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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

# ISO RECOMMENDATION
# R 1171

## DETERMINATION OF ASH OF SOLID MINERAL FUELS

(Replacing ISO Recommendations R 158-1960, R 586-1967 and R 1016-1969)

1st EDITION

January 1970

For each individual country the only valid standard is the national standard of that country.

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## BRIEF HISTORY

The ISO Recommendation R 1171, *Determination of ash of solid mineral fuels*, 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 to the adoption of a Draft ISO Recommendation.

In June 1968, this Draft ISO Recommendation (No. 1612) 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
Belgium
Canada
Czechoslovakia
Denmark
France
Germany
India
Iran
Israel
Netherlands
New Zealand
Peru
Poland
Portugal
Romania
South Africa, Rep. of
Spain
Sweden
Switzerland
Turkey
U.A.R.
United Kingdom
U.S.A.
U.S.S.R.
Yugoslavia

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in January 1970, to accept it as an ISO RECOMMENDATION, replacing ISO Recommendations R 158-1960, R 586-1967 and R 1016-1969.

ISO Recommendation R 1171 January 1970

# DETERMINATION OF ASH OF SOLID MINERAL FUELS

(Replacing ISO Recommendations R 158-1960, R 586-1967 and R 1016-1969)

## INTRODUCTION

The ash remaining after coal or coke has been incinerated in air is derived from inorganic complexes present in the original coal substance and from associated mineral matter. The amount of sulphur retained in the ash is in part dependent on the conditions of ashing and, in order to obtain values for the ash content on a comparable basis, it is necessary to adhere strictly to these conditions.

## 1. SCOPE

This ISO Recommendation describes a method of determining the ash content of all solid mineral fuels.

## 2. PRINCIPLE

The sample is heated in air at a specified rate up to a temperature of 815 ± 10 °C and maintained at this temperature until constant in mass. The characteristics of hard coal and coke differ from those of brown coal and lignite in such a manner that more rapid rates of heating are permitted for the former.

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

## 3. REAGENT

*Desiccant*. Either fresh or freshly regenerated, self-indicating, activated alumina, silica gel, anhydrous calcium sulphate or phosphorus pentoxide, or fresh magnesium perchlorate.

NOTE. – Regeneration of magnesium perchlorate should not be attempted, owing to the risk of explosion; when exhausted, the magnesium perchlorate should be washed down the sink with water.

## 4. APPARATUS

4.1 *Balance*, sensitive to 0.1 mg.

4.2 *Muffle furnace* capable of giving a zone of substantially uniform temperature at the levels required by the procedure and of reaching these levels in the specified times. The ventilation through the muffle furnace should be such as to give about five air changes per minute.

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

4.3 *Dish* of silica, porcelain, or platinum, 10 to 15 mm deep, with lid. The diameter of the dish should be such that the thickness of the sample layer does not exceed 0.15 g/cm$^2$ for coal or 0.10 g/cm$^2$ for coke.

4.4 *Insulating plate*. A silica plate, 6 mm thick, or its equivalent, of such size as to be an easy sliding fit into the muffle furnace.

## 5. SAMPLE

The coal or coke used for the determination of ash content is the analysis sample ground to pass a sieve of 0.2 mm aperture. If necessary, the sample should be exposed in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere.

## 6. PROCEDURE

Before commencing the determination, mix the analysis sample thoroughly for at least 1 minute, preferably by mechanical means.

Weigh accurately a clean, dry dish and lid (see Note), spread 1 to 2 g of sample evenly in the dish and re-weigh.

NOTE. – If a silica dish is used, then before its initial mass is determined, it should be heated to 815 ± 10 °C, maintained at this temperature for 15 minutes and then cooled under the conditions specified for the actual determination.

– *For brown coal and lignite*

Insert the uncovered dish in the muffle furnace at room temperature. Raise the temperature to 250 °C in 30 minutes; from 250 to 500 °C in a further 30 minutes; from 500 to 815 ± 10 °C in a further 60 minutes and maintain at this temperature for a further 60 minutes.

– *For hard coal*

Insert the uncovered dish in the muffle furnace at room temperature. Raise the temperature to 500 °C in 30 minutes; from 500 to 815 ± 10 °C in a further 30 to 60 minutes and maintain at this temperature for 60 minutes.

NOTE. – If the coal is of unknown origin, or if it has high sulphur and carbon dioxide contents (each more than 2 %), the heating rate specified for brown coal and lignite should be used.

– *For coke*

Place the uncovered dish on the insulating plate, insert in the muffle furnace at 815 ± 10 °C and maintain at this temperature for 75 minutes.

NOTE. – Alternatively, for coke the dish may be placed in a cold muffle and heated to 815 °C as rapidly as possible, the duration of heating being calculated from the time the muffle reaches 815 ± 10 °C. In this case, the insulating plate may be dispensed with.

Remove the dish from the furnace, cover the dish with its lid and allow it to cool, first on a thick metal plate for 10 minutes and finally in the desiccator for 15 minutes. For all samples of brown coal and lignite, or if the ash is light and fluffy, the dish should be covered with its lid before removal from the furnace.

Weigh the covered dish and ash. Reheat at 815 ± 10 °C for further 15 minute periods until any further change in mass does not exceed 1 mg.

## 7. CALCULATION AND REPORTING OF RESULTS

### 7.1 Calculation of result

The ash content ($A$) 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, in grammes, of the dish plus lid;
$m_2$ is the mass, in grammes, of dish plus lid plus sample;
$m_3$ is the mass, in grammes, of dish plus lid plus ash.

### 7.2 Precision of reporting

The result (preferably the mean of duplicate determinations – see section 8) should be reported to the nearest 0.1 %.

## 8. PRECISION OF THE METHOD

| Ash | Maximum acceptable differences between results obtained (calculated to the same moisture basis) | |
|---|---|---|
| | in the same laboratory (repeatability) | in different laboratories (reproducibility) |
| Less than 10 % | 0.2 % absolute | 0.3 % absolute |
| 10 % and over | 2.0 % of the mean result | 3.0 % of the mean result |

### 8.1 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator with the same apparatus on two representative portions taken from the same analysis sample (see Note below), should not differ by more than the above value.

### 8.2 Reproducibility

The means of the results of duplicate determinations carried out in two different laboratories, on representative portions taken from the same analysis sample after the last stage of sample preparation (see Note below), should not differ by more than the above value.

NOTE. – If the analysis sample has not been ground to pass a 0.2 mm sieve, the differences between duplicate determinations may be exceeded.

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