INTERNATIONAL STANDARD 602

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Coal — Determination of mineral matter

Charbon — Détermination du taux de matières minérales

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 602 and found it technically suitable for transformation. International Standard ISO 602 therefore replaces ISO Recommendation R 602-1967 to which it is technically identical.

ISO Recommendation R 602 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 602 into an International Standard.

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 coal.

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, *Hard coal and coke — Determination of chlorine — High temperature combustion method.*

ISO 587, *Coal and coke — Determination of chlorine using Eschka mixture.*

ISO/R 1171, *Determination of ash of solid mineral fuels.*

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. In addition, determination of the iron content of the ash so that the pyrites present in the extracted coal can be calculated. Determination also of the amount of hydrochloric acid absorbed by the coal substance.

4 REAGENTS

All reagents shall be of analytical reagent quality and distilled water shall be used throughout.

4.1 Hydrochloric acid, ρ 1,18 g/ml.

4.2 Hydrochloric acid, 5 N.

4.3 Hydrofluoric acid, ρ 1,13 g/ml.

5 APPARATUS

All the apparatus listed below shall be resistant to acids, especially hydrofluoric acid. A suitable material is polyvinyl chloride (PVC). The balance used shall be sensitive to 0,1 mg.

5.1 Beaker, 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, as shown for example in the figure.

5.6 Filter flask.

6 PREPARATION OF SAMPLE

The sample used for the determination of mineral matter is the analysis sample ground to pass a sieve of 0,2 mm 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 commencing the determination, mix the air-dried analysis sample of coal (see note 1) thoroughly for at least 1 min, preferably by mechanical means.

7 PROCEDURE

Weigh accurately about 6 g of the sample into the beaker and add 40 ml of the hydrochloric acid (4.2) (see note 2). Insert the stirrer and the tube carrying the 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. Decant the solution through the filter (5.5) under suction.

Wash any coal 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 3).

Add 40 ml of the hydrofluoric acid (4.3) to the beaker and repeat the heat treatment and filtration as previously described. Rinse any coal on the filter back 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. Decant the solution through the prepared filtering device and wash the coal with water three times, decanting each time. Transfer the coal entirely to the filter and wash twenty times, with 25 ml portions of hot water each time. Remove 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 compacted, wet coal and dry the filter top and coal in a vacuum oven at 50 °C and a pressure of 35 mbar absolute for about 1 1/2 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.

NOTES

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

2 For low rank and other reactive coals, the acids may be placed in the beaker before adding the sample to avoid local over-heating.

3 The first hydrochloric acid extraction is unnecessary for coals having a carbon dioxide content of less than 0,5 %.

8 EXPRESSION OF RESULTS

All results used in the calculation shall be on the dry basis. The mineral matter *MM*, in per cent, is given by the formula

$$\frac{m_1 - m_2 + P + \mathrm{HCl} + 1{,}1\,A}{m_1} \times 100 \text{ (see note)}$$

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;

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

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

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

The mineral matter factor F is given by the formula

$$\frac{MM}{A_1}$$

where A_1 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 duplicate determinations, see clause 9) shall be reported to the nearest 0,1 %.[1)]

NOTE – 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	
	Repeatability	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 evidence is available for this to be done.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

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

1) Calculation of the results to other bases is dealt with in ISO/R 1170.