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

Part 4:
Coal — Preparation of test samples

Houille et coke — Échantillonnage mécanique —

Partie 4: Charbon — Préparation des échantillons pour essai



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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 13909-4 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.

When difficulty in handling the coal or coals being sampled is expected at a particular stage in sample preparation, or if there is a likelihood of losing moisture by evaporation, it is necessary to withdraw the sample or increment from the on-line system at the stage immediately prior to the point of difficulty and proceed off-line.

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Hard coal and coke — Mechanical sampling —

Part 4:

Coal — Preparation of test samples

1 Scope

This part of ISO 13909 describes the preparation of samples of coal 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 589:1981, *Hard coal — Determination of total moisture*.

ISO 3310-1:2000, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*.

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

ISO 13909-2:2001, *Hard coal and coke — Mechanical sampling — Part 2: Coal — Sampling from moving streams*.

ISO 13909-3:2001, *Hard coal and coke — Mechanical sampling — Part 3: Coal — Sampling from stationary lots*.

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:

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

where

V_I is the primary increment variance;

n is the number of increments per sub-lot;

V_{PT} is the preparation and testing variance 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 0,2 or less for both ash and moisture tests. Better levels are expected when using mechanical dividers.

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 method given in ISO 13909-7.

5 Constitution of a sample

5.1 Introduction

Primary increments shall be taken in accordance with the procedures specified in ISO 13909-2 and ISO 13909-3.

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

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.

Samples may also be prepared by the combination of other samples (see 5.3).

5.2 Combination of increments

5.2.1 Time-basis sampling

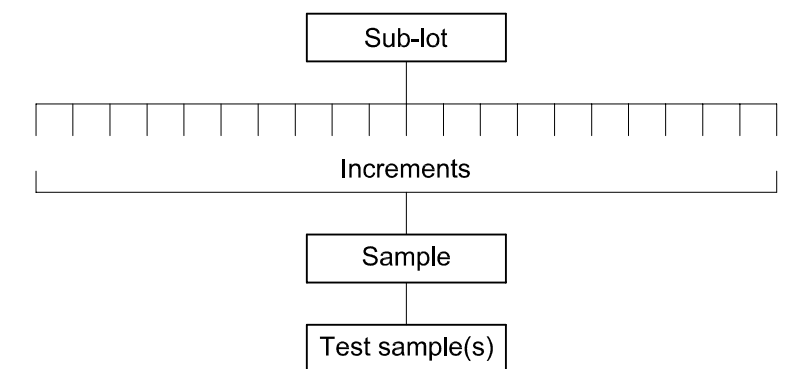
The mass of the primary increments shall be proportional to the flow rate at the time of sampling. The primary increments may be combined into a sample, either directly as taken or after having been prepared individually to an appropriate stage by fixed-ratio division (see clause 6).

5.2.2 Mass-basis sampling

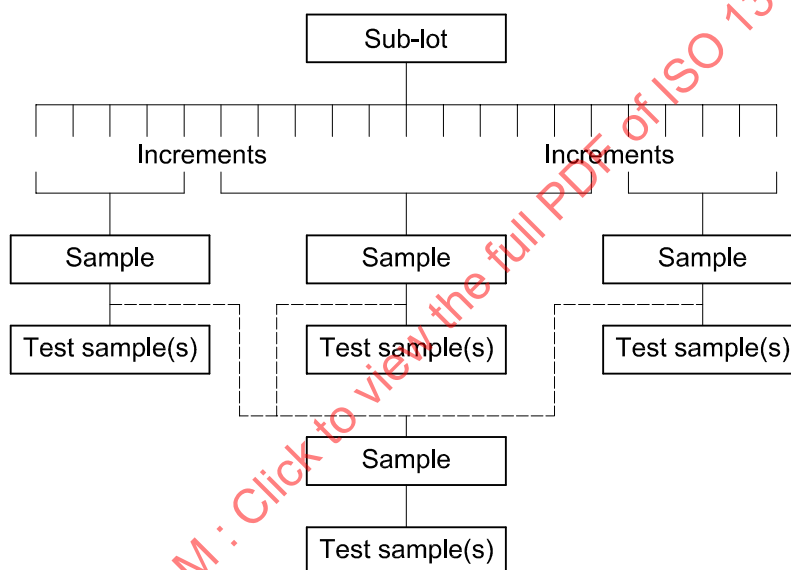
If the primary increments are of almost uniform mass (see note), they may be combined into a sample, either directly as taken or after having been prepared individually to an appropriate stage by fixed-ratio division (see clause 6).

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 13909-2:2001, annex B).

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 7).



a) Example 1



b) Example 2

Figure 1 — Examples of the constitution of samples

5.3 Combination of samples

When combining samples, the mass of the individual samples shall be directly proportional to the mass of the coal 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 division (see clause 6).

6 Division

6.1 General

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 one or more parts of the coal 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.

Coal which is visibly wet may not run freely through or may 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 clause 10 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 methods may themselves result in bias, particularly if the mass of coal 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 which has been crushed, if necessary, to an appropriate nominal top size. Division shall be either by fixed-mass division or by fixed-ratio division subject to the conditions set out in 6.2.3.

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 coal 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 coal 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.

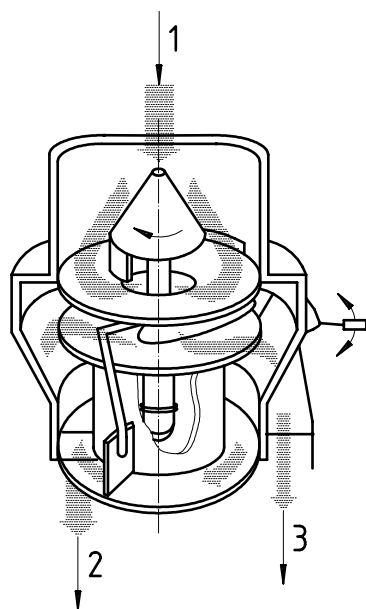
The cutting aperture shall be at least three times the nominal top size of the coal to be divided.

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 cutting interval. For secondary and tertiary dividers, the cycle time shall not be evenly divisible into the cycle time of the cutter which precedes it.

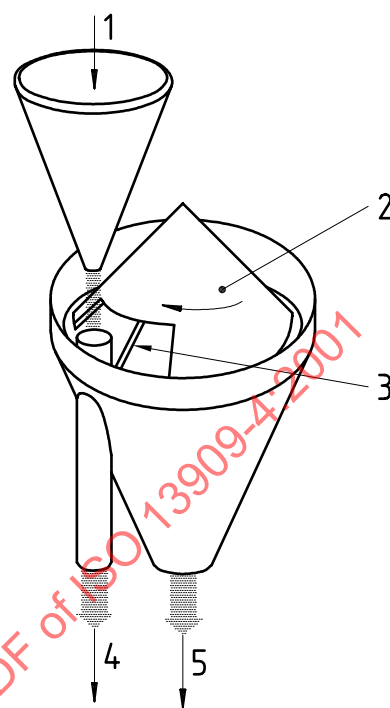
For fixed-mass division, the interval between taking cuts shall be varied proportionally to the mass of coal to be divided so that divided samples having almost uniform mass are obtained.

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

**Key**

- 1 Feed
- 2 Reject
- 3 Divided sample

The material from a mixing container is fed by scrapers to the centre of the dividing disc. From there it is discharged over the range of the disc via special clearing arms. The sample falls through adjustable slots into chutes; the reject is carried away via a cleaning conduit. The whole interior space is cleaned by scrapers.

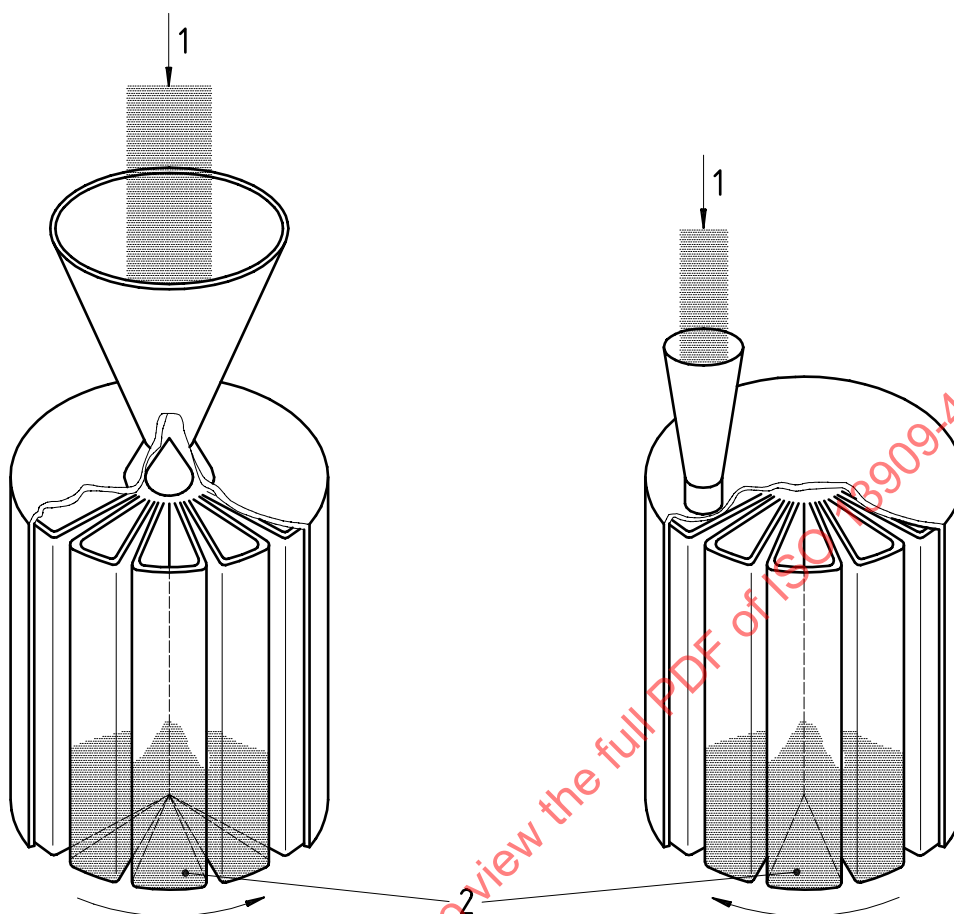
a) Rotating disc type**Key**

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

A stream of coal 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.

b) Rotating cone type

Figure 2 — Examples of dividers

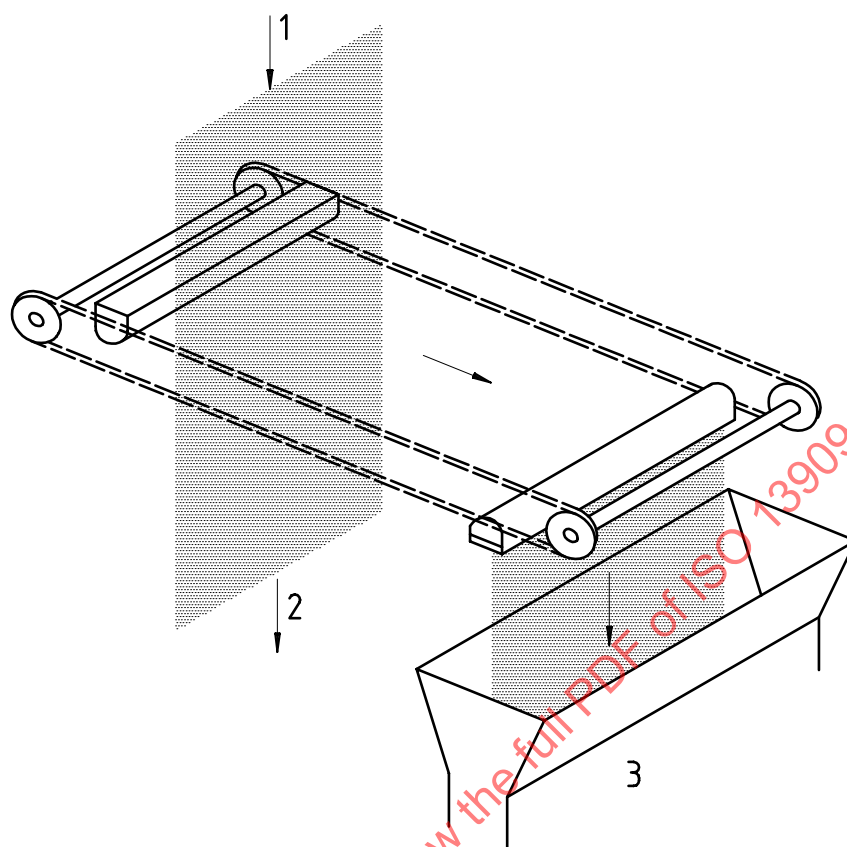
**Key**

- 1 Feed
- 2 Divided sample in rotating receivers

The coal 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:

- 1) for dividing;
- 2) for collecting duplicates;
- 3) for collecting replicates.

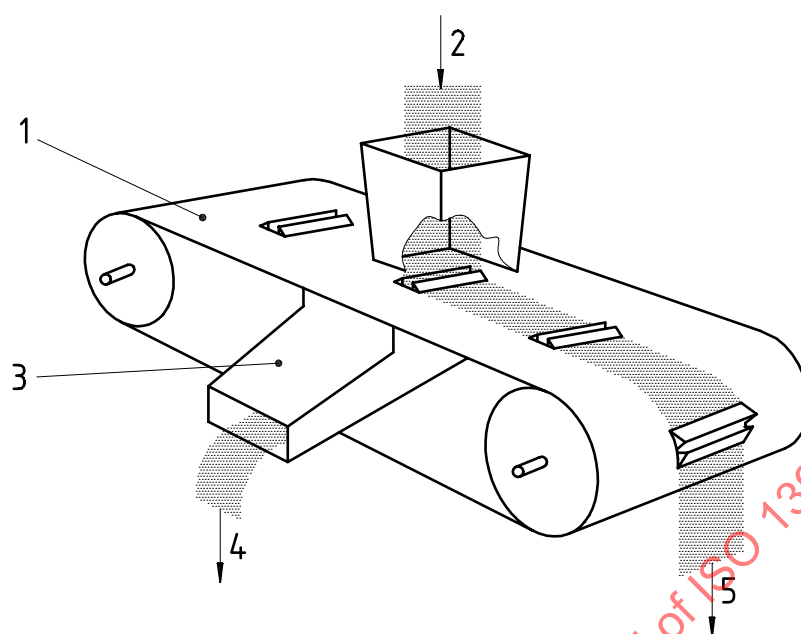
c) Container type**Figure 2 — Examples of dividers (continued)**

**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 coal stream to extract cuts which discharge to sample as the bucket inverts.

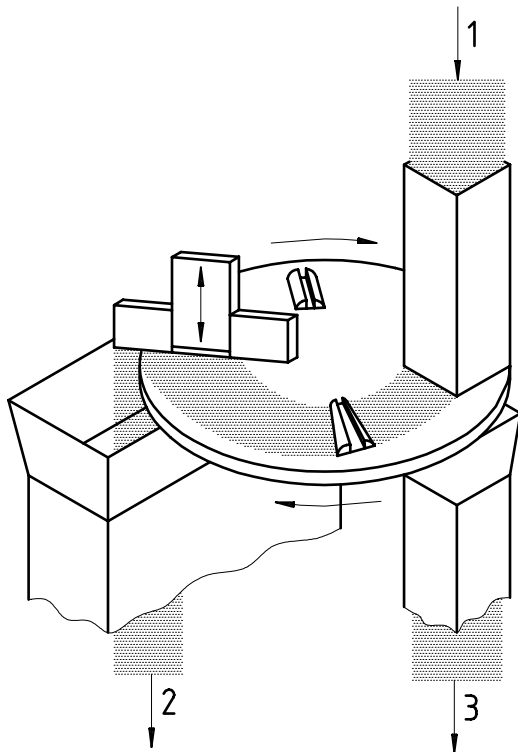
d) Chain-bucket type**Figure 2 — Examples of dividers** *(continued)*

**Key**

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

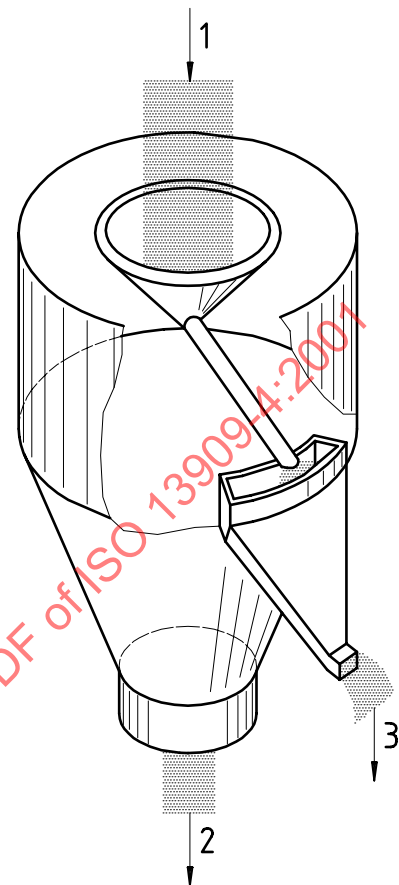
An endless belt having slots spaced at equal pitch with lips that act as cutting edges passes below a feed chute. The coal stream is fed to the chute and, as each slot passes through the stream, a cut is taken. The stream which falls onto the plain part of the belt is carried to rejects.

e) Slotted-belt type**Figure 2 — Examples of dividers (continued)**

**Key**

- 1 Feed
- 2 Reject
- 3 Divided sample

A flat plate with lipped slots spaced at equal pitch rotates beneath a feed chute. Coal is fed into the feed chute, then falls onto the rotating plate to form a ribbon bed which is carried to the plough and discharged to rejects. As a slot passes through the stream, a cut is taken.

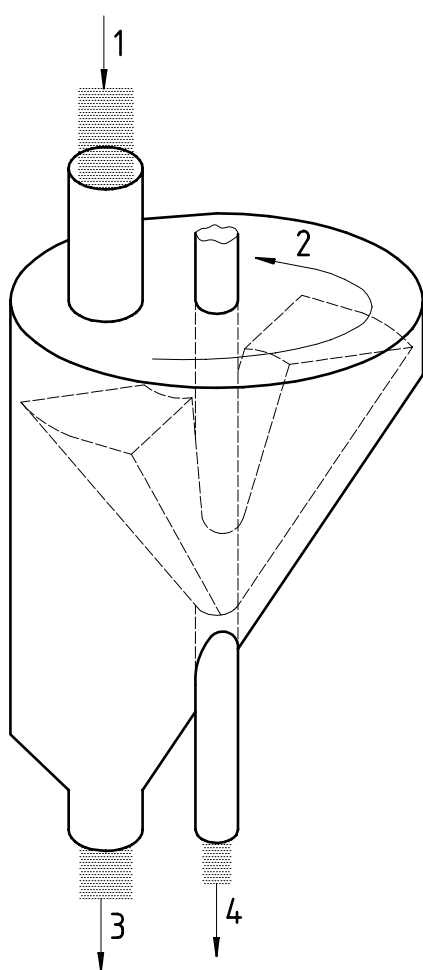
f) Rotating plate type**Key**

- 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 coal stream and to discharge via the hollow shaft.

g) Rotating chute type

Figure 2 — Examples of dividers (continued)

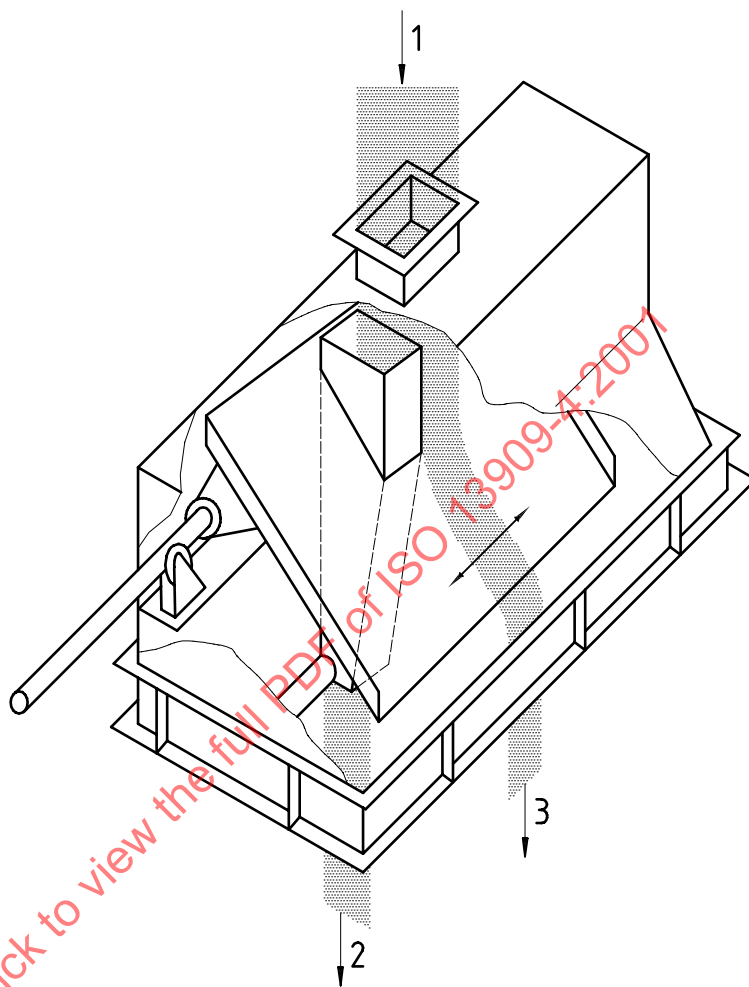


Key

- 1 Feed
- 2 Rotating hopper
- 3 Reject
- 4 Divided sample

A rotating hopper receives a coal 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.

h) Rotating hopper and spout



Key

- 1 Feed
- 2 Divided sample
- 3 Reject

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

i) Cutter-chute type

Figure 2 — Examples of dividers (continued)

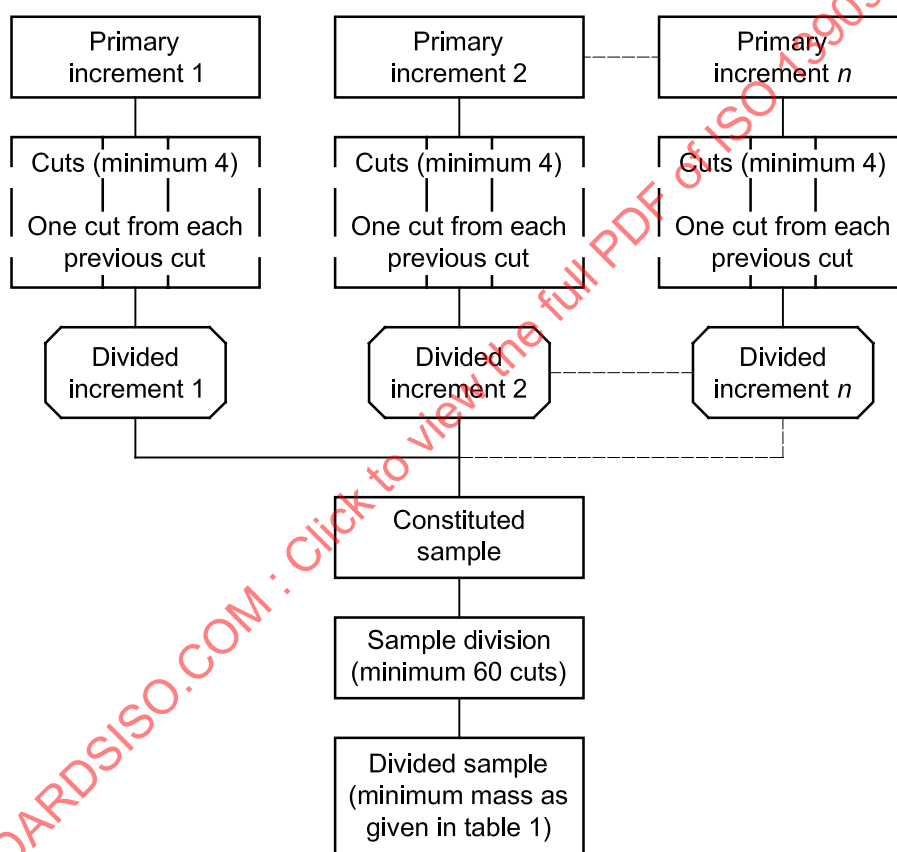
6.2.4 Division of individual increments

6.2.4.1 Number of cuts

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

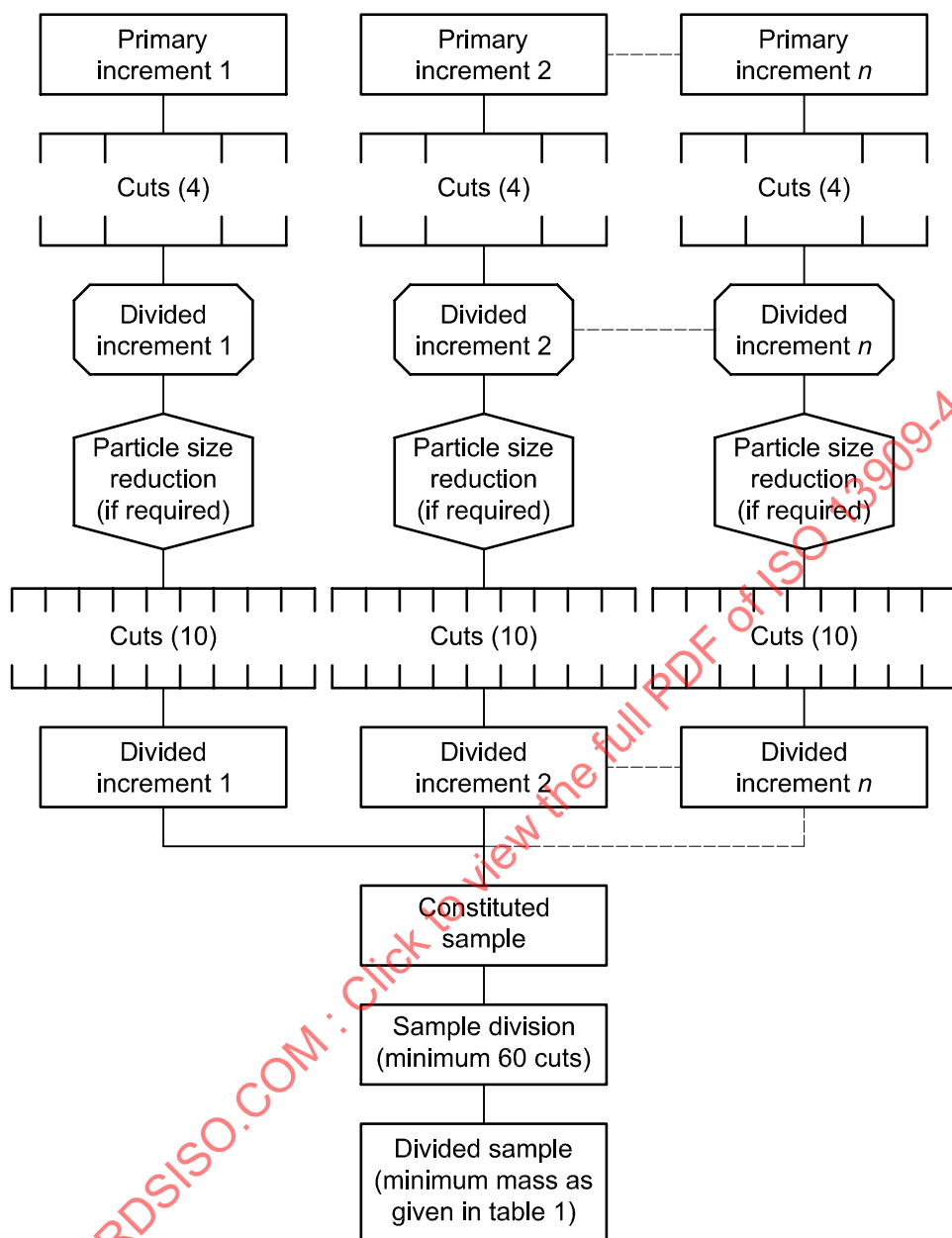
- For fixed-mass division, the minimum number of cuts for dividing primary increments shall be four. An equal number of cuts shall be taken from each primary increment in the sub-lot.
- For fixed-ratio division, the minimum number of cuts for dividing a primary increment of mean mass shall be four.
- For subsequent division of individual divided primary increments, a minimum of one cut shall be taken from each cut from the preceding division.

Examples of procedures for division of individual increments, and subsequent sample division are shown in Figure 3.



a) Example of division of individual increments (minimum number of cuts)

Figure 3 — Examples of procedures for division of increments and samples



b) Example of two-stage division of individual increments

Figure 3 — Examples of procedures for division of increments and samples (continued)

6.2.4.2 Minimum mass of divided increment

The minimum mass of a 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 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.

6.2.5 Division of samples

6.2.5.1 Number of cuts

The sample constituted from all increments, or divided increments, 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.

If the mass is too low, an alternative manual method of division should be used.

6.2.5.2 Minimum mass of divided samples (see Table 1)

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

The minimum mass of divided samples is dependent on the nominal top size of the coal, the precision required for the parameter concerned and the relationship of that parameter to particle size. The attainment of the required minimum mass after division will not, in itself, guarantee the required precision, because division precision is also dependent on the number of cuts taken during division (see 6.2.4.1 and 6.2.5.1).

Values for the minimum mass of divided samples for general analysis to reduce the variance due to the particulate nature of the coal to 0,01, corresponding to a precision of 0,2 % with regard to ash, are given in column 2 of Table 1 (see CSIRO report ^[1]). Column 3 of Table 1 gives the corresponding minimum masses of divided samples for total-moisture analysis, which are approximately 20 % of the minimum masses for general analysis, subject to an absolute minimum of 0,65 kg. Values for the minimum mass of divided samples for size analysis are given in columns 4 and 5 of Table 1 for division precisions of 1 % and 2 % respectively. These masses have been calculated on the basis of the precision of the determination of oversize, i.e. the coal above the normal top size. The precision for other size fractions will normally be better than this. Note that, in each case, the overall division precision is determined by the sum of the division variances for each sample-division stage.

The minimum mass of divided samples, m_S , for other desired levels of precision may be calculated from the following equation:

$$m_S = m_{S,0} \left(\frac{P_0}{P_R} \right)^2 \quad (2)$$

where

$m_{S,0}$ is the minimum mass of sample after division specified in Table 1 for a given nominal top size;

P_0 is the precision for a given division stage specified in Table 1;

P_R is the required precision for a given division stage.

When a coal is regularly sampled under the same circumstances, the precision obtained for all the required quality parameters shall be checked (see ISO 13909-7) and the masses may be adjusted accordingly. However, the masses shall not be reduced below the minimum requirements laid down in the relevant analysis standards.

When preparing coal to produce samples for multiple use, account shall also be taken of the masses and size distribution of the test samples required for each test.

Table 1 — Minimum mass of sample after division

Nominal top size of coal mm	General-analysis and common samples kg	Total-moisture analysis samples kg	Size-analysis samples	
			1 %	2 %
300	15 000	3 000	54 000	13 500
200	5 400	1 100	16 000	4 000
150	2 600	500	6 750	1 700
125	1 700	350	4 000	1 000
90	750	125	1 500	400
75	470	95	850	210
63	300	60	500	125
50	170	35	250	65
45	125	25	200	50
38	85	17	110	30
31,5	55	10	65	15
22,4	32	7	25	6
16	20	4	8	2
11,2	13	2,5	3	0,7
10	10	2,0	2	0,5
8	6	1,5	1	0,25
5,6	3	1,2	0,5	0,25
4	1,5	1,0	0,25	0,25
2,8	0,65	0,65	0,25	0,25
2,0	0,25	—	—	—
1	0,1	—	—	—
< 0,5	0,06	—	—	—

NOTE 1 The masses for the general-analysis samples and common samples have been determined to reduce the variance due to the particulate nature of coal to 0,01, corresponding to a precision of 0,2 % ash.

NOTE 2 These values are generally suitable for off-line division but, for nominal top sizes of 16 mm and below, the masses may not be sufficient to maintain the integrity of the sample when performing on-line division.

6.3 Manual methods

6.3.1 Riffle method

A riffle (see Figure 4) 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 coal being evenly distributed along its length. Adjacent slots feed opposite receivers.

The slot width shall be at least 3 times the nominal top size of the coal. 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 coal might rest shall have a slope of at least 60° to the horizontal.

The coal shall be allowed to fall steadily into the riffle, ensuring that it is evenly distributed over all the slots. The coal 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 coals 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.

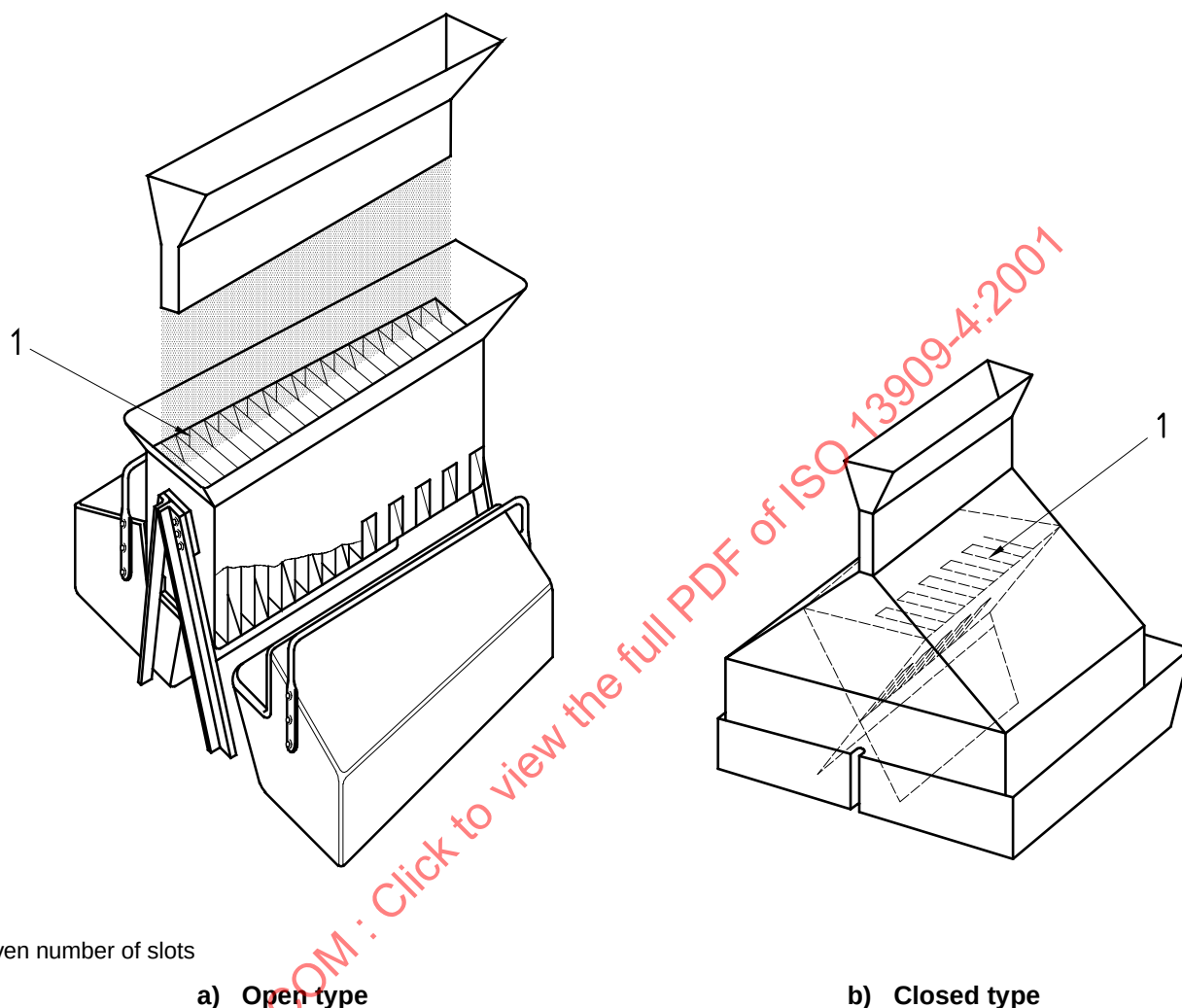


Figure 4 — Examples of riffles

6.3.2 Flattened-heap method

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

The sample is mixed thoroughly and spread to form a rectangle of uniform thickness on a mixing plate which is a smooth, non-absorbent and non-contaminating surface. The maximum thickness shall be 3 times the nominal top size of the coal. Avoid moisture loss from wet coals which can result from over-mixing.

If the mass of the coal 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 shall be taken from each heap.

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 are 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 is the mass of the divided sample (see Table 1) divided by the number of parts of the flattened heap. This mass is determined by using a scoop of appropriate dimensions.

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

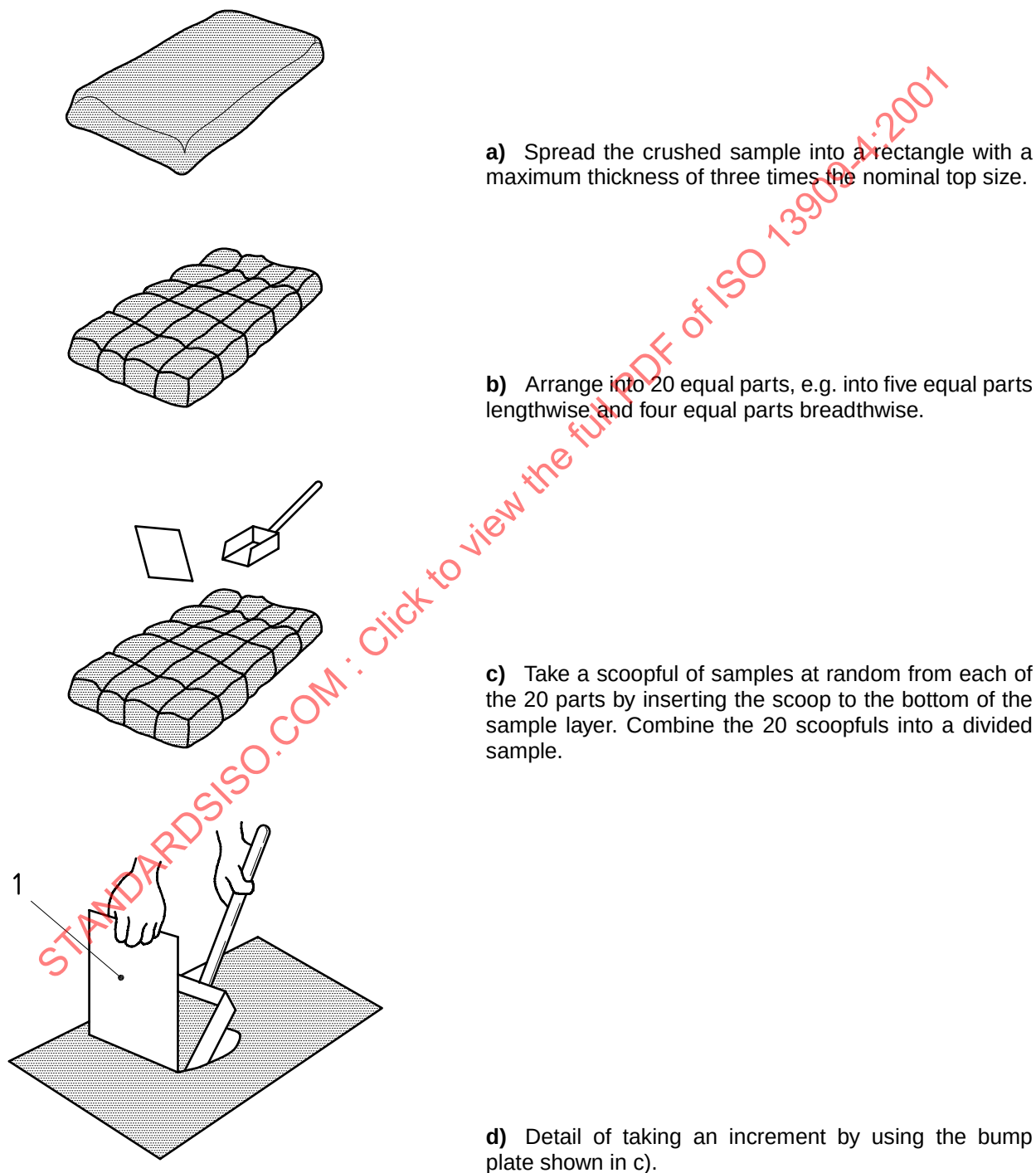


Figure 5 — Flattened-heap method

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 coal 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.

6.3.3 Strip-mixing and splitting method

The procedure, which is illustrated in Figure 6, is as follows:

The coal 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 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.

Increments shall be taken as a complete section across the strip. The width of each cross-section shall be not less than 3 times the nominal top size of the coal.

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 coal 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 may be achieved with fewer increments.

7 Reduction

7.1 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.

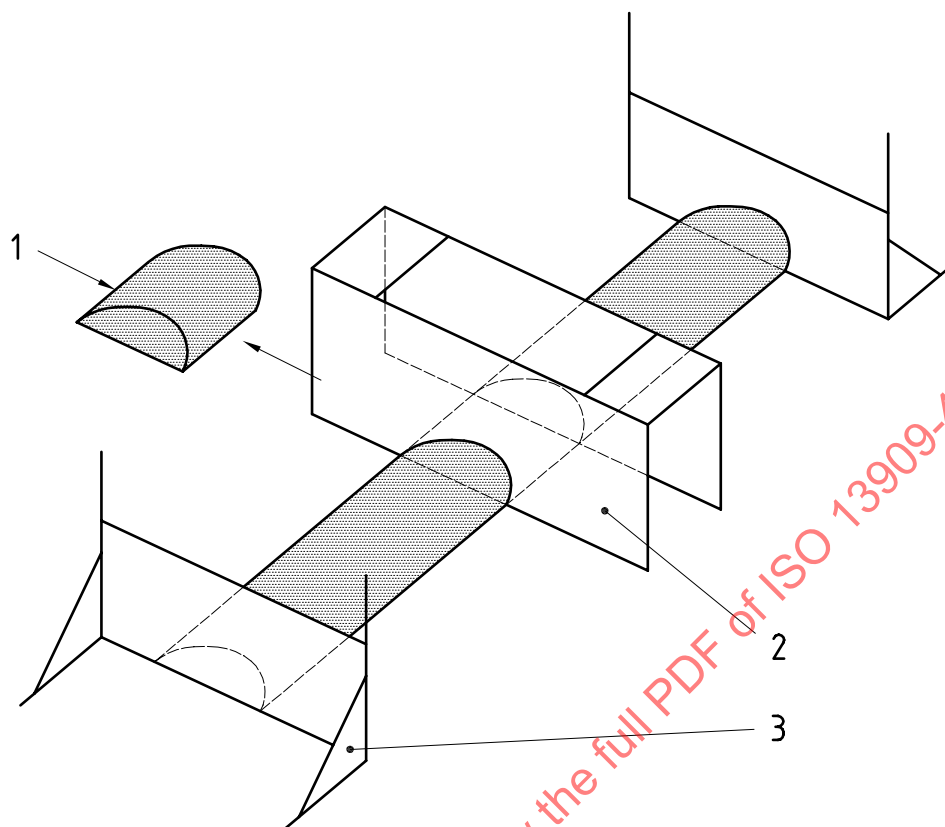
The mill settings should be checked regularly by sieving and determining the nominal top size produced by each mill.

7.2 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, which might contaminate succeeding samples, shall be minimized. Heating of the sample and airstream effects shall be minimized, particularly where the sample is to be used for the determination of total moisture, calorific value, and for coking tests.

There shall be no contact between the metal surfaces in order to avoid localized heating of the sample. 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 but the effect will depend on the speed range.

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

**Key**

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

Figure 6 — Strip-mixing and splitting method

8 Mixing

Mixing is not possible where samples or increments are flowing through any form of preparation system and is therefore restricted to off-line preparation.

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 may also result in loss of moisture.

One method that can be used is to pour the sample through a riffle (6.3.1) or a container-type sample divider [see Figure 2 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 may be useful at the final stage of preparation of test samples.

9 Air-drying

The sample is spread in a thin layer and allowed to attain equilibrium with the atmosphere at ambient temperatures.

The coal layer shall not exceed a thickness of 1,5 times the nominal top size of the coal or a loading of 1 g/cm², whichever is the greater.

The recommended times to attain equilibrium at different ambient temperatures up to 40 °C are given in Table 2. The times recommended in Table 2 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 coals susceptible to oxidation.

Table 2 — Recommended drying times for air-drying

Drying temperature °C	Drying time h
20	Preferably not to exceed 24
30	Preferably not to exceed 6
40	Preferably not to exceed 4

Drying temperatures above 40 °C shall not be used on samples likely to be susceptible to oxidation or if the sample is to be 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.

For drying temperatures above normal ambient, 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 will depend on the drying temperature. For example, 3 h is normally sufficient if the sample has been dried at 40 °C.

10 Preparation of samples for specific tests

10.1 Types of test samples

The following types of test sample may be prepared:

- a) samples for determination of total moisture only;
- b) samples for general analysis only (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 Hardgrove Grindability Index.

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

10.2 Preparation of samples for determination of total moisture only

10.2.1 General

The test sample for the determination of total moisture shall be prepared to meet the requirements of ISO 589. If air-drying is performed at any stage of preparation, the percentage loss in mass shall be recorded and included in the calculation of total moisture as specified in 8.2 of ISO 589:1981.

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 coal and the reduction and division equipment and procedures used.

A sample may be prepared as a test sample with or without preliminary air-drying but the preferred procedure is to divide the uncrushed sample to a mass not less than that given in Table 1 and air-dry it. The sample is then crushed and divided to the required test sample state.

Preliminary air-drying may be necessary in order to minimize moisture loss in any subsequent reduction/division stages. Reduction/division shall only be carried out prior to air-drying if it does not result in moisture bias.

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 as specified in ISO 589.

If the mass of the uncrushed sample is so large that air-drying is impracticable, the samples shall be crushed and divided before air-drying. Crushing shall be kept to the minimum necessary to allow division to a manageable mass. The absence of bias due to these procedures shall be checked using the procedures given in ISO 13909-8.

If the particle size of the sample is so large that the mass given in Table 1 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.

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 7.

10.2.2 Storage

Precautions shall be taken to minimize changes of moisture content during preparation due to the use of unsuitable containers and to evaporation during handling. All samples for moisture determination shall be kept in moisture-tight 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 to overcome these problems (see ISO 13909-2 or ISO 13909-3).

Weigh the samples stored for moisture determination before storage to allow determination of any moisture change that takes place during storage.

10.2.3 Sample reduction

Care shall be taken to minimize changes in moisture content during reduction, by using equipment in which there is no appreciable heating and by reducing to a minimum the flow of air through the mill. The uncrushed sample shall be air-dried prior to reduction, unless it can be shown that crushing causes no relevant bias.

10.2.4 Sample division

When carrying out sample division prior to air-drying of a sample or increment, care shall be taken to minimize changes of 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 which are too moist to flow through a sample divider and for which it is also impossible to air-dry the entire sample, it may be necessary to divide the sample by collecting increments from a flattened heap (6.3.2) or by strip-mixing and splitting (6.3.3). This divided sample is then air-dried.

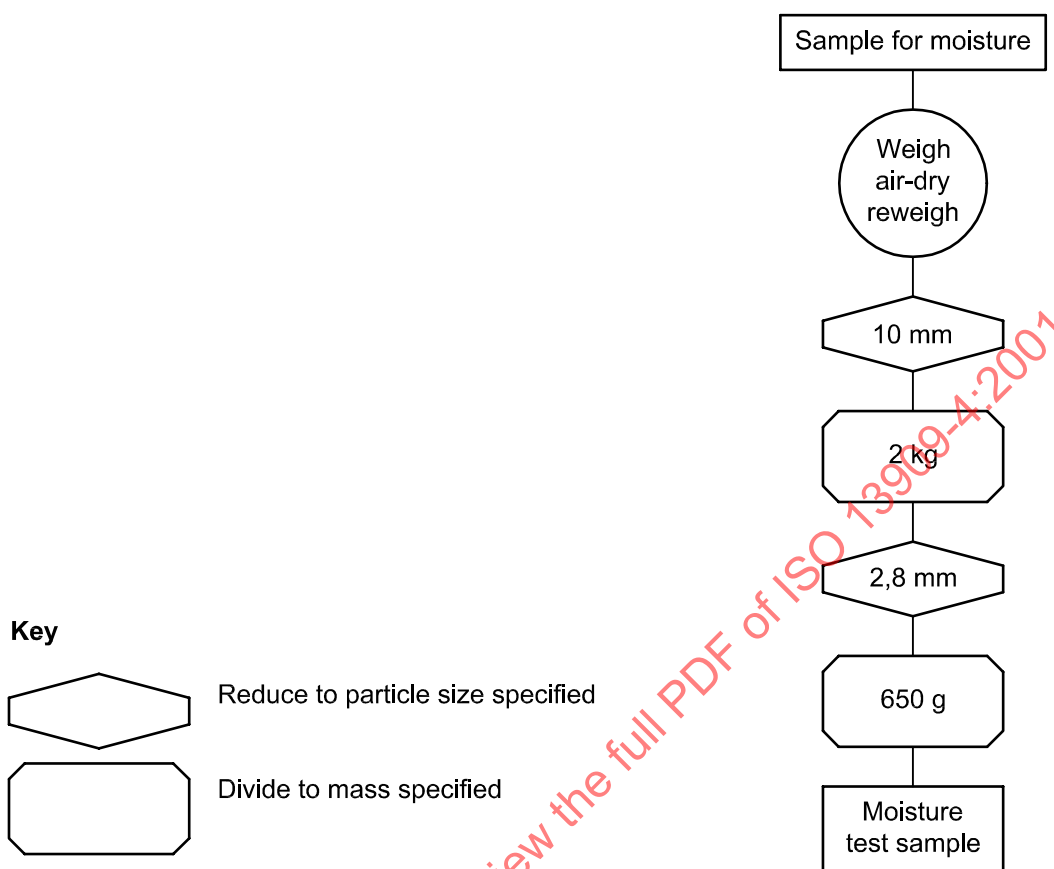


Figure 7 — Example of a two-stage moisture-test sample preparation

10.3 Preparation of samples for general analysis only

10.3.1 General

The objective of general-analysis sample preparation is to prepare a test sample which will pass a sieve of nominal size of openings 212 μm conforming to the requirements of ISO 3310-1. The mass of the test sample depends on the analysis required but is 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.

10.3.2 Air-drying

Air-drying in connection with preparation for general analysis is only carried out to ensure that the coals 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.

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 to be used for the determination of calorific value, coking or swelling properties, the maximum drying temperature shall be 40 °C. If drying can be avoided during the first stage of preparation, the procedure can be simplified.

10.3.3 Reduction and division

Reduction and/or division of increments takes place in accordance with the requirements of clause 6 and clause 7 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 may 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 errors due to sample division.

NOTE 2 It may 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.

The sample is reduced and divided, in one or two further stages, to the nominal top size and mass required for the test sample and is 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 coal is required. For manual division, a riffle may be used, or the sample 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 the scheme is given in Figure 8.

10.4 Common samples

10.4.1 General

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 in accordance with 10.4.2.

If the common sample is visibly wet and it is impossible to air-dry the entire sample, use a manual method in accordance with 10.4.3.

Examples of schemes for the preparation of separate test samples for moisture and for general analysis from a common sample are given in Figure 9. Sometimes a single test sample may be prepared for both moisture 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 preparation of the general-analysis test sample. Each part shall fulfil the requirements for minimum mass specified in Table 1 and further treatment of the parts shall be in accordance with 10.2 and 10.3 respectively.

10.4.2 Extraction of moisture sample by mechanical division

The extraction of the moisture sample may be carried out at any convenient stage of the preparation procedure consistent with the requirements of 10.2.3. Prior to extraction, the sample shall be treated in accordance with 10.2 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 of total moisture content as specified in ISO 589.