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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

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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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 4, *Sampling*.

This second edition cancels and replaces the first edition (ISO 13909-6:2001), which has been technically revised.

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 samples depend on the test to be carried out.

Examples of tests which require different masses are Shatter Index (ISO 616), Micum and Irsid Index (ISO 556), reactivity tests (ISO 18894), density (ISO 567, ISO 1013) and size distribution (ISO 728, ISO 2325).

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.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 687, *Solid mineral fuels — Coke — Determination of moisture in the general analysis test sample*

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

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

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*

ISO 21398, *Hard coal and coke — Guidance to the inspection of mechanical sampling systems*

3 Terms and definitions

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

4 Precision of sample preparation

From the formulae 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 Formula (1):

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

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 should 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 General

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.

5.2 Combination of increments

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).

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).

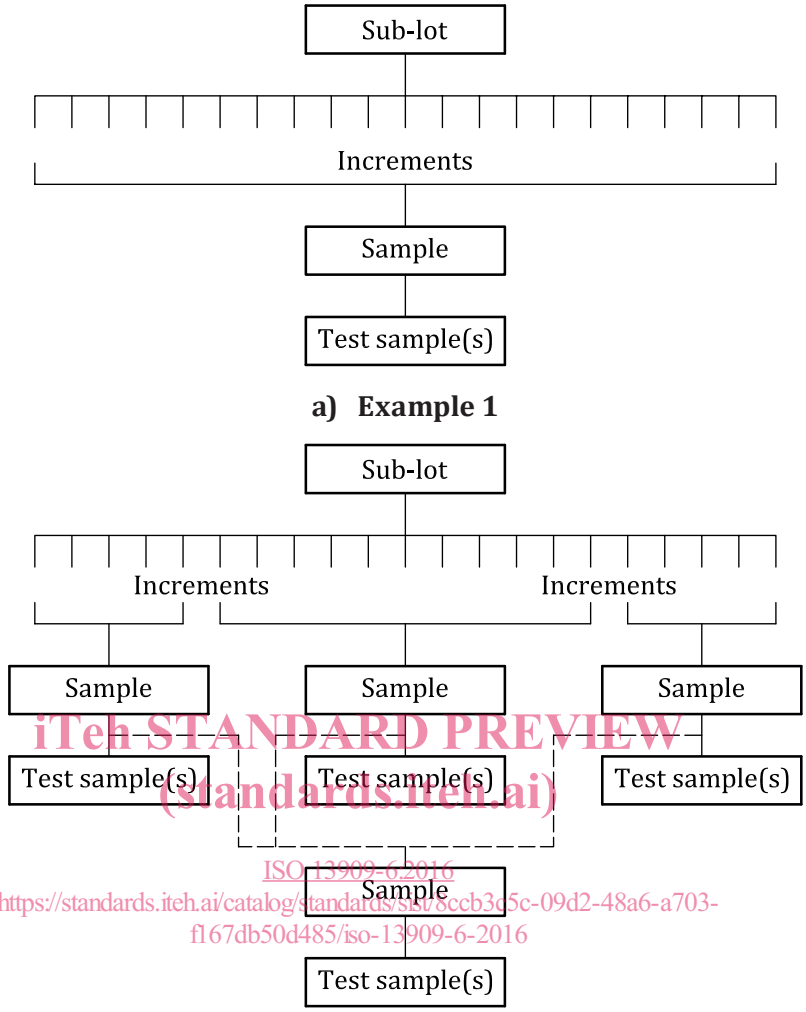


Figure 1 — Examples of the constitution of samples

6 Division

6.1 General

Since the cutter aperture will 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

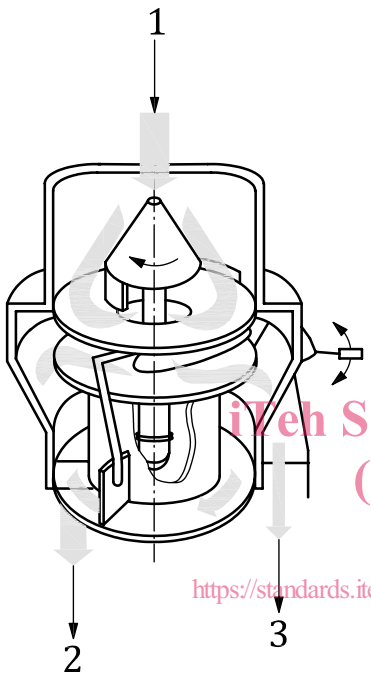
— on-line mechanically, or

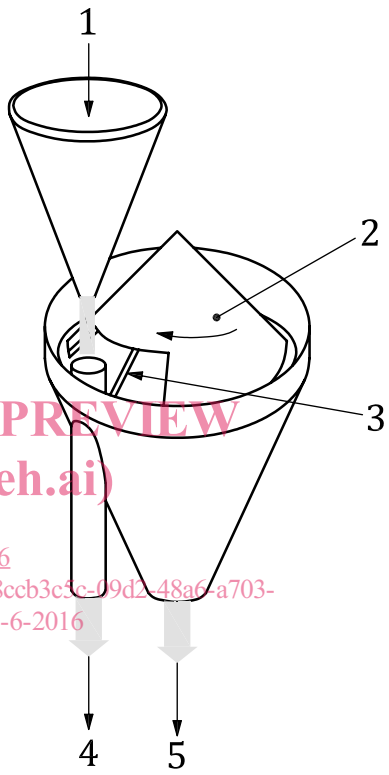
— off-line mechanically or manually.

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.





Key

1 feed
2 reject
3 divided sample

The material from a mixing container is fed by scrapers to the centre of the dividing disc. From there, it is discharged over the range of the disc through special clearing arms. The sample falls through adjustable slots into chutes; the reject is carried away through a cleaning conduit. The whole interior space is cleaned by scrapers.

a) Rotating disc type

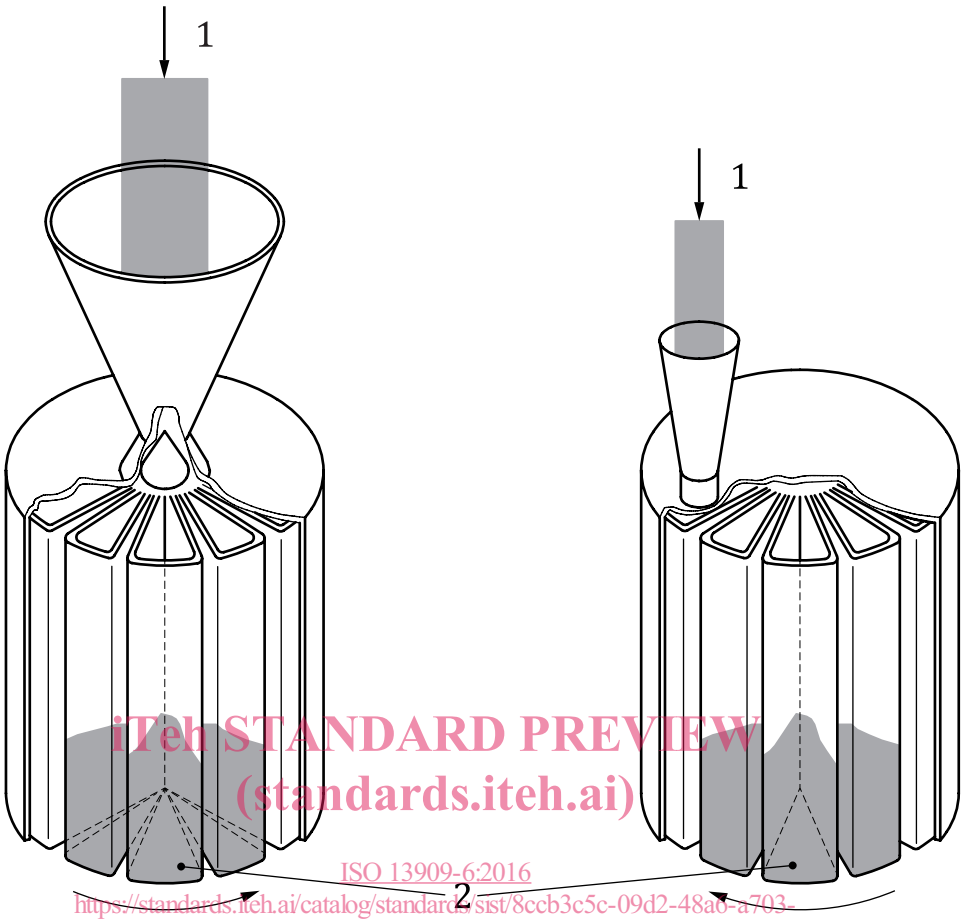
Key

1 feed
2 rotating cone
3 adjustable slot
4 divided sample
5 reject

As stream of coal is allowed to fall onto a rotating cone; the adjustable slot with lips in the cone allows the stream to fall directly onto the sample receiver for part of each revolution.

b) Rotating cone type

Figure 2 — *(continued)*



Key

1 feed
2 divided sample in rotating receivers

The coal stream flows to the hopper and this flow is intercepted by the top edge of a number of sector-shaped containers dividing the flow into equal parts. Either the hopper or the containers may rotate. The machine can be controlled for the following operations:

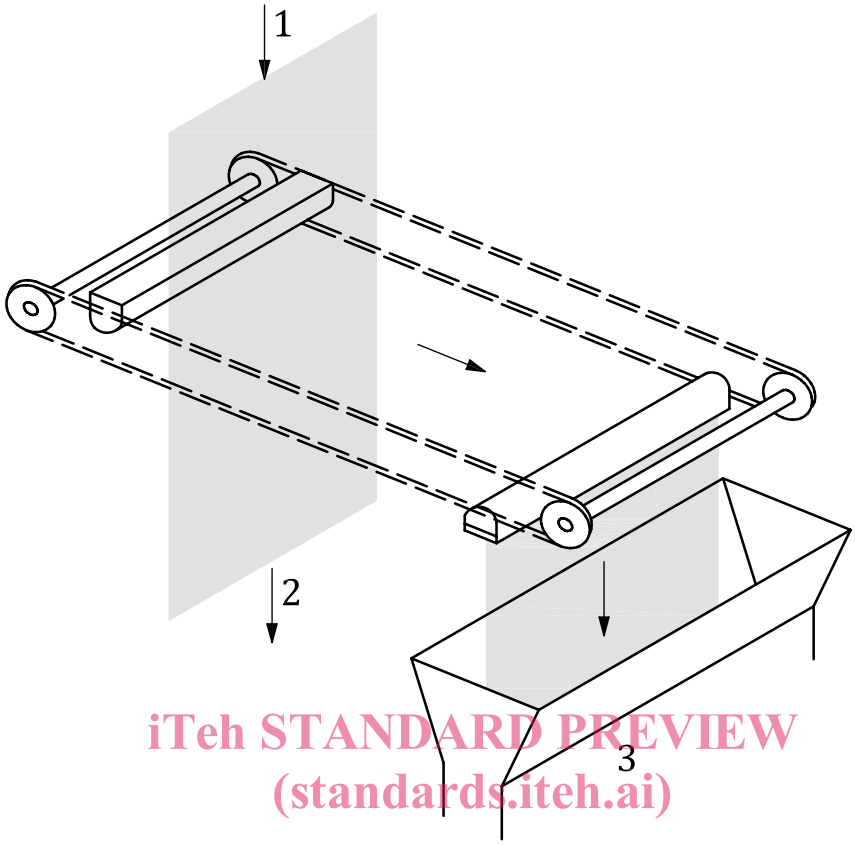
a) for dividing;

b) for collecting duplicates;

c) for collecting replicates.

c) Container type

Figure 2 — *(continued)*



Key

1 feed
2 reject
3 divided sample

A chain mechanism as shown is equipped with buckets spread at equal pitch. The buckets travel in a single direction or change direction at preset time periods. The bucket intercepts the free-falling coal stream to extract cuts which discharge the sample as the bucket inverts.

d) Chain bucket type

Figure 2 — *(continued)*