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SIST ISO 602:2016

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Premog - Določevanje mineralnih snovi

Coal - Determination of mineral matter

Charbon - Détermination du taux de matières minérales

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Coal — Determination of mineral matter

Charbon — Détermination du taux de matières minérales

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Contents

Page

Foreword ..... **iv**
1 Scope **1**
2 Normative references **1**
3 Principle **1**
4 Reagents **1**
5 Apparatus **1**
6 Preparation of sample **2**
7 Procedure **2**
8 Expression of results **3**
9 Precision of the method **3**
9.1 Repeatability 4
9.2 Reproducibility 4
10 Test report **4**
Annex A (normative) **Example of calculation** **6**
Bibliography **7**

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.

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 05, *Methods of analysis*.

This third edition cancels and replaces the second edition (ISO 602:1983), which constitutes a minor revision.

Coal — Determination of mineral matter

1 Scope

This International Standard specifies a method of determining the amount of mineral matter in all types of coal, including brown coals and lignites.

2 Normative references

There are no normative references cited in this document.

3 Principle

The following principle applies:

a) partial demineralization of a sample of the coal by treatment with hydrochloric and hydrofluoric acids under such conditions that the coal substance remains unaffected;

b) recording of the loss in mass of the coal due to the acid treatment and determination of the undissolved part of the mineral matter by ashing the partially demineralized coal;

c) determination of the iron content of the ash so that the pyrites present in the extracted coal can be calculated; and

d) determination of the amount of hydrochloric acid absorbed by the coal substance.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrochloric acid, ρ 1,18 g/ml.

4.2 Hydrochloric acid, solution, c(HCl) 5 mol/l.

4.3 Hydrofluoric acid, ρ 1,13 g/ml.

WARNING — Very toxic by inhalation, in contact with skin, and if swallowed. Causes severe burns.

Keep container tightly closed in a well-ventilated place. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

Wear suitable protective clothing and gloves. In case of accident or feeling unwell, seek medical advice immediately (show the label where possible).

5 Apparatus

All the apparatus listed below shall be resistant to acids, especially hydrofluoric acid. A suitable material is polyvinyl chloride (PVC).

5.1 Beaker, of capacity 200 ml, with a cover slip.

5.2 Thermometer pocket: a tube, sealed at one end, to carry a thermometer.

5.3 Stirrer.

5.4 Wash-bottle.

5.5 Filter, quantitative filter paper with 1 µm aperture and a sintered alumina filter plate, as shown in Figure 1.

5.6 Filter flask.

5.7 Balance, accurate to 0,1 mg.

6 Preparation of sample

The coal used for determination of mineral matter is the analysis sample, ground to pass a sieve of 200 µm aperture. If necessary, expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere (see ISO 11722 for moisture in the analysis sample).

Before starting the determination, mix the air-dried analysis sample of coal (see the note) thoroughly for at least 1 min, preferably by mechanical means.

NOTE Alternatively, the coal sample can be dried at 105 °C to 110 °C before carrying out the procedure.

7 Procedure

Weigh, to the nearest 0,1 mg, about 6 g of the sample into the beaker (5.1) and add 40 ml of the hydrochloric acid solution (4.2) (see Note 1). Insert the stirrer (5.3) and the tube (5.2) carrying a thermometer and place the cover slip over the beaker. Place the beaker in a water-bath adjusted to maintain the temperature of the solution between 55 °C to 60 °C. Stir the contents at 5 min intervals, remove the beaker after 45 min and allow the coal suspension to settle for 10 min. Filter the solution through the filter (5.5) under suction.

Wash the coal retained on the filter with water, drain and transfer the coal back to the beaker with the aid of not more than 5 ml of water. Care is required to avoid loss of coal by splashing (see Note 2).

Add 40 ml of the hydrofluoric acid (4.3) to the beaker and repeat the heat treatment and filtration as previously described. Rinse the coal retained on the filter into the beaker with not more than 5 ml of water. Add 50 ml of hydrochloric acid (4.1) to the beaker, replace it in the water-bath and repeat the heat treatment previously described. Filter and wash the coal three times, decanting each time. Transfer the coal entirely to the filter and wash 20 times with 25 ml portions of hot water each time. Recover any residual coal from the beaker by means of a rubber tipped rod and cold water. Drain the coal under suction for 5 min to 10 min.

Dismantle the filter, break up the wet coal, and dry the filter and coal in a vacuum oven at 50 °C and a pressure of 3,5 kPa[1)] absolute for about 1,5 h. Remove and allow to cool in air for about 1 h to attain equilibrium and then weigh. Recover the coal and transfer as much as possible to a glass stoppered bottle. Brush the filter top and filter paper free from coal and reweigh. Obtain the mass of extracted coal by difference.

Mix the extracted coal thoroughly and determine its moisture (see ISO 11722 for moisture in the analysis sample), ash (see ISO 1171 for determination of ash), and chlorine (see ISO 587 for determination of chlorine), as well as the total iron content of the ash (see ISO 157 for determination of forms of sulphur); determine also the moisture and ash content of the original sample, each determination being carried

1) 1 kPa = 10 mbar.

out according to the appropriate International Standards (see Bibliography). Calculate the hydrochloric acid equivalent to the chlorine content and the pyrites equivalent to the total iron content.

NOTE 1 For reactive coals, including brown coals and lignites, the acids can be placed in the beaker before adding the sample to avoid local overheating.

NOTE 2 The first hydrochloric acid extraction is unnecessary for coals having a carbon dioxide content of less than 0,5 % (*m*/*m*).

8 Expression of results

All results used in the calculation shall be on the dry basis. The mineral matter, *MM*, expressed as a percentage by mass, is given by Formula (1)[2].

$$\frac{m_1 - m_2 + m_3 + m_4 + 1{,}1\,m_5}{m_1} \times 100 \qquad (1)$$

where

m_1 is the mass, in grams, of the test portion taken;

m_2 is the mass, in grams, of the test portion after extraction;

m_3 is the mass, in grams, of pyrites in the extracted coal;

m_4 is the mass, in grams, of hydrochloric acid in the extracted coal;

m_5 is the mass, in grams, of ash, less iron oxide from the pyrites in the extracted coal

The mineral matter factor, F_{MM}, is given by Formula (2):

$$\frac{MM}{A} \qquad (2)$$

where

A is the percentage of ash in the original coal.

An example of the calculation is given in Annex A.

The result (preferably the mean of two determinations, see Clause 9) shall be reported to the nearest 0,1 %.

NOTE 1 If it is required to derive the mineral matter from the percentage of ash by the application of a formula, see ISO 1170.

NOTE 2 The factor 1,1 allows approximately for the water of hydration of the aluminium and silicon compounds in the demineralized coal. In most cases, the correction is small and can be ignored.

9 Precision of the method

Mineral matter	Maximum acceptable difference between results obtained	
	In the same laboratory (repeatability)	In different laboratories (reproducibility)
	0,4 % absolute	(see 9.2)

2) See the notes.