INTERNATIONAL STANDARD 332

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Coal — Determination of nitrogen by the macro Kjeldahl method

Charbon — Dosage de l'azote par la méthode Kjeldahl macrométrique

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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 now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 332 and found it technically suitable for transformation. International Standard ISO 332 therefore replaces ISO Recommendation R 332-1963, to which it is technically identical.

ISO Recommendation R 332 was approved by the Member Bodies of the following countries :

Austria	Greece	Portugal
Belgium	India	Romania
Brazil	Israel	South Africa, Rep. of
Canada	Italy	Spain
Chile	Japan	Turkey
Czechoslovakia	Mexico	United Kingdom
Denmark	Netherlands	U.S.A.
France	New Zealand	U.S.S.R.
Germany	Poland	Yugoslavia

No Member Body expressed disapproval of the Recommendation.

The Member Bodies of the following countries disapproved the transformation of ISO/R 332 into an International Standard :

Czechoslovakia
United Kingdom

Printed in Switzerland

Coal — Determination of nitrogen by the macro Kjeldahl method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the nitrogen content of hard coal, brown coal and lignite by the macro Kjeldahl method. An alternative semi-micro Kjeldahl method is given in ISO 333.[1)]

2 PRINCIPLE

Coal is heated with concentrated sulphuric acid in the presence of a mixed catalyst to convert the nitrogen into ammonium sulphate, from which the ammonia, released by distillation from alkaline solution, is absorbed in sulphuric acid, and the excess acid titrated with sodium or potassium hydroxide.

3 REAGENTS

All reagents shall be of analytical reagent quality, and distilled water shall be used throughout.

3.1 Potassium sulphate, anhydrous.

3.2 Selenium powder, or

3.3 Mercury(II) sulphate, or

3.4 Mixed catalyst, containing by mass :

32 parts of potassium sulphate (3.1);
1 part of selenium powder (3.2);
5 parts of mercury(II) sulphate (3.3).

Grind the above reagents in a mortar and mix them thoroughly.

3.5 Sucrose.

3.6 Sulphuric acid, ρ 1,84 g/l.

3.7 Sodium hydroxide, 400 g/l solution.

Dissolve 400 g of sodium hydroxide in water and dilute to 1 l.

3.8 Alkaline sodium sulphide solution.

Dissolve 20 g of sodium sulphide ($Na_2S.9H_2O$) in water, dilute to 50 ml, add 600 ml of the sodium hydroxide solution (3.7) and mix well.

3.9 Sulphuric acid, approximately 0,1 N.

3.10 Sodium hydroxide, 0,1 N solution, or

3.11 Potassium hydroxide, 0,1 N solution.

3.12 Methyl red indicator solution.

Dissolve 0,125 g of 4'-dimethylaminoazobenzene-2-carboxylic acid (methyl red) in 50 ml of ethanol.

4 APPARATUS

All volumetric apparatus shall be of the best analytical quality obtainable, and the balance used shall be sensitive to 0,1 mg.

4.1 Digestion flask Kjeldahl flask of borosilicate glass having a pear-shaped bulb of 200 to 500 ml effective capacity and a neck about 200 mm long and 23 mm in internal diameter. A suitable device for closing the mouth of the flask shall be provided, for example a light blown-glass stopper which fits loosely in the neck of the flask.

4.2 Heating arrangement, to heat one or more flasks inclined at about 35° from the vertical.

5 PREPARATION OF SAMPLE

The coal used for the determination of nitrogen content is the analysis sample ground to pass a sieve of 0,2 mm aperture. If necessary expose this sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrim with the laboratory atmosphere.

Before commencing the determination, mix the air-dried sample of coal for at least 1 min, preferably by mechanical means.

1) ISO 333, *Coal and coke — Determination of nitrogen by the semi-micro Kjeldahl method.*