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**ISO 18283**

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# Hard coal and coke — Manual sampling

*Houille et coke — Échantillonnage manuel*

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

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

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# Introduction

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

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

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

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

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

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

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a) incorrect location/timing of increments,

b) incorrect delimitation and extraction of increments,

c) loss of integrity of increments after extraction.

Methods for measuring bias are described in this International Standard.

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# Hard coal and coke — Manual sampling

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

## 1 Scope

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

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

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

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## 2 Normative references

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

ISO 579, *Coke — Determination of total moisture*

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

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

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

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

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

## 3 Terms and definitions

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

**3.1**
**air-drying**
process of bringing the moisture content of the sample near to equilibrium with the atmosphere in the area in which further reduction and division of the sample are to take place

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

**3.2**
**bias**
systematic error that leads to the average value of a series of results being persistently higher or persistently lower than those that are obtained using a reference sampling method

**3.3**
**common sample**
sample collected for more than one intended use

**3.4**
**continuous sampling**
taking of a sample from each consecutive sub-lot so that increments are taken at uniform intervals whenever the fuel is handled at the point of sampling

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**3.5**
**divided increment**
part obtained from the division of the increment in order to decrease its mass

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

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**3.6**
**fixed-mass division**
method of sample division in which the mass retained is predetermined and independent of the mass of the feed

**3.7**
**fixed-ratio division**
method of sample division in which the division ratio is predetermined, i.e. the mass of sample retained is a fixed proportion of the mass of the feed

**3.8**
**fuel**
hard coal or coke

**3.9**
**general analysis test sample**
sample prepared to pass a sieve of nominal size of openings 212 µm in accordance with ISO 3310-1, used for the determination of most chemical and some physical characteristics

**3.10**
**increment**
portion of fuel extracted in a single operation of the sampling device

**3.11**
**intermittent sampling**
taking of samples from only certain sub-lots of fuel

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

NOTE A lot can be divided into sub-lots.

**3.13**
**manual sampling**
extraction of increments by human effort

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

**3.15**
**mechanical sampling**
extraction of increments by mechanical means

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

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

**3.17**
**nominal top size**
aperture size of the smallest sieve in the range included in the R 20 Series (see ISO 565, square hole) on which not more than 5 % of the sample is retained

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

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

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

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

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

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

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

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

### 3.24
**sample division**

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

### 3.25
**sample preparation**

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

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

### 3.26
**sample reduction**

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

### 3.27
**size analysis sample**

sample taken specifically for particle size analysis

### 3.28
**standard deviation**

square root of the variance

### 3.29
**stratified random sampling**

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

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### 3.30
**sub-lot**

part of a lot for which a test result is required

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### 3.31
**systematic sampling**

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

### 3.32
**test sample**

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

### 3.33
**time-basis sampling**

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

### 3.34
**unit mass**

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

### 3.35
**variance**

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

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# 4 Establishing a sampling scheme

## 4.1 General

### 4.1.1 Sampling

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

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

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

### 4.1.2 Sampling scheme

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

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

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b) Define the lot.

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

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

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

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

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

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

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

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

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

### 4.1.3 Parameters

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

a) variability of the fuel;

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

c) number of increments comprising each sample;

d) mass of sample relative to nominal top size.

### 4.1.4 Sampling methods

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

a) continuous sampling;

b) intermittent sampling.

## 4.2 Sampling methods

### 4.2.1 Continuous sampling

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

### 4.2.2 Intermittent sampling

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

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## 4.3 Design of the sampling scheme

### 4.3.1 General

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

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

a) fuel to be sampled and considerations for sampling;

b) bias of results;

c) precision of results;

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d) lot size and number of sub-lots;

e) method of sampling;

f) requirements for test samples;

g) number of increments;

h) mass of sample.

### 4.3.2 Fuel to be sampled and considerations for sampling

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

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

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

b) location and the handling system;

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c) transport means/carriers;

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

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e) human safety risks.

### 4.3.3 Division of lots

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

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

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

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

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

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

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

### 4.3.4 Bias of results

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

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

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

### 4.3.5 Precision of results

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

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

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

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

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### 4.3.6 Requirements for test samples

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

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

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

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

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

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

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## 4.4 Precision of sampling

### 4.4.1 General

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

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

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

### 4.4.2 Precision and total variance

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

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

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

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where

$s$ is the sample estimate of the population standard deviation;

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

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

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

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

where

$V_{\mathrm{I}}$ is the primary increment variance;

$V_{\mathrm{PT}}$ is the preparation and testing variance;

$n$ is the number of primary increments in the sample.