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Coal and coke — Determination of ash

Charbon et coke — Détermination des cendres

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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 document 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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This document was prepared by Technical Committee ISO/TC 27, *Coal and coke*, Subcommittee SC 5, *Methods of analysis*.

This fifth edition cancels and replaces the fourth edition (ISO 1171:2010), which has been technically revised.

The main changes are as follows:

— title and references changed to be consistent with the new name of ISO/TC 27

— editorial updates to be in line with ISO 80000-1

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

After coal or coke has been incinerated in air, ash remains. The ash is derived from inorganic complexes present in the original coal substance and from associated mineral matter. Therefore, the result of the determination is “ash” and not “ash content” as coal does not contain any ash.

The amount of sulfur retained in the ash is in part dependent on the procedure to determine the ash. No deviations are expected when following the procedure specified in this document.

Coal and coke — Determination of ash

1 Scope

This document specifies a method for the determination of the ash of coal and coke.

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 1213-2, *Solid mineral fuels — Vocabulary — Part 2: Terms relating to sampling, testing and analysis*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1213-2 apply.

ISO and IEC maintain terminology 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/

4 Principle

The test portion is heated in air at a specified rate up to a temperature of 815 °C ± 10 °C and maintained at this temperature until constant in mass.

The ash is calculated from the mass of the residue after incineration.

5 Apparatus

5.1 Balance, capable of reading to the nearest 0,1 mg.

5.2 Furnace, capable of giving a zone of uniform temperature at the levels required by the procedure and reaching these levels in the specified times.

The ventilation through the furnace shall be such as to give five to ten air changes per min.

Alternatively, two furnaces may be used: one capable of achieving an adequate zone at a uniform temperature of approximately 500 °C; and the other capable of maintaining a temperature of 815 °C ± 10 °C.

5.3 Dish, of silica, porcelain or platinum, 8 mm to 15 mm deep, of such a size that the sample loading does not exceed 0,15 g/cm^2 for coal and 0,10 g/cm^2 for coke.

5.4 Plate, for use with coke samples, made from silica or heat-resistant steel, approximately 6 mm-thick and of such a size as to slide easily into the furnace (5.2).

5.5 Desiccator or other closed container.

6 Preparation of test sample

The sample shall be the general analysis test sample (see ISO 1213-2), prepared to a nominal top size of 212 µm. Sample preparation procedures are specified in ISO 13909-4, ISO 13909-6 or ISO 18283, whatever applies.

The sample shall be well mixed and in moisture equilibrium with the laboratory atmosphere.

7 Procedure

7.1 General

The amount of sulfur retained in the ash is in part dependent on the conditions of ashing and, in order to obtain values for the ash on a comparable basis, strictly follow the procedures specified in this document.

7.2 Ash determination

Before use, silica or porcelain dishes should be heated to 815 °C ± 10 °C, maintained at this temperature for 15 min and then cooled under conditions specified for the actual determination.

Determine the mass of an empty dish (5.3) to the nearest 0,1 mg by weighing on an analytical balance (5.1). Add 1 g ± 0,1 g of the test sample in an even layer and reweigh to the nearest 0,1 mg.

Insert the dish with the sample into the furnace (5.2) at room temperature. Raise the furnace temperature evenly to 500 °C over a period of 60 min and hold at this temperature for 30 min. For brown coals, hold this temperature over a period of 60 min.

Continue heating to 815 °C ± 10 °C in the same furnace. Alternatively, transfer the dish to a second furnace, previously heated to 815 °C ± 10 °C (see 5.2). Maintain this temperature for at least 60 min.

Alternatively for coke, the dish placed on the plate (5.4) may be inserted directly into a furnace at 815 °C ± 10 °C. Maintain at this temperature for at least 60 min.

When the incineration period is complete, remove the dish plus the ash from the furnace and allow to cool on a thick metal plate for 10 min. At the end of the 10 min cooling period, transfer the dish to a desiccator (5.5) or other closed container without desiccant and allow to cool to room temperature. Determine the mass of the dish plus the ash to the nearest 0,1 mg by weighing on an analytical balance (5.1).

The container may be flushed with dry gas in order to reduce the amount of moisture adsorbed during cooling. In this case, the dish should be covered with a lid.

If there is any doubt that incineration is complete (e.g. there are visible unburned carbon particles), reheat at 815 °C ± 10 °C for further 15 min periods until the change in mass does not exceed 1 mg.

8 Expression of results

The ash mass fraction, w_A, of the sample as analysed, expressed as per cent, is given in Formula (1):

$$w_A = \frac{100\,(m_3 - m_1)}{m_2 - m_1} \qquad (1)$$

where

m_1 is the mass of the empty dish, expressed in grams;

m_2 is the mass of the dish plus the test portion, expressed in grams;

m_3 is the mass of the dish plus the ash, expressed in grams;

100 is the conversion factor from the dimension one mass fraction to per cent.

Report the result as the mean of duplicate determinations to the nearest 0,1 % mass fraction.

The results of the determination specified in this document are reported on the air-dried basis. Calculation of results to other bases is dealt with in ISO 1170.

9 Precision

9.1 Repeatability limit

The results of duplicate determinations, carried out at different times, in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same general analysis sample, should not differ by more than the value given in Table 1.

Table 1 — Repeatability and reproducibility limits for ash

Ash	**Maximum acceptable difference between results** (calculated to the same moisture mass fraction)	
% mass fraction	**Repeatability limit (*r*)**	**Reproducibility limit (*R*)**
≤ 10 %	0,2 % absolute	0,3 % absolute
> 10 %	2,0 % of the mean result	3,0 % of the mean result

9.2 Reproducibility limit

The results of duplicate determinations, carried out in each of two laboratories, on representative portions taken from the same analysis sample after the last stage of sample preparation, should not differ by more than the value given in Table 1.

10 Test report

The test report shall include the following information:

1) identification of the sample tested;

2) the method used by reference to this document, i.e. ISO 1171:2024;

3) the date of the determination;

4) the results of the determination performed, together with the relevant analysis basis (e.g. air dry);