UDC 664.741/.742:620.1

Ref. No.: ISO/R 586 - 1967 (E)

Replaced by ISO/R 1171 F.V.

Annulée

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 586

DETERMINATION OF ASH OF COKE

1st EDITION

July 1967

Printed in Switzerland

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

BRIEF HISTORY

The ISO Recommendation R 586, *Determination of Ash of Coke*, 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 1958 and led, in 1961, to the adoption of a Draft ISO Recommendation.

In March 1964, this Draft ISO Recommendation (No. 680) 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	France	Republic of South Africa
Austria	Germany	Romania
Belgium	India	Switzerland
Canada	Italy	Turkey
Chile	Korea, Rep. of	U.A.R.
Colombia	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.A.
Denmark	Poland	U.S.S.R.

One Member Body opposed the approval of the Draft:

Japan

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 586 July 1967

DETERMINATION OF ASH OF COKE

1. SCOPE

This ISO Recommendation describes the method of determining the ash of coke.

2. PRINCIPLE

The coke is heated in air at 815 ± 10 °C and maintained at this temperature until constant in mass. The percentage of ash is calculated from the mass of residue after incineration.

Provided that steps are taken to prevent mechanical loss, the (precautionary) slow rate of heating necessary for determining the ash of coal is unnecessary for coke, as that part of the sulphur in coke which is combined with iron is present as ferrous sulphide which, unlike the iron pyrites in coal, does not liberate sulphur on heating to 500 °C. The alkaline earths and all forms of sulphur are usually present in only small amount in the hard coals used for coke-making, so that sulphation of the ash is less likely with coke than with coal. Because coke is less reactive than coal, a thinner layer is used to ensure that the test portion shall be burnt completely in a reasonable period of time. The temperature of incineration is conventional and for convenience the same temperature as that recommended for coal has been selected.

3. REAGENT

Desiccant. Either fresh or freshly regenerated, self-indicating, activated alumina, silica gel, anhydrous calcium sulphate or phosphorus pentoxide, or freshly dried 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 a current of water.

4. APPARATUS

4.1 Balance, sensitive to 0.1 mg.

4.2 Muffle furnace, capable of providing a substantially uniform temperature zone at 815 ± 10 °C, with a ventilation rate of at least four air changes per minute (see Note 1).

4.3 Dish or crucible, of silica, porcelain or platinum, with lid (see Note 2), and of such a size that the coke layer does not exceed 0.1 g/cm².

4.4 Insulating plate. A flat plate 6 mm thick, of silica or other suitable insulating material, which fits easily in the muffle.

4.5 Desiccator, containing a metal plate, preferably of aluminium, and a suitable desiccant (see section 3).

NOTES

1. The number of air changes in the muffle per minute can be determined by measurement of the air flow in the flue by means of a pitot-static tube and a sensitive manometer.
2. The use of a lid is optional and the references to a lid in sections 6 and 7 are not intended to indicate a preference.

5. PROCEDURE

Before commencing the determination, mix the general analysis sample of coke (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 a clean, dry dish (and lid) (see Note 2) and into the dish weigh accurately between 1 and 2 g of the sample. Spread the coke evenly over the dish, place the uncovered dish on the insulating plate, insert in the muffle furnace at 815 ± 10 °C (see Note 3) and maintain at this temperature until constant in mass (see Note 4).

When incineration is complete, remove the plate with the dish from the furnace, cover the dish with its lid (if used) and allow it to cool, first on a thick metal plate for 10 minutes and finally in the desiccator for 15 minutes. If a lid is not used, cool the dish on the thick metal plate for 2 or 3 minutes and then 25 minutes in the desiccator. Weigh the (covered) dish and deduct the initial mass of the dish (and lid) to obtain the mass of ash.

NOTES

1. If the sample of coke dried in the determination of total moisture is used for the preparation of the general analysis sample, air-drying after crushing is not required. If a separate sample is used for general analysis, it should be air-dried either before or after crushing to 0.2 mm.
2. Silica dishes, if used, should be heated to 815 ± 10 °C for 15 minutes and cooled under the conditions specified for the actual determination immediately before their initial weights are determined.
3. Alternatively, 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.
4. Under the conditions described, incineration should be complete in 75 minutes. Constancy in mass is defined as a change not exceeding 0.5 mg in a further period of heating of 15 minutes at 815 °C.

6. CALCULATION AND REPORTING OF RESULTS

If m_1 = mass of dish (plus lid), expressed in grammes,

m_2 = mass of dish (plus lid) plus coke, expressed in grammes,

m_3 = mass of dish (plus lid) plus ash, expressed in grammes,

and A = ash of the coke as analysed, per cent,

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

The result (preferably the mean of duplicate determinations – see section 7 below) should be reported to the nearest 0.1 %.

7. PRECISION OF DETERMINATION

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

7.1 In the same laboratory

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

7.2 In different laboratories

The means of the results of duplicate determinations, carried out in each of two different laboratories on test portions taken from the same analysis sample after the last stage of the reduction process, should not differ by more than the above values.

Date of the first printing:
September 1967

Price: Sw. fr. 6.—