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Hard coal and coke -- Mechanical sampling -- Part 6: Coke -- Preparation of test samples

Houille et coke -- Échantillonnage mécanique -- Partie 6: Coke -- Préparation des échantillons pour essai

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Hard coal and coke — Mechanical sampling —

Part 6: Coke — Preparation of test samples

Houille et coke — Échantillonnage mécanique —

Partie 6: Coke — Préparation des échantillons pour essai

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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 3.

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 part of ISO 13909 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13909-6 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

ISO 13909 cancels and replaces ISO 9411-1:1994, *Solid mineral fuels — Mechanical sampling from moving streams — Part 1: Coal* and ISO 9411-2:1993, *Solid mineral fuels — Mechanical sampling from moving streams — Part 2: Coke*, of which it constitutes a technical revision. It also supersedes the methods of mechanical sampling of coal and coke given in ISO 1988:1975, *Hard coal — Sampling* and ISO 2309:1980, *Coke — Sampling*.

ISO 13909 consists of the following parts, under the general title *Hard coal and coke — Mechanical sampling*:

- *Part 1: General introduction*
- *Part 2: Coal — Sampling from moving streams*
- *Part 3: Coal — Sampling from stationary lots*
- *Part 4: Coal — Preparation of test samples*
- *Part 5: Coke — Sampling from moving streams*
- *Part 6: Coke — Preparation of test samples*
- *Part 7: Methods for determining the precision of sampling, sample preparation and testing*
- *Part 8: Methods of testing for bias*

Introduction

The objective of sample preparation is to prepare one or more test samples from the primary increments for subsequent analysis. The requisite mass and particle size of the test sample depend on the test to be carried out.

The process of sample preparation may involve constitution of samples, reduction, division, mixing and drying or all or a combination of these.

Primary increments may be prepared individually as test samples or combined to constitute samples either as taken or after having been prepared by reduction and/or division. Samples may either be prepared individually as test samples or combined on a weighted basis to constitute a further sample.

On-line sampling and sample-preparation systems can be usefully implemented when dealing with a coke or cokes which are known to be free from handling problems, but only when this will not result in loss of moisture, contamination by the sampling equipment or size degradation of physical samples.

Hard coal and coke — Mechanical sampling —

Part 6:
Coke — Preparation of test samples

1 Scope

This part of ISO 13909 describes the preparation of samples of coke from the combination of primary increments to the preparation of samples for specific tests.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 13909. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13909 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 579:1999, *Coke — Determination of total moisture*.

ISO 687:1974, *Coke — Determination of moisture in the analysis sample*.

ISO 13909-1:2001, *Hard coal and coke — Mechanical sampling — Part 1: General introduction*.

ISO 13909-5:2001, *Hard coal and coke — Mechanical sampling — Part 5: Coke — Sampling from moving streams*.

ISO 13909-7:2001, *Hard coal and coke — Mechanical sampling — Part 7: Methods for determining the precision of sampling, sample preparation and testing*.

ISO 13909-8:2001, *Hard coal and coke — Mechanical sampling — Part 8: Methods of testing for bias*.

3 Terms and definitions

For the purposes of this part of ISO 13909, the terms and definitions given in ISO 13909-1 apply.

4 Precision of sample preparation

From the equations given in ISO 13909-7, the estimated absolute value of the precision of the result obtained for the lot at the 95 % confidence level, P_L, for continuous sampling is given by:

$$P_L = 2\sqrt{\frac{\frac{V_I}{n} + V_{PT}}{m}} \quad (1)$$

where

V_I is the primary increment variance;

n is the number of increments in the sample;

V_{PT} is the variance of preparation and testing for both off-line and on-line systems;

m is the number of sub-lots.

The procedures given in this part of ISO 13909 are designed to achieve levels of V_{PT} of 0,05 or less for moisture tests. Better levels may be expected for other chemical characteristics.

For some preparation schemes, however, practical restrictions may prevent the preparation and testing variance being as low as this. Under these circumstances, the user will have to decide whether to achieve the desired overall precision by improving the preparation scheme or by dividing the lot into a greater number of sub-lots.

The errors occurring in the various stages of preparation and analysis, expressed in terms of variance, may be checked by the methods given in ISO 13909-7.

5 Constitution of a sample

5.1 Introduction

Examples of the constitution of samples are shown in Figure 1.

Primary increments shall be taken in accordance with the procedures specified in ISO 13909-5.

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

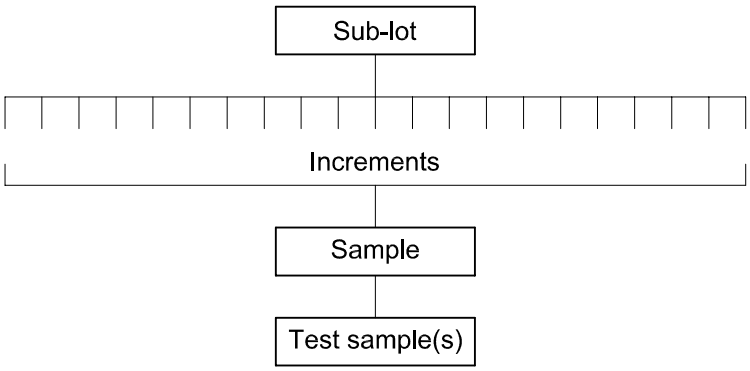
Samples may also be prepared by the combination of other samples.

The procedures for increment combination (5.2) may vary according to whether the primary increments were taken using a time-basis (5.2.1) or a mass-basis (5.2.2) sampling scheme.

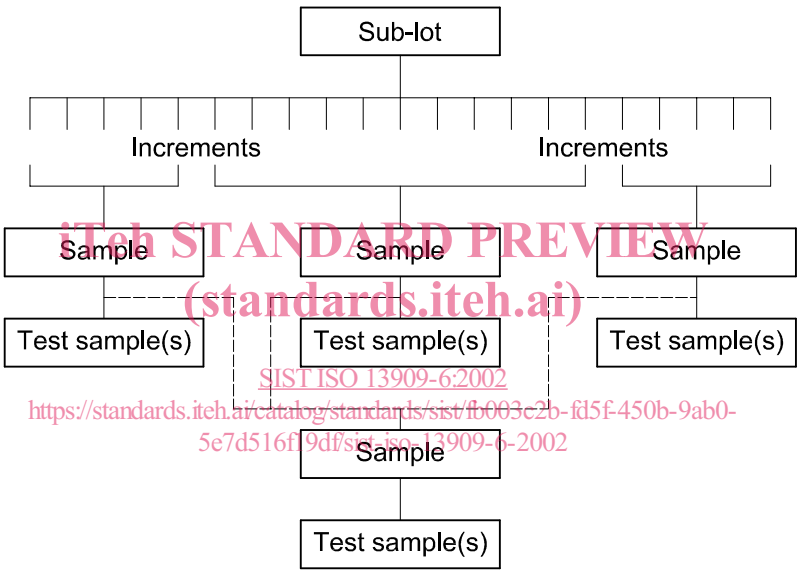
5.2 Combination of increments

5.2.1 Time-basis sampling

The mass of the primary increments shall be proportional to the flow rate at the time of sampling. The primary increments may be combined into a sample, either directly as taken or after having been prepared individually to an appropriate stage by fixed-ratio division (see clause 6).



a) Example 1



b) Example 2

Figure 1 — Examples of the constitution of samples

5.2.2 Mass-basis sampling

If the primary increments are of almost uniform mass (see note), they may be combined into a sample, either directly as taken or after having been prepared individually to an appropriate stage by fixed-ratio division (see clause 6).

NOTE Almost uniform mass has been achieved if the coefficient of variation of the increment masses is less than 20 % and there is no significant correlation between the flow rate at the time of taking the increment and the mass of the increment (see ISO 13909-5).

If the primary increments are not of almost uniform mass, they may only be combined into samples after having been divided individually by fixed-mass division (see clause 6).

5.3 Combination of samples

When combining samples, the mass of the individual samples shall be directly proportional to the mass of the coke from which they were taken in order to obtain a weighted mean of the quality characteristic for the sub-lot. Prior to combination, division shall be by fixed-ratio (see clause 6).

6 Division

6.1 General

Since the cutter aperture will normally be at least three times the nominal top size, this will result in a very large increment mass in many cases. The handling and preparation of such large increments would be either manpower or equipment intensive. Division prior to further treatment may be necessary to provide a manageable sample mass.

Sample division can be

— on-line mechanically, or

— off-line mechanically or manually.

Whenever possible, mechanical methods are preferred to manual methods to minimize human error. Examples of dividers are shown in Figure 2.

Mechanical dividers are designed to extract a part of the coke in a number of cuts of relatively small mass. When the smallest mass of the divided sample that can be obtained in one pass through the divider is greater than that required, further passes through the same divider or subsequent passes through further dividers may be necessary.

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

6.2 Mechanical methods

6.2.1 General

Mechanical sample division may be carried out on an individual increment or a sample. Where samples are for moisture or general analysis, it is permissible to install on-line crushing to a nominal top size of 16 mm followed by sample division. Division shall be either by fixed-mass division or by fixed-ratio division subject to the conditions set out in 6.2.2 and 6.2.3.

When crushing on-line, the risk of moisture loss should be considered, particularly if the coke is hot.

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

When a coke is regularly sampled under the same conditions, the precision obtained for all the required quality parameters shall be checked using the procedures of ISO 13909-7 and the minimum mass adjusted accordingly. The masses shall not be reduced, however, below the minimum requirements laid down in the relevant analysis standards.

NOTE The procedures described for fixed-ratio division are the simplest to implement. Other procedures may be used, however, provided that the mass of the divided sample is proportional to the mass of the feed, e.g. the number of cuts could be kept constant by making the feed rate of each division proportional to the mass of coke to be divided.

6.2.2 Mass of cut

The cuts shall be of uniform mass throughout the division of an increment. In order to achieve this, the flow of coke to the divider shall be uniform and the cutting aperture and speed of the cutter shall be constant. The method of feeding the divider shall be designed to minimize any segregation caused by the divider.

The cutting aperture shall be at least three times the nominal top size of the coke to be divided.