INTERNATIONAL STANDARD

ISO 1171

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Solid mineral fuels — Determination of ash content

Combustibles minéraux solides — Détermination du taux de cendres

Reference number
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Foreword

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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.

International Standard ISO 1171 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Sub-Committee SC 5, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 1171:1981), which has been technically revised.

Annex A of this International Standard is for information only.

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Introduction

The ash remaining after coal or coke has been incinerated in air is derived from inorganic complexes present in the original coal substance and from associated mineral matter. The amount of sulphur retained in the ash is in part dependent on the conditions of ashing and, in order to obtain values for the ash content on a comparable basis, it is necessary to adhere strictly to the conditions specified.

Solid mineral fuels — Determination of ash content

1 Scope

This International Standard specifies a method for the determination of the ash content of all solid mineral fuels.

2 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 content is calculated from the mass of the residue after incineration.

3 Apparatus

3.1 Balance, capable of weighing to the nearest 0,1 mg.

3.2 Furnace, capable of giving a zone of substantially 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 minute.

NOTES

1 The number of air changes per minute can be assessed by the measurement of the air flow in the furnace flue with a pitot-static tube and a suitable manometer.

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

3.3 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.

3.4 Plate, for use with coke samples, made from silica or heat-resistant steel, 6 mm thick and of such a size as to be an easy sliding fit into the furnace (3.2).

3.5 Desiccator or other closed container

4 Preparation of test sample

The coal or coke used for the determination of ash content is the general analysis test sample (ground to pass a sieve of 212 μm aperture).

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

5 Procedure

Weigh to the nearest 0,1 mg, the clean, dry dish (3.3) (see note below), spread approximately 1 g of the sample (clause 4) evenly in the dish and reweigh.

NOTE 1 If a silica or porcelain dish is used, before its initial mass is determined, it 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.

Insert the dish in the furnace (3.2) 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 a holding period of 60 min at 500 °C is necessary.

Continue heating to 815 °C ± 10°C in the same furnace or, alternatively, transfer the dish to a second furnace, previously heated to 815 °C ± 10°C. (See note 2, in 3.2). Maintain this temperature for at least 60 min.

Alternatively for coke, the dish placed on the plate (3.4) may be inserted directly in 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 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 or other closed container without desiccant and allow to cool to room temperature. When cool, weigh to the nearest 0,1 mg.

NOTE 2 The container may be flushed with dry gas in order to reduce the pick-up of moisture during cooling. In this case the dish should be covered with a lid.

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

NOTE 3 If results are to be calculated other than on an air dried basis, the moisture in the test sample shall be determined.

6 Expression of results

The ash content, A, of the sample as analysed, expressed as a percentage by mass, is given by the equation:

$$A = \frac{m_3 - m_1}{m_2 - m_1} \times 100$$

where

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

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

m_3 is the mass, in grams, of dish plus ash.

Report the result, as a mean of duplicate determinations, to the nearest 0,1 % (m/m).

The results of the determination described in this International Standard are reported on the air-dried basis. Calculation of results to other bases can be done according to ISO 1170 (see note 3 in clause 5).

7 Precision

Table 1

Ash content	Maximum acceptable difference between results (calculated to the same moisture content)	
	Repeatability limit	Reproducibility limit
< 10 %	0,2 % absolute	0,3 % absolute
⩾ 10 %	2,0 % of the mean result	3,0 % of the mean result

7.1 Repeatability limit

The results of duplicate determinations (carried out over a short period of time, but not simultaneously) in the same laboratory by the same operator with the same apparatus on two representative portions taken from the same analysis sample, shall not differ by more than the values given in table 1.

7.2 Reproducibility limit

The means of the results of duplicate determinations performed in each of two laboratories, on representative portions taken from the same analysis sample, shall not differ by more than the values given in table 1.

8 Test report

The test report shall include the following information:

a) the identification of the sample;

b) the method used;

c) the date of the determination;

d) the results and the calculation basis in which they are expressed;

e) any unusual features noted during the determination;

f) a reference to this International Standard;

g) any operation not included in this International Standard or regarded as optional.

Annex A
(informative)

Bibliography

[1] ISO 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method*.

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

[3] ISO 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method*.

[4] ISO 1170:1977, *Coal and Coke — Calculation of analyses to different bases*.

[5] ISO 1213-2:1992, *Solid mineral fuels— Vocabulary — Part 2: Terms relating to sampling, testing and analysis*.

[6] ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method*.

[7] ISO 11722:—[1)], *Solid mineral fuels — Hard coal — Determination of moisture in the analysis sample by drying in nitrogen*.

1) To be published.