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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 602

DETERMINATION OF MINERAL MATTER IN COAL

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July 1967

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BRIEF HISTORY

The ISO Recommendation R 602, *Determination of Mineral Matter in Coal,* was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1955 and led, in 1961, to the adoption of a Draft ISO Recommendation.

In March 1964, this Draft ISO Recommendation (No. 679) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 602 July 1967

DETERMINATION OF MINERAL MATTER IN COAL

1. SCOPE

This ISO Recommendation describes a method of determining the amount of mineral matter in coal.

2. PRINCIPLE

A sample of the coal is partially demineralized by treatment with hydrochloric and hydrofluoric acids under such conditions that the coal substance remains unaffected. The loss in mass of the coal due to the acid treatment is recorded and the insoluble part of the mineral matter determined by ashing the partially demineralized coal. In addition, the iron content of the ash is determined so that the pyrites present in the extracted coal can be calculated. The amount of hydrochloric acid absorbed by the coal substance is also determined.

3. REAGENTS

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

3.1 **Hydrochloric acid,** *d* 1.18 or 1.19.

3.2 **Hydrochloric acid,** 5 N.

3.3 **Hydrofluoric acid,** *d* 1.13 or 1.14.

4. APPARATUS

All the apparatus listed below should be resistant to acids, especially hydrofluoric acid. A suitable material is polyvinyl chloride (P.V.C.). The balance used should be sensitive to 0.1 mg.

4.1 **Beaker.** A 200 ml beaker with a cover slip.

4.2 **Thermometer pocket.** A tube, sealed at one end, to carry a thermometer.

4.3 **Stirrer.**

4.4 **Wash-bottle.**

4.5 **Filter,** with a sintered alumina filter plate, as shown for example in the Figure, page 4.

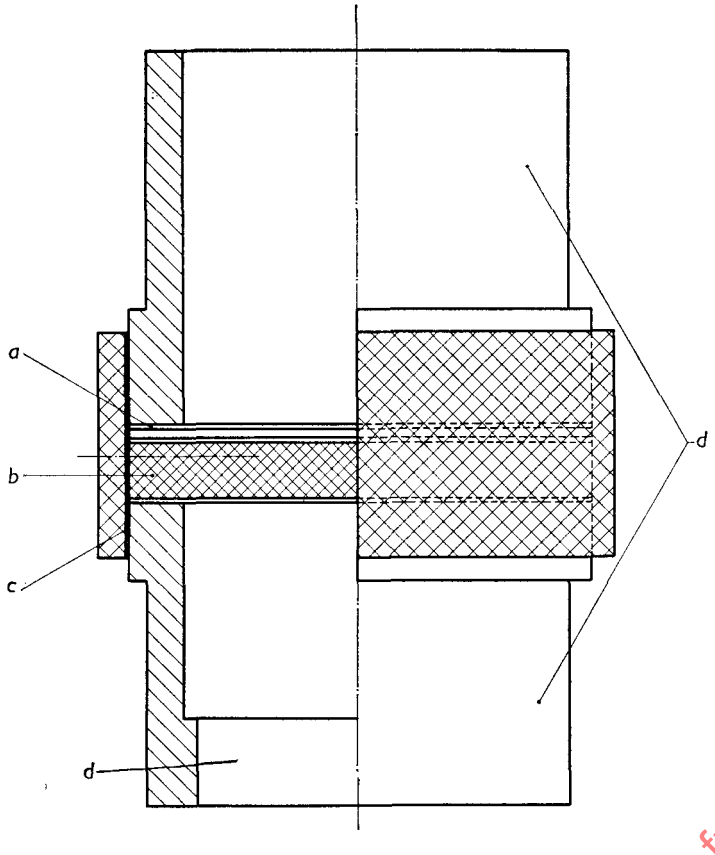
4.6 **Filter flask.**

5. PROCEDURE

Before commencing the determination, mix the air-dried analysis sample of coal (see Note 1), ground to pass a sieve of 0.2 mm aperture, thoroughly for at least one minute, preferably by mechanical means.

Weigh accurately about 6 g of the sample into the beaker and add 40 ml of the hydrochloric acid (3.2) (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 minute intervals, remove the beaker after 45 minutes and allow the coal suspension to settle for 10 minutes. Decant the solution through the filter (4.5) 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).



a: paper filter, connected to the upper cylinder with an adhesive substance

b: plate of sintered alumina

c: rubber ring

d: cylinder of acid resistant material (e.g. polyvinyl chloride)

FIGURE. — **Filtering device** (Left half shown in section)

Add 40 ml of the hydrofluoric acid (3.3) 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 water. Add 50 ml of hydrochloric acid (3.1) 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 minutes.

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 25 mmHg for about 1½ hours. Remove and allow to cool in air for about one hour to attain equilibrium and then weigh. Recover the coal and transfer as much as possible to a glass stoppered bottle. Wipe 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 content of the original sample, each determination being carried out according to the appropriate ISO Recommendations *. Calculate the hydrochloric acid equivalent to the chlorine content and the pyrites equivalent to the total iron content.

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* See ISO Recommendations: R 157, *Determination of Forms of Sulphur in Coal;* R 158, *Determination of Ash of Hard Coal;* R 331, *Determination of Moisture in the Analysis Sample of Coal by the Direct Gravimetric Method;* R 348, *Determination of Moisture in the Analysis Sample of Coal by the Direct Volumetric Method;* R 350, *Determination of Chlorine in Coal by the Bomb-combustion Method;* R 352, *Determination of Chlorine in Coal by the High Temperature Combustion Method;* R 587, *Determination of Chlorine in Coal and Coke using Eschka Mixture.*

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 per cent.

6. CALCULATION AND REPORTING OF RESULTS

6.1 All results should be quoted on a moisture-free basis; an example of the calculation is given in Annex A.

If m_1 = mass of test portion taken, expressed in grammes,

m_2 = mass of test portion after extraction, expressed in grammes,

P = mass of pyrites in the extracted coal, expressed in grammes,

HCl = mass of hydrochloric acid in the extracted coal, expressed in grammes,

A = mass of ash, less iron oxide from the pyrites in the extracted coal, expressed in grammes,

A_1 = percentage of ash in the original coal,

F = mineral matter factor,

and MM = percentage of mineral matter,

then $$MM = \frac{m_1 - m_2 + P + \mathrm{HCl} + 1.1\,A}{m_1} \times 100$$ (see Note below)

and $$F = \frac{MM}{A_1}$$

The result (preferably the mean of duplicate determinations – see clause 7) should be reported to the nearest 0.1 %. Calculation of the result to other bases is being studied * and will be the subject of a separate ISO Recommendation.

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 this correction is small and can be ignored.

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* by the Technical Committee ISO/TC 27, *Solid Mineral Fuels*.

7. PRECISION OF DETERMINATION

Mineral matter	Maximum acceptable difference between results obtained	
	in the same laboratory	in different laboratories
	0.4% absolute	(see 7.2)

7.1 In the same laboratory

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.

7.2 In different laboratories

No tolerances are quoted for determinations carried out in different laboratories, since insufficient evidence is available for this to be done.