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Hard coal — Sampling

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1988 was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels,* and circulated to the Member Bodies in June 1971.

It has been approved by the Member Bodies of the following countries :

Austria	India	Switzerland
Belgium	Ireland	Turkey
Canada	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.A.
Denmark	Portugal	Yugoslavia
Egypt, Arab Rep. of	Romania	
Germany	Sweden	

The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

France
South Africa, Rep. of

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Annexes

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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.