INTERNATIONAL STANDARD

ISO 1171

Fourth edition
2010-06-15

Solid mineral fuels — Determination of ash

Combustibles minéraux solides — Détermination des cendres

Reference number
ISO 1171:2010(E)

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Published in Switzerland

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 562 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This fourth edition cancels and replaces the third edition (ISO 1171:1997), of which it constitutes a minor revision. (It also incorporates the Technical corrigendum ISO 1171:1997/Cor.1:1998.)

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. 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 conditions of ashing and, in order to obtain values for the ash on a comparable basis, it is necessary to adhere strictly to the conditions specified.

Solid mineral fuels — Determination of ash

1 Scope

This International Standard specifies a method for the determination of the ash 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 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.

NOTE 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.

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 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 next paragraph), spread approximately 1 g of the sample (Clause 4) evenly in the dish and reweigh.

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 3.2, last paragraph). 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.

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 the change in mass does not exceed 1 mg.

6 Expression of results

The ash, A, of the sample as analysed, expressed as a percentage mass fraction, is given by Equation (1):

$$A = \frac{m_3 - m_1}{m_2 - m_1} \times 100 \qquad (1)$$

where

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

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

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

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

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

7 Precision

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.

Table 1 — Repeatability and reproducibility limits for ash

Ash % mass fraction	**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

7.2 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) reference to this International Standard, i.e. ISO 1171;

b) identification of the sample;

c) date of the determination;

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