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Hard coal and coke — Mechanical sampling —

Part 6:
Coke — Preparation of test samples

Houille et coke — Échantillonnage mécanique —

Partie 6: Coke — Préparation des échantillons pour essai

Reference number
ISO 13909-6:2001(E)

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Foreword

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

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

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

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

International Standard ISO 13909-6 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

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

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

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

Introduction

The objective of sample preparation is to prepare one or more test samples from the primary increments for subsequent analysis. The requisite mass and particle size of the test sample depend on the test to be carried out.

The process of sample preparation may involve constitution of samples, reduction, division, mixing and drying or all or a combination of these.

Primary increments may be prepared individually as test samples or combined to constitute samples either as taken or after having been prepared by reduction and/or division. Samples may either be prepared individually as test samples or combined on a weighted basis to constitute a further sample.

On-line sampling and sample-preparation systems can be usefully implemented when dealing with a coke or cokes which are known to be free from handling problems, but only when this will not result in loss of moisture, contamination by the sampling equipment or size degradation of physical samples.

Hard coal and coke — Mechanical sampling —

Part 6:
Coke — Preparation of test samples

1 Scope

This part of ISO 13909 describes the preparation of samples of coke from the combination of primary increments to the preparation of samples for specific tests.

2 Normative references

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

ISO 579:1999, *Coke — Determination of total moisture*.

ISO 687:1974, *Coke — Determination of moisture in the analysis sample*.

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

ISO 13909-5:2001, *Hard coal and coke — Mechanical sampling — Part 5: Coke — Sampling from moving streams*.

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

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

3 Terms and definitions

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

4 Precision of sample preparation

From the equations given in ISO 13909-7, the estimated absolute value of the precision of the result obtained for the lot at the 95 % confidence level, P_L, for continuous sampling is given by:

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

where

V_I is the primary increment variance;

n is the number of increments in the sample;

V_PT is the variance of preparation and testing for both off-line and on-line systems;

m is the number of sub-lots.

The procedures given in this part of ISO 13909 are designed to achieve levels of V_PT of 0,05 or less for moisture tests. Better levels may be expected for other chemical characteristics.

For some preparation schemes, however, practical restrictions may prevent the preparation and testing variance being as low as this. Under these circumstances, the user will have to decide whether to achieve the desired overall precision by improving the preparation scheme or by dividing the lot into a greater number of sub-lots.

The errors occurring in the various stages of preparation and analysis, expressed in terms of variance, may be checked by the methods given in ISO 13909-7.

5 Constitution of a sample

5.1 Introduction

Examples of the constitution of samples are shown in Figure 1.

Primary increments shall be taken in accordance with the procedures specified in ISO 13909-5.

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. size analysis or bias testing, the sample consists of a single increment which is prepared and tested.

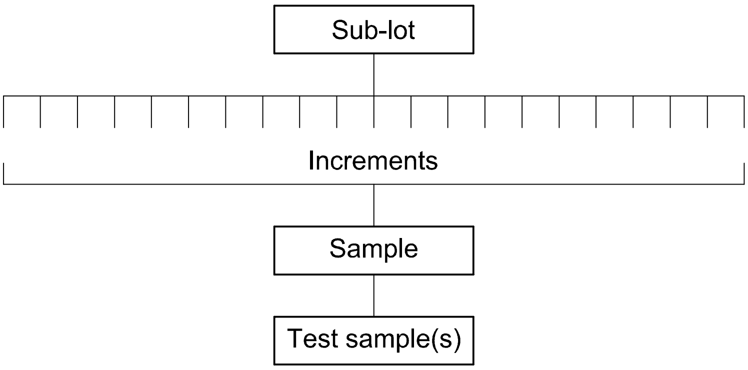
Samples may also be prepared by the combination of other samples.

The procedures for increment combination (5.2) may vary according to whether the primary increments were taken using a time-basis (5.2.1) or a mass-basis (5.2.2) sampling scheme.

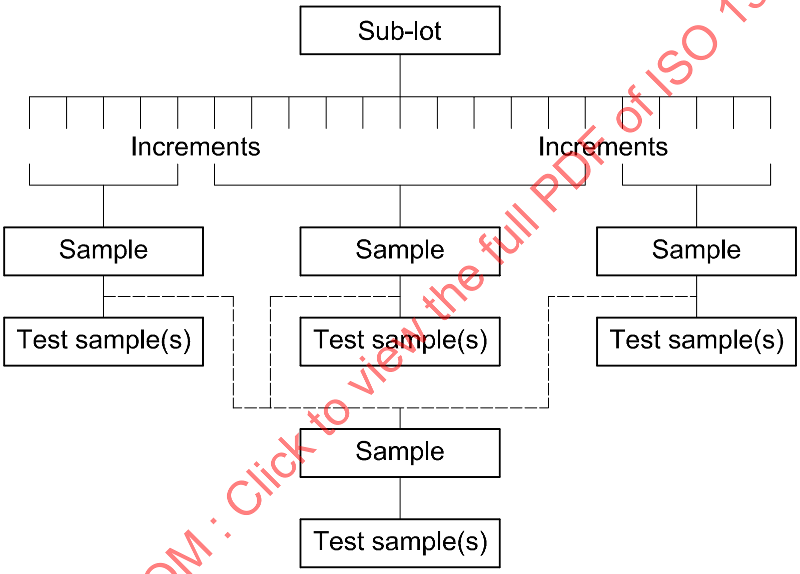
5.2 Combination of increments

5.2.1 Time-basis sampling

The mass of the primary increments shall be proportional to the flow rate at the time of sampling. The primary increments may be combined into a sample, either directly as taken or after having been prepared individually to an appropriate stage by fixed-ratio division (see clause 6).



a) Example 1



b) Example 2

Figure 1 — Examples of the constitution of samples

5.2.2 Mass-basis sampling

If the primary increments are of almost uniform mass (see note), they may be combined into a sample, either directly as taken or after having been prepared individually to an appropriate stage by fixed-ratio division (see clause 6).

NOTE Almost uniform mass has been achieved if the coefficient of variation of the increment masses is less than 20 % and there is no significant correlation between the flow rate at the time of taking the increment and the mass of the increment (see ISO 13909-5).

If the primary increments are not of almost uniform mass, they may only be combined into samples after having been divided individually by fixed-mass division (see clause 6).

5.3 Combination of samples

When combining samples, the mass of the individual samples shall be directly proportional to the mass of the coke from which they were taken in order to obtain a weighted mean of the quality characteristic for the sub-lot. Prior to combination, division shall be by fixed-ratio (see clause 6).

6 Division

6.1 General

Since the cutter aperture will normally be at least three times the nominal top size, this will result in a very large increment mass in many cases. The handling and preparation of such large increments would be either manpower or equipment intensive. Division prior to further treatment may be necessary to provide a manageable sample mass.

Sample division can be

- on-line mechanically, or
- off-line mechanically or manually.

Whenever possible, mechanical methods are preferred to manual methods to minimize human error. Examples of dividers are shown in Figure 2.

Mechanical dividers are designed to extract a part of the 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 may be necessary.

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

6.2 Mechanical methods

6.2.1 General

Mechanical sample division may be carried out on an individual increment or a sample. Where samples are for moisture or general analysis, it is permissible to install on-line crushing to a nominal top size of 16 mm followed by sample division. Division shall be either by fixed-mass division or by fixed-ratio division subject to the conditions set out in 6.2.2 and 6.2.3.

When crushing on-line, the risk of moisture loss should be considered, particularly if the coke is hot.

The uses to which the sample is to be put, the numbers, masses and size distribution of the test samples required shall also be taken into account when deciding on the minimum mass of the sample.

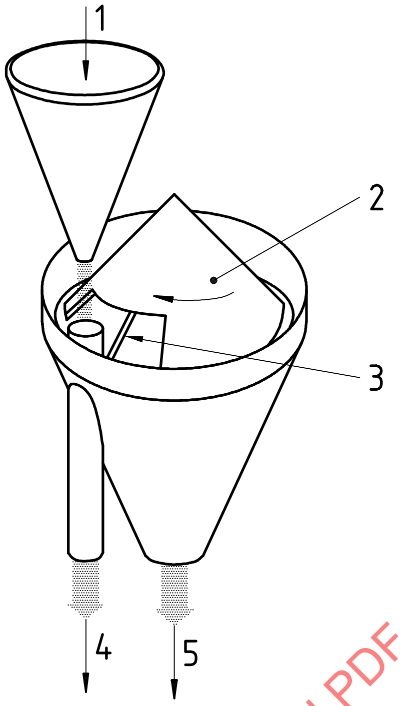
When a coke is regularly sampled under the same conditions, the precision obtained for all the required quality parameters shall be checked using the procedures of ISO 13909-7 and the minimum mass adjusted accordingly. The masses shall not be reduced, however, below the minimum requirements laid down in the relevant analysis standards.

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

6.2.2 Mass of cut

The cuts shall be of uniform mass throughout the division of an increment. In order to achieve this, the flow of coke to the divider shall be uniform and the cutting aperture and speed of the cutter 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 coke to be divided.



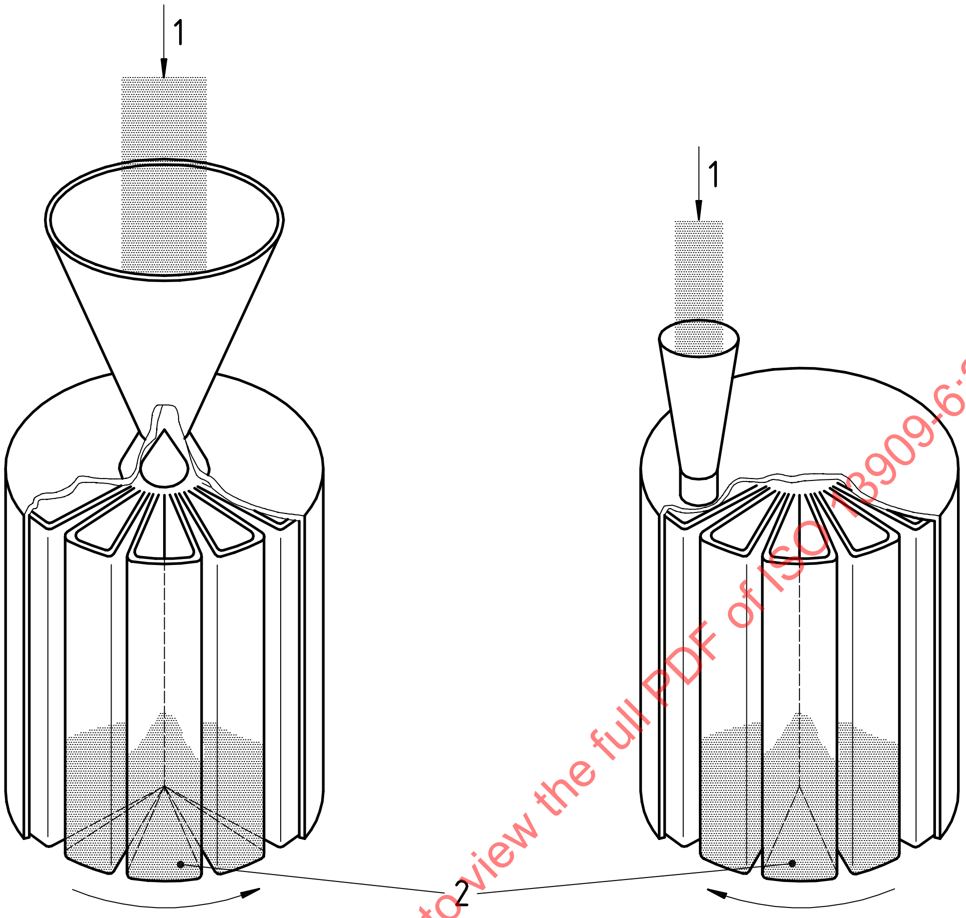
Key

1	Feed
2	Rotating cone
3	Adjustable slot
4	Divided sample
5	Reject

A stream of coke is allowed to fall onto a rotating cone; the adjustable slot with lips in the cone allows the stream to fall directly onto the sample receiver for part of each revolution.

a) Rotating cone type

Figure 2 — Examples of dividers



Key

1	Feed
2	Divided sample in rotating receivers

The coke stream flows to the hopper and this flow is intercepted by the top edge of a number of sector-shaped containers dividing the flow into equal parts. Either the hopper or the containers may rotate. The machine can be controlled for the following operations:

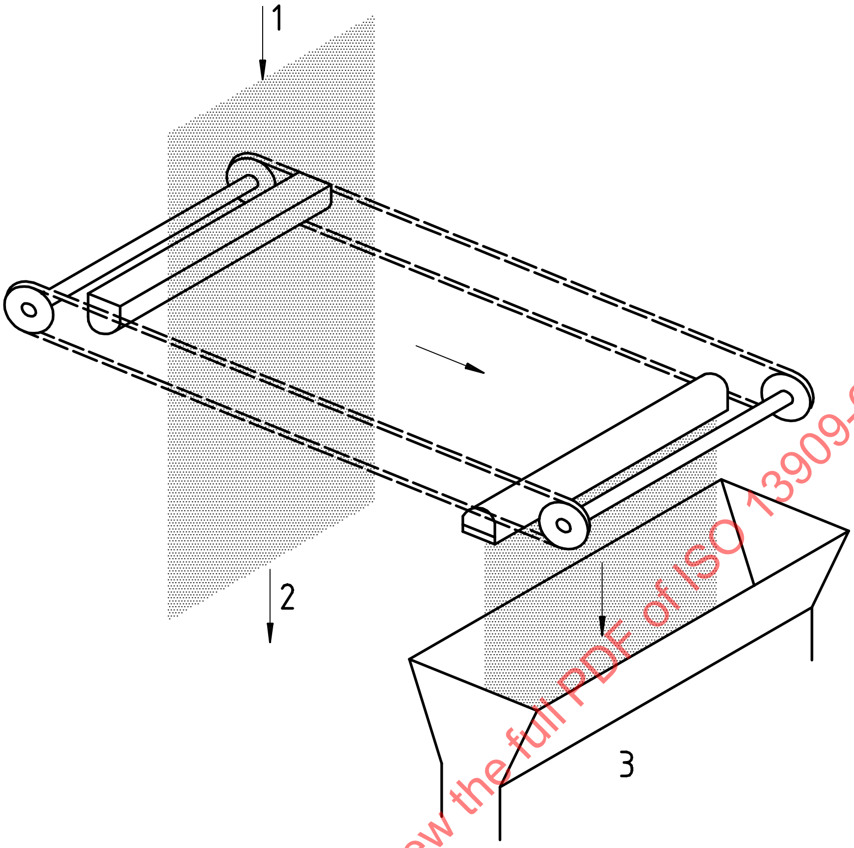
a) for dividing;

b) for collecting duplicates;

c) for collecting replicates.

b) Container type

Figure 2 — Examples of dividers (*continued*)



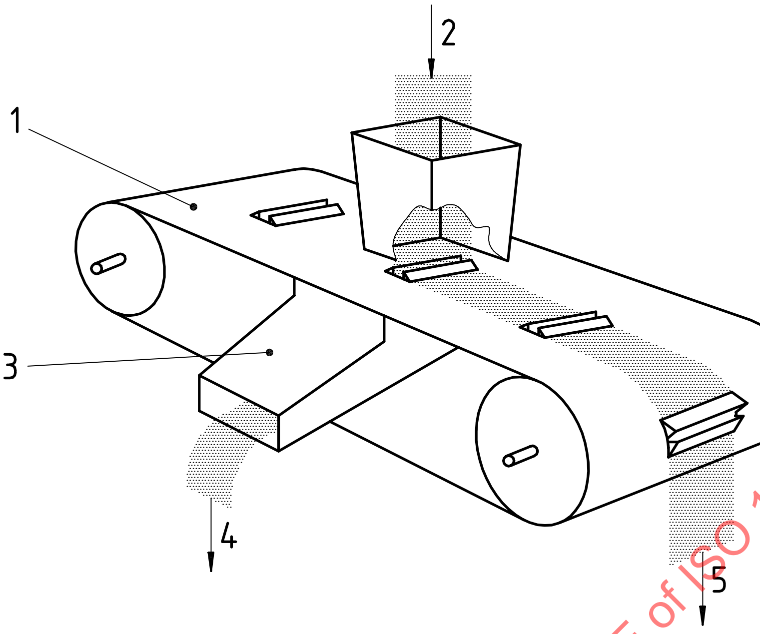
Key

1	Feed
2	Reject
3	Divided sample

A chain mechanism as shown is equipped with buckets spread at equal pitch. The buckets travel in a single direction or change direction at pre-set time periods. The bucket intercepts the free-falling coke stream to extract cuts which discharge to sample as the bucket inverts.

c) Chain-bucket type

Figure 2 — Examples of dividers (*continued*)



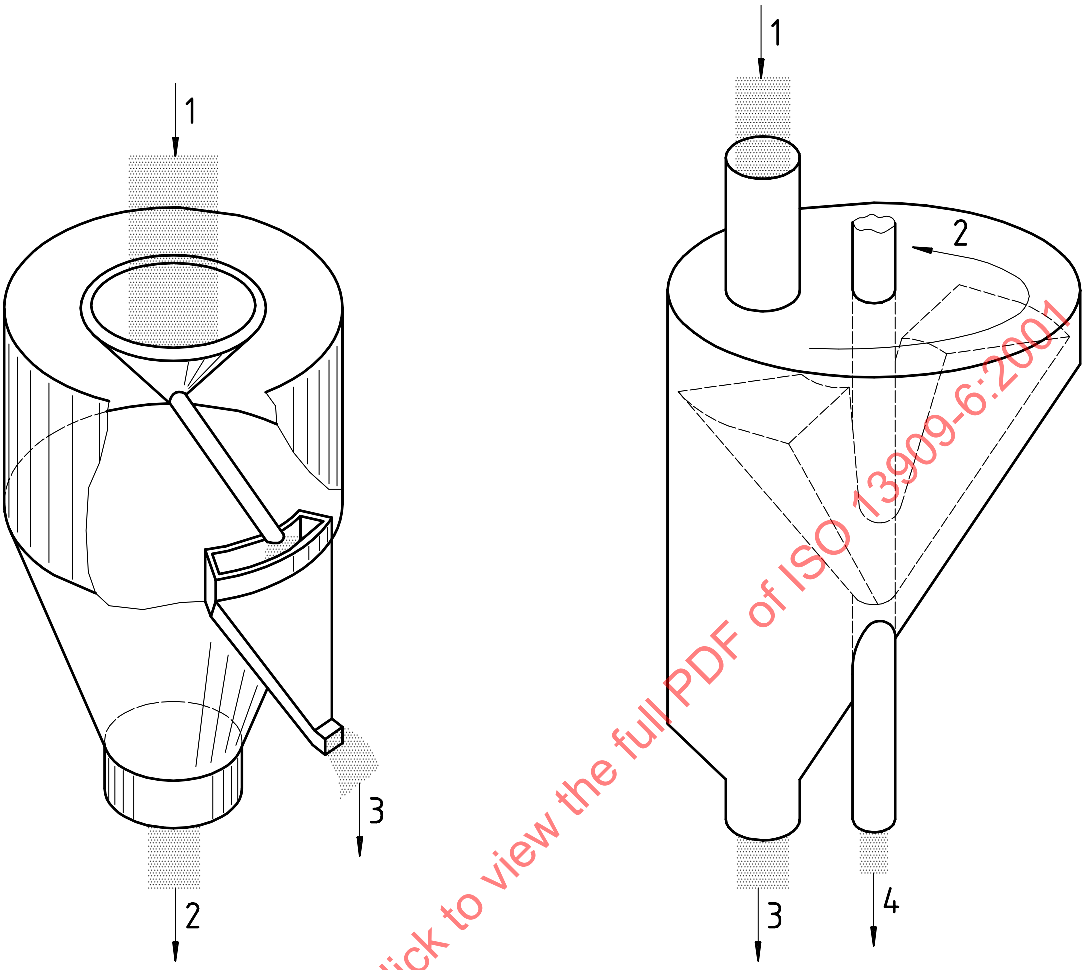
Key

1	Slotted belt
2	Feed
3	Inclined chute
4	Divided sample
5	Reject

An endless belt having slots spaced at equal pitch with lips that act as cutting edges passes below a feed chute. The coke stream is fed to the chute and, as each slot passes through the stream, a cut is taken. The stream which falls onto the plain part of the belt is carried to rejects.

d) Slotted-belt type

Figure 2 — Examples of dividers (*continued*)



Key

1 Feed
2 Reject
3 Divided sample

A hollow shaft which is attached to one or more cutters rotates within a housing as shown. Each cutter is designed to take cuts from the coke stream and to discharge via the hollow shaft.

e) Rotating chute type

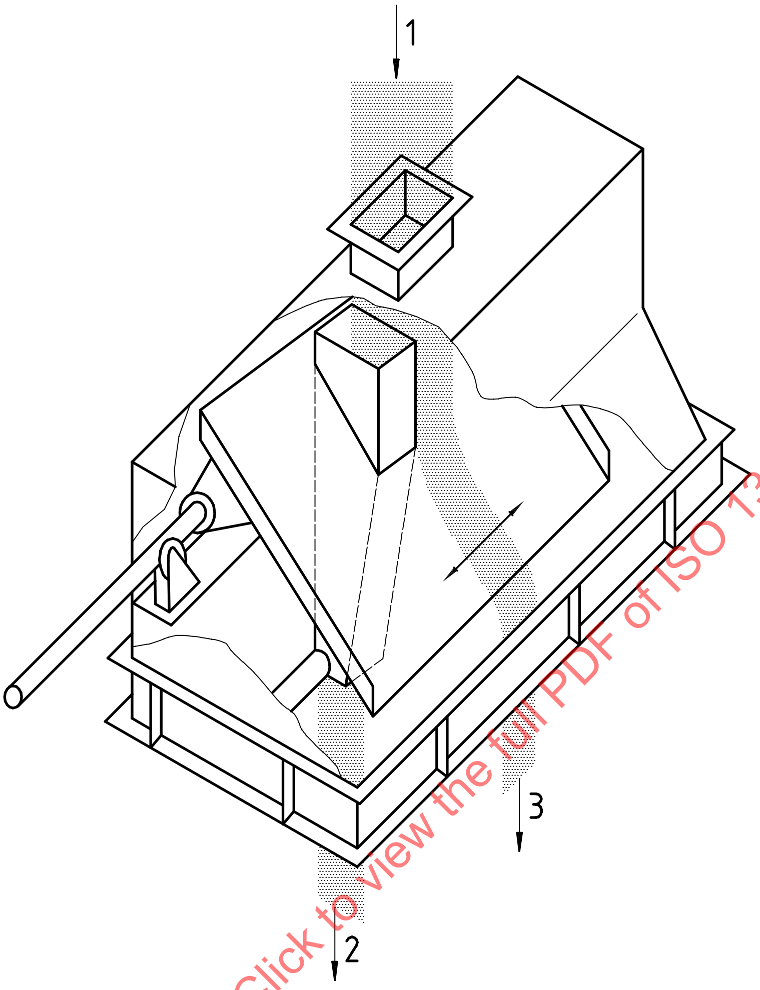
Key

1 Feed
2 Rotating hopper
3 Reject
4 Divided sample

A rotating hopper receives a coke flow and discharges through a spout. Stationary cutters are positioned in the path of the spout outlet, cuts are taken as each cutter is passed. One or more cutters may be fitted.

f) Rotating hopper and spout

Figure 2 — Examples of dividers (*continued*)



Key

1	Feed
2	Divided sample
3	Reject

The cutter-chute traverses the full coke stream and diverts a portion from the stream. When the coke stream is not being cut by the chute, it is deflected by the angle plate to reject.

g) Cutter-chute type

Figure 2 — Examples of dividers (*continued*)

6.2.3 Interval between cuts

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

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

6.2.4 Division of individual increments

6.2.4.1 Number of cuts

The number of cuts for dividing an increment shall be determined as follows.

a) For fixed-mass division, the minimum number of cuts at any stage shall be six. An equal number of cuts shall be taken from each increment in the sub-lot.

b) For fixed-ratio division, the minimum number of cuts from an increment of mean mass at any stage shall be six.

For subsequent division of divided increments, a minimum of one cut shall be taken from each cut from the preceding division.

6.2.4.2 Minimum mass of divided increment

The minimum mass of the divided increment shall be such that the combined masses of all the divided increments in the sub-lot shall, at each stage, be greater than the mass given in Table 1, corresponding to 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.

For moisture samples, there is a restriction of 70 kg on the maximum mass of sample to be crushed at one time during off-line preparation.

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

Table 1 — Minimum mass of sample after division

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

6.2.5 Division of samples

6.2.5.1 Number of cuts

The sample shall be divided by taking a minimum of 60 cuts.

NOTE If, during preparation, the sample is thoroughly mixed and it can be established that the required precision can be achieved, the number may be reduced to 20.

6.2.5.2 Minimum mass of divided sample

The mass of the divided sample shall not be less than the minimum mass in Table 1.

6.3 Manual methods

6.3.1 General

For the manual division methods given in 6.3.2 to 6.3.4, the mass of the divided sample shall not be less than the minimum mass in Table 1, subject to there being sufficient mass for all test portions required.

6.3.2 Riffle method

A riffle (see Figure 3) is a sample divider that will, in a single pass of a sample, divide it into halves, one of which is retained and the other normally rejected. The device is normally portable and, for sample division, is usually fed manually, the coke being evenly distributed along its length. Adjacent slots feed opposite receivers.

The slot width shall be at least 2,5 times and, preferably, not more than 3 times the nominal top size of the coke. Each half of the riffle shall have the same number of slots, which shall be at least eight and preferably more. All the surfaces on which the coke might rest shall have a slope of at least 60° to the horizontal.

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

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 cokes 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.

6.3.3 Flattened-heap method

The procedure, which is illustrated in Figure 4, is as follows.

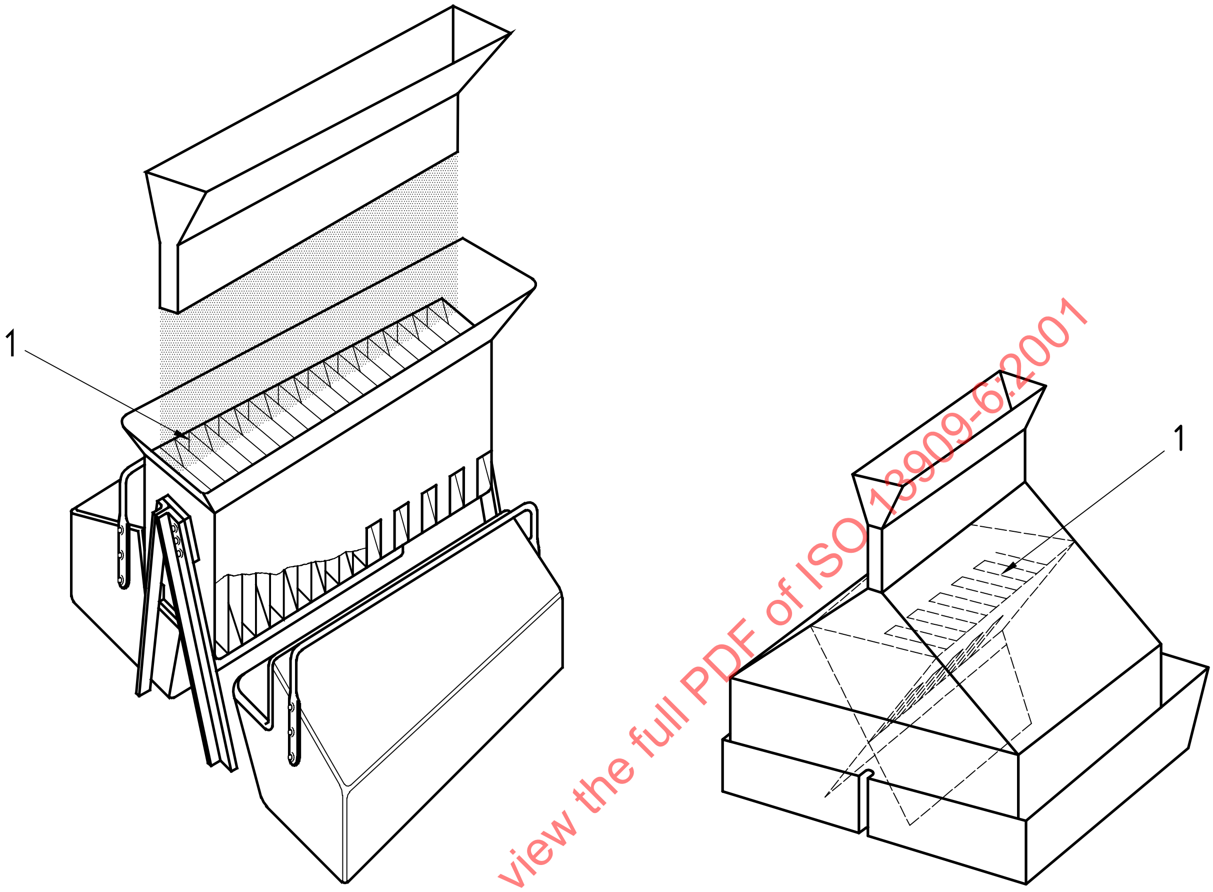
The sample is mixed thoroughly and spread to form a rectangle of uniform thickness of 40 mm to 50 mm on a mixing plate, which is a smooth, non-absorbent and non-contaminating surface.

Avoid moisture loss from wet cokes which can result from over-mixing.

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 with a bump plate (see the last paragraph of this subclause) to the bottom of the matrix layer. The increments shall be combined into a divided sample. It is essential that these operations be performed quickly if loss of moisture is to be prevented.

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

The scoop shall be flat bottomed and the width of the entry shall be at least three times the nominal top size of the 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 increment to be taken.



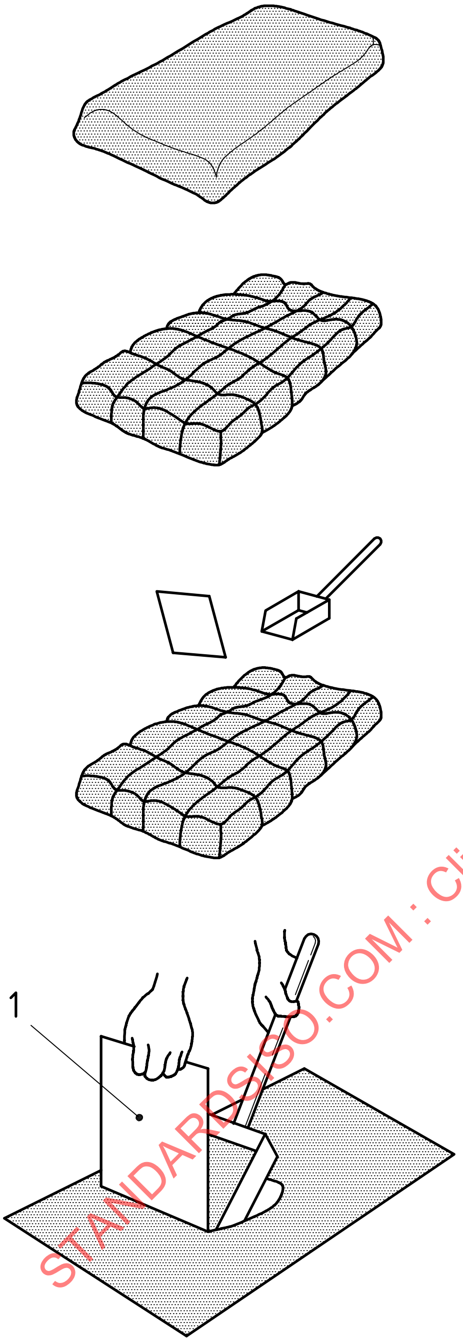
Key

1 Even number of slots

a) Open type **b) Closed type**

Figure 3 — Examples of riffles

Take the scoop sample 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 to the bottom of the spread 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 of 40 mm to 50 mm.

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

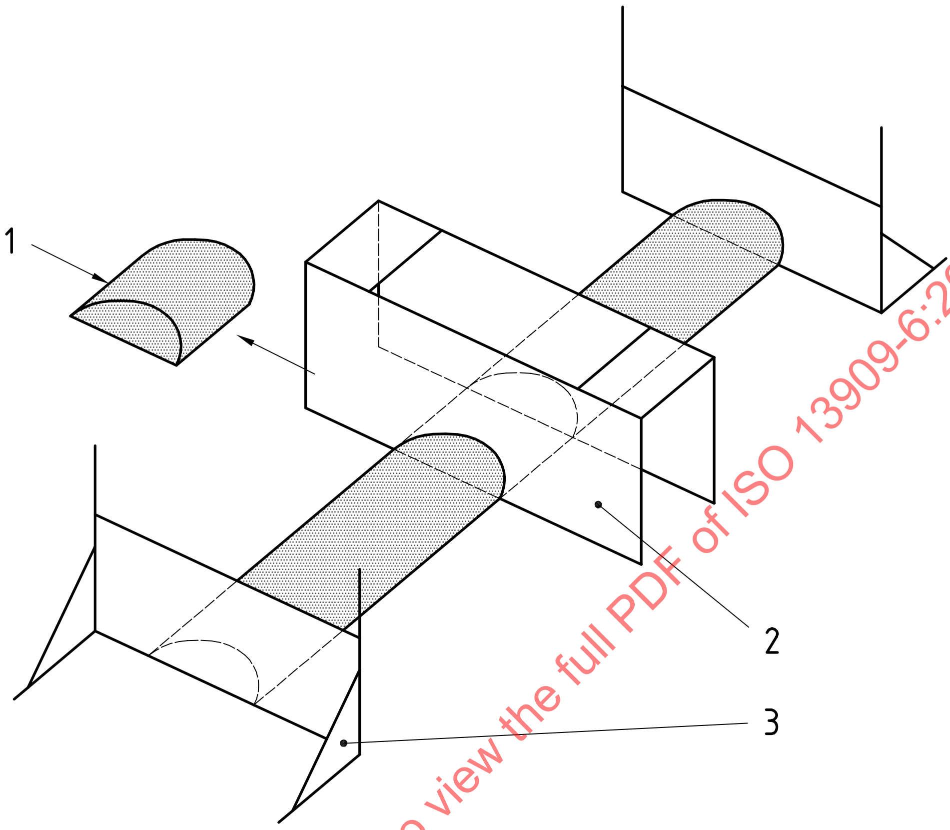
c) Take a scoopful of samples at random from each of the 20 parts by inserting the scoop to the bottom of the sample layer. Combine the 20 scoopfuls into a divided sample.

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

Figure 4 — Flattened-heap method

6.3.4 Strip-mixing and splitting method

The procedure, which is illustrated in Figure 5, is as follows.



Key

1	Increment
2	Sampling frame
3	End plates (“book-ends”)

Figure 5 — Strip-mixing and splitting method

The 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 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 shall be used to ensure that size segregation only occurs laterally.

Increments shall be taken as a complete section across the strip. The width of each cross-section shall be not less than 2,5 times the nominal top size of the coke.

NOTE 1 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 coke is regularly prepared under the same conditions and it has first been established that the required precision can be obtained (see ISO 13909-7).

NOTE 2 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.

7 Preparation of samples for specific tests

7.1 Types of test sample

The methods of preparation depend on the purpose for which the original sample was collected. Three types of sample are usually required:

a) sample for determination of total moisture;

b) sample for general analysis;

c) sample for physical tests.

Additional samples may be required for testing for special properties. Where samples are to be used for the determination of other special properties, it will be necessary to set aside part of the sample, either prior to preparation or after an appropriate stage in the preparation.

A schematic diagram showing the alternative ways of constituting and preparing the various types of test sample is shown in Figure 6.

7.2 Preparation of samples for determining total moisture

7.2.1 General

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 of coke, and the reduction and division procedures used.

Precautions shall be taken to minimize loss of moisture due to the use of unsuitable containers and to evaporation during handling.

All samples for moisture 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, increase the number of sub-lots units to overcome these problems (see ISO 13909-5).

Care shall also be taken to minimize loss of moisture during division by carrying out the operation as quickly as possible and using mechanically operated dividers with limited ingress of air.

7.2.2 Procedure

The procedure for preparing the 1 kg sample for the determination of total moisture is shown schematically in Figure 6.

In the case of small coke, having a top size of 20 mm or less, only the process of sample division described in 7.2.5 is required.

It is essential that precautions be taken to prevent loss of moisture during these operations which shall be carried out in an enclosed space, roofed over and made free from draughts. The total time taken to crush and divide the coke shall not exceed 15 min and it is therefore essential to crush the coke quickly and mechanically. In addition, the mass of sample crushed shall not exceed 70 kg.

Samples which are visibly wet and those for which the moisture is expected to exceed 15 % are partially dried before reduction and division (see 7.2.3).

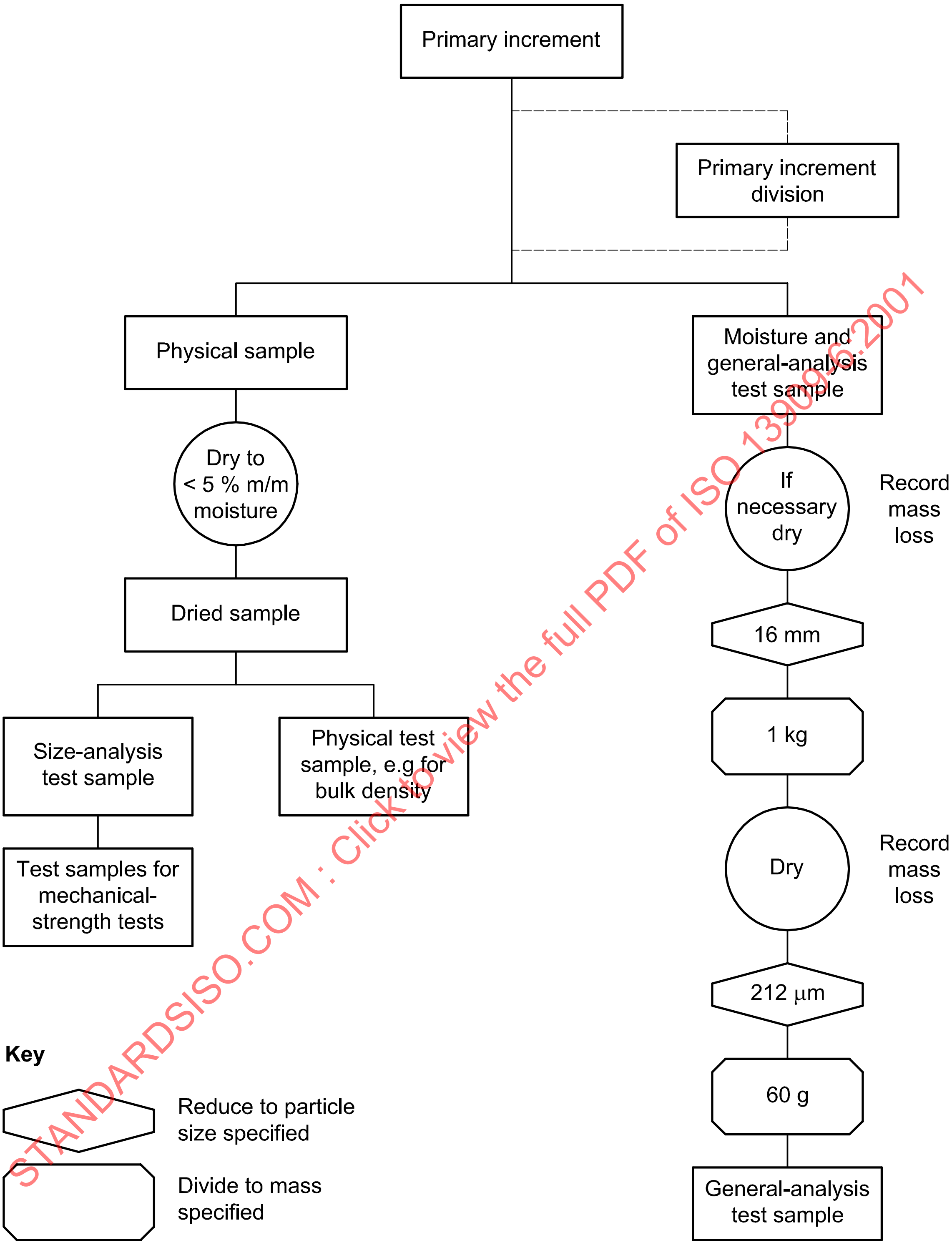


Figure 6 — Constitution and preparation of samples

7.2.3 Wet samples

The procedure for air-drying is as follows.

Weigh the whole of the sample in its container on a weighing machine which can be read to an accuracy of at least 100 g. Spread the sample in a thin layer on a steel plate in a warm, well-ventilated room and leave for a minimum of 12 h until there is no visible moisture on either the coke or the plate. Meanwhile dry and weigh the container. Collect the air-dried sample in the original container and reweigh and calculate the percentage loss in mass. Continue the preparation according to 7.2.4 and 7.2.5.

When air-drying has been 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 7.2.6, calculate the total moisture, M_T as a percentage, from the following equation:

$$M_\mathrm{T} = X + M\left(1 - \frac{X}{100}\right)$$

where

X is the percentage loss on partial drying;

M is the percentage of moisture determined as described in 7.2.6.

7.2.4 Reduction of moisture test sample

The primary increments or divided primary increments shall be fed through a jaw crusher to reduce the nominal top size to 16 mm. The time taken to crush 50 kg shall not exceed 5 min. The process shall be timed occasionally to ensure that this time is not exceeded.

Take care to avoid loss of moisture by undue ventilation or loss of sample as dust.

7.2.5 Division

A test portion of 1 kg shall be prepared by dividing the 16 mm sample using mechanical dividers of the cone or container type [see Figures 2 a) and 2 b)].

The divider may be installed so that the product of the jaw crusher is discharged directly into the feed bin of the divider so that reduction and division may proceed concurrently. Precautions shall be taken against undue ventilation.

For manual division, the strip-splitting and mixing procedure, the flattened-heap procedure (see 6.3) or a closed riffle may be used.

7.2.6 Determination of moisture

The 1 kg sample taken as described in 7.2.2 to 7.2.5 shall be transferred to the laboratory in a closed container. For the determination of moisture, one of the methods specified in ISO 579 or ISO 687 shall be used, as appropriate.

7.2.7 Reserve sample

If a second sample is needed as a reserve or check sample, duplicate 1 kg samples shall be taken at the same time either by modifying the division procedure or by taking alternate increments when carrying out the procedure of manual division.

7.3 Preparation of sample for general analysis

7.3.1 General

The procedure for the preparation of a 60 g sample for general analysis is shown in schematic form as part of Figure 6. Normally, the starting material is the 1 kg sample of coke which has been dried in the determination of total moisture (see 7.2.6). Alternatively, a separate 1 kg sample may be prepared as described in 7.2 and dried.

For some methods of reduction to − 212 µm, it may be necessary to reduce the coke from − 16 mm to an intermediate size, e.g. − 4 mm.

7.3.2 Preparation errors

7.3.2.1 General

The process of sample preparation, which involves two distinct operations of reduction and division, may lead to errors due to contamination, segregation and loss of fines.

7.3.2.2 Contamination errors

The contamination error arises from the abrasive nature of coke, which tends to wear the surface of mild steel, cast iron, porcelain and, if a rubbing action is involved, most of the surfaces used in grinding equipment. This error may be as high as 3 % of ash if the dried moisture sample is reduced to 212 µm by rubbing on a cast-iron surface.

Although contamination usually implies the presence in the ash of iron oxide derived from a ferrous grinding surface, it may also involve an increase in the phosphorus content if cast-iron surfaces are used.

The contamination error can never be entirely eliminated but it can be reduced to negligible proportions if the recommended methods and equipment are used. Methods of size reduction other than those recommended, such as rubbing against a metal surface, are unsatisfactory and shall not be used.

Equipment also used for other samples shall be flushed with some excess sample material to remove any bituminous or high phosphorous material.

7.3.2.3 Segregation errors

The segregation error arises from the heterogeneous nature of coke and because of the difficulty in uniformly mixing any coke in which there is a wide range of sizes. It is therefore essential to mix the coke thoroughly in order to obtain a representative sample.

The segregation error increases with both the ash in the coke and the size at which division occurs.

This error can be made negligible even for high-ash cokes if the particle size is reduced to 212 µm, the size required for general analysis, before division occurs.

7.3.3 Sample reduction

The 1 kg sample shall be reduced to pass a 212 µm sieve by any suitable means, such as crushing in a crusher with chromium steel rolls. The coke shall be crushed in successive passes. The rolls shall be capable of crushing to smaller than 212 µm without causing contamination.

Alternatively, the coke may be reduced using a totally enclosed planetary ball mill or ring grinder. The materials in contact with the coke shall be of such hardness that no contamination of the sample occurs by abrasion.