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Hard coal — Sampling

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FOREWORD

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Hard coal — Sampling

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods of sampling hard coal, for both routine and special purposes, to provide samples for general analysis and for the determination of total moisture. It also outlines the principles to be taken into consideration when taking the sample and preparing it for analysis.

The principles of this International Standard may also be used for taking samples for the determination of physical characteristics, such as particle size and density, and the determination of rheological properties. For physical characteristics it may be necessary to collect a greater mass of gross sample than the minimum specified, either by increasing the mass of each increment or by taking more increments, and for rheological properties the top size of the prepared laboratory sample may have to be different from that of either the general analysis sample or the total moisture sample (see 2.9).

NOTES

1 The term "hard coal" refers to all coals so defined by the ECE classification (see ISO/R 1213). It may include certain coals known in the French classification as "hard lignites".

2 Attention is drawn to ISO/R 1213, *Vocabulary of terms relating to solid mineral fuels — Part I : Terms relating to coal preparation,* and *Part II : Terms relating to coal sampling and analysis,* the terms and definitions in which apply to this International Standard.

2 INTRODUCTION

2.1 Guide to the reader : Layout

This International Standard is a comprehensive document dealing with all aspects of the sampling of coal and is accordingly lengthy. The following notes are given as a brief guide to the layout.

Clauses 2 and 3 refer to general problems which arise in the sampling of coal and should be studied thoroughly. One of the clauses 4 to 7 — whichever is appropriate depending on the location of the coal — should be followed to obtain detailed instructions for sampling from the particular location. Annex A describes typical items of sampling equipment which may be required.

After reading clause 2, it will be apparent that certain decisions have to be made before sampling can start and an outline of the sort of instructions which may have to be devised for the sampling operator is given in annex B.

Clause 3 gives general principles and describes the procedure of replicate sampling which is used to determine whether the desired sampling precision has been attained. This procedure, once understood, is very simple to operate; the numerical checks on the results obtained are described in annex C and the theory underlying it is explained in annex G.

The whole of the above refers to taking the sample. When the gross sample has been obtained, laboratory samples must be prepared from it and instructions for these procedures are given in clauses 8 and 9. A procedure for checking errors of sample preparation is given in annex D, and the theory underlying this procedure is explained in annex H. If there is any doubt as to whether the sampling method is suitable, annex E should be studied since this gives instructions for testing sampling procedures for bias.

2.2 General procedure for collecting a sample

The object of collecting a sample of coal is to obtain a portion which serves for the determination of the qualities of the coal concerned. Normally coal consists of particles of varied shapes and sizes, which may have different physical and chemical properties. In order that the sample shall represent the coal from which it is taken, it is collected by taking a definite number of portions, known as increments, distributed throughout the whole of the coal being sampled. The term "increment" refers to the quantity of the coal obtained by a single operation of the sampling instrument.

An essential condition of sampling is that the whole bulk of the coal to be sampled should be exposed, so that all parts are equally accessible to the sampling implement and have the same chance of being included in the sample.

Three methods for spacing the increments have been proposed :

a) systematic sampling : increments are spaced evenly in time or in position over the unit;

b) random sampling : the increments are spaced at random in time or in position over the unit;

c) stratified random sampling : the unit is divided by time or quantity into a number of equal strata and one or more increments are taken at random from each stratum.

Systematic sampling would lead to serious bias if there were a periodic variation in quality coinciding with the frequency of taking increments; experience shows that a

strictly periodic variation is unlikely to occur in practice without the knowledge of the technician concerned. The chance of bias arising from such a coincidence is therefore very small.

Stratified random sampling and random sampling are difficult to operate as routine procedures for automatic or manual use; they would give better results only if the periodic variation mentioned above occurred without the knowledge of the technician.

In a few cases random sampling techniques have been accepted but this International Standard is based mainly on the principle of systematic sampling. Care must, therefore, be taken to avoid any possible coincidence between the taking of the increments and a periodic variation of quality.

Bias, i.e. a tendency to obtain results which are persistently too high or persistently too low, occurs very easily during sampling and is difficult to detect; the greatest care should, therefore, be taken to prevent its occurrence. The two main causes of bias are :

a) the selection of increments from an unrepresentative part of the coal being sampled; for example, from only one side of a belt;

b) the collection of increments in such a way that they are not representative of the coal in their immediate vicinity; for example, by using a scoop which is too small to collect the larger pieces of coal.

To avoid bias, it is essential that the dimensions of the sampling equipment and the mass of increment should be in accordance with the maximum dimensions of the coal (see 3.3). If bias is suspected it may be possible to improve the accuracy by changing the shape and/or location of the sampling implement, or by changing to another sampling system, but in practice it has been found that neither accuracy nor precision (see 2.5) can be improved merely by increasing the mass of individual increments above the minimum specified. A change in the precision of sampling may be effected by altering the number of increments, but this will not alter any bias which is inherent in the sampling system.

The most favourable situation, in which the whole of the coal is exposed for sampling, is when it is being conveyed on a belt or similar device so that it passes the sampling point in a stream. If the belt is stopped and a section of adequate length is taken across the whole width of the belt, all the coal particles in this section can be taken so that there will be no bias due to causes a) and b) above. Sampling from a stopped belt is normally the most satisfactory way of ensuring that the sample is free from bias and it is therefore recommended as the most reliable reference method, which should be used for checking other methods.

In many installations it is not possible to stop the belt without considerable interference with the work of the installation and other methods of sampling must be used. The next most satisfactory methods are those by which cross-sections of a moving stream are collected, but it is necessary to ensure that each increment is a representative cross-section. When sampling stationary coal, the essential condition that each part of the coal is equally accessible to the sampling implement is not fulfilled; for example, when coal is sampled in a wagon there is no possibility of the particles in the bottom corners of the wagon being taken. Consequently a distinction is drawn in this International Standard between coal in a stream (whether moving or stopped) and stationary coal.

Experience has shown that satisfactory samples can be taken from stationary material in wagons, ships and even in stockpiles, provided that special precautions are taken to avoid bias. As stationary material tends to be highly segregated the sampling points must be carefully selected and the number of increments collected must be larger than from moving material.

References in this International Standard to stationary coal imply coal which is in a wagon, ship or stockpile. References to a moving stream imply coal which is being handled by a belt or other conveyor; whether the conveyor is moving or not at the time of sampling is of no significance in this definition.

Whatever method of sampling is adopted, careful consideration is required to find a point where unbiased increments can be collected with safety and without undue physical strain. It is frequently desirable to make permanent arrangements such as the provision of a special platform for the safety and convenience of the sampler. Special arrangements are also desirable for the removal of the samples, where they are taken from an exposed point to an enclosed location for further treatment.

If increments are collected manually a trained sampler should be employed and the instructions given to him 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 (see 2.12). It is for this reason that mechanical sampling is preferable to manual sampling, but it is necessary, in the first place, to check that the sampling machine is unbiased.

2.3 Differences between suppliers and customers

The supplier is always handling the same type or types of coal of which the general characteristics are known; he is, therefore, usually interested in the average characteristics of the coal over a specified period rather than the characteristics of an individual consignment. If a customer's supplies are loaded at random the average analyses over a period may be a better estimate of the quality supplied to him than an analysis taken from an individual consignment.

So far as sampling is concerned a customer does not usually know any more about a coal than its reputed quality and must regard the coal as a single consignment the characteristics of which are not known. If he receives the same type of coal regularly, he may be in a similar position to the supplier — though generally conditions will be somewhat different because the coal may have suffered segregation or mixing by being loaded into wagons, barges or ships.

When coal from the same source is sampled regularly and random errors only are present, the difference between the means of the sample values obtained by supplier and customer should tend towards zero as the number of consignments sampled increases.

2.4 Samples for general analysis and for moisture determination

In some circumstances it is necessary or convenient to collect separate samples[1)] for the determination of total moisture and for general analysis. In other circumstances, it is more convenient to take a common sample for both moisture and general analysis; for example, it may be necessary to take a common sample when an automatic sampler is in use, or a separate moisture sample when the coal is very wet.

This International Standard gives figures for the collection of two separate samples, one for ash and one for total moisture. If it is desired to collect a common sample, the mass of sample specified for ash may need to be increased according to the instructions given later.

2.5 "Accuracy" and "Precision"

No method of sampling, sample preparation, or analysis can be perfect, since the true value can never be known exactly. The **accuracy** of the experimental results obtained from a method of sampling is the closeness with which they agree with the true value. But, as the true value is not known, it is necessary to assess the closeness with which the experimental values agree among themselves. This is known as the **precision**.

This means that it is not possible to determine the **accuracy** of a series of determinations, but only their precision. Provided that there is no bias in the method, the precision will be the same as the accuracy. For convenience in this International Standard, the word **precision** is used hereafter.

2.6 Precision of sampling

This International Standard is based on a reference standard precision for moisture and ash (see 3.1.4).

Experience shows that when a sample is collected which provides this precision for ash, a precision better than this will usually be obtained in the determination of other common characteristics.

In this International Standard it is assumed that when sampling to the reference standard, the variance of sample preparation and analysis will be approximately one-fifth of the total variance and the remaining variance will be due to sampling. Thus, for a coal having an ash of 10 %, a precision of ± 1 % absolute (95 times out of 100) is equivalent to an overall variance of 0,25, arising from a sampling variance of 0,20 and a sample preparation and analysis variance of 0,05.

The characteristics of coal vary considerably, so that a specified sampling procedure will provide different precisions for different coals. For example, the precision obtained by taking a certain number of increments from a uniform product from a single seam will be much better than if the same number of increments were taken from a product of the same average quality, but derived from a number of different seams. In order to ensure that results are not worse than a particular limit of precision, it is therefore necessary to specify the number of increments appropriate to the most variable coals that have to be examined. This means that in the majority of cases the precision obtained will be better than the specified limit. It is strongly recommended that the method of replicate sampling (described in 3.5) should be used to check the sampling precision so that, if necessary, the number of increments can be adjusted to the minimum number needed to give the required precision (see annex F).

2.7 Sample preparation

When the sample or samples have been collected it is usually necessary to prepare from them two laboratory samples, one for the determination of ash and other chemical characteristics, the other for the determination of total moisture.

The object of sample preparation is to treat the samples so that the small sample of coal received in the laboratory for analysis will be representative of the original gross sample. The laboratory general analysis sample should consist of at least 60 g of coal with a top size of not more than 0,2 mm. The mass of the moisture sample depends on the method of moisture determination which is to be used, but will be 300 g or more.

Instructions for the preparation of the moisture sample are given in clause 8 and for the preparation of the general analysis sample in clause 9.

2.8 Treatment of sample

When a separate moisture sample is taken the increments 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. The sample should be kept in a cool place during storage, preferably at a temperature which is not above that of the sample when it was taken.

For a common sample, the same procedure should be followed until the moisture sample has been extracted as described in clause 8.

For an ash sample, the increments may be kept in sacks, but they must be protected from contamination or loss and treated by the methods of clause 9.

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

1) In the remainder of this International Standard a sample which is collected for the preparation of the general analysis sample is referred to briefly for convenience as an **ash sample** and the other sample is referred to as a **moisture sample**. If a single sample is taken for the determination of ash and moisture, it is referred to as a **common sample**.

2.9 Physical and other tests

A number of physical tests are frequently carried out on coal, of which the most common are float and sink analysis and size analysis. The results of all physical tests are affected by the size distribution of the coal and, provided special care is taken to avoid breakage, the procedures of this International Standard are applicable to the collection of samples for physical tests. In particular, the **minimum** mass of increment needed for physical tests will be the same as the minimum mass of increment needed for the determination of ash or moisture content as specified in this International Standard.

For all physical and other tests the total mass of sample required depends on the test involved and will generally be greater than the mass of sample required for ash and moisture. These masses are (or will be) given in the appropriate ISO publications and reference should be made to these to determine the appropriate mass.

For these tests the sample should be collected in accordance with this International Standard, but either the mass of the individual increments or the number of increments should be increased to give the greater sample mass. It is preferable to increase the number of increments rather than the mass of individual increments; but it may be more convenient on some occasions to collect larger increments.

For certain tests, for example, coking or other physical test, it may be necessary to use the coal in its original state, or at sizes other than the 0,2 mm referred to above. In such cases, sub-clause 2.7 is not relevant.

2.10 Report

The sampler should prepare a report stating the number and size of increments, details of the sampling procedure, full details of the coal and the precision adopted. This report should be attached to the sample or otherwise made available to the recipient of the final results.

2.11 Theories of sampling

There are many theories of sampling, some of which have been found to give an adequate explanation of the factors involved in some circumstances, whereas others are satisfactory in other circumstances, but none is satisfactory in all circumstances. For this reason, this International Standard is based primarily on practical experience, including a substantial volume of experimental data collected in several countries. The theoretical basis for the procedures is discussed in the annexes, where the derivation of the empirical formulae is also dealt with.

2.12 Instructions for sampling operators

This International Standard gives methods and principles of sampling which should cover all sampling problems likely to be encountered in international trade. It has been necessary, therefore, to describe a large number of alternative methods, with the result that the document is lengthy and is too complicated to be handed directly to a sampling operator. It is important that the sampling operator should receive instructions which are simple, easily understood and capable of only one interpretation. 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. Instructions should be set out under the headings listed in table 1, which also lists those parts of the document which should be consulted before preparing the instructions for the sampling operator.

Before the sheet of instructions can be prepared, the supervisor himself must have information on the following :

a) for what purpose is the sample required ?

b) what is the estimated maximum size, quality and ash content of the coal ?

c) what analyses are required (for example moisture, ash, physical tests) ?

d) is a separate moisture sample to be taken; or a common sample ?

e) from where is the sample to be taken (from a stream of coal, wagons, ship or stockpile) ?

f) is the coal to be treated as a single consignment or as a regular delivery ?

g) what is the size of the consignment (is it to be sampled as a whole or in 1 000 tonne lots ?) and what information is available on its heterogenity ?

h) is the reference standard of precision adequate, or is a different precision required ?

j) is the precision to be checked by replicate or duplicate sampling ?

Table 1 gives references to the relevant sub-clauses, clauses or annexes of the document required for various methods of sampling.

Examples of suitable instructions are given in annex B.

TABLE 1 — **References to information required**

Information required	Reference : For sampling from			
	streams	wagons	ships	stockpiles
General considerations	4.1	5.1	6.1	7.1
Collecting the sample	4.3	5.3	6.3	7.3
Sampling equipment	annex A	annex A	annex A	annex A
Mass of increment	3.3	3.3	3.3	3.3
Number of increments	4.2	5.2	6.2	7.2
Treatment of sample	2.8	2.8	2.8	2.8
Preparation for analysis	8 and 9	8 and 9	8 and 9	8 and 9
Precision	3.1	3.1	3.1	3.1
Check on precision	annex C	annex C	annex C	annex C

3 FUNDAMENTALS OF SAMPLING

3.1 Precision

3.1.1 *General*

In this International Standard, all references to precision are related to 95 % probability.

This means that determined values (for example, for ash or moisture) on samples taken from the same coal (i.e. coal of the same quality from a single source) can be expected to lie within the specified limits of precision 95 times out of 100 : in the absence of bias these limits would be spread uniformly about the time value. Conversely, applying these limits to a single value, there is a 95 % probability that the spread includes the time value.

3.1.2 *Precision and number of increments*

The standard of precision selected is to some extent arbitrary since taking more increments will give a better precision. Subject to the limitations discussed below (see 3.2.5), any desired precision may be attained by suitable adjustment of the number of increments.

Nevertheless, it is convenient to select a reference standard of precision to which can be related the number of increments necessary for different types of sampling or different types of coal. The references in 3.2.4 and in clauses 4 to 7 to an "initial number of increments" imply the number of increments required for this reference standard of precision, which is set out in table 2. Instructions are given in 3.2.4 for adjusting the initial number of increments if a different precision is required.

In general, unless there are special reasons to the contrary, the reference standard of precision should be adopted.

3.1.3 *Replicate sampling*

By using the procedure of replicate sampling, it is possible to test the precision obtained by a particular sampling scheme.

In particular, the application of replicate sampling allows adjustments to be made in the number of increments taken. As explained in 2.6, the recommended number of increments is determined by the requirement to attain the reference standard of precision when sampling the most variable coals. With other coals, therefore, this number of increments should give a better precision than is usually required. If repeated consignments of the same coal are being sampled, the application of replicate sampling may enable the initial number of increments to be reduced progressively for successive consignments until the desired precision is attained with the minimum number of increments.

If only a single consignment of a coal is being sampled, it is not possible to reduce the number of increments in this way, but by applying replicate sampling the actual precision obtained can be determined.

3.1.4 *Reference standard of precision*

The reference standard of precision for coals of all quantities and all forms of sampling is ± one-tenth of the true ash for values up to 20 %[1] ash and ± 2 % absolute for higher values. The same standard is used for moisture content. This standard is set out in table 2 and represents the deviation from the true value (ash or moisture) corresponding to the sum total of errors arising from sampling, sample preparation and analysis.

TABLE 2 — **Reference standards of precision for sampling**

Characteristic	Type of coal	Standard of precision
Ash	Less than 20 % ash	± one-tenth* of the true ash
	More than 20 % ash	± 2 % absolute**
Moisture	Less than 20 % moisture	± one-tenth* of the true moisture
	More than 20 % moisture	± 2 % absolute **

* For example, a coal of 15 % ash or moisture should give a result between 13,5 and 16,5 %.

** For example, a coal of 25 % ash or moisture should give a result between 23,0 and 27,0 %.

3.1.5 *Other standards of precision*

If a standard of precision other than those in 3.1.4 is required, the sampling procedure set out in this International Standard should be followed, but the number of increments should be adjusted as described in 3.2.4 and the standard of precision stated. The mass of increment must be neither increased nor reduced. Increase will not improve the precision and reduction may introduce bias.

3.2 Number of increments

3.2.1 *Principle*

The number of increments to be taken from a consignment from a single source in order to attain a certain precision is a function of the variability of the coal in the consignment, irrespective of its mass. This variability depends on the amount of segregation present, the size range and whether the coal is cleaned or uncleaned. The numbers of increments specified in tables 3 and 4 take account of these differences as well as of differences in the technique of sampling. Moreover, the variability of the coal in large consignments is usually greater than that in small consignments and for this reason the recommended number of increments for reference standards of precision is applied only to consignments of up to 1 000 tonnes.

1) In this International Standard, all references to ash are on the "dry" basis.

3.2.2 *For reference standard of precision*

The number of increments to be taken to attain the reference standard of precision when sampling from moving streams, wagons, ships and stockpiles for ash and moisture is given in clauses 4 to 7 respectively. For convenience the numbers are also given in tables 3 and 4.

TABLE 3 — **Initial number of increments for sampling for ash**

Condition of coal	Number of increments : For sampling from			
	conveyors and falling streams	wagons and barges	sea-going ships	stockpiles
Cleaned	16	24	32	32
Uncleaned	32	48	64	64

TABLE 4 — **Initial number of increments for sampling for moisture**

Condition of coal	Number of increments for all methods of sampling
Unwashed or dry coal; washed graded coal	16
Washed smalls	32

The number of increments given above is the initial number of increments for the standard precision, but it may be adjusted for the mass of the consignment or for a different standard of precision (see 3.2.3 and 3.2.4).

3.2.3 *Larger consignments*

For consignments over 1 000 tonnes, there are two alternative procedures :

a) preferably the consignment should be divided into a number of portions, each of 1 000 tonnes or less, from each of which a separate sample with the specified number of increments is taken;

b) alternatively, one sample only may be taken, but the initial number of increments for the particular case should be multiplied by the following empirical factor :

$$\sqrt{\frac{\text{consignment mass (in tonnes)}}{1\,000}}$$

3.2.4 *Adjustment of increments*

If replicate (or duplicate) sampling is carried out, the initial number of increments may be reduced in accordance with the test so that the desired standard is attained with the minimum number of increments (see 3.5).

3.2.5 *Warning*

In 3.1.2 it is stated that the standard of precision is arbitrary and that any standard, either better or worse than the reference standard, may be obtained by suitable adjustment of the number of increments as indicated in 3.2.4. The adjustments are, however, based on certain assumptions about the behaviour of the coal (see annex F). Deviations from this typical behaviour will not introduce significant errors provided that the precision aimed at is of the same order as the reference standard, but it is generally inadvisable to attempt to attain a precision of numerically less than 0,5 % absolute, particularly with stationary coal. If a better standard is required, it is advisable to attain this by averaging the results of several samples, so that the average results for a week or a month will have the desired "high" precision.

Moreover, to be on the safe side, the initial number of increments should never be reduced below 12, whatever standard of precision is required.

3.3 Minimum mass of increment

3.3.1 *Principle*

The minimum mass of increment is defined in such a way that bias should not be incurred. The mass of increment must be such that it is large enough to ensure that the large particles of coal are not excluded, and that the particles are present in the same proportion as in the unit of coal being sampled.

The minimum mass of increment is therefore dependent mainly on the size of the coal being sampled. In general, it is inadvisable to collect increments larger than specified, unless it is unavoidable, as for example when taking sections from a moving or falling stream; the increased mass of sample makes the problem of reducing it to the laboratory sample more difficult. The numbers of increments **must not** be reduced merely because larger increments have been taken.

3.3.2 *For coals up to 150 mm top size*

1) The minimum mass of increment, P kg, should be determined from the empirical formula : P (kg) = 0,06 D (mm), when D is the nominal upper size[1)], except that P should never be less than 0,5 kg.

2) In addition, the following conditions should be satisfied :

a) When sampling from a stopped belt :

The minimum width of the cross-section taken should be 2,5 times the upper size of the coal.

b) When sampling from a moving stream :

the minimum opening of the sampling implement should be 2,5 times the upper size of the coal.

1) The square mesh sieve size such that not more than 5 % of the coal is oversize.

c) When sampling from a wagon, ship or stockpile :

the minimum width of the sampling scoop, or the minimum diameter of the probe used, should be 2,5 times the upper size of the coal.

d) The relevant dimension in a), b) and c) should **never** be less than 30 mm.

3) Manual sampling of coal of 80 mm or more is recommended only when the coal is stationary.

3.3.3 *For coals over 150 mm top size*

1) The requirements of 3.3.2 (1 to 3) should be satisfied.

2) The minimum mass of increments should be 10 kg.

3) In addition the following procedure should be adopted.[1)]

The proportion by mass of the material over 150 mm in the coal should be estimated, or preferably obtained from a size analysis. One method of size analysis is described in 5.4.7. If a suitable screen is not available the sample should be provided with a test ring of 150 mm diameter (see A.4.4 of annex A) to enable him to divide roughly one or more increments into "large" ($>$ 150 mm) and "small" ($<$ 150 mm), which are then weighed.

The initial number of increments required should be read from table 3.

The initial number of increments should be multiplied by the above proportion to give the number of increments for "large" coal (i.e. over 150 mm).

The number of increments for "small" coal (i.e. under 150 mm) is obtained by subtraction.

The increments from the "small" coal (i.e. the coal under 150 mm in size), each of 10 kg, should be taken according to the sampling system in use.

The contribution from the "large" coal is obtained in the following manner. An adequate number of pieces over 150 mm in size should be taken to given much more than the relevant number of 10 kg increments. These should be reduced to a size below 80 mm and mixed, then divided by quartering to a mass equal to the required number of increments times 10 kg.

3.3.4 *Example*

An uncleaned coal has 21 % ash and is to be sampled from a moving stream. According to table 3, 32 increments should be taken. It is estimated that 10 % by mass of the coal consists of lumps above 150 mm. Therefore, 3,2 (say 3) increments are required from pieces above 150 mm and the remainder from coal less than 150 mm : each of the increments should be of 10 kg in accordance with 3.3.3.

Thus 29 increments of 10 kg are collected from the coal less than 150 mm, rejecting the pieces over 150 mm.

At the same time about 30 or more pieces over 150 mm are collected. The mass of 30 such pieces will be about 150 kg. These are crushed by blows at right angles to the bedding planes until all coal is less than 80 mm in size. The coal is thoroughly mixed and then quartered to give a portion of about 30 kg (i.e. 3 increments).

3.3.5 *Sub-division of overweight increments*

When increments consist of the full cross-section of a coal stream, they may be much heavier than the minimum mass required, particularly when an automatic sampler is used, and it is then permissible to add only a given proportion of each increment to the gross sample. Sub-division should be carried out by a suitable sample divider and the increment should preferably be crushed before the division is carried out. It is essential that **the same proportion** of each increment should be taken and the division should be such that the amount added to the sample is, on average, not less than the minimum mass of increment appropriate to the original size of coal.

The apparatus for division may be coupled automatically to mechanical sampling equipment, but the whole of the processes after collection, including storage, must be enclosed and draught-proof to prevent loss of moisture.

3.4 Organization of sampling schemes

When the precision required for a given quantity of coal has been decided, the number of increments to be collected should be determined as described in 3.2. The mass of each increment should be determined as described in 3.3.

3.4.1 *Single consignment*

If coal is to be sampled from an isolated consignment, the required number of increments, each of the appropriate mass, should be taken from the consignment as described in clause 4, 5, 6 or 7, whichever is relevant. The result should be of the required precision, but if it is desired to confirm this the procedure of replicate sampling described in 3.5 should be applied.

3.4.2 *Regular consignments*

If the coal to be sampled is part of a regular series of deliveries from the same source, the required precision will usually be related to a certain period, for example the weekly mean may be required to a precision of ± 1 in terms of ash percentage. The coal handled during the period is considered to be made up of a number of units of coal, for example a shift's production, a day's production, a wagon load. The units can be fixed at will. When sampling regular consignments from a stream, there are two possible methods of arranging the collection of the increments during the period; they can be collected either continuously

1) This procedure is unsuitable for mechanized sampling.

or intermittently. However, when sampling from wagons, ships or stockpiles a coal which is received regularly, continuous sampling should normally be used.

3.4.3 *Continuous sampling*

In "continuous" sampling every unit is sampled and the same number of increments should be collected from each unit. Thus the number of increments required to give the specified precision should be divided by the total number of units in the period to give the number of increments for each unit. This number of increments, each of the appropriate mass, should be taken from each unit as described in clause 4, 5, 6, or 7, whichever is relevant. The increments from each unit should be pooled and a laboratory sample prepared therefrom, so that one result is obtained for each unit. There are as many sample results for each period as there are units. The average should be of the required precision, but if it is desired to check that the required precision has been attained with the least possible number of increments, it is possible to do so by using the procedure of duplicate sampling described in 3.5.3.

3.4.4 *Intermittent sampling*

It is often convenient to collect increments from some of the units of coal, but not from others. Thus it may be desired to collect samples on, say, 2 days but not on other days in a week. This is called "intermittent" sampling. The same number of increments is taken from every unit that is sampled. The number of units to be sampled should be decided and the total number of increments required should be divided by this number of units to give the number of increments to be taken from each unit sampled. The units to be sampled may be chosen at random; for example, if the sample is to be taken on only 2 days a week, the days for sampling should be varied each week.

The necessary number of increments each of the specified mass should be taken from each selected unit as described in clauses 4 to 7. The increments from each unit are put together and a laboratory sample prepared therefrom so that there is one analysis for each unit sampled. There are therefore as many sample results per period as there are units sampled, but the number of units **available** is greater because there are some which are not sampled. In this case it is not possible to say that the average of these results will have the required precision until information about the variation between units is available. This can be obtained by following the procedure described in C.3.4 of annex C, preferably in conjunction with duplicate sampling. If the variation between units is too large, it may be necessary to introduce "continuous" sampling to achieve the desired precision.

"Intermittent" sampling cannot be carried out when sampling from ships or stockpiles and in such cases it is improbable that regular sampling can be carried out in any form since it is usually necessary to regard the coal in a ship or in a stockpile as a single consignment. Nevertheless, the conditions of continuous sampling might apply occasionally if coal from a single source were regularly received by ship or by barge.

3.5 Replicate sampling

3.5.1 *General*

As explained in 3.4, a check on the precision which has been obtained can be carried out by adopting the procedure of replicate sampling. 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 will be noted that each replicate sub-sample will be composed of a smaller number of increments than normal.

Replicate sampling cannot be used to test whether bias is absent since if it were present all the results would be equally affected by it. The precision of the sampling as assessed from replicate sampling, therefore, applies only if it has been established that no bias is present. A procedure for testing a sampling procedure for bias is given in annex E.

Unless a large number of sub-samples is considered, there is a large margin of error attached to the calculated precision. Thus it is generally preferable to test whether the desired precision has been attained instead of calculating the precision.

When a coal is regularly sampled at the same point, it is possible to test whether the desired precision has been attained and, if not, to adjust the sampling procedure progressively so as to attain the desired level.

When sampling from a stream of material, it is necessary to make a distinction between "continuous" sampling and "intermittent" sampling (see 3.4.3 and 3.4.4). In continuous sampling, a sample is taken from each consignment or "unit" of coal received. The average quality of the coal over a period is then known with a precision that is directly related to the precision of each sample and the number of samples taken. With intermittent sampling, some units are not sampled and the precision of the period average then depends on the variation in quality from unit to unit as well as on the precision of each sample result. It

It is recommended that replicate sampling should be used :

a) WHEN SAMPLING SINGLE CONSIGNMENTS :

to provide a retrospective check as to whether the desired precision has been attained and, if not, to calculate the actual precision;

b) WHEN SAMPLING REGULAR CONSIGNMENTS :

to determine whether the desired precision has been attained and, if not, to adjust the procedure so that the required precision will be attained in the future with the least possible number of increments.

The testing procedure is different for single consignments and for regular consignments.

3.5.2 *Single consignments*

1) PRINCIPLE

For a single consignment the sample is collected in six replicate sub-samples and each is analysed separately. A statistical test is carried out on the six results to determine whether the desired precision has been attained. The procedure is described in annex C.

2) PROCEDURE

Determine the initial number of increments to be taken by reference to table 3. If this is not divisible by six, increase it to the next multiple of six. Check whether there will be sufficient coal in each of the samples to provide laboratory samples for ash (and, if necessary, moisture); if not, increase the number of increments to the next multiple of six until there will be sufficient coal.

Provide six sub-sample containers labelled A to F, then collect the specified number of increments in the usual way; place the first increment into the container labelled A, the second increment into B and so on; the seventh increment is again placed in A, the eighth in B and so on. Proceed in this way, placing the increments successively into the six containers, so that each has the same number of increments in it.

When the six replicate sub-samples have been collected, prepare laboratory samples from each container in accordance with clauses 8 and 9. Determine the moisture, ash and any other characteristic required on each of these. Thus six results are obtained for each characteristic.

Tabulate the results and carry out the statistical analysis as described in annex C.

3.5.3 *Regular consignments*

1) PRINCIPLE

For regular consignments, each sample is collected in duplicate; however, since the coal is regularly received there will be a number of such duplicate samples. From the series of duplicate results, statistical tests are carried out to determine whether the desired precision has been achieved. The procedure is described in annex C.

Although the sampling procedures for continuous and intermittent sampling are the same, the testing of the results differs — see annex C.

2) PROCEDURE

Determine the initial number of increments to be taken by reference to table 3. Provide two sub-sample containers labelled A and B, then collect the increments in the usual way, place the first increment into container A, the second into B, the third into A, the fourth into B and so on.

Proceed in this manner so that alternate increments are placed successively into the two containers. The increments for B should be taken approximately midway between two successive increments for A. When the whole of the sample has been collected, prepare a laboratory sample from each of the duplicate sub-samples in accordance with clauses 8 and 9. Determine the moisture, ash or any other characteristic required on each duplicate sub-sample.

Continue this procedure of duplicate sampling and analysis for ten consignments. Tabulate the results and carry out the statistical tests described in annex C.

Once a satisfactory sampling level has been achieved, it is necessary to take only occasional samples in duplicate as described in annex C.

3.5.4 *Determination of characteristics other than moisture and ash*

During replicate sampling, a number of different sub-samples will be obtained. These are reduced, mixed and divided as separate laboratory samples, which are then tested separately for ash, and possibly for moisture, depending on which characteristics are being checked.

Usually it will not be necessary to carry out a statistical check on the other characteristics and accordingly all the laboratory samples may be put together and thoroughly mixed in order to provide one sample for the determination of the other characteristics.

4 SAMPLING FROM A STREAM OF COAL

4.1 Scope

This clause describes the method of sampling from a stream of material, whether moving or stationary. It includes the reference method of removing a section from a stopped belt, against which any other method may be checked.

4.2 Number of increments

4.2.1 *Isolated consignments*

The initial number of increments for an ash or moisture sample, for consignments of up to 1 000 tonnes from a single source (for example a single seam at a colliery), is given in table 5. For larger consignments see 3.2.3. If there is any doubt as to the condition of the coal, it should be assumed that it falls into the class which requires the greater number of increments.

TABLE 5 — **Initial number of increments for sampling from a stream of material**

Condition of coal	Number of increments for sampling for ash	Condition of coal	Number of increments for sampling for moisture
Cleaned	16	Unwashed or dry coal; washed graded coal	16
Uncleaned	32	Washed smalls	32

4.2.2 *Regular consignments*

For both continuous and intermittent sampling (see 3.4.2), sufficient increments should be taken from each unit or, for intermittent sampling, from the selected units, so that the total number of increments taken over the period which the sample is required to represent is that given in table 5.

For continuous sampling the precision specified in clause 3 will then refer to the period chosen. It is strongly recommended that the method of replicate sampling described in 3.5 should be applied and the number of increments of the next delivery adjusted, if necessary.

For intermittent sampling, it is essential to use the methods of 3.5. Unless these methods are used there will be no information about the unit to unit variation, the results obtained will always be less precise than specified in clause 3 and the precision achieved will not be known.

4.2.3 *Common sample*

Where a moisture sample is to be extracted from a common sample, the initial number of increments collected should be that required for ash **or** moisture, whichever is the greater. If there will not be sufficient coal left for the ash sample after the removal of the moisture sample in accordance with clause 8, the mass of sample given by this number of increments must be increased, if necessary by taking extra increments.

4.3 Taking the sample

4.3.1 *Reference method*

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 taking an increment by removing a section from a stopped belt (see 4.4) is the **only** way of ensuring that all the particles are collected and hence that the sample is free from bias. Therefore, this is the reference method against which any other method may be checked. It should always be used to check sampling machines because these are particularly subject to bias, although many models free from bias have been devised (see annex A).

4.3.2 *General*

It is important that the interval of time between successive increments should not coincide with any natural periodicity, either known or possible, in the quantity or quality of the coal being sampled, since this would introduce a bias. Such a periodicity may arise from the mining cycle or system of working and particular care should be taken to avoid this. If the increments are taken at equal intervals of time, their masses should be proportional to the flow density; otherwise, they should be equal in mass.

Increments should be taken from the whole width and thickness of the coal stream. If possible this should be done in a single operation and the width of the section should be at least 2,5 times the maximum size of the coal. Biased samples will be obtained if part of the coal is excluded. It is therefore important that the sampler should be able to reach the whole cross-section of the stream in safety and without undue physical strain.

Increments must be taken while there is a normal load on the belt at the point of sampling; special care must be taken not to take increments from the beginning or end of a flow.

The method of sampling depends on whether the coal is sampled

a) from a stopped belt;

b) from a point of discharge of a continuously moving stream;

c) from a moving stream on a belt;

d) from a moving stream which is discontinuous (for example a bucket conveyor).

Automatic sampling machines should be used if possible (see 4.5.1) preferably at a point of discharge (b) or, if this is not possible, from a moving stream (c).

For coals of 80 mm top size and above, manual sampling from a moving stream may be dangerous and the belt should be stopped if possible, or a sampling machine should be used.

4.4 Sampling from a stopped belt

If it is practicable to arrange to stop the belt periodically, increments can be collected from the whole cross-section of the stream without difficulty. A suitable frame may be placed on the stationary belt so that it is in contact with the belt across its full width; all coal lying inside the frame should be swept off into a container. Any large pieces of coal obstructing the insertion of the frame are pushed

a) at the left side of the frame, into the sample;

b) at the right side of the frame, out of the sample.

4.5 Sampling from a point of discharge of a continuously moving stream

This is the most reliable method of obtaining satisfactory increments when the coal is in motion. The increments may be taken by means of sampling machines or by hand.

4.5.1 *Sampling machines*

Sampling machines, controlled by hand or automatically, are available which will traverse a falling stream of coal at constant speed. They should be adjusted carefully to ensure that the whole thickness and width of the stream is taken. The increment must not fill the sample container completely. The sample must be retained in a closed container. Sampling machines which have been proved to be free from bias are preferable to manual sampling methods because they always behave in the same way and eliminate the subjective influence of the sampler.

Machines of any design should have their performance tested against a reference method, i.e. a belt which has been temporarily stopped for this purpose in accordance with 4.4. A procedure for checking for bias is described in annex E. It will usually be found possible to arrange a special trial of this nature purely for test purposes – if necessary by arranging a telephone system so that interlinked belts can be stopped and the load of coal on the sample belt stopped or reduced to avoid difficulty in restarting; however, care must be taken to avoid taking an increment from the beginning or end of a flow.

4.5.2 *Sampling by hand*

Increments may be taken from a falling stream by means of a hand scoop or ladle which is moved **across** the width of the stream at a constant rate. When sampling by hand alternate increments should be taken by crossing the stream in opposite directions.

The increment should not fill the sample container completely.

4.5.3 *Sampling wide streams*

Sampling wide and high-capacity streams is best done by mechanical methods. Where these are not available and it is impossible to sample across the whole of the width of the stream in one movement without over-filling the container, the stream should be sampled systematically taking the increments from parts of the stream in turn.

The following scheme shows a method of taking increments from a wide stream of uniform depth in two parts :

X X X etc.
X X X

This scheme can be extended for falling streams to any number of portions depending on the width of the stream. Three positions will normally be sufficient, but for very wide streams four or five positions may be necessary.

The stream must be sampled by passing the scoop through it once and then withdrawing it in such a way that the full scoop is not passed a second time through the stream; this may be achieved by inverting the scoop, passing it to the back of the stream and withdrawing it through the stream; alternatively the scoop may be filled in passing from front to rear provided that it can then be withdrawn away from the stream – for example, by moving it sideways.

Whichever method is used the increment should not fill the sample container after it has traversed the stream.

It may be necessary to support the handle of the sample container across a bar when it is passed into the falling stream or to erect a special gantry with adequate supports.

4.5.4 *Duplicate sampling from wide streams*

If the width of the stream is too great to be sampled as a whole, successive increments for the A and B samples should be taken from the same part of the stream followed by successive increments from the other parts of the stream. In principle the two sub-samples should be identical as regards the methods of taking the increments which compose them.

Thus duplicate samples may be taken from a stream of uniform depth in two parts, as follows :

A B A B etc.
A B

This scheme can be extended for falling streams to any number of portions depending on the width of the stream.

4.6 Sampling from a moving belt

Sampling from a moving belt may be necessary if it is impossible to sample satisfactorily at a point of discharge. This procedure demands skill and good judgement on the part of the sampler. Care should be taken to ensure that the whole thickness of the stream is sampled. The scoop should move along with the flow and should sweep the bottom of the conveyor, otherwise there will be a tendency to leave behind some of the small coal. This method is unsuitable where there are several layers of different coals on the conveyor belt.

If it is impossible to sample the whole width of the stream from one side, increments should be taken alternately from both sides of the belt (see 4.5.3).

Belts moving at high speeds or carrying heavy loads are dangerous; manual sampling from moving belts is therefore recommended only when the speed of the belt is not greater than 1,5 m/s, the height of the coal is not greater than 0,3 m and the flow is not greater than 200 tonnes per hour.

Sampling machines are also available which will sweep an increment from a moving belt.

4.7 Sampling from discontinuously moving streams

Such devices are discharging conveyors, bucket elevators, bucket conveyors or aerial ropeways. Sampling may be carried out provided that the mass of the contents of a bucket is not less than the mass of increment required, a condition that is fulfilled in nearly all cases. Increments should be collected from the coal stream at the point of discharge or at any point by stopping the line. The whole contents of a bucket should be taken as an increment. With large buckets, each bucket may be divided into sections if each is larger than the specified mass of increment; one of these only is taken, but in successive buckets each section must be taken in rotation.

4.8 Apparatus

For suitable sampling devices, see annex A.

5 SAMPLING FROM WAGONS

5.1 Scope

This clause describes the method of sampling from railway wagons when the preferred procedure of sampling from a conveyor belt during loading or unloading cannot be used.

5.2 Number of increments

5.2.1 *Ash or moisture sample*

The initial number of increments to be taken from consignments up to 1 000 tonnes is shown in table 6. For larger consignments, see 3.2.3.

The increments specified in table 6 should be distributed uniformly over the whole consignment and, as a minimum, one increment should be taken from every wagon even when the number of increments specified is less than the number of wagons. When the number of increments specified is greater than the number of wagons in the consignment, the number of increments taken from each wagon should be determined by dividing the total number of increments by the number of wagons; if after this division there is a remainder of increments, these should be distributed uniformly over the consignment.

TABLE 6 — **Initial number of increments for sampling from wagons**

Condition of coal	Number of increments for sampling for ash	Condition of coal	Number of increments for sampling for moisture
Cleaned	24	Unwashed or dry coal; washed graded coal	16
Uncleaned	48	Washed smalls	32

5.2.2 *Common sample*

Where a moisture sample is to be extracted from a common sample the initial number of increments collected should be that required for ash or moisture, whichever is the greater. The mass of each increment or the number of increments should be increased if there will not be sufficient coal left for the ash sample after the removal of the moisture sample in accordance with clause 8.

5.3 Taking the sample

5.3.1 *General*

The following methods of reaching all the coal in a wagon are available :

a) sampling from the tops of wagons by means of probes;

b) sampling from bottom or side door wagons during discharge;

c) sampling from the exposed faces of wagons as they are being tipped into bunkers or ships;

d) sampling from wagons being emptied by side-tipplers.

The method of sampling should be recorded in the report.

5.4 Sampling from the tops of wagons

5.4.1 *Sampling by probes*

Owing to the difficulty of insertion, a probe can only be used for coals up to about 25 mm. Probes suitable for larger coal may be developed. The aperture of the probe should be not less than 2,5 times the upper size of the coal, with a minimum dimension of 30 mm. A probe which penetrates to the full depth of the coal must be used. Care should be taken to ensure that a full column of coal is extracted, so that a representative increment is removed. Large and hard pieces of coal or rock should not be deliberately pushed aside when an increment is collected and no portion of the increment should be lost during extraction of the probe from the wagon. In addition, contamination of the increment by particles falling in from outside the hole should be prevented. Wet coal should not be allowed to adhere to the probe, but the probe must not be heated to stop wet coal sticking to it. The effectiveness of a probe, and whether it is free from bias, depends on the nature of the coal being sampled and the method of loading. Tests for bias should be carried out (see annex E).

5.4.2 *Sampling by scoops*

It is not always possible to insert a probe to the full depth of the wagons and it is then necessary to collect an increment from the bottom of a shallow hole made in the coal. Care is needed in digging the hole. The coal dug out of the hole should be thrown clear and the angle of the sides should be less than the angle of rest, otherwise large pieces will trickle down the sides and the increment will contain too much large coal. The increment, of the minimum mass given in 3.3, should be taken from the bottom of the hole with a shovel (see annex A).

Alternatively, with some coals the hole may be dug with an auger, the last portion extracted being the increment. Again, care should be taken to ensure that the walls of the hole do not collapse and that no portion of the increment is lost from the auger. Sampling by these methods is not regarded as satisfactory, since the whole of the coal is not exposed for sampling, and tends to give biased results. These methods should be adopted only when all other methods are impossible.

5.4.3 *Coals which are visibly wet — Moisture or common sample*

Water tends to descend to the lowest level. Consequently, for coals which are visibly wet the holes for moisture samples should be dug to one half the depth of the wagon. This method is laborious and should be avoided if possible. It gives unbiased results only if the coal is thoroughly drained and should not be used while drainage is actually in progress.

5.4.4 *Coals which are not visibly wet — Moisture or common sample*

For coals which are not visibly wet the holes should be at least 0,3 m deep to avoid contamination of the sample from the surface.

5.4.5 *Sampling for ash only*

When sampling from wagon tops for ash only, the increments should be taken from the bottom of holes at least 0,3 m deep, whether the coal is visibly wet or not.

5.4.6 *Position of the increments*

The positions of the increments should be varied from wagon to wagon so that all parts of the surface are correctly represented. There are various methods of doing this and different schemes may be preferred for use with different designs or sizes of wagons.

It is recommended that the surface of the wagon be divided into squares each of side about 1 m, the number of squares being dependent on the size of the wagon. Increments should be taken from the squares in random order, but can be taken from any position within the appropriate square (see 5.10 and 5.11).

Care should be taken to see that the coal near the periphery of the wagon is adequately represented.

As indicated in clause 6, the same type of scheme should be used for the positions of increments from the surface of barges. All schemes used should be tested for bias (see annex E). This method is inapplicable if the wagon has been loaded in layers with different types of coal.

5.4.7 *Sampling of large coals*

When the coal includes particles larger than 150 mm, the number of increments specified in table 6 should be taken in proportion to the mass of the pieces above and below 150 mm. To determine the percentage of large pieces, each coal should be mechanically tested every 3 months, by taking not less than 10 tonnes of the coal before loading into railway wagons. This should be passed over a horizontal sieve with apertures 150 mm × 150 mm : the coal which does not pass through the sieve should be weighed and expressed as a percentage of the whole mass. If the coal size varies appreciably in the 3 months, a test should be carried out every month.

Increments should be taken from the wagons and from the areas according to the system described above. At each position a hole should be dug to a depth of 0,3 m. Any large pieces in the hole should be removed and collected in boxes, **which should be kept separate from the increments.** If there are no large pieces in the hole, they should be taken from the next hole according to the sampling plan. The total mass of large pieces should be at least 8 kg. The large pieces should be reduced to 80 mm, divided and mixed into the sample in proportion to the appropriate percentage as determined by the mechanical sizing test described above.

The number of increments from the smaller coal in the hole should be taken in accordance with the normal procedure.

5.5 Sampling from wagons during bottom discharge

In this method the coal is being sampled while the wagons are unloaded. As large a container as can be handled is swung into the stream, the position of entry being varied from wagon to wagon. Owing to the rapid fall of coal, it is difficult to obtain increments from all parts of the stream and mechanical aids are required to render the method reasonably safe.

In general, the same sample should be used for both ash and moisture since there is insufficient time to take two increments from the same wagon. This method is difficult to operate and is dangerous. Other methods should be used if possible.

5.6 Sampling from bottom door wagons

In wagons with eight doors only the four centre doors are used; in those with only four doors, all are used. The wagons may be sampled either from exposed faces or during discharge.

5.6.1 *Exposed face method*

Two doors side by side are opened and part of the coal is discharged into the bunker. The doors are then shut and bolted, leaving two sloping faces at rest in the wagon; it is assumed that the faces are numbered in areas as shown in figure 1 and increments are taken from the numbered areas. The doors to be opened are taken at opposite ends of the wagon in alternate wagons.

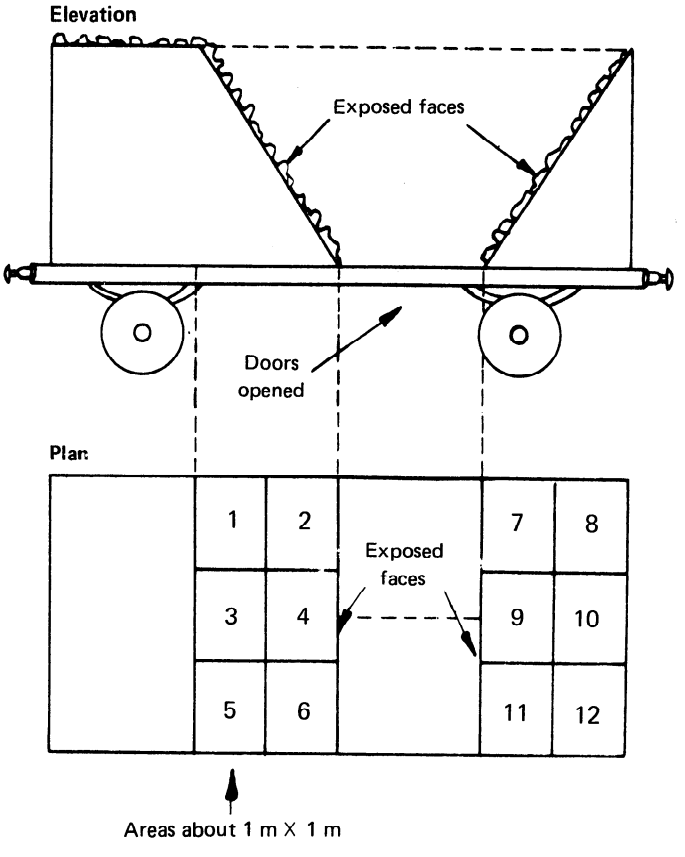

FIGURE 1 — **Exposed faces in wagon**

5.6.2 *Discharge method*

Increments are collected from underneath the wagon as the stream of coal falls from the doors; the method described in 5.5 should be used.

5.7 Sampling from wagon side-tipplers

When wagons are side-tippled the coal is sampled from the exposed surface of the coal after the wagon has been partly emptied. The wagon is half-emptied so that the bottom is just visible, leaving a face at rest. The wagon is then lowered slightly to prevent the face slipping. A suitable number of sampling areas are allocated to this face as in wagon top sampling and the increments are collected from the points in the same manner as above. It may be necessary to erect special gantries on rollers which can be pushed forward or retracted; alternatively, use a scoop with a long handle.

5.8 Sampling for moisture

A wagon loaded with washed coal will gradually lose water by drainage and it is therefore undesirable to sample for moisture until equilibrium is reached, which may take 2 or 3 days. However, if a sample is required for other purposes, or if it is desired to determine the moisture content at a particular time, a sample can be collected before equilibrium is reached.

Even when equilibrium has been reached as a result of drainage, the lower layers of coal in the wagon will normally contain more water than the upper layers because of gravitation and evaporation from the surface. Increments taken from near the top of the wagon after any drainage has taken place will therefore be **biased** with regard to their moisture content. The bias will normally be such as to give a low moisture figure, unless the coal has been recently subjected to a heavy fall of rain or snow.

5.9 Duplicate sampling

When every wagon is sampled, duplicate sub-samples are formed by placing alternate increments into different containers. When some wagons are not sampled, the increments from **alternate wagons sampled** are placed into different containers to form the two sub-samples.

5.10 Example

The following example shows a typical procedure. A further example is given in annex B.

A sample of 48 increments is required from a train of 40 wagons, each approximately 3 m wide and 6 m long.

Each wagon should be divided into 18 sections (3 × 6) as follows :

1	4	7	10	13	16
2	5	8	11	14	17
3	6	9	12	15	18

where numbers refer to the positions to be used for taking the increments.

In order to obtain the required number of increments, one or two will be required from every wagon; the position numbers and the numbers of the wagons to have 2 increments should be selected at random (see 5.11).

5.11 Random samples

One method for obtaining random samples is as follows :

Provide a set of discs, one disc for each position, suitably marked; for example, a set of discs numbered 1 to 18 for the example in 5.10. The discs should be placed in a bag close to the sampling point, together with a diagram painted on a fixed board showing the locations of the points over the surface of the wagon. On sampling from the first selected wagon, the sampling operator should remove from the bag one, two or three discs, to correspond with the number of increments to be taken from this wagon. An increment should be collected from each area indicated by the discs. The discs should be placed in a second bag after use. For the second wagon the same procedure is used, the discs being removed from those remaining in the first bag. This process continues until all the discs are used up. The position of the bags is then reversed and the procedure continued, so that the order of the positions from which increments are taken is always different.

This procedure can also be used for selecting the wagons to be sampled, when some are sampled and some are not. For example, suppose 36 wagons are to be sampled out of a consignment of 100. A set of discs numbered 1 to 100 is placed in a bag and the sampling operator draws from the bag 36 numbered discs in succession. The selected discs may be hung on hooks on a reference board and the wagons numbered serially with chalk as they pass. The wagons corresponding to the numbers drawn should be sampled.

6 SAMPLING FROM SHIPS

6.1 Scope

This clause describes the method of sampling from sea-going ships and barges where the preferred procedure of sampling from a conveyor belt during loading or unloading cannot be used.

6.2 Number of increments

6.2.1 *Ash and moisture sample*

The initial number of increments to be taken from a consignment in a sea-going ship of up to 1 000 tonnes from a single source is given in table 7. For larger consignments, see 3.2.3. For a consignment in a barge the numbers given in table 8 should be used, which are the same as for wagons.

TABLE 7 — **Initial number of increments for sampling from sea-going ships**

Condition of coal	Number of increments for sampling for ash	Condition of coal	Number of increments for sampling for moisture
Cleaned	32	Unwashed or dry coal; washed graded coal	16
Uncleaned	64	Washed smalls	32

TABLE 8 — **Initial number of increments for sampling from barges**

Condition of coal	Number of increments for sampling for ash	Condition of coal	Number of increments for sampling for moisture
Cleaned	24	Unwashed or dry coal; washed graded coal	16
Uncleaned	48	Washed smalls	32

6.2.2 *Common sample*

Where a moisture sample is extracted from a common sample the initial number of increments collected is that required for ash or moisture, whichever is the greater. The mass of each increment or the number of increments should be increased if there will not be sufficient coal left for the ash sample after the removal of the moisture sample, in accordance with clause 8.

6.2.3 *Mixture of coals*

If it is known that different coals are loaded into different holds, each coal should be sampled separately. If no information is available on the origin or characteristics of the coal, each hold should be considered as a unit and 48 increments should be taken from each.

6.3 Taking the sample

6.3.1 *General*

This recommendation for the sampling of coal in sea-going ships and barges is based on collecting increments from a number of points distributed over various layers of the coal in the hold which are exposed from time to time as the ship is unloaded.

If during loading or unloading the coal is moved by conveyor, the sample should, preferably, be taken from some point in the conveying system where bias can be avoided more easily. The procedure set out in clause 4 should then be followed, but in view of the segregation that occurs during the loading of a ship, the number of increments indicated in table 8 should be taken.

Usually, however, it is necessary to sample from the hold of the ship, and it is essential to employ a trained and experienced sampler who works under the direction of an expert. The size distribution of the cargo should be estimated so as to ensure that the increments taken are representative.

It is important to note that segregation during loading often results in the accumulation of lumps near the walls of the hold. It is necessary to take this into account when making an estimate of the size distribution.

The sampler's skill in estimating the size distribution should be regularly checked by such means as are convenient. One suitable method is to use a grab to remove a portion of the cargo of substantial size and to check the sampler's estimate of the size distribution of the contents by a size analysis carried out on the whole of this portion. Alternatively, his estimate can be checked by that of an expert who is not actually responsible for carrying out the sampling.

When sampling from a sea-going ship the opportunities for sampling are often limited since delays during unloading must be avoided. For this reason, procedures that are not ideal may have to be adopted on occasions. So that the sample will contain the same proportion of lumps and smalls as are present in the cargo, the percentage of lumps should be estimated; then the smalls and lumps are sampled separately. The lumps are picked by hand until a reasonable mass is available. From every lump one or two pieces perpendicular to the bedding planes are knocked off with the aid of a hammer, these pieces are collected and an amount is added to the sample of the smalls according to the estimate of the proportion of lumps in the cargo. Reference should also be made to 3.3.3 regarding other procedures for taking increments from large coal.

In collecting each increment care should be taken to ensure that it represents the coal in the vicinity and, in particular, that large particles are not allowed to roll from or into the scoop when the increment is extracted.

When sampling fines, a probe is preferable to a scoop (see 5.4.1).

Care should be taken to ensure that the increments are not contaminated because of prevailing atmospheric conditions. As the top layer is liable to be affected by rain, or dried by the wind, it is desirable to collect increments 0,2 to 0,3 m below the surface unless this has been uncovered very recently.

6.3.2 *Position of the increments*

The position of the increments should be distributed over the surface of the coal and the procedure of 5.4.6 and 5.11 should be used.

6.3.3 *Sampling from barges*

If the depth of coal in the hold is less than 4 m, it should be sampled in one stage during the unloading. The sampling should be carried out when the unloading has partly uncovered the bottom of the hold.

6.3.4 *Sampling of fines from barges*

For the sampling of fines from a barge, a longer probe of a type such as is described in annex A can often be used with advantage. In this case 2 increments, comprising a complete cross-section, should be taken from each hold. The position of the increments should be varied from hold to hold.

6.3.5 *Sampling from coasters or sea-going vessels*

Coasters or sea-going vessels should be sampled in two or more stages, there being one stage for each 4 m of the depth of coal in the hold. Figure 2 illustrates the stages at which sampling should be carried out when it is done in three stages.

The first stage of sampling should not take place until sufficient of the surface of the coal has been removed to permit an estimate to be made of the proportions of fine and lump coal in the consignment.

At each stage increments should be collected from points evenly distributed over the exposed surface of the coal, taking particular care that all portions of the surface are covered satisfactorily.

6.3.6 *Sampling for moisture*

If free moisture is present in the cargo this will settle towards the bottom of the hold so that a steady increase in moisture content will occur through the depth of the coal. This increase of moisture with depth makes the collection of a moisture sample particularly difficult when sampling is carried out in one stage (for example from barges). Increments should then be taken at three different levels, namely, at an upper and lower level, each equidistant from the centre level, and at half-depth (see figure 2).

Provided that the full depth of coal is exposed, the upper level should be chosen 0,1 to 0,2 m from the top and the lower level at an equal distance from the bottom. If this is not possible (because the bottom has not been exposed, or because the top coal has been removed) then the distance between the upper and lower levels should be as great as possible, subject to the condition that they are equidistant from the centre. Equal numbers of increments should be taken from the lower, centre and upper levels.

6.3.7 *Sampling from grabs or conveyors*

When the coal in a ship or barge is unloaded by means of grabs or conveyors, it may be preferable to use the sampling scheme recommended in 4.7. However, the number of increments recommended in table 7 or 8, as appropriate, should be collected.

6.4 Example

The following example shows a method of sampling from a barge.

A sample of 48 increments is required from a barge of width 5 m and length 20 m.

The barge should be divided into 100 sections (5 X 20), as follows, and 48 of the positions should be selected at random.

1																			96
2																			97
3																			98
4																			99
5																			100

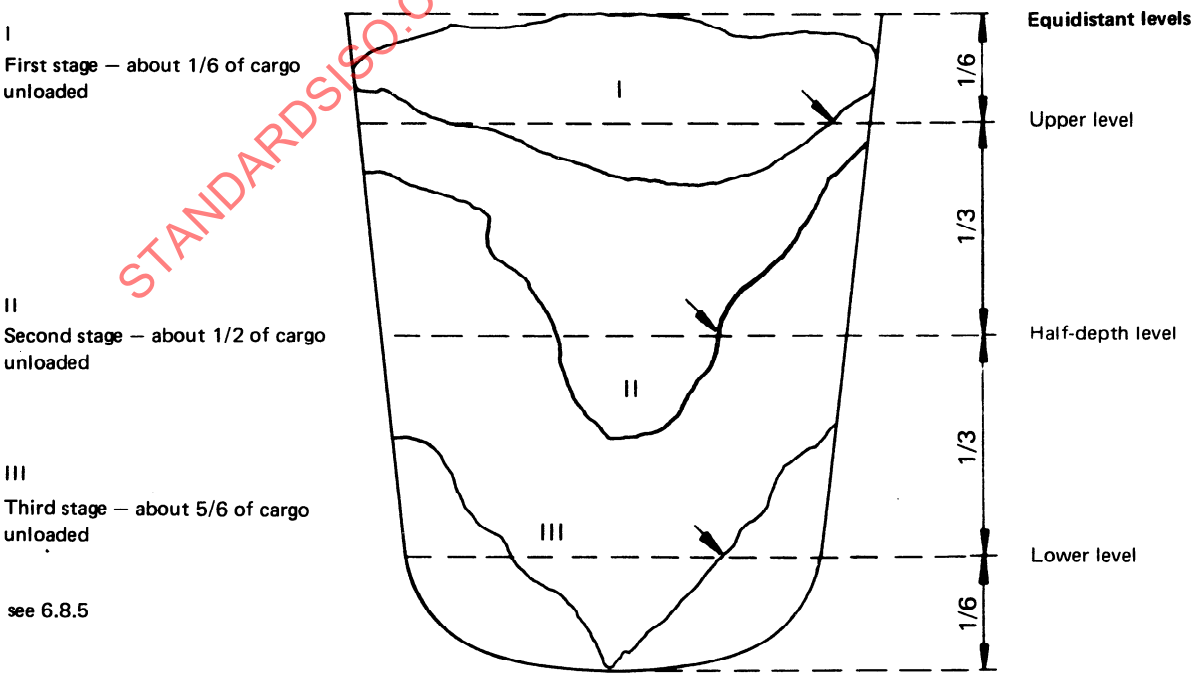

FIGURE 2 — **Sampling the hold of a vessel (moisture and ash)**

7 SAMPLING FROM STOCKPILES

7.1 Scope

This clause describes the method of sampling from stockpiles where the preferred procedure of sampling from a conveyor belt during stocking or unstocking cannot be used.

7.2 Number of increments

7.2.1 *Ash or moisture sample*

The initial number of increments to be taken from a pile of up to 1 000 tonnes from a single source is shown in table 9. For larger consignments see 3.2.3.

TABLE 9 — **Initial number of increments for sampling from stockpiles**

Condition of coal	Number of increments for sampling for ash	Condition of coal	Number of increments for sampling for moisture
Cleaned	32	Unwashed or dry coal; washed graded coal	16
Uncleaned	64	Washed smalls	32

7.2.2 *Common sample*

Where a moisture sample is extracted from a common sample the initial number of increments collected is that required for ash **or** moisture, whichever is the greater. The mass of each increment or the number of increments should be increased if there will not be sufficient coal left for the ash sample after the removal of the moisture sample in accordance with clause 8.

7.2.3 *Mixture of coals*

If a stockpile is known to consist of different coals piled in separate areas of the total pile, a separate gross sample must be taken from each such area.

If the sampling is carried out by digging holes in the stockpile, for example by means of grabs, increments of very different masses may be taken from different positions. Consequently, if the coal is not uniform or has come from different sources, the increments should not be put together, but each should be prepared and analysed separately. For each increment, the value of the required characteristic should be determined; the value for the whole pile should then be obtained by taking an average of the increment values, weighted according to the masses of the different coals from which the increments were taken.

7.3 Taking the sample

7.3.1 *General*

This recommendation for the sampling of coal in stockpiles is based on collecting increments spaced as evenly as possible over the surface and layers of the stockpiles. The usual methods of sampling stationary material, using relatively small probes or digging holes to traverse the layers, are not adequate to fulfil the basic requirement of sampling, that the whole of the consignment shall be equally accessible. Moreover, the coal in the top layer of a stockpile is almost always different in quality from the rest, due to exposure, segregation and other causes. Consequently, if in the course of stocking or lifting it is possible to take a representative sample from a conveyor or from a falling stream, this method of sampling is to be preferred and the procedure described in clause 4 should be used except that the number of increments should be as indicated in table 9.

The position of the increments should be spaced as evenly as possible over the surface of the stockpile. It is advisable, especially in the case of large stockpiles, to work to a plan, to indicate the sampling positions on a scale drawing or map of the area and to mark the positions before sampling.

In all cases the sample can only represent that part and that depth of the coal from which it is collected. It is essential to use trained and experienced samplers.

7.3.2 *Sampling for ash*

In order that the sample will contain the same proportions of large pieces and small pieces as are estimated for the stockpile, and at the same time to maintain individual increments at a reasonable mass, it may be necessary to break some of the large lumps perpendicular to the bedding planes and to add a proportion of the broken coal to the sample.

In collecting each increment care should be taken to ensure that it represents the coal in the vicinity and, in particular, that large particles are not allowed to roll from or into the scoop when the increment is extracted.

When sampling coal of up to 25 mm particle size, the increment should be taken with a probe (see annex A). Care should be taken that the full column of coal is taken out and that no particles are lost when the probe is extracted.

When sampling coal of more than 25 mm particle size, or when it is impossible to sample with a probe, it is necessary to dig holes by means of a shovel or a grab-dredger. The angle of the sides should be less than the angle of rest of the coal so that no particles will trickle down the sides. Holes should be dug to different depths and from the bottom of each hole an increment should be taken by shovel so that samples of approximately the same mass are collected from different layers.

7.3.3 *Sampling for moisture*

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

Stored coal gradually loses water by drainage until equilibrium is reached. After rainfall or snow, the moisture content will not change below a certain depth. This depth depends on the size distribution and the arrangements made during stocking, for example, continuous rolling during stocking, covering with dust coal, or rolling the dust coal.

The moisture content of samples collected from the surface of the stockpile largely depends on the weather. It will always be too low unless the samples are collected after rainfall or snow. Hence it is necessary to dig holes to such a depth as will avoid the surface layer.

8 SAMPLE PREPARATION FOR DETERMINATION OF TOTAL MOISTURE

8.1 Types of samples to be tested

The sample to be treated will be either a special moisture sample or a common sample (see 2.4). If the sample is a common sample, it is necessary to extract a moisture sample from it and leave the remainder for treatment as the ash sample.

The moisture sample may be removed

a) before any reduction is carried out, or

b) at various stages of the reduction process.

The method to be adopted depends on the apparatus available for reduction and the preference at the laboratory.

For a special moisture sample, proceed as described in 8.3. For a common sample, extract the moisture sample as described in 8.4.1; then treat this moisture sample by the methods described in 8.3.

8.2 General principles

8.2.1 *Precautions against loss of moisture*

One of the main difficulties in ascertaining the total moisture is to avoid changes in the moisture content of the sample during the handling which is necessary while preparing the final sample. Every precaution should therefore be taken to avoid loss of water due to the use of unsuitable containers and by evaporation during handling, particularly when the coal is extremely wet. All moisture samples should therefore be kept in closed containers in a cool place before and after preparation as well as during any interval between stages of sample preparation.

Care should also be taken to avoid loss of moisture during reduction, by using equipment in which there is no appreciable heating and by reducing to a minimum the amount of air passing through the mill. Machines that crush are preferable to those that grind, as the latter have a greater tendency to generate heat.

In addition, care should be taken to avoid loss of moisture when carrying out sample division and all such operations should be carried out as quickly as possible. If the coal is "visibly wet" and has not been milled, increments should be collected from a flattened heap as this will lead to less loss of moisture. In all other cases, it is preferable to use mechanically operated dividers to which access of air is restricted.

8.2.2 *Methods available*

The method of sample preparation depends on the method of analysis to be used and whether a preliminary air-drying is necessary. Three alternative methods[1] are permissible for the determination of total moisture in hard coal, each of which may be preceded by a preliminary air-drying if necessary :

Method A — sample heated with toluene

Method B — sample dried in an oven in nitrogen

Method C — sample dried in an oven in air

Methods A and B are applicable to all hard coals and require a sample of not less than 300 g of coal of size 3 mm.

Method C is applicable only to coals which are not susceptible to oxidation[2] and requires a sample of size from 20 to 3 mm with a mass of 1 kg for 20 mm coal, 0,6 kg for 10 mm coal or 0,3 kg for 3 mm coal.

If the coal is "visibly wet", a one-stage method of moisture determination is used provided that a closed mill is available; otherwise a two-stage method is used. If the coal is apparently dry a one-stage method is used.

8.3 Procedure

8.3.1 *Arrangement of scheme*

The procedure differs according to the origin of the sample, the particle size and mass of the sample to be treated and the method to be used for the moisture determination. The possible schemes are shown in figure 3, for Methods A and B, and figure 4 for Method C : figure 5 shows the procedure of removing the moisture sample from a common sample. For ease of reference the initial samples are labelled P, Q, R and S in figures 3 and 4, and corresponding references are made in the text.

For Method C the particle size of the moisture sample depends on the particle size of the original sample, the apparatus available, the preference of the laboratory and on whether the sample is to be removed from a common sample. In this last case the size may be determined by the procedure to be used for crushing the ash sample and the size at which this sample is divided. If the original sample is over 20 mm, it must be reduced.

If the sample is to be reduced to 3 mm, this should be done for preference in a closed mill, which reduces coal of any moisture content (i.e. whether visibly dry or visibly wet) to this size without loss of moisture. With other types of mill, it is difficult to reduce the sample to 3 mm.

1) Described in ISO 589, *Hard coal — Determination of total moisture.*

2) For the purposes of this International Standard, such coals are presumed to be those included in clauses 0 to 5 inclusive of the International Classification of Hard Coals by Type, adopted by the United Nations Economic Commission for Europe.

Therefore, if a closed mill is not available, the sample should be reduced to 10 mm and divided to 1 kg before being reduced to 3 mm. In this case, if the sample is "visibly wet" it should first be air-dried and the loss in mass determined, this being the first stage of a two-stage method.

8.3.2 *Reserve sample*

It is recommended that a reserve sample should be collected so that, in the case of dispute or if the results of the first moisture determination are lost or invalid, this sample may then be examined. The reserve sample should be collected in the same way and at the same time as the final sample for the determination of moisture. If the division is done mechanically, two sample containers should be used. If not, twice as many increments as are specified above should be taken and placed alternately into two containers, one for the moisture sample and one for the reserve sample.

If more than one reserve sample is required, appropriate steps should be taken to collect the necessary number of inter-leaved increments.

8.3.3 *Sample preparation for one-stage moisture determination*

This procedure may be used for coals that are "visibly dry" and also for coals that are "visibly wet" prepared in a closed mill.

a) SAMPLE DIVISION

Preferably sample division should be done mechanically or with a riffle. Suitable mechanical sample dividers and riffles are described in annex A.

If suitable dividers are not available, or if the sample is too damp to flow freely through the dividers, use the following procedure : The sample should be carefully mixed, formed into a cone and the cone flattened. At least ten increments of equal mass, sufficient to give the required total mass, should be extracted from positions evenly spaced over the flattened pile (see table "Mass of moisture sample", figure 3).

b) FOR METHODS A AND B (see 8.2.2)

1) Closed mill available

The closed mill should be conditioned by crushing in it some of the coal from which the sample was taken. The product should be discarded and then the whole of the moisture sample should be passed through the mill and reduced to 3 mm in size.

The sample should be divided to 300 g (see figure 3, path S).

The 300 g sample should be placed in a bottle fitted with an air-tight stopper and the bottle labelled, giving details of the sample.

2) Closed mill not available, coal "visibly dry"

If the maximum particle size exceeds 20 mm, the sample should be reduced mechanically so that it just passes 10 mm. If the coal is smaller than 20 mm, this reduction in size is not necessary.

The sample should then be divided to 1 kg (see figure 3, path R).

The 1 kg sample should be reduced to 3 mm in size and then divided to 300 g.

The 300 g sample should be placed in a bottle fitted with an air-tight stopper and the bottle labelled, giving details of the sample.

c) FOR METHOD C (see 8.2.2)

1) Preparation of sample of 20 mm or 10 mm coal

If the maximum particle size of the coal exceeds 20 mm, the sample should be reduced mechanically to 10 mm in size. If the coal is "visibly wet", a closed mill should be used, or the coal should be air-dried (see 8.3.4). The sample should then be divided to 600 g.

If the maximum particle size of the coal is 20 mm or less, the sample should be divided to give a mass (in kilograms) not less than 0,06 times the maximum particle size (in millimetres).

The container should be labelled, giving details of the sample.

2) Preparation of sample of 3 mm coal

If the sample required is of 3 mm coal, it should be prepared in the same way as described for Methods A and B in 8.3.3 b).

8.3.4 *Sample preparation for two-stage moisture determination*

This sub-clause only applies if the coal is "visibly wet" and a closed mill is not available, so that a preliminary air-drying is necessary. This constitutes a **two-stage** procedure.

a) FOR METHODS A AND B (see 8.2.2)

The sample should be air-dried as described in 8.4.2. After air-drying, the procedure of 8.3.3 b) 2) should be followed and the label on the bottle containing the 300 g sample thus obtained should also show the percentage loss of moisture on air-drying.

b) FOR METHOD C (see 8.2.2)

The sample should be air-dried as described in 8.4.2. After air-drying, the procedure of 8.3.3 c) should be followed and the label on the bottle containing the final sample should show also the percentage loss of moisture on air-drying.

8.4 Methods

8.4.1 *Extraction of moisture sample from common sample*

This sub-clause applies only if the sample is a common sample and the moisture sample has to be removed from it. This may be done before or after reduction, as convenient.

When the coal is reduced before the sample is removed, this reduction forms the first part of the preparation of the general analysis sample (in accordance with clause 9), as well as of the preparation of the moisture sample. Whether the coal is reduced or not, the residue after removal of the moisture sample is treated as the ash sample, in accordance with clause 9, and the next process may be a further division or a further reduction.

This procedure will give one sample for moisture and a second sample for general analysis, which is normally the most convenient procedure.

However, when a closed mill is available and reduction to 3 mm is being practised, it may be preferred to take only one sample for both moisture and general analysis (see c below).

a) EXTRACTION OF MOISTURE SAMPLE BEFORE REDUCTION

Without previous mixing, the gross sample should be tipped on a plate to form a cone and then flattened. The moisture sample (see figure 3, path P, and figure 4, path P) should be extracted by taking ten increments, each increment being taken from a different position and the positions being evenly spaced over the flattened pile. It is essential that this operation be performed quickly to prevent loss of moisture.

The mass of each increment depends on the maximum particle size of the coal, as follows :

For coal up to 25 mm — 0,5 kg

For coal up to 50 mm — 1,0 kg

For coal up to 80 mm — 1,5 kg

For coal over 80 mm — 5,0 kg

For coal over 80 mm, the resulting 50 kg sample should be reduced to about 80 mm, mixed, coned and flattened.

The sample should then be divided to 15 kg. The remainder of the 50 kg sample should be returned to the original sample.

If air-drying or further reduction is not carried out immediately the sample container should be sealed.

b) EXTRACTION OF MOISTURE SAMPLE AFTER REDUCTION

When a closed mill which will crush to 3 mm is available, the whole of the common sample — whether "visibly wet" or "visibly dry" — may be reduced to this size; then the moisture sample of 300 g should be extracted, as described in 8.3.3 b) 1) (see figure 3, path R, and figure 4, path S).

If a closed mill is not available, "visibly wet" coals must be air-dried as described in 8.4.2, then treated as "visibly dry" as described in the next paragraph.

"Visibly dry" coals may be crushed to 10 mm before extracting the moisture sample. If the sample is to be left at 10 mm for Method C, 600 g of coal is required (see figure 4, path Q); if the sample is to be further reduced to 3 mm, for Methods A, B or C, 1 kg is required (see figure 3, path R, and figure 4, path R).

The sample should be reduced further immediately or placed in a clean container and sealed.

c) EXTRACTION OF ONE SAMPLE FOR MOISTURE AND GENERAL ANALYSIS

If the common sample is reduced to 3 mm in a closed mill, **one** sample may be taken for moisture and general analysis. However, in this case the mass retained must be increased so that two laboratory samples (of up to 100 g each) may be taken for moisture determination and sufficient coal left to fulfil the requirements of clause 9. This will require the following masses (but see 9.1.1) :

Condition A
300 g for ash + 200 g for moisture = 500 g

Condition B
2 kg for ash + 200 g for moisture = 2,2 kg

8.4.2 *Determination of loss of moisture on air-drying*

Air-drying should be carried out at a temperature not more than 10 to 15 °C above that of the atmosphere. In countries with hot climates the temperature at which air-drying may be carried out should not exceed 45 °C. It is necessary to permit free circulation of air above the samples but dust should be excluded. The procedure for air-drying differs according to the form in which the coal is received for this purpose.

METHOD a)

If air-drying is to be carried out immediately after a stage in the course of sample preparation, a dry tray should be weighed (m_1) and the coal to be air-dried should be placed directly into the tray. The coal should be spread evenly to a depth not exceeding 20 mm (except for lumps greater than this size). The tray plus coal should then be weighed (m_2). The coal should be allowed to air-dry until the change in mass of the tray plus coal over a period of 1 h is less than 0,1 % of the initial mass of the coal ($m_2 - m_1$). The final mass of the tray plus air-dried coal should be noted (m_3).

Calculation of results :

The percentage loss of moisture on air-drying, M, is given by the formula

$$M = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the dry tray;

m_2 is the mass, in grams, of the tray plus coal;

m_3 is the mass, in grams, of the tray plus coal after air-drying.

METHOD b)

If the sample is delivered in a sealed tin and air-drying is required, the container and coal should be weighed as received, before opening the tin. After weighing, the coal should be transferred to a dry tray and spread evenly to a depth not exceeding 20 mm (except for lumps greater than this size). The tray plus coal should then be weighed. The container, the lid and the coal should be allowed to air-dry until the change in mass of the tray plus coal over a period of 1 h is less than 0,1 % of their original combined mass. Any dried coal should be brushed from the container and lid into the tray and the dry empty container and its lid should be re-weighed. The coal from the tray should be returned to the container, the lid replaced and the whole re-weighed.

Calculation of results :

The percentage loss of moisture on air-drying, M, is given by the formula

$$M = \frac{m_1 - m_3}{m_1 - m_2} \times 100$$

where

m_1 is the mass, in grams, of the closed container plus coal before air-drying;

m_2 is the mass, in grams, of the dry empty container plus lid;

m_3 is the mass, in grams, of the closed container plus coal after air-drying.

8.4.3 *Particle size reduction*

If a closed mill system is used, some of the coal from which the gross sample was extracted should be passed through the system to condition the interior surfaces, before the actual moisture sample itself is milled.

Information about equipment and apparatus is given in annex A.

9 SAMPLE PREPARATION FOR GENERAL ANALYSIS

9.1 General principles

The object of sample preparation is to prepare a small sample, representative of the original sample, which can be sent to the laboratory for analysis. This analysis sample should consist of coal milled to a top size of not greater than 0,2 mm. The mass of the analysis sample depends on the analysis required. Normally 60 to 150 g is sufficient but 30 g may be adequate in some cases. However a greater mass is required if certain caking tests are to be carried out.

The process of sample preparation involves several distinct operations, which may sometimes be preceded by drying :

a) a reduction in particle size by milling[1], sometimes referred to briefly as "size reduction" or "reduction";

b) mixing in order to achieve homogeneity;

c) a decrease in the mass of sample by dividing it into two or more parts, sometimes referred to briefly as "division".

Figure 6 shows the procedure.

Reduction must always precede division. The above operations constitute **one stage** of sample preparation, the completion of the stage being marked by the process of division.

Two kinds of error can occur in the course of sample preparation :

a) systematic errors;

b) errors of division.

Systematic errors cause a bias in the results which will normally always be of the same sign. Errors of division are random and may be sometimes positive, sometimes negative, larger or smaller.

Systematic errors are caused by extraneous material being added to the sample or, conversely, by some of the sample material, such as dust or moisture being lost; such errors can be avoided by care in handling and by operating according to instructions, for example, by the use of suitable equipment in suitable sample preparation rooms.

Errors of sample division arise because part of the sample is retained and the remainder is rejected; in general these errors will be increased the smaller the proportion of coal is retained, and vice versa. **In principle,** to reduce random errors, it is desirable at each stage of division to retain as large a mass of coal as possible but **in practice,** to reduce the quantity handled, it is desirable to retain as little as possible. The stages of sample preparation should therefore be selected in such a way as to give sufficiently small variance of sample preparation without having to retain too large a mass.

The amount of coal which should be retained after sample division depends upon the **maximum particle size** of the coal at which the division is performed, its ash content and the precision required for the sample preparation process.

It is preferable for all stages of sample preparation to be carried out mechanically.

9.1.1 *Two-stage preparation*

Two stages of sample preparation should be used (except as provided in 9.1.2). In the first stage the coal is reduced from its initial size to an intermediate size; in the second stage it is reduced from that size to an intermediate size; in the second stage it is reduced from that size to the final size of 0,2 mm required for analysis. Thus it is necessary to select only one intermediate size between the initial size and the final size of 0,2 mm. This intermediate size would normally be 10 mm or 3 mm.

1) Sometimes referred to as crushing or grinding.

In the first stage the gross sample should be reduced to the selected intermediate size, then divided to a reasonably small mass; in the second stage the sample should be reduced to 0,2 mm then divided to the final mass required for the general analysis sample.

It is essential that the mass retained at the intermediate size should be sufficiently large to ensure that the variance of sample preparation is as small as is required and the relevant masses for certain intermediate sizes are given in table 10. Two conditions are specified :

CONDITION A applies to cleaned coal containing less than 10 % of ash.

CONDITION B applies to all other conditions.

TABLE 10 – **Minimum mass of sample retained after division in two-stage preparation**

Upper size of coal* after reduction**	Minimum mass of sample retained	
	Condition A	Condition B
mm	kg	kg
10	1,5	10
3	0,3	2
1	0,15	0,6

* This is the nominal size, such that 99 % of the coal is undersize.

** Intermediate sizes are little used but if necessary may be obtained from this table by interpolation.

Condition A allows lower masses to be retained than Condition B; these lower masses may be used for particular coals if they have been shown by using the procedure of annex D to give a sample preparation variance which is sufficiently low.

The errors of sample division at a size of 0,2 mm are small and can be disregarded **provided that the sample has been adequately mixed.** Thus, a variation is permissible in the second stage. The sample may be reduced from the chosen intermediate size to 1 mm and divided to the appropriate mass given in table 10 ; it is then reduced to 0,2 mm, mixed and divided to the mass required for the laboratory sample.

An outline of the procedure is given in figure 6.

9.1.2 *Three-stage preparation*

Three-stage preparation should be used only if the initial size of the coal is 120 mm or greater. In this case two intermediate sizes should be selected from table 10. However, if the sample preparation is carried out in three stages the total errors will be correspondingly higher and will exceed the value assumed unless larger masses are taken at each stage. To a first approximation the variance is inversely proportional to the mass of sample taken at each stage, provided that the coal is uniform. Thus, the appropriate masses given in table 10 must be increased by approximately 50 % (see table 11).

When a common sample has been collected, the precaution given in 8.2 should be carefully observed.

TABLE 11 – **Minimum mass of sample retained after division in the three-stage preparation**

Upper size of coal* after reduction**	Minimum mass of sample retained	
	Condition A	Condition B
mm	kg	kg
10	2,5	15
3	0,45	3
1	0,25	1

* This is the nominal size, such that 99 % of the coal is undersize.

** Intermediate sizes are little used but if necessary may be obtained from this table by interpolation.

9.2 Procedure

9.2.1 *Arrangement of scheme*

As indicated in 9.1.1, sample preparation is normally carried out in two stages, each consisting of drying (if necessary), size reduction, mixing (if necessary) and sample division.

Choose the intermediate size which is to be used for division at the end of the first stage, this being preferably the size of 3 mm.

In the second stage, two alternatives are permitted, of which the first is preferred.

a) reduce the sample to 0,2 mm in size and then divide it to the mass required for the laboratory sample.

or alternatively,

b) reduce the sample to 1 mm, then divide it to the appropriate mass in table 10. Then reduce it to 0,2 mm in size, mix thoroughly[1)] and divide it to the mass required for the laboratory sample.

The possible schemes of sample preparation are shown in figure 6.

9.2.2 *Precautions*

The place set apart for the treatment of samples should be enclosed, roofed over and free from draughts. Samples should be treated as soon as possible after being taken. If they cannot be treated immediately, they should be stored in a place where the atmospheric conditions are not markedly different from those in the sample preparation

1) Mixing can be omitted if a mechanical divider is used, but is always preferable if a lower variance of sample division is desired.

room. All surfaces with which the sample comes into contact should be clean and made of material which will not contaminate the sample. Precautions against loss are important, especially in the handling of fine coal, which is easily blown away. Equipment for extraction of dust in suspension in the room may be sometimes necessary to maintain a satisfactory standard of health and cleanliness, particularly when workers are subject to continued exposure.

9.2.3 *Extraction of moisture sample*

If the moisture sample is to be extracted from the common sample, the methods described in 8.4.1 should be used to obtain the moisture sample before or during the preparation of the ash sample.

9.2.4 *Drying*

If at any stage of the procedure the coal seems too moist to pass freely through the equipment, the sample should be dried as described in 9.3.1. If drying can be avoided in the first stage of the sample preparation, the procedure is simplified.

9.2.5 *First stage of sample preparation*

The whole of the gross sample should be passed through the first mill. It may be necessary to use a stamp or maul to break up the large lumps before feeding the coal into the mill, but no other form of hand reduction is permitted (see 9.3.2).

Care should be taken to ensure that the coal is sufficiently dry to pass through the milling and dividing equipment used (see 9.2.4). The equipment should be conditioned with a part of the sample used, to avoid loss of moisture.

If possible, the coal should be milled to pass 3 mm in the first stage in order to reduce the mass of sample retained for the next stage as well as to minimize errors due to sample division.

The mass of sample for coal of size greater than 10 mm is so large that a reduction to a size larger than 10 mm should only be used in special circumstances.

After milling, the sample should be divided as described in 9.3.4 by a suitable sample divider, to the mass corresponding to the selected size as given in table 10.

9.2.6 *Second stage of sample preparation*

In the second stage of sample preparation, the sample is reduced in particle size as described in 9.3.2 to 1 mm or 0,2 mm.

After reduction, the sample should be divided to the required mass of 60 g or 150 g or more as described in 9.3.4 either by a mechanical sample divider or by mixing and then dividing, using a riffle for both operations.

9.2.7 *Final preparation of the ash sample*

The sample received in the laboratory should be reduced as described in 9.3.2 so that it passes a 0,2 mm sieve.[1)]

The sample should then be placed in an airtight container, which is then sealed. A label should be attached to the container giving full details of the original sample and its subsequent method of treatment.

9.3 Methods

9.3.1 *Methods of drying*

The purpose of drying the ash sample is to ensure that it will pass through the mills and sample dividing equipment freely and without loss or contamination.

Drying may be carried out at any stage of the sample preparation procedure at which it may be found to be necessary. Care should be taken that samples are not oxidized during drying, or exposed to the action of warm or overheated mills during reduction. The sample should be dried until it is visibly dry. The temperatures and times of drying in table 12 should be sufficient to dry the sample but, if necessary, a longer drying time may be used.

TABLE 12 — **Drying times**

Temperature	**Time** hours
15 °C above ambient but not exceeding 25 °C	preferably not to exceed 24
30 °C	6
45 °C*	3
105 °C* (high rank coals only**)	1

* If caking or swelling tests are to be carried out on the coal sample, drying should not be carried out at a temperature above 30 °C, unless it has been established by experience of the coal in question that the drying procedure used does not significantly affect the results of the tests.

** For the purposes of this International Standard, high rank coals are defined as those included in clauses 0 to 5 inclusive of the International Classification of Hard Coals by Type, adopted by the United Nations Economic Commission for Europe.

In order to achieve these temperatures, a cabinet or oven may be used and the rate of air change should then be not less than once per minute. The samples should be spread out in a uniform layer, to give a concentration not exceeding 1 g/cm^2.

1) The sample should be milled so that at least 99 % passes a 0,2 mm sieve (see 9.3.5).

If a common sample is taken for ash and moisture, the drying then forms part of the determination of total moisture; the method of air-drying in this case is specified in 8.4.2, together with details of the necessary calculation.

If a closed mill preventing the loss of moisture is used there is no need to dry the sample during the first stage of sample preparation.

If drying is carried out during the second stage there is no need to take into account any loss of mass, as the moisture sample will have been removed previously.

9.3.2 *Methods of particle size reduction*

The particle size should be reduced mechanically, using the mills described in A.6.1 of annex A. In the second stage of preparation, power-operated mills should be used and high speed mills are preferable.

The precautions necessary with high speed mills are given in A.6.1 of annex A.

Samples which are intended for analysis should not be sieved to remove the oversize for re-crushing. This is a bad practice since the material which is difficult to crush is usually shale and it cannot be satisfactorily mixed back with the milled coal.

As mentioned above, errors of sample division are greatly increased by the presence of coarse material. Accordingly, it is essential that the 99 % pass level should be used for checking mills (see 9.3.5).

The mills should be thoroughly cleaned at regular intervals and should be cleaned each time a different kind of coal is to be milled. The use of a stream of compressed air or an industrial type of vacuum cleaner is suitable for this purpose.

Hand reduction with stamps or mauls should only be used to break oversize particles to the maximum feed of the mill in the sample preparation room, or in exceptional circumstances (for example, in field work, though preferably portable mills should be used).

9.3.3 *Methods of mixing*

The errors of sample division can be considerably reduced by a thorough mixing of the sample prior to division. Mixing can be carried out by one of the following methods :

a) passing the sample through a riffle three times in succession and re-uniting the two portions after each riffling;

b) the use of mechanical mixers (applicable to the second stage of sample division);

c) in a rotary divider, a mixing pass can be obtained by removing the segmental receivers and substituting a cylindrical container. In general, however, mechanical sample dividers mix the sample adequately and no additional mixing pass is necessary.

Hand methods of mixing are not recommended. In particular, methods which involve forming the sample into a conical pile usually lead to segregation rather than mixing. The hand mixing of fine material less than 2 mm in top size is particularly difficult, as fine, dry material tends to segregate and to be lost as dust.

9.3.4 *Methods of sample division*

Sample division is the process whereby a sample of required mass is extracted from a larger sample of coal without change in particle size. The division may be carried out either by a mechanical sample divider or a riffle. In both cases the samples are effectively collected by taking a large number of small increments.

Division by coning and quartering is not recommended.

Each stage of sample division may be carried out either in two or more passes (for example when using a riffle, three passes are required to divide a sample to a one-eight fraction), or in a single pass (for example when using a rotary sample divider, a one-twenty-fourth fraction may be taken in one pass). If the total capacity of the receiver of the sample divider is less than the mass of the gross sample, each pass will have to be carried out in a series of steps. Thus a pass is defined as the passage of all the sample once through a sample divider and a step as the passage of part of the material once through the sample divider.

Coal which is visibly wet may not run freely, or may tend to adhere to the sides or containers of sample dividers or riffles. In such circumstances, it may be necessary, before sample dividing, to dry the sample as described in 9.3.1.

a) DIVISION BY MECHANICAL SAMPLE DIVIDER

The main advantage of mechanical sample dividers is that they extract a part of the coal by a large number of increments. If less than about fifty increments are taken by the sample divider, the accuracy may be substantially reduced, so that the part to be collected should not be too small a fraction of the whole. In such circumstances it may be preferable to carry out the division in two passes.

b) DIVISION BY RIFFLE

With a riffle the sample is divided into two parts of approximately the same mass. One part is used for further division, the other part may be rejected. Subdivision to any required mass can be carried out by successive passes.

The coal should be fed steadily into the riffle from a sample container, ensuring that the coal is evenly distributed over all the slots. The coal should be allowed to fall freely, i.e. not towards one side of the riffle, and the rate of feed should be controlled so that the slots are never choked. When a stage of sample dividing requires two or more steps or passes, the sub-sample retained at each step or pass should be taken alternately from each side of the riffle.

Care should be taken that loss of moisture does not occur from a moisture sample when division is carried out by a riffle. Closed riffles should be used for dividing moisture samples, or for dry coals to prevent loss of dust.

9.3.5 *Methods of checking mills*

It is essential to confirm that the mills are milling the coal sufficiently finely. Accordingly, the mills in use should be tested regularly, say once a week. This is best done by passing through the mill a sample of the "most difficult" coal which is handled at the laboratory. The coal should be treated by the normal procedure and the milled coal should then be sieved over a 0,2 mm sieve to confirm that 99 % by mass will pass. If it will not, the mill should be adjusted or repaired, as necessary, until the operation is satisfactorily performed.

Coal which is used for testing in this way should not be part of a normal sample which is to be used for analysis.

9.4 Equipment

Equipment is required for reducing particle size, mixing and dividing samples, viz., mills, mixers, mechanical sample dividers and riffles. Descriptions and essential details are given in annex A.

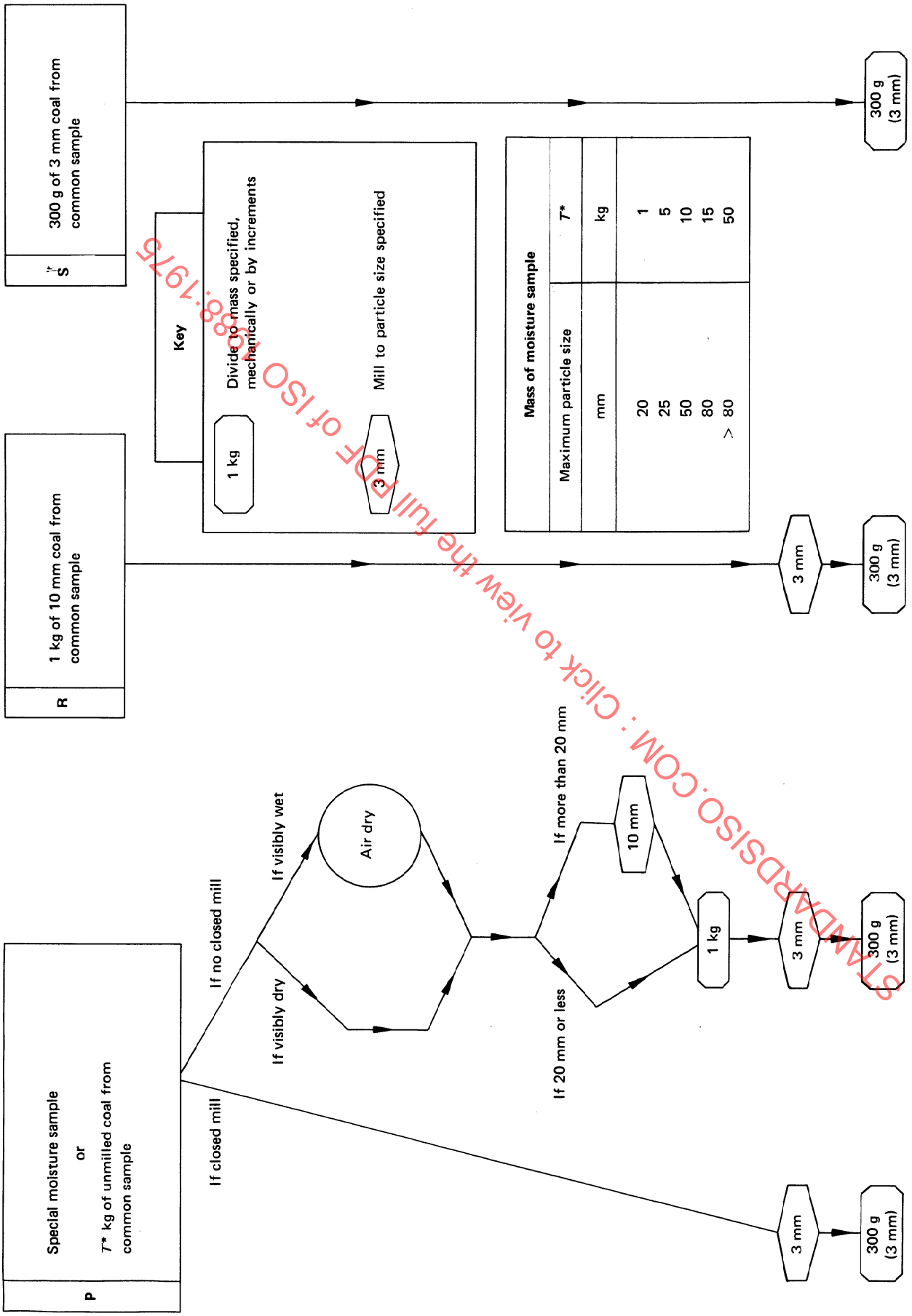

FIGURE 3 – Determination of moisture by method A or B

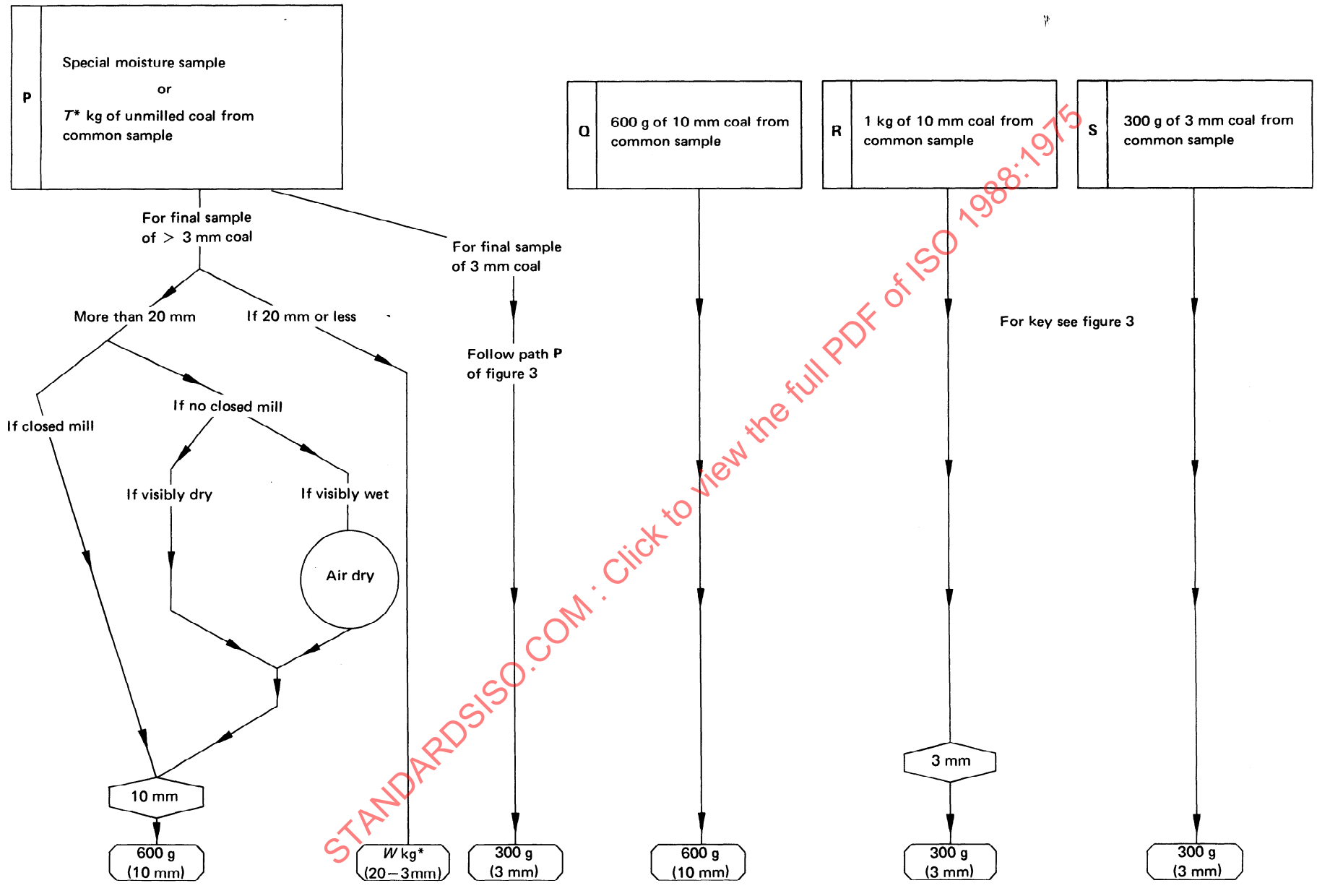

FIGURE 4 — **Determination of moisture by method C (Applicable only to coals not susceptible to oxidation)**

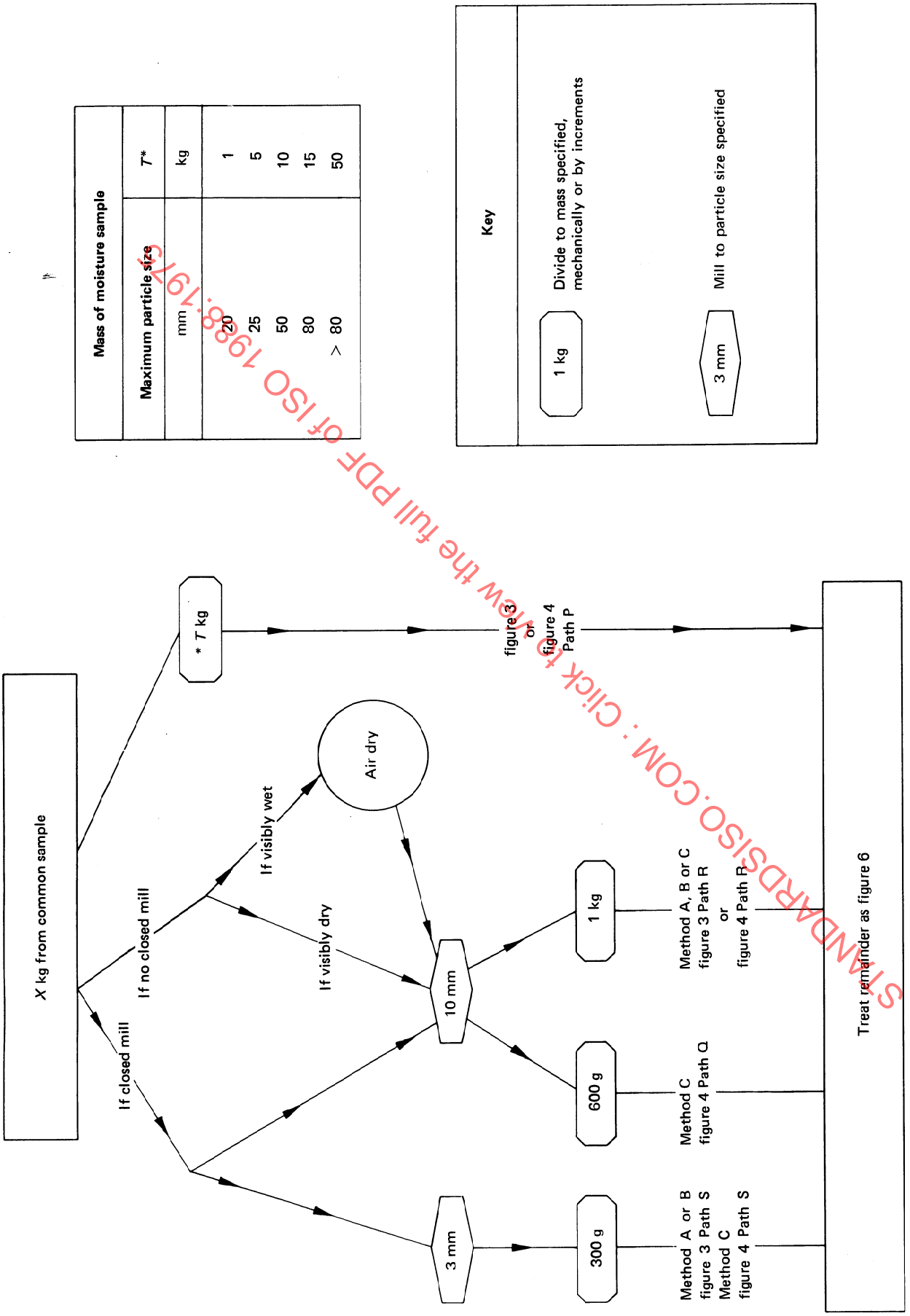

FIGURE 5 – Treatment of common sample

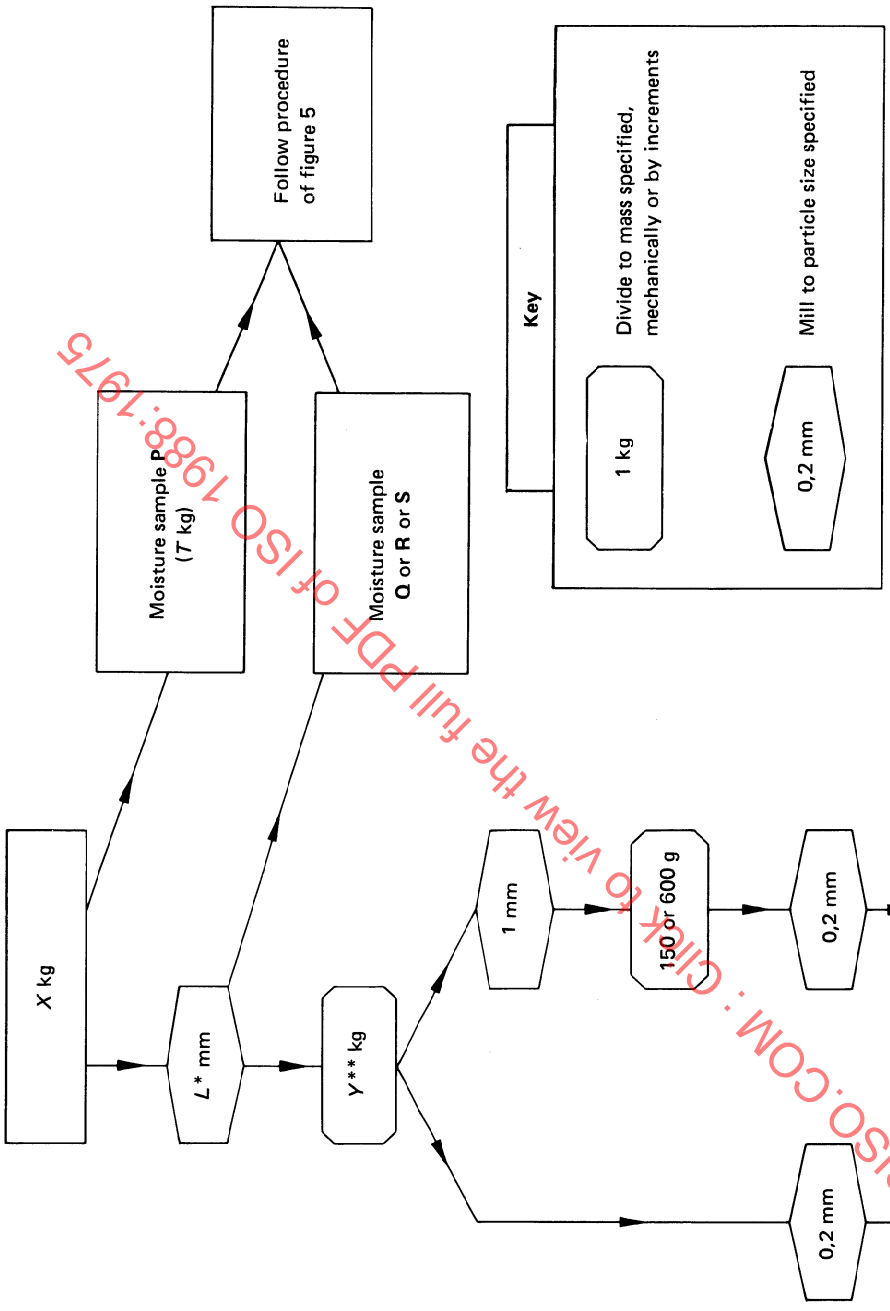

FIGURE 6 – **Preparation of general analysis sample**

Mass of moisture sample		
Maximum particle size		*T*
mm		kg
20		1
25		5
50		10
80		15
> 80		50

*L** mm	*Y*** kg	
	Condition A	Condition B
10	1,5	10
3	0,3	2
1	0,15	0,6

ANNEX A

SAMPLING EQUIPMENT

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A.1 SCOPE

This annex describes typical examples of equipment used in the collection of coal samples, their transport and their preparation for analysis.

A.2 INTRODUCTION

The annex is divided into clauses, each describing in general terms one type of equipment; schematic drawings are also provided to illustrate the principles involved.

A.3 GENERAL REQUIREMENTS

A.3.1 Sampling

Depending on the conditions under which the coal samples are collected, sampling equipment is required for the following purposes :

a) collection of samples from a falling stream;

b) collection of samples from conveyor belts;

c) collection of samples from coal loaded in railway wagons or other means of transport, or in stockpiles.

Composite equipment consists of appropriate sample collecting and sample preparation machines combined into a single unit by means of which, in a continuous chain of operations, coal increments are collected and prepared into samples.

The sampling equipment used must be such that the sample obtained is representative : it must also fulfil the following requirements :

a) the capacity of increment-collecting vessels must be such that during the collection of a single increment they are not filled to more than three-quarters of their capacity;

b) the length of the collecting vessel and of the traversing equipment must be such as to ensure that the whole width of the coal stream is traversed;

c) the width of the inlet aperture of the increment-collecting equipment must not be less than 2,5 times the top size of the coal, or about 30 mm, whichever is the larger;

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

e) none of the coal collected for the sample must be lost during the removal of the sample;

f) the sampling equipment must not become clogged with moist coal while the sample is collected.

A.3.2 Sample preparation

Sample preparation machines are required :

a) for preparing laboratory samples from gross samples;

b) for preparing analysis samples from laboratory samples;

c) for preparing analysis samples from gross samples.

The performance of machines for the reduction and division of samples must be such that the samples remain representative, i.e. that mass or moisture is not lost and that the sample does not become contaminated.

Sample preparation machines should ensure the following :

a) when laboratory samples are prepared from larger samples : a particle size reduction to less than 3 mm and a division of the sample into the required number of laboratory samples of the required mass;

b) when analysis samples are prepared from laboratory samples : a particle size reduction to less than 0,2 mm and the production of the required number of analytical samples of the required mass;

c) when analysis samples are prepared from gross samples : a particle size reduction to less than 0,2 mm and the production of the required number of analytical samples of the required mass.

A.4 HAND EQUIPMENT FOR THE COLLECTION OF INCREMENTS

This clause includes tools for sampling by hand, but not those which require a source of power for their operation.

A.4.1 Augers (see figure 7)

Augers are used for sampling washed smalls up to 25 mm in size in wagons and stockpiles. An auger consists of two fixed curved blades mounted on a cross-piece attached to a steel or aluminium tubular stem 1 m long. The two blades, which together form part of a cone, fork and interlace at the leading end; the auger is inserted into the bed of coal with a screwing motion.

A.4.2 Ladles[1] (see figure 8)

A suitable design of ladle for use with falling streams is shown diagrammatically. The ladle opening should be at least 2,5 times the top size of the coal to be sampled; ladles are not suitable for sampling moving coal larger than about 80 mm in size.

A.4.3 Scoops (see figure 9)

Suitable designs of scoops for use on conveyors or with stationary coal are shown diagrammatically. The scoop opening should be at least 2,5 times the top size of the coal to be sampled; scoops are not suitable for sampling moving coal larger than about 80 mm in size.

1) A ladle is a container with an open **top**; a scoop is a container with an open **side**.

A.4.4 Test ring

A ring of 150 mm diameter fitted with a handle, to enable the sampler to determine the proportion of lumps over this size when sampling large coal.

A.4.5 Sampling frame (see figure 10)

The device is an aid to sampling on a stopped belt. The frame should be inserted into the coal until it is in contact with the belt across its full width and the increments collected by sweeping off the whole of the coal lying between the sides of the frame.

Any large lumps bisected by the frame on the left hand side are pushed into the sample and those on the right hand side out of the sample, or conversely.

The distance between the sides of the frame should be at least 2,5 times the maximum particle size. The height of the device must be greater than the thickness of the layer on the belt.

A.4.6 Probes (see figures 11 a) to 11 d)

Probes are used for sampling coal of small particle size – up to 25 mm at present – over the whole depth of a pile without the need to disturb the pile; for example, a loaded wagon can be sampled in this way without it being necessary to unload the wagon. They take the form of cylindrical tubes which are inserted vertically or at a slight angle into the coal until they touch the bottom of the pile.

When using this equipment, the insertion into the coal is sometimes very difficult and the tube tends to empty when it is withdrawn.

The internal diameter of the device should be at least 2,5 d, where d is the largest dimension of the coal being sampled. On opening the probe the sample can be analysed layer by layer, thus obtaining its composition and giving a complete record of the heterogeneity of the coal.

For the sampling of dry dust the same types of probe can be used but should be equipped with a device to permit the injection of a jet of water at the base of the probe before withdrawing it.

Four designs are described :

a) The probe consists of two half tubes, made of cold drawn steel plate, which are designed in such a way as to slide together to form a closed cylinder. Such probes can be made in various lengths up to 3,5 m; long probes are easiest to use for coal up to 15 mm.

b) The probe consists of a cylindrical tube which is slightly tapered and is slit along an axis in such a way that the taper tends to grip the material contained in the tube. By means of a handle it is possible to give a rotary movement to the tube in order that it may be inserted. The tube may be awkward to empty and clean.

c) The probe consists of a channel, the two edges of which have grooves, and a plate which can slide along the grooves. The channel is inserted open, the plate is slid along the grooves and the complete apparatus is withdrawn.

d) The probe is a tube of circular cross-section, equipped with apertures as shown in the diagram. This probe is pushed down into the coal, then twisted round so that the action of twisting causes the probe to fill itself. There is a danger that the probe may form an oval hole during the twisting; this can be prevented by the use of a rod of wood which completely fills the probe and seals the apertures; when the probe has reached the bottom of the coal, the rod is withdrawn as the probe is twisted, in such a way that it maintains contact between the apertures and the coal, and the probe is gradually filled. In this design the sample will not necessarily be suitable for the detection of heterogenity.

A.5 POWERED EQUIPMENT FOR THE COLLECTION OF INCREMENTS

Various types of power operated automatic samplers are available commercially and this clause describes only those which illustrate different general principles. Automatic samplers may be provided with a secondary crushing and reduction system to reduce the mass of the large increments normally taken. It is important that samples should be stored in closed containers.

All mechanical or automatic sampling equipment should be checked by comparing the samples obtained with samples taken manually by skilled personnel, to ensure freedom from bias.

The types are classified according to the sampling system to which they relate.

Machines for sampling from falling streams should have a bucket of width equal to the full width of the stream and cutting through it in a direction either parallel to or at right angles to the movement of the belt. It is essential that the box passes through the stream at uniform speed and that the slot width should be properly related to the size of coal. In addition, this type of sampler usually passes through the stream twice to collect an increment (from front to back and reverse) and the container must be of such a size that it is not full at the end of its travel. The change in direction at the back of the stream must take place where there is no falling coal. The bucket usually tips at the end of each double movement and the sample is emptied down a chute. Alternatively, the bucket may fill on one movement and then tip, or close for its return.

A.5.1 Falling stream – Breeches chute (see figure 12)

This device takes increments from a falling stream of coal by diverting the whole stream into one or the other leg of a breeches chute. Movement of the flap governing the direction of flow is usually time-controlled and the proportionality of the mass of sample to the mass of consignment depends on the period during which the flap is allowed to remain in its alternative positions. If the flow rate is erratic it may be necessary to control the device according to the quantity conveyed (for example by means of a belt weigher).

The flap must move with a rapid "snap" action so that the time taken for the flap to traverse the stream is negligible compared with the time for which the whole stream is diverted. Such action can be satisfactorily obtained by compressed air operation, or for small-sized coal by electromagnetic operation.

A.5.2 Falling stream — Slotted vessel (see figure 13)

A vessel with an aperture in the form of a rectangular slot is drawn through a falling stream of the material to be sampled, usually with the slot at right angles to the direction of the conveyor. The slotted vessel traverses the whole width of the stream and a continuous "ribbon" of the material is collected. The width of the slot must be at least 2,5 times the upper size of the coal, otherwise bridging will occur; the device is not therefore usually employed for coals larger than 20 mm.

The mass of increment collected depends on the quantity conveyed per unit time and the rate of passage of the slotted vessel through the falling stream. The volume of the vessel must be such that it is not overfilled when the layer of coal on the belt is at its maximum.

A.5.3 Falling stream — Swinging arm (see figures 14 a) and 14 b))

Swinging arm samplers are designed to sample at the discharge end of a conveyor and are suitable for positions where the vertical height or headroom is limited.

a) In this arrangement a heavy bucket is allowed to swing once, like a pendulum, through the full width of the stream of material. The increment collected is discharged into a sample hopper. Power for the operation of the sampler is sometimes derived from the drum of the conveyor and the device may be set to operate automatically either at equal intervals of time or, if a belt weighing machine is incorporated, after specified masses of coal have passed.

b) A further type of swinging arm sampler consists of a rectangular frame with a number of holes of sufficient dimension drilled through the top surface. The arm swings across the full width of the stream and the coal passing through the holes falls on to a small conveyor carried within the frame. This belt discharges into a chute or storage hopper.

A.5.4 Falling stream — Ram operated cart (see figure 15)

This device is intended for taking a sample from a falling stream. The sample collecting device is a moving cart of the full width of the stream, with a removable bottom. It is propelled by a ram and in its idle position it is clear of the falling stream at the top. When the ram operates, the cart is pushed through the stream from the front to the back with the bottom open. The bottom is then closed and the cart is withdrawn by the ram so that it passes through the falling stream and collects the sample. When it has reached the "home position" at the end of "up" stroke, the bottom is automatically opened so that its contents are dumped into a hopper. From this hopper the coal is fed to a crusher and then to a feeder from which a second sample is collected by a rotating nozzle, the remainder of the coal being discharged. The coal can then be fed down further chutes past further dividers until the final laboratory sample is prepared. The ram can be operated by pneumatic, hydraulic or mechanical power. The unit can be installed at the head of a conveyor without major alterations.

A.5.5 Moving belt — Scraper arm (see figure 16)

The scraper arm operates by sweeping off increments across the width of a conveyor belt. Since the motion of the conveyor causes fines to be segregated to the bottom of the material on the belt, it is essential to have the sampler properly adjusted to the belt curvature across its width, to avoid taking a biased sample. The shape of the scraper is also important since in sweeping through the stream of coal it creates a "bow-wave" in front of it; the tendency is to push aside the large coal pieces and thus reject them. The speed must also be carefully adjusted.

When properly adjusted the scraper arm is satisfactory for coal up to 50 mm in size, the mass of increment depending on the load on the belt and the width of the scraper. The device may be set to operate automatically either at equal intervals of time or, if a belt weigher is incorporated, after specified masses of coal have passed.

A.5.6 Bucket elevator — Box car or drawer (see figure 17)

This device is suitable for positioning at the discharging point of a bucket elevator and consists of a vessel for holding the contents of at least one whole bucket or sometimes of two buckets. The device is not sensitive to segregation and is useful for all sizes of coal, but its efficient operation and the accuracy of the sample depends on uniform filling of the individual buckets. It should be set to function at equal intervals of time or after the passage of a specified number of buckets. A deflecting plate directs the contents of the selected buckets into the car.

A box car may also be used in connection with a breeches chute.

A.6 EQUIPMENT FOR SAMPLE PREPARATION

Sample preparation equipment (excluding that for drying wet samples) may be divided into three classes :

a) size reduction apparatus;

b) sample mixing apparatus;

c) sample dividing apparatus.

A.6.1 Size reduction apparatus

The equipment for reducing particle size should be mechanical and such machines are called **mills** for the purposes of this International Standard. High speed mills are desirable to ensure a product of specified size even from hard coals of high ash content, or coals with shale.

Reduction mills vary in type from jaw crushers to roll crushers and from plate mills to high speed impact pulverisers, which are particularly suitable for the finer stages of milling (to 0,2 mm).

All types of mill should be made of heat-resistant material. Those parts of mills coming into contact with coal should be of wear-resistant material in order to prevent contamination of the samples and to avoid overheating, which would induce oxidation of the coal or loss of moisture from it. Mills should be easy to clean.

High speed mills tend to become heated and samples must not be allowed to remain in them long enough to be affected. If a mill is used for a series of samples, it should be allowed to cool between samples in order to avoid oxidation and loss of moisture with subsequent samples.

Modern high speed mills can be seriously damaged by the presence of extraneous iron or other ferrous material in the sample. A magnetic separator placed on the chute leading to the machine is a convenient safeguard against this.

The efficiency of mills used for grinding to 0,2 mm should ensure that at least 99 % of the sample will pass this size.

Although high speed pulverisers are the most efficient with a wide range of coals, they entrain air with the samples due to the fan-like effect of the rotating hammer; to avoid the loss of fine material or dust, it is essential either to limit the air flowing through the mill by using closed inlet and outlet hoppers or by fitting a breather bag to the mill outlet.

A new type of totally enclosed slow speed conical mill is of particular interest in that it crushes the sample between a tungsten carbide rotating cone and a fixed jacket and is superimposed on a dust-tight rotary sample divider (see A.6.3). An advantage is that the product is fairly uniform in particle size and remarkably free from fines.

The mills should be cleaned at regular intervals and always before use on a different coal; the use of a stream of compressed air or an industrial-type vacuum cleaner may be found suitable for this purpose.

A.6.2 Sample mixing apparatus

The apparatus should not cause breakage of the coal nor produce dust nor allow loss of moisture. It should be conditioned with the coal being used or with another coal of similar moisture content.

Three types of mixer have been found satisfactory — the paddle mixer, the drum mixer and the double cone mixer.

A.6.2.1 *Double cone mixer for coal of 1,0 — 0,2 mm size* (see figure 18)

A double cone mixer is suitable for mixing the residue from the first stage of sample division. It has a short cylindrical portion, an inclined baffle plate and a closure plate at each end secured by a wing nut. An axle passes through the centre of the mixer and rotates in bearings mounted on a suitable support. The mixer is rotated by hand at about 60 rev/min.

For quantities up to 0,25 kg, mixing for 1 min is sufficient; for larger quantities, mixing for 4 min is required.

A.6.2.2 *Riffle*

A riffle may also be used for mixing samples (see A.6.3.1).

A.6.3 Sample dividing apparatus

Sample division is accomplished either by riffles or by power driven rotary dividers. The latter have the advantage that they take a very large number of small increments so that it is virtually unnecessary to mix the sample except before taking the analysis sample; as a precautionary measure mixing is carried out before the final stage of division for a high ash coal, or when very low errors of sample division are desirable.

Dividers should be conditioned with the coal being used or with another coal of similar moisture content.

A.6.3.1 *Riffles* (see figure 19)

A riffle is a non-mechanical sample divider by the use of which the coal fed onto it is divided into two halves, one being retained and the other rejected, for example by allowing the coal to fall through a set of parallel slots of uniform width.

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

A riffle should be symmetrical (so that a part-sample may be taken from either side). All surfaces on which coal might rest should be inclined at not more than 30° to the vertical. The receivers should fit closely against the body of the riffle, to minimize loss of dust. It is essential to use a riffle which is appropriate to the upper size of the coal to be divided as serious errors may be introduced if the slots in the riffle are either too large or too small, or if the number of slots on each size is insufficient to give an adequate number of divisions for each half of the sample. In general, the slot width should be about 2,5 times the upper size of the coal; a suitable slot width for an upper size of 10 mm is 25 mm and for an upper size of 3 mm is 10 mm. The number of slots for each half of the riffle should be at least eight and preferably more. Effective mixing of the sample is obtained by passing the whole of the coal through the riffle three times before sub-dividing.

A.6.3.2 *Mechanical sample dividers*

Most mechanical sample dividers are of the rotary type, in which a receiver is placed on a turntable so as to intercept a falling stream of coal once or twice in each revolution or, alternatively, to collect a continuous ribbon from a falling stream of coal produced by allowing the coal to fall from the hopper onto the apex of a cone. The slot or dimension where the coal passes from the reservoir should be at least 2,5 times the upper particle size.

Other mechanical dividers may be used, provided that the sample is effectively collected by a large number of increments and that bias does not occur.

a) DOUBLE CONE SAMPLE DIVIDER (see figure 20 a))

A stream of coal from a hopper is allowed to fall onto a rotating cone; one or two slots in the cone allow the stream to fall directly into a sample receiver for a part of each revolution. Within limits the width of the slots can be varied so that the proportion of sample retained can be adjusted between about 1/5 and 1/10, but the opening should always be at least 2,5 times the upper particle size of the coal. The divider is rotated at about 60 rev/min. There are models suitable for coals up to 10 mm in size.

b) ROTARY SAMPLE DIVIDER (see figure 20 b))

The rotary sample divider consists of a number (for example twelve) of containers shaped like segments mounted on a rotating turntable, revolving at about 60 rev/min. The bottom edge of the input reservoir must be large enough to prevent coal bridging the outlet. The minimum size of the opening at the point where it intercepts the coal must be at least 2,5 times the upper particle size of the coal. Thus, a number of separate increments are collected in each revolution and the proportion of sample retained may be chosen by taking the relevant number of containers; for example if there are twelve segments, one container is retained for 1/12 fraction, two containers for 1/6 fraction and so on.

There are models suitable for coals up to 1,5 mm in size, 6 mm in size or 10 mm in size, while a smaller version can be used to divide samples of 0,2 mm in size.

A.7 GENERAL EQUIPMENT

A.7.1 Weighing machines

Any type of weighing machine which will weigh to a precision of 0,1 % of its maximum load is satisfactory provided that the maximum load is not much greater than the amount usually being weighed. Platform scales with dial readings are recommended. For large samples a scale of 100 kg capacity reading to 100 g is suitable; for samples of medium mass a scale of 25 kg reading to 25 g; for small samples a scale of 10 kg reading to 10 g.

A.7.2 Air-drying cabinet (see figure 21)

The principles of the air-drying cabinet are shown in the figure; a stream of air is passed over a number of trays on which the coal is spread out in thin layers. The inlet air may be heated, in which case there should be thermostatic control of the temperature.

A.7.3 Sample containers (see figure 22)

Containers used for moisture or common samples should be water-tight and made of impermeable non-corrodible material, of adequate strength with well fitting lids. Containers of metal or of plastics material have been found suitable. Lids incorporating a rubber gasket and tightened down to the body of the container by means of levers or screws are recommended.

A.8 COMPLETE SAMPLING EQUIPMENT

This clause gives details of some possible arrangements for complete sampling equipment by which samples of coal can be taken and sub-divided to give a sample for analysis. Size reduction may also be included to these arrangements.

A.8.1 Falling stream — Moving shutter (see figure 23)

This arrangement incorporates a complete sampling apparatus, with equipment for sample reduction and division. The coal usually flows down an inclined chute fitted with an automatic shutter over a trap-door which can divert the coal down a vertical chute. This shutter is periodically (about every 7 min) opened for about 4 to 5 s by a cylinder of compressed air. The increment is then conveyed on a sampling belt at a speed of 80 mm/s over a magnetic drum where extraneous iron can be removed. An automatic shutter subsequently diverts a part of this coal into another chute at the base of which there is a grinder. The sample passes through the latter and is then divided by passing over a grating, a certain proportion of the sample being collected in the ground condition in a collecting device. Alternatively, by a manual shutter the coal can be fed into the sample collecting jars in the uncrushed conditions.

Arrangements are made, as will be seen in the figure, for unwanted material and for scrapings off the sample belt to be returned to the original circuit.

A.8.2 Falling stream (small coal) — Moving orifice (see figure 24)

This device incorporates a moving box with a slit which can traverse the discharge of fine coal by means of a trolley. It moves across the stream at pre-arranged intervals of 15 min and takes a section across the discharge.

This is fed into a box, the contents of which are discharged through a tapered tube. Under this tapered tube can be positioned a pair of rotating collecting pipes so that a fraction of the sample is taken into the sample container while the collecting pipes are under the tapered tube. The residue is returned to the main stream.

This equipment cannot be used for wet coal, including slime.

A.8.3 Falling stream — Swinging flap (see figure 25)

This type of equipment may be used for the collection of increments from a falling stream of coal of up to 50 mm size and for preparing the gross sample into the required number of laboratory samples weighing 0,5 kg each. The particle size of the laboratory samples is less than 3 mm.

The equipment consists of a flap (3) with an increment collector which can be swung across the stream, a mechanism (2) by means of which the collecting device is operated, a hopper for accumulating the collected increments, a hammer mill (8) for reducing the original particle size of the gross sample to 3 mm maximum and a two-stage divider for reducing the mass of the ground sample. If required a worm feed is installed between the hopper (7) and the mill (8).

The collecting device (3) periodically traverses the whole width of the falling stream of coal and dumps the collected increments into the chute (4) through which the coal falls into the hopper (7).

The gross sample is ground to 3 mm in the mill (8) and prepared so as to obtain one to four laboratory samples in the two-stage dividers.

A.8.4 Falling stream — Moving bucket (see figure 26)

This type of sample collecting machine is intended for the collection of coal samples from a falling stream.

The machine consists of an electric motor (4), reducing gear (8), dividing sprocket (7), idling sprocket (6), two endless chains (2), bucket (1), limit switch (5), and the frame (3) on which all the components of the equipment are mounted.

This machine will deal with coal up to 200 mm in size with a moisture content of up to 8 %, in a stream of capacity up to 500 tonnes per hour.

The machine is about to 4 to 5 ml long and about 1 to 2 m wide.

A.8.5 Falling stream — Slotted vessel (see figure 27)

This type of sample collecting machine is intended for the collection of coal samples directly from conveyor belts.

The equipment consists of an electric motor (5), reducing gear (6), driving sprocket (4), idling sprocket (2), scraper-type collecting vessel (3), limit switch (8), a hopper (9) in which the increments are collected and a frame (7) on which the components of the equipment are mounted.

The equipment will deal with coal up to 200 mm in size with a moisture content of up to 14 %, in a stream with capacity up to 450 tonnes per hour. The width of the conveyor belt is between 0,8 and 1,0 m and the apparatus is about 1,2 m long and 2,4 m wide.

A.8.6 Wagons — Sampling tubes (see figure 28)

The automatic wagon sampler consists of one or more steel pipes mounted on the side of a dumper cradle (or side tippler) on the side which tips downwards.

The tubes extend a certain distance above the side of the wagon and the section extending above the cradle slopes towards the centre of the wagon in the cradle. As the wagon of coal is tipped, the coal flows over the sampling tubes and a portion of it flows into openings in the sloping part of the tubes shown in figure 28. When the cradle is tilted back to its normal position the coal flows down the tube into a container from which it may be discharged or fed into crushing and reduction apparatus.

The section of the wagon from which the sample is taken is determined by the angle of the tube, the location of the openings in the tube relative to the top of the wagon and the flow characteristics of the coal being dumped.

A.9 COMBINED REDUCTION AND DIVISION APPARATUS

A.9.1 Machine for preparation of 3 mm samples (see figure 29)

This machine is intended for the preparation of coal samples by reducing their particle size to 3 mm and by dividing them into the required number of laboratory samples of the required mass.

The machine consists of a hopper (2) for collecting the sample, a belt-type feeder (1), a hammer mill (3), a bucket-type drum divider (4) and a riffle (6) for producing two laboratory samples. There is also a worm conveyor (5) for removing the unwanted portion of the original sample, and the necessary electrical equipment. The apparatus will deal with coal of up to 150 mm in size with a moisture content of up to 12 % and will give a final sample of particle size 3 mm. The mass of the laboratory sample obtained is not less than 500 g and the mill will deal with up to 2 tonnes of coal per hour. The overall size of the machine is about 3 m long and 3 m wide.

A.9.2 Machine for the preparation of 0,2 mm samples (see figure 30)

This machine is intended for the preparation of samples from coal of a size up to 150 mm and for automatic determination of the moisture content of the sample.

The machine consists of a hammer mill (3), a divider (2), a drying chamber (4) for drying the laboratory sample, a hammer mill (5) for reducing the particle size of the laboratory samples to the size required for the analytical samples, and a divider for the analytical samples (6).

The sample preparation procedure involves the following consecutive operations; grinding the sample to a size of 3 mm, reduction of this product to a mass between 0,5 and 0,85 kg, drying the reduced sample of particle size 3 mm to an air-dry condition and grinding the 3 mm sample to the laboratory sample of particle size 0,2 mm. It is possible for this machine to be fitted with a device for the automatic determination and recording of the moisture content of the milled sample by electrical means.

This equipment will deal with coal up to 150 mm in size with a moisture content up to 15 %; a sample weighing 150 kg can be completely dealt with in 25 min. The operation of the mechanism is intermittent as and when the sample collects. The machine is about 2 m long and 1,5 m wide.

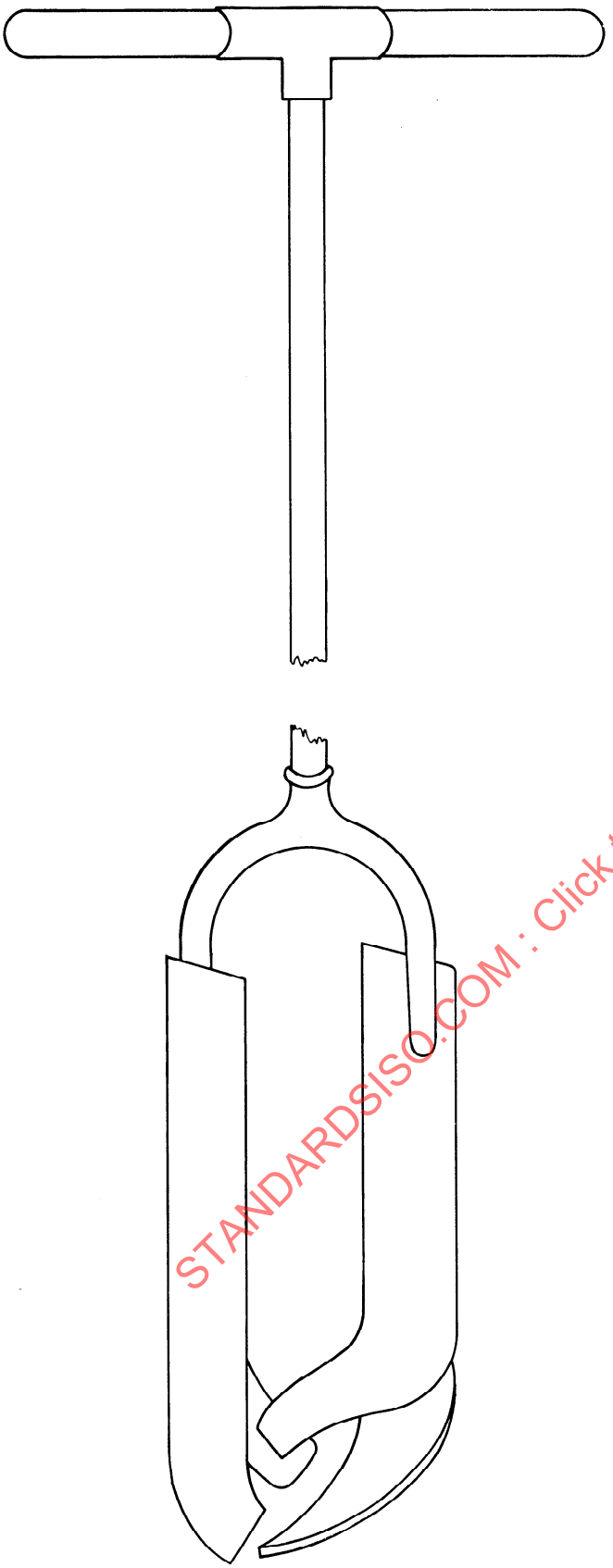


FIGURE 7 — **Auger**

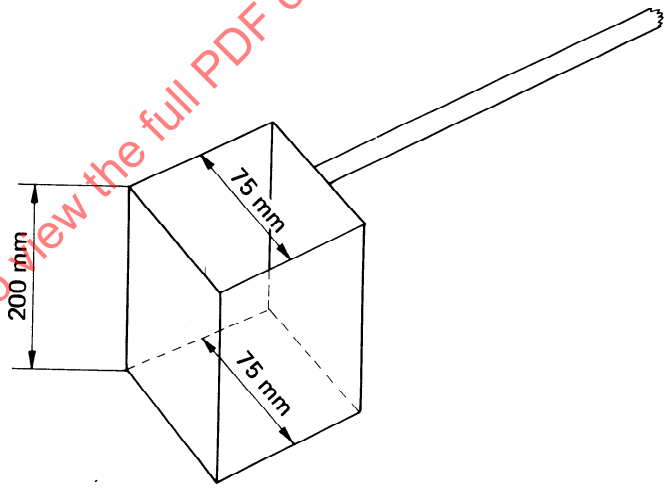


FIGURE 8 — **Ladle for smalls up to 25 mm**

Dimensions in millimetres

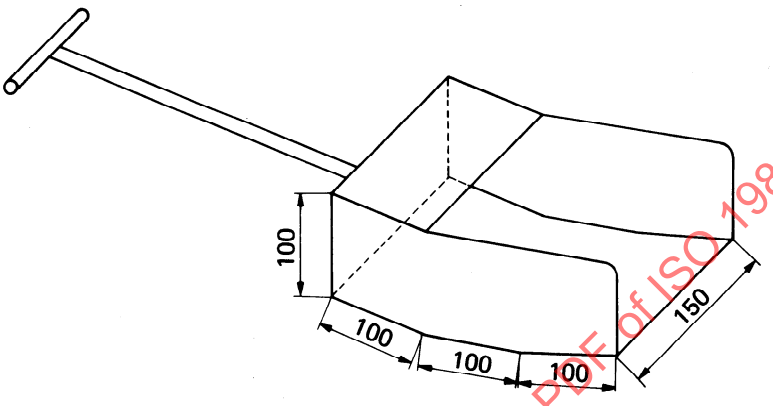


FIGURE 9 – **Scoop (suitably dimensioned for coal of 50 mm top size)**

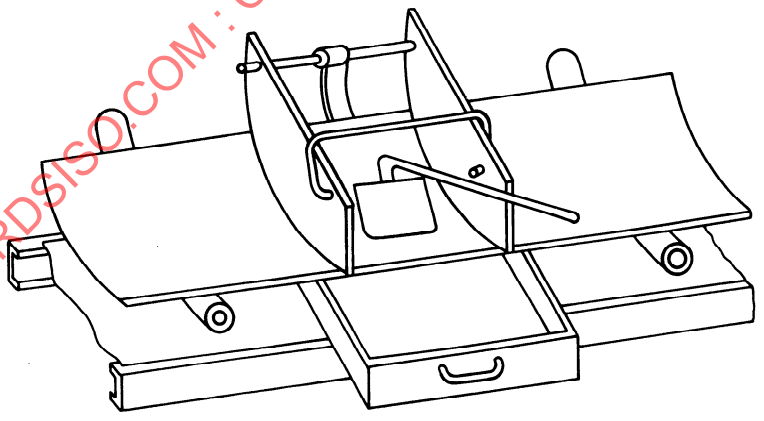


FIGURE 10 – **Sampling frame**

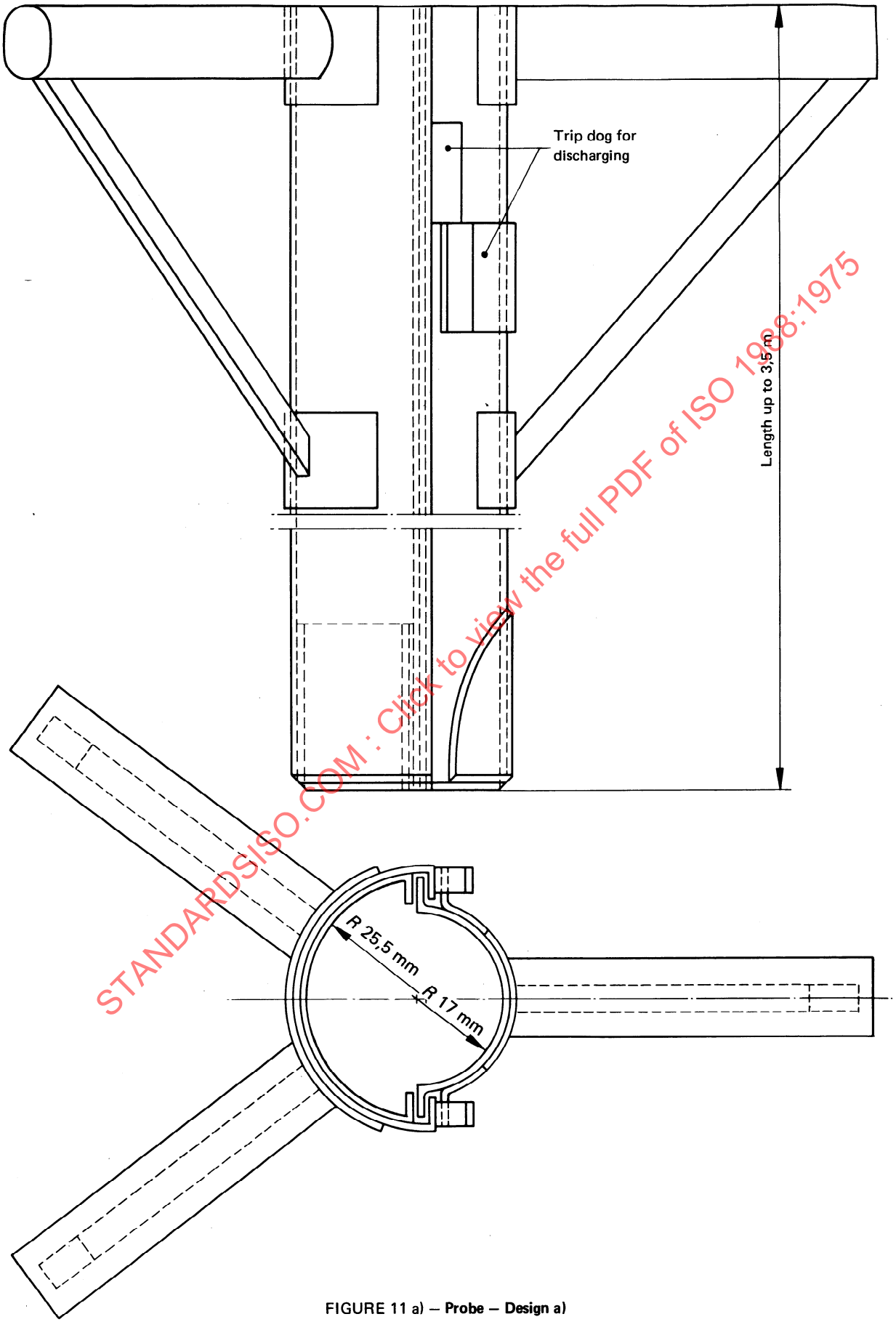


FIGURE 11 a) — **Probe — Design a)**

Dimensions in millimetres

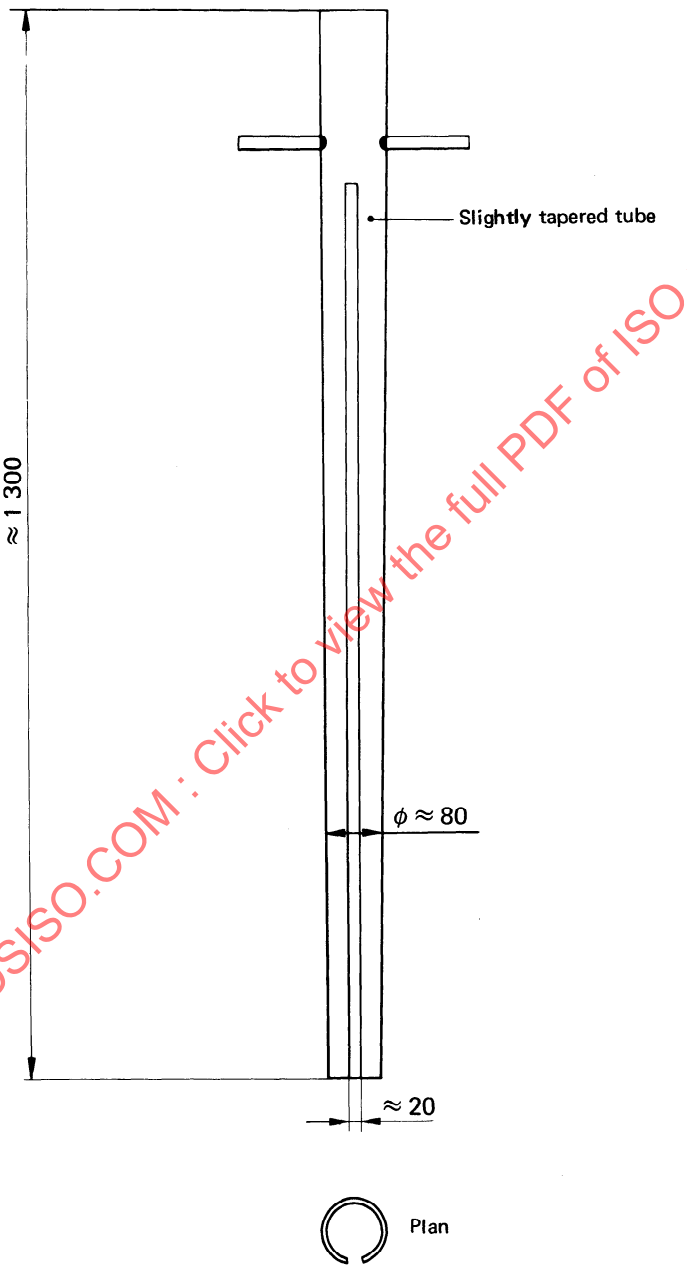


FIGURE 11 b) — **Probe — Design b)**

Dimensions in millimetres

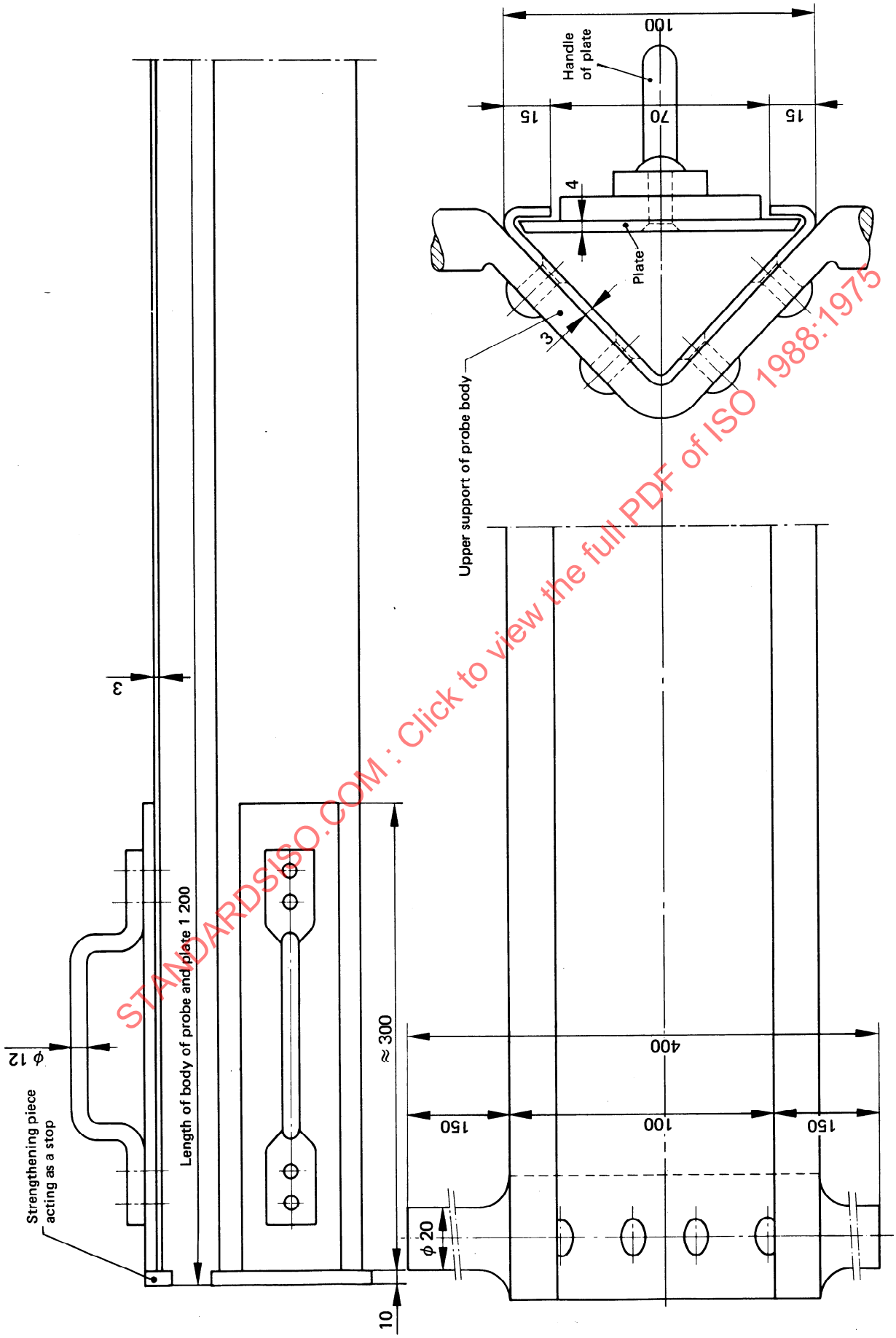


FIGURE 11 c) — Design c)

Dimensions in millimetres

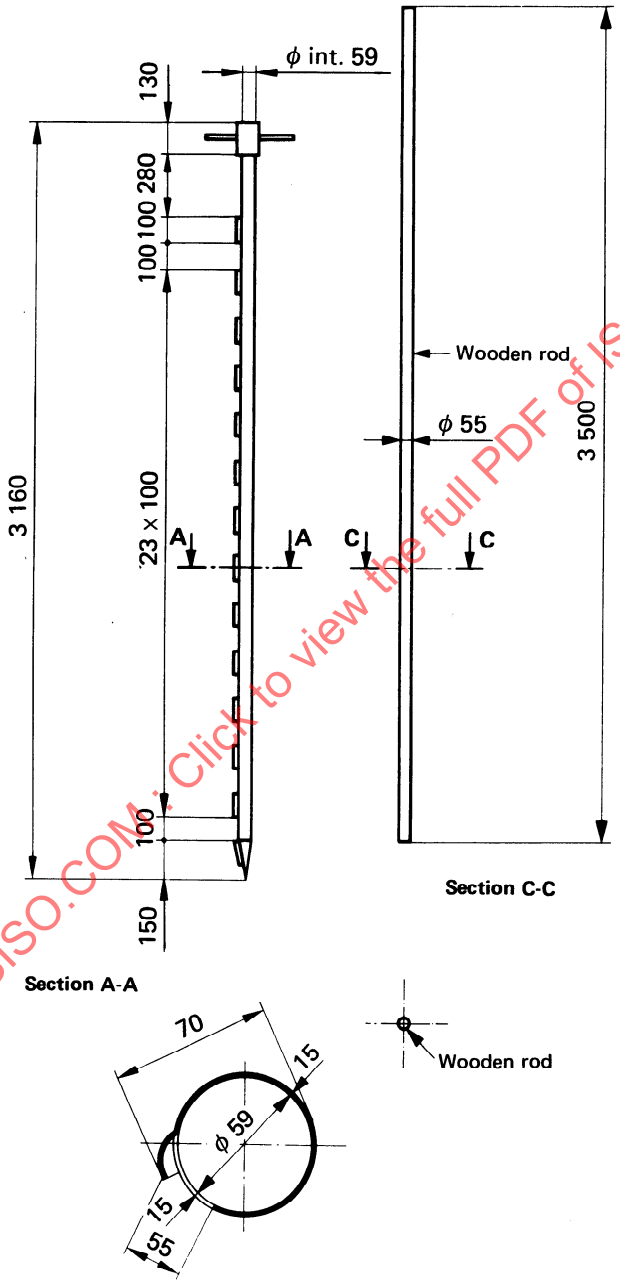


FIGURE 11 d) — **Design d), window probe**

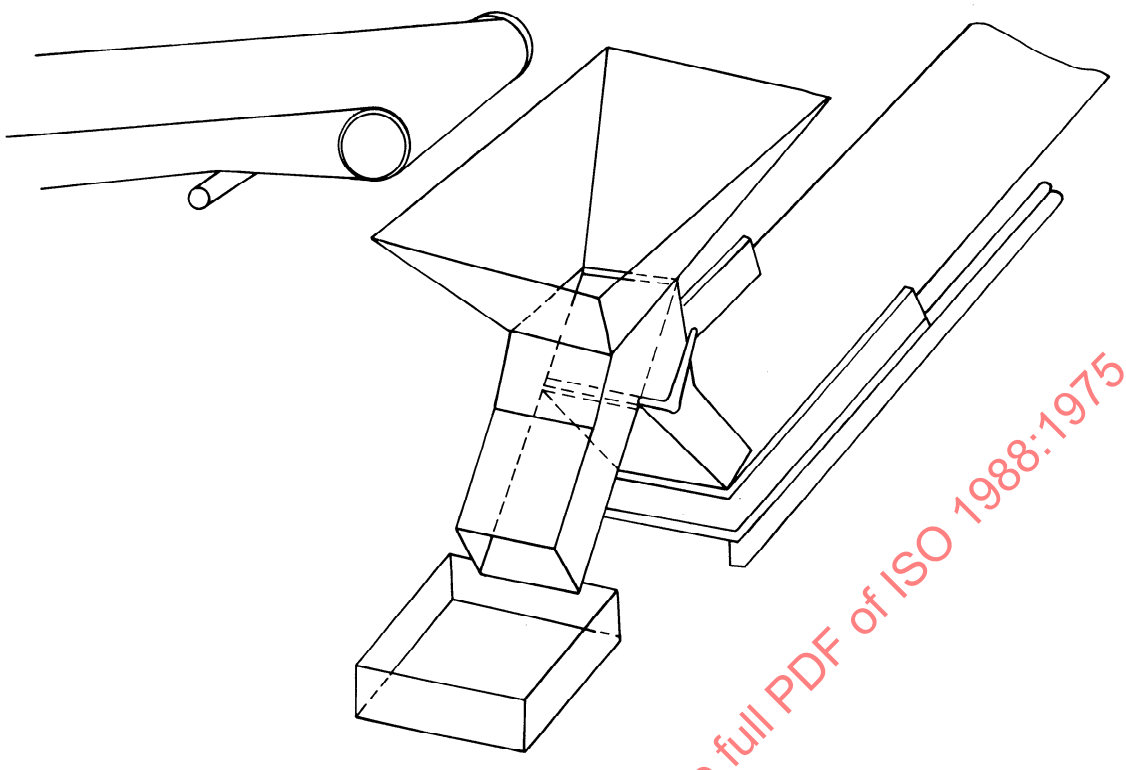


FIGURE 12 — **Falling stream — Breeches chute**

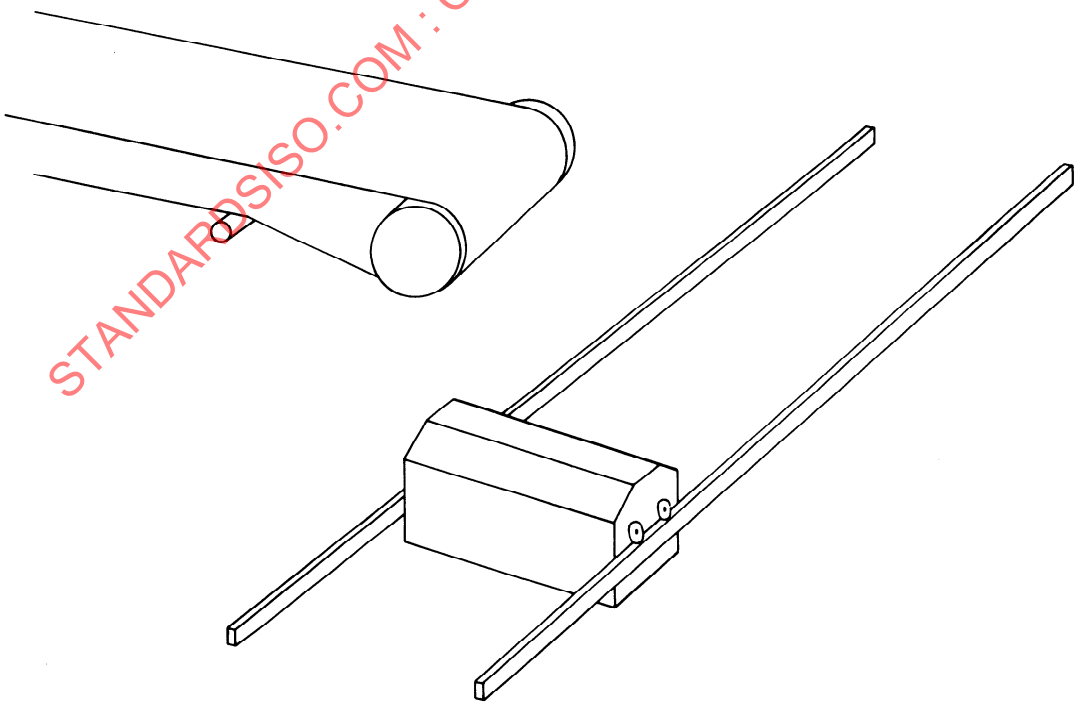


FIGURE 13 — **Falling stream — Slotted vessel**

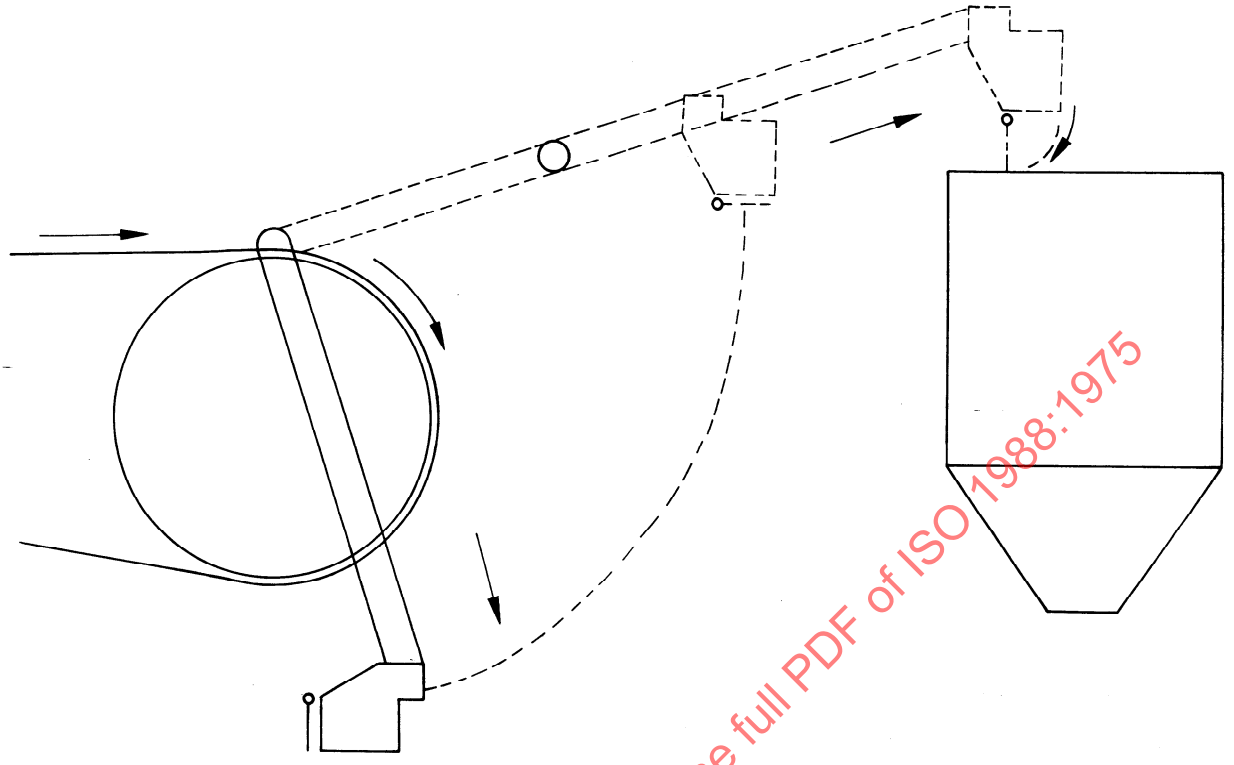


FIGURE 14 a) — **Falling stream — Swinging arm**

Dimensions in millimetres

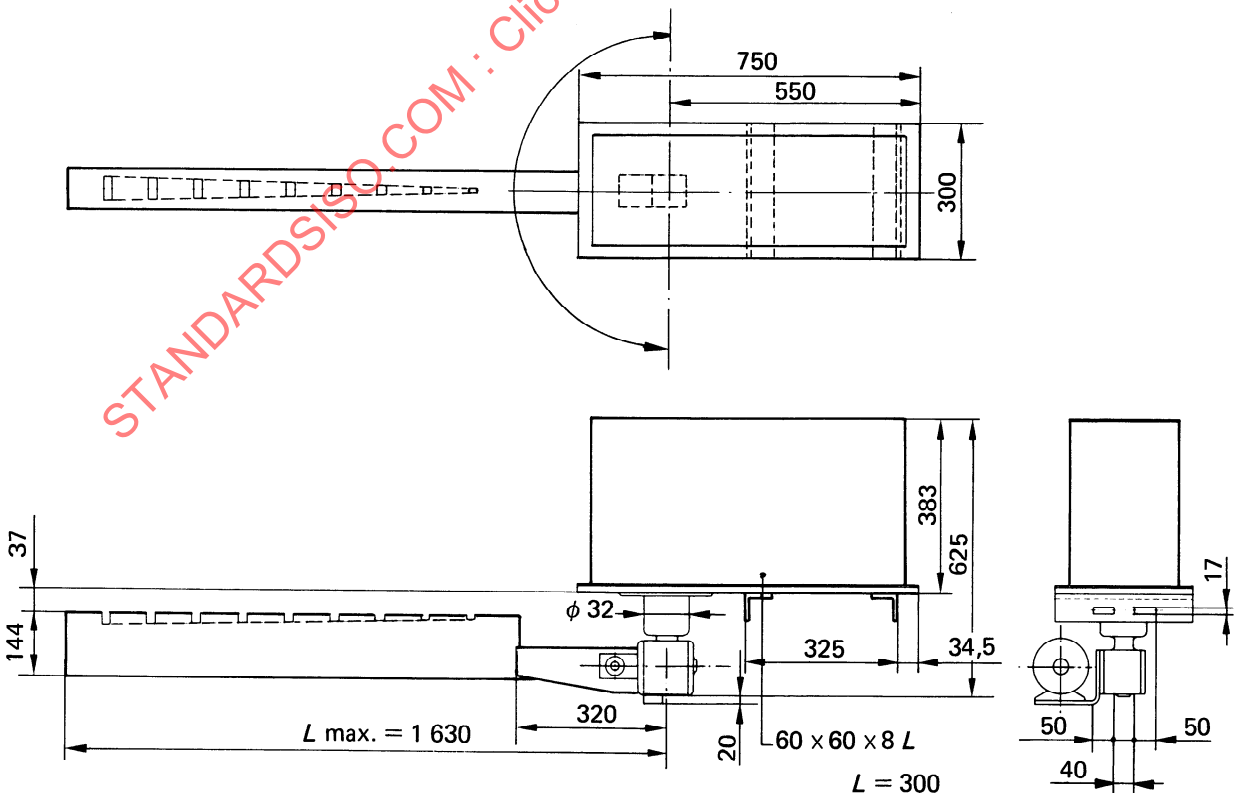


FIGURE 14 b) — **Falling stream — Swinging arm**

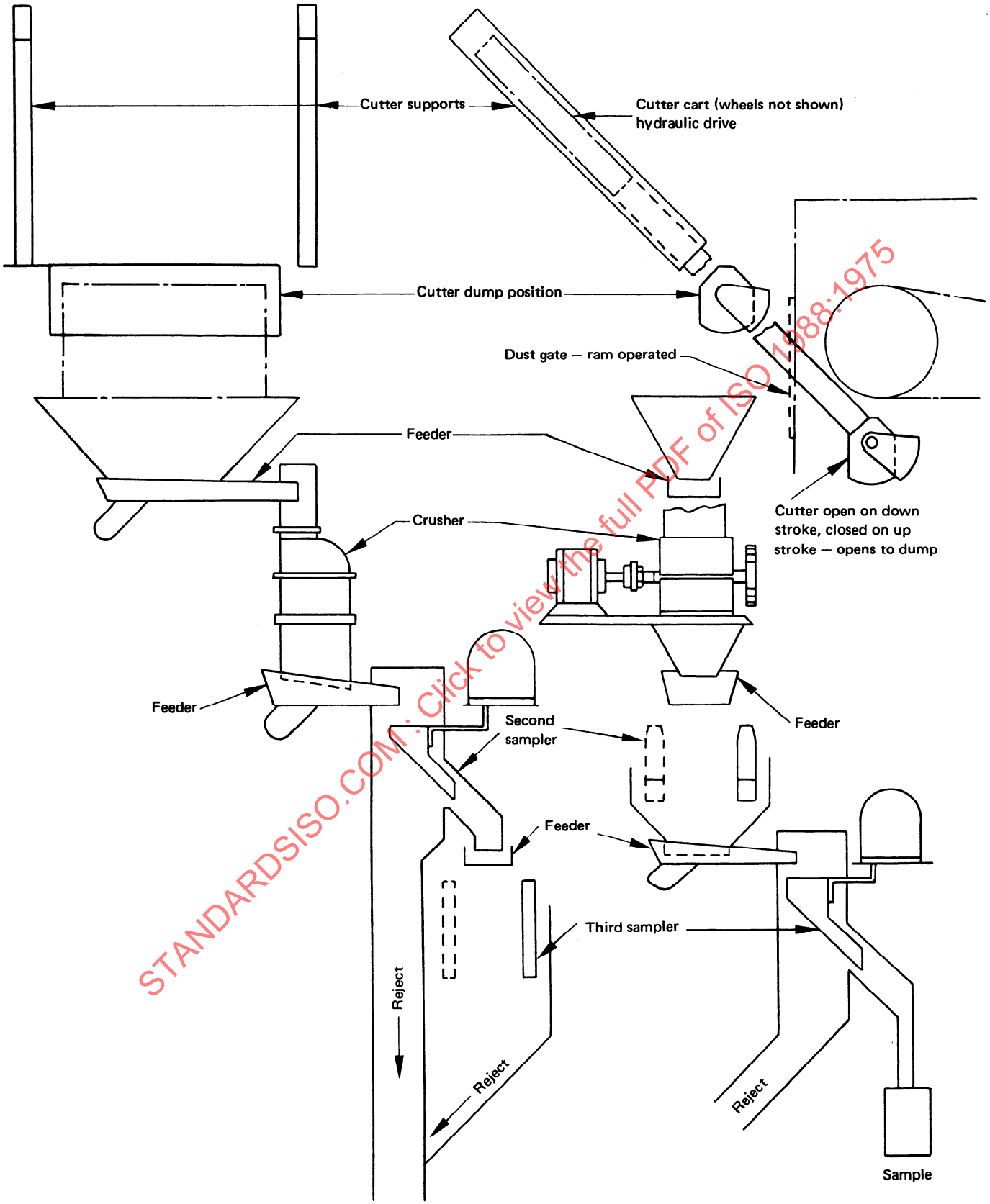


FIGURE 15 — **Falling stream — Ram operated cutter**

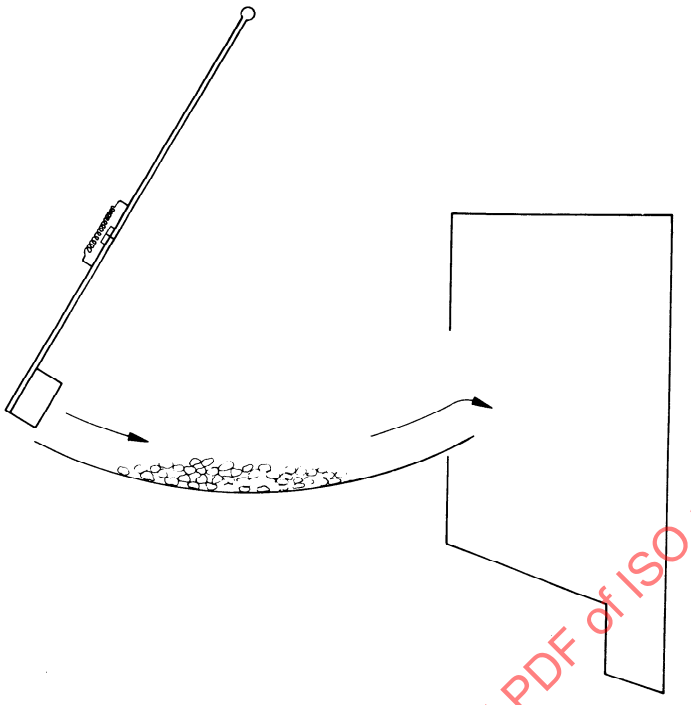


FIGURE 16 — **Moving belt — Scraper arm**

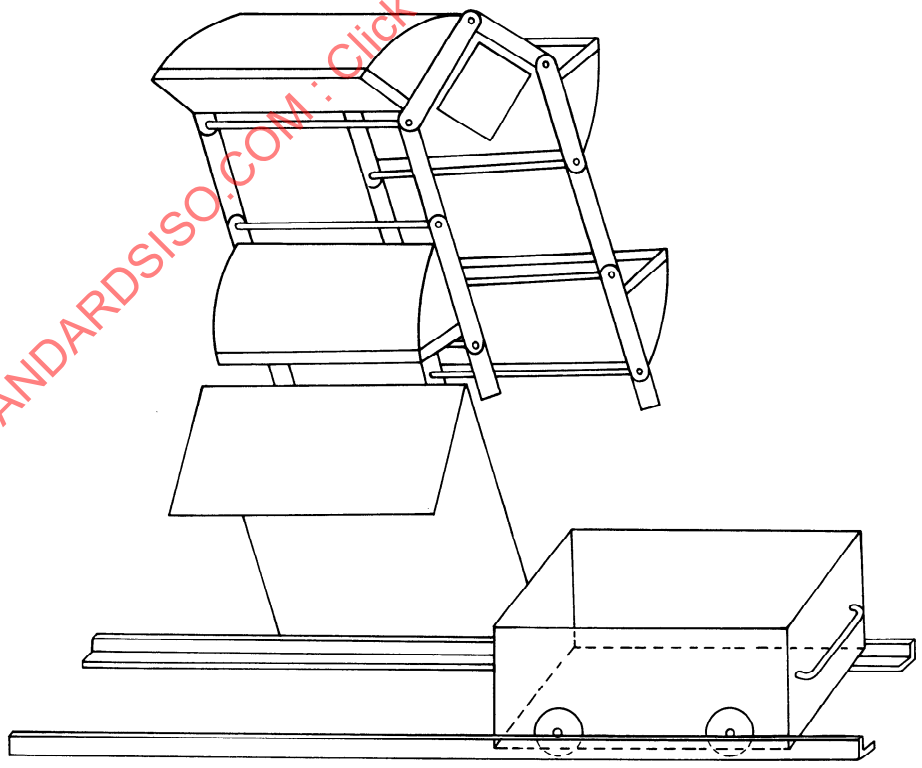
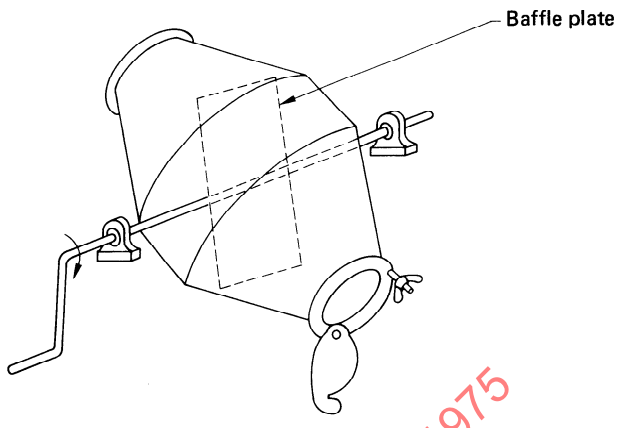


FIGURE 17 — **Bucket elevator — Box car or drawer**



Length 400 mm
Maximum diameter 330 mm

FIGURE 18 — **Double cone mixer**

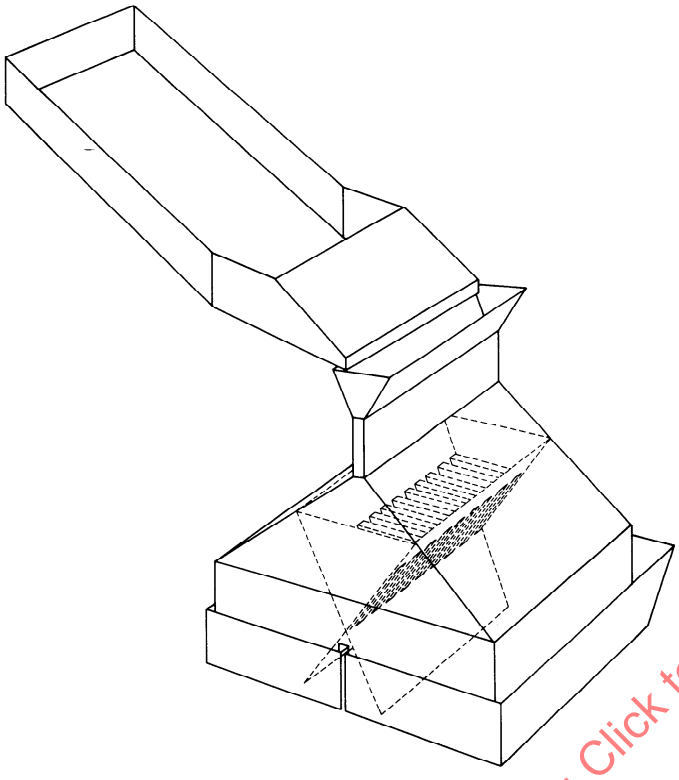


FIGURE 19 — **Riffle**

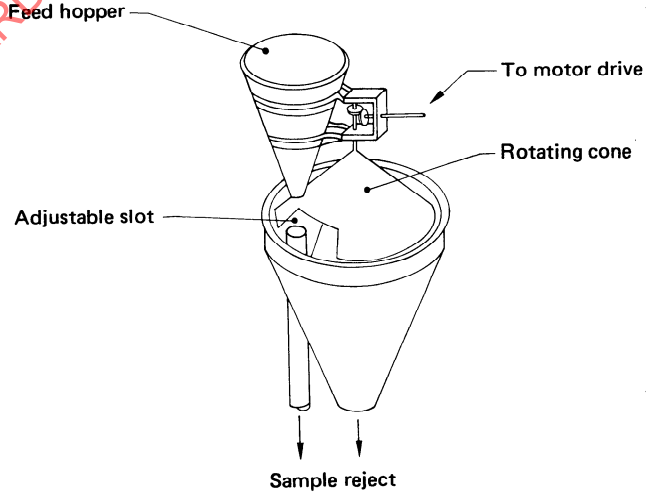
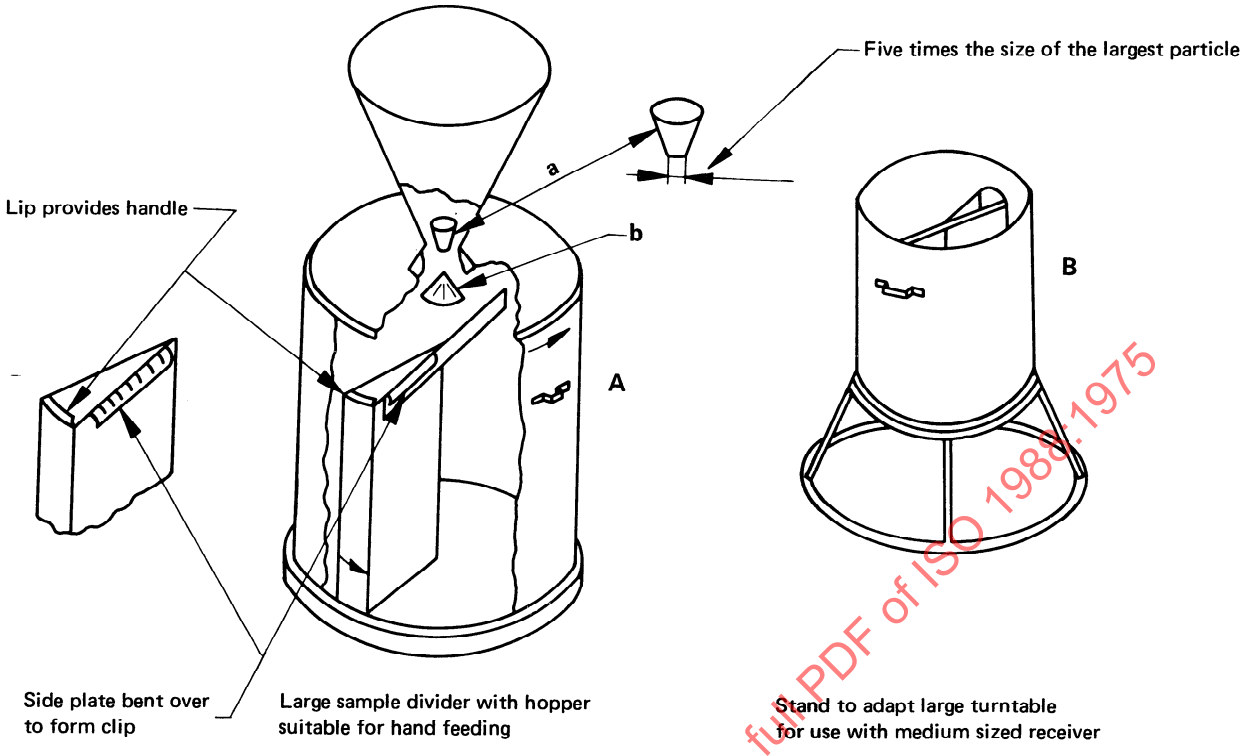
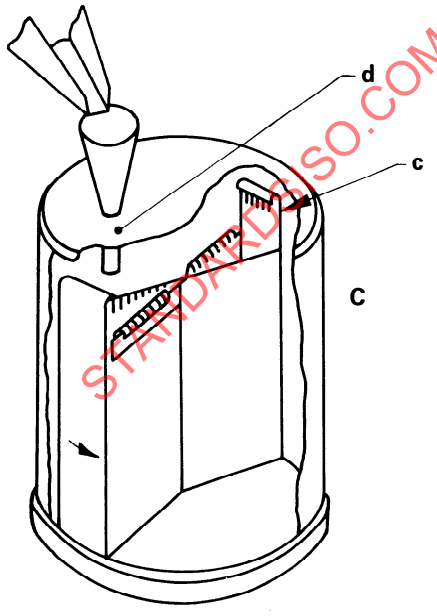


FIGURE 20 a) — **Double cone sampler divider**





High speed sample divider fed from crusher via a chute

Type	Capacity	Height of receiver	Diameter of receiver	Height of container
	kg	mm	mm	mm
A	35	450	450	400
B	7	280	300	190
C	1	150	130	150

KEY

a : interchangeable orifice
b : distributing zone
c : sample receiver segment
d : feed funnel

FIGURE 20 b) — **Rotary sample dividers**

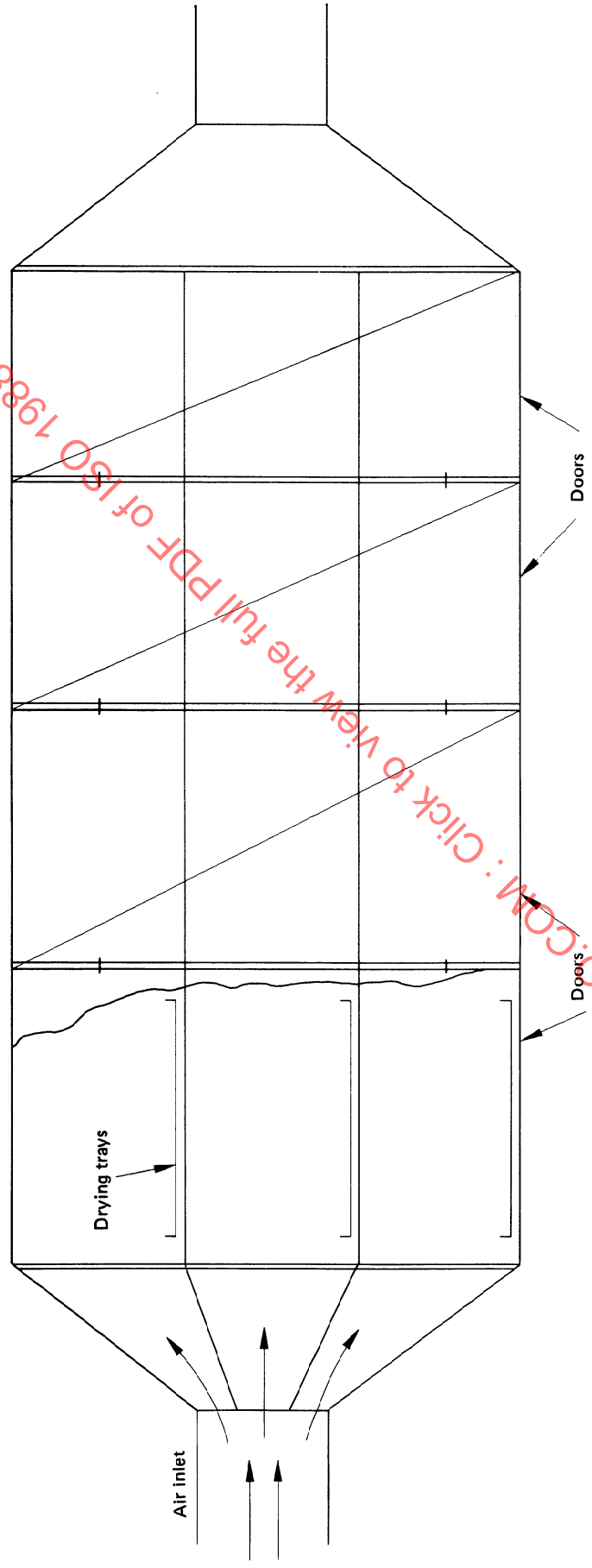


FIGURE 21 – Air-drying cabinet

Dimensions in millimetres

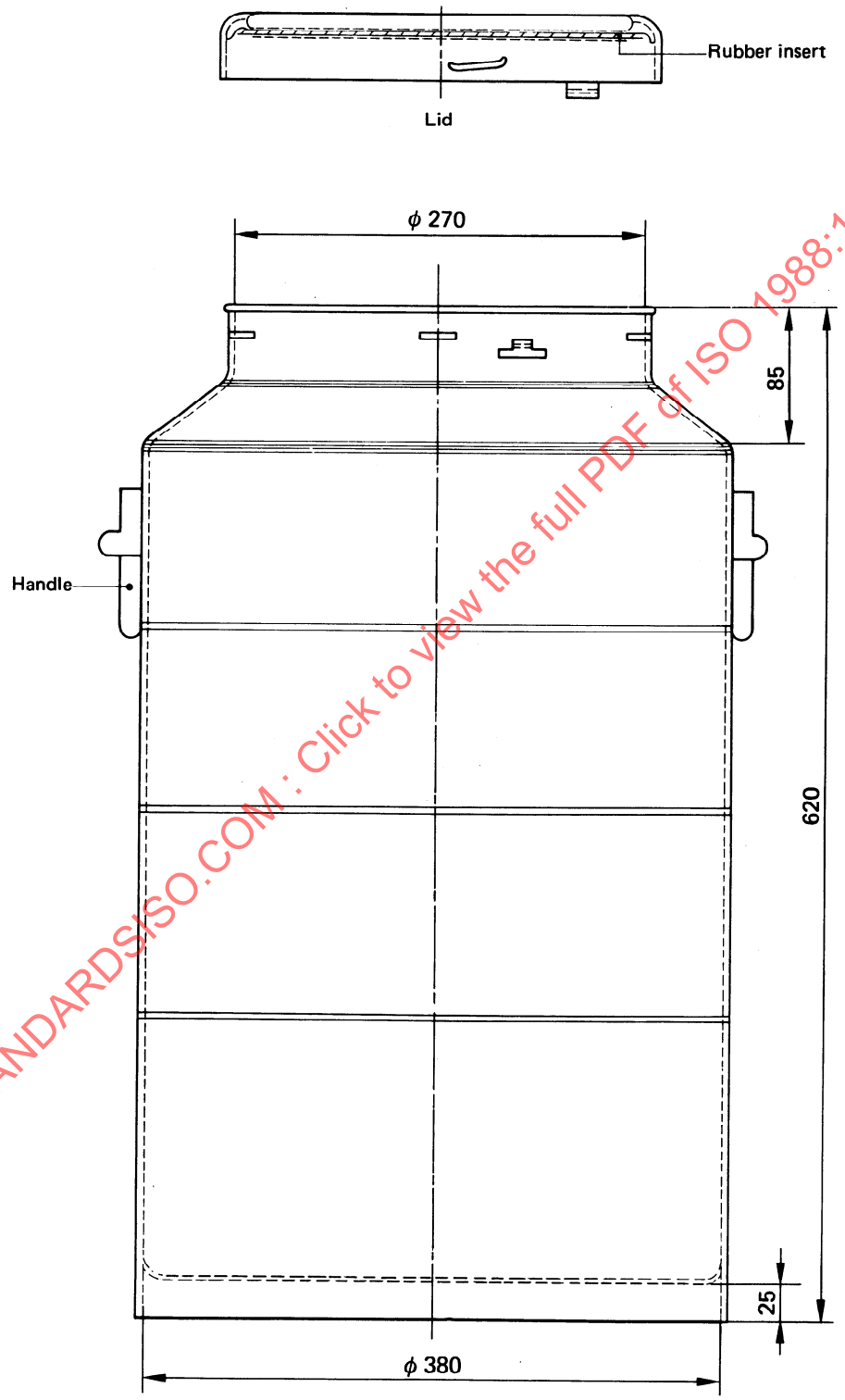


FIGURE 22 — **Sample container (galvanized iron)**

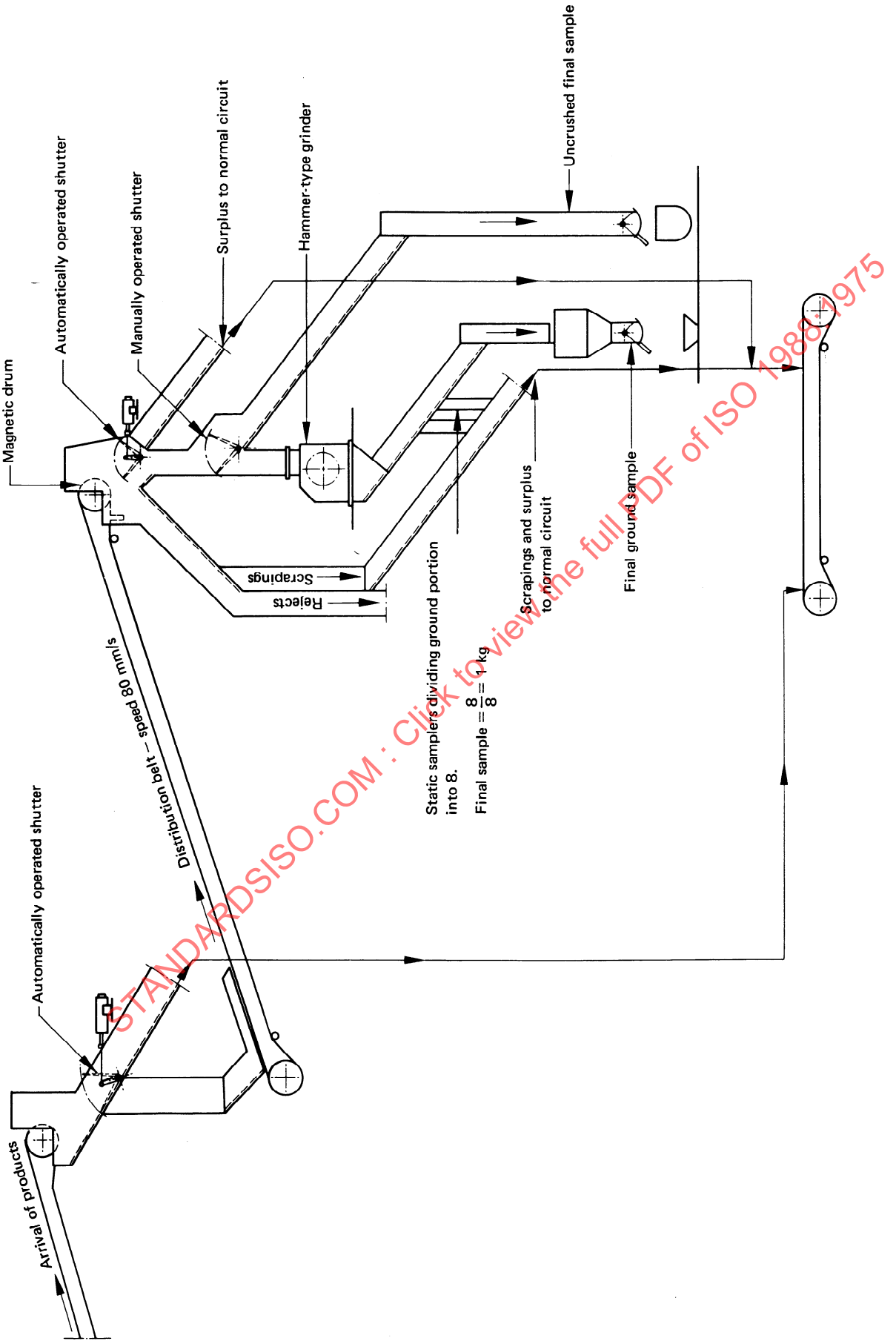


FIGURE 23 – **Falling stream – Moving shutter**

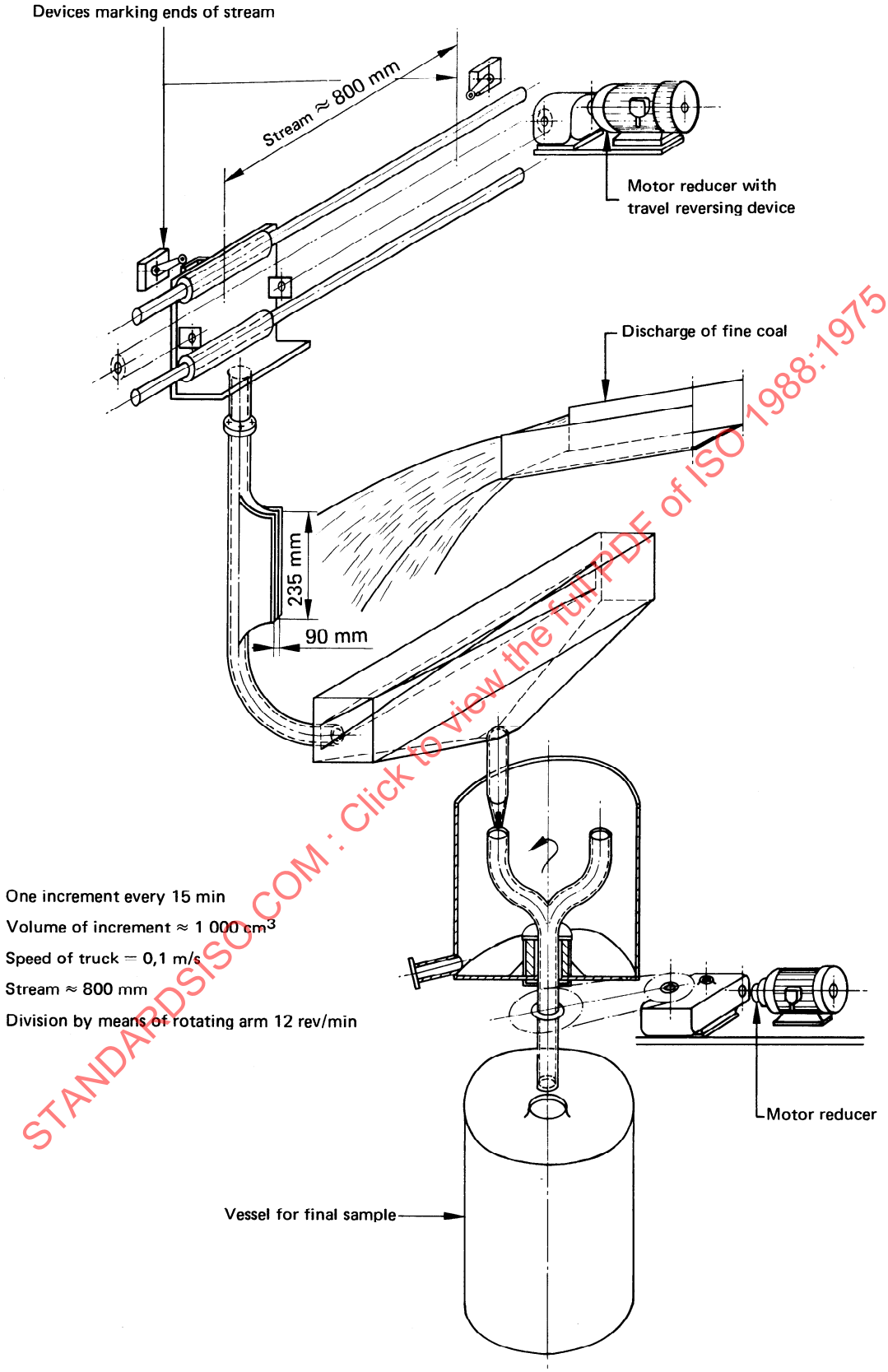
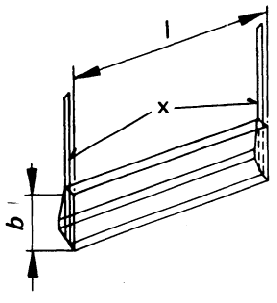


FIGURE 24 — **Falling stream (small coal) — Moving orifice**



Increment collecting device (3)

l = length of the collecting device, slightly exceeding the width of the coal stream

b = width of inlet aperture

x = lever of the collecting device

Designation of parts

1 Delivery drum
2 Operating mechanism
3 Collecting device
4 Chute
5 Apparatus for opening the chute
6 Chute cover
7 Hopper
8 Mill
9 Funnel
10 Distributing cone
11 Dividing sector
12 Hollow shaft
13 Sector funnel
14 Beaker
15 Container
16 Electric motor with reducing gear
17 Chain drive
18 Intermediate container
19 Housing of the 1st stage of the divider
20 Chute of the 1st stage of the divider
21 Hose

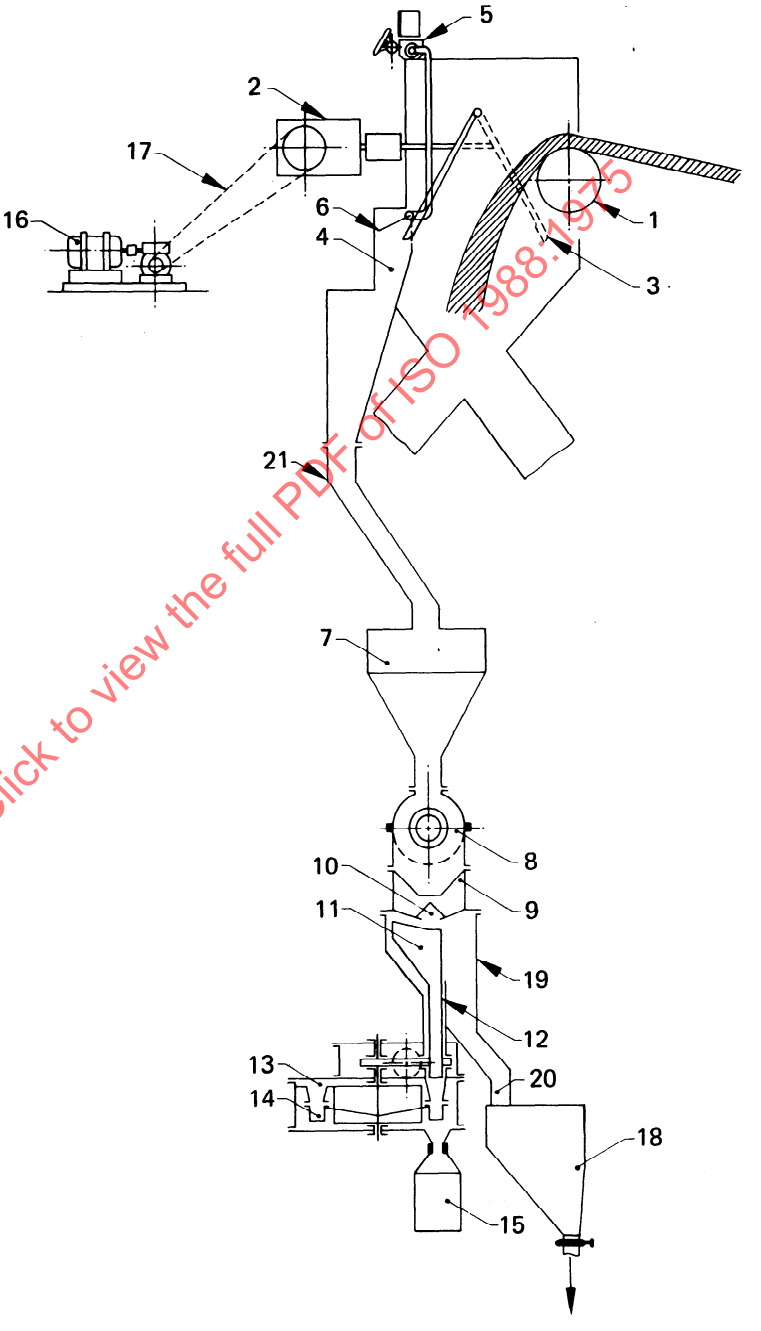


FIGURE 25 — **Falling stream — Swinging flap**

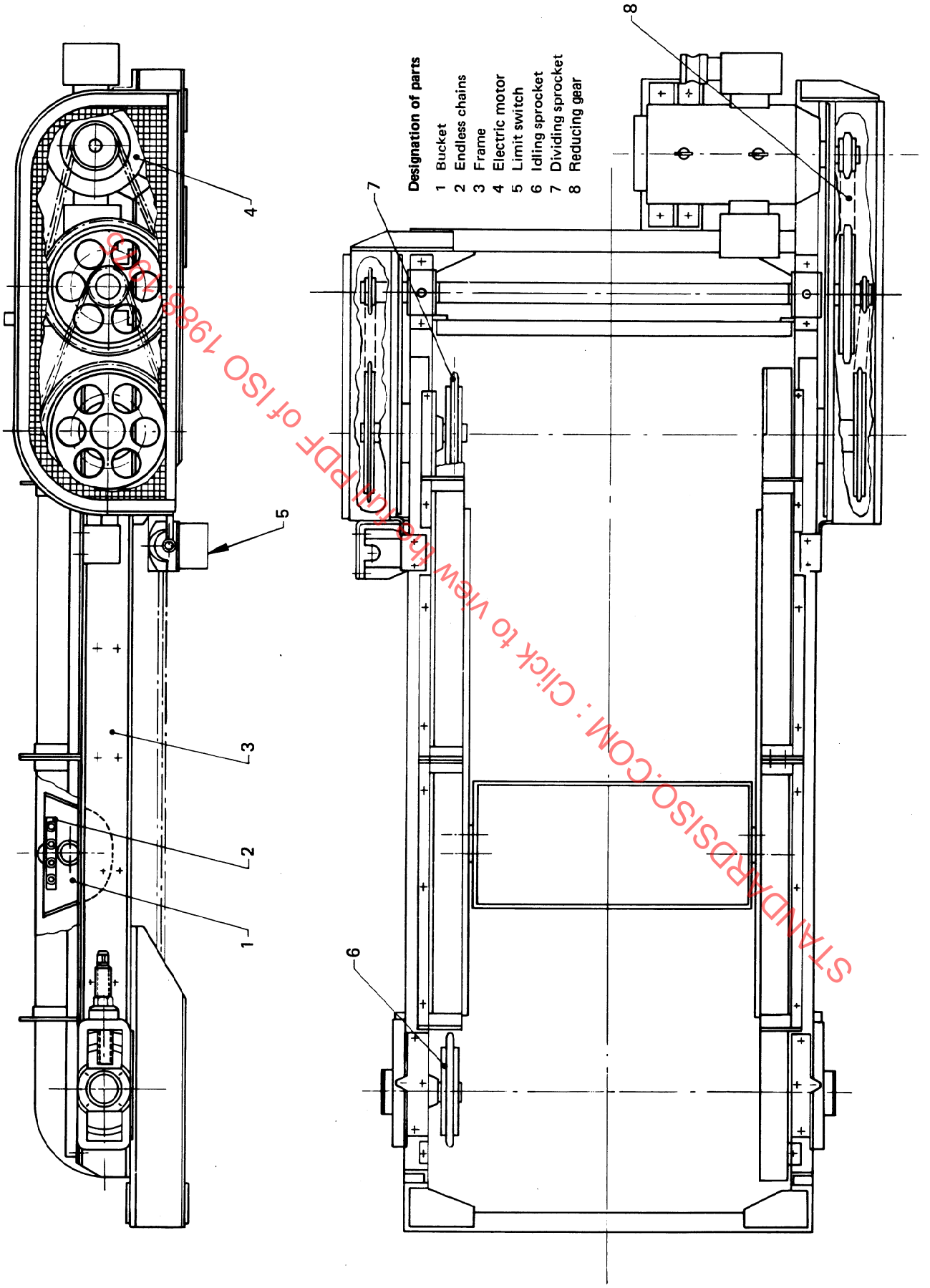


FIGURE 26 – Falling stream – Moving bucket

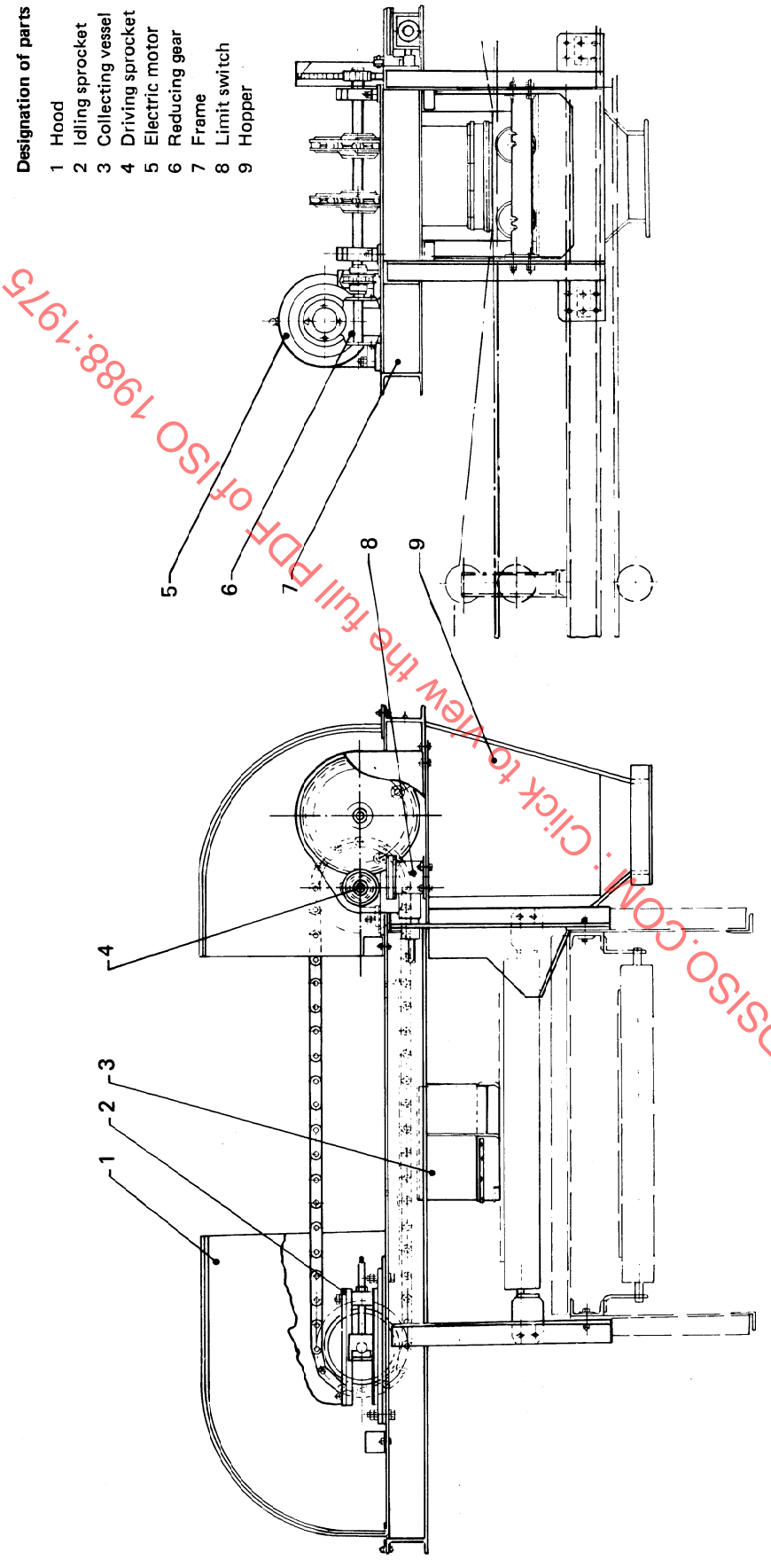


FIGURE 27 – Falling stream – Slotted vessel

Dimensions in millimetres

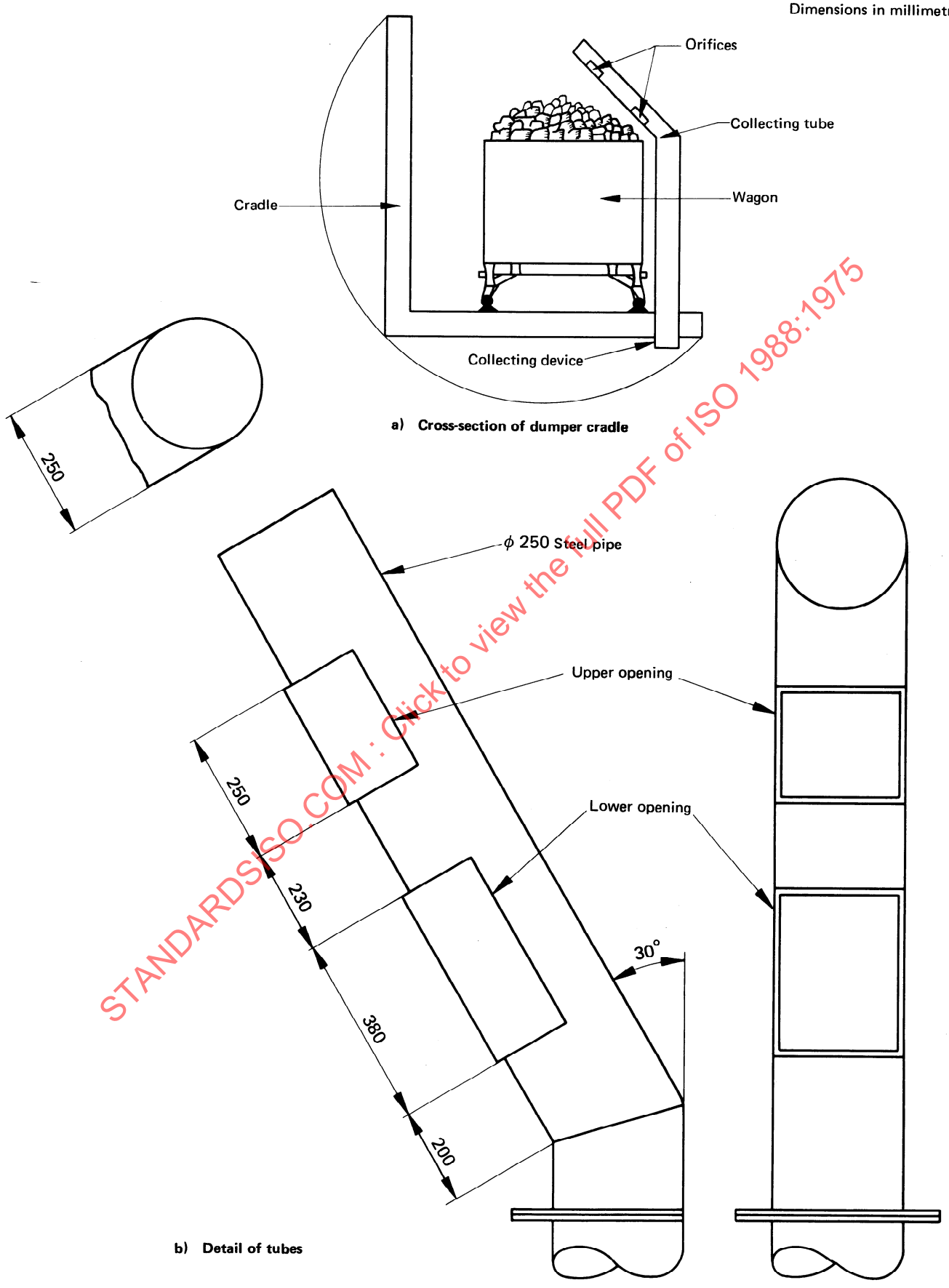


FIGURE 28 – **Wagons – Sampling tubes**

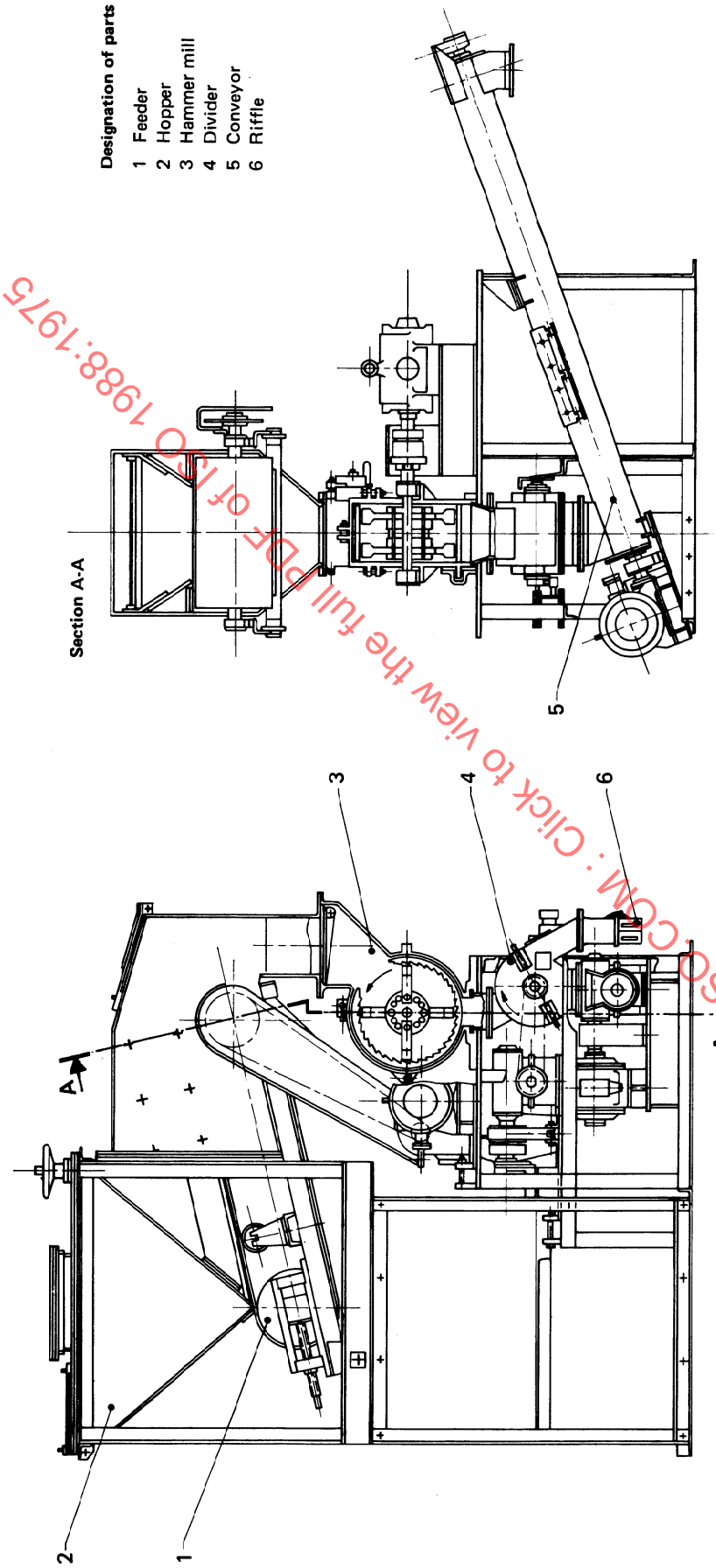


FIGURE 29 — Machine for preparation of 3 mm samples

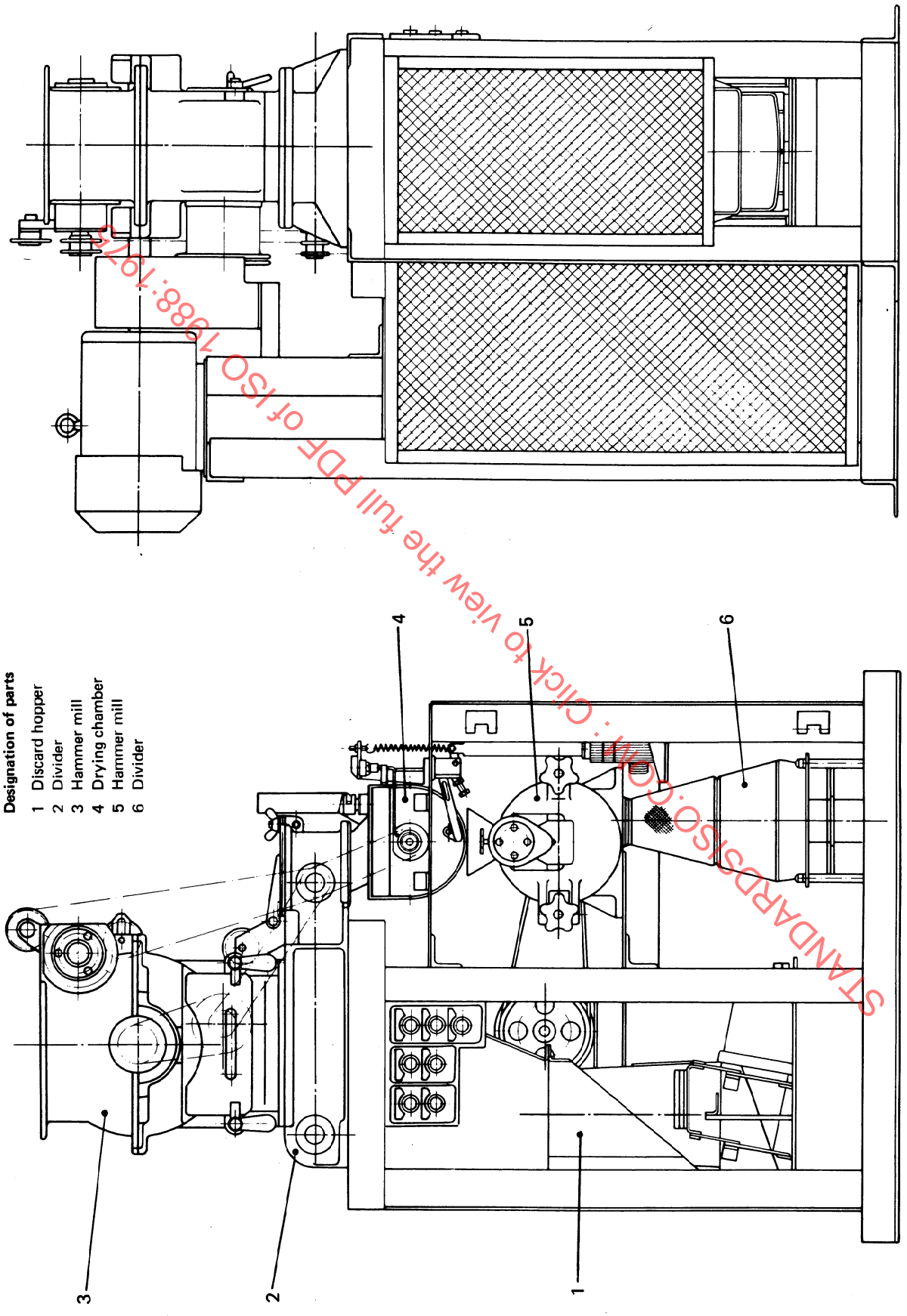


FIGURE 30 – Machine for preparation of 0,2 mm samples

ANNEX B

EXAMPLES OF INSTRUCTIONS TO SAMPLING OPERATORS

B.1 EXAMPLE 1 – FALLING STREAM

a) The sample is required for boiler trials.

b) The coal is washed smalls with particle size less than 25 mm and reputedly 12 % ash (dry basis).

c) The analyses required are moisture, proximate analysis, ultimate analysis and calorific value.

d) A separate moisture sample is required.

e) The sample is to be taken from a falling stream.

f) The size of the consignment is about 800 tonnes in one lot.

g) The ash is to be determined to ± 0,5 % but the moisture to the reference standard for moisture; replicate sampling is to be employed, taking six sub-samples.

Proceed as follows :

1) Take 128 increments (32 for a separate moisture sample; $96 = 16 \times 6$ for general analysis, as calculated from 3.2.4, and table 6).

2) The minimum mass of each increment to be 1,5 kg.

3) Place 96 increments in rotation into 6 containers, such that these hold 16 increments each, to form 6 sub-samples for general analysis; a seventh container, capable of being sealed between the insertion of increments is required to hold 32 increments (evenly spread over the consignment), to provide a separate sample for moisture.

4) Use a sampling ladle with a width of at least 65 mm.

5) Sampling should be carried out at the point of discharge of the stream.

6) Label each of the seven containers for later identification.

7) Prepare a sampling report.

B.2 EXAMPLE 2 – WAGONS

a) The sample is required for routine control

b) The coal is dry uncleaned slack with particle size less than 15 mm and with about 25 % ash (dry basis), ascertained from past records.

c) The analyses required are moisture "as received" and ash.

d) A common sample is required.

e) The size of the consignment is 1 800 tonnes, in 3 train loads of 33 wagons; each wagon is 3 m wide × 5 m long; the coal is 1,75 m deep; each train load is to be sampled separately.

f) The ash is to be determined to ± one-tenth of the true ash content; duplicate sampling is to be employed.

Proceed as follows for each train load :

1) Take 48 increments (table 3).

2) The minimum mass of each increment should be 0,90 (say 1,0) kg (see 3.3.2).

3) Sampling should be carried out from wagons tops using a probe inserted to the depth of the coal.

4) Take 2 increments from 15 wagons and 1 increment from each of the other wagons. Select the 15 wagons as follows : Provide two boxes P and Q. In box P, put a set of discs numbered 1 to 33. Select a disc from box P, record the number and put the disc into box Q. Continue in this way until 15 numbers out of 33 have been selected. Number the wagons with chalk and take 2 increments from the wagons with the selected numbers.

5) Divide the top of each wagon into 15 areas as follows :

1	4	7	10	13
2	5	8	11	14
3	6	9	12	15

6) Take either 1 or 2 increments from each wagon from the positions 1 to 15 selected at random as described above by using discs numbered 1 to 15 in box P; each disc should be put into box Q until the 15 have been used; then start again.

7) Place the increments alternately into sample containers labelled A and B.

8) Repeat the whole procedure for each train load.

9) Prepare a sampling report.

B.3 EXAMPLE 3 – BARGE

a) The sample is required for trade purposes.

b) The coal is washed fines (0 to 10 mm) ash content below 10 %, moisture content 8 to 10 %.

c) The analyses required are moisture "as received", ash, volatile matter and calorific value.

d) A common sample is required.

e) The size of the consignment is 400 tonnes, divided over 8 compartments of a barge in which the coal is loaded approximately 3 m high.

f) The ash content is to be determined to the reference standard of ± one-tenth of the true value.

Proceed as follows :

1) Take 36 increments (minimum number 32; table 3).

2) The minimum mass of each increment should be 0,5 kg.

3) Collect the increments with the aid of a short probe (see annex A) and store them in a closed container.

4) Sample the compartments in twos after they have been so far unloaded that the bottom is just visible.

5) Take 12 increments at each of 3 levels, 0,2, 1,5 and 2,8 m above the bottom respectively; i.e. take 9 increments from every two compartments, 3 from each level.

6) Label and seal the container and state date and place of sampling, origin and particulars of the cargo (size) and the name of the barge.

7) Prepare a sampling report, giving details of the coal, sampling method employed, number and mass of increments, etc.

B.4 EXAMPLE 4 – SHIP

a) The sample is required to control whether the consignment has the warranted quality.

b) The consignment is run of mine coal of size under 80 mm with about 15 % of ash (dry basis).

c) The analyses required are moisture, ash, volatile matter and calorific value.

d) A common sample is required.

e) The size of the consignment is 10 800 tons, divided over 5 holds :

Hold	1	contains about	1 200	tonnes
"	2	"	2 400	"
"	3	"	3 200	"
"	4	"	2 400	"
"	5	"	1 600	"

The depth of a hold is approximately 12 m.

f) The precision required is ± 1 unit in ash percentage.

Proceed as follows :

1) Take 675 increments (the actual number is calculated as

$$64\sqrt{10,8} \times \frac{4 \times 1,5^2}{5 - 1,5^2} = 690$$

see 3.2.3 and 3.2.4 and table 3; the number of 675 will be sufficiently close and can be distributed more easily).

2) Divide them over the holds as follows :

Hold 1 :	75	increments
" 2 :	150	"
" 3 :	200	"
" 4 :	150	"
" 5 :	100	"
Total	675	increments

3) The minimum mass of each increment should be 5 kg.

4) Collect the increments with a scoop, at least 200 mm wide, and store them in a closed container.

5) Sample every hold in 3 stages (see figure 2) and take one-third of the increments in each stage.

6) Label the containers and state data and place of sampling, origin and particulars of the cargo and the name of the ship. Prepare a sampling report giving details of the coal, sampling method employed, number and mass of increments, etc.

B.5 EXAMPLE 5 – BARGES

a) The sample is required to be accurately determined for experimental trials.

b) The coal is under 12 mm in size.

c) The coal is a mixture of six washed and unwashed coals and middlings with ash contents from 6 to 35 % – a reputed ash content of say, 25 %.

d) The analyses required are moisture "as received" and ash.

e) A common sample is required.

f) There are 12 barges each with four holds and each barge contains about 100 tons of coal; the height of the coal is 2,5 m; each barge is to be sampled separately.

g) The moisture and ash are to be determined to the reference of standards of precision; duplicate sampling is to be employed.

Proceed as follows :

1) The minimum mass of each increment should be 0,75 kg.

2) Imagine that each hold is marked out into 6 parts on its length and 6 parts on its width making 36 squares altogether (see figure 31).

3) In each hold of 36 squares make 6 insertions with a probe to the bottom of the barge for sample A and the same number for sample B, using pattern 1 of figure 31.

4) Place the set of 6 increments from the first hold into a container labelled A then the second set of 6 increments from this hold into a container B. Repeat this procedure for the other three holds of the same barge. Thus, each container will hold 24 increments : 48 increments will have been taken from the whole barge.

5) Analyse samples A and B from the first barge separately for moisture and ash thus obtaining two moisture figures and two ash figures.

6) Repeat the same procedure on the second barge using pattern 2 given in figure 31.

7) Repeat the procedure on the other barges taking the increments from successive barges from the positions indicated on the successive patterns given in figure 31.

8) Label each of the containers clearly to indicate whether it is an A sample or a B sample and from which barge it is taken.

9) Report the figures for subsequent statistical analysis.

In each barge there are 64 central areas and 80 peripheral areas, a percentage of 45 and 55 respectively. Thus in each hold there is the same density of increments taken from the squares, around the edge of the hold and from the centre of the hold. Thus from the central squares, 11 A or B increments are required on average and from the peripheral square 13 on average, giving a total of 24 increments.

Note than in each system of 6 × 6 each horizontal row contains one position for increment A and one position for increment B; similarly for each vertical column of 6.

A = 24 positions for the probe for sample A

B = 24 positions for the probe for sample B

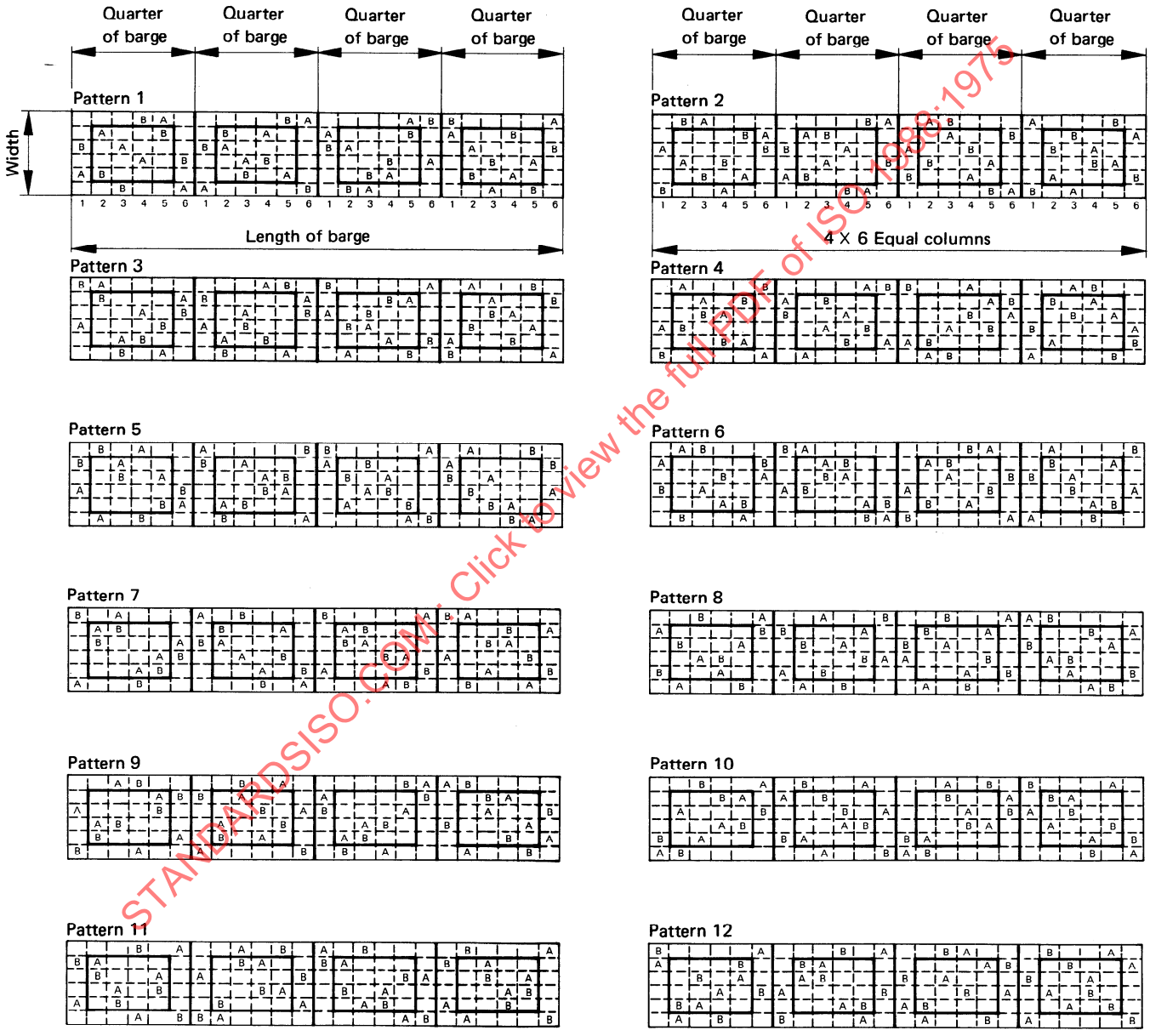


FIGURE 31 — **Scheme of sampling from barges using twelve stratified random patterns**

ANNEX C

METHODS OF CHECKING PRECISION BY MEANS OF REPLICATE SAMPLING

C.1 INTRODUCTION

An estimate of the precision of sampling, though not of its bias, can be obtained by taking the sample in a number of parts (replicate sampling) and comparing the results obtained from these parts.

Sub-clause 3.5 describes the collection of samples in a number of parts and this annex describes the treatment of the results.

C.2 CONTINUOUS SAMPLING

C.2.1 Outline of procedure

The precision which is being achieved is checked by comparing the mean difference between duplicates obtained from ten successive pairs of duplicate samples with the theoretical value that would be obtained if the desired precision was being achieved. If it is necessary, the number of increments to be taken for further samples of the same coal can then be adjusted.

C.2.2 Recording results

Record the results as in table 13 and calculate for each sample the mean value and the difference between the duplicates. When the results of ten pairs of duplicate samples are available, calculate the mean difference, d, between duplicates.

C.2.3 "Rogue" results

A "rogue" is a result which appears to be in disagreement with others from the same coal and which arouses suspicion that there has been a mistake in sampling or sample preparation. The following test is used to decide whether such a result should be rejected.

If a difference between duplicates is greater than 3,5 times the mean difference, then reject that difference and take a further sample in duplicate before analysing the results. In the test report any results considered to be "rogue" results should be noted.

C.2.4 Testing results

When ten samples have been taken in duplicate, the results are examined. The observed mean difference between duplicates, d, is compared with the theoretical value, D, that would be expected if the desired precision was achieved. The appropriate value of D should be obtained from table 14.

Calculate the ratio ;

$$\frac{D}{d} = \frac{\text{expected mean difference between duplicates}}{\text{observed mean difference between duplicates}}$$

This ratio is used to check whether sampling is at the desired level, as follows :

a) if the ratio D/d is greater than 2,0, too many increments have been taken;

b) if the ratio D/d is between 2,0 and 0,67, there is no evidence that the number of increments is incorrect;

c) if the ratio D/d is less than 0,67, too few increments have been taken.

C.2.5 Adjustment procedure

If condition b) applies take a further series of ten duplicate samples; if too many or too few increments have been taken, adjust the number of increments according to table 15 and take a further series of ten duplicate samples. Continue duplicate sampling, the number of increments being adjusted if necessary, until two successive sets of ten results give values of the ratio D/d lying between 2,0 and 0,67.

C.2.6 Check procedure

Thereafter duplicate sampling may be carried out as a check. It is not necessary to take every sample in duplicate; for example one sample in five may be taken in duplicate or, alternatively, a group of ten duplicate samples may be taken periodically. When ten duplicate samples have been taken, test the results as described in C.2.2 to C.2.4. For this purpose ignore the routine samples which are not taken in duplicate.

If, for any set of ten duplicate samples, the ratio D/d lies outside the limits 2,0 and 0,67, adjust the number of increments and resume full duplicate sampling until two successive sets of ten give the ratio D/d between 2,0 and 0,67. Then resume check sampling.

C.2.7 Application to other characteristics

Table 14 and the tests can be used for any characteristic. For convenience the precisions quoted are those which will normally be encountered when determining the percentage of some constituent of the raw coal. The value of D for a given number of units is, however, simply proportional to the precision required. For example, to obtain the calorific value of coal to ± 50 calories look in the row of table 14 which gives values for a precision of ± 0,5 and multiply the values by 100. Thus if the determination of calorific value is wanted to an accuracy of ± 50 for each unit, the

appropriate value of D is 40. If the mean calorific value over 15 units of coal is required to within ± 25 calories the value of D is 77.

NOTE – The sampling precision is tested after ten duplicate samples have been obtained irrespective of whether the precision of each individual sample or that of the average of any number of samples is required.

C.2.8 Example

Coal is being received at a plant where heat balances are required every month; the mean ash value for the month is required to be within ± 0,5 %. Duplicate samples are taken each day from the coal going to the furnace and the mean difference between duplicates after 10 days is found to be 0,48 %.

From table 14 we find that, if the mean of 25 samples is required to a precision of ± 0,5 %, the value of D is 2,0. Thus $D/d = 4,2$ and too many increments are being taken from each unit. The number of increments taken in subsequent samples is therefore halved, in accordance with table 15, and duplicate sampling is continued.

For the next two sets of ten samples d is found to be 1,2 % and 2,2 %, giving values of D/d of 1,7 and 0,9 respectively. Sampling is therefore continued at this level. At this plant it was decided to keep a check on the precision by taking duplicate samples 1 day per week. It will be noted that ten daily samples were sufficient to show that the average figure which would be obtained at the end of the month would be more precise than was needed and that a reduction in the number of increments could be made.

C.3 INTERMITTENT SAMPLING

(This section is only applicable in certain cases when sampling from a stream of coal or from wagons).

C.3.1 Outline of procedure

The precision related to one period is affected by :

a) The number of units sampled in this period;

b) The number of increments in the sample taken from each unit.

The variation between the unit values contributes to the total variation; thus there is no point in taking a large number of increments from each unit (to determine the unit values to high precision) if there is an excessive variation between the units. Consequently the number of units sampled is checked to ensure that the desired precision is being obtained and then the number of increments in each sample is checked to ensure that the number of increments taken from each unit is correct. The two factors are checked separately and each is adjusted as appropriate.

C.3.2 Recording results

Record the results as shown in table 16; calculate the mean value of and the differences between each pair of duplicate values : Calculate the mean difference between duplicates, d and the range, c, of the mean sample values, i.e., the difference between the greatest and least.

C.3.3 "Rogue" results[1)]

The following tests are used to decide whether a result is a "rogue" and should be rejected :

a) If a difference between duplicates is greater than 3,5 times the mean difference, d, obtained by excluding that difference, then reject that difference and take a further sample in duplicate before analysing the results.

b) If the value of c is reduced to one half of its original value by excluding one mean sample value, reject that sample value and take a further sample in duplicate before analysing the results.

C.3.4 Testing results – Number of units

When ten duplicate samples have been taken, test the results by comparing the observed range, c, with the theoretical value, C, which would be obtained if the correct number of units was sampled. Calculate C from the equation

$$C = kA$$

where A is the required accuracy and the value of k is obtained from table 17.

Calculate the ratio C/c :

a) If C/c is less than 0,6, too few units have been sampled;

b) If C/c is between 0,6 and 1,8, there is no evidence to suggest that the number of units sampled is incorrect;

c) If C/c is greater than 1,8, too many units have been sampled.

The decision **must** be made on the results of ten samples, irrespective of whether it is desired to know the precision of each individual sample, or of the mean of any number of units sampled.

If condition b) applies, test the **number of increments** using the procedure given in C.3.6.

When testing the number of units, but not taking duplicate samples, the procedure is the same as the above except that the first ten sample values should be used instead of the ten mean values of the duplicate samples.

1) See C.2.3.

C.3.5 Number of units — Adjustment

If condition a) or c) applies, more or less units must be sampled and there is no point in adjusting the number of increments; adjust the **number of units** in accordance with table 18. If the adjustment to the number of units indicates that all units should be sampled, adopt continuous sampling. Continue with this procedure of checking the number of units and then, if necessary, checking the number of increments until two successive sets of ten samples fulfil the conditions of C.3.4 b) and C.3.6 b).

C.3.6 Testing results — Number of increments

When indicated by C.3.4 b), examine the results of ten duplicate samples and calculate the ratio c/d. This is used to check whether the correct number of increments has been taken from each unit as follows :

a) If the ratio c/d is greater than 9,3, too many increments have been taken from each unit;

b) If the ratio c/d is between 9,3 and 2,3, there is no evidence that the number of increments taken from each unit is incorrect;

c) If the ratio c/d is less 2,3, too few increments have been taken from each unit.

C.3.7 Number of increments — Adjustment

If condition b) applies, take a further series of ten duplicate samples; if condition a) or c) applies, adjust the number of increments according to table 19 and take a further series of ten duplicate samples. Continue duplicate sampling until two successive sets of ten samples give values of the ratio c/d lying between 2,3 and 9,3 and also satisfy the condition of C.3.4 b). Thereafter duplicate sampling may be carried out as a check, as described in C.3.8.

C.3.8 Check procedure

After the initial period, the values for all samples should be listed and the range, c, calculated for each group of ten successive samples. If some of the samples have been taken in duplicate the mean of the duplicate values is taken as the sample value.

For each group, compare the range, c, with the last mean difference of ten successive duplicate samples, d. If c/d lies outside the prescribed limits of 2,3 and 9,3, adjust the number of increments and resume full duplicate sampling until two successive sets of ten duplicate samples give values of c/d lying between 2,3 and 9,3.

If c/d remains satisfactory, compare c with C; if C/c lies outside the limits of 0,6 and 1,8, compare c with the previous values for c. If it differs from previous values by a factor of 3 or more, then it is probable that the natural variability of the coal has changed. In this case, use table 18 to calculate the number of units to be sampled.

If, on the other hand, c differs from previous values by a factor of less than 3, the last few values of c should be averaged before calculating the number of units to be sampled.

If only the number of units has been changed, but not the number of increments, there is no need to revert to full duplicate sampling.

When ten duplicate values have been collected, calculate a new figure for d and compare it with the latest value of c. If the ratio c/d is within the limits of 2,3 and 9,3, the new value of d can be used for testing.

C.3.9 Intermittent sampling of consistent coals

If, on studying the ash contents of a set of ten duplicate samples, it is found that the range, c is less than 3 %, it is recommended that the c/d ratio be not used but that a fixed standard of precision for each sample be aimed for. This may be conveniently fixed at ± 1 %, so that the D/d test for continuous sampling may be used (see C.2.4) with $D = 0,8$.

C.3.10 Application to other characteristics

The test described in C.3.4 and C.3.6 can be used for any characteristic : table 17 requires no modification.

C.3.11 Example 1

A grade of coal was being sampled at a colliery once a week, samples of 24 increments being taken from units of approximately 20 tonnes, the total daily tonnage being 250 tonnes; i.e. the proportion of units being sampled each week was $\frac{20}{250 \times 6} = 0,013$.

The quarterly ash figure was required to within ± 0,5 %.

The results of the first ten duplicate samples, taken so that each day of the week and each time of the day was represented, are given in table 16.

For 15 samples per quarter and a proportion of 0,013 of the units being sampled, k (table 17) would be 6,0, making $C = 3,0$ % ash, and $C/c = 0,29$. This shows that too few units were sampled. Therefore the units to be sampled should be increased by 50 % (table 18). For convenience, it was arranged to take samples on 2 days each week.

A second set of ten duplicate samples were taken, two samples being taken each week and each sample consisting of 24 increments. The results of this set of ten duplicate samples showed that the new number of units sampled was satisfactory, but that too many increments were being taken from each unit. Therefore the number of increments in each sample was reduced to 16. The results of the next two sets of ten duplicate samples were tested and showed that this scheme (two samples per week, 16 increments per sample) was satisfactory.

C.3.12 Example 2

In the example given in C.2.8, continuous sampling was carried out at a plant to provide a precision of ± 0,5 % ash at the end of a month.

It was considered that continuous sampling unnecessarily occupied a man's full time and it was decided that a sample should be collected for a period of 1 h twice a day, i.e., for one-quarter of a coaling period, and that each sample should contain half the number of increments previously taken for a full day's sample. This was carried out experimentally for 2 weeks, the following results being obtained (ten samples were taken each week, in such a manner that each part of the working shift was represented).

1st week		2nd week	
c	= 3,5 % ash	c	= 5,0 % ash
d	= 1,3 % ash	d	= 0,8 % ash
c/d	= 2,6	c/d	= 6,2
k	= 12,6	k	= 12,6
C	= 6,3 % ash	C	= 6,3 % ash
C/c	= 1,8	C/c	= 1,3

The value of k is that given in table 17 corresponding to 50 samples per month and a proportion of 0,3 of units sampled (this entry is the nearest to the actual conditions, i.e. 40 samples per month and a proportion of 0,25; an interpolated value for the actual conditions would be 11,0).

It is seen that the value of C/c is more than 0,6 in both cases so that the final precision may be assumed to be ± 0,5 % ash or better.

Also both values of c/d are within the limits; thus it appears that taking two samples a day for a period of 1 h provides the precision required. Furthermore, it appears that no greater total mass of sample need be collected than if continuous sampling were carried out.

C.4 SINGLE CONSIGNMENT

C.4.1 Outline of procedure

An estimate of the precision achieved by sampling a single consignment can be obtained provided that at least six replicate sub-samples have been taken. The range between the highest and lowest replicate values is calculated. This observed difference is then compared with the theoretical value that would be obtained if sampling were of the correct precision, so that it is possible to say whether the correct precision has been achieved or not. Alternatively, it is possible to calculate the actual precision which has been obtained.

C.4.2 Recording results

Record the results as in table 20 and calculate the range, w, the difference between the highest and lowest sub-sample values. The squares of the values need not be calculated unless the procedure of C.4.4 is to be used.

C.4.3 Testing results

Compare the range, w, with the lowest and highest values that would be expected if the desired precision was achieved. These limiting values, W_L and W_U, are determined from the equations :

$$W_L = g_1 A$$
$$W_U = g_2 A$$

where A is the required precision and g_1 and g_2 are constants that depend on the number of replicate samples taken; the values of g_1 and g_2 are obtained from table 21.

a) If w is below the lower limit W_L, it may be assumed that a higher precision than that desired has been obtained. This does not call for any action at the time but if a consignment of the same coal is received later smaller number of increments may be taken. The number originally taken should be reduced by 33 %.

b) If w is between the two limits it may be assumed that the desired precision has been obtained and no further action is necessary.

c) If w is greater than the higher limit W_U, then the desired precision has not been obtained and the actual precision achieved may be estimated by the method of C.4.4. If the same coal is received again, it will be necessary to increase the number of increments and the number originally taken should be increased by 50 %.

C.4.4 Determination of precision achieved

Calculate the squares of the sub-sample values and sum them as shown in the example of table 20. Calculate the standard deviation S of the mean from :

$$S = \sqrt{\frac{1}{n\,(n-1)}\left(G - \frac{M^2}{n}\right)}$$

where

n is the number of replicate sub-samples;

M is the sum of the sub-sample values;

G is the sum of squares of the sub-sample values.

The precision of the mean is given by $\pm tS$ where t is obtained from table 22 using $f = (n - 1)$.

In the particular case of six sub-samples, the precision becomes

$$\pm 0{,}47\sqrt{G - \frac{M^2}{6}}$$

C.4.5 Comparison of the two methods of calculation

The methods of C.4.4 may have to be used in disputes or in other special cases, when the methods of C.4.3 have shown that the required precision has not been attained. On most occasions, however, the methods of C.4.3 will be appropriate.

C.4.6 Application to ships

These tests can be used when several holds of a ship are sampled separately. However, it is essential that the figures used to test or estimate the precision must be obtained from at least six separate sample values.

C.4.7 Example

A consignment of washed smalls in a ship, nominally of 16 % ash, was sampled by taking six samples, each of 64 increments. The results are shown in table 20. The required precision was ± 0,5 %. The values of g obtained from table 21 are $g_1 = 1,2$, $g_2 = 4,9$ and therefore $W_U = 2,45$ and $W_L = 0,60$. The range, w, is 1,9 and thus lies between the limits 2,45 and 0,60. We therefore assume that the sample has been collected to the required precision of ± 0,5 %.

If, on the other hand, we do not use the information about the expected precision, the precision of the mean is calculated as follows :

$$\pm 0,47 \sqrt{1\,613,19 - \frac{(98,3)^2}{6}}$$

$$= \pm 0,47 \sqrt{1\,613,19 - 1\,610,48}$$

$$= \pm 0,8$$

The precision of ± 0,8 % is thus worse than the ± 0,5 % assumed by the former approach. In the absence of further information the figure of ± 0,8 % should be quoted; but the first approach shows that the precision of ± 0,8 % is not sufficiently different from the desired precision of ± 0,5 % to assume that the desired precision was not obtained. There is thus no contradiction between the methods of approach. Apparent difficulties of this type are inevitable when basing estimates of error on so few degrees of freedom.

C.5 DIRECT CALCULATION OF PRECISION

Circumstances may arise when it is necessary to estimate the precision achieved rather than to compare it with an expected value; for example to determine the overall reliability of a sampling procedure. Results are required for this purpose from at least ten pairs of duplicate samples having the same sampling characteristics. The standard deviation of a single result is calculated from

$$S = \sqrt{\frac{1}{2(n-1)} \left[\Sigma d^2 - \frac{(\Sigma d)^2}{n} \right]}$$

where

S is the standard deviation of a single result;

d is the difference between pairs of duplicates;

n is the number of pairs.

The precision of a single result is calculated from :

$$A = \pm tS$$

where

A is the precision of a single result;

t is Student's t, numerical values of which at the 95 % probability level are given in table 22.

TABLE 13 – **Results of continuous duplicate sampling – Ash content, %**

Sample No.	Duplicate values		Mean sample value	Difference between duplicates
	Higher	Lower		
1	11,1	10,5	10,8	0,6
2	12,4	11,9	12,1	0,5
3	12,5	12,2	12,4	0,3
4	10,6	10,3	10,4	0,3
5	12,5	11,6	12,1	0,9
6	12,0	11,8	11,9	0,2
7	12,2	11,8	12,0	0,4
8	10,8	10,0	10,4	0,8
9	8,2	7,9	8,1	0,3
10	10,8	10,3	10,5	0,5
TOTALS	$P = 113,1$	$Q = 108,3$	$R = 110,7$	$T = 4,8$

Mean $= R/10 = 11,1$

Mean difference between duplicates $d = T/10 = 0,48$

(A check on the calculations can be carried out since since $P - Q = T = 4,8$ and $P + Q = 2R = 221,4$)

TABLE 16 – **Results of duplicate intermittent sampling – Ash content**

Sample No.	Duplicate values		Mean sample values	Difference between duplicates
	Higher	Lower		
1	11,0	10,8	10,9	0,2
2	10,4	10,0	10,2	0,4
3	20,0	19,8	19,9	0,2
4	15,5	15,3	15,4	0,2
5	15,1	14,9	15,0	0,2
6	18,6	16,2	17,4	2,4
7	16,6	14,2	15,4	2,4
8	19,7	18,0	18,9	1,7
9	17,1	14,4	15,7	2,7
10	9,7	9,2	9,5	0,5
Totals	153,7 (H)	142,8 (L)	148,3 (M)	10,9 (K)

Mean $= M/10 = 14,8$ %

Range of sample values, $c = 19,9 - 9,5 = 10,4$ %

Mean difference between duplicates, $d = K/10 = 1,09$ %

(A check on the calculations can be carried out since $H - L = K$ and $H + L = 2M$)

TABLE 14 – **Continuous sampling – Values of D**

Precision required	Number of units for which the stated precision is needed										
	1	2	3	4	5	10	15	20	25	30	50
± 0,25	0,20	0,28	0,35	0,40	0,45	0,63	0,77	0,89	1,0	1,1	1,4
± 0,5	0,4	0,6	0,7	0,8	0,9	1,3	1,5	1,8	2,0	2,2	2,8
± 0,75	0,6	0,8	1,0	1,2	1,3	1,9	2,3	2,7	3,0	3,3	4,2
± 1,0	0,8	1,1	1,4	1,6	1,8	2,5	3,1	3,6	4,0	4,4	5,6
± 1,5	1,2	1,7	2,1	2,4	2,7	3,8	4,6	5,4	6,0	6,6	8,5
± 2,0	1,6	2,3	2,8	3,2	3,6	5,0	6,2	7,1	8,0	8,8	11,3

TABLE 17 – **Values of k**

Proportion of units sampled	Number of units sampled in the period being considered										
	1	2	3	4	5	10	15	20	25	30	50
0,05	1,5	2,2	2,7	3,1	3,4	4,9	6,0	6,9	7,7	8,4	10,9
0,10	1,6	2,3	2,8	3,2	3,6	5,1	6,2	7,2	8,0	8,8	11,4
0,30	1,8	2,5	3,1	3,6	4,0	5,6	6,9	8,0	8,9	9,8	12,6
0,50	2,0	2,8	3,5	4,0	4,5	6,4	7,8	9,0	10,1	11,0	14,2
0,60	2,2	3,1	3,8	4,4	4,9	6,9	8,4	9,7	10,9	11,9	15,4
0,70	2,4	3,4	4,1	4,8	5,3	7,5	9,2	10,7	11,9	13,1	16,9
0,80	2,7	3,8	4,6	5,3	6,0	8,4	10,3	11,9	13,3	14,6	18,8
0,90	3,1	4,4	5,3	6,2	6,9	9,7	11,9	13,8	15,4	16,9	21,8

TABLE 15 – **Continuous sampling – Adjusting the number of increments**

D/d greater than 2,0		D/d less than 0,67	
D/d	Decrease number of increments by	D/d	Increase number of increments by
2,0 to 2,6	33 %	0,67 to 0,50	50 %
> 2,6	50 %	< 0,50	100 %

TABLE 18 – **Intermittent sampling – Adjusting the number of units**

$C/c > 1,8$	Decrease number of units by 33 %	$C/c < 0,6$	Increase number of units by 50 %

TABLE 19 — **Intermittent sampling — Adjusting the number of increments**

$c/d > 9,3$		$c/d < 2,3$	
c/d	Decrease number of increments by	c/d	Increase number of increments by
$< 11,2$	33 %	$> 1,8$	50 %
$\geqslant 11,2$	50 %	$\leqslant 1,8$	100 %

TABLE 20 — **Results of consignment sampling — Ash content**

Sub-sample No.	Sub-sample value	(Sub-sample value)^2
1	15,3	234,09
2	17,1	292,41
3	16,5	272,25
4	17,2	295,84
5	15,8	249,64
6	16,4	268,96
Totals	$M = 98,3$	$G = 1\,613,19$

Mean $= M/n = 16,4$ %

Range of sub-sample values, $w = 17,2 - 15,3 = 1,9$ %

Sum of sub-sample values, $M = 98,3$

Sum of squares of sub-sample values, $G = 1\,613,19$

TABLE 21 — **Values of g**

n	6	7	8	9	10
g_1	1,2	1,5	1,8	2,1	2,4
g_2	4,9	5,4	5,9	6,4	6,9

TABLE 22 — **Values of t**

f	5	6	7	8	9	10	15	20	25	50	∞
t	2,57	2,45	2,37	2,31	2,26	2,23	2,13	2,09	2,06	2,01	1,96

ANNEX D

METHODS OF CHECKING SAMPLE PREPARATION ERRORS

D.1 INTRODUCTION

As described in clause 9, sample preparation should be carried out in two[1] (or possibly three) stages, each stage consisting of a reduction in particle size, possibly mixing, and division of the sample into two parts, one of which is retained and one rejected. All the errors occur in the course of the two divisions, in the selection of the final 1 or 2 g of 0,2 mm size and in the determination of the ash of this portion. The same remarks apply to all other determinations, but for convenience the remainder of this annex refers to ash only. Moreover, if the variance is satisfactory for ash it will normally be so for the other characteristics of the proximate and ultimate analysis. Errors in moisture and calorific value, however, should be checked. All characteristics may be checked if desired.

This method of checking the precision of sample preparation is designed to estimate the random errors arising in the various stages of the process; the important factors are the size distribution of the sample after crushing and before division, and the mass remaining after division. The errors are expressed in terms of variance. Separate tests are necessary to ensure that bias is not introduced either by contamination or losses during the sample preparation process. The procedure described is simple to carry out, and the results are simple to analyse, but it is not the best statistical design; such a design is shown in figure 34, but the analysis required is different and considerably more complicated; if it is desired to apply this scheme, the advice of a statistician should be sought.

It is assumed that the original sample weighs X kg. This is milled to L mm, then divided to Y kg. This is the first stage. The sample is then milled to 0,2 mm and divided to between 60 and 150 g. This is the second stage. A diagram of the procedure is shown in figure 32.

D.2 REQUIRED VALUE OF VARIANCE OF SAMPLE PREPARATION AND ANALYSIS

The methods of sample preparation recommended in clause 9, using the masses specified, should achieve for the most variable coals a variance of sample preparation of $0{,}04\,A^2$ or less, a variance of $0{,}01\,A^2$ being allowed for analysis; where A is the overall precision of sampling, sample preparation and analysis. This gives a total variance of $0{,}05\,A^2$.

D.3 TO CHECK PROCEDURE AS A WHOLE

The first step is to check that the overall variance of preparation and analysis does not exceed the value of $0{,}05\,A^2$ required for this International Standard. The exact value of $0{,}05\,A^2$ will not be obtained and the method provides a test of whether the difference between the determined value and $0{,}05\,A^2$ is significant.

This is done by taking two portions at the first division of the sample; these are thereafter treated entirely separately to give the two analysis samples (figure 32). The two samples provide an unbiased estimate of the variance of sample preparation and analysis. Ten pairs of analysis samples are obtained in this way.

Let the mean difference between the ten pairs of results be h; then h should lie between $0{,}37\,A$ and $0{,}13\,A$.

Provided that **two successive sets** of ten duplicate samples fall between these upper and lower limits, it may be assumed that the procedure is satisfactory.

If the difference is below $0{,}13\,A$, the variance is low, but no adjustment is necessary since it is always desirable to have the variance as low as possible.

If the mean difference is greater than $0{,}37\,A$, the variance is too high and the values given in table 10 (giving the mass of sample to be retained at various stages of the sample preparation process) will not be valid. Therefore, the variance of the errors arising at each stage should be estimated as described in the following clause, so that steps may be taken to improve the procedures as shown to be necessary.

D.4 TO CHECK STAGES SEPARATELY

D.4.1 Procedure

The following procedure[2] should be used to measure errors at different stages if the test described in clause D.3 shows that the variance of the procedure as a whole is too high. The same procedure may be used to measure the errors at different stages when installing a new piece of equipment or when considering the introduction of a new kind of procedure. Particular care is needed in the interpretation of the results, especially in estimating the errors at each stage of division.

1) Hereinafter assumed to be two. If three stages are used, the checking procedure is similar, but the advice of a statistician should be sought.

2) Based on that given by R.C. Tomlinson, *Journal of the Institute of Fuel*, London 1954, **27**, 515.