampling of coal or coke in continuous motion from a moving conveyor may require mechanical means and/or mechanical assistance to collect the primary increment safely.

5.6.2 Stopped belt

Some methods of sampling tend to collect too many of either the large or the small particles and hence are liable to introduce bias. The method of extracting an increment by removing a whole cross-section from a stopped belt is the only way of ensuring that all particles are collected and hence that the sample is free from bias. Therefore, this is the reference method against which any other method should be checked and is the most ideal method of sampling. Increments should be extracted from the whole width and thickness of the coal or coke stream when there is a normal load at the point of sampling.

If it is practicable to arrange with the operator to stop the belt periodically, increments can be extracted from the whole cross-section of the stream. The risk assessment should include factors such as ease of access, the mass of the stopped-belt cutter, and strict measures to block electrical power to conveyor drive motors during the process of sample collection.

While the actual increment is collected when the coal or coke on the conveyor belt is not in motion, stopped-belt sampling is still considered to be sampling coal or coke in continuous motion, because the stopping of the belt is determined by sampling personnel.

Stopped-belt increments shall be extracted with a sampling frame [see Figure 2 j)], or equivalent, from a complete cross-section of the coal or coke on the belt at a fixed position. The width of the complete section shall be at least three times the nominal top size of the coal or coke to be sampled, with a minimum dimension of 150 mm. The frame shall be placed on the stationary belt so that it is in contact with the full width and depth of the belt.

Particles obstructing the insertion of the end plate on the left-hand side of the collection end of the sampling frame shall be pushed into the increment, while those obstructing the insertion of the end

plate on the right-hand side shall be pushed out of the increment or vice versa. Whichever practice is used initially, this practice shall be implemented throughout the test.

All pieces of coal or coke on the belt lying within the frame shall be swept into the sample container. No portion of the increment should be lost during extraction. Wet coal or coke should not be allowed to adhere to the sampling frame, but the sampling frame shall not be heated to stop wet coal or coke sticking to it.

NOTE Proper insertion of the stopped-belt frame becomes increasingly difficult at top sizes larger than 100 mm and caution should be taken at these larger top sizes.

5.6.3 Falling stream

Samples are taken from coal or coke in continuous motion at a transfer point for a continuously moving stream.

The sampling personnel shall be able to reach the whole cross-section of the stream in safety and handle the resulting increment without undue physical strain or loss of sample material. Since safety is a significant concern at high flow rates, the risk assessment should include factors such as the mass of the increment collected and the force exerted by the falling stream of coal or coke. It may be necessary to provide mechanical support for the sampling device when it is passed through the falling stream or to erect a special gantry with adequate support.

Increments shall be extracted from a falling stream by means of a sampling device that is moved across the width of the stream, as far as possible at a constant rate, less than 0,6 m/s. The aperture of the sampling device shall be at least three times the nominal top size of the coal or coke, with a minimum dimension of 30 mm (see 6.2.1 and 6.2.5) and larger, as necessary, to ensure that the larger particles are not excluded from the increment and that the increment does not overflow the sampling device.

The sampling device shall traverse the full cross-section of the stream. The coal or coke near the periphery shall be adequately represented and, if layering takes place with different types of coals or cokes or size distribution, these shall be proportionally represented.

This may be achieved by passing the sampling device through the stream from right to left, or vice versa, or by inverting the sampling device, passing it to the back of the stream and withdrawing it through the stream, provided the sampling device completely intersects the whole stream and clears the stream in each pass. Alternatively, the sampling device may be filled in passing from front to rear, provided that it can then be withdrawn away from the stream, e.g. by moving it sideways.

Proper manual extraction of samples from falling streams becomes increasingly difficult at stream widths greater than 300 mm and top sizes larger than 50 mm, so caution should be taken.

5.6.4 Moving belt

Sampling coal or coke from any moving belt is hazardous. However, under some circumstances part-stream sampling from a moving belt, or at a transfer point, may be considered, subject to conducting a risk analysis at the outset. The risk assessment shall include the width, speed and throughput of the conveyor, and adequate guards shall be constructed to protect sampling personnel from all moving parts. This type of sampling will only take place on slow moving belts or at low flow rates, or as a temporary backup method when mechanical sampling equipment is broken down. This can be accomplished with the use of a long-handled scoop or sided shovel. Increments should be collected by inserting the sampling instrument into the coal or coke in the same direction as the flow of material. For wide conveyors, increments should be collected from both sides. However, this type of sampling shall not be used for large, fast moving belts, because the safety of sampling personnel is paramount. At higher flow rates, mechanical assistance is necessary to ensure that primary increments can be collected safely.

Other key considerations when applying part-stream sampling are the lack of mixing of the coal or coke in the case of blends, and the strong tendency of the coal or coke to segregate by particle size during movement by conveyor.

5.6.5 **Stockpiles** (building/reclaiming)

Sampling of stockpiles shall be carried out during the process of building up or reclaiming from the stockpile. Sampling of stationary stockpiles is not recommended because it is impossible to penetrate to the bottom of the stockpile to collect a representative sample with manual sampling implements. However, if it is the only alternative, the procedures in Annex B should be used, but the results are merely indicative of the quality of the coal or coke.

Increments shall be extracted from the working face of the stockpile provided it is safe to do so, from the bucket of a front-end loader (see 5.6.6), or from a single, discrete load delivered to the stockpile before being pushed into the main stockpile. When extracting increments from a working face, the surface shall be sufficiently compacted to bear safely the weight of personnel and equipment.

A manual sampling shovel, probe or scoop shall be used to extract increments. The use of augers is not permitted. The width of the sampling shovel and scoop and the aperture of the probe shall be at least three times the nominal top size of the coal or coke, with a minimum dimension of 30 mm (see Clause 6) and larger, if necessary, to ensure that the larger particles are not excluded from the increment and that the increment never fills the sampling shovel, probe or scoop completely. Owing to the difficulty of insertion, a probe shall be used only for coal or coke with a particle size of up to about 25 mm. Probes shall not be used for coal or coke that require size analysis. Increments shall be spaced as evenly as possible over the working face or the surface of the load in the front-end loader bucket.

Selected front-end loader buckets of coal or coke can also be discharged onto a clean surface, if required, and then the coal or coke can be sampled by either full-depth sampling or by taking increments from the freshly exposed coal or coke.

When extracting increments, the manual sampling shovel, probe or scoop shall be inserted at right angles to the surface of the coal or coke after the top surface of the coal or coke has been removed. Large pieces of coal or coke shall not be deliberately pushed aside when an increment is extracted and no portion of the increment shall be lost during extraction of the sampling shovel, probe or scoop from the surface. A full column of coal or coke shall be extracted so that a representative increment is taken.

5.6.6 Grabs/front-end loaders

When the coal or coke is handled by grabs or front-end loaders, the only solution may be to extract the increments from the grabs or front-end loaders.

For safety reasons, it is important that sampling personnel coordinate the extraction of the increments unambiguously with the operator of the grab or front-end loader. The risk assessment should include factors such as lighting, communications, hi-visibility clothing, etc.

A manual sampling shovel, probe or scoop shall be used. The use of augers is not permitted. The width of the sampling shovel and scoop and the aperture of the probe shall be at least three times the nominal top size of the coal or coke, with a minimum dimension of 30 mm (see Clause 6) and larger, as necessary, to ensure that the larger particles are not excluded from the increment. Probes that result in attrition of the sample shall not be used for coal or coke that requires size analysis. Increments shall be spaced as evenly as possible over the surface of the front-end loader bucket or the grab.

With large grabs and front-end loaders, each grab or front-end loader load may be divided into sections, only one of which is taken. When sampling successive grabs or front-end loader loads, each section shall be taken in rotation.

Selected grabs and front-end loaders full of coal or coke may be discharged on to a clean surface and then the coal or coke can be sampled either by full-depth sampling or by taking increments from the freshly exposed coal or coke. Sufficient grabs or front-end loaders of coal or coke shall be selected to ensure that the required number of increments is obtained.

When extracting increments, the manual sampling shovel, probe or scoop shall be inserted at right angles to the surface of the coal or coke. Large pieces of coal or coke should not be deliberately pushed aside when an increment is extracted, and no portion of the increment should be lost during extraction

of the sampling shovel, probe or scoop from the surface. Owing to the difficulty of insertion, a probe shall be used only for coal or coke with a particle size of up to about 25 mm. A full column of coal or coke shall be extracted so that a representative increment is taken.

5.6.7 **Barges/trucks/railcars/wagons** (loading/unloading)

Sampling of coal or coke during its loading or unloading is based on progressively extracting increments from a number of points distributed over the freshly exposed surfaces. It is not permitted to sample the tops of fully loaded barges, trucks or railcars before these are unloaded, due to possible segregation or weather influences during transportation. Sampling of the full depth is recommended using a mechanical device such as a mechanical auger or a rotary cylinder sampler (see ISO 13909-3). For coke, mechanical augers are not recommended due to coke breakage and problems with penetrating the coke. The use of manual augers and manual probes is not recommended, because, in most cases, it is not physically possible to sample the full depth with these devices.

Sampling of coal or coke in barges shall be based on extracting increments from a number of points distributed over various layers of the coal or coke in the hold, which are exposed from time to time as the barge is loaded or unloaded. Sampling shall be done from sequential layers during loading or unloading. For safety reasons, it is important that sampling personnel coordinate the extraction of the increments unambiguously with the operator of the barge, truck, railcar or wagon loader or unloader. The risk assessment should include factors such as lighting, communications, hi-visibility clothing, etc. When extracting increments from a working face, the surface shall be sufficiently compacted to bear safely the weight of personnel and equipment.

If it is not possible to reach all of the coal or coke in the hold, the sample may be seriously biased. Sampling the top surface of the coal or coke in barges, trucks, railcars or wagons immediately after these are loaded is permitted, provided it has been confirmed that loading by layering of coal or coke of different qualities did not take place.

A manual sampling shovel or scoop shall be used to extract increments. The width of the sampling shovel or scoop shall be at least three times the nominal top size of the coal or coke, with a minimum dimension of 30 mm (see 6.2.2 and 6.2.3) and larger, as necessary, to ensure that the larger particles are not excluded from the increment.

Increments shall be spaced as evenly as possible over the surface. It is important to note that segregation during handling often results in the accumulation of lumps, e.g. near one or more walls of the hold depending on the handling system.

When extracting increments, the manual sampling shovel or scoop shall be inserted at right angles to the surface of the coal or coke after the top surface of the coal or coke has been removed. Large pieces of coal or coke shall not be deliberately pushed aside when an increment is extracted, and no portion of the increment shall be lost during its extraction by the sampling shovel or scoop.

5.7 Moisture/common sample

A moisture sample is a sample taken specifically for the purpose of determining total moisture, while a common sample is a sample collected for more than one intended use. A physical sample is a sample taken specifically for the determination of physical characteristics, e.g. physical strength indices or size analysis.

Where a moisture sample is extracted from a common sample, the initial number of increments extracted shall be that required for ash or moisture, whichever is the greater. The number of increments should be increased only if there will not otherwise be sufficient coal or coke left for the ash sample and/or the samples for other intended uses after the removal of the moisture sample. As a consequence, when it is necessary to increase the number of increments, the sampling interval shall be decreased.

There can be circumstances that make it necessary or convenient to collect separate samples for the determination of total moisture and/or for the other intended uses, e.g. a separate moisture sample when the coal or coke is very wet/visibly wet.

The following additional points should be considered when sampling for moisture content:

a) stored coal or coke, washed coal or coke, etc. gradually lose water by drainage until an equilibrium is reached;

b) if free moisture is present in the lot, this migrates towards the bottom so that a steady increase in moisture content occurs as the depth of coal or coke increases;

c) it may be necessary, when collecting samples for moisture determination from lots over an extended period, to limit the standing time for samples.

Under all these conditions, it is highly preferable that increments be extracted from sub-lots representing the different levels or restricted time periods.

5.8 Different coals or cokes

If a lot is known to consist of coal or coke of different qualities piled/located in separate areas of the total lot, a separate sample shall be taken, prepared and analysed for each of these areas (covered by separate sampling schemes) and the sampling scheme established according to the most variable coal or coke. Each area shall consist of at least one sub-lot. The number of sub-lots and increments should be calculated in accordance with 4.3.6.

If insufficient knowledge about the lot is available to sample separately the different coal or coke qualities or sources, or if they are not identifiably separated or stowed separately, the lot should be divided into a number of sub-lots. Each sub-lot shall be prepared and analysed separately, and the values of the characteristics for each sub-lot reported. If necessary, for some parameters that can be averaged e.g. proximate analysis and calorific value, the values of the characteristics for the whole lot can be obtained by taking a weighted average of the sub-lot values. For characteristics that cannot be averaged, a weighted composite sample shall be formed for analysis, e.g. ash fusion properties.

It is preferable to separately sample and analyse different quality coal or coke, or coal or coke from different sources, just before mixing takes place.

5.9 Random selection of increments

All the possible sampling areas (grabs, front-end loaders, barges, barge-holds or parts thereof, trucks, railcars, wagons or stockpiles when sampling from horizontal surfaces) shall be identified and numbered, although sampling of stationary lots may not provide representative samples (see 4.1.1 and 5.1) and this shall be stated in the sampling report. The areas to be sampled are selected by one of the following methods:

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

b) Provide a set of numbered discs that are uniform in dimensions and mass, one disc corresponding to each sampling area, and then proceed as follows:

   1) When selecting grabs, front-end loaders, barges or barge-holds, trucks or railcars (units), place the discs in a bag, mix the contents of the bag, and draw sufficient discs from the bag to coincide with the total number to be sampled. Attach the selected discs to a reference board and sample those units corresponding to the numbers on the selected discs;

   2) When selecting sampling areas within units or on stockpiles (see Figure 1) for sampling from horizontal surfaces, place the discs in a bag close to the sampling point and provide a diagram on a fixed board showing the locations of the areas across the surface of the coal or coke. To sample the first selected unit or the first horizontal layer of the stockpile, draw sufficient discs from the bag to coincide with the total number of increments to be extracted from that unit or that layer and extract an increment from those areas corresponding to the numbers on the selected discs. Place these discs in a second bag after use. For the second unit or layer, follow the same procedure by drawing discs from those remaining in the first bag. Continue this

process for subsequent units or layers until all the discs are used up and then swap the bags over so that discs are drawn from the second bag and placed in the first bag;

This procedure can also be used for selecting the units to be sampled, when some are sampled and some are not. For example, suppose 50 units are to be sampled out of a lot of 100. A set of discs numbered 1 to 100 is placed in a bag and the sampling operator draws from the bag 50 numbered discs in succession. The selected discs may be hung on hooks on a reference board and in case of a series of different units (e.g. railcars), the units may be numbered serially with chalk as they pass. In case of sampling from grabs, the total number of grab volumes should be estimated, and each grab shall be counted. The units corresponding to the numbers drawn should be sampled.

1	4	7	10	13	16	19	22
2	5	8	11	14	17	20	23
3	6	9	12	15	18	21	24

Figure 1 — Example of sampling areas within a unit or a horizontal layer of a stockpile

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

6 Sampling equipment

6.1 General

Clause 6 describes equipment (devices/tools) for the extraction of increments and equipment for dividing samples. This equipment shall be such that the sample obtained is representative and it should fulfil the following requirements:

a) the width, depth and height as appropriate of the sampling device, which determines the minimum mass of increment, shall satisfy the condition given in Formula (18), subject to a minimum dimension of 30 mm;

$$W \geq 3d \tag{18}$$

where

W is the minimum width, depth and height of the sampling device, expressed in millimetres;

d is the nominal top size of the coal or coke, expressed in millimetres.

b) the capacity of the device shall be such that, during the extraction of a single increment, it is not overfilled, and it can contain at least the required minimum mass (see 4.3.6.3);

c) if the device is being used for falling streams, the length of the entry aperture shall be such as to ensure that the whole width of the stream is intercepted;

d) large and hard pieces of coal, coke or rock or shale shall not be pushed aside during the extraction of increments;

e) none of the coal or coke shall be lost from the device during the extraction and handling of the increment;

f) wet coal or coke adhering to the devices should be minimized.

Examples of equipment for extracting increments are ladles, shovels/scoops, probes, manual cutters, and sampling frames (see 6.2 and Figure 2).

6.2 Examples

6.2.1 Ladles

A ladle [see Figure 2 a)] is a box or container or slotted device for extracting increments from falling streams of coal or coke. The dimensions of the ladle shall be such that, during the extraction of a single increment, it contains at least the complete increment of minimum mass (see 6.1). Ladles are not suitable for sampling in any operation where overflow of the sampling device occurs.

Ladles should be made of materials that avoid sample build-up, corrosion and contamination.

6.2.2 Shovels

A suitable design of a shovel is shown diagrammatically in Figure 2 b). The dimensions of the shovel shall be such that the width shall be at least three times the nominal top size of the coal or coke being sampled, with a minimum dimension of 30 mm. Shovels shall be constructed from durable and light weight material that avoids sample build-up, corrosion and contamination, and should be fitted with a handle of appropriate length for the safety of sampling personnel.

6.2.3 Scoops

A suitable design of a scoop is shown diagrammatically in Figure 2 c). The dimensions of the scoop shall be such that the width shall be at least three times the nominal top size of the coal or coke being sampled, with a minimum dimension of 30 mm. Scoops shall be constructed from durable and light weight material that avoids sample build-up, corrosion and contamination, and should be fitted with a handle of appropriate length for the safety of sampling personnel.

The scoop used for the flattened heap division method (see Figure 7) shall be flat bottomed and the width shall again be at least three times the nominal top size of the coal or coke being sampled. The side walls shall be higher than the height of the heap and the depth shall be sufficient to allow the desired mass of increment to be extracted using a suitable bump plate.

The scoop can be either flat bottomed or curved. If curved, ensure the minimum depth dimension requirements are achieved at all points on the curve. The scoop can be expanded with a handle.

6.2.4 Probes

Probes take the form of tubes constructed from durable materials that avoid sample build-up, corrosion and contamination, which are inserted vertically or at a slight angle into the coal or coke. Insertion into the coal or coke is sometimes difficult and the tube tends to empty when it is withdrawn. The dimensions of the probe shall be such that the inner diameter shall be at least three times the nominal top size of the coal or coke being sampled, with a minimum dimension of 30 mm.

Probes are used for sampling coal or coke of up to 25 mm nominal top size. An example of a probe is shown in Figure 2 d).

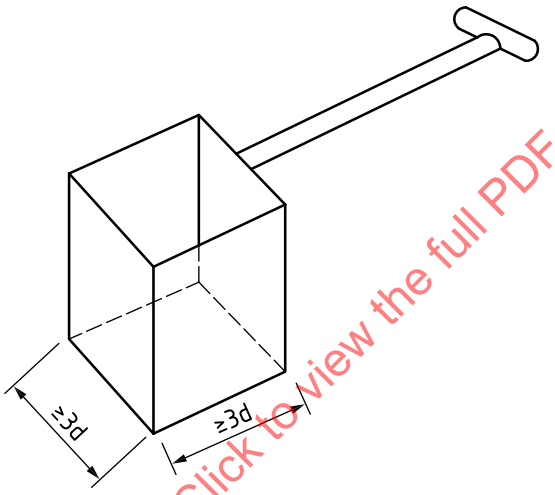
6.2.5 Manual cutter

A manual cutter [see Figure 2 e)] is a sampling device that can be moved through a falling stream, manually or with mechanical assistance. Manual cutters are not suitable for sampling in any operation where overflow of the cutter occurs. The dimensions of the cutter aperture shall be at least three times the nominal top size of the coal or coke being sampled, with a minimum dimension of 30 mm.

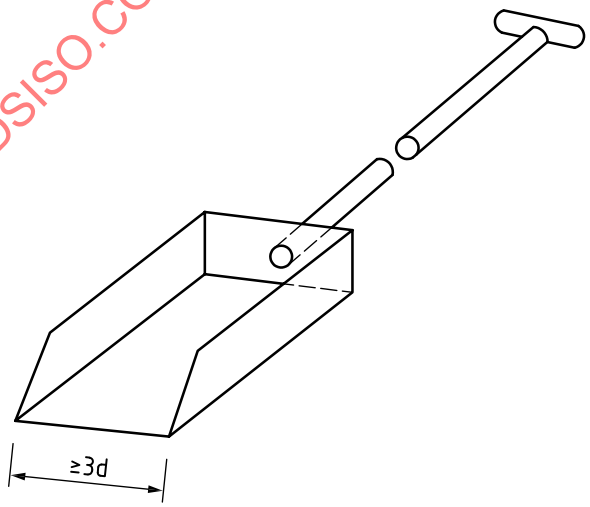
6.2.6 Stopped-belt sampling frame

A stopped-belt sampling frame [see Figure 2 f)] is a parallel-sided frame of robust construction used for extracting a stopped-belt increment or for strip mixing and splitting increments. The width of the frame shall be at least three times the nominal top size of the coal or coke being sampled, with a minimum dimension of 150 mm. The frame shall be inserted into the coal or coke until it is in contact with the belt across its full width and the increment is extracted by sweeping off the whole of the coal or coke lying between the sides of the frame.

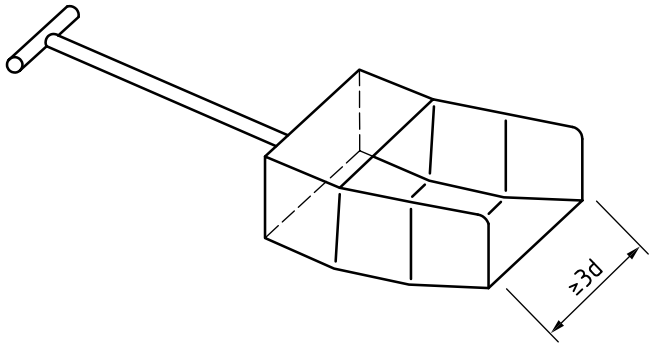
Dimensions in millimetres, unless otherwise specified



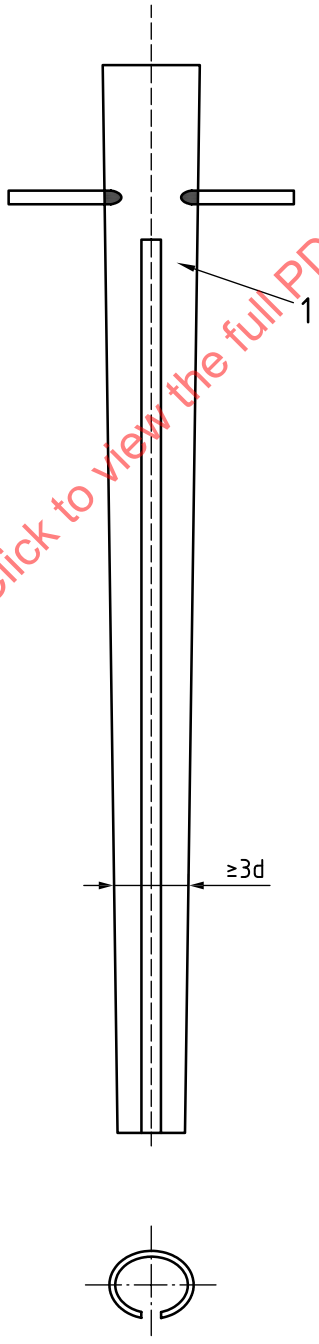
a) Example of ladle suitable for sampling coal or coke with a width and breadth of at least three times the nominal top size of the coal or coke being sampled, subject to a minimum of 30 mm



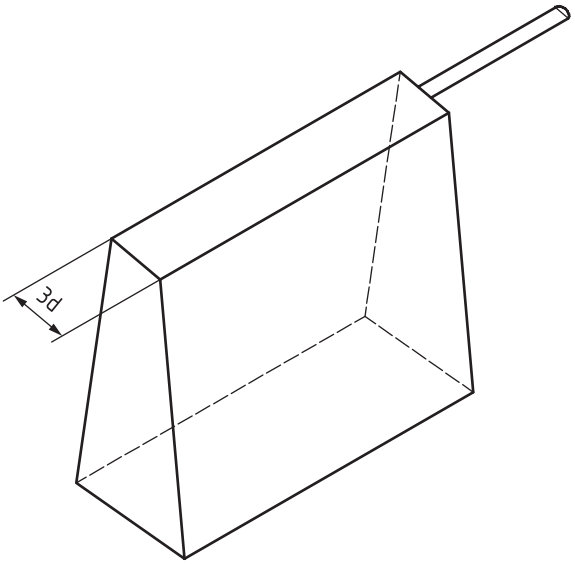
b) Example of a shovel suitable for sampling coal or coke with a width of at least three times the nominal top size of the coal or coke being sampled, subject to a minimum of 30 mm



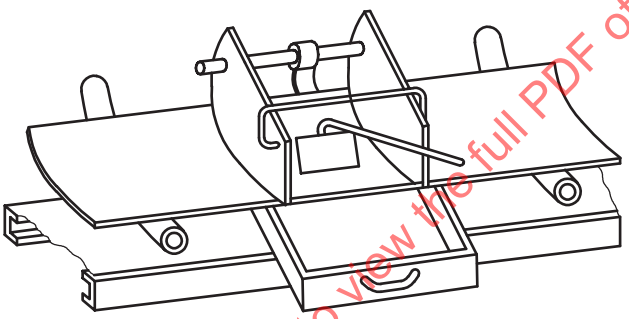
c) Example of a scoop suitable for sampling coal or coke with a width of at least three times the nominal top size of the coal or coke being sampled, subject to a minimum of 30 mm



d) Example of a probe suitable for sampling coal or coke with minimum internal diameter of 30 mm



e) Manual cutter



f) Sampling frame

Key

1 slightly tapered tube

d nominal top size of the coal or coke

Figure 2 — Examples of sampling equipment

NOTE Equipment shown in Figure 2 are examples. Other equipment that meets the criteria in 6.2 also meet the standard.

7 Handling and storage of samples

7.1 Sample size

The size of the samples to be handled and stored has to conform to the minimum masses and relevant nominal top sizes given in this document (see Tables 1 and 2 and ISO 1953).

7.2 Time

Increments or divided increments shall be placed as quickly as possible into sample containers with tight-fitting lids and the lids shall be immediately replaced after each increment has been inserted.

Test samples shall be kept available under good custody for an agreed period after issuance of the formal final sampling report (see Clause 10). Certain types of coal or coke do not allow a long storage time.

7.3 Divided sample

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.

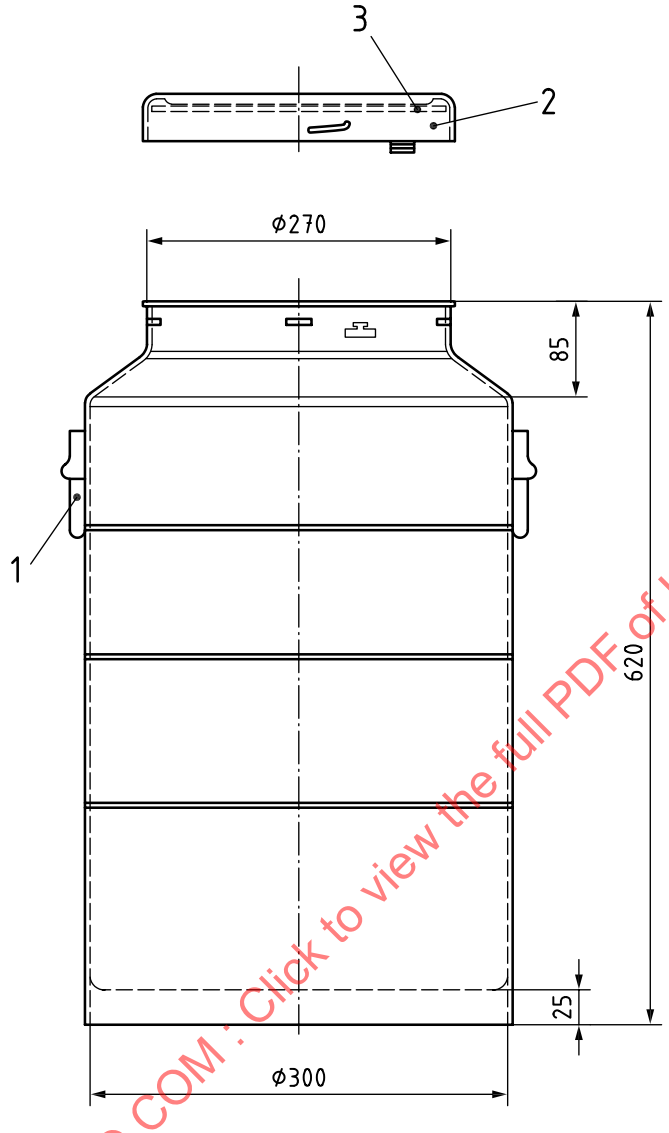
7.4 Containers

Containers (refer to Figure 3 for an example) used for moisture or common samples should be watertight and made of impermeable non-corrodible material, of adequate strength and, where applicable, with well-fitting lids. Containers made from metal or plastic material have been found suitable.

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 is not impaired during removal and transport.

If general test samples are required, e.g. size samples, the containers for such samples shall give adequate protection against contamination and loss of sample material, but they are not required to be fully impervious to water and vapour.

Dimensions in millimetres



Key

1 handle
2 lid
3 rubber insert

Figure 3 — Example of a sample container

7.5 Moisture loss/breakage or degradation

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

Common samples and moisture samples shall be kept in a cool place during any storage, preferably at a temperature that is not above that of the sample when it was taken, and the moisture shall be determined as quickly as possible after taking the sample.

7.6 Identification/labelling

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

It is recommended that for this purpose the container be provided with two waterproof tags, each marked by means of waterproof ink with adequate identifying information, one tag being placed on the outside of the container and one being placed inside the container; if a plastic inner liner is used, the latter tag should be placed inside this liner. It is recommended that digital methods, e.g. bar coding, also be used to identify samples at all levels of preparation from sub-lot sample to total moisture and analysis samples for testing.

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

It is recommended that the information described in Clause 9 be shown on the label/tag or accompanying documents.

8 Sample preparation

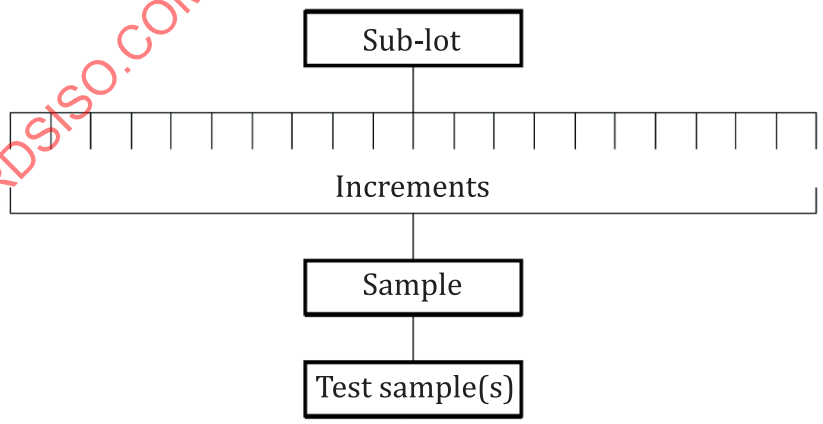
8.1 General

Clause 8 describes the preparation of samples of coal or coke from the combination of primary increments to the preparation of samples for specific tests. For the precision of sample preparation, see 4.3.5.

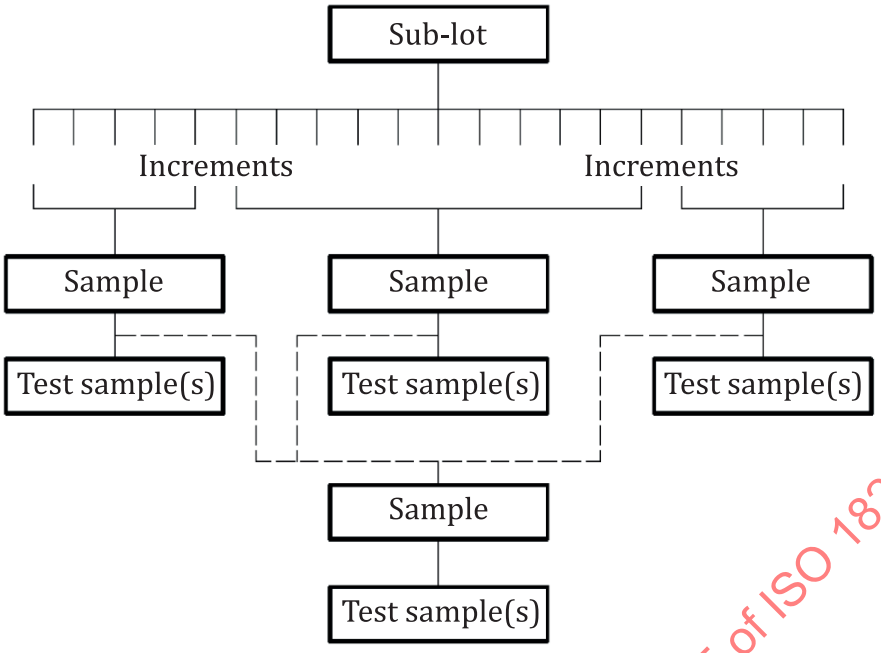
8.2 Constitution of a sample

Examples of the constitution of samples are shown in Figure 4.

Individual increments are usually combined to form a sample. A single sample may be constituted by combination of increments taken from a complete sub-lot or by combining increments taken from individual parts of a sub-lot. Under some circumstances, e.g. stopped belt sampling, the sample consists of a single increment, which is prepared and tested.



a) Example 1



b) Example 2

Figure 4 — Examples of the constitution of test samples

When combining samples, the mass of the individual samples shall be directly proportional to the mass of the coal or coke from which they were taken to obtain a weighted mean of the quality characteristic for the sub-lot. Prior to combination, division shall be by fixed-ratio division (see 8.3).

8.3 Division

8.3.1 General

Division can be carried out mechanically or manually. Whenever possible, mechanical methods are preferred to manual methods to minimize human error. Examples of dividers are rotary dividers and riffles shown in Figures 5 a) to d) and Figures 6 a) to c).

Mechanical dividers are designed to extract one or more parts of the coal or coke in a number of cuts of relatively small mass. When the smallest mass of the divided sample that can be obtained in one pass through the divider is greater than that required, further passes through the same divider or subsequent passes through further dividers is necessary.

Coal or coke that is visibly wet might not run freely through or might tend to adhere to the surfaces of a sample divider. In such circumstances, it may be necessary to air-dry the sample as described in 8.6 before sample division is undertaken.

Manual division is normally applied when mechanical methods would result in loss of integrity, e.g. loss of moisture or size degradation. Manual division is also applied when the nominal top size of the coal or coke is such as to make the use of a mechanical divider impracticable. Manual methods can themselves result in bias, particularly if the mass of coal or coke to be divided is large.

8.3.2 Mechanical methods

8.3.2.1 General

Mechanical sample division may be carried out on an individual increment, multiple increments or a sample, that has been crushed, if necessary, to an appropriate nominal top size. Division shall be either by fixed-mass division or by fixed-ratio division.

The uses to which the sample shall be put and the numbers, masses and size distribution of the test samples need to be considered when deciding on the minimum mass of the sample.

NOTE The procedures described for fixed-ratio division are the simplest to implement. Other procedures can be used, however, provided that the mass of the divided sample is proportional to the mass of the feed. For example, the number of cuts can be kept constant by making the feed rate of each division proportional to the mass of coal or coke to be divided.

The cuts shall be of uniform mass throughout division. In order to achieve this, the flow of coal or coke to the divider shall be uniform and the cutting aperture shall be constant. The method of feeding the divider shall be designed to minimize any segregation caused by the divider.

The cutting aperture shall be at least three times the nominal top size of the coal or coke to be divided.

Division devices shall

a) have sufficient capacity to retain completely or to pass the entire sample without loss or spillage,

b) not introduce bias, for example by selective collection (or rejection) on the basis of particle size or by loss of moisture. It is recommended that they be totally enclosed or as airtight as possible,

c) use a method of feeding that minimizes the segregation of the coal or coke, and

d) provide a controlled uniform flow to the equipment at each stage of division.

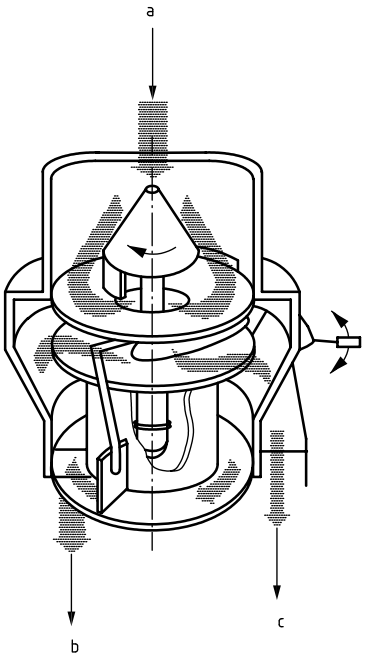
In order to minimize bias, the first cut for each mass to be divided shall be made at random within the first cutting interval. For secondary and tertiary dividers, the cycle time shall not be evenly divisible into the cycle time of the cutter that precedes it.

For fixed-mass division, the interval between taking cuts shall be varied proportionally to the mass of coal or coke to be divided, so that divided samples having almost uniform mass are obtained. The mass shall be fixed for the whole sub-lot.

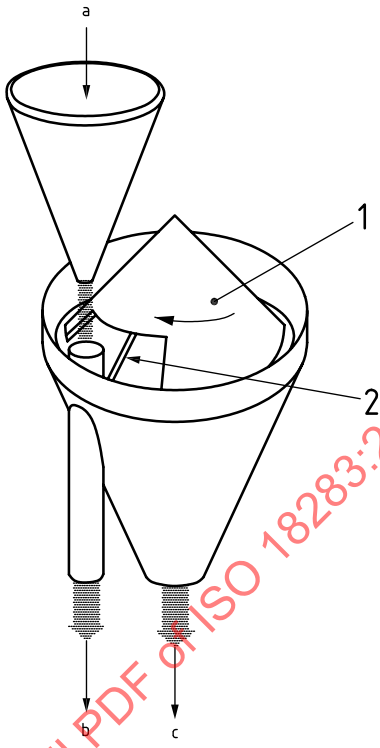
For fixed-ratio division, the interval between taking cuts shall be constant irrespective of the variations of masses of coal or coke to be divided, so that the divided sample masses are proportional to the mass of the feed. The ratio shall be fixed for the whole sub-lot.

8.3.2.2 Rotary dividers

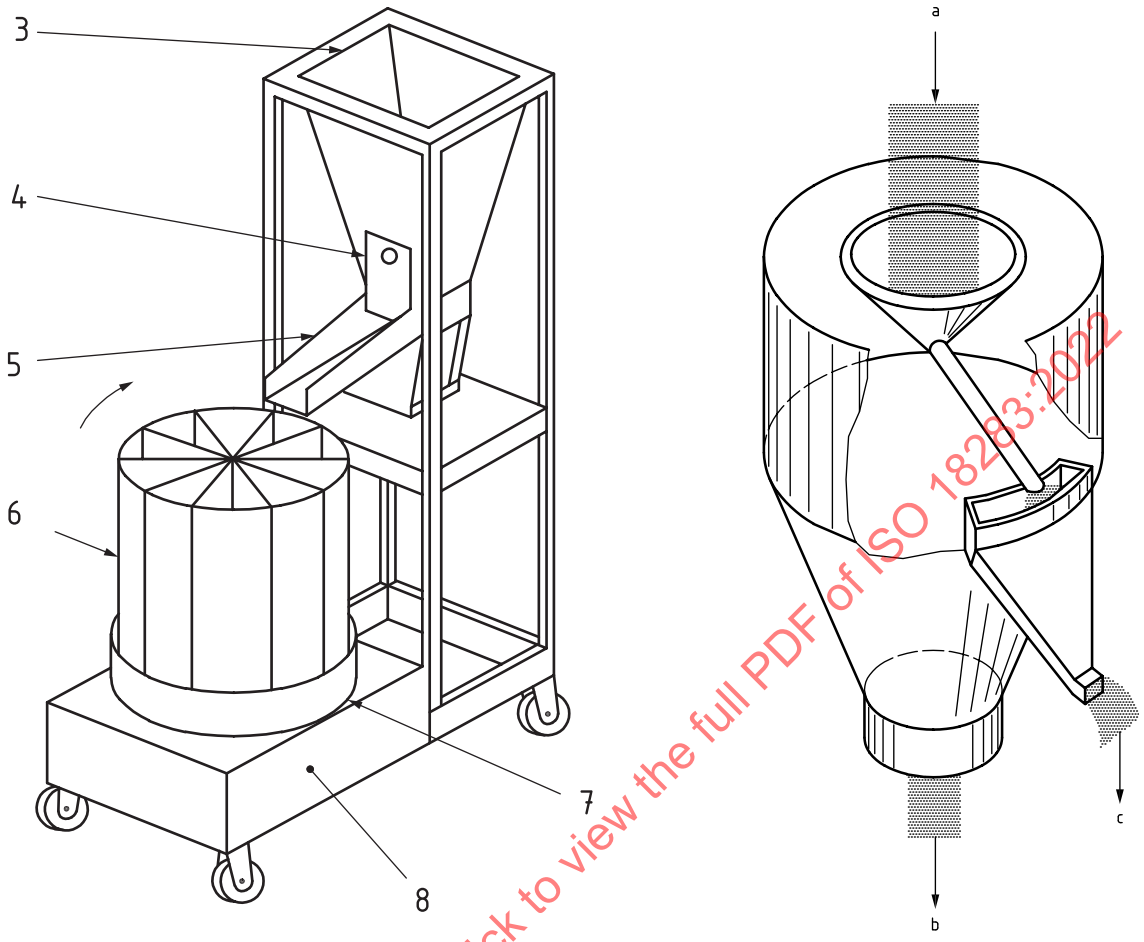
Rotary dividers are suitable for mechanical sample division. Examples of typical rotary type dividers are shown in Figures 5 a) to d).



a) Rotating disk type



b) Rotating cone type



c) Container type **d) Rotating chute type**

Key

1 rotation cone
2 adjustable slot
3 feed hopper
4 slide gate
5 vibratory feeder
6 removable canisters
7 turntable
8 drive (enclosed)
a Feed.
b Reject.
c Divided sample.

Figure 5 — Examples of rotary type dividers

8.3.2.3 Division of samples

The sample constituted from all increments or divided increments shall be divided by taking a minimum of 60 cuts.

If, during preparation, it can be established that the required precision can be achieved, the number of cuts can be reduced to 20. If the mass is too low, an alternative manual method of division should be used.

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 adequately represent all the particle sizes in the mass of coal or coke being sampled. The attainment of the required minimum mass after division does not, of itself, guarantee the required precision, because division precision is also dependent on the number of cuts taken during division.

8.3.3 Manual methods

8.3.3.1 Riffle method

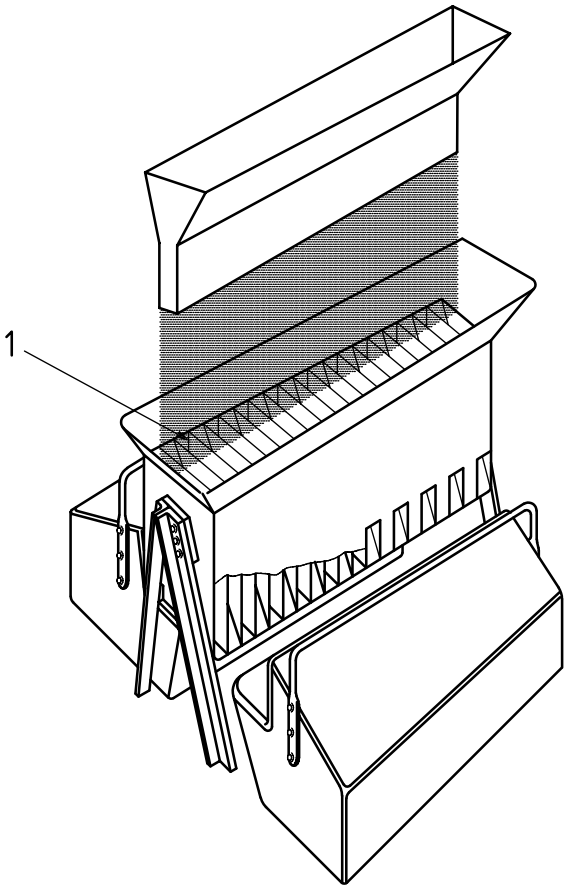
A riffle is a manual sample divider that, in a single pass of a sample, divides it into halves, one of which is retained and the other normally is rejected. The device is normally portable and, for sample division, is usually fed manually, the coal or coke being evenly distributed along its length. Adjacent slots feed opposite receivers. Examples of riffles are shown in Figures 6 a) to b). Riffle dividers fitted with a feed gate enable even placement of the feed before opening the gate [see Figure 6 c)].

The slot width shall be at least three times the nominal top size of the coal or coke. Each half of the riffle shall have the same number of equally sized slots. The minimum number of slots on each side of the riffle shall be eight, corresponding to a total of 16 slots in total. All the surfaces on which the coal or coke might rest shall have a slope of at least 60° to the horizontal.

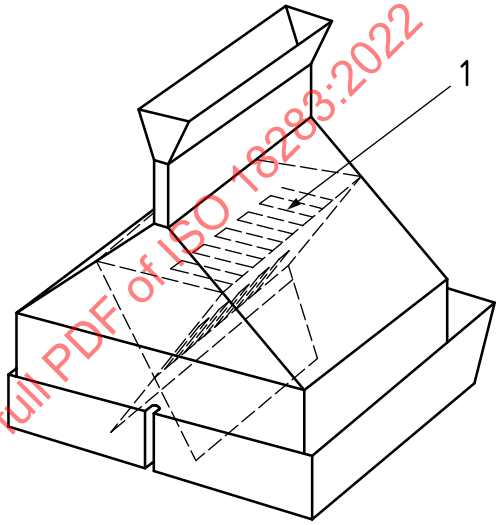
The coal or coke shall be allowed to fall freely into the riffle, ensuring that it is evenly distributed over all the slots and not towards one side of the riffle, and the rate of feed shall be controlled such that the slots are never choked.

Care shall be taken to minimize loss of dust and moisture. To this end, the receiver shall fit closely against the body of the riffle, and, for dry coal and coke and moisture samples, closed-type riffles shall be used.

When a stage of sample division requires two or more steps or passes, the sample retained at each step shall be taken alternately from each side of the riffle.



a) Open type



b) Closed type

c) Gated riffle

Key

1 even number of slots

Figure 6 — Examples of riffles

8.3.3.2 Flattened-heap method

The flattened-heap procedure, which is illustrated in Figure 7, is as follows.

a) The sample is mixed thoroughly and spread to form a rectangle of uniform thickness on a mixing plate, which is a smooth, non-absorbent, non-contaminating surface. The maximum thickness shall be three times the nominal top size of the coal or coke. If the mass of the coal or coke is greater than can be formed into a heap of 2 m × 2,5 m, two or more heaps of equal mass shall be formed and separate samples taken from each heap.

For wet coal or coke, thorough mixing, which might result in loss of moisture, shall be avoided.

b) A matrix is marked on the spread sample to give a minimum of 4 × 5 equal parts. An increment is taken, at random, from each of the parts by inserting a scoop vertically with a bump plate to the bottom of the matrix layer. The increments are combined into a divided sample.

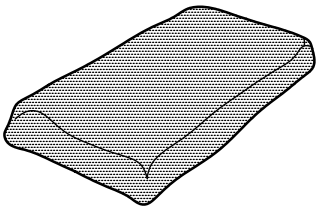
The increments shall be of uniform mass. The minimum mass required for each nominal top size is the mass of the divided sample (see Tables 1, 2 and 4) divided by the number of parts of the flattened heap.

The scoop shall be flat bottomed and the width shall be at least three times the nominal top size of the coal or coke. The side walls shall be higher than the height of the heap and the depth shall be sufficient to allow the required mass of increments to be taken.

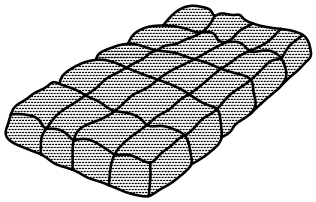
It is essential that these operations be performed quickly if loss of moisture is to be prevented.

Increments are taken with the aid of a bump plate, which is inserted vertically through the flattened heap until it is in contact with the bottom of the sample layer. The scoop is then inserted vertically to

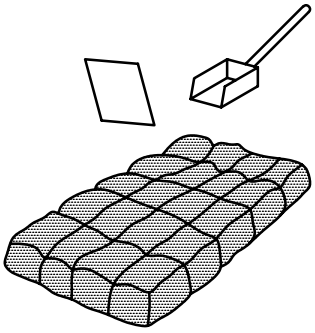
the bottom of the spread coal or coke and moved horizontally until its open end comes into contact with the vertical bump plate. The scoop and bump plate are lifted together to ensure that all particles are collected off the top of the mixing plate and that none fall off during lifting



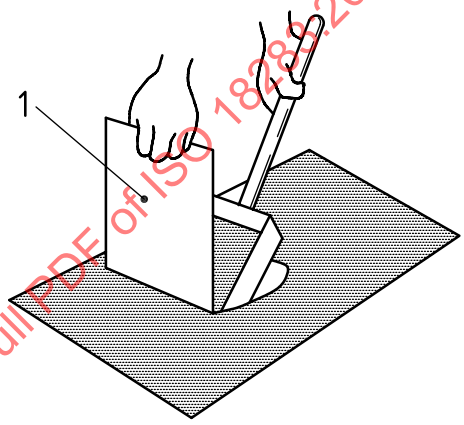
a) Spread the crushed sample into a rectangle with a maximum thickness three times the nominal top size



b) Arrange into 20 equal parts, e.g. into five equal parts lengthwise and four equal parts breadthwise



c) Take a scoopful of samples at random from each of the 20 parts by inserting the scoop to the bottom of the sample layer. Combine the 20 scoopfuls into a divided sample



d) Detail of taking an increment by using the bump plate shown in c)

Key

1 bump plate

Figure 7 — Flattened-heap method

8.3.3.3 Strip-mixing and splitting method

The strip-mixing and splitting procedure, which is illustrated in Figure 8, is as follows.

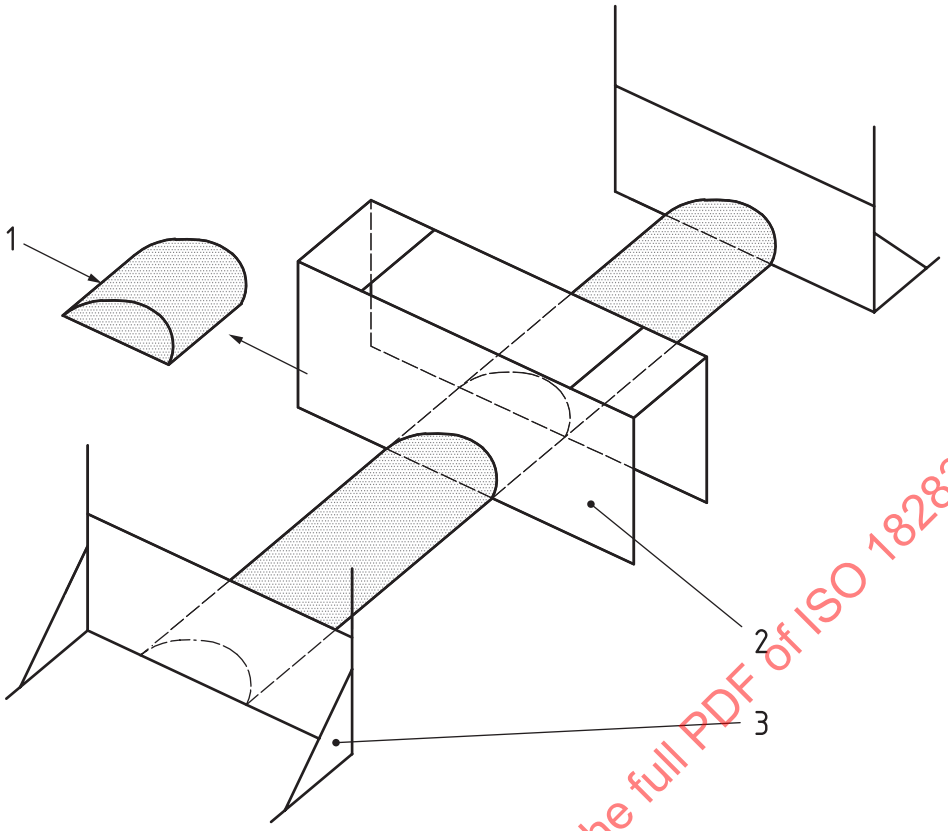
a) The coal or coke sample is formed on a mixing plate, which is a smooth, non-absorbent, non-contaminating surface, into a strip at least 10 times as long as it is wide by distributing the coal or coke along the length of the strip as evenly as possible, working randomly from end to end and from both sides of the strip. End plates are used to ensure that size segregation only occurs laterally,

b) Increments are taken systematically along the length of the strip as complete sections across the strip. The width of each cross-section shall be not less than three times the nominal top size of the coal or coke.

A special apparatus for the cutting-out of increments may be constructed if desired.

Normally 20 increments are required. Fewer increments may be taken, subject to a minimum of 10, where the same quality coal or coke is regularly prepared under the same conditions and it has first been established that the required precision can be obtained (see 4.4).

NOTE Because of the efficient longitudinal mixing achieved in the formation of a strip, the same precision as that obtainable with the flattened heap method can be achieved with fewer increments.



Key

1 increment
2 sampling frame
3 end plates (“book ends”)

Figure 8 — Strip-mixing and splitting method and device illustrating the extraction of one of the required number of increments

8.4 Reduction

8.4.1 General

Mechanical equipment shall be used to reduce the particle size, but manual crushing is permitted for the breakage of large material to meet the maximum feed size acceptable to the first-stage mill.

The test sample shall be reduced to the particle size specified in the relevant test method.

The mill settings shall be checked regularly by sieving and determining the nominal top size produced by each mill.

8.4.2 Reduction mills

The particle size produced depends on the speed of the mill and its design. Mills shall be designed such that the required particle size of the reduced sample can be achieved without using extreme settings. Loss of sample or retention of material from previous samples that might contaminate succeeding samples shall be minimized. Heating of the sample and air-stream effects shall be minimized, particularly where the sample is used for the determination of total moisture, calorific value and for coking tests.

There shall be no contact between the metal surfaces of the mill in order to avoid localized heating of the sample, except for ring grinders used for crushing to 212 µm nominal top size for determination of ash. Totally closed, high-speed (>20 Hz) ball mills shall not be used. The particle size of the output is influenced by the hardness of the coal or coke, but the effect depends on the speed range.

For certain tests, specific size grades are required, and the type of mill shall be chosen to ensure that the required size is obtained.

Hammer mills, jaw crushers and roll mills are most suitable for crushing coal during preparation, taking care to minimize heating. Coal samples for general analysis are milled to pass 212 µm top size using a small high-speed hammer mill with a screen. Ring grinders are acceptable for crushing coal samples for determination of ash content and other parameters not sensitive to heating, but are not suitable for preparation of samples for determining swelling index or dilatation. Suitable mills for crushing coke during preparation are jaw crushers, roll mills and ring grinders.

8.5 Mixing

In theory, thorough mixing of a sample prior to its division reduces errors due to sample preparation. In practice, this is not easy to achieve and some methods of hand mixing, e.g. forming and reforming into a conical pile, can have the opposite effect, leading to increased segregation. Mixing can also result in loss of moisture.

One method that can be used is to pour the sample through a riffle [see Figures 6 a) and 6 b)] or a container-type sample divider [see Figure 5 c)] three times, reuniting the parts after each pass. If mechanical sample dividers are used in the course of preparation, an additional mixing step is not normally necessary to meet the required precision.

NOTE Mechanical mixing can be useful at the final stage of preparation of test samples.

8.6 Air-drying of coal

The procedure for air-drying of coal is as follows.

a) Determine the mass of the whole of the sample in its container on a balance which can be read to an accuracy of at least 0,1 %.

b) Spread the sample in a thin layer that shall not exceed a thickness of 1,5 times the nominal top size of the coal on a dry flat smooth surface in a warm, well-ventilated room and allow the sample to attain equilibrium with the atmosphere at ambient temperature.

The recommended times to attain equilibrium at different ambient temperatures up to 40 °C are given in Table 4. The times recommended in Table 4 will normally be sufficient; but, if necessary, a longer drying time may be used, provided that any increase is kept to a minimum, especially for coal susceptible to oxidation.

Table 4 — Recommended drying times for air-drying of coal

Drying temperature °C	Drying time h
20	Preferably ≤24
30	Preferably ≤6
40	Preferably ≤4

Drying temperatures above 40 °C shall not be used on samples likely to be susceptible to oxidation or if the sample is used for any of the following tests:

a) calorific value;

b) caking properties;

c) swelling properties;

d) air-drying as part of a determination of total moisture content.

For drying temperatures above normal ambient temperature, a cabinet or oven with appropriate air-change facilities shall be used. If drying has been carried out at such temperatures, the sample shall be cooled until moisture equilibrium at normal ambient temperature is achieved before reweighing. The cooling period required depends on the drying temperature. For example, for high rank coals, 3 h is normally sufficient if the sample has been dried at 40 °C. If the change in mass of the sample over a period of 1 h is less than 0,1 % of its original mass, the sample is considered as air-dried. However, for low rank coals, it is quite difficult to achieve a change of less than 0,1 % of its original mass by drying at 40 °C for 4 h. In this case, to avoid heating the sample beyond 4 h and possibly causing oxidation of the sample, it is recommended that the sample be considered air-dried after 4 h drying at 40 °C.

Dry and determine the mass of the container. Collect the air-dried sample in the original container, redetermine the mass and calculate the percentage loss in mass. Continue the preparation for other tests.

When air-drying is used, the percentage loss of moisture in this operation shall be recorded on the label with a reference to the method of sampling and preparation used.

After the moisture of the partially dried sample has been determined as described in 8.7.2, calculate the total moisture content, M_T, as a percentage, from Formula (19):

$$M_T = X + M\left(1 - \frac{X}{100}\right) \tag{19}$$

where

X is the loss on partial drying, expressed as a percentage;

M is the moisture content, determined as described in 8.7.2 and expressed as a percentage; and

100 is the conversion factor from dimensionless mass fraction to percent, %.

8.7 Coal — Preparation of test samples

8.7.1 Types of test samples

The following types of test samples may be prepared:

a) samples for determination of total moisture;

b) samples for general analysis (i.e. not to be used for determining total moisture);

c) common samples for both total moisture and for general analysis;

d) samples for size analysis;

e) samples for other tests, e.g. determination of the Hardgrove grindability index.

The methods of preparation depend on the purpose for which the original sample was collected.

8.7.2 Preparation of samples for determination of total moisture content

The test sample for the determination of total moisture content shall be prepared to meet the requirements of ISO 589. If air-drying is performed at any stage of preparation, the percent loss in mass is recorded and included in the calculation of total moisture, as specified in ISO 589.

A major problem with the preparation of test samples for the determination of moisture content is the risk of bias due to inadvertent loss of moisture. The amount of this loss is dependent on such factors as

the effectiveness of the sealing of the sample containers, the level of moisture content of the sample, the ambient conditions, the type and rank of coal and the reduction and division procedures used.

Precautions shall be taken to minimize changes in moisture content during preparation and due to the use of unsuitable containers. All samples for moisture content determination shall be kept in sealed containers in a cool place, under cover, before and during preparation as well as during any interval between steps of sample preparation. If excessive standing time causes bias, the number of sub-lots should be increased to overcome this problem (see 4.3.6).

Preliminary air-drying using the procedure described in 8.6 may be necessary in order to minimize moisture loss in any subsequent reduction/division stages. When carrying out division prior to air-drying of a sample or increment, take care to minimize change in moisture content. To this end, all divisions shall be carried out as quickly as possible and mechanically operated dividers with limited ingress of air shall be used.

NOTE For coals that are too moist to flow through a sample divider and for which it is also impossible to air-dry the entire sample, it can be necessary to divide the sample by collecting increments from a flattened heap (8.3.3.2) or by strip-mixing and splitting (8.3.3.3). This divided sample is then air-dried.

It is recommended that the mass of samples stored for moisture content determination should be recorded before storage to allow determination of any moisture loss that takes place during storage.

If the coal is so wet that water separates from the coal in the sample container, the whole of the sample and the container shall be air-dried and the loss in mass recorded and included in the calculation of total moisture content as specified in ISO 589.

If the particle size of the sample is so large that the mass given in Table 1 (see 4.3.7) makes its air-drying impracticable, the sample shall be crushed and divided before air-drying. Crushing shall be kept to the minimum necessary to allow division to a manageable mass, but in any event not less than 2,8 mm nominal top size.

Care shall be taken to minimize changes in moisture during reduction by using totally enclosed equipment in which there is no appreciable heating and by reducing to a minimum the flow of air through the mill.

The preparation process shall be tested for relevant bias using the procedures given in ISO 13909-8, by comparison with the method of drying samples without reduction.

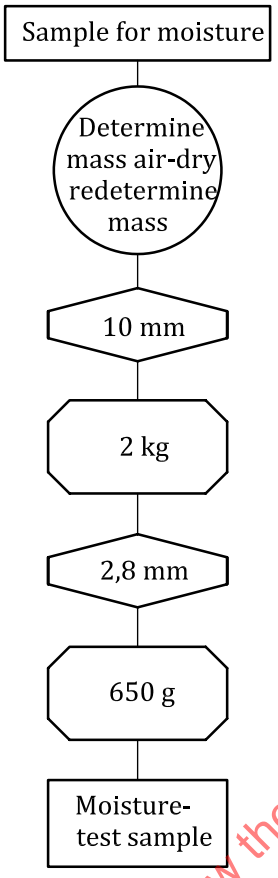
An example of a scheme for the preparation of a sample for a two-stage moisture test is given in Figure 9.

8.7.3 Preparation of samples for general analysis

The objective of general analysis sample preparation is to prepare a test sample that passes a sieve of nominally 212 µm size of openings conforming to the requirements of ISO 3310-1. The mass of the test sample depends on the analysis required, but is typically between 60 g and 300 g.

Sample preparation is normally carried out in two or three stages, each consisting of drying (if necessary), size reduction, mixing (if necessary) and division.

Air-drying (see 8.6) in connection with preparation for general analysis is carried out only to ensure that the coal can pass freely through the equipment. Loss of moisture during the preparation is of no relevance and consequently it is not necessary to measure the loss of mass.



Key

reduce to particle size specified

divide to mass specified

Figure 9 — Example of a two-stage moisture test sample preparation for coal

Air-drying may be carried out at any stage, provided that it does not affect the quality of the sample. For example, if the sample is used for the determination of calorific value, the maximum drying temperature shall be 40 °C. If drying can be avoided during the first stage of preparation, the procedure can be simplified.

Reduction and/or division of increments takes place in accordance with the requirements of 8.3 and 8.4 down to a nominal top size of 2,8 mm prior to their combination to form samples.

NOTE 1 If the coal is wet, it might not be possible to crush it so finely, because of blocking of chutes, dividers, mills, feeders, etc.

If possible, reduce the coal to a nominal top size of 2,8 mm in the first stage in order to minimize the mass of sample retained for the next stage as well as to minimize potential errors due to sample division.

NOTE 2 It might be necessary to use a stamp or a maul to break oversize particles to the maximum feed size of the crushing device.

If the original nominal top size of the coal is too large, or if the coal is too wet, an intermediate stage may be required. In this case, the retained sample from the first stage shall be passed through a second mill to reduce the nominal top size to 2,8 mm.

The sample shall be divided by means of a suitable sample divider to the mass corresponding to the nominal top size in accordance with Table 1 (see 4.3.7).

The sample shall be reduced and divided in one or two further stages to the nominal top size and mass required for the test sample and finally thoroughly mixed.

Mechanical or manual division may be used, the former being preferred. For mechanical division, a suitable divider to give 60 g to 300 g of 212 µm nominal top size coal is required. For manual division, a riffle may be used or the sample shall be spread out and 60 g to 300 g taken by hand in not less than 20 increments from various parts of the flattened heap.

An example of a scheme for preparation of a general analysis sample for coal is given in Figure 10.

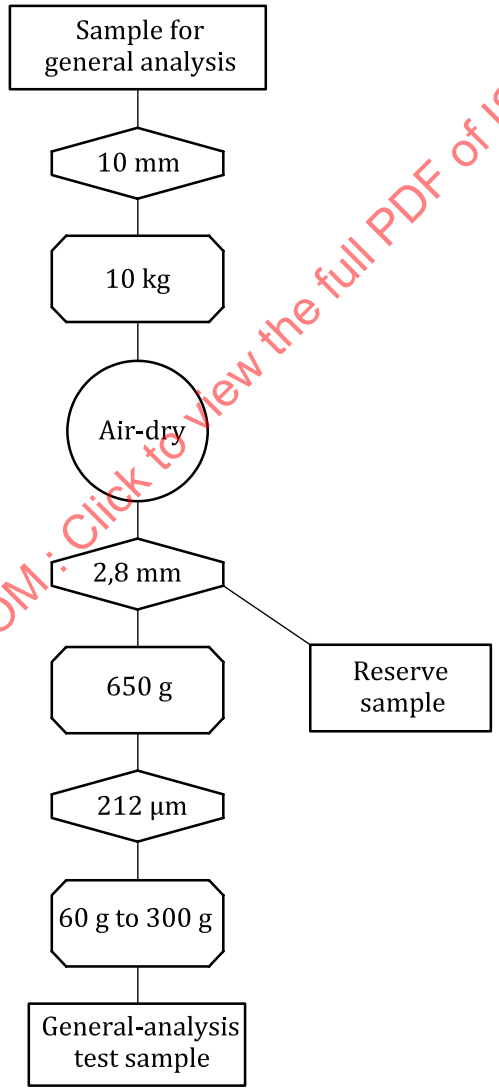


Figure 10 — Example of preparation of a general analysis test sample for coal

8.7.4 Common samples

In some circumstances, it is more convenient to take a common sample for both moisture and general analysis.

It is preferable to extract the moisture sample by use of a mechanically operated divider. The extraction of the moisture sample may be carried out at any convenient stage of the preparation procedure consistent with the requirements of 8.7.2. Prior to extraction, the sample shall be treated in accordance with 8.7.2 in order to avoid any inadvertent loss of moisture. If air-drying is part of the preparation prior to extraction, the loss of mass during the drying shall be measured, recorded and included in the calculation for total moisture content as specified in ISO 589.

If the common sample is visibly wet and it is impossible to air-dry the entire sample, use a manual method. Extract a moisture sample by collecting increments by the flattened heap method (8.3.3.2) or by the strip-mixing and splitting method (8.3.3.3). Avoid further treatment of the moisture sample before air-drying to reduce the risk of bias in the moisture determination. Further treatment after air-drying shall be carried out as described in 8.7.2. The residual coal after extraction constitutes the sample from which the general-analysis sample is prepared and is treated as described in 8.7.3.

Examples of schemes for the preparation of separate test samples for moisture and for general analysis from a common sample are given in Figure 11. Sometimes a single test sample may be prepared for both moisture and general analysis.

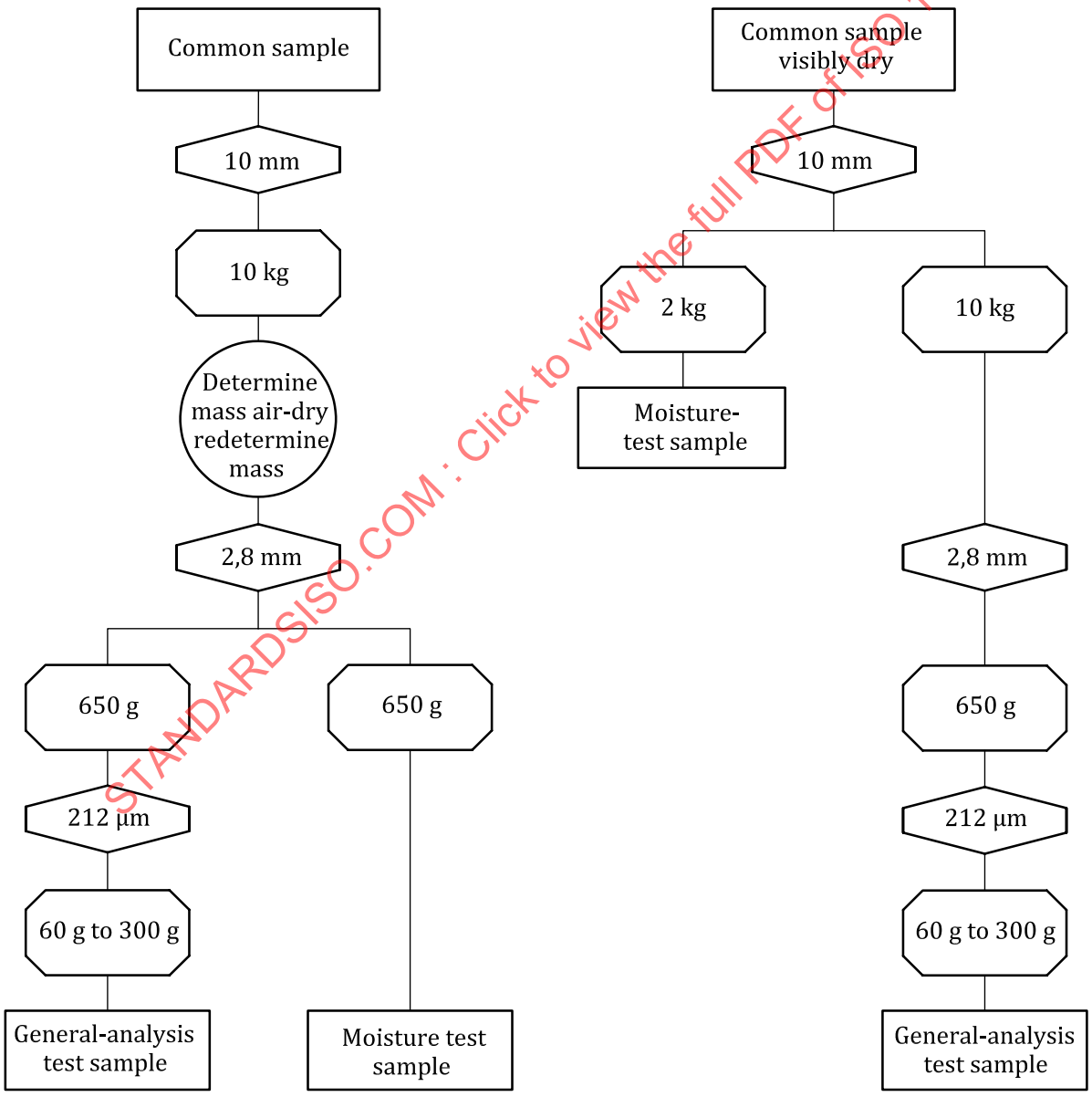


Figure 11 — Examples of preparation of coal test samples from a common sample for moisture determination and general analysis

As a result of the extraction, the common sample has been divided into two parts, one for preparation of the moisture-test sample and one for the preparation of the general-analysis test sample. Each part shall fulfil the requirements for minimum mass specified in Table 1 (see 4.3.7) and further treatment of the parts shall be in accordance with 8.7.2 and 8.7.3, respectively.

8.7.5 Preparation of size-analysis sample

If the mass of the size-analysis sample is more than that specified in ISO 1953 for the appropriate nominal top size, it may be divided to a mass not less than that specified in ISO 1953 provided that the requirements for division (see 8.3) are satisfied

During division, precautions shall be taken to avoid breakage. An example of a scheme for preparation of test samples for size analysis is given in Figure 12.

If the nominal top size of the coal is greater than one-third of the cutting aperture of the sample divider, oversize material may be removed by sieving out and the whole of this oversize portion subjected to size analysis. The undersize coal should be divided to a mass not less than that given in ISO 1953 for the appropriate nominal top size. The divided sample should then be subjected to size analysis and the results combined with those from the oversized coal, weighted according to their relative proportions in the original sample.

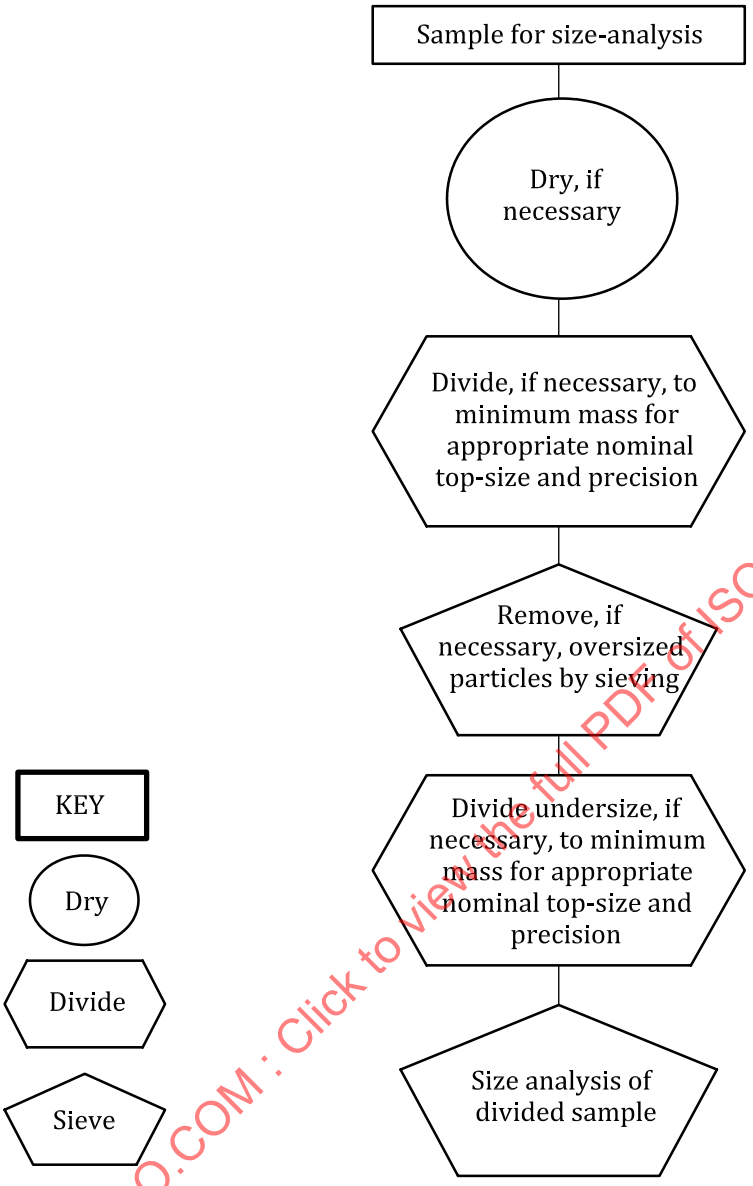


Figure 12 — Example of preparation of coal samples for size analysis

8.7.6 Preparation of samples for other tests

Preparation shall be as described in 8.7.3 or 8.7.4, except that the nominal top size and mass of the test sample shall be as required in the relevant test method. An example of a scheme for preparation of such test samples is given in Figure 13.