UDC 662.642 : 662.613.11 : 543.062

Ref. No. : ISO/R 1016-1969 (E)

Replaced by ISO/R 1171

27

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

Annulée

ISO RECOMMENDATION

R 1016

DETERMINATION OF ASH OF BROWN COALS AND LIGNITES

1st EDITION
March 1969

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1016, *Determination of ash of brown coals and lignites,* 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 led, in 1966, to the adoption of a Draft ISO Recommendation.

In August 1967, this Draft ISO Recommendation (No. 1283) 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	Iran	South Africa, Rep. of
Austria	Italy	Spain
Canada	Japan	Switzerland
Czechoslovakia	Korea, Rep. of	Turkey
Denmark	Netherlands	U.A.R.
France	New Zealand	United Kingdom
Germany	Portugal	U.S.S.R.
India	Romania	Yugoslavia

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 March 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1016 March 1969

DETERMINATION OF ASH OF BROWN COALS AND LIGNITES

1. SCOPE

This ISO Recommendation describes a method of determining the ash of brown coals and lignites.

2. FIELD OF APPLICATION

When brown coal or lignite is incinerated completely, the ash remaining differs from the mineral matter originally present in the brown coal or lignite. This is because various changes occur during incineration, such as loss of water of constitution from shaly matter and of carbon dioxide from carbonates, and the oxidation of iron pyrites to iron oxide. The fixation of oxides of sulphur by bases also occurs. The method for the determination of ash of brown coals and lignites is, therefore, empirical, because the conditions of incineration determine the extent to which these reactions occur. It is essential, therefore, to adhere strictly to the procedure laid down to obtain reproducible results.

3. PRINCIPLE

Brown coal or lignite is heated in air to a temperature of 250 °C in 30 minutes; from 250 °C to 500 °C in a further 30 minutes; from 500 °C to 815 °C in a further 60 minutes; and is maintained at this last temperature until it is constant in mass.

The percentage of ash is calculated from the mass of residue after incineration.

4. APPARATUS

4.1 *Balance,* sensitive to 0.1 mg.

4.2 *Muffle furnace,* capable of giving a substantially uniform temperature zone at 250 °C after 30 minutes of heating from room temperature, of being raised to 500 °C in a further 30 minutes, of being raised to 815 ± 10 °C in a further 60 minutes, and of maintaining this last temperature at the end of the run-up period. The ventilation should be such as to give at least four air changes per minute.

NOTE. – The air changes per minute can be assessed by measurement of the air flow in the flue by means of a Pitot static tube and sensitive manometer.

4.3 *Dish,* of silica, porcelain or platinum, 15 mm deep, with a lid, and of such a size that with the mass of sample used, the coal layer does not exceed 0.15 g/cm^2.

5. PROCEDURE

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

Weigh a clean, dry dish with its lid (see Note 1, below) and spread uniformly into it about 1 g of the sample, to form a layer of not more than 0.15 g/cm^2. Weigh the covered dish and its contents to determine by difference the mass of sample taken. Insert the uncovered dish and the lid separately in the muffle furnace at room temperature, raise the temperature to 250 °C in 30 minutes, from 250 °C to 500 °C in another 30 minutes and from 500 °C to 815 °C in another 60 minutes. Maintain the final temperature for a further 60 minutes.

Cover the dish with the lid, then remove the dish from the muffle furnace and allow it to cool, first on a cold metal slab for 5 minutes and finally in a desiccator standing at the side of the balance. Weigh the dish with its contents and lid after it has been in the desiccator for 15 minutes. Re-ignite at the final temperature until constant in mass (see Note 2, below).

NOTES

1. Silica and porcelain dishes, if used, should be heated to 815 ± 10 °C for 15 minutes and cooled under the conditions specified in the actual determination immediately before their initial masses are determined.

2. Constancy in mass is defined as a change not exceeding 1 mg in a further period of heating of 15 minutes.

6. EXPRESSION OF RESULTS

The ash (A) of the sample as analysed, expressed as a percentage, by mass, is calculated from the following formula :

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

where

m_1 is the mass of dish with lid, in grammes;

m_2 is the mass of dish with lid and sample, in grammes;

m_3 is the mass of dish with lid and ash, in grammes.

The final result should be reported to the nearest 0.1 %.

7. PRECISION OF THE METHOD

Ash	Maximum acceptable difference between results obtained	
	in the same laboratory (Repeatability)	in different laboratories (Reproducibility)
Less than 10 %	0.3 % absolute	0.4 % absolute
10 % and over	3.0 % of result	4.0 % of result

7.1 Repeatability

The results of duplicate determinations, carried out at different times, in the same laboratory, by the same operator, with the same apparatus, on representative test portions taken from the same analysis sample, should not differ by more than the value mentioned in the table on the previous page.

7.2 Reproducibility

The means of the results of duplicate determinations, carried out in two different laboratories, on representative test portions taken from the same analysis sample, should not differ by more than the value mentioned in the table on the previous page.

Date of the first printing :
May 1969

Price based on 3 pages