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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 158

DETERMINATION OF ASH OF HARD COAL

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BRIEF HISTORY

The ISO Recommendation R 158, *Determination of Ash of Hard Coal*, was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels*, the Secretariat of which is held by the British Standards Institution (B.S.I.).

At the first meeting of ISO/TC 27, held in London, in March 1950, national standards for the determination of ash were compared.

At the second meeting, held in London, in November 1951, this subject was further discussed and provisional decisions were reached on certain details.

At the third meeting, held in London, in November 1953, it was agreed that a draft proposal should be prepared and that Working Group No. 2, *Ash and Moisture*, should attempt to resolve certain outstanding difficulties experimentally.

The first draft proposal was circulated in June 1954. It was considered by the Working Group, together with a counter-proposal from Belgium, at a meeting in October 1954. Six methods for determining ash were demonstrated. After considering all the results, it was agreed that the one-furnace method should be adopted and that the temperature should be raised from cold to 500 °C in 30 minutes and from 500 °C to the final temperature in 30 to 60 minutes. For the final temperature, a compromise figure of 815 ± 10 °C was adopted.

A second draft proposal incorporating these decisions was circulated in January 1955 to the members of the Committee and was studied, together with the report of the Working Group, at the fourth meeting of ISO/TC 27, held in Stockholm in June 1955. It was agreed that the method should only apply to hard coal, since the question of the final temperature for brown coals and lignites required further investigation. A decision reached at that time was later revoked, and the choice of using a 1 g or a 2 g sample was restored. It was agreed that a new draft proposal should be drawn up on that basis.

This draft proposal for an ISO Recommendation was circulated in June 1956 to the Members of ISO/TC 27, and, with minor changes, was adopted as a Draft ISO Recommendation.

On 29 June 1957, the Draft ISO Recommendation (No. 170) was distributed to all the ISO Member Bodies and was approved, subject to some modifications, by the following 23 (out of a total of 38) Member Bodies:

Austria	*Ireland	Sweden
*Canada	Italy	*Switzerland
Czechoslovakia	Mexico	*Union of South Africa
Denmark	Netherlands	United Kingdom
*Germany	*New Zealand	U.S.A.
*Greece	*Portugal	*U.S.S.R.
Hungary	Romania	*Yugoslavia
India	Spain	

One Member Body opposed the approval of the Draft: Belgium.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in June 1960, to accept it as an ISO RECOMMENDATION.

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* These Member Bodies stated that they had no objection to the Draft being approved.

ISO Recommendation R 158 June 1960

DETERMINATION OF ASH OF HARD COAL

1. INTRODUCTION

When coal is incinerated completely, the ash remaining differs from, and is less than, the mineral matter originally present in the coal. 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 in coal 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.

2. PRINCIPLE

The coal is heated in air to 500 °C in 30 minutes and from 500 to 815 °C in a further 30 to 60 minutes and maintained at this temperature until constant in weight. The percentage of ash is calculated from the weight of residue after incineration.

3. APPARATUS

3.1 *Balance*, sensitive to 0.1 mg.

3.2 *Muffle furnace*, capable of giving a substantially uniform temperature zone at 500 °C after 30 minutes of heating from room temperature, of being raised to 815 ± 10 °C in a further 30 to 60 minutes, and of maintaining this latter temperature at the end of the run-up period. The ventilation should be such as to give at least 4 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 tube and sensitive manometer.

3.3 *Dish*, of silica, porcelain or platinum, 10 to 15 mm deep, with a lid, of such a size that, with the amount of sample used, the thickness of the coal layer does not exceed 0.15 g/cm².

4. PROCEDURE

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

Weigh a clean, dry dish with its lid (see Note 1) and spread uniformly into it 1 to 2 g of the sample, to a depth of not more than 0.15 g/cm². Weigh the covered dish and its contents to determine by difference the weight of sample taken. Insert the uncovered dish in the muffle furnace at room temperature, raise the temperature to 500 °C in 30 minutes and to 815 ± 10 °C in a further 30 to 60 minutes and maintain at this temperature for 60 minutes (see Note 2). Remove the dish from the muffle furnace (see Note 3) and allow it to cool, first on a cold metal slab for 10 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 weight (± 0.001 g). Use the initial weight of the dish and lid to obtain the weight of ash and report the result as a percentage to the first decimal place.

NOTES

1. Silica dishes, if used, should be heated to 815 ± 10 °C and maintained at this temperature for 15 minutes and then cooled under the conditions specified in the actual determination immediately before their initial weights are determined.
2. If the coal is of unknown origin, or if it has high sulphur and carbon dioxide contents (each more than 2 per cent), the heating rate should be changed as follows: to reach 500 °C in 60 minutes, from 500 to 815 °C in a further 60 minutes, and held at 815 °C for 60 minutes.
3. If the ash is light and fluffy, the dish should be covered before removal from the furnace.

5. CALCULATION OF RESULTS

If W_1 = weight of dish plus lid, expressed in grammes,
W_2 = weight of dish plus lid plus sample, expressed in grammes,
W_3 = weight of dish plus lid plus ash, expressed in grammes,
A = percentage of ash in the sample,

then
$$A = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

6. TOLERANCES

The following table gives the maximum acceptable differences between

the results of determinations carried out at different times in the same laboratory, on the same sample, by the same operator using the same apparatus, and

the means of two determinations carried out in different laboratories on representative samples taken from the same bulk sample after the last stage of the reduction process.

Ash	Maximum acceptable differences between results obtained	
	in the same laboratory	in different laboratories
Less than 10% 10% and over	0.2% absolute 2.0% of result	0.3% absolute 3.0% of result

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