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Coal and coke — Manual sampling

Houille et coke — Échantillonnage manuel

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 27, *Coal and Coke*, Subcommittee SC 4, *Sampling*.

This second edition cancels and replaces the first edition (ISO 18283:2006), which has been technically revised. It also incorporates the Technical Corrigendum ISO 18283:2006/Cor.1:2006.

The main changes are as follows:

— Removal of any reference to intermittent sampling. Only continuous sampling is permitted.

— Discussion of the need to eliminate bias prior to discussing precision.

— Deletion of the separate tables on calculated numbers of increments.

— Deletion of the table on reference increment mass.

— Separation of tables for minimum sample masses for coal and coke.

— Removal of the table for reduced minimum sample mass for large sizes of coal and coke.

— Inclusion of manual sampling from a moving conveyor, provided a risk assessment is conducted at the outset and that this type of sampling is only permitted on a slow-moving belt or at low flow rates. Furthermore, at higher flow rates, mechanical assistance is necessary to ensure that primary increments can be collected safely.

— Restriction of the type of probes that can be used.

— Deletion of augers for manual sampling.

— Inclusion of a photograph of a gated riffle.

— Exclusion of sampling of large fuels in excess of the nominal top sizes in Tables 1, 2 and 4, because it is not practical.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

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

The fundamental requirements of sampling are that all particles of the coal or coke in the lot are accessible to the sampling instrument and thus have a non-zero chance of being selected, and that each individual particle of equal mass 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 document are intended to obtain the most representative sample that can be safely achieved. Manual sampling should only be applied if no possibility for mechanical sampling exists.

The purpose of taking and preparing a sample of coal or coke is to provide a test sample that, when analysed, provides test results representative of the lot or sub-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 coal or coke portions known as primary increments. The primary increments are then combined into a sample. From this sample, the required number and types of test samples are prepared by a series of processes jointly known as sample preparation.

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

a) incorrect location/timing of increments,

b) incorrect delimitation and extraction of increments,

c) particle size segregation at the point of sampling,

d) loss of integrity of increments after extraction.

Methods for measuring bias are described in ISO 13909-8.

Coal and coke — Manual sampling

WARNING — This document can involve hazardous materials, operations and equipment, and does not purport to address all the safety issues associated with its use. It is the responsibility of the user of this document to establish appropriate health and safety practices.

1 Scope

This document provides the basic terms used in manual sampling of coal and coke and describes the general principles of sampling. It provides 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, and applies to manual sampling during the transfer of coal or coke. Guidelines for manual sampling 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.

This document covers sampling of brown coals and lignites, but does not include sampling from coal seams, for which guidance is given in ISO 14180. Mechanical sampling of coal and coke is covered in ISO 13909.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Hard coal — Determination of total moisture*

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

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 terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

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 1 to entry: 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 every consecutive *sub-lot* (3.30) so that *increments* (3.10) are taken at uniform intervals over the entire *lot* (3.11) being handled

3.5
cut
see *increment* (3.10)

3.6
divided increment
part obtained from the division of the *increment* (3.10) in order to decrease its mass

Note 1 to entry: Such division can be done with or without prior size reduction.

3.7
fixed-mass division
method of sample division in which the mass retained is predetermined and independent of the mass of the feed

3.8
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

3.9
general analysis test sample
sample prepared to pass a sieve of nominal size of openings of 212 µm used for the determination of most chemical and some physical characteristics

3.10
increment
portion of coal or coke extracted in a single operation of the sampling device

Note 1 to entry: Cut is an equivalent term.

3.11
lot
defined quantity of coal or coke for which the quality is to be determined

Note 1 to entry: A lot can be divided into sub-lots.

3.12
manual sampling
extraction of *increments* (3.10) by human effort

3.13
mass-basis sampling
taking of *increments* (3.10) whereby the position of each *increment* (3.10) to be extracted from the stream of coal or coke is measured by a mass interval of stream flow and the *increment* (3.10) mass is fixed

3.14
mechanical sampling
extraction of *increments* (3.10) by mechanical means

3.15
moisture sample
sample taken specifically for the purpose of determining total moisture

Note 1 to entry: For coke, this sample can also be used for general analysis.

3.16
nominal top size
aperture size of the smallest sieve in the range included in the R 20 Series on which not more than 5 % of the mass of the sample is retained

Note 1 to entry: See ISO 565, square hole.

3.17
physical sample
sample taken specifically for the determination of physical characteristics, e.g. physical strength indices or size distribution

3.18
precision
closeness of agreement between independent test results obtained under stipulated conditions

Note 1 to entry: This is often defined using an index of precision, such as 2 standard deviations.

Note 2 to entry: 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.

3.19
primary increment
increment (3.10) extracted at the first stage of sampling, prior to any sample division and/or sample reduction

3.20
random sampling
extracting of *increments* (3.10) at random mass or time intervals

3.21
replicate sampling
extracting, at intervals, of *increments* (3.10) that are combined in rotation into different containers to give two or more samples of approximately equal mass

3.22
representative sample
sample collected in such a manner that the analyses, size distribution and moisture content represent that of the *lot* (3.11)

3.23
sample
quantity of coal or coke, 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 1 to entry: Sample preparation covers mixing, particle size reduction, sample division and sometimes *air-drying* (3.1) of the sample and may be performed in several stages

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

3.27
size analysis sample
sample taken specifically for particle size analysis

3.28
standard deviation
square root of the variance

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

3.30
sub-lot
part of a *lot* (3.11) for which a test result is required

3.31
systematic sampling
extracting of *increments* (3.11) at uniform 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 coal or coke 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
variance
measure of dispersion, which is the sum of the squared deviations of observations from their average divided by one less than the number of observations

4 Establishing a sampling scheme

4.1 General

4.1.1 Sampling

Mechanical sampling of coal and coke in accordance with ISO 13909-2, ISO 13909-3 and ISO 13909-5 is the preferred method. However, where this is not possible, manual sampling may be conducted. The recommended method for manual sampling of coal and coke is while it is being transferred, e.g. loading or unloading of ships, barges, wagons, rail cars and trucks, transferring coal or coke using fixed/moveable conveyors and/or stackers, or during the formation of or reclaiming from stockpiles. For safety and practical reasons, manual sampling of coal or coke being transferred 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 sampling personnel. 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 sampling personnel. These instructions, which should preferably be set out in writing, should be prepared by the sampling supervisor from the information given in this document.

4.1.2 Sampling scheme

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

a) define the quality parameters to be determined and the types of samples required;

b) define the lot;

c) select or assume the required overall precision for the lot (see 4.3.2);

d) determine or assume the variability of the coal or coke (see 4.3.3 and, if relevant, 4.3.4) and the variance of preparation and testing (see 4.3.5);

e) ascertain the nominal top size of the coal or coke for the purpose of determining the mass of increment and sample (see 4.3.6.3 and 4.3.7);

f) 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;

g) select the sampling device (see Clause 6);

h) establish the number of sub-lots and the number of increments per sub-lot required to attain the desired precision (see 4.3.6);

i) determine the method of combining the increments into samples and the method of sample preparation (see Clause 8);

j) define the sampling interval in terms of time or mass (see Clause 5);

k) determine where to collect 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 coal or coke;

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

c) number of sub-lot samples to be constituted for the lot;

d) number of increments comprising each sub-lot sample;

e) mass of sample relative to nominal top size.

4.1.4 Sampling methods

In this document, only continuous sampling methods are considered.

4.2 Design of the sampling scheme

4.2.1 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 document. The following items in the following list and described in 4.2.2 to 4.2.6 should be considered by the supervisor when compiling instructions:

a) coal or coke to be sampled and considerations for sampling;

b) lot size and number of sub-lots;

c) method of sampling;

d) requirements for test samples;

e) number of increments per lot or sub-lot;

f) mass of sample;

g) precision of results;

h) bias of results.

4.2.2 Coal or coke to be sampled and considerations for sampling

The first stage in the design of the scheme is to identify the coal or coke to be sampled. Samples may 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 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) coal or coke properties, e.g. fines, lump and, more specifically, the nominal top size; furthermore, whether dry, wet or free flowing;

b) location and the handling system;

c) transport means/carriers;

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

e) human safety risks.

4.2.3 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. The size of each sub-lot should be selected for convenience of sampling, e.g. coal or coke despatched or delivered over a period of time, a train load, a wagon load, or coal or coke produced during a certain period, e.g. a shift. For large lots, such as ocean-going vessels, it is recommended to sample in multiple sub-lots for the reasons below. It is common industry practice to limit sub-lot sizes to a maximum of 10 000 t. However, when the loading or discharge rate is less than

1 000 t/h, it is recommended that sub-lot sizes be limited to 5 000 t to avoid moisture bias due to long periods of sample collection.

Such division into a number of sub-lots may be necessary to achieve the following:

a) 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 changes of moisture due to standing or 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 of operators;

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

4.2.4 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 4.3.6 and the mass of the primary increments shall be determined as described in 4.3.6.3.

For single lots, the quality variation shall be assumed as the worst case (see 4.3.3). The precision of sampling achieved may be measured using the procedure of replicate sampling (see 4.5).

At the start of regular sampling of unknown coal or coke, the worst-case quality variation shall be assumed in accordance with 4.3.3 and 4.3.4.

If any subsequent change in precision is required, the number of sub-lots and of increments shall be changed as determined in 4.3.6 and the precision attained rechecked. The precision shall also be checked if there is any reason to suppose that the variability of the coal or coke being sampled has increased. The number of increments determined in 4.3.6 applies to the precision of the result when the sampling errors are large relative to the sample preparation and testing errors, e.g. moisture. However, in some tests, the testing errors are themselves large. In this case, it can be necessary to prepare two or more test portions from the sample and use the mean of the determinations to give a better precision.

4.2.5 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 4.3.6.3 and 4.3.7).

It may 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 due to standing (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 (see ISO 13909-8).

4.2.6 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, some tests require