International Standard 602

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Coal — Determination of mineral matter

Charbon — Détermination du taux de matières minérales

Second edition — 1983-02-01

UDC 662.66 : 543.84 **Ref. No. ISO 602-1983 (E)**

Descriptors : coal, chemical analysis, determination of content, mineral matter.

Price based on 4 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 602 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels.*

This second edition was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 602-1974), which had been approved by the member bodies of the following countries:

Australia	Egypt, Arab Rep. of	Romania
Austria	France	South Africa, Rep. of
Belgium	Germany, F.R.	Switzerland
Brazil	India	Turkey
Canada	Italy	United Kingdom
Chile	Korea, Rep. of	USA
Colombia	Netherlands	USSR
Czechoslovakia	New Zealand	
Denmark	Poland	

No member body had expressed disapproval of the document.

Printed in Switzerland

Coal — Determination of mineral matter

1 Scope and field of application

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

2 References

ISO 157, *Hard coal — Determination of forms of sulphur.*

ISO 331, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method.*

ISO 348, *Hard coal — Determination of moisture in the analysis sample — Direct volumetric method.*

ISO 352, *Solid mineral fuels — Determination of chlorine — High temperature combustion method.*

ISO 587, *Solid mineral fuels — Determination of chlorine using Eschka mixture.*

ISO 1170, *Coal and coke — Calculation of analyses to different bases.*

ISO 1171, *Solid mineral fuels — Determination of ash.*

3 Principle

Partial demineralization of a sample of the coal by treatment with hydrochloric and hydrofluoric acids under such conditions that the coal substance remains unaffected. 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. Determination of the iron content of the ash so that the pyrites present in the extracted coal can be calculated. 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, with a sintered alumina filter plate, for example as shown in the figure.

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.

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 may be dried at 105 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 maintained at 55 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 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, ash, and chlorine, as well as the total iron content of the ash; determine also the moisture and ash content of the original sample, each determination being carried out according to the appropriate International Standards (see clause 2). Calculate the hydrochloric acid equivalent to the chlorine content and the pyrites equivalent to the total iron content.

1) 1 kPa = 10 mbar

2) See the notes.

NOTES

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

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 the formula[2]

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

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 the formula

$$\frac{MM}{A}$$

where A is the percentage of ash in the original coal.

An example of the calculation is given in the annex.

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

NOTES

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

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)

9.1 Repeatability

The result of duplicate determinations, carried out at different times in the same laboratory by the same operator with the same apparatus on the same analysis sample, should not differ by more than the above value.

9.2 Reproducibility

No value for reproducibility can be quoted for determinations carried out in different laboratories, since insufficient data are available.

10 Test report

The test report shall include the following information:

a) an identification of the product tested;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

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

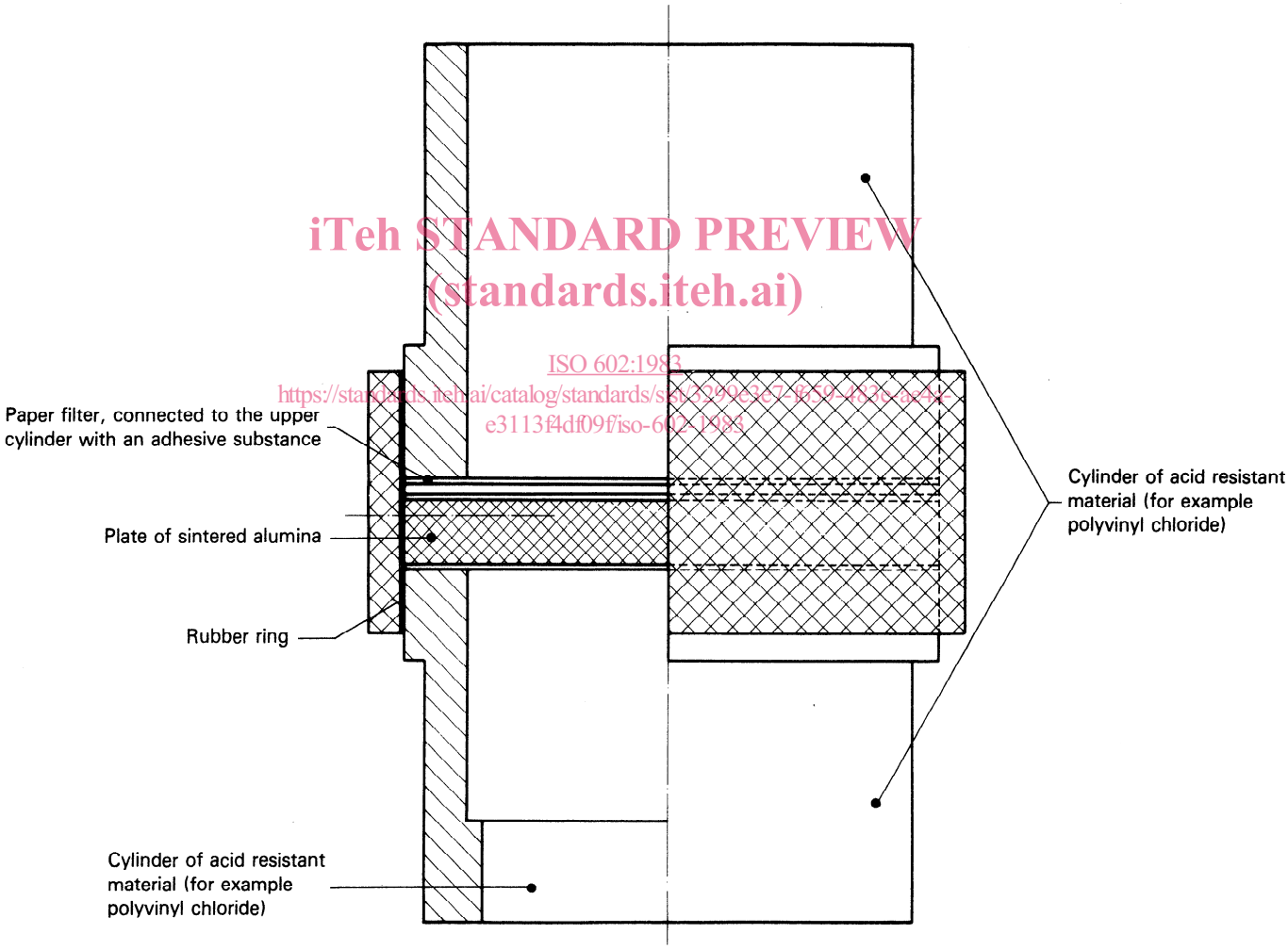

Figure — Filtering device (Left half shown in section)

Annex

Example of calculation

The following example illustrates the method of calculation. All results are quoted on the dry basis.

			% (m/m)	g	
1	Mass of original coal	=		6,836	(m_1)
2	Ash content of original coal	=	52,8		(A)
3	Mass of extracted coal	=		3,205	(m_2)
4	Ash content of extracted coal	=	6,03		(A_1)
5	Amount of hydrochloric acid absorbed by the extracted coal	=	1,06		(P_1)
6	Amount of iron oxide in the ash of the extracted coal	=	4,2		(P_2)
7	Loss in mass of the sample ($m_1 - m_2$)	=		3,631	
8	Mass of hydrochloric acid absorbed ($P_1 m_2/100$)	=		0,034	(m_4)
9	Mass of the pyrites in the extracted coal (1,5 $P_2 m_2/100$)	=		0,202	(m_3)
10	Mass of the residual ash [1,1 m_2 ($A_1 - P_2$)/100]	=		0,065	(1,1 m_5)
11	Total mass of mineral matter ($m_1 - m_2 + m_3 + m_4 + 1,1\ m_5$)	=		3,932	(m_6)
12	Percentage of mineral matter (100 m_6/m_1)	=	57,5		(MM)
13	Mineral matter factor (MM/A)	=	1,09		(F_{MM})

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