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Hard coal and coke — Manual sampling

Houille et coke — Échantillonnage manuel

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 18283 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

This first edition of ISO 18283 cancels and replaces ISO 1988:1975 and ISO 2309:1980, which have been technically revised.

Introduction

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

The fundamental requirements of sampling are that all particles of the fuel in the lot are accessible to the sampling instrument and that each individual particle has an equal probability of being selected and included in the sample.

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

The purpose of taking and preparing a sample of fuel is to provide a test sample that, when analysed, provides test results representative of the lot sampled.

The first stage of sampling, known as primary sampling, is the taking from positions distributed over the entire lot of an adequate number of fuel portions known as primary increments. The primary increments are then combined into a sample, either "as taken" or after having been divided, in order to reduce the mass of the sample to a manageable size. From this sample, the required number and types of test samples are prepared by a series of processes jointly known as sample preparation.

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

a) incorrect location/timing of increments,

b) incorrect delimitation and extraction of increments,

c) loss of integrity of increments after extraction.

Methods for measuring bias are described in this International Standard.

Hard coal and coke — Manual sampling

CAUTION — Sampling can involve hazardous materials, operations, equipment and situations. However, it is beyond the scope of this International Standard to address all of the safety problems associated with the use of this International Standard. It is, therefore, the responsibility of the parties concerned to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

ISO 18283 defines the basic terms used in manual sampling of hard coal and coke and describes the general principles of sampling. It specifies procedures and requirements for establishing a manual sampling scheme, methods of manual sampling, sampling equipment, handling and storage of samples, sample preparation and a sampling report.

This International Standard applies to manual sampling from fuels in movement. Guidelines for manual sampling from fuels in stationary situations are given in Annex B, but this method of sampling does not provide a representative test sample and the sampling report shall state this.

ISO 18283 does not include sampling of brown coals and lignites, which is described in ISO 5069-1 and ISO 5069-2, nor sampling from coal seams, for which guidance is given in ISO 14180. Mechanical sampling of coal and coke is covered in ISO 13909 (all parts).

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 579, *Coke — Determination of total moisture*

ISO 589:2003, *Hard coal — Determination of total moisture*

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

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

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

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

3 Terms and definitions

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

3.1

air-drying

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

NOTE Air-drying to equilibrium with the atmosphere applies to coal. Drying of coke is generally to facilitate sample preparation.

3.2

bias

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

3.3

common sample

sample collected for more than one intended use

3.4

continuous sampling

taking of a sample from each consecutive sub-lot so that increments are taken at uniform intervals whenever the fuel is handled at the point of sampling

3.5

divided increment

part obtained from the division of the increment in order to decrease its mass

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

3.6

fixed-mass division

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

3.7

fixed-ratio division

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

3.8

fuel

hard coal or coke

3.9

general analysis test sample

sample prepared to pass a sieve of nominal size of openings 212 µm in accordance with ISO 3310-1, used for the determination of most chemical and some physical characteristics

3.10

increment

portion of fuel extracted in a single operation of the sampling device

3.11

intermittent sampling

taking of samples from only certain sub-lots of fuel

3.12
lot
defined quantity of fuel for which the quality is to be determined

NOTE A lot can be divided into sub-lots.

3.13
manual sampling
extraction of increments by human effort

3.14
mass-basis sampling
taking of increments whereby the position of each increment to be extracted from the stream of fuel is measured by a mass interval of stream flow and the increment mass is fixed

3.15
mechanical sampling
extraction of increments by mechanical means

3.16
moisture sample
sample taken specifically for the purpose of determining total moisture

NOTE For coke, this sample can also be used for general analysis.

3.17
nominal top size
aperture size of 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

3.18
physical sample
sample taken specifically for the determination of physical characteristics, e.g. physical strength indices or size distribution

3.19
precision
closeness of agreement between independent test results obtained under *stipulated conditions*

NOTE 1 This is often defined using an index of precision, such as 2 standard deviations.

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

3.20
primary increment
increment extracted at the first stage of sampling, prior to any sample division and/or sample reduction

3.21
random sampling
extracting of increments at random mass or time intervals

3.22
replicate sampling
extracting, at intervals, of increments that are combined in rotation into different containers to give two or more samples of approximately equal mass

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

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

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

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

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

3.27
size analysis sample
sample taken specifically for particle size analysis

3.28
standard deviation
square root of the variance

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

3.30
sub-lot
part of a lot for which a test result is required

3.31
systematic sampling
extracting of increments at uniform mass or time intervals according to a predetermined plan

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

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

3.34
unit mass
quantity of material, which is defined by the sampling process (usually the primary increment)

3.35
variance
measure of dispersion, which is the sum of the squared deviations of observations from their average divided by one less than the number of observations

4 Establishing a sampling scheme

4.1 General

4.1.1 Sampling

The preferred method for manual sampling of fuels is during handling: e.g. (un)loading of ships, barges, wagons, trucks or during formation of or reclaiming from stockpiles. For safety and practical reasons, manual sampling of moving streams is sometimes not possible.

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

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

4.1.2 Sampling scheme

The general procedure for establishing a sampling scheme is as follows.

a) Define the quality parameters to be determined and the types of samples required.

b) Define the lot.

c) Select or assume the required overall precision for the lot (see 4.4.2).

d) Determine or assume the variability of the fuel (see 4.4.5 and, if relevant, 4.4.6) and the variance of preparation and testing (see 4.4.7).

e) Ascertain the nominal top size of the fuel for the purpose of determining the mass of increment and sample (see 4.4.9 and 4.4.10).

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

f) Select the sampling device (see Clause 6).

g) Establish the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see 4.5).

h) Determine the method of combining the increments into samples and the method of sample preparation (see Clause 8).

i) Define the sampling interval in terms of time or mass (see Clause 5).

j) Determine where to take the increments (see Clause 5).

4.1.3 Parameters

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

a) variability of the fuel;

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

c) number of increments comprising each sample;

d) mass of sample relative to nominal top size.

4.1.4 Sampling methods

In this International Standard, the following sampling methods are considered:

a) continuous sampling;

b) intermittent sampling.

4.2 Sampling methods

4.2.1 Continuous sampling

In continuous sampling, every sub-lot is sampled and the number of increments collected from each sub-lot shall be determined in accordance with 4.4.8.2. There are as many sample results for the lot as there are sub-lots. Each sub-lot should be approximately the same size; however, for practical reasons sometimes sub-lots of different sizes are used. The mean result for the lot should be of the required precision, but if it is desired to check that the required precision has been attained, it is possible to do this by using the procedures of replicate sampling (see 4.6).

4.2.2 Intermittent sampling

If fuel of the same source and quality is sampled frequently, it can be satisfactory to collect increments from some of the sub-lots but not from others. This is called intermittent sampling. The same number of increments shall be taken from every sub-lot that is sampled (see 4.4.8.3). The sub-lots to be sampled shall be chosen at random, unless it can be demonstrated that no bias, for example as a result of time-dependent variance, is introduced by choosing sub-lots systematically. Such demonstration shall be repeated from time to time and at random intervals. There are as many sample results per lot as there are sub-lots sampled, but because some sub-lots are not sampled, it is not possible to say whether the average of these results has the required precision for the lot unless information about the variation between sub-lots is available (see 4.4.4 and 4.4.6). If the variation between sub-lots is too large, it can be necessary to introduce continuous sampling to achieve the desired precision. Use of intermittent sampling shall be agreed between contracting parties and shall be recorded in the sampling report.

4.3 Design of the sampling scheme

4.3.1 General

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

It is important that the sampling operator receive instructions that are simple, easily understood and capable of only one interpretation. These instructions, which should be set out in writing, should be prepared by the sampling supervisor after inspecting the sampling site and referring to the information given in this International Standard. The following items in the following list and described in 4.3.2 to 4.3.6 should be considered by the supervisor when compiling instructions:

a) fuel to be sampled and considerations for sampling;

b) bias of results;

c) precision of results;

d) lot size and number of sub-lots;

e) method of sampling;

f) requirements for test samples;

g) number of increments;

h) mass of sample.

4.3.2 Fuel to be sampled and considerations for sampling

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

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

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

b) location and the handling system;

c) transport means/carriers;

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

e) human safety risks.

4.3.3 Division of lots

The lot may be sampled as a whole, resulting in one sample, or divided into a number of sub-lots resulting in a sample from each. A lot may be sampled as a whole or as a series of sub-lots each to a maximum of 10 000 t, e.g. fuel despatched or delivered over a period of time, a ship load, a train load, a wagon load, or fuel produced during a certain period, e.g. a shift.

Such division into a number of sub-lots can be necessary to

a) achieve the required precision (calculated by the procedure in 4.5),

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

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

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

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

4.3.4 Bias of results

It is of particular importance in sampling to ensure as far as possible that the parameter to be measured is not altered by the sampling and sample preparation process or by subsequent storage prior to testing. This can require, in some circumstances, a limit on the mass of the primary increment, the divided sample and the test sample to maintain integrity (see 4.4.9 and 4.4.10).

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

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

4.3.5 Precision of results

After the overall precision of the lot has been decided, the number of sub-lots and the number of increments per sub-lot collected shall then be determined as described in 4.4.8 and the reference mass of the primary increments shall be determined as described in 4.4.9.

For single lots, the quality variation shall be assumed as the worst case (see 4.4.5). The precision of sampling achieved may be measured using the procedure of replicate sampling (see 4.6).

At the start of regular sampling of unknown fuels, *the worst-case* quality variation shall be assumed in accordance with 4.4.3 and 4.4.5.

If any subsequent change in precision is required, the number of sub-lots and of increments shall be changed as determined in 4.5 and the precision attained rechecked. The precision shall also be checked if there is any reason to suppose that the variability of the fuel being sampled has increased. The number of increments determined in 4.5 applies to the precision of the result when the sampling errors are large relative to the sample preparation and testing errors, e.g. moisture. However, in some tests, the testing errors are themselves large. In this case, it can be necessary to prepare two or more test portions from the sample and use the mean of *the determinations* to give a better precision.

4.3.6 Requirements for test samples

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

A number of tests are carried out on crushed or pulverized samples of prepared top sizes as mentioned in the relevant testing standards, e.g. ash on a – 0,212 mm sample. However, a number of tests require samples either *in the original state* or prepared to a particle size somewhere between original state and 0,212 mm.

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

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

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

In case the mass of the sample as calculated in accordance with this International Standard is insufficient for the masses of the required test samples, the number of increments should be increased to provide the greater mass.

4.4 Precision of sampling

4.4.1 General

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

It is possible to design a sampling scheme that, in principle, can achieve an arbitrary level of precision, such level to be determined.

The required overall precision on a lot should be agreed between the parties concerned. In the absence of such agreement, a value of 10 % of the ash content may be assumed.

4.4.2 Precision and total variance

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

If a large number of replicate samples are taken from a sub-lot of fuel, prepared and analysed separately, the precision, P, of a single observation is given by Equation (1):

$$P = 2s = 2\sqrt{V_{\mathrm{SPT}}} \qquad (1)$$

where

s is the sample estimate of the population standard deviation;

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

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

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

$$V_{\mathrm{SPT}} = \frac{V_{\mathrm{I}}}{n} + V_{\mathrm{PT}} \qquad (2)$$

where

V_{I} is the primary increment variance;

V_{PT} is the preparation and testing variance;

n is the number of primary increments in the sample.

4.4.3 Continuous sampling

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

$$V_{\mathrm{SPT}} = \frac{V_{\mathrm{I}}}{Nn} + \frac{V_{\mathrm{PT}}}{N} \tag{3}$$

where

n is the number of primary increments comprising each sample;

N is the number of sample results used to obtain the mean.

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

$$P_{\mathrm{L}} = \frac{P_{\mathrm{SL}}}{\sqrt{N_{\mathrm{SL}}}} = 2\sqrt{\frac{V_{\mathrm{I}}}{N_{\mathrm{SL}}n} + \frac{V_{\mathrm{PT}}}{N_{\mathrm{SL}}}} \tag{4}$$

$$P_{\mathrm{SL}} = P_{\mathrm{L}} \cdot \sqrt{N_{\mathrm{SL}}} \tag{5}$$

where

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

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

V_{I} is the primary increment variance;

n is the number of increments per sub-lot;

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

V_{PT} is the preparation and testing variance.

If the quality of a fuel of a type not previously sampled is required, then in order to devise a sampling scheme, assumptions have to be made about the variability (see 4.4.5).

4.4.4 Intermittent sampling

Whilst the value used for the primary increment variance is assumed to be consistent for all the sub-lots in a lot, there can be variations between the means of sub-lots. Providing all sub-lots are sampled and tested, this is not a source of additional variance. However, if only one or some sub-lots are sampled and tested (i.e. intermittent sampling), then a term to correct for sub-lot variance should be included in Equation (3) and the equations derived from it, as given in Equation (6):

$$V_{\mathrm{SPT}} = \frac{V_{\mathrm{I}}}{N_{\mathrm{SLS}}n} + \frac{V_{\mathrm{PT}}}{N_{\mathrm{SLS}}} + \left(1 - \frac{N_{\mathrm{SLS}}}{N_{\mathrm{SL}}}\right)\frac{V_{\mathrm{SL}}}{N_{\mathrm{SLS}}} \tag{6}$$

where

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

N_{SLS} is the number of sub-lots sampled;

V_{SL} is the sub-lot variance.

The term $\left(1-\frac{N_{\mathrm{SLS}}}{N_{\mathrm{SL}}}\right)V_{\mathrm{SL}}$ compensates for the fact that, as the proportion of sub-lots sampled and tested increases, the influence of sub-lot variance decreases, until it disappears when $N_{\mathrm{SLS}}=N_{\mathrm{SL}}$.

The equivalent to Equation (4) can be derived by combining Equations (1) and (6), as given in Equation (7):

$$P_{\mathrm{L}}=2\sqrt{\left[\frac{V_{\mathrm{I}}}{N_{\mathrm{SLS}}n}+\frac{V_{\mathrm{PT}}}{N_{\mathrm{SLS}}}+\left(1-\frac{N_{\mathrm{SLS}}}{N_{\mathrm{SL}}}\right)\frac{V_{\mathrm{SL}}}{N_{\mathrm{SLS}}}\right]} \tag{7}$$

4.4.5 Primary increment variance

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

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

The value of the primary increment variance, V_{I}, required for the precision using Equation (4) can be obtained by either

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

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

For calculating the variance, Equation (8) can be used:

$$V_{\mathrm{I}}=\frac{1}{n-1}\left[\sum x_i^2-\frac{\left(\sum x_i\right)^2}{n}\right]-V_{\mathrm{PT}} \tag{8}$$

where

V_{I} is the primary increment variance;

n is the number of increments taken;

x_i is the value of the analysed parameter;

V_{PT} is the preparation and testing variance.

If neither of these values is available, a value of $V_{\mathrm{I}}=20$ for ash content can be assumed initially and checked after sampling has been carried out.

4.4.6 Sub-lot variance

In some cases (e.g. see 4.4.4), the sub-lot variance, V_{SL}, can be calculated, because, just like the primary increment variance, this value gives an indication of the homogeneity of the fuel. For calculation of V_{SL} Equation (9) can be used:

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

where

V_{SL} is the sub-lot variance;

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

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

V_{PT} is the preparation and testing variance.

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

4.4.7 Preparation and testing variance

The value of the preparation and testing variance, V_{PT}, required for the calculation of the precision using Equation (4) or (7) can be obtained by either

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

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

$$V_{\mathrm{PT}} = \frac{\sum d_i^2}{2n_{\mathrm{p}}} \tag{10}$$

where

V_{PT} is the preparation and testing variance;

d_i is the difference between individual pair members;

n_{p} is the number of pairs.

Alternately, split one or more sub-lot samples into a minimum of 20 test samples. Prepare and analyse each test sample for the parameters of interest, preferably ash (dry basis) and total moisture. The preparation and testing variance shall be calculated as given in Equation (11):

$$V_{\mathrm{PT}} = \frac{1}{n-1}\left[\sum x_i^2 - \frac{\left(\sum x_i^2\right)^2}{N_{\mathrm{TS}}}\right] \tag{11}$$

where

V_{PT} is the preparation and testing variance;

N_{TS} is the number of test samples;

x_i is the value of the analysed parameter.

If neither of these values is available, a value of $V_{PT} = 0,2$ for ash content can be assumed initially and checked if necessary after preparation and testing has been carried out.

If high overall precision, P_L, is required, then lower V_{PT} values of 0,1 or 0,05 for ash content are required to obtain the required overall precision using a practical number of primary increments and sub-lots (see 4.4.8).

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

4.4.8.1 General

The number of increments taken from a lot in order to achieve a particular precision is a function of the variability of the quality of the coal in the lot irrespective of the mass of the lot. The lot may be sampled as a whole, resulting in one sample, or divided into a number of sub-lots resulting in a sample from each. Such division can be necessary in order to achieve the required precision and the necessary number of sub-lots shall be calculated using the procedure given in 4.4.8.2 or 4.4.8.3 as appropriate.

Another important reason for dividing the lot is to maintain the integrity of the sample, i.e. to avoid bias after taking the increment, particularly in order to minimize loss of moisture due to standing. The requirement to do this is dependent on factors such as the time taken to collect the samples, ambient temperature and humidity conditions, the ease of keeping the sample in sealed containers during collection and the particle size of the coal. It is recommended that, if moisture loss is suspected, a bias test is carried out to compare the quality of a reference sample immediately after extraction with that of the sample after standing for the normal time. If bias is found, the sample standing time should be reduced by collecting samples more frequently, i.e. increasing the number of sub-lots.

There can be other practical reasons for dividing the lot:

a) for convenience when sampling over a long period,

b) to keep sample masses manageable.

Establish the number of sub-lots and number of increments required per sub-lot in accordance with 4.4.8.2 or 4.4.8.3 as appropriate.

NOTE The equations given in 4.4.8.2 and 4.4.8.3 generally give an overestimation of the required number of increments. This is because they are based on the assumption that the quality of coal has no serial correlation; however, serial correlation is always present to some degree. In addition, because a certain amount of preparation and testing is required when measuring the increment variance or the sub-lot variance, the preparation and testing errors are included more than once.

The designer of a sampling scheme should make provisions for the worst case anticipated and then tend to use higher values for V_I and V_{SL} than may actually occur when the system is in operation. On implementing a new sampling scheme, a check on the actual precision being achieved should be carried out using the methods described in ISO 13909-7. This can be necessary to achieve the required precision, in which case the number of sub-lots is calculated using the procedures given in 4.4.8.2 and 4.4.8.3.

4.4.8.2 Continuous sampling

Determine the number of sub-lots required for practical reasons (see 4.4.8.1) and then estimate the number of increments for a desired precision from Equation (12), obtained by transposing Equation (4):

$$n = \frac{4V_I}{N \cdot P_L^2 - 4V_{PT}} \qquad (12)$$

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 sub-lots. In such cases, or if n is impracticably large, reduce the errors of sample preparation and testing or increase the number of sub-lots by one of the following means.

a) Choose a new number of sub-lots corresponding to a convenient sub-lot mass, recalculate n from Equation (12) and repeat this process until n is a practicable number.

b) Decide on the maximum practicable number of increments per sub-lot, n_1, and calculate N from Equation (13):

$$N = \frac{4\left(V_\mathrm{I} + n_1 V_\mathrm{PT}\right)}{n_1 P_\mathrm{L}^2} \qquad (13)$$

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

Take n as 10 if the final calculated value is less than 10.

Examples of calculations for continuous sampling from moving streams for a range of values of overall precision, P_L, and preparation and testing variance, V_PT, for ash assuming $V_\mathrm{I} = 5$ for washed coal and $V_\mathrm{I} = 10$ for unwashed coal are given in Table 1.

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

Table 1 — Examples of calculated number of increments per lot and sub-lot for ash for specified conditions [a]

Lot and sub-lot characteristics			$P_L = 0,2$ %											
			$V_{PT} = 0,05$				$V_{PT} = 0,1$				$V_{PT} = 0,2$			
Mass of lot	Mass of sub-lot	Number of sub-lots	Number of increments				Number of increments				Number of increments			
			Washed coal		*Unwashed coal*		Washed coal		Unwashed coal		Washed coal		Unwashed coal	
t	t		Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot
150 000	10 000	15	50	750	100	1 500	100	1 500	200	3 000	b	b	b	b
100 000	10 000	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
50 000	5 000	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
40 000	4 000	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
30 000	3 000	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
20 000	2 000	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
10 000	1 000	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
8 000	500	16	45	720	91	1 456	83	1 328	167	2 672	b	b	b	b
6 000	500	12	71	852	143	1 716	250	3 000	500	6 000	b	b	b	b
5 000	500	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
4 000	250	16	45	720	91	1 456	83	1 328	167	2 672	b	b	b	b
3 000	250	12	71	852	143	1 716	250	3 000	500	6 000	b	b	b	b
2 000	200	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b
1 000	100	10	100	1 000	200	2 000	b	b	b	b	b	b	b	b

[a] Conditions: Overall P_L ranges from 0,2 % to 0,6 % ash and preparation and testing variances, V_{PT}, of 0,05, 0,1 and 0,2 for ash *content assuming* $V_1 = 5$ for washed coal and $V_1 = 10$ for unwashed coal.

[b] Smaller V_{PT} or more sub-lots required.

Table 1 (*continued*)

Lot and sub-lot characteristics			$P_L = 0,3$ %											
			$V_{PT} = 0,05$				$V_{PT} = 0,1$				$V_{PT} = 0,2$			
Mass of lot	Mass of sub-lot	*Number of* sub-lots	Number of increments				Number of increments				Number of increments			
			Washed coal		Unwashed coal		Washed coal		Unwashed coal		Washed coal		Unwashed coal	
t	t		Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot
150 000	10 000	15	17	255	35	525	21	315	42	630	36	540	73	1 095
100 000	10 000	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000
50 000	5 000	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000
40 000	4 000	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000
30 000	3 000	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000
20 000	2 000	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000
10 000	1 000	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000
8 000	1 000	8	38	304	77	616	63	504	125	1 000	b	b	b	b
6 000	750	8	38	304	77	616	63	504	125	1 000	b	b	b	b
5 000	500	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000
4 000	500	8	38	304	77	616	63	504	125	1 000	b	b	b	b
3 000	500	6	59	354	118	708	143	858	286	1 716	b	b	b	b
2 000	250	8	38	304	77	616	63	504	125	1 000	b	b	b	b
1 000	100	10	29	290	57	570	40	400	80	800	200	2 000	400	4 000

[a] Conditions: Overall P_L ranges from 0,2 % to 0,6 % ash and preparation and testing variances, V_{PT}, of 0,05, 0,1 and 0,2 for ash content assuming $V_1 = 5$ for washed coal and $V_1 = 10$ for unwashed coal.

[b] Smaller V_{PT} or more sub-lots required.

Table 1 (*continued*)

Lot and sub-lot characteristics			$P_L = 0,4$ %											
			$V_{PT} = 0,05$				$V_{PT} = 0,1$				$V_{PT} = 0,2$			
Mass of lot	Mass of sub-lot	Number of sub-lots	Number of increments				Number of increments				Number of increments			
			Washed coal		Unwashed coal		Washed coal		Unwashed coal		Washed coal		Unwashed coal	
t	t		Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot
150 000	10 000	15	10 [c]	150	18	270	10	150	20	300	13	195	25	375
100 000	10 000	10	14	140	29	290	17	170	33	330	25	250	50	500
50 000	10 000	5	33	165	67	335	50	250	100	500	b	b	b	b
40 000	8 000	5	33	165	67	335	50	250	100	500	b	b	b	b
30 000	6 000	5	33	165	67	335	50	250	100	500	b	b	b	b
20 000	4 000	5	33	165	67	335	50	250	100	500	b	b	b	b
10 000	2 000	5	33	165	67	335	50	250	100	500	b	b	b	b
8 000	1 000	8	19	152	37	296	23	184	45	360	42	336	83	664
6 000	1 000	6	26	156	53	318	36	216	71	426	125	750	250	1 500
5 000	1 000	5	33	165	67	335	50	250	100	500	b	b	b	b
4 000	500	8	19	152	37	296	23	184	45	360	42	336	83	664
3 000	500	6	26	156	53	318	36	216	71	426	125	750	250	1 500
2 000	250	8	19	152	37	296	23	184	45	360	42	336	83	664
1 000	200	5	33	165	67	335	50	250	100	500	b	b	b	b

[a] Conditions: Overall P_L ranges from 0,2 % to 0,6 % ash and preparation and testing variances, V_{PT}, of 0,05, 0,1 and 0,2 for ash content assuming $V_1 \approx 5$ for washed coal and $V_1 = 10$ for unwashed coal.

[b] Smaller V_{PT} or more sub-lots required.

[c] This is the minimum value, the calculated values are less than 10. Due to uncertainties in the variability of the fuel, a minimum of 10 increments per sub-lot shall be taken.

Table 1 (*continued*)

Lot and sub-lot characteristics			$P_L = 0,6\ \%$											
			$V_{PT} = 0,05$				$V_{PT} = 0,1$				$V_{PT} = 0,2$			
Mass of lot	Mass of sub-lot	Number of sub-lots	Number of increments				Number of increments				Number of increments			
			Washed coal		Unwashed coal		Washed coal		Unwashed coal		Washed coal		Unwashed coal	
t	t		Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot	Sub-lot	Lot
150 000	10 000	15	10 [b]	150	10 [c]	150	10 [c]	150	10 [c]	150	10 [c]	150	10 [c]	150
100 000	10 000	10	10 [c]	100	12	120	10 [c]	100	13	130	10 [c]	100	14	140
50 000	10 000	5	13	65	25	125	14	70	29	145	20	100	40	200
40 000	10 000	4	16	64	32	128	19	76	38	152	31	124	63	252
30 000	5 000	6	10	60	20	120	11	66	23	138	15	90	29	174
20 000	5 000	4	16	64	32	128	19	76	38	152	31	124	63	252
10 000	2 500	4	16	64	32	128	19	76	38	152	31	124	63	252
8 000	2 000	4	16	64	32	128	19	76	38	152	31	124	63	252
6 000	1 500	4	16	64	32	128	19	76	38	152	31	124	63	252
5 000	1 000	5	13	65	25	125	14	70	29	145	20	100	40	200
4 000	1 000	4	16	64	32	128	19	76	38	152	31	124	63	252
3 000	750	4	16	64	32	128	19	76	38	152	31	124	63	252
2 000	500	4	16	64	32	128	19	76	38	152	31	124	63	252
1 000	250	4	16	64	32	128	19	76	38	152	31	124	63	252

[a] Conditions: Overall P_L ranges from 0,2 % to 0,6 % ash and preparation and testing variances, V_{PT}, of 0,05, 0,1 and 0,2 for ash content assuming $V_1 = 5$ for washed coal and $V_1 = 10$ for unwashed coal.

[c] This is the minimum value, the calculated values are less than 10. Due to uncertainties in the variability of the fuel, a minimum of 10 increments per sub-lot shall be taken.

4.4.8.3 Intermittent sampling

Initially decide on the number of sub-lots, N_{SL}, and the minimum number, N_{SLS}, required to be sampled for practical reasons (see 4.4.8.1) and then estimate the number of increments in each sub-lot for a desired precision in a lot from Equation (14), obtained by transposing Equation (7):

$$n = \frac{4V_i}{N_{\mathrm{SLS}} P_{\mathrm{L}}^2 - 4\left(1 - \frac{N_{\mathrm{SLS}}}{N_{\mathrm{SL}}}\right) V_{\mathrm{SL}} - 4V_{\mathrm{PT}}} \tag{14}$$

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 sub-lots. In such cases or if n is impracticably large, increase the number of sub-lots to be sampled by one of the following means.

a) Choose a larger value for N_{SLS}, the number of sub-lots actually sampled, recalculate n and repeat this process until the value of n is a practicable number.

b) Decide on the maximum practicable number of increments per sub-lot, n_1, and calculate N_{SLS} from Equation (15):

$$N_{\mathrm{SLS}} = \frac{4N\left(\frac{V_{\mathrm{I}}}{n_1} + V_{\mathrm{SL}} + V_{\mathrm{PT}}\right)}{NP_{\mathrm{L}}^2 + 4V_{\mathrm{SL}}} \tag{15}$$

Adjust N_{SL} upwards if necessary to a convenient number and recalculate n from Equation (14).

Take n as 10 if the final calculated value is less than 10.

4.4.9 Mass of increments

The reference mass of increments can be found in Table 2. These values were established empirically and should be used as a starting point.

Table 2 — Reference increment mass

Nominal top size of fuel mm	Reference mass of increment for sampling kg	Nominal top size of fuel mm	Reference mass of increment for sampling kg
150	9,0	25	1,5
125	7,5	22,4	1,3
100	6,0	20	1,2
90	5,5	16	1,0
80	5,0	11,2	0,70
75	4,5	10	0,60
70	4,0	8	0,50
63	4,0	5,6	0,50
60	3,5	4	0,50
50	3,0	2,8	0,50
45	3,0	2	0,50
30	2,0	1	0,50
25	1,5	< 0,5	0,50

4.4.10 Minimum mass of samples

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

The minimum mass of a sample (primary as well as after division) is dependent on the nominal top size of the fuel, the precision required for the parameter concerned and the relationship of that parameter to particle size. Some such relationship applies at all stages of preparation. The attainment of this mass does not, of itself, guarantee the required precision. This is also dependent on the number of increments in the sample and their variability (see 4.5).

Values for the minimum mass of samples are shown in Table 3 and for the reduced minimum sample mass in Table 4.

Table 3 — Minimum mass of sample for general analysis (coal), determination of total moisture content (coal), coke sample and size analysis (coal and coke)

1	2	3	4	5	6
Nominal top size of fuel	Coal		Coke	Coal and coke	
	General analysis samples and common samples [a]	Samples for determination of total moisture content [b]	Coke samples [c]	Samples for size analysis for a precision of 1 % [d]	Samples for size analysis for a precision of 2 % [d]
mm	kg	kg	kg	kg	kg
150	2 600	500	2 000	6 750	1 700
125	1 700	350	1 000	4 000	1 000
90	750	125	500	1 500	400
75	470	95	375	950	250
63	300	60	250	500	125
50	170	35	175	280	70
45	125	25	125	200	50
38	85	17	90	130	30
31,5	55	10	60	65	15
22,4	32	7	30	25	6
16	20	4	15	8	2
11,2	13	2,5	8	3	0,70
10	10	2	6	2	0,50
8	6	1,5	4	1	0,25
5,6	3	1,2	2	0,50	0,25
4	1,50	1	2	0,25	0,25
2,8	0,65	0,65	2	0,25	0,25
2	0,25	0,65	2	0,25	0,25
1	0,10	0,65	2	0,25	0,25
< 0,5	0,06	0,65	2	0,25	0,25

[a] Minimum mass of coal sampled for general analysis to reduce the variance due to the particulate nature of the coal to 0,01, corresponding to a division precision of 0,2 % with regard to ash.

[b] Minimum mass of coal samples for total moisture analysis, which is approximately 20 % of the minimum mass for general analysis, subject to an absolute minimum of 0,65 kg.

[c] Minimum masses for coke samples.

[d] Minimum masses for coal and coke samples for size analysis corresponding to a division precision of 1 % and 2 %, respectively. These masses have been calculated on the basis of the precision of the determination of oversize, i.e. the fuel above nominal top size. The precision for other size fractions is normally better than this.

NOTE 1 For fuels with top sizes > 150 mm, see Clause B.2.

NOTE 2 If the specified masses for 90 mm, 125 mm or 150 mm mentioned in the columns 2, 4 and 5 are impractically large, the specified minimum masses for 75 mm can be taken. This results in a worse division precision.

Table 4 — Reduced minimum sample masses for large sizes

1	2		3		4	
Nominal top size of fuel	**General analysis samples and common samples**		**Coke samples**		**Samples for size analysis**	
mm	kg	Division precision	kg	Division precision	kg	Division precision
150	470	0,47	375	0,46	950	2,7
125	470	0,38	375	0,33	950	2,1
90	470	0,25	375	0,23	950	1,3
75	470	0,20	375	0,20	950	1,0

NOTE For coke, the masses given in column 4 are for guidance on the minimum mass for unknown or heterogeneous cokes. Whilst they can usually be reduced for the moisture sample, they can be inadequate for the determination of, for example, oversize, to 1 % division precision, particularly on very large coke.

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 3 corresponding to the purpose for which the sample has been taken and the nominal top size. If the increment masses are too low to satisfy this requirement, the divided increment shall be crushed prior to further division.

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

The minimum sample mass, m_S, for other desired levels of division precision may be calculated from Equation (16):

$$m_S = m_{0,S}\left(\frac{P_D}{P_R}\right)^2 \qquad (16)$$

where

$M_{0,S}$ is the mass given in Table 3;

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

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

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

When a fuel is regularly sampled under the same circumstances, the overall precision obtained for all the required parameters shall be checked (see 4.4.1) and the masses may be adjusted accordingly. However, the masses shall not be reduced below the minimum requirements laid down in the relevant standard specifying the test method.

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

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

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

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

Examples of the results of such calculations for continuous sampling from moving streams for a range of values of overall precision, P_L, and preparation and testing variance, V_{PT}, for ash assuming $V_I = 5$ for washed coal and $V_I = 10$ for unwashed coal are given in Table 1.

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

4.6 Determination of acquired precision by replicate sampling

4.6.1 General

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

4.6.2 Method and calculation

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

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

A typical calculation for fuel using the results in Table 5 is given below.

Table 5 — Results of single-lot sampling of % ash, dry basis

Sample No.	Sample value, x_i %	x_i^2
A	15,30	234,09
B	17,10	292,41
C	16,50	272,25
D	17,20	295,84
E	15,80	249,64
F	16,40	268,96
G	15,70	246,49
H	16,30	265,69
I	18,00	324,00
J	16,70	278,89
Total	165,00	2 728,26

The number, j, of replicate samples is 10.

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

The sample estimate of the population standard deviation, s, is (see Equation 5):

$$s = \sqrt{V}$$

$$s = \sqrt{\frac{2\,728{,}26 - \frac{165^2}{10}}{9}}$$

$$= 0{,}800$$

The best estimate for the precision, P, achieved for the lot is given by Equation (17):

$$P = \frac{2s}{\sqrt{N_{\text{RS}}}} \tag{17}$$

i.e.

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

4.6.3 Precision obtained using normal sampling scheme

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

4.7 Size analysis

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

These factors should be taken into account when applying the techniques for calculating the numbers of increments for a particular precision (see 4.4.2). In the absence of any information on increment variance, etc., initially take 24 increments per sample.

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

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

The minimum masses of sample for size analysis are given in Table 3, columns 5 (precision of 1 %) and 6 (precision of 2 %). The masses have been calculated on the basis of the precision of the determination of oversize, i.e. the fuel above the nominal top size. Precision for other size fractions is normally better than this.

5 Methods of sampling

5.1 General

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

Biased samples are obtained if part of the fuel is excluded. Extra care should be taken when sampling particularly heterogeneous or layered fuels or mixtures of fuels. Cyclical variations in coal quality can occur during sampling. Every effort shall be made to eliminate coincidence of the cycle with the taking of increments in systematic sampling. If this cannot be done, a bias is invariably introduced that can be of unacceptable proportions. In such circumstances, stratified random sampling may be adopted.

The methods described in Clause 5 are applicable to fuel in motion as follows:

a) stopped belt;

b) falling stream;

c) moving belt;

d) stockpiles (building/reclaiming);

e) grabs/front-end loaders;

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

Annex B presents informative methods of sampling from fuel in stationary lots and of sampling from large fuels (> 150 mm). Sampling from lots consisting of different fuels is described in 5.8; the use of random selection of increments is described in 5.9.

5.2 Sampling by time interval

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

The time interval, Δt, expressed in minutes, between taking primary increments is determined from Equation (18):

$$\Delta t \leqslant \frac{60 m_{\text{sl}}}{q_{\text{max}} n} \tag{18}$$

where

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

q_{max} is the maximum flow rate of the fuel, in metric tonnes per hour;

n is the number of primary increments in the sample (see 4.5).

5.3 Sampling by mass interval

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

The mass interval, Δm, expressed in tonnes, between taking increments is determined from Equation (19):

$$\Delta m = \frac{m_{\text{sl}}}{n} \tag{19}$$

where

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

n is the number of primary increments in the sample.

The mass interval between increments shall be equal to or smaller than that calculated from the number of increments specified in 4.5 in order to ensure that the number of increments is at least the minimum number specified.

5.4 Stratified random sampling

5.4.1 General

Stratified random sampling means that, for each time or mass interval, the actual taking of the increment is displaced by a random amount of time or mass, respectively, subject to the limitation that it shall be taken before that interval has expired.

During stratified random sampling, it is possible that two increments are collected very close together even though they are collected in different mass or time intervals.

5.4.2 Stratified random sampling by time interval

The sampling interval shall be determined as in 5.2 and the increment mass as in 4.4.9. Prior to the start of each sampling interval, a random number between zero and the sampling interval, in seconds or minutes, shall be generated. The increments shall then be taken after the time indicated by the random number.

5.4.3 Stratified random sampling by mass interval

The sampling interval shall be determined as in 5.3. and the increment mass as in 4.4.9. Prior to the start of each sampling interval, a random number between zero and the mass of the sampling interval (metric tonnes) shall be generated. The increment shall be taken after the passage of the mass of coal indicated by the random number.

5.5 Extracting the increment

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

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

The aperture of the sampling device shall be at least three times the nominal top size of the fuel, with a minimum dimension of 30 mm (see Clause 6) and larger, as necessary, to ensure that the larger particles are not excluded from the increment.

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

5.6 Fuel in motion

5.6.1 Stopped belt

Some methods of sampling tend to collect too many of either the large or the small particles and hence are liable to introduce bias. The method of extracting an increment by removing a whole cross-section from a stopped belt is the only way of ensuring that all particles are collected and hence that the sample is free from bias. Therefore, this is the reference method against which any other method should be checked. This is the most ideal method of sampling; however, in most cases, it is not practical for a standard sampling operation. Increments should be extracted from the whole width and thickness of the fuel stream when there is a normal load at the point of sampling.

If it is practicable to arrange to stop the belt periodically, increments can be extracted from the whole cross-section of the stream without difficulty. Safety measures to prevent the stopped belt from starting unexpectedly shall be in place.

Stopped-belt increments shall be extracted with a sampling frame (see 6.2.7), or equivalent, from a complete cross-section of the fuel on the belt at a fixed position.

The width of the complete section shall be at least three times the nominal top size of the fuel to be sampled, with a minimum dimension of 30 mm. The frame shall be placed on the stationary belt so that it is in contact with the full width of the belt.

Particles obstructing the insertion of the end plate on the left-hand side shall be pushed into the increment, while those obstructing the insertion of the end plate on the right-hand side shall be pushed out of the increment or vice versa. Whichever practice is used initially, this practice shall be implemented throughout the test.

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

5.6.2 Falling stream

Samples are taken from a transfer point for a continuously moving stream.

The sampler shall be able to reach the whole cross-section of the stream in safety and handle the resulting increment without undue physical strain. For systems with a capacity of approximately 100 t/h and more, this method should not be used.

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

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

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

It can be necessary to support the handle of the sampling device across a bar when it is passed into the falling stream or to erect a special gantry with adequate support.

5.6.3 Moving belt

Sampling from a moving belt is not recommended.

5.6.4 Stockpiles (building/reclaiming)

Sampling of stockpiles shall be carried out during the process of building up or reclaiming from the stockpile. Sampling of stationary stockpiles is not recommended. However, if it is the only alternative, the procedures in Annex B should be used, but the results are merely indicative of the quality of the fuel.

Increments shall be extracted from the working face of the stockpile, from the bucket of a front-end loader (see 5.6.5) or from a single, discrete load delivered to the stockpile before being pushed into the main stockpile. When extracting increments from a working face, the surface shall be sufficiently compacted to bear safely the weight of personnel and equipment.

A manual probe/auger or a scoop shall be used to extract increments. The aperture of the probe/auger or scoop shall be at least three times the nominal top size of the fuel, with a minimum dimension of 30 mm (see Clause 6) and larger, if necessary, for a scoop to ensure that the increment never fills the scoop completely. Probes and augers shall not be used for fuels that require size analysis. Increments shall be spaced as evenly as possible over the working face or the surface of the load in the front-end loader bucket.

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

When extracting increments, the manual probe/auger or scoop shall be inserted at right angles to the surface of the fuel after the top surface of the fuel has been removed. Large pieces of fuel should not be deliberately pushed aside when an increment is extracted and no portion of the increment should be lost during extraction of the scoop from the surface. Owing to the difficulty of insertion, a probe/auger shall be used only for fuels with a particle size of up to about 25 mm. A full column of fuel shall be extracted so that a representative increment is taken.

5.6.5 Grabs/front-end loaders

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

It is important that the sampler co-ordinate the extracting of the increments unambiguously with the operator of the grab or front-end loader.

A manual probe/auger or a scoop shall be used. The aperture of the probe/auger or scoop shall be at least three times the nominal top size of the fuel, with a minimum dimension of 30 mm (see Clause 6) and larger, as necessary, for a scoop to ensure that the larger particles are not excluded from the increment. Probes and augers shall not be used for fuels that require size analysis. Increments shall be spaced as evenly as possible over the surface of the front-end loader bucket or the grab.

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

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

When extracting increments, the manual probe/auger or scoop shall be inserted at right angles to the surface of the fuel. Large pieces of fuel should not be deliberately pushed aside when an increment is extracted and no portion of the increment should be lost during extraction of the scoop from the surface. Owing to the difficulty of insertion, a probe/auger shall be used only for fuels with a particle size of up to about 25 mm. A full column of fuel shall be extracted so that a representative increment is taken.

5.6.6 **Barges/trucks/railcars** (loading/unloading)

Sampling of fuels during its loading or unloading is based on progressively extracting increments from a number of points distributed over the freshly exposed surfaces. It is not permitted to sample the tops of fully loaded barges, trucks or railcars before these are unloaded, due to possible segregation or weather influences during transportation. Sampling of the full depth is permitted only when probed (see 6.2.4).

Sampling of fuels in barges shall be based on extracting increments from a number of points distributed over various layers of the fuel in the hold, which are exposed from time to time as the barge is loaded or unloaded. Sampling shall be done from sequential layers during (un)loading. If it is not possible to reach all of the fuel in the hold, the sample can be seriously biased. Sampling the top surface of the fuel in barges, trucks or railcars immediately after these are loaded is permitted, provided loading by layering of fuels of different qualities did not take place.

When extracting increments from a working face, the surface shall be sufficiently compacted to bear safely the weight of personnel and equipment.

A manual probe/auger or a scoop shall be used to extract increments. The aperture of the probe/auger or scoop shall be at least three times the nominal top size of the fuel, with a minimum dimension of 30 mm (see Clause 6) and larger, as necessary, for a scoop to ensure that the larger particles are not excluded from the increment. Probes and augers shall not be used for fuels that require size analysis.

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

When extracting increments, the manual probe/auger or scoop shall be inserted at right angles to the surface of the fuel after the top surface of the fuel has been removed. Large pieces of fuel should not be deliberately pushed aside when an increment is extracted and no portion of the increment should be lost during extraction of the scoop from the surface. Owing to the difficulty of insertion, a probe/auger shall only be used for fuels with a particle size of up to about 25 mm. A full column of fuel shall be extracted so that a representative increment is taken.

5.7 Moisture/common sample

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

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

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

The following additional points should be considered when sampling for moisture.

a) Stored fuel, washed fuel, etc. gradually lose water by drainage until an equilibrium is reached.

b) If free moisture is present in the lot, this settles towards the bottom so that a steady increase in moisture content occurs with increasing depth of the fuel.

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

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

5.8 Different fuels

If a lot is known to consist of different fuels piled/located in separate areas of the total lot, a separate sample shall be taken, prepared and analysed for each of these areas (covered by separate sampling schemes) and the sampling scheme established according to the most variable coal.

Each area shall consist of at least one sub-lot. The number of sub-lots and increments should be calculated in accordance with 4.5.

If insufficient knowledge about the lot is available to sample separately the different fuel qualities or sources, or if they are not identifiably separated or stowed separately, the lot should be divided into a number of sub-lots. Each sub-lot should be prepared and analysed separately. The values of the characteristics for each sub-lot should be reported. If necessary, for some parameters that can be averaged, the values of the characteristics for the whole lot can be obtained by taking a weighted average of the sub-lot values.

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

5.9 Random selection of increments

All the possible sampling areas (grabs, front-end loaders, barges, barge-holds or parts thereof, trucks, railcars or stockpiles when sampling from horizontal surfaces) shall be identified and numbered. The areas to be sampled are selected by one of the following methods.

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

b) Provide a set of numbered discs, one disc corresponding to each sampling area, and then proceed as follows.

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

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

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

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

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

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

6 Sampling equipment

6.1 General

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

a) The width of the sampling device, which determines the minimum mass of increment, shall satisfy the condition given in Equation (20), subject to a minimum dimension of 30 mm:

$$W \geqslant 3D \tag{20}$$

where

W is the minimum width of the cross-section, the width of the opening of the sampling device, expressed in millimetres;

D is the nominal top size of the fuel, expressed in millimetres.

b) The capacity of the device shall be such that during the extraction of a single increment, it is not overfilled and it can contain at least the required minimum mass (see 4.4.9).

c) The width of the entry aperture of the device shall be at least three times the nominal top size of the fuel or 30 mm, whichever is the larger.

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

e) Large and hard pieces of fuel or rock or shale shall not be pushed aside during the extraction of increments.

f) None of the fuel shall be lost from the device during the extraction and handling of the increment.

g) Wet fuel adhering to the devices should be minimized.

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

6.2 Examples

6.2.1 Ladles

A ladle [see Figure 2 a)] is a box or container or slotted device for extracting increments from falling streams of fuel. Ladles are not suitable for sampling in any operation where overflow of the sampling device occurs.

Ladles and scoops should be made of such materials as stainless steel to avoid sample build-up.

6.2.2 Shovels

A suitable design of a shovel is shown diagrammatically [see Figure 2 b)]. Shovels are constructed from sheet steel of approximately 2,5 mm thickness and should be fitted with a handle of appropriate length.

6.2.3 Scoops

Suitable designs of scoops [see Figures 2 c) and 2 d)] are shown diagrammatically. Scoops are made of about 2,5 mm sheet steel and should be fitted with a handle of appropriate length.

The scoop used for the flattened heap division method (see Figure 8) shall be flat bottomed and the width of the entry shall be at least three times the nominal top size of the fuel. 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 extracted.

6.2.4 Probes

Probes take the form of tubes which are inserted vertically or at slight angle into the fuel. Insertion into the fuel is sometimes difficult and the tube tends to empty when it is withdrawn.

Probes are used for sampling fuels of up to 25 mm nominal top size.

Three designs are described:

- Figure 2 e): The probe consists of two half-tubes that are designed to slide together to form a closed cylinder. Such probes can be used in various lengths up to 3,5 m; long probes are easiest to use for fuel up to 15 mm nominal top size.
- Figure 2 f): The probe consists of a cylindrical tube that is slightly tapered and is slit along an axis so that the taper tends to grip the fuel contained in the tube. Using a handle, it is possible to rotate the tube in order that it can be inserted. The tube can be awkward to empty and clean.
- Figure 2 g): The probe consists of a channel, the two edges of which have grooves, and a plate that can slide along the grooves. The channel is inserted open, the plate is inserted along the grooves and the complete device is withdrawn.

6.2.5 Augers

An auger [see Figure 2 h)] consists of a single spiral blade mounted on a hand crank. The auger is inserted into the bed of fuel with a screwing motion.

Augers are used for sampling fuels up to 25 mm nominal top size.

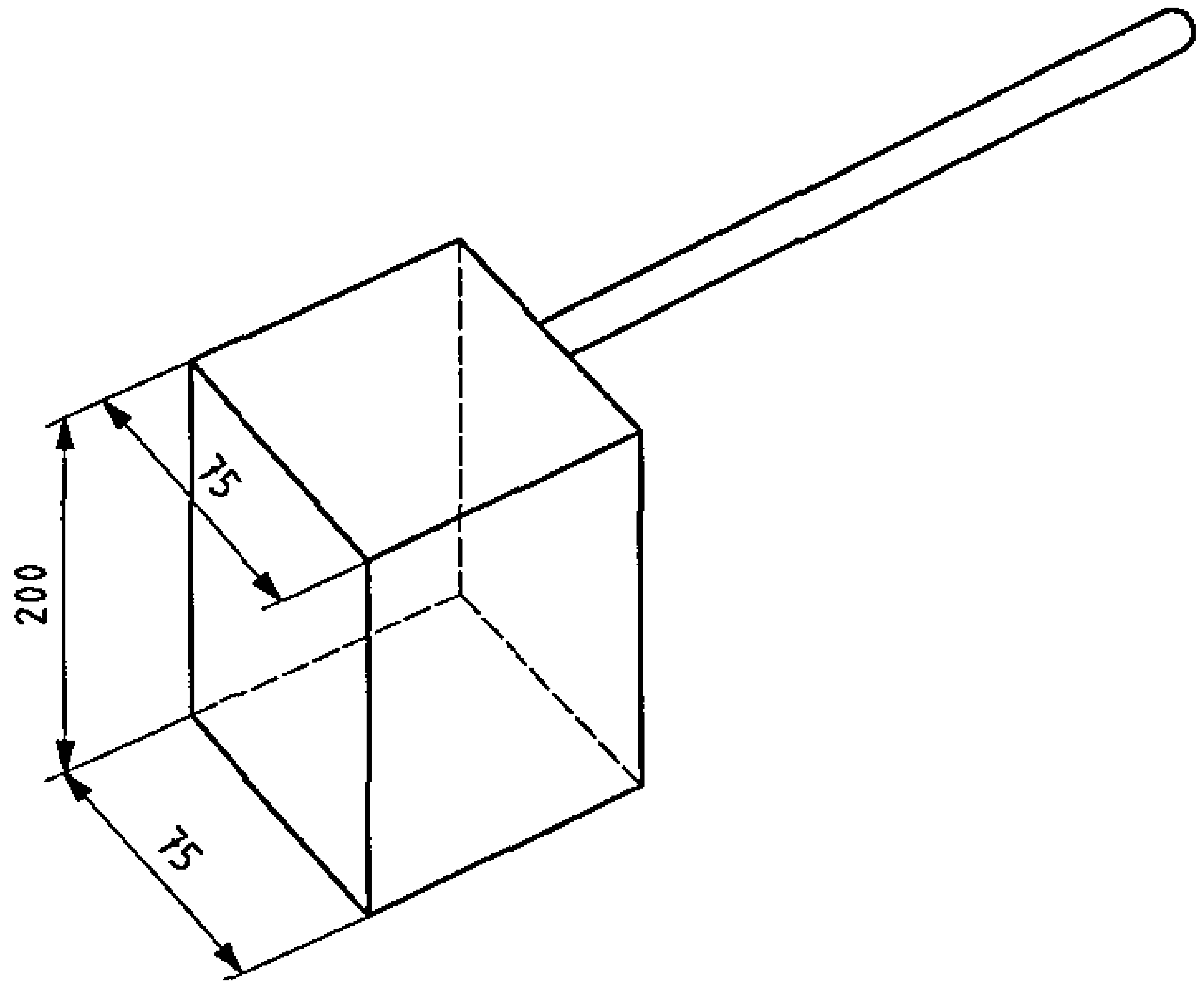
6.2.6 Manual cutter

A manual cutter [see Figure 2 i)] is a sampling device that can be moved through a falling stream, manually or with mechanical assistance. Manual cutters are not suitable for sampling in any operation where overflow of the cutter will occur.

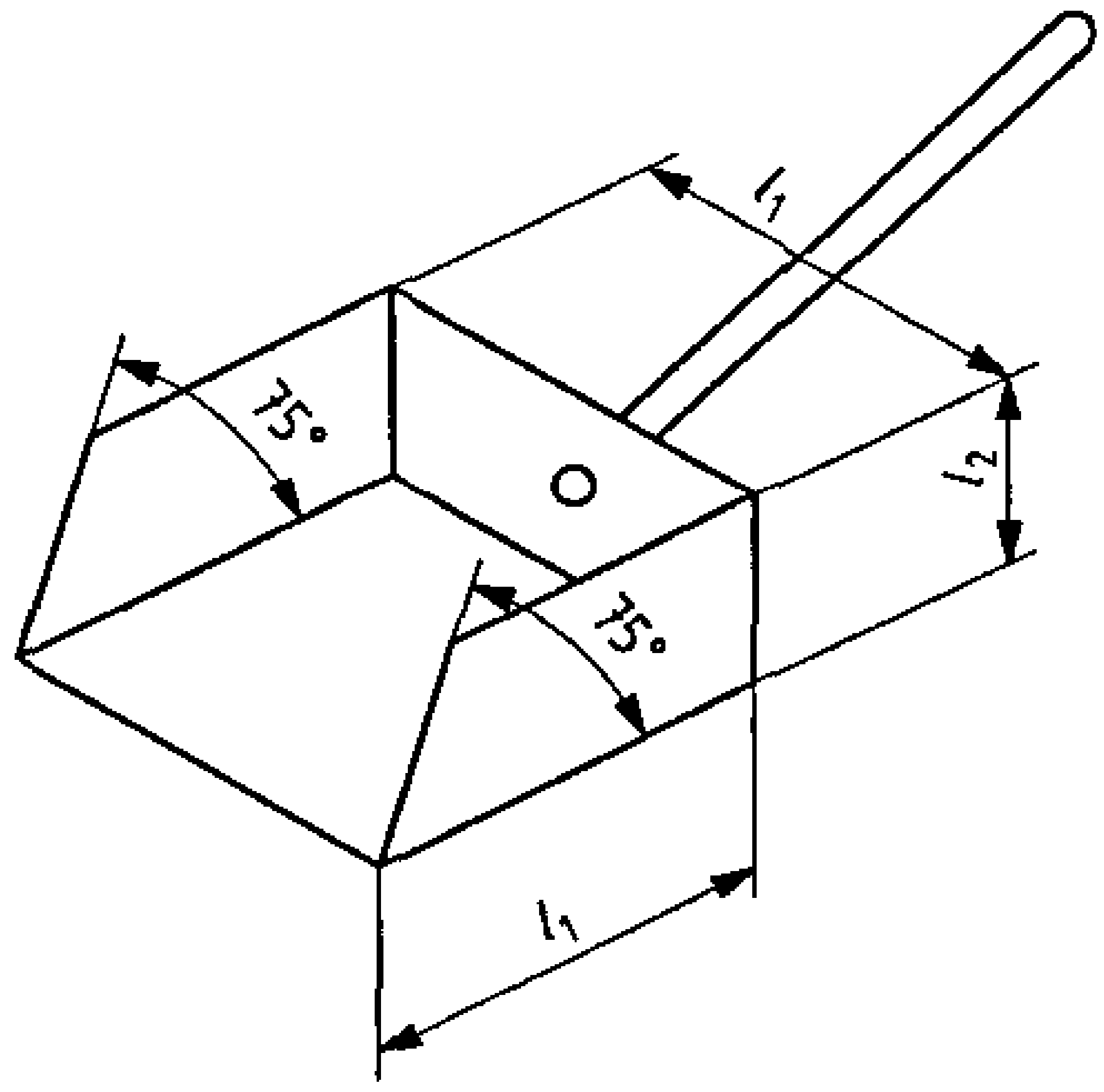
6.2.7 Stopped-belt sampling frame

A stopped-belt sampling frame [see Figure 2 j)] is a parallel-sided frame used for extracting a stopped-belt increment or for strip mixing and splitting increments. The width of the frame shall be at least three times the nominal top size of the fuel, with a minimum dimension of 30 mm. The frame should be inserted into the fuel until it is in contact with the belt across its full width and the increment is extracted by sweeping off the whole of the fuel lying between the sides of the frame.

Dimensions in millimetres, unless otherwise specified

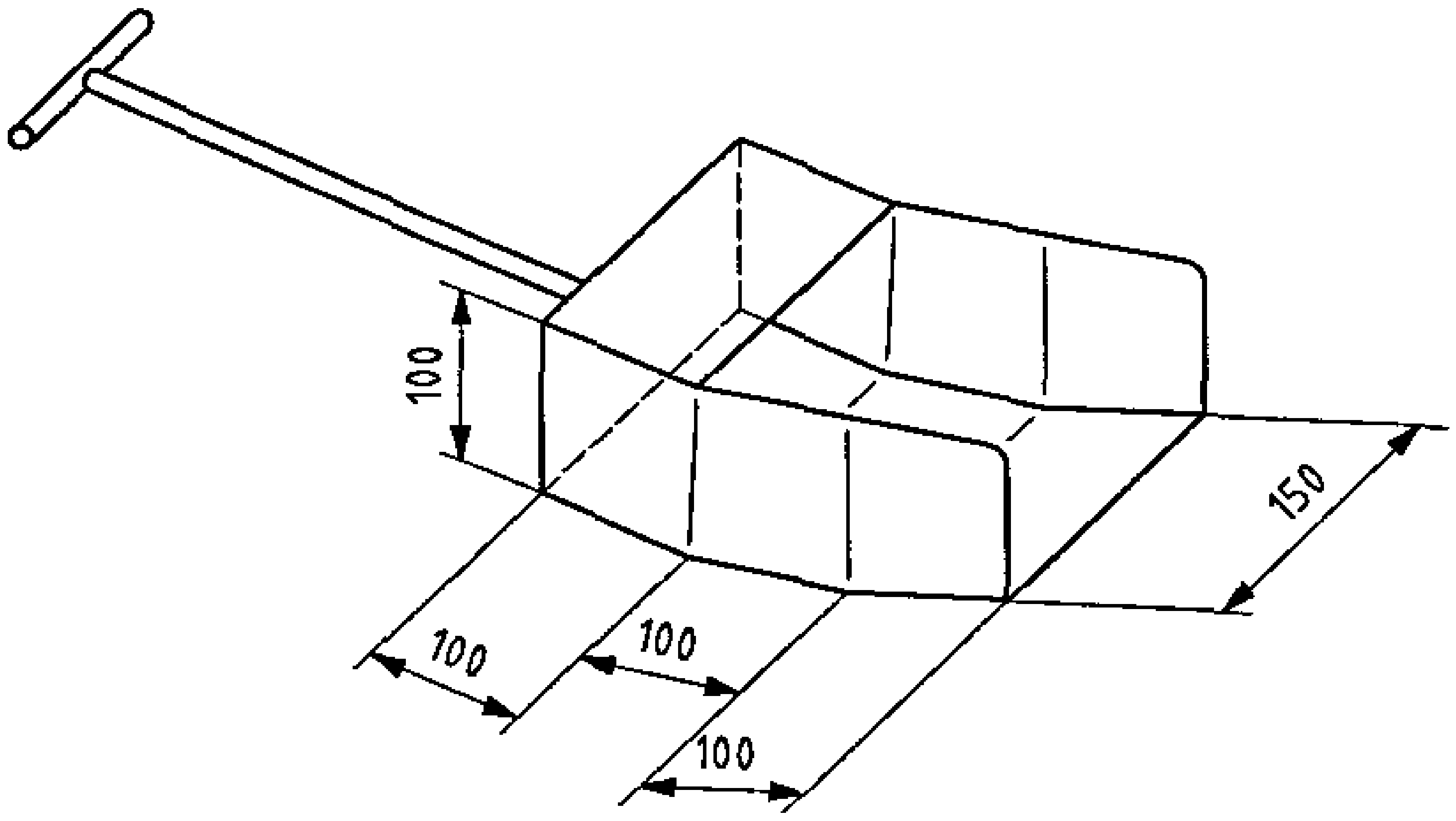


a) **Ladle for smalls up to 25 mm**

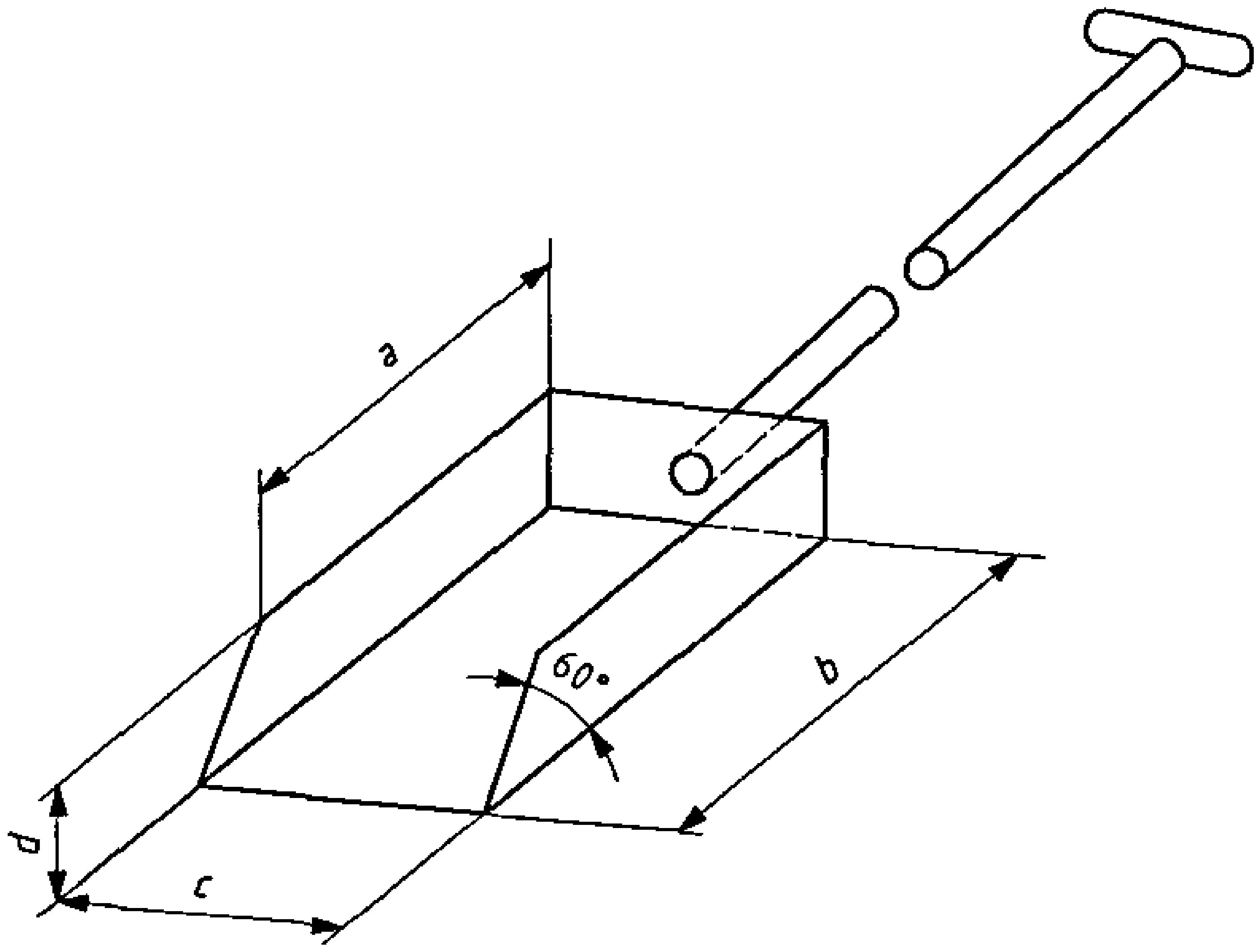


b) **Shovel**

Figure 2 (*continued*)

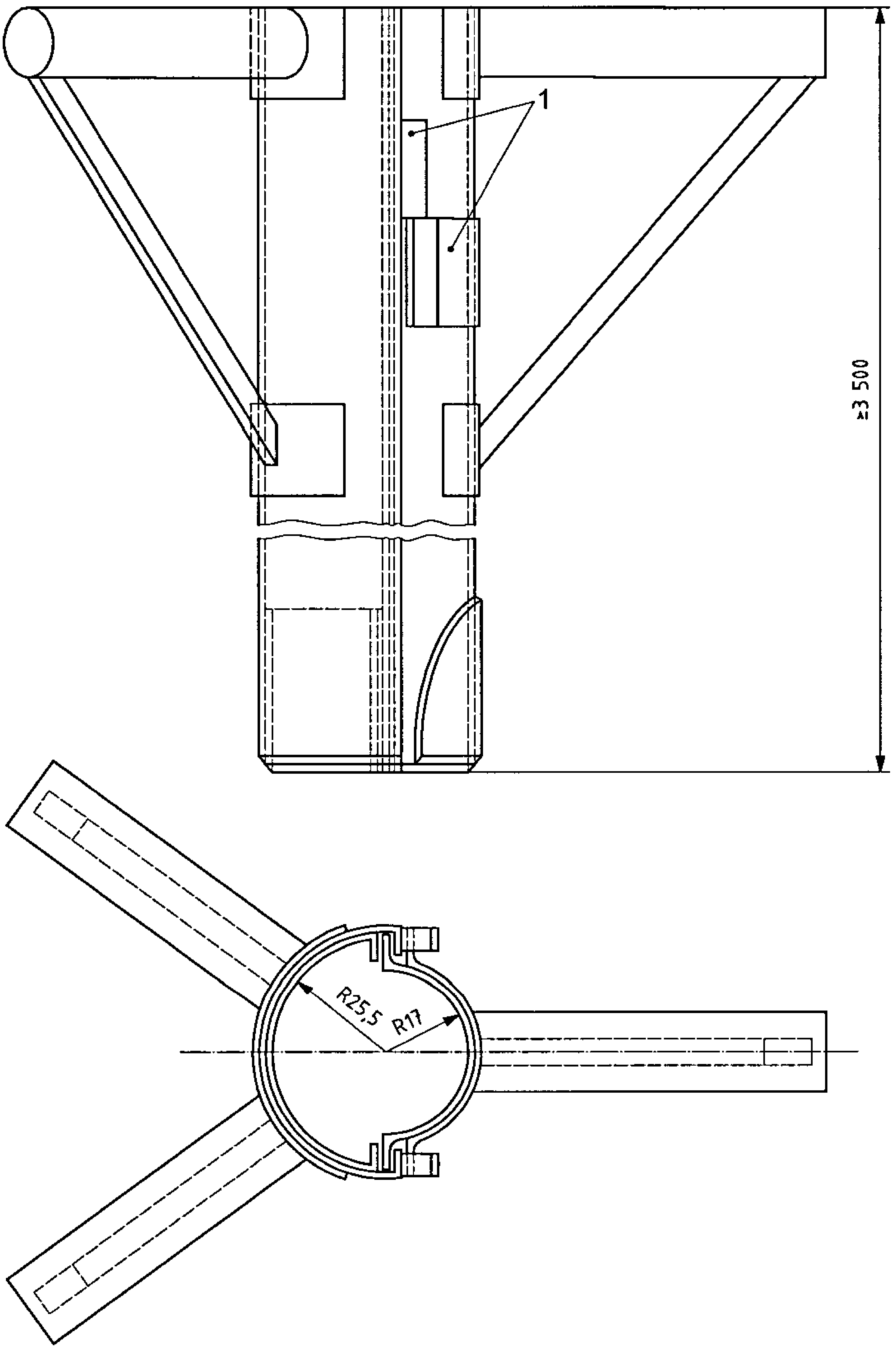


c) Scoop, suitably dimensioned for coal of 50 mm top size



d) Scoop

Figure 2 (*continued*)

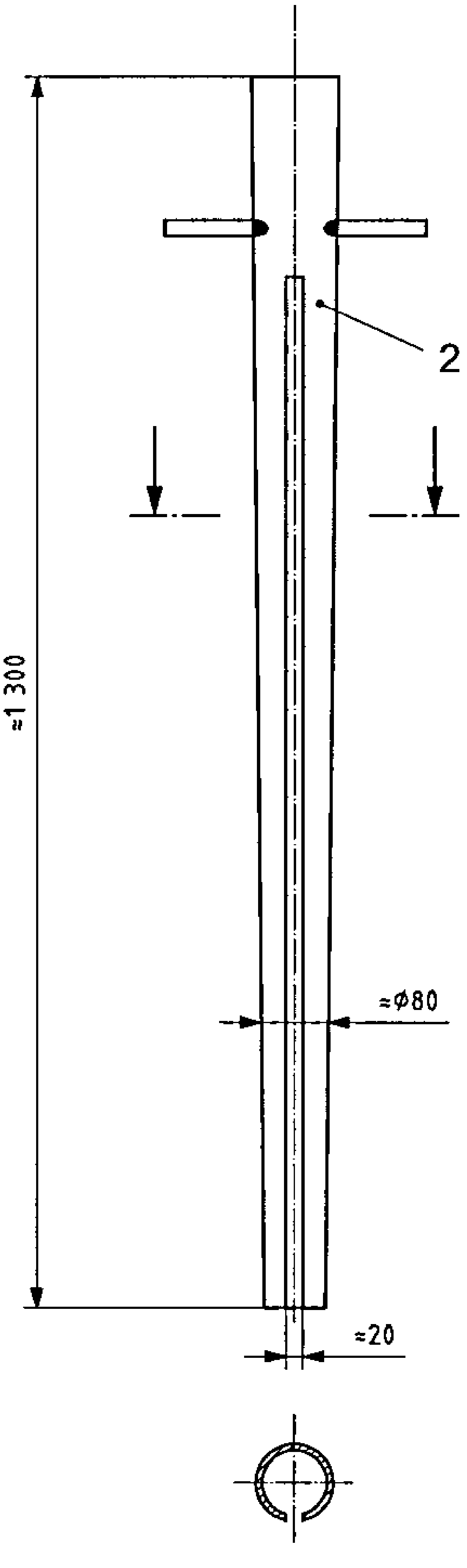


Key

1 trip dog for discharging

e) **Probe**

Figure 2 (*continued*)

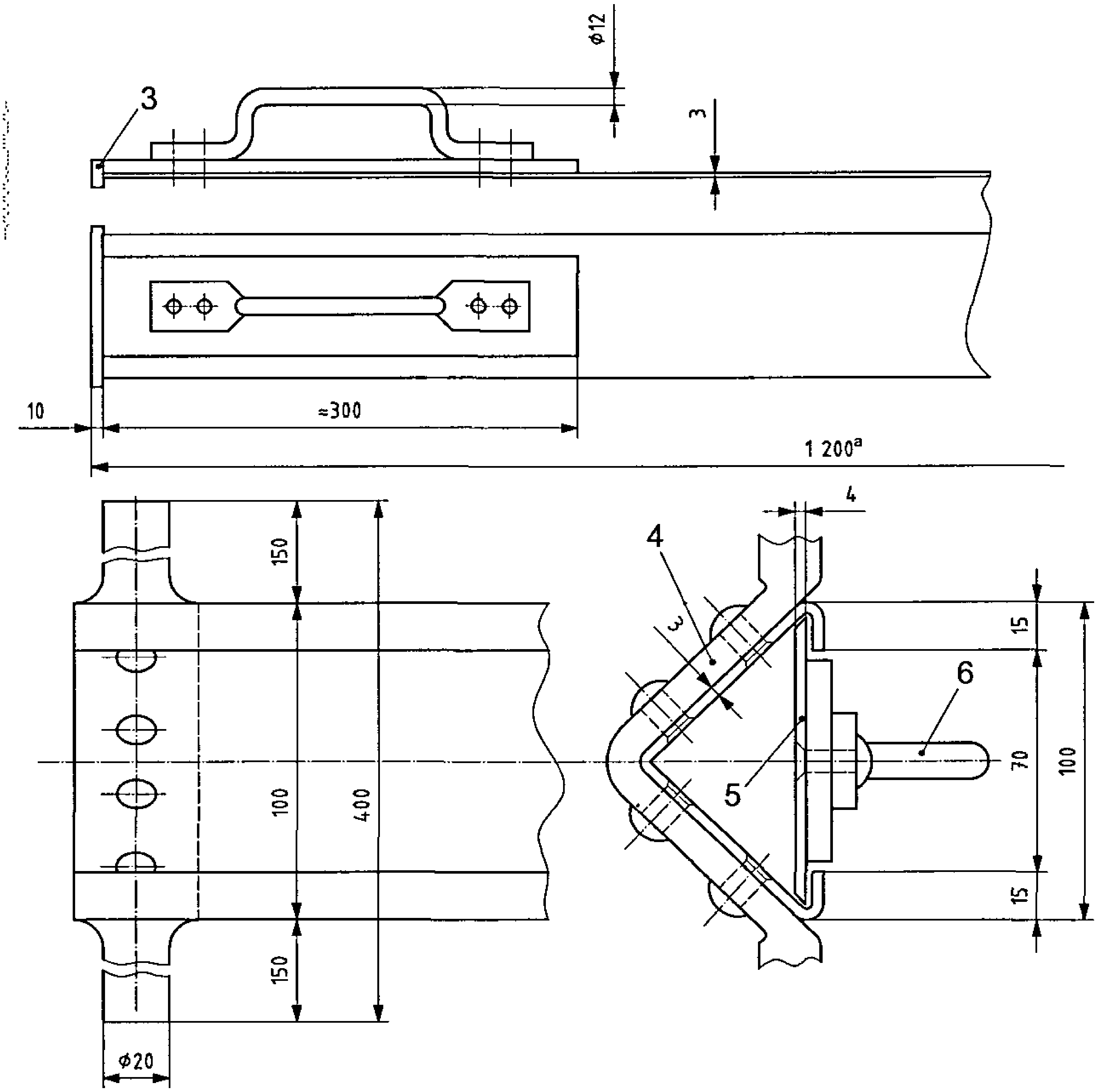


Key

2 slightly tapered tube

f) **Probe**

Figure 2 (*continued*)



Key

3 strengthening piece acting as a stop

4 upper support of probe body

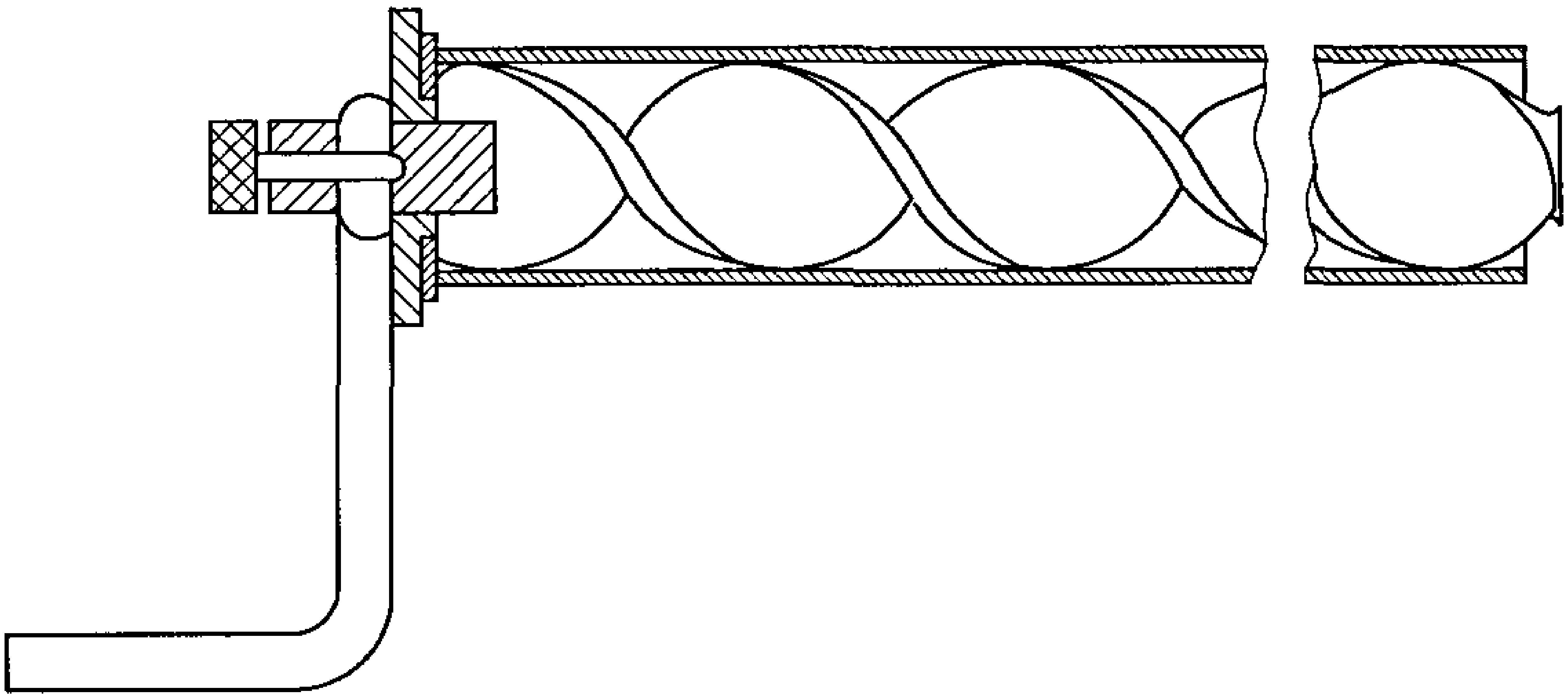
5 plate

6 handle of plate

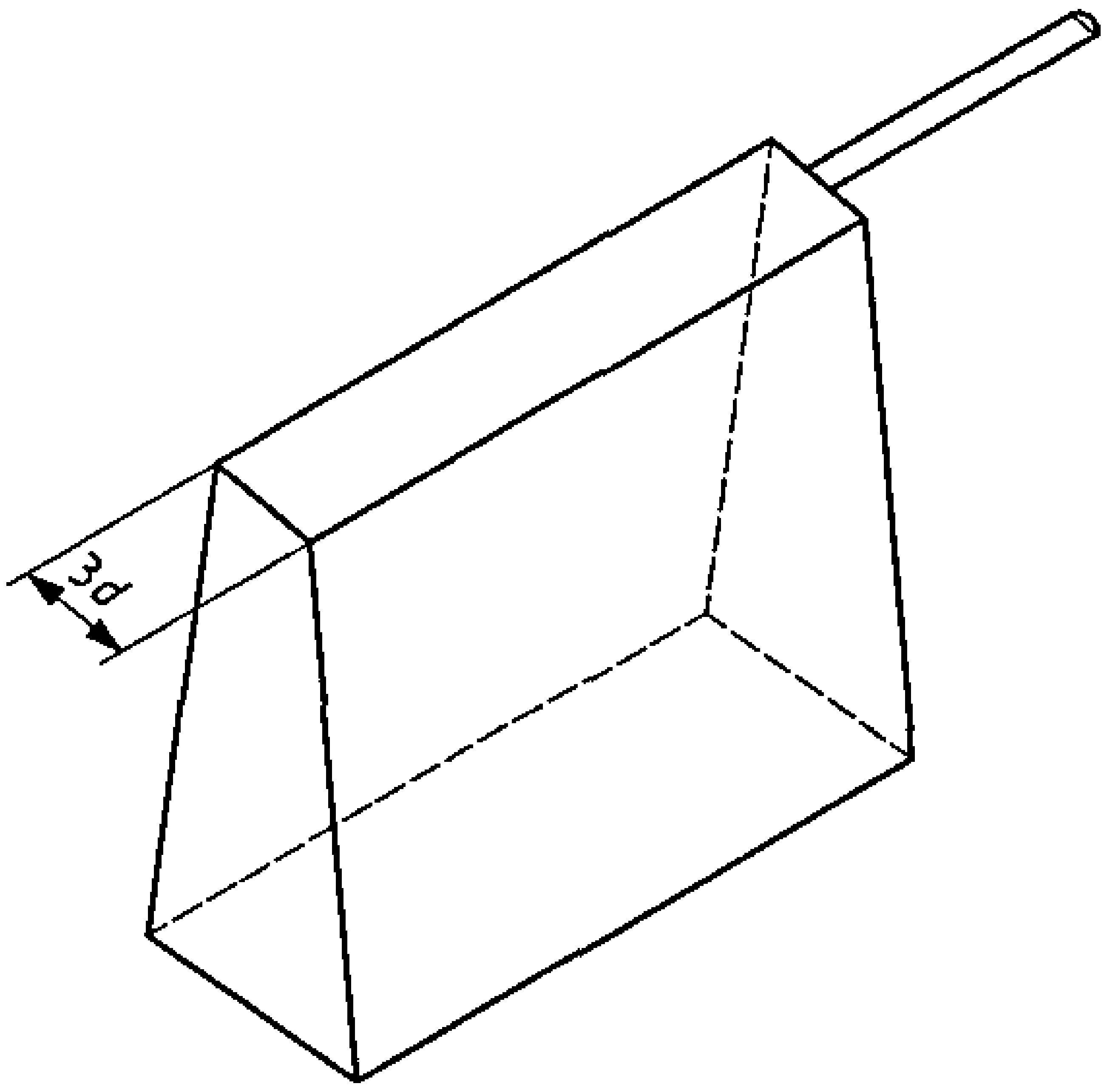
a Length of body of probe and plate.

g) Probe

Figure 2 (*continued*)

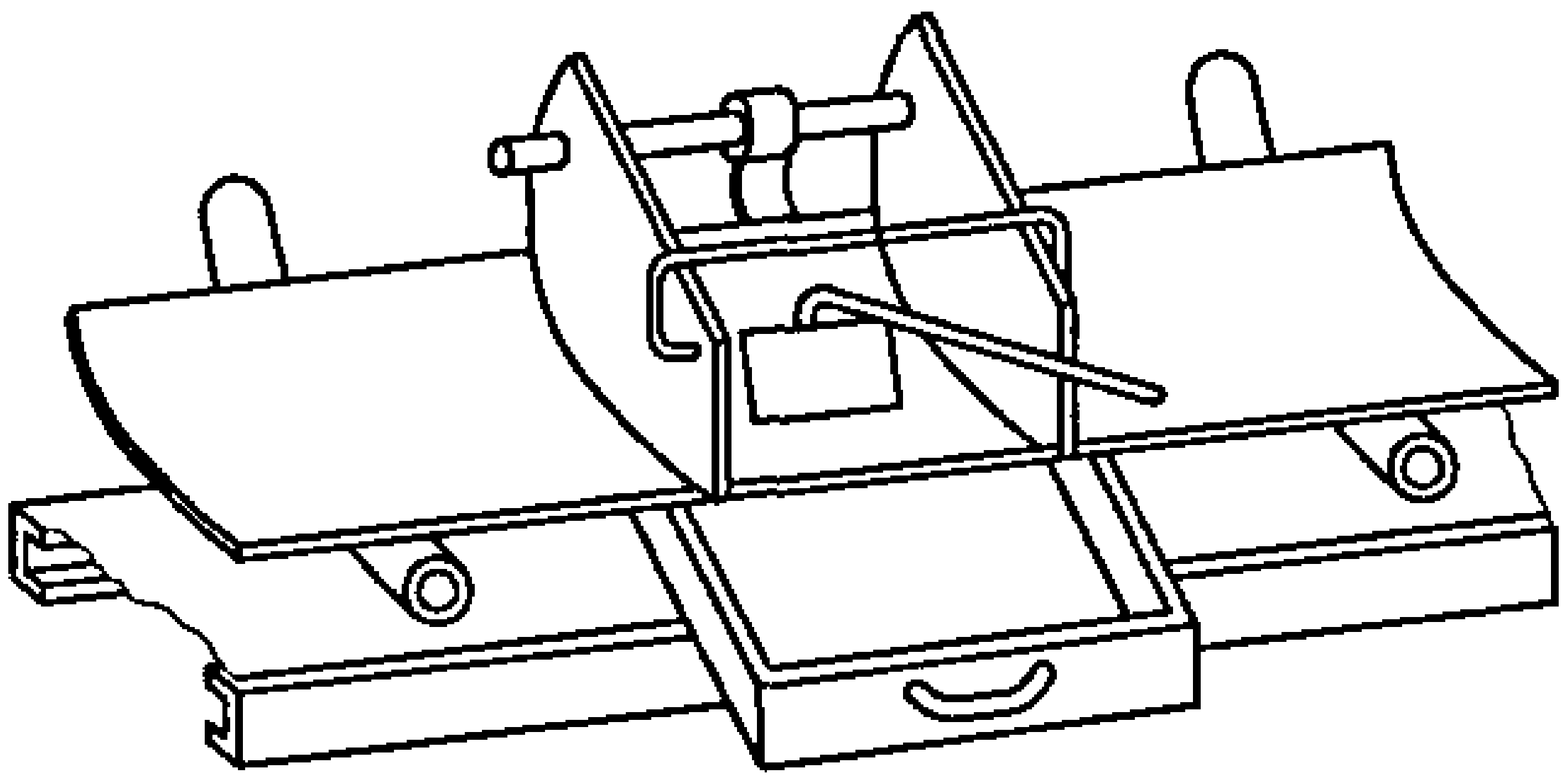


h) Auger



d nominal top size of the fuel

i) Manual cutter



j) Sampling frame

Figure 2 — Examples of sampling equipment

7 Handling and storage of samples

7.1 Sample size

The size of the samples to be handled and stored has to conform to the minimum masses and relevant nominal top sizes given in this International Standard (see Table 3).

7.2 Time

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

Test samples shall be kept available under good custody for an agreed period after issuance of the formal final sampling report (see Clause 9). Certain types of fuel do not allow a long storage time.

7.3 Divided sample

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

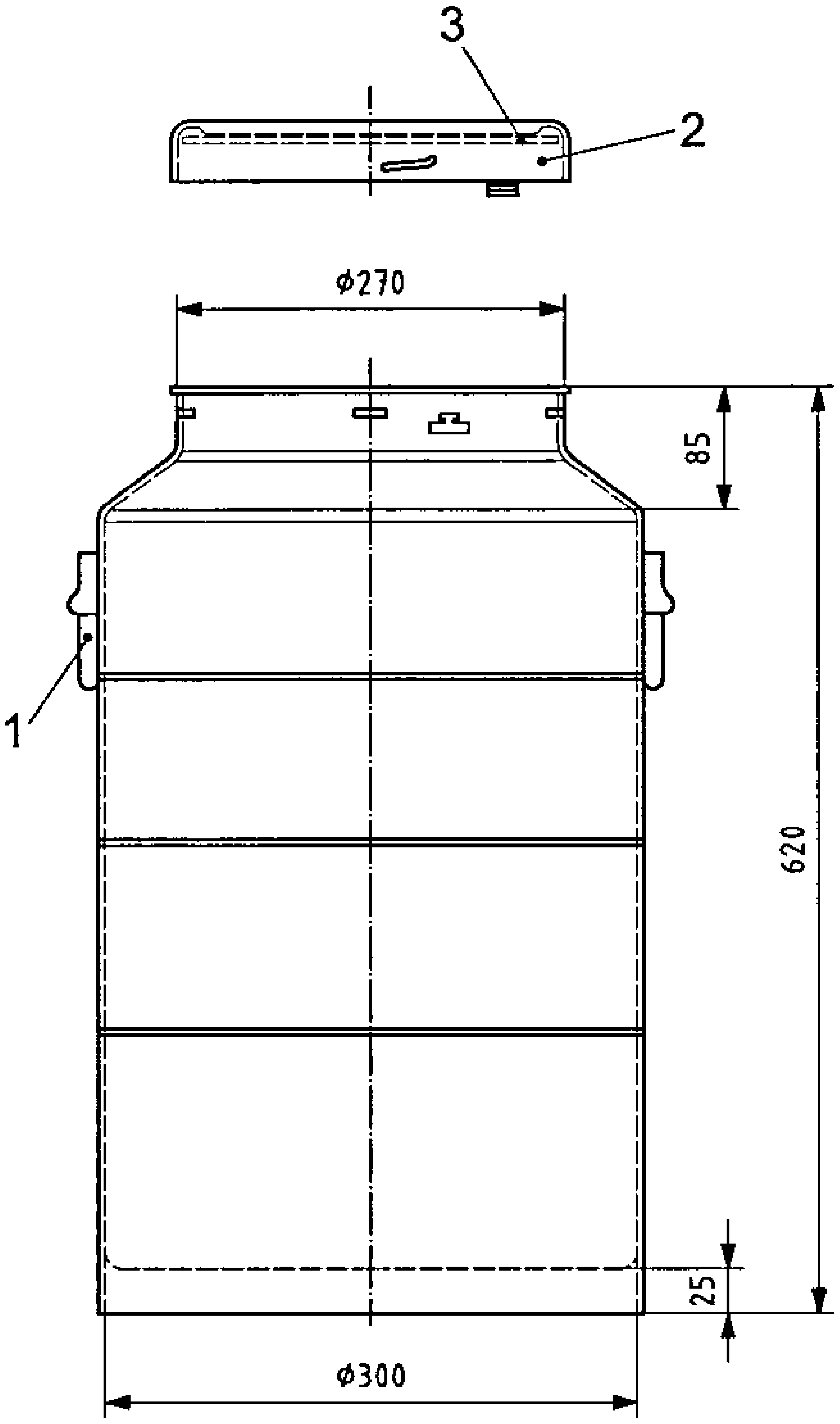
7.4 Containers

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

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

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

Dimensions in millimetres



Key

1 handle
2 lid
3 rubber insert

Figure 3 — Example of a sample container

7.5 Moisture loss/breakage or degradation

If physical test samples are required, the containers for such samples shall give adequate protection against loss of sample material, but they are not required to be fully impervious to water and vapour. Such samples should be carefully handled in all stages and under all circumstances to prevent breakage and/or degradation.

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

7.6 Identification/labelling

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

It is recommended that for this purpose the container be provided with two waterproof tags, each marked by means of waterproof ink with adequate identifying information, one tag being placed on the outside of the container and one being placed inside the container; if a plastic inner liner is used, the latter tag should be placed inside this liner.

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

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

8 Sample preparation

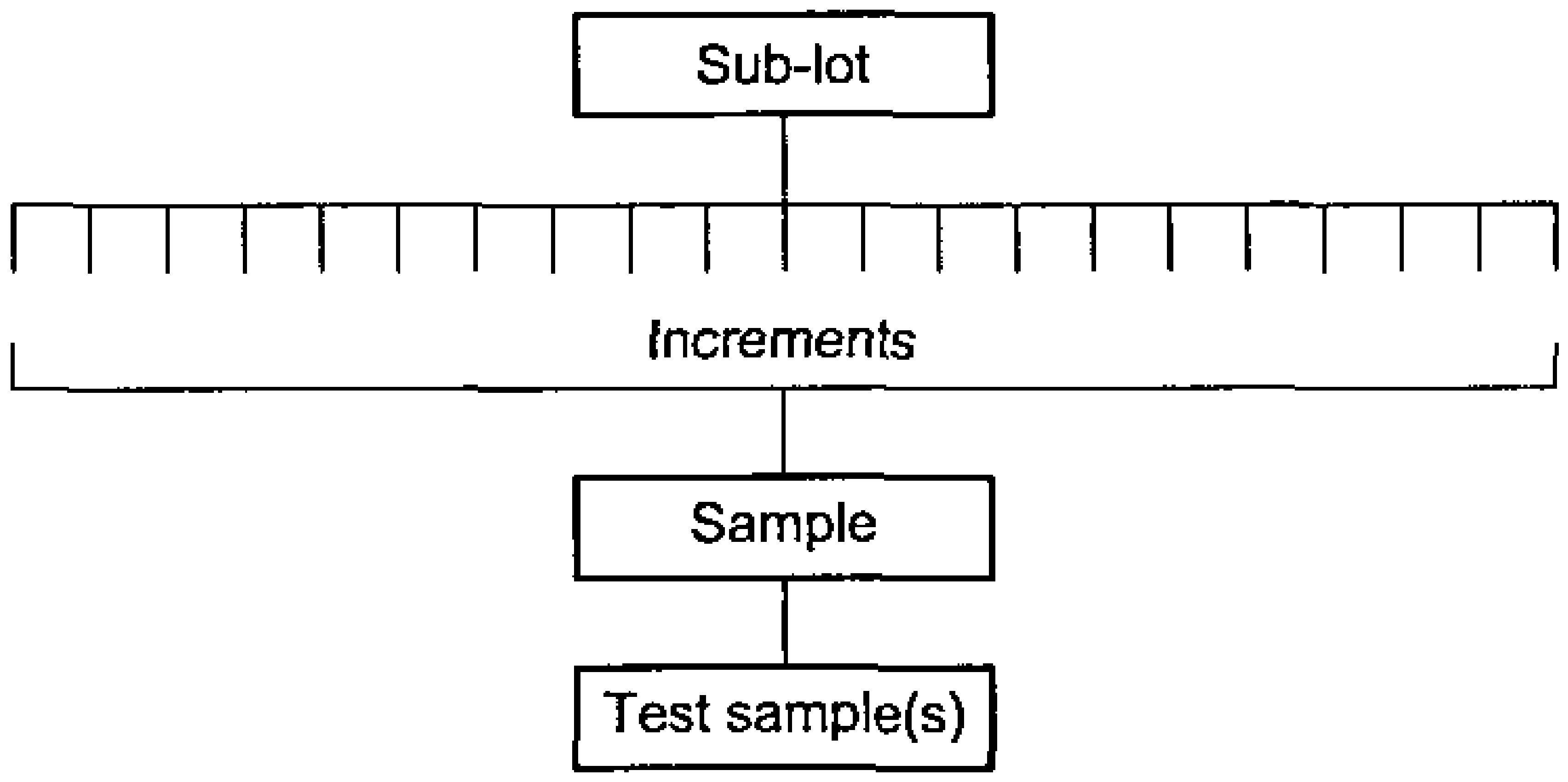
8.1 *General*

Clause 8 describes the preparation of samples of coal and coke from the combination of primary increments to the preparation of samples for specific tests. For the precision of sample preparation, see 4.4.

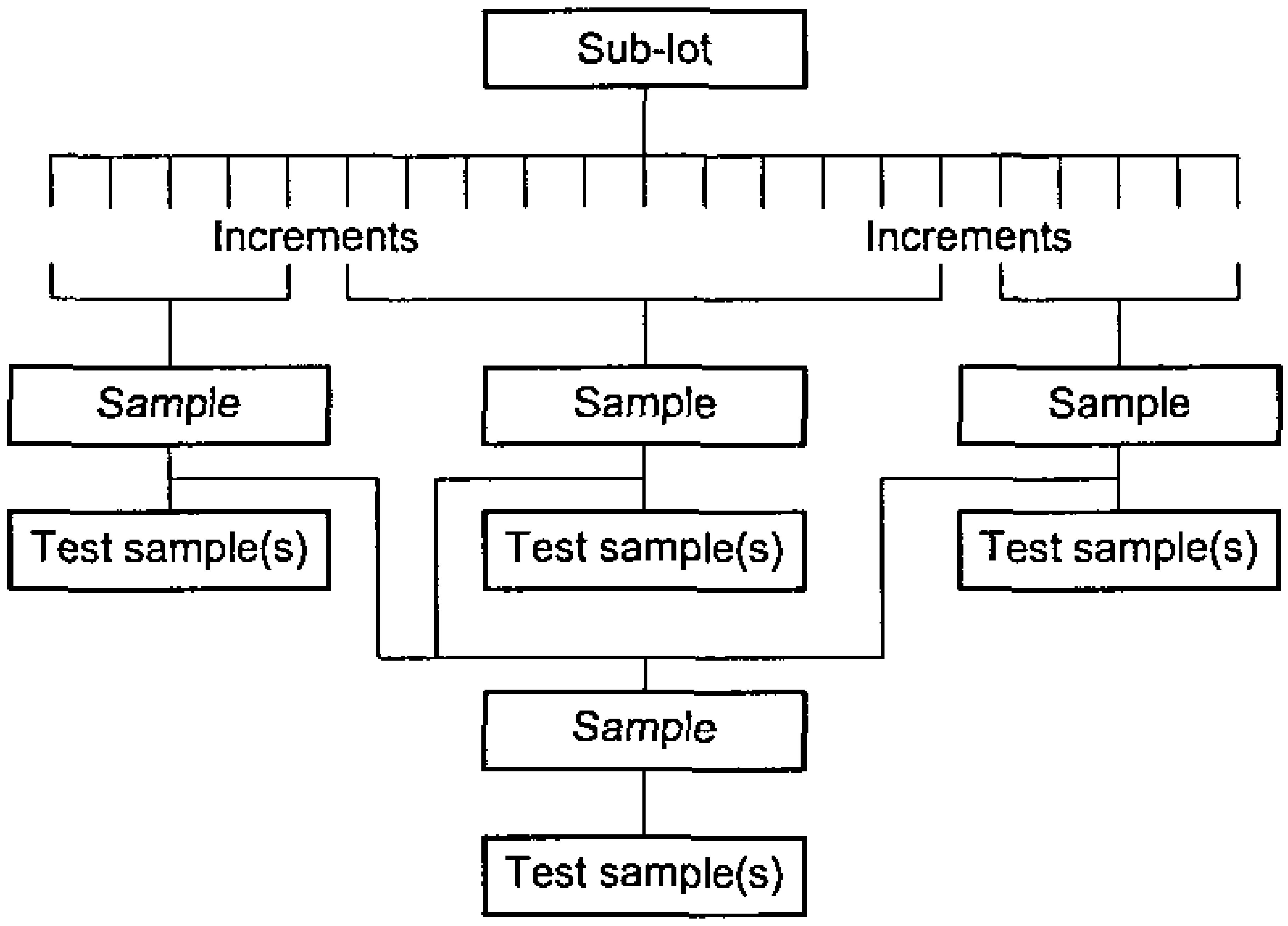
8.2 Constitution of a sample

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

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



a) **Example 1**



b) **Example 2**

Figure 4 — Examples of the constitution of test samples

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

8.3 Division

8.3.1 General

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

Mechanical dividers are designed to extract one or more parts of the fuel 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 can be necessary.

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

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

Examples of equipment for dividing samples are riffles and rotary dividers.

8.3.2 Riffle divider

A *riffle* [*see* Figures 5 a) and 5 b)] is a sample divider that, in a single pass of a sample, divides it into halves, one of which is retained and the other *normally* is rejected. The device is normally portable and, for sample division, is usually fed manually, the fuel being evenly distributed along its length. Adjacent slots feed opposite receivers.

NOTE Closed types of riffle dividers fitted with a feed gate allow actual placement of the feed evenly before opening the slots.

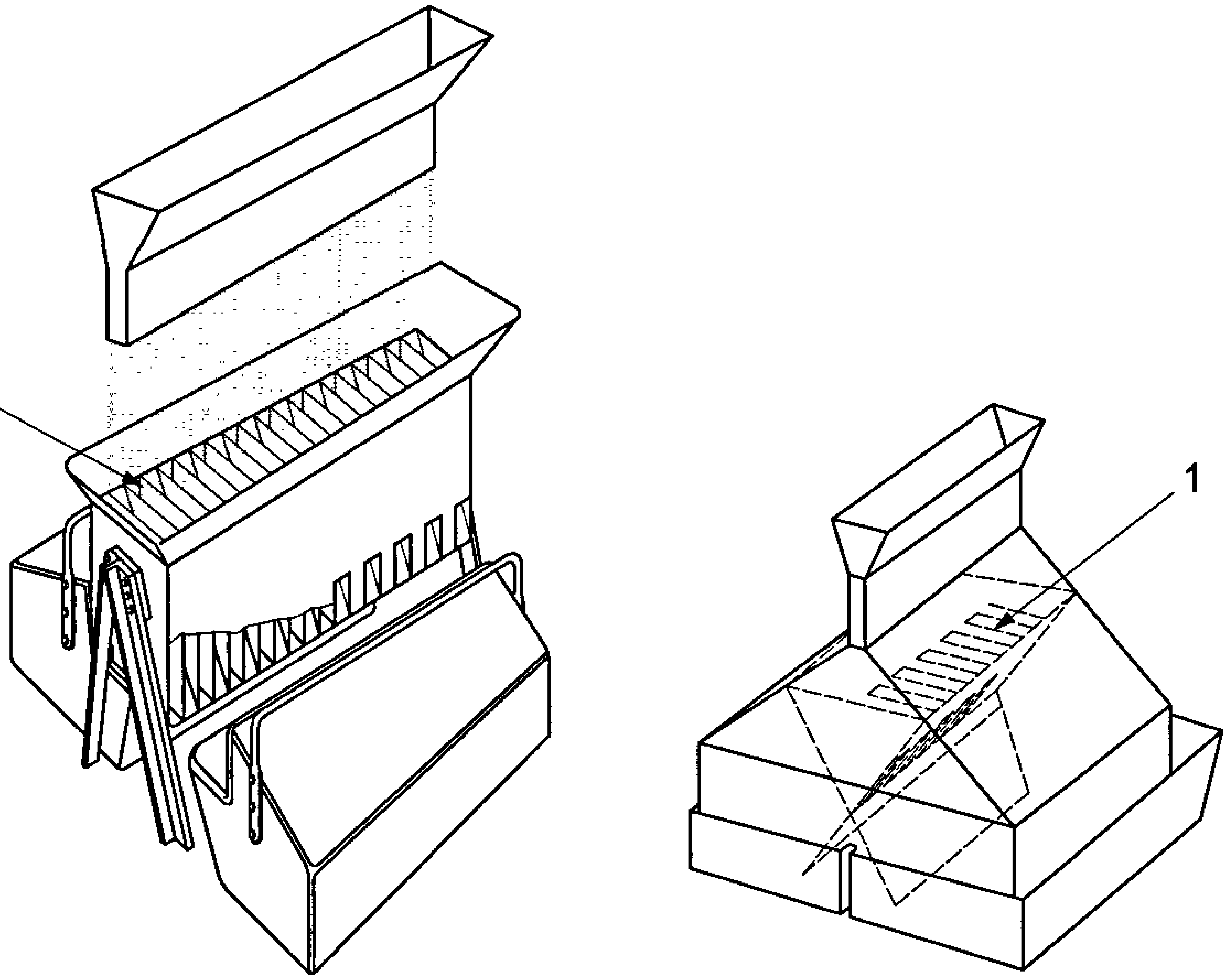
The slot width shall be at least two times the nominal top size. Each half of the riffle shall have the same number of slots, which shall be at least eight and preferably more. All the surfaces on which the fuel might rest shall have a slope of at least 60° to the horizontal.

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

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.

8.3.3 Rotary divider

Figures 6 a) to d) illustrate examples of rotary dividers.



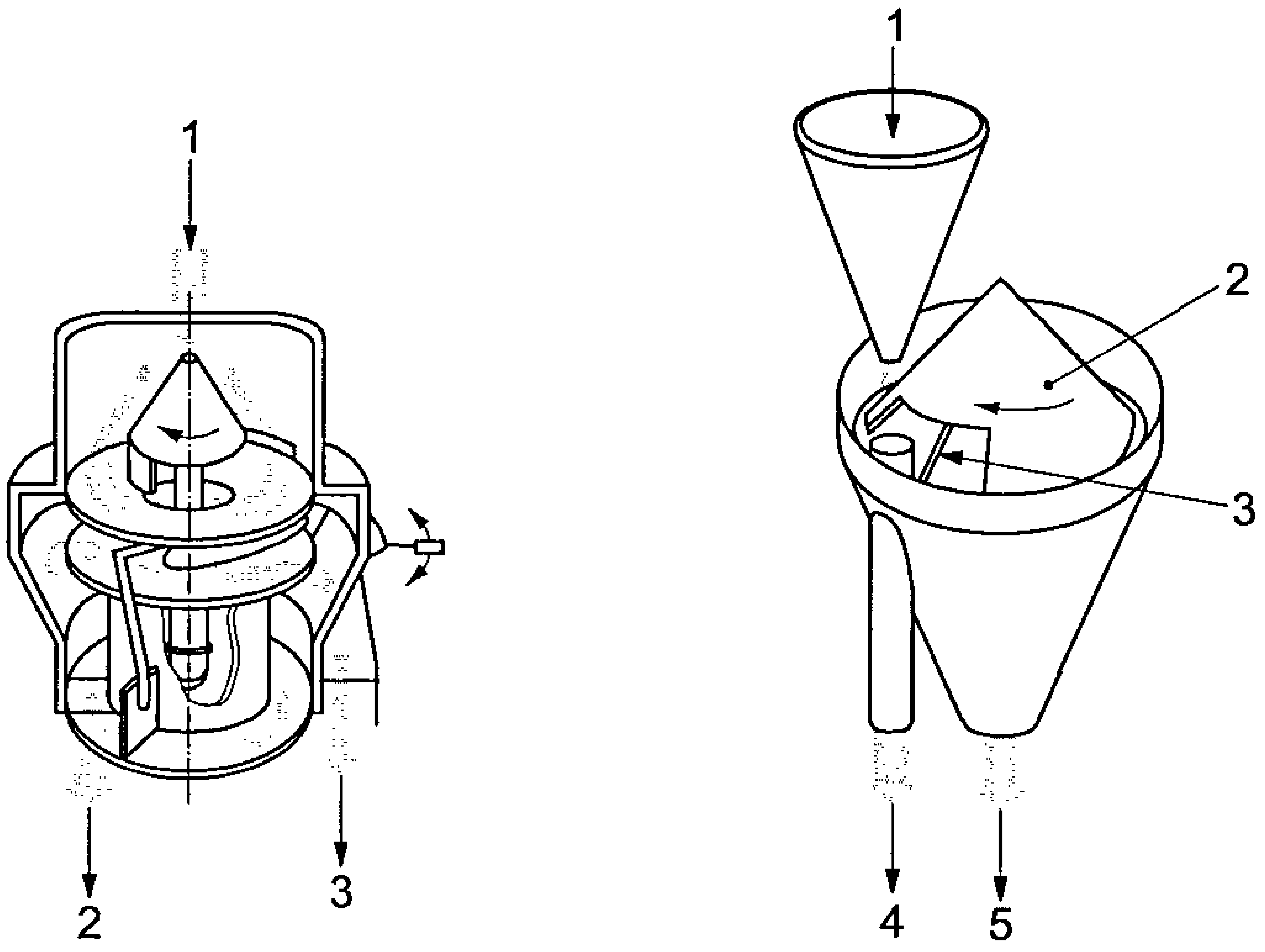
a) Open type

b) Closed type

Key

1 even number of slots

Figure 5 — Examples of riffles



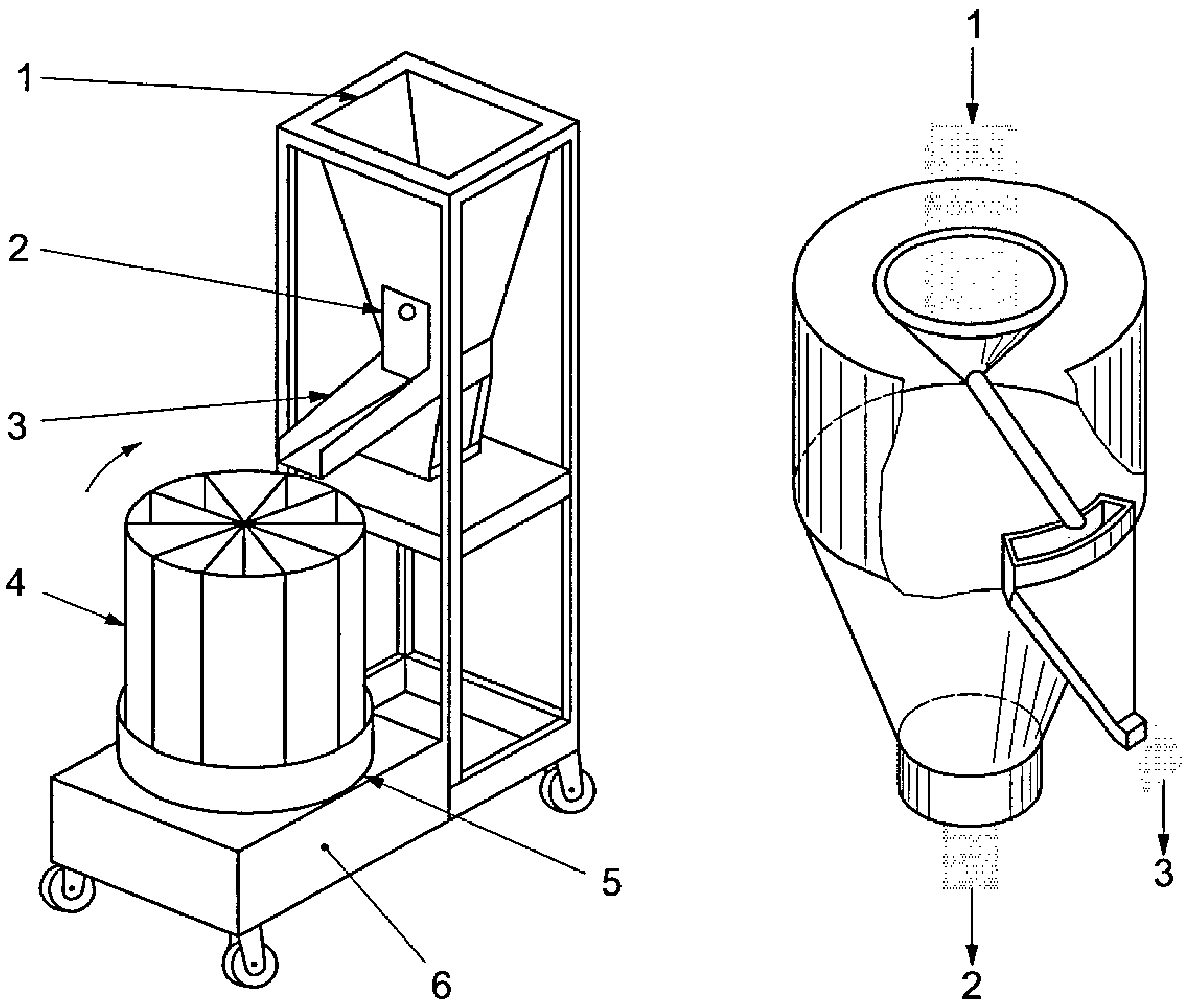
a) Rotating disk type

b) Rotating cone type

Key

1 feed hopper
2 slide gate
3 vibratory feeder
4 removable canisters
5 turntable

Figure 6 — Examples of dividers



c) **Container type**

d) **Rotating chute type**

Key

1	feed hopper
2	slide gate
3	vibratory feeder
4	removable canisters
5	turntable
6	drive (enclosed)

Figure 6 (*continued*)

8.3.4 Mechanical methods

8.3.4.1 General

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

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

NOTE 1 The procedures described for fixed-ratio division are the simplest to implement. Other procedures can be used, however, provided that the mass of the divided sample is proportional to the mass of the feed. For example, the number of cuts can be kept constant by making the feed rate of each division proportional to the mass of fuel to be divided.

The cuts shall be of uniform mass throughout division. In order to achieve this, the flow of fuel 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 fuel to be divided.

Division devices shall

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

b) not introduce bias, for example by selective collection (or rejection) on the basis of particle size or by loss of moisture,

NOTE 2 In order to avoid moisture loss, in some circumstances totally enclosed dividers can be necessary.

c) use a method of feeding that minimizes the segregation of the fuel,

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

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

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

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

8.3.4.2 Division of individual increments

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

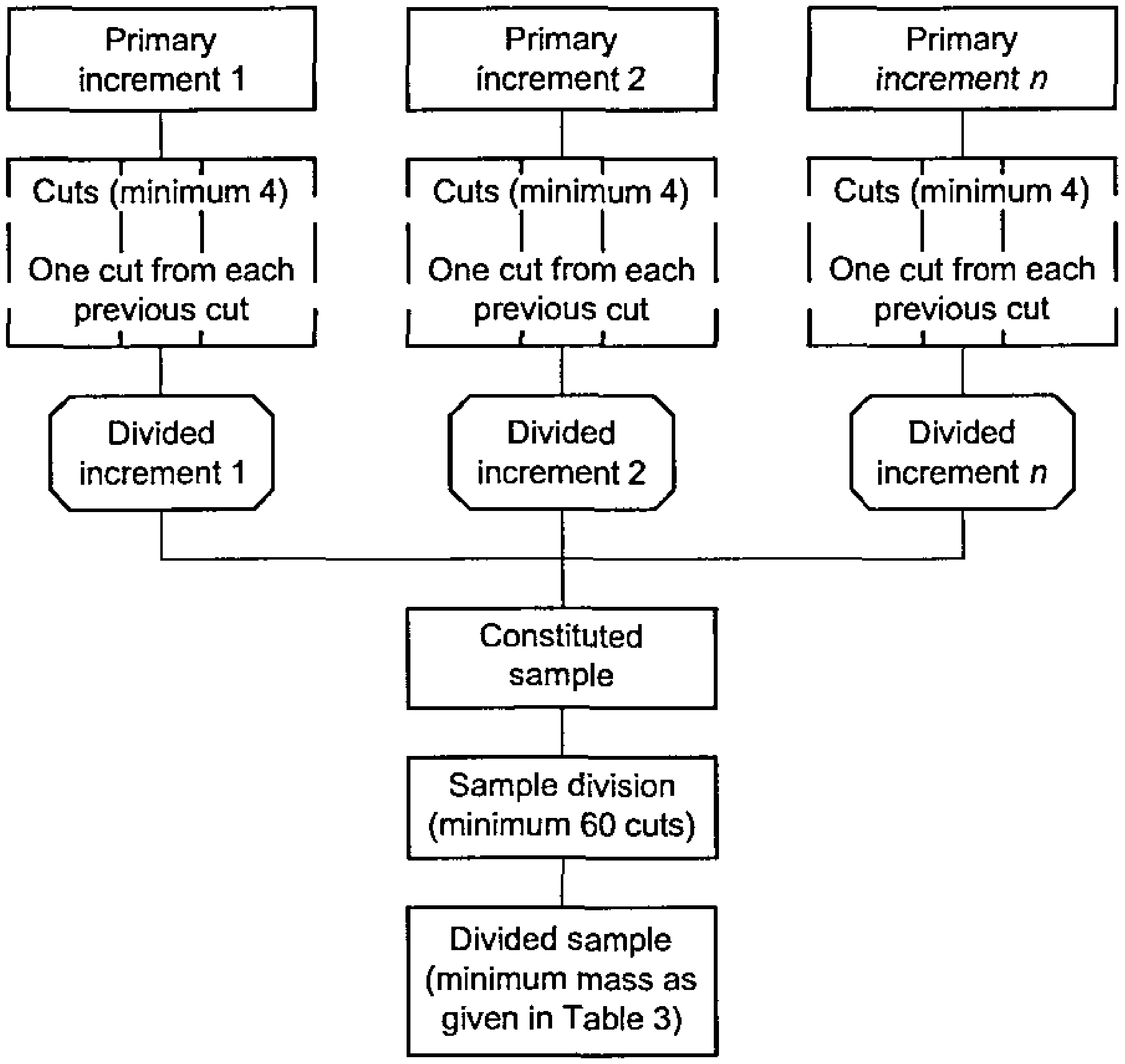
a) For fixed-mass division, the minimum number of cuts for dividing primary increments shall be four. An equal number of cuts shall be taken from each increment in the sub-lot.

b) For fixed-ratio division, the minimum number of cuts for dividing a primary increment of mean mass shall be four.

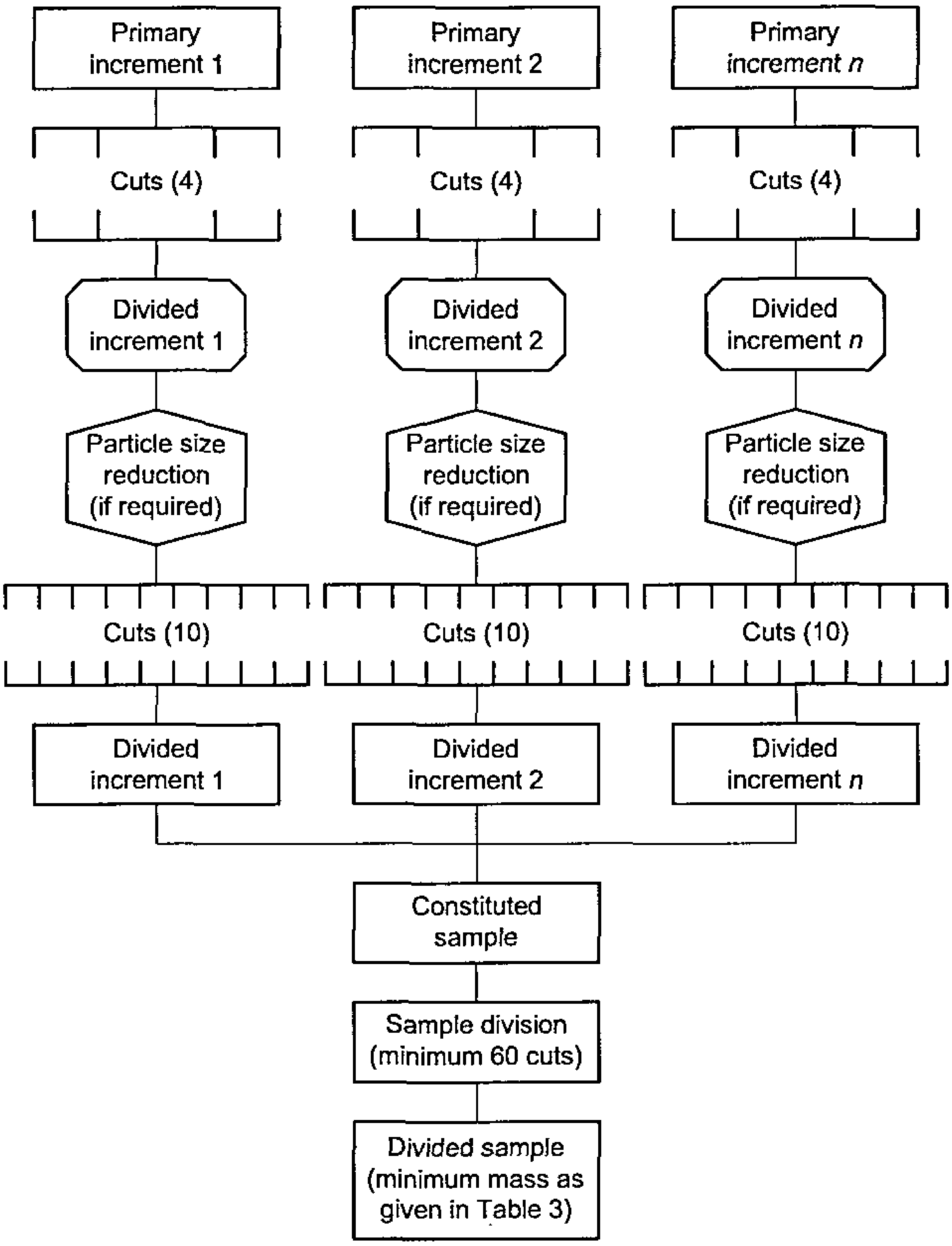
c) For subsequent division of individual divided primary increments, a minimum of one cut shall be taken from each cut from the preceding division.

Examples of procedures for division of individual increments and subsequent sample division are shown in Figure 7.

Values for the minimum mass of samples (primary as well as after division) are shown in Table 3 (see 4.4.10).



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



b) Example of two-stage division of individual increments

Figure 7 — Examples of procedures for division of increments and samples

8.3.4.3 Division of samples

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

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

For most parameters, particularly size analysis and those that are particle-size related, the precision of the result is limited by the ability of the sample to adequately represent all the particle sizes in the mass of fuel being sampled. The attainment of the required minimum mass after division does not, of itself, guarantee the required precision, because division precision is also dependent on the number of cuts taken during division (see 8.3.4.2).

8.3.5 Manual methods

8.3.5.1 Riffle method

A riffle (see Figure 5) is a sample divider that, in a single pass of a sample, divides it into halves, one of which is retained and the other is normally rejected. The device is normally portable and, for sample division, is usually fed manually, the fuel being evenly distributed along its length. Adjacent slots feed opposite receivers.

For a description of a riffle, see 8.3.2.

8.3.5.2 Flattened-heap method

The procedure, which is illustrated in Figure 8, is as follows.

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

   For wet fuels, thorough mixing, which might result in loss of moisture, should be avoided.

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

The increments shall be of uniform mass. The minimum mass required for each nominal top size is the mass of the divided sample (see Table 3) divided by the number of parts of the flattened heap.

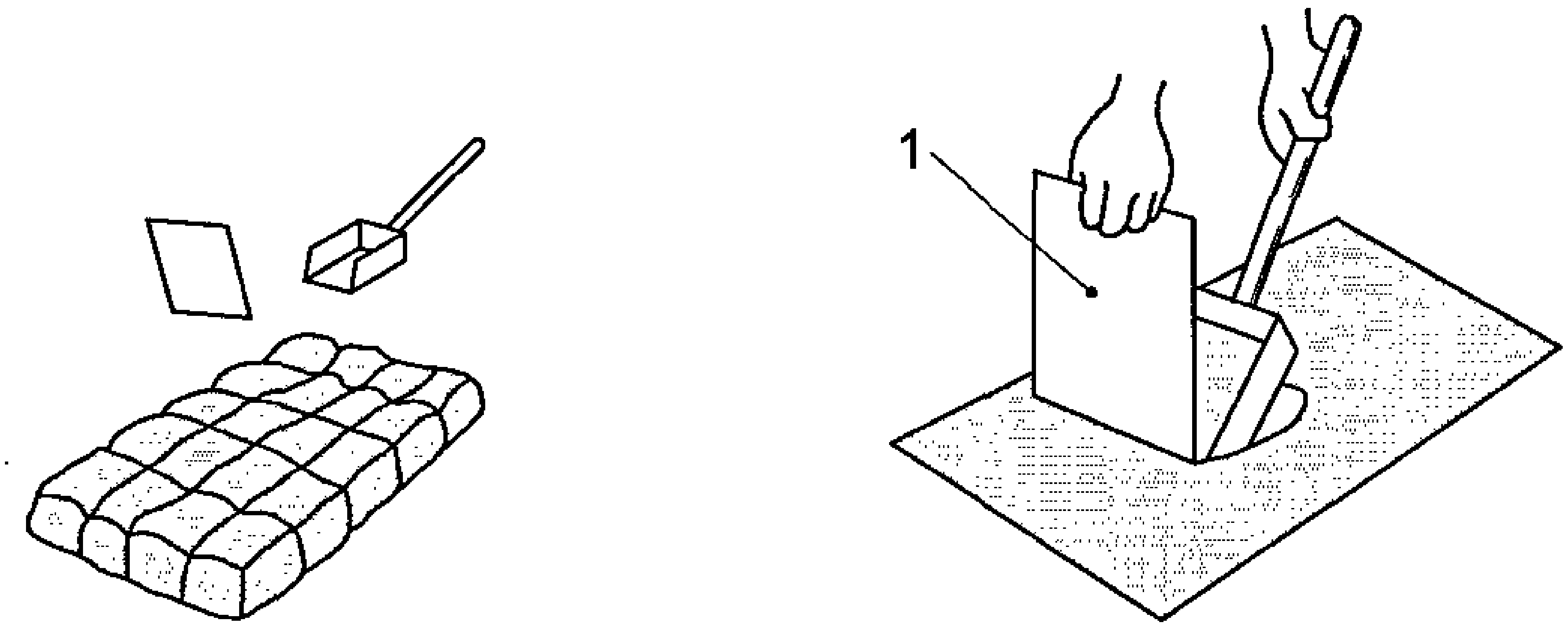
The scoop shall be flat bottomed and the width of the entry shall be at least three times the nominal top size of the fuel. The side walls shall be higher than the height of the heap and the depth shall be sufficient to allow the required mass of increments to be taken.

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

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

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

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



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

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

Key

1 bump plate

Figure 8 — Flattened-heap method

8.3.5.3 Strip-mixing and splitting method

The procedure, which is illustrated in Figure 9, is as follows.

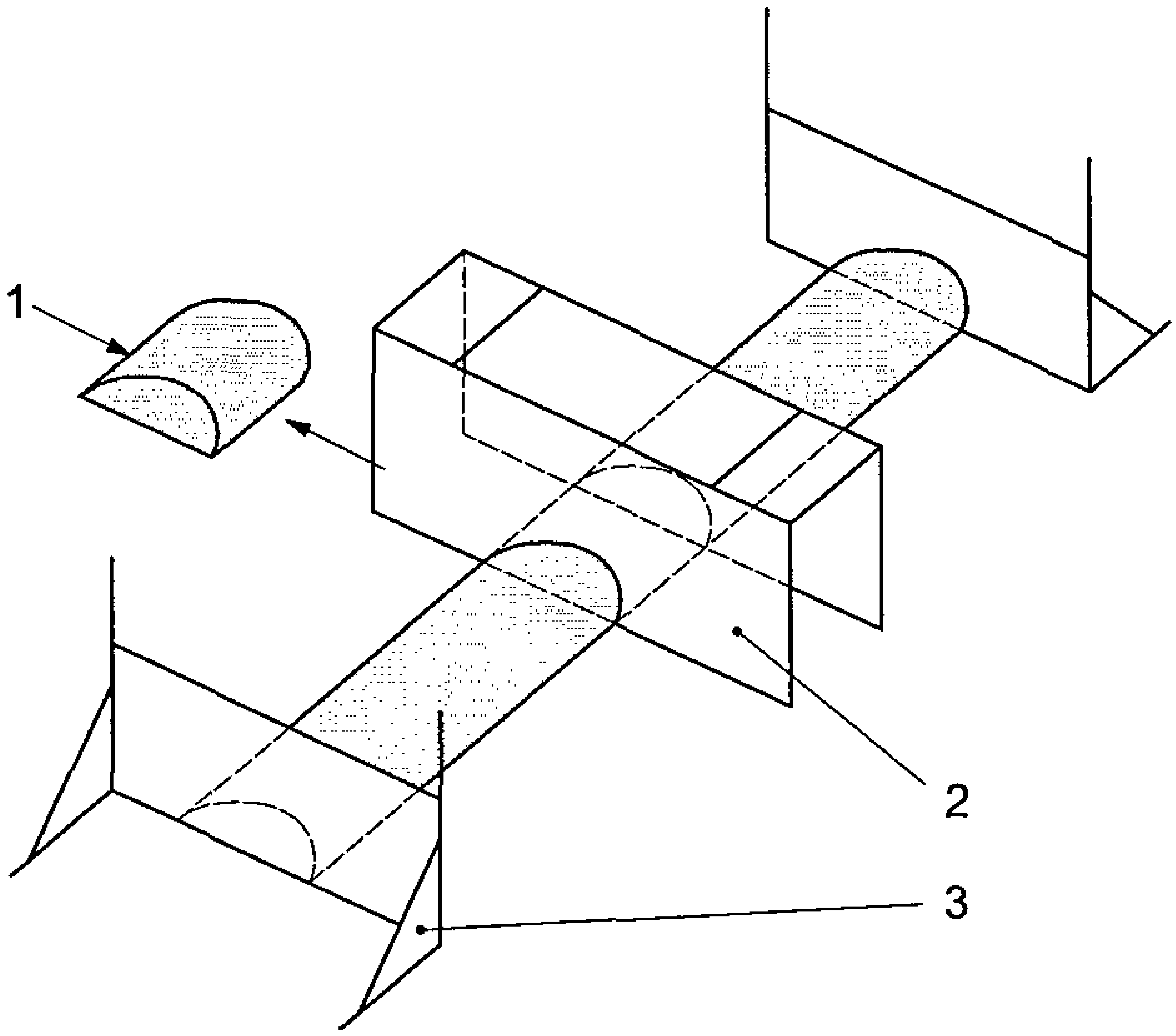
a) The fuel sample is formed on a mixing plate, which is a smooth, non-absorbent, non-contaminating surface, into a strip at least 10 times as long as it is wide by distributing the fuel along the length of the strip as evenly as possible, working randomly from end to end and from both sides of the strip.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

b) Increments shall be taken as a complete section across the strip. The width of each *cross-section shall be* not less than three times the nominal top size of the fuel.

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

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

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



Key

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

Figure 9 — Strip-mixing and splitting method and device

8.4 Reduction

8.4.1 General

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

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

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

8.4.2 Reduction mills

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

There shall be no contact between the metal surfaces of the mill in order to avoid localized heating of the sample. 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 depends on the speed range.

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

Hammer mills or roll mills are most suitable for crushing coals during preparation. In general practice, samples for general analysis are milled to pass 212 µm top size using a small high-speed hammer mill with a screen.

8.5 Mixing

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

One method that can be used is to pour the sample through a riffle (8.3.5.1) or a container-type sample divider [see Figure 6 c)] three times, reuniting the parts after each pass. If mechanical sample dividers are used in the course of preparation, an additional mixing step is not normally necessary to meet the required precision.

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

8.6 Air-drying

The procedure for air-drying is as follows.

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

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

The recommended times to attain equilibrium at different ambient temperatures up to 40 °C are given in Table 6. The times recommended in Table 6 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 fuels susceptible to oxidation.

Table 6 — 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 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 temperature, a cabinet or oven with appropriate air-change facilities shall be used. If drying has been carried out at such temperatures, the sample shall be cooled until moisture equilibrium at normal ambient temperature is achieved before reweighing. The cooling period required depends on the drying temperature. For example, 3 h is normally sufficient if the sample has been dried at 40 °C. If the change in mass of the sample over a period of 1 h is less than 0,1 % of its original mass, the sample is considered as air-dried.

Dry and weigh the container. Collect the air-dried sample in the original container and reweigh and calculate the percentage loss in mass. Continue the preparation for other tests.

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

After the moisture of the partially dried sample has been determined as described in 8.7.2 (coal) or 8.8.2.3 (coke), calculate the total moisture, M_T, as a percentage, from Equation (21):

$$M_T = X + M\left(1 - \frac{X}{100}\right) \qquad (21)$$

where

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

M is the moisture, determined as described in 8.7.2 (coal) or 8.8.2.3 (coke) and expressed as a percentage.

8.7 Coal — Preparation of test samples

8.7.1 Types of test samples

The following types of test samples may be prepared:

a) samples for determination of total moisture;

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

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

d) samples for size analysis;

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

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

8.7.2 Preparation of samples for determination of total moisture

The test sample for the determination of moisture shall be prepared to meet the requirements of ISO 589. If air-drying is performed at any stage of preparation, the percent loss in mass is recorded and included in the calculation of total moisture, as specified in ISO 589:2003, 8.2.

NOTE 1 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 procedures used.

Precautions shall be taken to minimize changes in moisture during preparation and due to the use of unsuitable containers. All samples for moisture determination shall be kept in sealed containers in a cool place, under cover, before and during preparation as well as during any interval between steps of sample preparation.

If excessive standing time causes bias, the number of sub-lots should be increased to overcome this problem (see 4.5).

Preliminary air-drying using the procedure described in 8.6 can be necessary in order to minimize moisture loss in any subsequent reduction/division stages. When carrying out division prior to air-drying of a sample or increment, take care to minimize change in moisture. 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 2 For coals that are too moist to flow through a sample divider and for which it is also impossible to air-dry the entire sample, it can be necessary to divide the sample by collecting increments from a flattened heap (8.3.5.2) or by strip-mixing and splitting (8.3.5.3). This divided sample is then air-dried.

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

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

If the particle size of the sample is so large that the mass given in Table 3 (see 4.4.10) makes its air-drying impracticable, the sample shall be crushed and divided before air-drying. Crushing shall be kept to the minimum necessary to allow division to a manageable mass, but in any event not less than 2,8 mm.

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

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

An example of a scheme for the preparation of a sample for a two-stage moisture test is given in Figure 10.

8.7.3 Preparation of samples for general analysis

The objective of general analysis sample preparation is to prepare a test sample that passes a sieve of 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 typically between 60 g and 300 g.

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

Air-drying (see 8.6) in connection with preparation for general analysis is carried out only to ensure that the fuels 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.

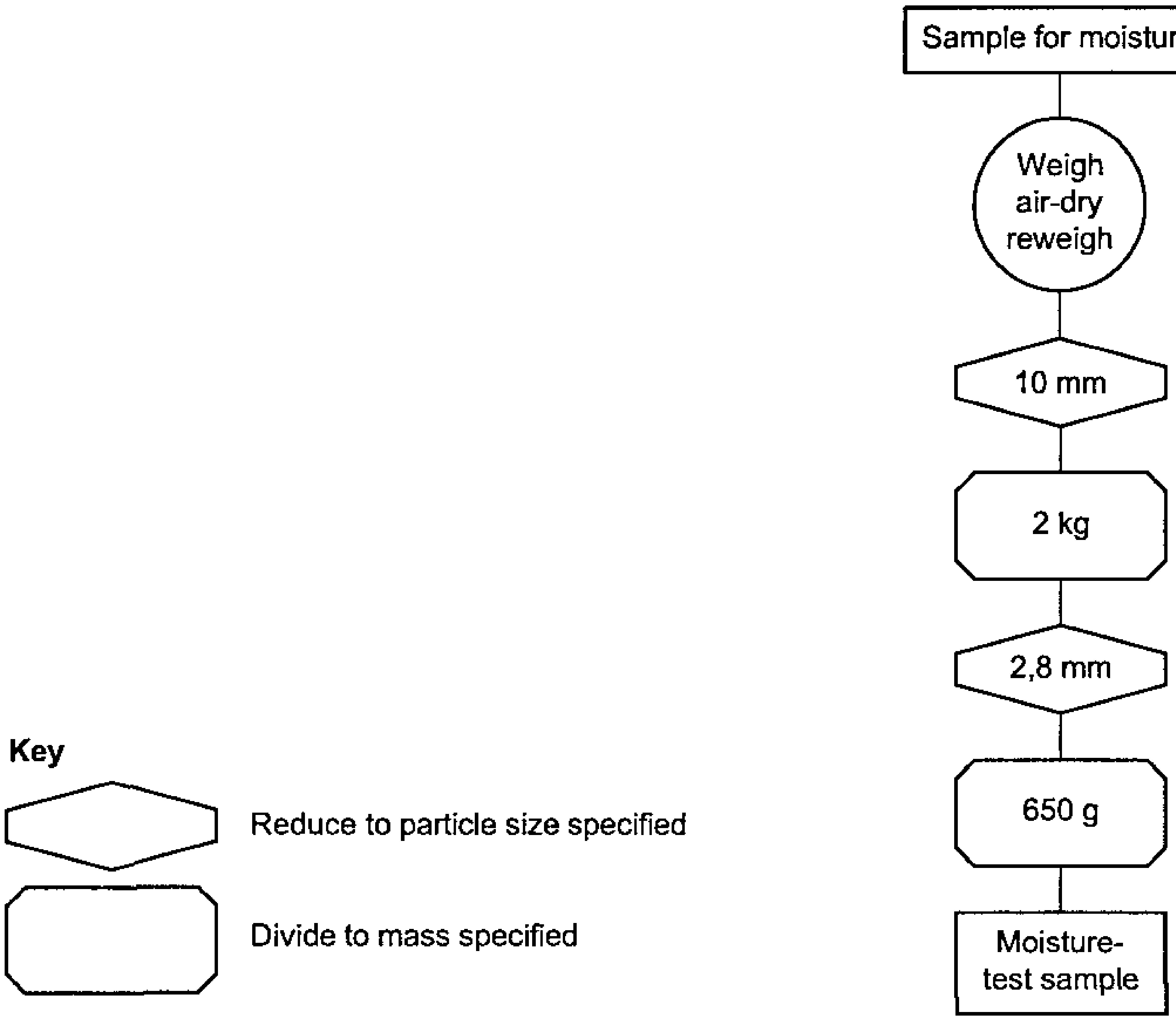


Figure 10 — Example of a two-stage moisture-test-sample preparation

Air-drying may be carried out at any stage, provided that it does not affect the quality of the sample. For example, if the sample is used for the determination of calorific value, coking properties 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.

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

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

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

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

If the original nominal top size of the coal is too large, or if the coal is too wet, an intermediate stage can 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 3 (see 4.4.10).

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 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 shall be spread out and 60 g to 300 g taken by hand in not less than 20 increments from various parts of the flattened heap.

An example of a scheme for preparation of general analysis is given in Figure 11.

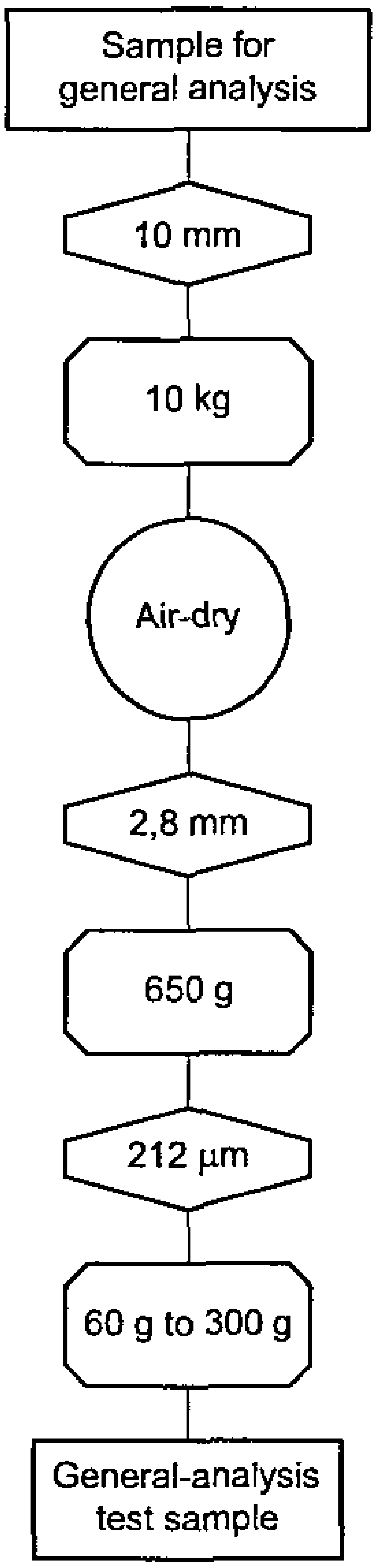


Figure 11 — Example of preparation of general-analysis test sample

8.7.4 Common samples

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

It is preferable to extract the moisture sample by use of a mechanically operated divider. The extraction of the moisture sample may be carried out at any convenient stage of the preparation procedure consistent with the requirements of 8.7.2. Prior to extraction, the sample shall be treated in accordance with 8.7.2 in order to avoid any inadvertent loss of moisture. If air-drying is part of the preparation prior to extraction, the loss of mass during the drying shall be measured, recorded and included in the calculation for total moisture as specified in ISO 589:2003, 8.2.

If the common sample is visibly wet and it is impossible to air-dry the entire sample, use a manual method. Extract a moisture sample by collecting increments by the flattened heap method (8.3.5.2) or by the strip-mixing and splitting method (8.3.5.3). Avoid further treatment of the moisture sample before air-drying to reduce the risk of bias in the moisture determination. Further treatment after air-drying shall be carried out as described in 8.7.2. The residual coal after extraction constitutes the sample from which the general-analysis sample is prepared and is treated as described in 8.7.3.

Examples of schemes for the preparation of separate test samples for moisture and for general analysis from a common sample are given in Figures 12 and 13. Sometimes a single test sample may be prepared for both moisture and general analysis.

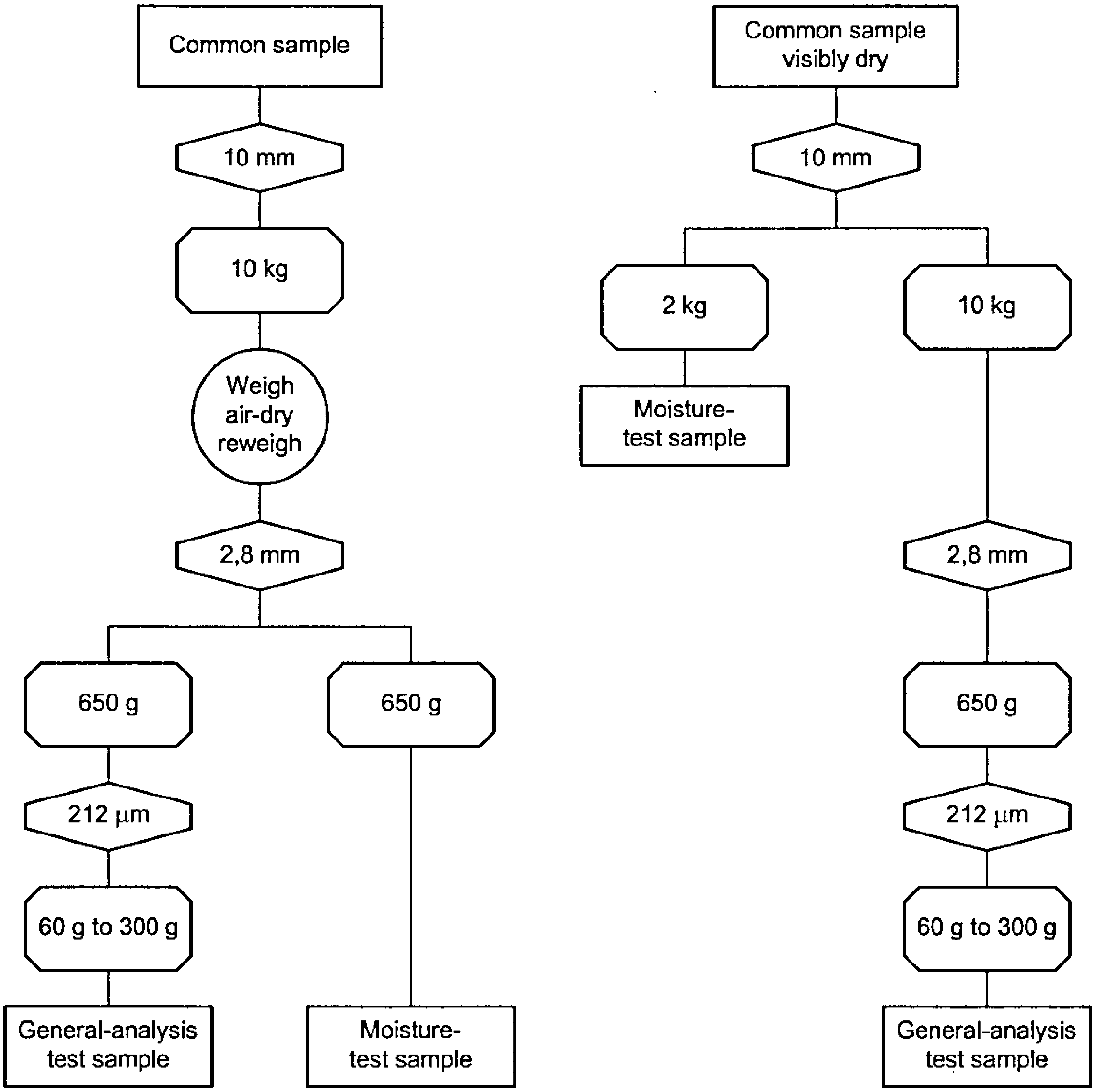


Figure 12 — Examples of preparation of test samples from a common sample for moisture and general analysis

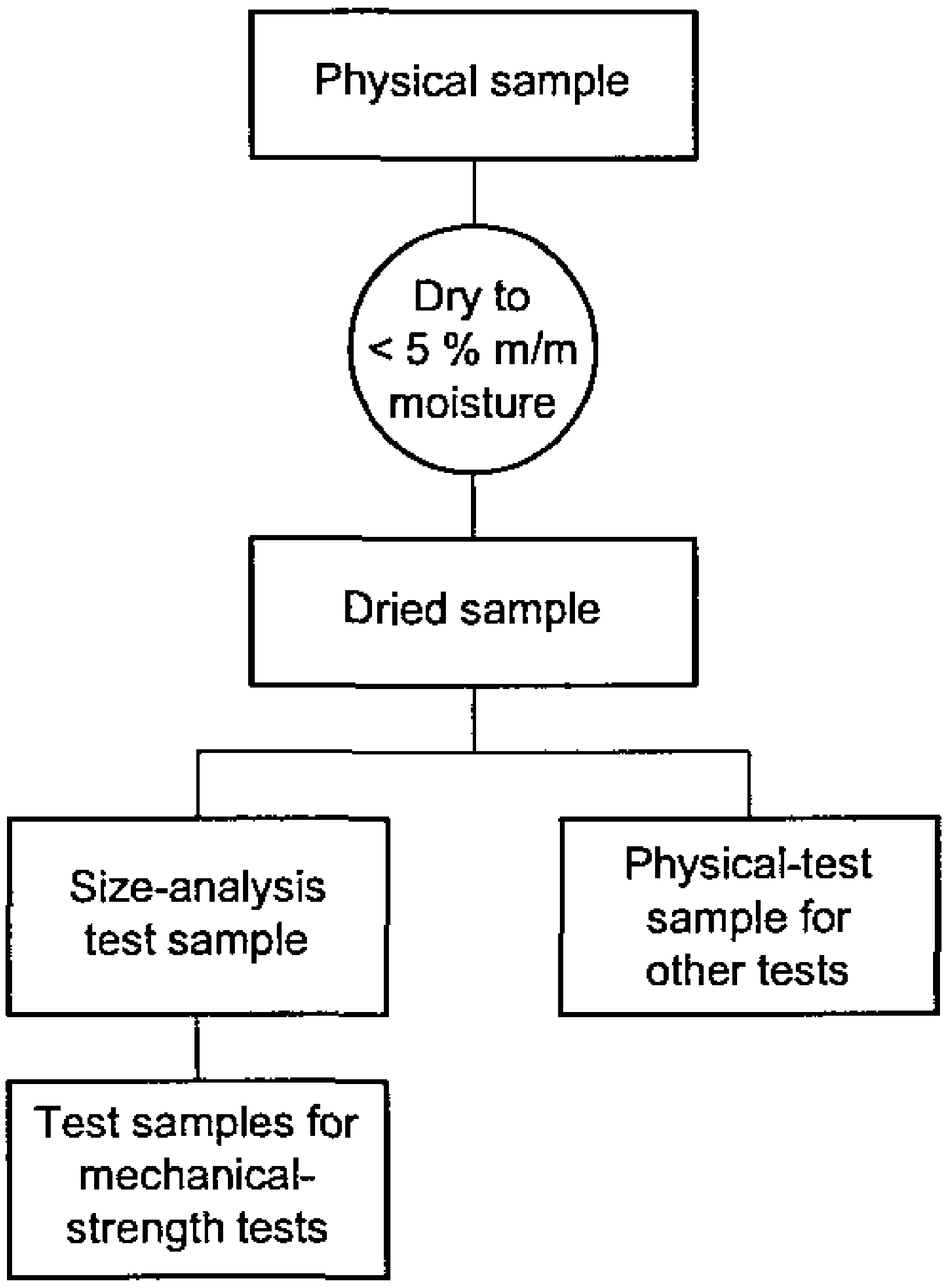


Figure 13 — Example of preparation of samples for size analysis and/or other tests

As a result of the extraction, the common sample has been divided into two parts, one for preparation of the moisture-test sample and one for the preparation of the general-analysis test sample. Each part shall fulfil the requirements for minimum mass specified in Table 3 (see 4.4.10) and further treatment of the parts shall be in accordance with 8.7.2 and 8.7.3, respectively.

8.7.5 Preparation of size-analysis sample

If the mass of the size-analysis sample is more than twice that given in Table 3 (see 4.4.10) for the appropriate nominal top size, it may be divided to a mass not less than given in the table, provided that the requirements for division (see 8.3) are satisfied. During division, precautions shall be taken to avoid breakage. An example of a scheme for preparation of test samples for size analysis is given in Figure 13

If the nominal top size of the coal is greater than one-third of the cutting aperture of the sample divider, oversize material may be removed by sieving out and the whole of this oversize portion subjected to size analysis. The undersize coal should be divided to a mass not less than that given in Table 3 (see 4.4.10) for the appropriate nominal top size. The divided sample should then be subjected to size analysis and the results combined with those from the oversized coal, weighted according to their relative proportions in the original sample.

8.7.6 Preparation of samples for other tests

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

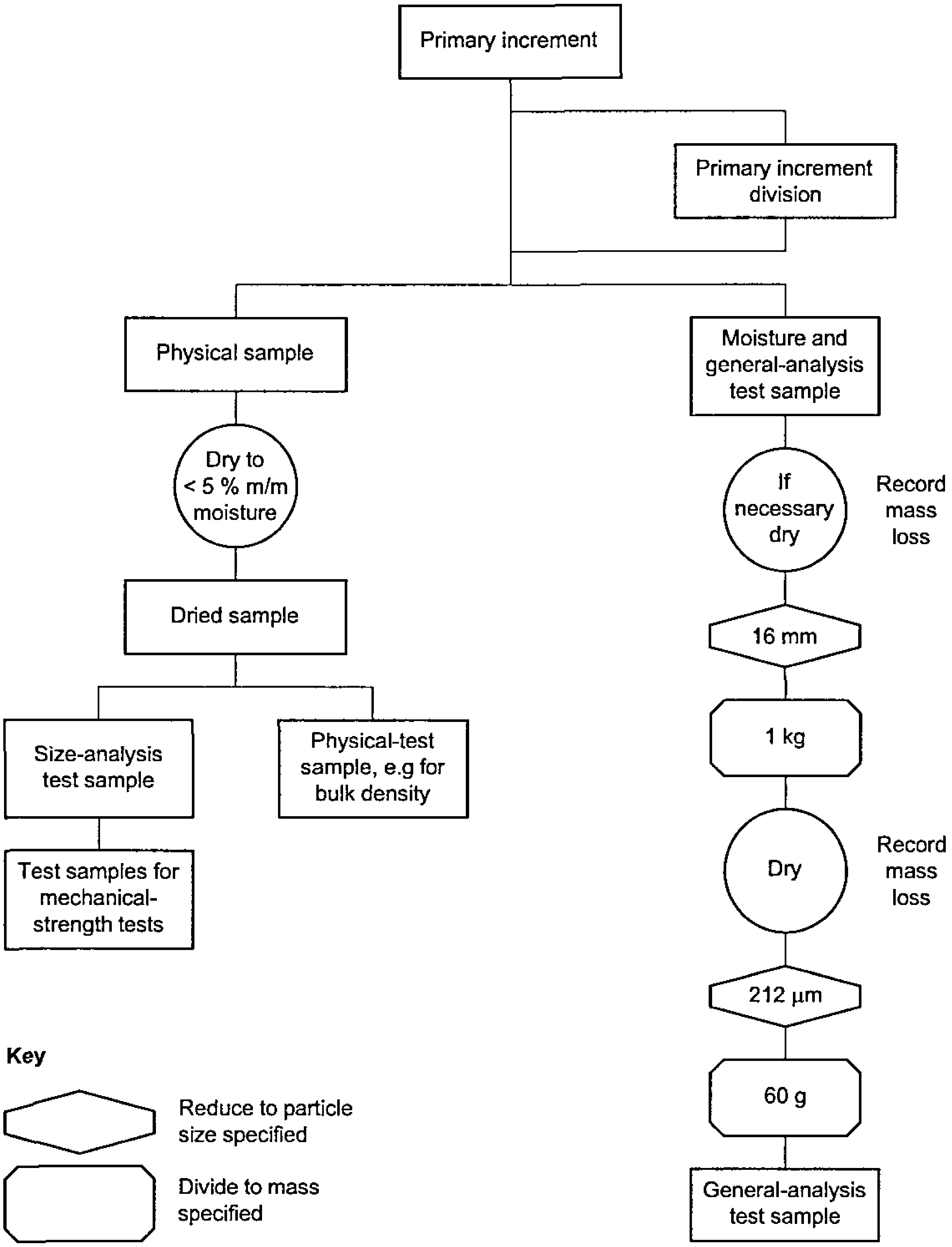


Figure 14 — Constitution and preparation of samples

8.7.7 Reserve sample

If a reserve sample is taken for examination in the event of a dispute or in case the results of the first tests are lost or invalid, it shall be collected at the same time and prepared in the same way as the ordinary sample.

It is recommended that the reserve sample be divided as little as possible and no further than to the maximum mass that it is still practicable to store. It should not be reduced further than to the nominal top size consistent with the mass given in Table 3 (see 4.4.10 and 4.5).

8.8 Coke — Preparation of test samples

8.8.1 Types of test samples

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

f) sample for determination of total moisture;

g) sample for general analysis;

h) physical-test sample.

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

A schematic diagram showing the alternative ways of constituting and preparing the various types of test samples is shown in Figure 14.

8.8.2 Preparation of samples for determination of total moisture

8.8.2.1 General

The test sample for the determination of moisture shall be prepared to meet the requirements of ISO 579 or ISO 687, as appropriate. If air-drying is performed at any stage of preparation, the percent loss in mass is recorded and included in the calculation of total moisture.

NOTE 1 A major problem with the preparation of test samples for the determination of moisture content is the risk of bias due to inadvertent loss of moisture. The amount of this loss is dependent on such factors as the effectiveness of the sealing of the sample containers, the level of moisture content of the sample, the ambient conditions, the type of coke, and the reduction and division procedures used.

Precautions shall be taken to minimize changes in moisture during preparation and due to the use of unsuitable containers. All samples for moisture determination shall be kept in sealed containers in a cool place, under cover, before and during preparation as well as during any interval between steps of sample preparation.

If excessive standing time causes bias, the number of sub-lots should be increased to overcome these problems (see 4.5).

Preliminary air-drying may be necessary in order to minimize moisture loss in any subsequent division stages, in accordance with the procedure described in 8.6. When carrying out division prior to air-drying of a sample or increment, take care to minimize change in moisture. 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 2 For cokes that are too moist to flow through a sample divider and for which it is also impossible to air-dry the entire sample, it can be necessary to divide the sample by collecting increments from a flattened heap (8.3.5.2) or by strip-mixing and splitting (8.3.5.3.). This divided sample is then air-dried.

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

If the coke is so wet that water separates from the coke 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.

If the particle size of the sample is so large that the mass given in Table 3 (see 4.4.10) 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.

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

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

8.8.2.2 Procedure

The procedure for the preparation of a 1 kg sample for the determination of total moisture is shown schematically in Figure 14.

In the case of small coke, having a top size of 20 mm or less, only the process of sample division described in 8.8.2.1 is required.

Precautions shall be taken to prevent loss of moisture during these operations, which shall be carried out in an enclosed space, roofed over and made free from draughts.

The total time taken to crush and divide the coke shall not exceed 15 min; therefore, crush the coke quickly and mechanically. In addition, the mass of sample crushed at one time shall not exceed 70 kg.

Samples that are visibly wet and those for which the moisture is expected to exceed 15 % are partially dried before reduction and division (see 8.6).

8.8.2.3 Reduction and division of moisture-test sample

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

Precautions should be taken against loss of moisture by undue ventilation or loss of sample as dust.

Starting with the masses given in Table 3 (see 4.4.10), the crushed sample shall be divided into a mass of 1 kg using mechanical dividers of the cone or container type [see Figures 6 b) and 6 c)].

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

For manual division, the strip-mixing and splitting procedure (8.3.5.3), the flattened-heap procedure (8.3.5.2) or a closed riffle may be used.

The 1 kg sample taken as described in 8.8.2 shall be removed to the laboratory in a closed container. For the determination of moisture one of the methods specified in ISO 579 or ISO 687 shall be used, as appropriate.

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

8.8.3 Preparation of samples for general analysis

8.8.3.1 General

A procedure for the preparation of a 60 g test sample for general analysis is shown in schematic form as part of Figure 14. The material used may be the 1 kg sample of coke which has been dried in the determination of total moisture (see 8.8.2). Alternatively, a separate 1 kg sample may be prepared as described in 8.8.1 and dried.

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

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

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

Mechanical or manual division may be used, the former being preferred. For mechanical division a suitable divider to give 60 g of – 212 µm coke is required. For manual division, a riffle may be used or the sample shall be spread out and 60 g extracted by hand in not less than 20 increments taken from various parts of the heap using a small-scale version of the technique described in 8.3.5.2.

The sample shall be well mixed and it is preferred that mixing be carried out using a mechanical device, such as a mixing wheel. After mixing, the sample shall be placed in an air-tight container for storage. A glass jar with a screw on lid is suitable

8.8.3.2 Preparation errors

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

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

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

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

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

The segregation error arises from the heterogeneous nature of coke and because of the difficulty in mixing uniformly any coke in which there is a wide range of sizes. Therefore, the coke shall be mixed thoroughly in order to obtain a representative sample.

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

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

8.8.4 Physical-test sample

The physical-test sample may be used for the determination of size, physical characteristics or both. It is essential that collection and preparation procedures be such that breakage is minimized.

The sample may be divided to a mass not less than those given in Table 3 (see 4.4.10).

8.8.5 Samples for special properties

Samples required for special tests shall be extracted from an appropriate stage in the preparation process and prepared to the size and mass specified in the relevant International Standard test method.

Some examples of tests that can be required in this context are Shatter Index (ISO 616), mechanical strength (ISO 556), reactivity tests (ISO 18894), density (ISO 567 and ISO 1013) and particle size distribution (ISO 728 and ISO 2325).

Requirements concerning particle size, sample size and other factors in these standards should be taken into account in the preparation of the test sample if the standard is cited in the sampling protocol.

8.8.6 Reserve sample

If a reserve sample is taken for examination in the event of a dispute or in case the results of the first tests are lost or invalid, it shall be collected at the same time and prepared in the same way as the ordinary sample.

9 Packing and marking of samples and sampling report

Samples shall be packed in non-absorbent, air-tight containers and tightly sealed. All samples shall be labelled in such a way as to identify them uniquely.

A complete and final report or certificate, duly signed, stating all relevant information on the sampling, sample preparation and sample distribution shall be issued.

Any deviation from prescribed methods and the reason for the deviation shall be stated in the report, as shall any anomalies observed during sampling.

Relevant information from the sampling report shall be attached to the sample(s) or otherwise made available to the person(s) in charge of subsequent sample preparation, analysis and testing.

It is recommended that the following information be shown on the label or accompanying documents in the sampling report or certificate:

a) title, e.g. "Report of Sampling", "Sample Preparation" and "Distribution of Samples" (report/certificate only);

b) name and address of the client (report/certificate only);

c) type, grade and nominal top size of the fuel and name of the lot (identity of the ship or train, etc.);

d) method of sampling, with reference to the appropriate parts of this International Standard and its publication date (e.g. "sampled in accordance with ISO 18283:2006");

e) mass of the lot and the number of sub-lots;

f) mass of fuel represented by the sample(s), and the lot or sub-lot number(s);

g) sample, lot and sub-lot number(s);

h) place, date and time of sampling;

i) place, date and time of sample preparation;

j) name(s) of the sampler(s);

k) type of sample and its intended uses, e.g. common sample, moisture sample, etc.;

l) final mass of sample and its nominal top size, as collected before any sample preparation;

m) description and number of test sample(s), e.g. test sample for general analysis, test sample for moisture, etc.;

n) weather or other condition that can affect the results;

o) any other relevant information, e.g. percentage loss by air-drying of the moisture sample, sealing of the samples, etc.;

p) distribution of test samples and minimum period of storage of arbitration and reserve samples (report/certificate only).

Annex A
(informative)

Example of calculation of precision, mass of increments, number of sub-lots and number of increments per sub-lot

A.1 Introduction

A sampling scheme has to be established prior to sampling in order to obtain the required minimum sample mass, depending on nominal top size of the fuel and the desired degree of precision of the results.

The following parameters are used in the calculations:

P_L	the overall precision of sampling of the lot (characterizes the precision for the lot, either as a specification which has to be reached or for the result which has been obtained);
P_{SL}	the sampling precision for the sub-lot at 95 % confidence level expressed as % absolute;
V_I	the primary increment variance (variance of an analytical parameter in the fuel by increment size, dependent on increment mass, top size of fuel, etc.; it serves as a measure of the variability of a fuel with respect to the sampling conditions);
V_{PT}	the preparation and testing variance (variance of an analytical parameter due to sample preparation and testing);
N	the number of sub-lots (to achieve a required precision for a lot, it may be necessary to split the whole lot into several sub-lots and to sample and analyse them separately);
n	the number of increments per sub-lot.

The main equation for calculating sampling precision, P_L, is Equation (4) (see 4.4.3.), while Equation (12) (see 4.4.8.2) is used for calculating the required number of primary increments, n.

$$P_L = 2\sqrt{\frac{V_I}{Nn} + \frac{V_{PT}}{N}} \tag{4}$$

$$n = \frac{4V_I}{N \cdot P_L^2 - 4V_{PT}} \tag{12}$$

Depending on the chosen precision, the number of sub-lots and the number of primary increments can be calculated if a value for the primary increment variance is given (either by determination, by experience or initially assumed).

If the calculation for the number of increments yields a negative or a non-defined value, preferably the number of sub-lots should be increased. Otherwise, preparation and testing shall be carried out with a higher precision to reduce the preparation and testing variance, V_{PT}, which generally is more difficult to achieve. Take n as 10 if the final calculated value for the number of primary increments is less than 10.

A.2 Example

In Table A.1, the results for calculations of variables for different sampling schemes are shown. Before commencing these calculations, it is necessary to establish the following parameters:

- nominal top size of the fuel to be sampled;
- lot size;
- overall precision, P_L, required for the entire lot;
- preparation and testing variance, V_{PT};
- primary increment variance, V_I.

Referring to each row in Table A.1:

a) row "a" specifies the number of sub-lots;

b) row "b" calculates the sub-lot size given the lot size and the specified number of sub-lots;

c) row "c" specifies the reference increment mass based on Table 2 (see 4.4.9);

d) row "d" uses Equation 12 to calculate the number of primary increments that need to be taken from each sub-lot for primary increment variances of 5 and 10 to achieve the required overall precision;

e) row "e" specifies the minimum mass of sample for general analysis;

f) rows "f" calculates the mass of sub-lot samples by multiplying the reference increment mass by the number of primary increments.

In Table A.1, calculations for $V_I = 5$ and $V_I = 10$ are worked out for some different numbers of sub-lots. The figures between brackets indicate that there is a non-conformance with an aspect of this International Standard, in this case, the mass of sub-lot samples.

Table A.1 — Calculations of variables for different sampling schemes for washed and unwashed coal

Row	Reference	Description	Results for the specified conditions [a]								
			Washed coal ($V_I = 5$)				Unwashed coal ($V_I = 10$)				
a	—	Number of sub-lots	2	4	5	10	2	4	5	6	10
b	—	Sub-lot size	10 000	5 000	4 000	2 000	10 000	5 000	4 000	3 000	2 000
c	Table 2	Reference increment mass, kilograms	3	3	3	3	3	3	3	3	3
d	Equation (12)	Number of primary increments	167	45	33	14	333	91	67	53	29
e	Table 3	Minimum mass of samples for general analysis	170	170	170	170	170	170	170	170	170
f	Row c times row d	Mass of sub-lot sample, in kilograms, for ($V_I = 10$)	501	(135)	(99)	(42)	999	273	201	(159)	(87)

[a] Nominal top size, 50 mm; overall precision for the lot, $P_L = 0,4$ %; lot size, 20 000 *t*; preparation and testing variance, $V_{PT} = 0,05$.

A.3 Interpretation

Table A.1 clearly shows that considerable effort is necessary to sample a highly variable fuel with a high precision, particularly when the number of sub-lots is small. Hence, there can be considerable advantages in increasing the number of sub-lots.

However, it is important to ensure that the mass of sub-lot samples meets the minimum mass requirements of Table 3. In case of washed coal ($V_I = 5$), this is not achieved when the lot is divided up into 5 or more sub-lots.

In case of unwashed coal ($V_I = 10$), the minimum sample mass is not achieved when the lot is divided up into 6 or more lots.

In these cases, increase the mass of primary increments above the reference mass specified in Table 2 to achieve the minimum sample mass.

In both cases, it is possible to divide the lot into only a few sub-lots, but in such cases the sample mass can become very high. See, for instance, the case of unwashed coal ($V_I = 10$), where the lot is divided up into 2 sub-lots. In that case, an amount of around 1 000 kg sample mass is obtained.

Annex B
(informative)

Methods of sampling large fuels and fuels from stationary lots

B.1 General

Sampling of stationary lots does not provide representative test samples. When analysed, these samples provide only indicative test results; therefore, the use of this method does not comply with this International Standard.

B.2 Large fuels

When the fuel includes particles larger than 150 mm after sampling in accordance with this International Standard, the mass of the material over 150 mm in the sample can be considered as a separate sub-sample. The proportion by mass of the material over 150 mm in the fuel should be obtained from a particle-size analysis done at 150 mm. Other sizes can be chosen based on the fuel or on requirements.

Each sub-sample shall be crushed, if necessary, using a hammer and divided separately and preferably tested separately. Alternatively, the sub-samples may be mixed after division and preparation to uniform top size in the proportions by masses of over and under 150 mm to one sample.

B.3 Sampling from stationary lots

B.3.1 General

It can be necessary to sample a stationary lot. This is a static situation where the fuel is not being handled.

The purpose of this sampling may be to get an indication of certain parameters of the stationary lot. Only the outer surface of the stationary lot can be sampled.

B.3.2 Extracting the increment

The extraction of increments should be carried out by suitably trained and experienced samplers. The appropriate equipment should be used (see Clause 6).

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

When extracting the increments, it should be assured that the surface is sufficiently compacted to bear safely the weight of personnel and equipment.

A manual probe or auger shall be used. The aperture of the probe or auger shall be at least three times the nominal top size of the fuel, with a minimum dimension of 30 mm (see Clause 6).

Increments shall be spaced as evenly as possible over the surface of the stockpile. The fuel in the top layer of a stockpile is almost always different in quality from the rest, due to exposure, segregation and other causes. It is important to note that segregation during handling results in the accumulation of lumps at the bottom of the pile. It is necessary to take this into account when making an estimate of the particle-size distribution.

Divide the surface of the stockpile into a number of squares using an imaginary grid system. The number of squares is dependent on the size of the surface. Increments should be extracted from the squares from any position within the appropriate square (see 5.9).

When extracting increments, the manual probe/auger or scoop shall be inserted at right angles to the surface of the fuel after the top surface of the fuel has been removed. Large pieces of fuel should not be deliberately pushed aside when an increment is extracted and no portion of the increment should be lost during extraction of the scoop from the surface. Owing to the difficulty of insertion, a probe/auger shall only be used for fuels with a particle size of up to about 25 mm. A full column of fuel shall be extracted so that a representative increment is taken.

Drainage and water spraying shall be taken into account when sampling from a freshly exposed surface of a stockpile.

B.3.3 Sample storage

The increment should be placed as quickly as possible in metal or impermeable containers provided with well-fitting lids, which should be replaced after each increment has been inserted. Sample containers shall be protected from direct sunlight and precipitation during collecting of samples. The sample should be kept in a cool place during storage, preferably at a temperature that is not above that of the sample when it was taken.

For samples where the moisture content is not important, e.g. for determination of the ash, the increment may be kept in sample bags, but they shall be protected from contamination or loss.

A label giving a clear and sufficient description of the sample should be attached to the sample container.

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