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Hard coal — Determination of oxygen content

Houille — Dosage de l'oxygène

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27, *Solid mineral fuels*, has reviewed ISO Recommendation R 1994-1971 and found it technically suitable for transformation. International Standard ISO 1994 therefore replaces ISO Recommendation R 1994-1971, to which it is technically identical.

ISO Recommendation R 1994 had been approved by the member bodies of the following countries :

Australia	Greece	Spain
Belgium	India	Sweden
Canada	Italy	Switzerland
Chile	Japan	Thailand
Czechoslovakia	Netherlands	Turkey
Denmark	New Zealand	United Kingdom
Egypt, Arab Rep. of	Poland	U.S.A.
France	Portugal	Yugoslavia
Germany	South Africa, Rep. of	

No member body had expressed disapproval of the Recommendation.

No member body disapproved the transformation of the Recommendation into an International Standard.

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Hard coal — Determination of oxygen content

0 INTRODUCTION

This International Standard follows methods developed by Schütze[1], Unterzaucher[2], and Oita and Conway[3] for the direct determination of the oxygen content of organic compounds. Modifications have been incorporated to avoid errors due to the presence of moisture in coal. Oxygen is evolved (as water and carbon dioxide) from the mineral matter associated with the coal when the sample is pyrolysed and, to reduce errors from this source, coals containing more than 5 % of ash and all samples of unknown characteristics should be demineralized before analysis (see ISO 602).

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies semi-micro methods for the direct determination of the oxygen content of hard coal.

2 REFERENCES

ISO 331, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method.*

ISO 348, *Hard coal — Determination of moisture in the analysis sample — Direct volumetric method.*

ISO 602, *Coal — Determination of mineral matter.*

3 PRINCIPLE

The sample is dried at 105 to 110 °C in a stream of nitrogen and then pyrolysed under conditions in which the organic matter leaves an oxygen-free char. The volatile products, containing the organically bound oxygen and also water and carbon dioxide from any mineral matter, are decomposed with either pure carbon or platinized carbon to convert the oxygen to carbon monoxide. The carbon monoxide is oxidized to carbon dioxide and determined by a titrimetric[4] or a gravimetric procedure.

4 REAGENTS

4.1 Nitrogen.

The nitrogen used for the pyrolysis shall not contain more than 10 ppm of oxygen. If nitrogen of this purity is available commercially, further purification is not necessary if the total "blank" is within the limits specified in clause 7.

4.2 Alternative reagents for converting the volatile pyrolysis products to carbon monoxide

4.2.1 Pure carbon, particle size 0,7 to 2,0 mm[1], ash content not exceeding 0,01 %.

The carbon shall be ignited to dull red heat in an inert atmosphere to remove any oil before being placed in the pyrolysis tube.

Carbon containing up to 0,05 % ash can usually be purified as follows :

To remove any residual oil, heat to dull red, in an inert atmosphere, enough carbon to pack the pyrolysis tube and to provide a sample for the determination of ash. Allow to cool and digest the residue with hydrochloric acid, ρ 1,18 g/ml, at incipient boiling for 1 h. Allow the carbon to settle and decant the liquid through a hardened filter paper in a Buchner funnel. Wash the carbon several times by decantation, then transfer it to the filter and continue until the washings are free from chloride. Dry the cake first by suction, then in an oven at 120 °C.

Crush and sieve the dried cake carefully to obtain the maximum yield of 0,7 to 2,0 mm material. Determine the ash content of the granules.

or

4.2.2 Platinized carbon, particle size 0,7 to 2,0 mm[1], containing about 50 % of platinum.

A suitable method of preparation is as follows :

Dissolve 5 g of platinum in aqua regia and evaporate the solution to near dryness. Add 5 ml of hydrochloric acid, ρ 1,18 g/ml, and again evaporate to near dryness. Continue

1) To avoid too great a resistance to gas flow, it is important that material with particle size below 0,7 mm should be removed by sieving. Excessive resistance to gas flow may necessitate increasing the maximum size to 3 mm.