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Solid mineral fuels — Mechanical sampling from moving streams —

Part 1: Coal

*Combustibles minéraux solides — Échantillonnage mécanique sur
minéraux en mouvement —*

Partie 1: Charbon



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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.

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.

International Standard ISO 9411-1 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

ISO 9411 consists of the following parts, under the general title *Solid mineral fuels — Mechanical sampling from moving streams*:

- *Part 1: Coal*
- *Part 2: Coke*

Annexes A, B, C and D form an integral part of this part of ISO 9411. Annexes E and F are for information only.

Introduction

Mechanical sampling from moving streams of coal has increasingly replaced manual methods of coal sampling and, consequently, the recommended practices for mechanical sampling provided by International Standards have come under review.

Solid mineral fuels — Mechanical sampling from moving streams —

Part 1: Coal

1 Scope

This part of ISO 9411 provides recommended practices for the mechanical sampling of solid mineral fuels from moving streams. It has been developed primarily to give guidance on falling-stream samplers. This part of ISO 9411 is applicable to the mechanical sampling and preparation of coal samples for the determination of total moisture, general analysis, including physical and chemical tests, and size analysis. ISO 9411-2 deals with the methods of sampling of coke and preparation of coke samples in a similar way.

NOTE 1 The methods of analysing or testing the test samples of coal obtained are described in other International Standards.

In this part of ISO 9411 the basic requirements, together with typical examples of applications, are described for guidance in the design, installation and operation of systems for mechanical sampling and mechanical preparation of samples.

Notes on the operation of mechanical sampling systems are given in annex A.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9411. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9411 are encouraged to investigate the possibility of applying the most recent editions of the

standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 3310-1:1990, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*.

3 Definitions

For the purposes of this part of ISO 9411, the following definitions apply.

3.1 accuracy: The closeness of agreement between an observation and the “true” value.

NOTE 2 The accuracy of a result should not be confused with its precision.

3.2 air drying: The process of partial drying of the sample to bring its moisture content near to equilibrium with the atmosphere in the area in which further reduction and division of the sample are to take place.

3.3 bias: A systematic error which leads to results which are persistently higher or persistently lower than the “true” value.

3.4 coefficient of variation: The standard deviation, s , expressed as a percentage of the absolute value of the arithmetic mean, $|\bar{x}|$.

NOTE 3 This term is usually designated as v .

$$v = \frac{s}{|\bar{x}|} \times 100$$

3.5 common sample: A sample collected for more than one intended use.

3.6 constant-mass division: The method of increment or sample division in which the portions retained from individual increments, partial samples or gross samples are of uniform mass.

3.7 correlation coefficient: A measure of the degree of correlation between the members of paired sets.

3.8 cut: An increment taken by a sample divider.

3.9 cutter: A mechanical device which takes a single increment of coal.

3.10 divided increment: The part obtained from the division of the increment in order to decrease its mass.

NOTE 4 Such division may be done with or without prior size reduction.

3.11 duplicate sampling: A particular case of replicate sampling with only two replicate samples.

3.12 error: The difference between the observation and the "true" value, which can be designated systematic (bias) or random.

NOTE 5 The procedures of sampling, sample preparation and analysis are not perfect and the observations will be dispersed about the "true" values.

3.13 fixed-rate division: The method of increment or sample division in which the portions retained from individual increments, partial samples or gross samples have a mass proportional to the mass of the increment, partial sample or gross sample.

3.14 general analysis test sample: A sample, crushed to pass a sieve of nominal size of openings 212 µm complying with ISO 3310-1, used for the determination of most physical and chemical characteristics of coal.

3.15 gross sample: The quantity of coal consisting of all the increments or partial samples taken from a sampling unit, either in the condition as taken or after the increments have been individually reduced and/or divided.

3.16 increment: A portion of coal collected in a single operation of the sampling instrument.

3.17 laboratory sample: A sample prepared from the gross or partial sample as delivered to the laboratory, and from which further samples are prepared for test purposes.

3.18 lot: A discrete quantity of coal for which the overall quality to a particular precision needs to be determined.

3.19 manual sampling: The collection of increments by human effort.

3.20 mass-basis sampling: The taking of increments in uniform mass intervals throughout the sampling unit.

NOTE 6 Each increment or divided increment constituting the partial sample or the gross sample should be of almost uniform mass.

3.21 mechanical sampling: The collection of increments by mechanical means.

3.22 nominal top size: The smallest sieve in the range included in the R 20 series (see ISO 565, square hole) on which not more than 5 % of the sample is retained.

3.23 off-line sample preparation: Sample preparation performed manually or by mechanical equipment not integral with the mechanical sampling system.

3.24 on-line sample preparation: Sample preparation by mechanical equipment integral with the sampling system.

3.25 outlier: A result which appears to be in disagreement with others from the same coal and which arouses suspicion that there has been a mistake in the sampling, sample preparation or analysis.

3.26 partial sample: A sample representative of a part of the whole sampling unit, constituted in order to prepare laboratory samples or test samples.

NOTE 7 Partial samples may be obtained by combining all increments from a sampling unit into two or more sets, each set being composed of consecutive increments, the number of which need not be the same in all sets (see figure 1).

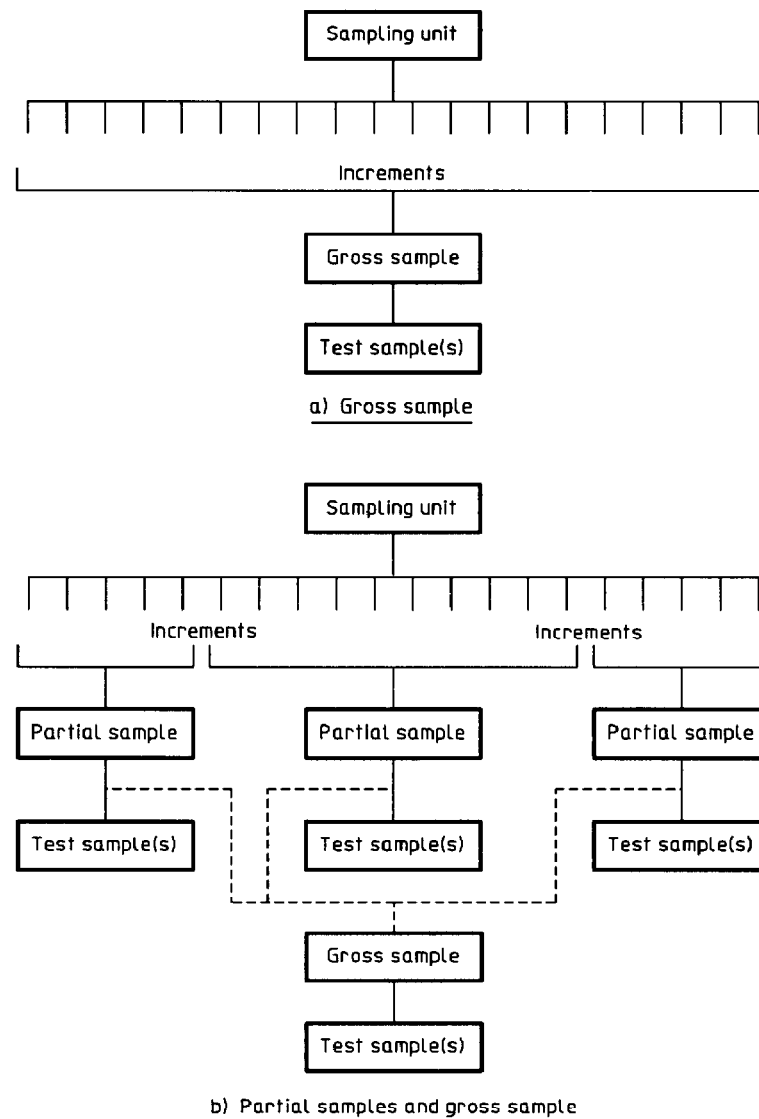


Figure 1 — Examples of the constitution of gross sample and/or partial samples

3.27 precision: A measure of the extent to which observations within a set agree with each other.

NOTE 8 A determination may be made with great precision and the standard deviation of a number of determinations on the same sampling unit may therefore be low, but the results will be accurate only if they are free from bias.

3.28 primary increment: The increment taken at the first stage of sampling, prior to any sample division and/or sample reduction.

3.29 random error: Error that has an equal probability of being positive or negative.

NOTE 9 The mean of the random errors resulting from a series of observations tends towards zero as the number of observations increases.

3.30 range: The difference between the greatest and least values of a number of observations.

3.31 reference increment mass: The minimum average mass of an increment which should be collected when taking primary or divided increments from a coal stream with a mechanical sampler.

3.32 relevant bias: Bias that is of practical importance, whether economic, scientific, legal or social.

3.33 replicate sampling: The taking from the sampling unit of increments which are combined in rotation into different containers to give two or more samples of approximately equal mass, each being representative of the whole sampling unit.

3.34 sample division: The process in sample preparation whereby the sample is divided into separate portions, one or more of which is retained.

3.35 sample preparation: The process of bringing samples to the condition required for analysis or testing.

NOTE 10 Sample preparation covers mixing, sample division, particle size reduction and sometimes air drying of the sample, and may be performed in several stages.

3.36 sample reduction: The process in sample preparation whereby the particle size of the sample is reduced by crushing or grinding.

3.37 sampling unit: A quantity of coal, the sampling of which results in one gross sample.

NOTE 11 There may be one or more sampling units per lot.

3.38 size analysis sample: A sample taken specifically for particle size analysis.

3.39 standard deviation: The positive square root of the variance.

NOTE 12 This term is usually designated as s .

3.40 stratified random sampling: The taking of an increment at random within the mass interval or time interval determined for mass-basis sampling or time-basis sampling respectively.

3.41 test sample: A sample which is prepared to meet the requirements of a specific test.

3.42 time-basis sampling: The taking of increments in uniform time intervals throughout the sampling unit.

NOTE 13 Each increment or divided increment constituting the partial sample or the gross sample should be of a mass proportional to the flow rate of the coal stream at the time of taking the increment.

3.43 total moisture sample: A sample taken specifically for the determination of total moisture.

3.44 variance: The mean square of deviations from the mean value of a set of observations.

NOTE 14 This term is usually designated as V .

4 Establishing a sampling scheme

4.1 General procedure

The following shall be the general procedure for establishing a sampling scheme.

- a) Define the quality parameters to be determined and the types of samples required.
- b) Define the lot.
- c) Define the precision required.
- d) Determine the variability of the coal (see 4.5.2) and establish the number of sampling units u (see 4.5.4) required to attain the desired precision and the minimum number of increments n (see 4.5.5). Methods for determining variability are given in annex B.
- e) Decide whether to use time-basis or mass-basis sampling (see clause 5) and define the sampling intervals in minutes or tonnes respectively.
- f) Ascertain the nominal top size of coal for the purpose of determining the reference increment mass.
- g) Determine the method of combining the increments into gross samples or partial samples (see 6.2) and the method of sample preparation.
- h) Determine the minimum mass of increments at each stage of division (see 6.5.2).

4.2 Types of samples

It is permitted to collect either separate samples or a common sample for the determination of total moisture, for size analysis and for general analysis and other tests.

4.3 Division of lots

A lot may be a single sampling unit or a series of sampling units, for example, coal despatched or delivered over a period of time, a ship load, a train load, a wagon load, or coal produced during a certain period, e.g. a shift.

A sampling unit may be represented by a gross sample or by two or more partial samples.

A division of a lot into a number of sampling units or the representation of a sampling unit by a number of

partial samples may be necessary, in order to improve the precision of the results.

For lots sampled over long periods, it may be convenient to divide the lot into a series of sampling units, obtaining a gross sample for each sampling unit.

If the quality is to be determined for parts of a lot in addition to that of the lot as a whole, the lot shall be sampled as two or more part-lots and each part-lot shall be treated as a lot in its own right. The resulting samples shall conform to the requirements for gross samples. The quality for the lot as a whole shall be calculated on the weighted average of the quality of the part-lots.

4.4 Basis of sampling

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

4.5 Precision of sampling

4.5.1 General

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

It is possible to design a sampling scheme by which, in principle, an arbitrary level of precision can be achieved. However, limits will be imposed on the precision that should be aimed for because of practical considerations.

NOTE 15 The required overall precision on a lot is agreed upon by the parties concerned.

The theory of the estimation of precision is given in annex B. The following equation is derived from equation (B.5):

$$P_L = \pm 2 \times \left(\frac{\frac{V_I}{n} + V_{PT}}{u} \right) \quad \dots (1)$$

where

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

V_I is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of increments to be taken from each sampling unit;

u is the number of sampling units in the lot.

If the quality of a coal of a type not previously sampled is required, then in order to devise a sampling scheme assumptions will have to be made about the variability (see 4.5.2). The precision actually achieved for a particular lot by the scheme devised can be measured by the procedures given in annex B.

If the same type of coal is sampled regularly, sampling schemes can be laid down using data derived from previous sampling. The procedures given in 4.5.5 can be used to devise the optimum scheme, thus keeping the sampling costs to a minimum.

4.5.2 Primary increment variance

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

For many coals, the variability for ash is higher than that for moisture and hence, for the same precision, the number of increments for ash will be adequate for moisture. If, however, a higher precision is required for moisture, the relevant primary increment variance shall be applied for each sample.

If at all possible, the primary increment variance, V_I , should be determined on the coal to be sampled using one of the methods described in annex B. If this cannot be done, V_I should be determined for a similar coal from a similar sampling system. If neither of these procedures is possible, assume an initial value of 20 for V_I and check this, after the sampling has been carried out, using one of the methods described in annex B.

NOTE 16 A method using the variogram technique is described in annex F.

4.5.3 Variance of preparation and testing

If at all possible, the variance of preparation and testing, V_{PT} , should be determined on the coal to be sampled using one of the methods described in annex C. If this cannot be done, V_{PT} should be determined for a similar coal from a similar sampling system. If neither of these procedures is possible, assume an initial value of 0,2 for V_{PT} and check this, after the preparation and testing has been carried out, using one of the methods described in annex C.

4.5.4 Number of sampling units

The number of increments taken from a lot in order to attain a certain precision is a function of the variability of the quality of coal in the lot, irrespective of the mass of the lot. When designing sampling schemes, the measure of variability, i.e. the primary increment variance, often has to be determined from the results of sampling relatively small sampling units. This may be a serious underestimation of the variability of the whole lot, for example, when segregation occurs during transport of very large masses of coal, during stockpiling, or when coal is despatched or received over extended periods during which long-term changes in quality may occur. Therefore, lots should be divided into a convenient number of sampling units.

It is recommended that a lot be divided into a number of sampling units, u , not less than the number given in table 1.

Table 1 — Recommended minimum number of sampling units in a lot

Mass of lot tonne	Number of sampling units
< 5 000	1
5 001 to 20 000	2
20 001 to 45 000	3
45 001 to 80 000	4
80 001 to 125 000	5
125 001 to 180 000	6
180 001 to 245 000	7

This number may be increased so that the sampling unit coincides with a convenient mass or time, or because it is necessary in order to achieve the desired precision (see 4.5.5). It may also be necessary to increase the number of sampling units, if ambient conditions make it necessary to limit the time over which the sample is collected.

The quality of the lot shall be calculated as the weighted average of the values found for the sampling units.

4.5.5 Number of increments per sampling unit

As stated in 4.5.1, the precision is determined by the variability of the coal, the number of increments and sampling units and the preparation and testing variance. By transposing equation (1) it can be shown that the number of increments for a desired precision in a lot can be estimated from the following equation:

$$n = \frac{4V_I}{uP_L^2 - 4V_{PT}} \quad \dots (2)$$

If n is a practicable number, the initial scheme is established. However, if n is less than 10, take 10 increments per sampling unit.

If n is impracticably large, increase the number of sampling units either by

- increasing u to a number corresponding to a convenient mass or time, recalculating n and continuing this until n is a practicable number, or
- deciding on the maximum practicable number of increments per sampling unit, n_1 , and calculating u from equation (3) [derived from equation (B.7)]:

$$u = \frac{4V_I + 4n_1V_{PT}}{n_1P_L^2} \quad \dots (3)$$

If necessary, adjust u upwards to a convenient number and recalculate n .

This method of calculating the number of increments required per sampling unit for a certain precision from the primary increment variance and the preparation and testing variance will generally give an overestimate of the required number. This is because it is based on the assumption that the quality of coal varies in a random manner. In addition, because a certain amount of preparation and testing is required when measuring the increment variance, the preparation and testing errors are included more than once.

The designer of a sampling system shall cater for the worst case anticipated, and will use higher values for V_I than may actually occur when the system is in operation. On implementing a new sampling scheme, it is therefore recommended that a check on the actual precision being achieved be carried out using the methods described in annex B. The number of increments/sampling units for the type of coal in

question can then be adjusted according to that procedure, so that the required precision can be achieved with the minimum sampling cost.

4.5.6 Examples

EXAMPLE 1

The lot is 80 000 tonnes delivered in 1 000 tonne train loads and the required precision, P_L , is $\pm 0,25\%$.

The quality variation is known and the following values have been determined:

$$V_i = 0,5, \quad V_{PT} = 0,05.$$

- a) Initial number of sampling units

From table 1, $u = 4$

Therefore, take 4 sampling units of 20 000 tonnes each.

- b) Number of increments per sampling unit

Using equation (2)

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

Therefore, take 4 sampling units of 40 increments each.

EXAMPLE 2

The lot is 100 000 tonnes delivered as 5 000 tonnes/day over two shifts and the required precision, P_L , is $\pm 0,25\%$.

As the quality variation is unknown, assume $V_i = 20$ and $V_{PT} = 0,20$.

From table 1, $u = 5$, but for convenience take daily samples i.e. $u = 20$, in order to avoid storage of samples overnight.

Using equation (2)

$$n = \frac{4 \times 20}{(20 \times 0,25^2) - (4 \times 0,2)} = 178$$

This number is too large for a single sample, therefore try taking shift samples, e.g. $u = 40$.

$$n = \frac{4 \times 20}{(40 \times 0,25^2) - (4 \times 0,2)} = 47$$

It would then be sensible to take 48 increments per shift (1 every 10 min).

EXAMPLE 3

The lot is 8 000 tonnes in a single load and the required precision, P_L , is $\pm 0,5\%$.

The quality variation is known and the following values have been determined:

$$V_i = 15, \quad V_{PT} = 0,20.$$

From table 1, $u = 2$

$$n = \frac{4 \times 15}{(2 \times 0,5^2) - (4 \times 0,2)} = \frac{60}{-0,3} = -200$$

A value of infinity or a negative number indicates that the errors of preparation and testing are such that the required precision cannot be achieved with this number of sampling units.

It could be decided that 50 increments is the maximum practicable number in a sampling unit, and from equation (3):

$$u = \frac{(4 \times 15) + (4 \times 50 \times 0,2)}{50 \times 0,5^2} = 8$$

This gives a practical sampling method of dividing the lot into 8 sampling units and taking 50 increments from each.

4.6 Mass of primary increment

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

$$m = \frac{CA}{3,6S} \times 10^{-3} \quad \dots (4)$$

where

- C is the flow rate, in tonnes per hour;
- A is the cutting aperture, in millimetres;
- S is the cutter speed, in metres per second.

Since the design of the cutter shall conform to the requirements of 8.7 and 8.8, whatever the flow rate at the time of taking an increment, its extraction from the coal stream will be unbiased. The mass of increment, of itself, is irrelevant when considering extraction bias.

Within the constraints of coal nominal top size, cutter speed/aperture and coal flow rate, mechanical sampling increments/systems are normally designed in terms of the mass of coal they will sample, i.e. in

crement mass. In order to give guidance to system designers, this part of ISO 9411 specifies a reference increment mass.

In most mechanical systems, the mass of primary increment collected [see equation (4)] will greatly exceed that necessary to make up a sample of the required mass. In some systems, the primary increments are therefore divided, either as taken or after reduction, in order to prevent the mass of the sample becoming excessive. The reference increment mass should be used as a guideline for the minimum average mass for primary increments or divided increments, but not as an absolute lower limit. In addition, when measuring primary increment variance (see B.2) at preliminary stages in the system design, these are examples of masses that can be used.

When the system is commissioned, the precision of the result can be estimated and adjusted (see annex B), by increasing or decreasing the number of increments in the sample, while keeping the same increment mass.

Table 2 gives values for the reference increment mass for a series of coal nominal top sizes. Reference increment masses corresponding to nominal top sizes between those given in table 2 may be estimated by interpolation.

NOTES

17 The comments on bias apply to increment extraction only. In systems with widely varying flow rates and hence, for flow-proportional increments, wide variations in increment mass, very small increments may suffer disproportionate changes in quality, e.g. loss in moisture. Precautions can be taken to prevent this. If such losses cannot be prevented and are found to cause relevant bias, either the flow of coal can be made more regular by such means such as buffer hoppers or a variable speed cutter (mass-basis sampling) should be used. Alternatively, increments can themselves be retained temporarily in a buffer hopper until there is sufficient mass to ensure bias-free passage through an on-line preparation system. On no account should a primary sampler, in a time-basis system or a mass-basis system, be switched off at low flow rates to avoid low mass increments.

18 The reference increment masses given in table 2 apply only to primary increments or to the divided increments resulting from their subsequent preparation. They should not be applied to the masses of individual cuts of sample dividers, either on individual increments or samples. Mass restrictions applying to division are given in 6.5.2 (divided increment) and 6.8 (sample).

Table 2 — Reference increment mass

Nominal top size of coal mm	Reference increment mass kg
300	100
200	25
150	15
125	10
90	5
63	3
45	2
31,5	1
22,4	0,75
16,0	0,50
11,2	0,25
8,0	0,15
5,6	0,10
4,0	0,10
2,8	0,10

4.7 Minimum mass of gross sample for general analysis/moisture determination

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

The minimum mass of gross sample is dependent on the nominal top size of the coal, the precision required for the parameter concerned and the relationship of that parameter to particle size. Such a relationship applies at all stages of preparation. The attainment of this mass will not, in itself, guarantee the required precision. This is also dependent on the number of increments in the sample and their variability (see 4.5.5).

Values for the minimum mass of gross sample or of the sample after division are given in table 3.

NOTE 19 The values in table 3 are based on a division variance of 0,01 with respect to ash. 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. When a coal is regularly sampled under the same circumstances, the precision obtained should be checked (see annexes B and C) and the masses adjusted accordingly. However, the masses should not be reduced below the minimum specified in the relevant standards for methods of analysis.

Table 3 — Minimum mass of gross sample/minimum mass of sample after division

Nominal top size of coal	General analysis and common samples	Moisture samples
mm	kg	kg
300	15 000	3000
200	5 400	1 100
150	2 600	500
125	1 700	350
90	750	125
63	300	60
45	125	25
31,5	55	10
22,4	32	7
16,0	20	4
11,2	13	2,50
8,0	6	1,50
5,6	3	1,20
4,0	1,50	1,00
2,8	0,65	0,65
2,0	0,25	—
1,0	0,10	—

4.8 Size analysis

Within the scope of this part of ISO 9411, the coals to be sampled will exhibit large differences in size, size range and size distribution. In addition, the parameters to be determined (percentage retained on a particular sieve, mean size, etc.) may differ from case to case. Furthermore, when sample division is applied, division errors shall be taken into account, whereas they are non-existent if sizing is performed without any preceding division.

These factors should be taken into account when applying the techniques for calculating numbers of increments for a particular precision (see 4.5). In the absence of any information on increment variance etc., it is recommended that 25 increments be taken initially to form the gross sample.

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

NOTE 20 Due to probable size degradation, mechanical sampling may not be applicable for size analysis. Trial tests should be carried out using the method given in annex D to determine the degree of degradation.

The minimum mass of gross sample for size analysis is given in table 4. The masses have been calculated

on the basis of the precision of the determination of oversize, i.e. the coal above the nominal top size. Precision for other size fractions will normally be better than this.

5 Methods of sampling

5.1 General

Sampling should be carried out by systematic sampling either on a time basis or on a mass basis, or by stratified random sampling. For operational and economic reasons, time-basis sampling is the preferred method.

If possible, sampling should be performed from a stream, the flow rate and cross-section of which are substantially uniform. The sampling ratio shall be constant over the entire range of the feed rates to the system. The sampling ratio is the mass of retained sample per unit mass of the feed to the primary sample cutter.

With mechanical sampling devices, the primary increment mass may be considerably larger than the reference increment mass. A system of primary increment division may be necessary to allow division of the increment to a manageable mass (see 6.5).

Coincidence between variations in flow rate, quality or segregation and the operation of sampling devices is a potential source of bias and care needs to be exercised to prevent such conditions from occurring.

Table 4 — Minimum mass of gross sample for size analysis

Nominal top size of coal	Minimum mass of gross sample	
	Precision of $\pm 1\%$ absolute	Precision of $\pm 2\%$ absolute
mm	kg	kg
300	54 000	13 500
200	16 000	4 000
150	6 750	1 700
125	4 000	1 000
90	1 500	400
63	500	125
45	200	50
31,5	65	15
22,4	25	6
16,0	8	2
11,2	3	0,70
8,0	1	0,25
5,6	0,50	0,25
4,0	0,25	0,25
2,8	0,25	0,25

5.2 Time-basis sampling

5.2.1 Method of taking primary increments

Each increment shall be taken by a single motion or by a complete cycle of the sampling device, so that a full cross-section of the coal stream is taken. The speed of the cutter shall be constant throughout the sampling of the entire sampling unit (see 8.8.2.2).

Primary increments shall be taken at preset equal time intervals throughout the lot or sampling unit. If the calculated number of increments has been taken before the handling has been completed, additional increments shall be taken at the same interval until the handling operation is completed.

5.2.2 Sampling interval

The time interval T , in minutes, between taking increments by time-basis sampling shall be calculated from the following equation:

$$T \leq \frac{60Q}{Gn} \quad \dots (5)$$

where

- Q is the mass, in tonnes, of sampling unit;
- G is the maximum flow rate on the conveyor belt, in tonnes per hour;
- n is the number of primary increments in the gross sample (see clause 4).

When the handling time of sampling units varies, special consideration shall be given to the shortest handling time anticipated, to ensure that the time interval is practical and that the requisite number of increments is obtainable and can be processed in the handling time available.

In order to minimize the possibility of any bias being introduced, a random start within the first sampling interval is recommended.

5.2.3 Mass of increment

The mass of the primary increment corresponding to the average flow rate (total mass/operating time) of the coal stream shall be not less than the reference increment mass (see table 2).

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

5.3 Mass-basis sampling

5.3.1 Method of taking primary increments

Each increment shall be taken by a single motion or by a complete cycle of the sampling device, so that a full cross-section of the coal stream shall be taken.

Either a fixed or variable speed cutter may be used (see 5.3.3).

The required number of increments shall be taken by sampling at a preset mass interval. This interval shall not be changed during the sampling of the sampling unit.

If the calculated number of increments has been taken before the handling has been completed, additional increments shall be taken at the same mass interval until the handling operation is completed.

5.3.2 Sampling interval

The increments shall be spread uniformly on a tonnage basis throughout the mass of the lot or the sampling unit.

The mass interval m_i , in tonnes, between taking increments by mass-basis sampling shall be determined from the following equation:

$$m_i \leq \frac{Q}{n} \quad \dots (6)$$

where

- Q is the mass, in tonnes, of the sampling unit;
- n is the number of primary increments in the gross sample.

If the lot is to be divided into sampling units, the mass of the sampling unit should be chosen so that the requisite number of increments to be taken from the sampling unit is obtainable and can be processed in the handling time available.

The mass interval between increments shall be equal to or smaller than that calculated from the number of increments specified in 4.5.5, in order to ensure that the number of increments will be at least the minimum number specified.

In order to minimize the possibility of introduction of bias, a random start within the first sampling interval is recommended.

5.3.3 Mass of increment

The masses of the individual primary or divided increments added to the gross sample or partial sample shall be almost uniform, i.e. the coefficient of variation shall be less than 20 %, and there should be no correlation between the flow rate at the time of taking the increment and the mass of the increment. The method for determining whether or not these criteria have been met is given in annex E. These criteria may be achieved by either of the following procedures.

- a) Taking primary increments of almost uniform mass using a variable speed cutter whose cutter speed is constant while cutting the stream but can be regulated, increment by increment, in proportion to the flow rate of the coal at the point of sampling. The average increment mass shall be not less than the reference increment mass (see 4.6). This is the preferred procedure.
- b) Taking primary increments using a fixed speed cutter, with subsequent division of the individual increments, if necessary, to almost uniform mass at a practical stage prior to the constitution of the partial or gross sample. The mass of the primary increment taken at the average flow rate of the coal stream shall be not less than the reference increment mass (see 4.6).

5.4 Stratified random sampling

5.4.1 General

Cyclical variations in coal quality may occur during systematic sampling, i.e. time-basis or mass-basis sampling. Whilst every effort should be made to avoid such variations, it is not always practicable to eliminate the possibility that they may coincide with the taking of increments. In the case of coincidence, a bias will invariably be introduced that may be of unacceptable proportions. In such circumstances, stratified random sampling may be adopted in which for each time or mass interval, as the case may be, 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.

NOTE 21 During stratified random sampling it is possible that two increments will be collected very close together even though they are collected in different mass or time intervals. It is therefore necessary that the discharge bin of the primary sampler is large enough to accept a minimum of two primary increments at the maximum flow rate.

Stratified random sampling may also be used in circumstances where cyclical variations do not occur.

5.4.2 Time-basis stratified random sampling

The sampling interval shall be determined as in 5.2.2.

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 increment shall then be taken after the time indicated by the random number. The mass of the increment shall be proportional to the flow rate of the coal (see 5.2.3).

5.4.3 Mass-basis stratified random sampling

The sampling interval shall be determined as in 5.3.2. Prior to the start of each sampling interval, a random number between zero and the mass, in tonnes, of the sampling interval shall be generated. The increment shall be taken after the passage of that mass of coal indicated by the random number. The masses of the individual increments added to the gross sample or the partial sample shall be independent of the flow rate of the coal (see 5.3.3).

5.5 Reference sampling

Reference samples should be taken by the stopped-belt method to enable checking for bias (see D.5).

6 Methods of sample preparation

6.1 Fundamentals

6.1.1 General

The object of sample preparation is to prepare one or more test samples from the primary increments for subsequent analysis. The requisite mass and particle size distribution of the test sample depend on the test to be carried out. Sample preparation for moisture determination and general analysis usually includes division and reduction. It may also include mixing and air drying.

Increments taken by the procedure specified in clause 5 may be prepared individually to constitute test samples or combined to constitute partial samples or a gross sample, either as taken or after having been prepared by reduction and/or division.

Partial samples may either be prepared individually to constitute test samples or combined on a weighted basis to constitute a gross sample.

Examples of methods of constituting partial samples or a gross sample are given in figure 1.

NOTE 22 An on-line preparation scheme should be designed for anticipated fluctuations in flow rate and quality. However, there may be occasions when the rules in 6.4 to 6.6 appear not to be met, for example when an increment is taken at an extremely low flow rate. The rules have been laid down so that, provided they are met under normal conditions, when unusual circumstances occur they will have no significant effect on the precision or bias of the result obtained for the gross sample.

On-line sampling and sample preparation systems can be usefully implemented when dealing with a coal or coals which are known to be free from handling problems, whilst it is more prudent to adopt an off-line sample preparation system when the type or types of coal are unknown or varied in their ability to pass through the system without intermediate air drying.

However, when difficulty and handling the coal or coals being sampled is known or expected at a particular stage in sample preparation, or 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.

6.1.2 Precision of sample preparation

From equation (B.5) derived in annex B, it can be shown that the overall precision for the test result on a single gross sample is

$$P_G = \pm 2\sqrt{\frac{V_I}{n} + V_{PT}} \quad \dots (7)$$

where

- P_G is the absolute value of precision of the result obtained for the gross sample at the 95 % confidence level;
- V_I is the primary increment variance;
- V_{PT} is the variance of preparation and testing;
- n is the number of increments in the gross sample.

The procedures given in this part of ISO 9411 are designed to achieve levels for V_{PT} of 0,2 or less for both ash and moisture. Better levels should be expected when using mechanical dividers.

The errors occurring in the various stages of preparation and analysis expressed in terms of variance may be checked by the method given in annex C.

6.2 Method of constitution of a gross sample or partial samples

6.2.1 General

According to the requirements of sample preparation and testing, it shall be decided whether a gross sample is to be constituted for a sampling unit or partial samples are to be constituted for individual parts of the sampling unit. In this subclause, the constitution of partial samples or a gross sample is described.

NOTE 23 The constitution of partial samples may more properly result from scheduled breaks in the sampling of units, e.g. shift, change of producer, etc.

6.2.2 Method of constitution for time-basis sampling

Partial samples and gross samples may be formed in one of the following ways.

- a) The primary increments as taken may be combined into partial samples or a gross sample, irrespective of the variation of the masses of the primary increments.
- b) A gross sample may be prepared by the combination of partial samples, provided that the masses of the partial samples so combined are directly proportional to the mass of coal from which they were taken, in order to obtain the weighted mean of the quality characteristic for the lot.
- c) Primary increments may be divided and the gross sample or partial sample may be prepared by combining divided increments, provided that the mass of the divided increment is proportional to that of the primary increment, so that the weighted mean of the quality characteristic for the lot is retained.

6.2.3 Method of constitution for mass-basis sampling

Partial samples and gross samples may be formed in one of the following ways.

- a) When the primary increments are of almost uniform mass (see 5.3.3) they may be combined into partial samples or a gross sample, either directly as taken or after having been prepared individually at an appropriate stage by fixed-rate division.
- b) When condition a) cannot be satisfied, the primary increments as taken shall not be combined into partial samples or a gross sample.

However, increments, after having been divided individually by constant-mass division, may be combined into partial samples or a gross sample at a practical stage.

- c) Partial samples may be combined to constitute a gross sample, provided that any division prior to that constitution is by fixed-rate division.

6.3 Methods of division

6.3.1 General

Sample division can be done on-line mechanically, or off-line mechanically or manually. Whenever possible, mechanical methods should be used in preference to manual methods as the former minimize human error. Each stage of sample division may be carried out in one or more passes.

Division by coning and quartering is not recommended.

6.3.2 Mechanical division

Mechanical sample dividers are designed to extract a part of the coal in a number of increments of relatively small mass. When the required mass of the divided sample is less than that which can be obtained by one pass through the divider, the division shall be carried out either in two passes (through the same divider) or in two steps (using two sample dividers in series).

Coal which is visibly wet may not run freely, or may tend to adhere to the surfaces of sample dividers. In

such circumstances, it may be necessary to air dry the sample as described in 6.11 before sample division.

6.3.3 Manual division

Manual division is normally applied only to gross samples or partial samples and shall be used when the mechanical methods would result in loss of sample integrity, for example loss of moisture or size degradation. Manual methods may themselves result in bias, particularly if the mass of coal to be divided is large and weather conditions are unfavourable.

6.4 Principles of mechanical sample division

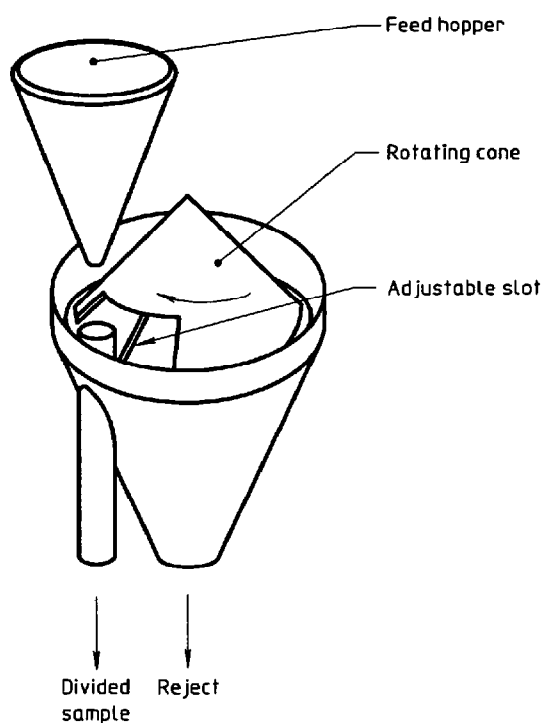
6.4.1 General

Mechanical sample division may be carried out on an individual increment, partial sample or gross sample, crushed if necessary to an appropriate nominal top size.

Division shall be either by constant-mass division or by fixed-rate division subject to the conditions set out in 6.5 and 6.6. The increments taken by sample dividers are referred to in 6.4.2 and 6.4.3 as cuts.

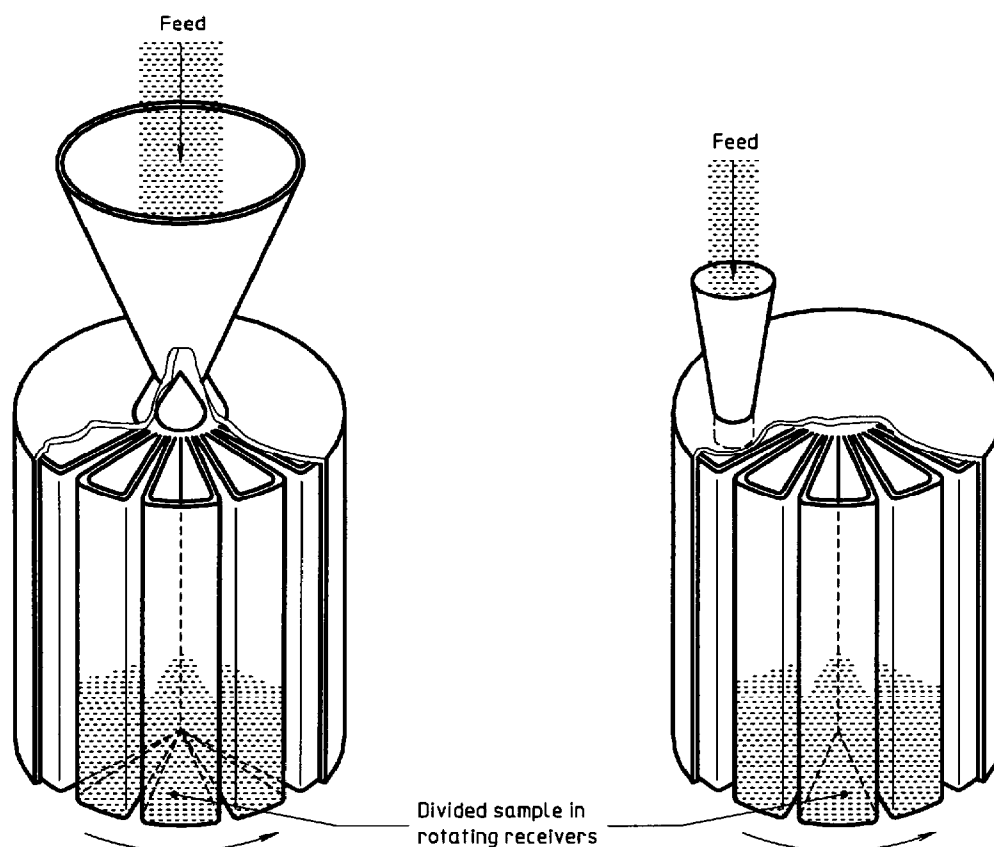
NOTE 24 The procedure recommended in 6.4.2 and 6.4.3 for fixed-rate division is the simplest to implement. However, other procedures are permitted, provided that the mass of the divided sample is proportional to the mass of the feed. For example, the number of cuts could be kept constant by making the feed rate for each division proportional to the mass of coal to be divided.

Examples of sample dividers are shown in figure 2. In addition, some types of primary samplers (figure 9) can be used.



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

a) Rotating cone type

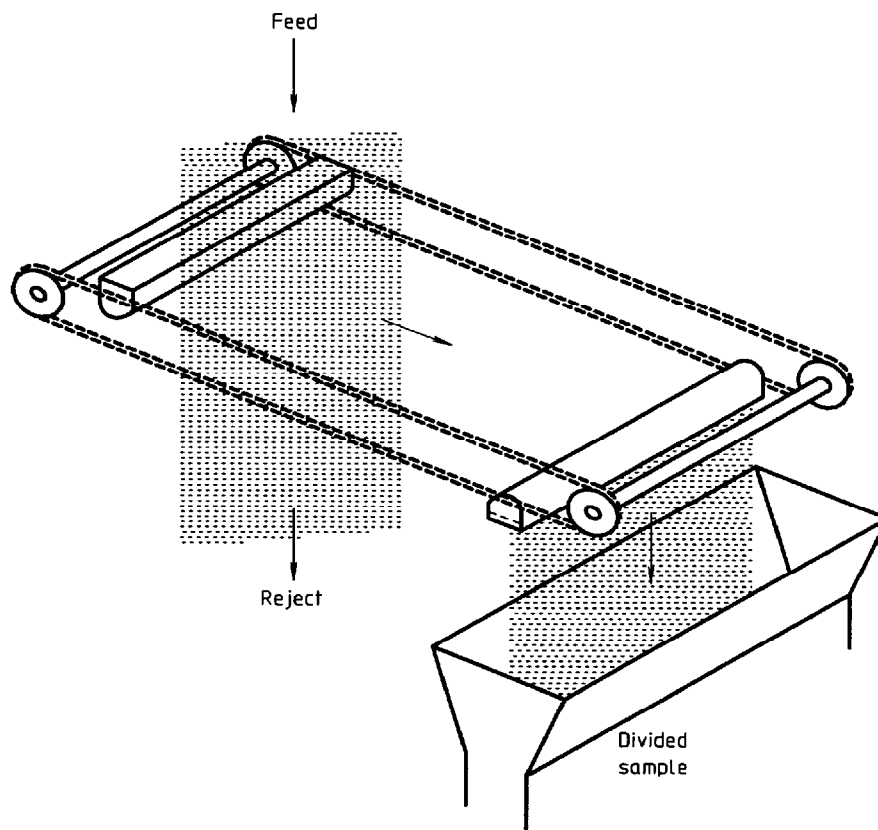


A uniform coal stream flows to the hopper and this flow is intercepted by the top edge of a number of sector-shaped containers dividing the flow into equal parts. Depending on the design, either the hopper or the containers may rotate. The machine can be controlled for the following operations:

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

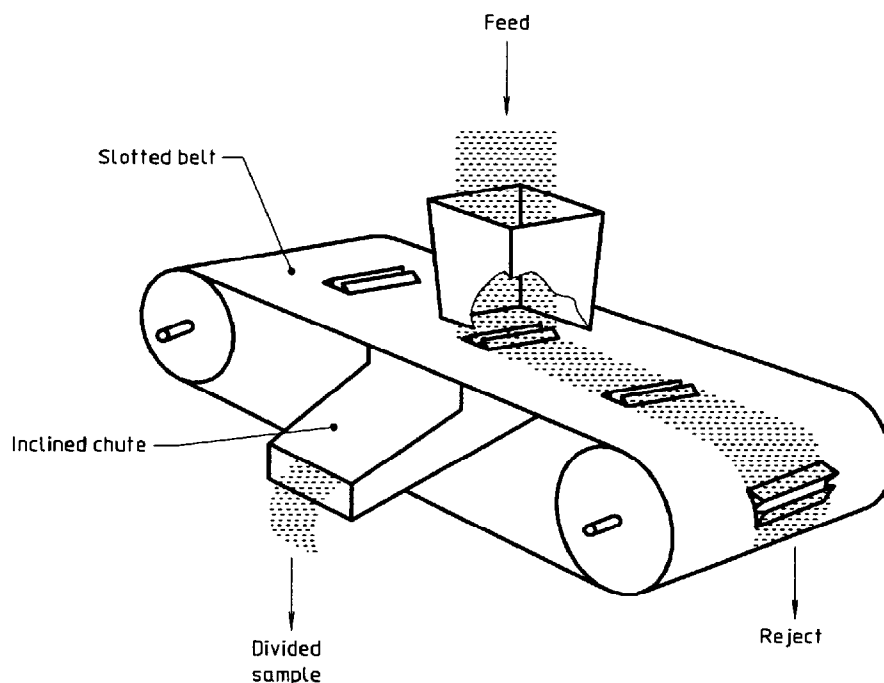
b) Container type

Figure 2 — Diagrammatic illustrations of examples of dividers



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 increments which discharge to the sample as the bucket inverts.

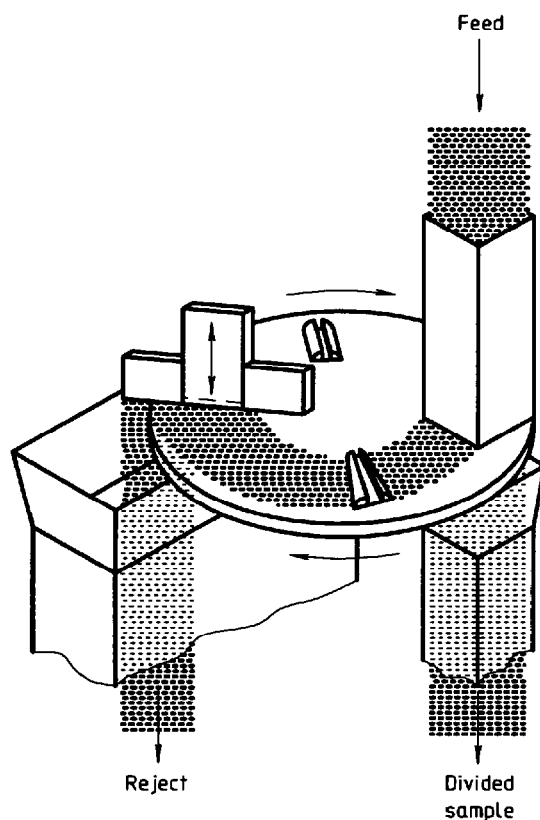
c) Chain-bucket type



An endless belt, having slots spaced at equal pitch with lips that act as cutting edges, passes below a feed chute. The belt is fed with a vibrating feeder to produce a uniform coal stream, 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 with lips type

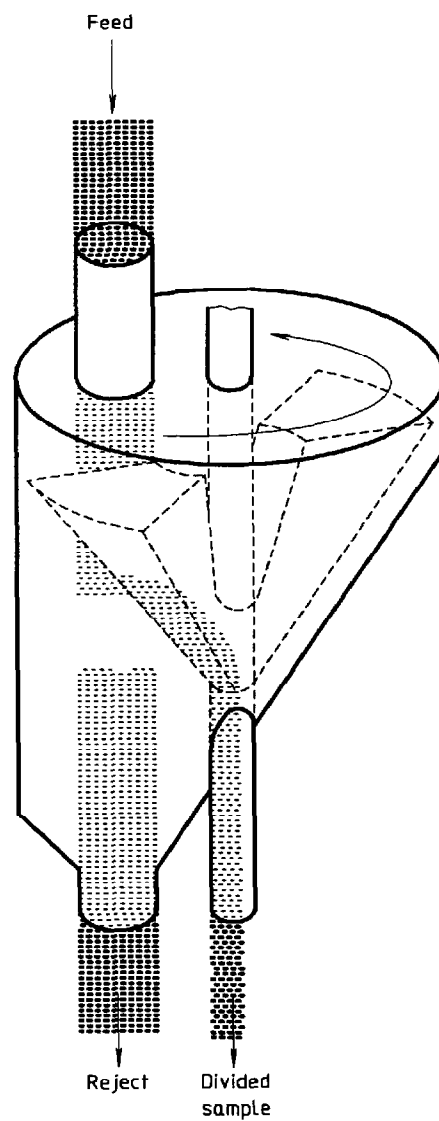
Figure 2 — Diagrammatic illustrations of examples of dividers (continued)



A flat plate, with slots spaced at equal pitch rotates beneath a feed chute. The slots shall have lips. Coal is fed into the feed chute at a controlled rate, 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.

e) Rotating plate type

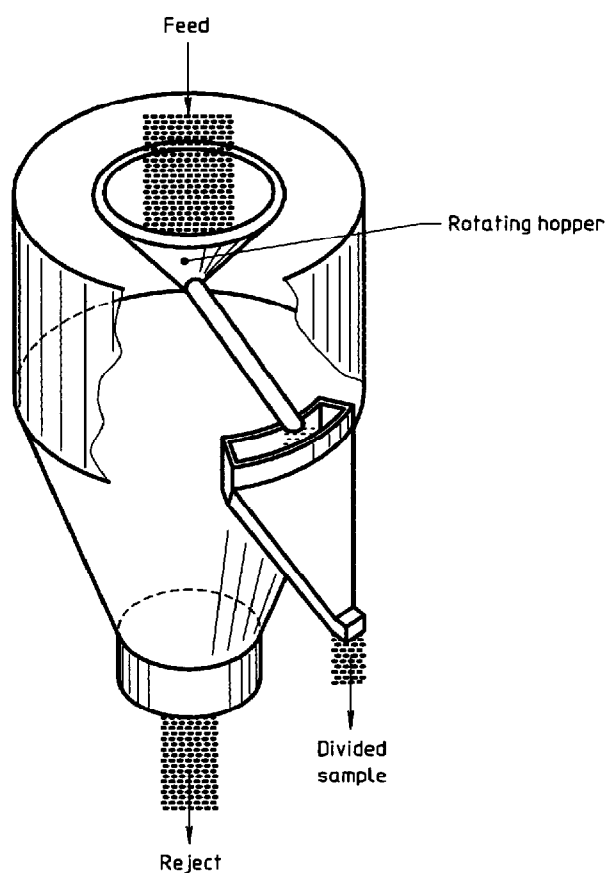
Figure 2 — Diagrammatic illustrations of examples of dividers (continued)



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 a uniform flow and to discharge via the hollow shaft.

f) Rotating chute type

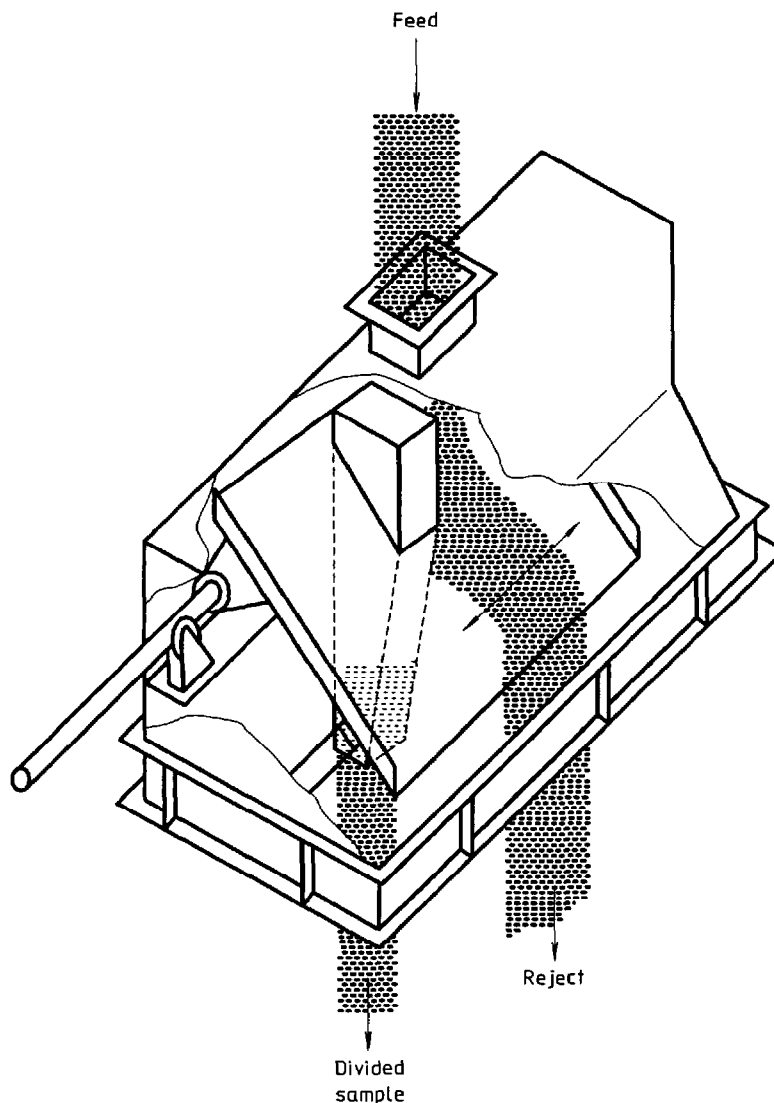
Figure 2 — Diagrammatic illustrations of examples of dividers (continued)



A rotating hopper receives a uniform 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.

g) Rotating hopper and spout

Figure 2 — Diagrammatic illustrations of examples of dividers (continued)



The cutter-chute traverses the full coal stream and diverts a portion from the stream.

h) Cutter-chute type

Figure 2 — Diagrammatic illustrations of examples of dividers (concluded)

6.4.2 Mass of cut

6.4.2.1 The cuts shall be of uniform mass throughout a division. In order to achieve this, the flow of coal to the divider shall be uniform and the cutter 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 feeder.

6.4.2.2 The cutter aperture shall be at least 3 times the nominal top size of the coal to be divided [see 8.8.1 e)]. When the coal is wet and may form a bridge, the minimum aperture shall be 30 mm.

6.4.3 Interval between cuts

6.4.3.1 Constant-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.

6.4.3.2 Fixed-rate division

The interval between taking cuts shall be constant, irrespective of the variations of masses of coal to be divided, so that divided sample masses are proportional to the original mass of increment.

6.4.3.3 Taking the first cut

In order to minimize bias, it is necessary to have a random start within the first sampling interval for the first cut on each mass of coal to be divided.

6.4.3.4 Secondary and tertiary cutters

The cycle time of a cutter shall not be evenly divisible into the cycle time of a cutter preceding it.

6.5 Mechanical division of individual increments

6.5.1 Number of cuts

The following conditions shall be met.

- a) For fixed-rate division, the minimum number of cuts from an increment of mean mass at any stage shall be six.
- b) For constant-mass division, the minimum number of cuts shall be six. The number of cuts from each increment in the sampling unit shall be equal.
- c) A minimum of one cut shall be taken for each cut from the preceding division.

6.5.2 Minimum mass of divided increment

The combined masses of all the divided increments in the sampling unit shall, at each stage, be greater than the masses given in table 3, depending on the purpose for which the sample has been taken and the nominal top size. If the combined mass of the individual increments is too low to satisfy this requirement, division is not permitted.

For a constant-mass division stage, the mass of each divided increment shall be at least the reference increment mass for the appropriate nominal top size given in table 2.

For a fixed-rate division stage, the mass of the divided increment resulting from the division of an average-mass primary increment or divided increment from the preceding stage shall be at least the reference increment mass for the appropriate nominal top size given in table 2.

NOTE 25 For mass-basis sampling schemes using a fixed-speed primary cutter, consideration should also be given to the masses achieved at the lowest anticipated flow rate. It may be necessary, when the flow rate is highly variable, to take increments which are, at the normal flow rate, considerably greater than the reference increment mass given in table 2, in order that there is sufficient coal

at the constant mass division stage when the flow rate is low.

6.6 Mechanical division of gross samples, partial samples and single increment samples

6.6.1 General

Individual increments are usually combined to form partial samples or gross samples. However, in some circumstances, for example size analysis of single increments for bias testing, individual increments are prepared and tested.

6.6.2 Number of cuts

6.6.2.1 Gross samples

The number of cuts shall be either.

- a) a minimum of 20, if thoroughly mixed; or
- b) a minimum of 60, if unmixed.

6.6.2.2 Partial samples

- a) Partial samples to be prepared to form test samples

Partial samples shall be treated in the same way as gross samples.

- b) Partial samples to be combined to form gross samples

The sum of the number of cuts on the partial samples in the sampling unit shall be at least 60. The number of cuts on each partial sample shall be proportional to the mass of that partial sample.

6.6.2.3 Single increment samples

Take a minimum of 20 cuts from each single increment sample.

6.7 Methods of manual division

6.7.1 Riffles

A riffle (see figure 3) is a non-mechanical sample divider that will, in a single pass of a sample, divide it into halves, one being retained and the other normally rejected.

Adjacent slots feed opposite receivers. The device is normally portable and is usually fed by hand for sample division.

The slot width should be at least 2,5 times, and preferably not more than 3 times, the nominal top size of the coal. Each half of the riffle shall have the same number of slots, and that number shall be a minimum of eight and preferably more. All surfaces on which the coal can rest should 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 so that the slots are never choked. When a stage of sample division requires two or more steps or passes, the sample retained at each step or pass shall be taken alternately from each side of the riffle.

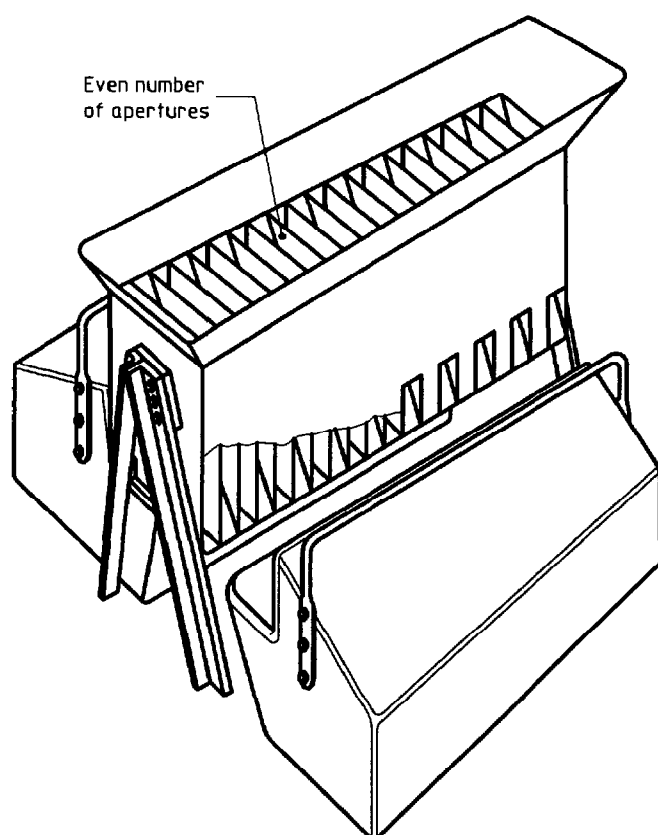
The receivers shall fit closely against the body of the riffle to minimize loss of dust.

It is necessary that care be taken to ensure that loss of moisture does not occur from a sample for moisture determination when division is carried out by a riffle. Enclosed riffles shall be used for dividing samples for moisture determination, or for dry coals to prevent loss of dust.

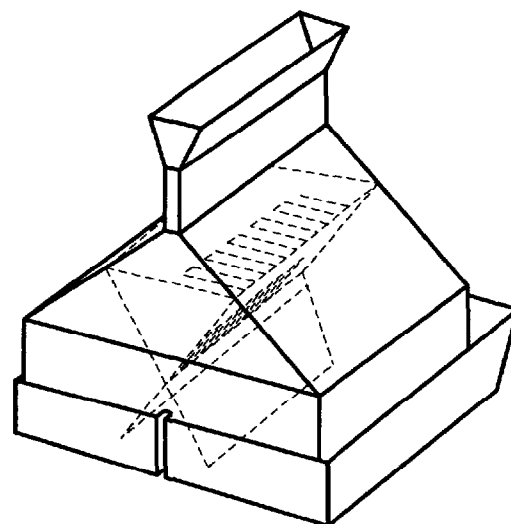
6.7.2 Flattened heap method

This procedure is illustrated in figure 4.

The sample shall be spread on a smooth, non absorbent, non-contaminating surface in the form of a rectangle of uniform thickness depending on the nominal top size as given in table 5. If the mass of coal is greater than that which can be formed into a 2 m × 2,5 m heap, two or more heaps of equal mass should be formed and separate samples taken from each heap.

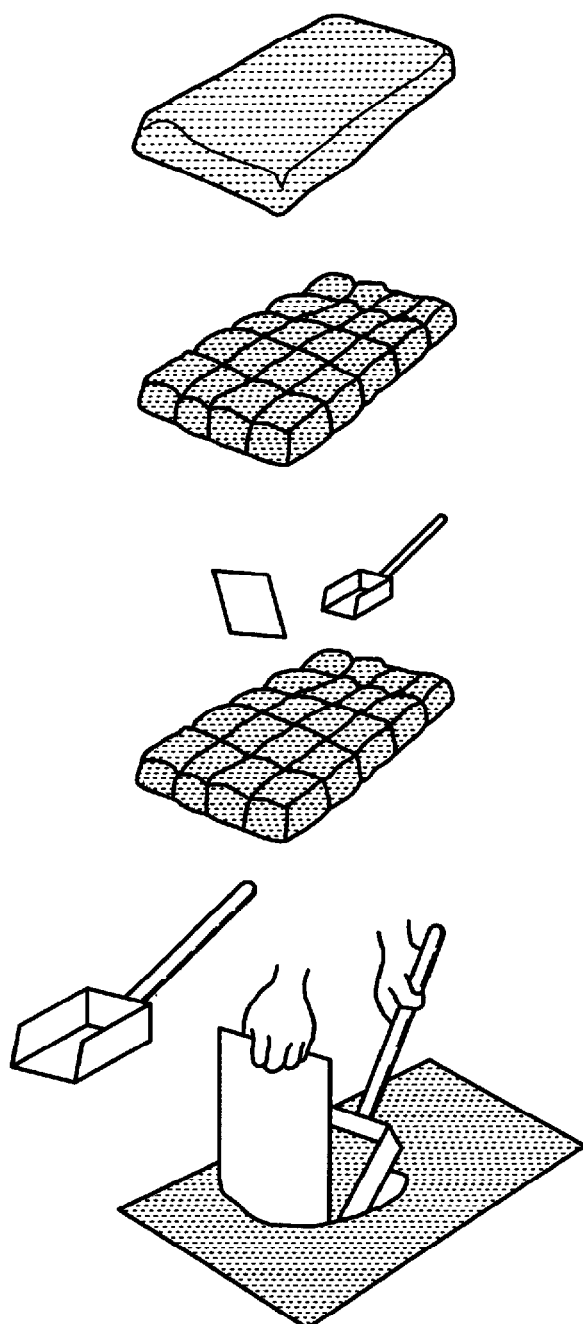


a) Open type



b) Closed type

Figure 3 — Examples of riffles



a) Spread the crushed gross sample into a rectangle with a thickness as specified in table 5.

b) Arrange into 20 equal parts, e.g. into 5 equal parts lengthwise and 4 equal parts breadthwise.

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

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

Figure 4 — Flattened heap method

Table 5 — Thickness of flattened heap

Nominal top size (NTS) mm	Thickness mm
> 45,0	(2,5 × NTS) to (3 × NTS)
45,0	110 to 120
31,5	80 to 90
22,4	55 to 65
16,0	40 to 50
11,2	30 to 35
8,0	20 to 25
≤ 5,6	15 to 20

Mark a matrix on the spread sample to give a minimum of 4 × 5 equal parts.

Take an increment, at random, from each of the 20 parts by inserting a scoop to the bottom of the matrix layer. The width of the scoop entry shall be at least 2,5 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 reference increment mass to be taken. The increments shall be of uniform mass. The minimum mass required is

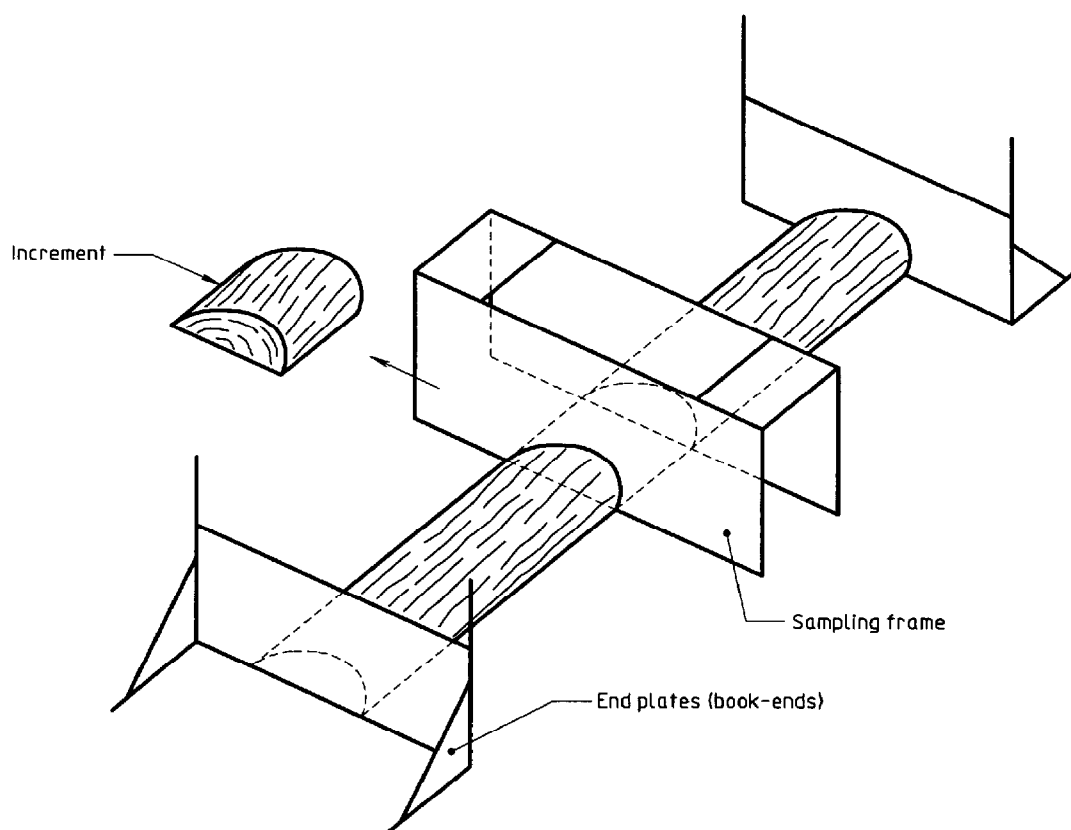
given in table 2 and depends on the nominal top size of the coal, subject to a minimum of 100 g.

Combine the increments into a divided sample. It is essential that these operations are performed quickly if loss of moisture is to be prevented.

NOTE 26 The scoop sample should be taken with the aid of a bump plate which is inserted vertically through the flattened heap until it is in contact with the mixing plate. 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 the 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.

6.7.3 Strip mixing and splitting method (see figure 5)

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

**Figure 5 — Strip mixing and splitting method**

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

Special apparatus for the cutting out of increments may be constructed if desired.

Because of the efficient longitudinal mixing achieved in the formation of a strip, the same precision can be achieved by taking fewer increments than in the case of the flattened heap method; 20 increments are normally required for unknown or heterogeneous coals. Where the same quality is regularly prepared under the same conditions, the precision obtained should be checked (see annex B) and the number of increments adjusted accordingly, subject to a minimum of 10.

6.8 Minimum retained mass of gross samples, partial samples and single increment samples

A gross sample (or a partial sample if it is intended to prepare it as a test sample) shall not be divided to less than a mass (given in table 3 or 4) corresponding to its nominal top size and depending on its intended use.

When partial samples are to be combined to form a gross sample, the total mass of all the partial samples in the sampling unit shall be at least the mass given in table 3 or 4.

When single increment samples that are to be prepared to form a test sample are of a mass less than that given in table 2 corresponding to their nominal top size, they shall be crushed before any division is carried out.

6.9 Methods of reduction

6.9.1 General

Equipment used to reduce the particle size should be mechanical. Manual crushing shall be limited to 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 test method.

Samples which are intended for general analysis should only be sieved for the purpose of checking the mill setting. It is bad practice to sieve out, recrush and mix back oversize material.

6.9.2 Reduction mills

6.9.2.1 Introduction

Reduction mills may be described as being of low, medium or high speed. Pulverizing to 4 mm size may be carried out in all types of mill, to 2,8 mm or 1 mm size in medium or high-speed mills. High-speed mills are preferable for pulverizing to 500 μm size or less. If the loss of moisture has to be minimized, only totally closed mills should be used to crush to any size less than 2,8 mm.

NOTE 27 In general, high-speed mills, if the screen used is changed, are more adaptable to a wide range of sizes than are slow-speed or medium-speed mills. Also, their use is of particular value in ensuring, with a standard setting, a constant nominal top size of the product for coals of different hardness.

Mills should be such that the coal is neither contaminated nor unduly heated in the course of the crushing. They should be designed so that no material from a previous sample is retained to contaminate succeeding samples, and so that there is no significant loss from the sample as it passes through the machine.

There should be no contact between the metal surfaces of the mill. This will reduce the danger of localized heating of the coal which might modify its properties. Furthermore, it should not be necessary to set the mill at its finest limit to obtain the specified size grading with coal. This will avoid excessive wear of the working parts which would result in contamination of the coal sample and increased replacement costs.

Examples of mills suitable for producing products of different sizes are outlined in 6.9.2.2 to 6.9.2.5.

NOTE 28 For certain tests, specific size gradings are required and the type of mill used should be chosen to ensure that such size gradings are obtained. The term "mills" refers to all types of reduction equipment described in 6.9.2.

6.9.2.2 Open low-speed mills (< 5 Hz)

Examples are jaw crushers and roll crushers.

6.9.2.3 Open medium-speed mills (5 Hz to 30 Hz)

Cone or ring crushers, consisting of a rotating cone or ring whose position in relation to a fixed cone or ring can be adjusted, plate mills or swing hammers.

6.9.2.4 Open high-speed mills (> 30 Hz)

Beater or swing-hammer mills, consisting of a fixed beater (or cross arms), or arms with a swing hammer

at the end, rotating in a casing with a screen which controls the size of the product. The size of the product is less influenced than in slow-speed and medium-speed machines by the hardness of the coal.

High-speed mills, however, entrain air with the samples due to the fan-like effect of the rotating hammer. To minimize the loss of fine material, dust or moisture, it is essential to limit the air flowing through the mill, either by using closed inlet and outlet hoppers or by fitting a breather bag to the mill outlet.

6.9.2.5 Totally closed mills

The most suitable totally closed mills are various types of ball mill. Ball mills may be low-speed, medium-speed, or high-speed mills; for certain types, the speed may be adjustable. High-speed ball mills (> 20 Hz) are liable to heat the coals so much that some coal parameters are affected: these types shall therefore not be used.

6.10 Mixing

Mixing is normally restricted to off-line preparation of gross or partial samples. Mixing is not possible while samples or increments are flowing through any form of preparation system.

In theory, thorough mixing of a sample prior to its division should reduce errors due to sample preparation. In practice, this is not easy to achieve and some methods of hand mixing, for example forming and reforming into a conical pile, can have the opposite effect to that intended, leading to increased segregation. Such hand mixing methods are not recommended but one manual method which may be used is the riffle (see 6.7.1). The sample may be passed through a riffle, or a container-type sample divider [see figure 2 b)], three times, and the parts reunited after each pass.

If only mechanical sample dividers are used in the course of preparation, an additional mixing step should not be necessary to meet the required precision.

Mechanical mixing may be found useful at the final stage of preparation of the test samples.

6.11 Air drying

The purpose of air drying of the sample is to ensure that it will pass through the mills and sample division

equipment freely and without loss of mass and with minimal change in moisture content. If a sample has been taken for the determination of total moisture and, in the case of a common sample for the preparation of the analysis sample, air drying forms part of the determination of total moisture. Air drying may be carried out at any stage of sample preparation.

For air drying, the coal is spread in a thin layer and allowed to attain equilibrium with the atmosphere. The depth of the layer shall not exceed either 1,5 times the nominal top size of the coal or a depth which gives a loading of $1,0 \text{ g/m}^3$, whichever is the greater.

The drying temperature shall not exceed 40°C if the sample is to be used to determine calorific value, or caking properties, or swelling properties, or if the coal is susceptible to oxidation, or if air drying is part of the determination of total moisture.

The drying times given in table 6 should be sufficient to dry the sample but, if necessary, a longer time may be used. Any unnecessary lengthening of the drying time shall be avoided, especially for coals susceptible to oxidation.

If the mass of the uncrushed sample is large enough to make air drying impracticable, it will be necessary to crush and divide it before air drying. This procedure shall be checked for the absence of bias.

Table 6 — Drying time (for guidance only)

Temperature $^\circ\text{C}$	Time h
20	Preferably ≤ 24
30	6
40	4

When drying at an elevated temperature, a cabinet or oven with the air changed not less than once per minute should be used.

If the air drying is part of the determination of total moisture content, the sample shall be weighed before as well as after drying. If drying has been performed at an elevated temperature, the sample shall be allowed to cool to the ambient temperature before reweighing, in order to achieve moisture equilibrium with the ambient atmosphere. If a temperature of 40°C is used for drying, a period of 3 h will be sufficient to achieve equilibrium.