International Standard 332

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Coal — Determination of nitrogen — Macro Kjeldahl method

Charbon — Dosage de l'azote — Méthode macrométrique de Kjeldahl

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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.

International Standard ISO 332 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This second edition was submitted directly to the ISO Council, in accordance with clause 5.10.1 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (ISO 332-1974), which had been 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	USA
France	New Zealand	USSR
Germany, F. R.	Poland	Yugoslavia

No member body had expressed disapproval of the document.

Coal — Determination of nitrogen — 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 method for coal is given in ISO 333.

2 References

ISO 333, *Coal — Determination of nitrogen — Semi-micro Kjeldahl method.*

ISO 1170, *Coal and coke — Calculation of analyses to different bases.*

3 Principle

A known mass of the sample 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.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Potassium sulphate, anhydrous.

4.2 Selenium powder, or

4.3 Mercury(II) sulphate, or

4.4 Mixed catalyst, containing by mass :

32 parts of the potassium sulphate (4.1);

1 part of the selenium powder (4.2);

5 parts of the mercury(II) sulphate (4.3).

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

NOTE — Other catalysts may be used if these can be shown to give the same results as those above.

4.5 Sucrose.

4.6 Sulphuric acid, ϱ 1,84 g/ml.

4.7 Sodium hydroxide, 400 g/l solution.

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

4.8 Sodium sulphide, alkaline 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 (4.7) and mix well.

4.9 Sulphuric acid, standard volumetric solution, $c(1/2\ H_2SO_4) = 0{,}1$ mol/l.

4.10 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0{,}1$ mol/l, or

4.11 Potassium hydroxide, standard volumetric solution, c(KOH) = 0,1 mol/l.

4.12 Methyl red indicator solution.

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

5 Apparatus

Ordinary laboratory apparatus : graduated glassware shall conform to the International Standards drawn up by ISO/TC 48, *Laboratory glassware and related apparatus*.

5.1 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.

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

5.3 Balance, accurate to 0,1 mg.

6 Preparation of sample

The coal used for the determination of nitrogen content is the analysis sample ground to pass a sieve of 200 µm aperture. If necessary, expose the sample in a thin layer for the minimum time required for the moisture content to reach approximate equilibrium with the laboratory atmosphere.

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

7 Procedure

7.1 Digestion

Weigh, to the nearest 0,1 mg, about 1 g of the sample, and transfer it to the dry digestion flask (5.1). Add 10 g of the potassium sulphate (4.1), followed by 0,2 g of the selenium powder (4.2) or 1 g of the mercury(II) sulphate (4.3) and 30 ml of the sulphuric acid (4.6) [when the mixed catalyst (4.4) is used, add 10 g of the mixture followed by 30 ml of the sulphuric acid (4.6)]. Place the digestion flask on the heating arrangement (5.2), close the open end of the neck, for example by means of the loose glass stopper, to prevent loss of sulphuric acid or intrusion of dust, and heat the mixture gently until the initial frothing has ceased. Heat the liquid to the boiling point, continue boiling freely until the solution becomes almost colourless, and then boil for a further period of 2 h.

7.2 Determination

Determine the quantity of ammonia present in the liquid by liberating it with sodium hydroxide [or alkaline sodium sulphide solution, where mercury(II) sulphate has been used] and distilling into an excess of sulphuric acid which is then titrated.

The following procedure is convenient :

Transfer the whole of the contents of the digestion flask, with about 200 ml of cold water, into a round-bottomed flask, fit it with a tap or thistle funnel and an efficient splash head and connect it, through a condenser if desired, to a bulbed tube dipping into a measured volume (say 25 ml) of the sulphuric acid solution (4.9) contained in a conical flask.

Add through a funnel 125 ml of the sodium hydroxide solution (4.7) [or the alkaline sodium sulphide solution (4.8) where mercury(II) sulphate has been used] and distil 150 to 200 ml of the liquid into the conical flask. Titrate the excess of sulphuric acid with the sodium hydroxide solution (4.10) or the potassium hydroxide solution (4.11), using the methyl red indicator solution (4.12).

7.3 Blank test

Carry out a blank test in exactly the same manner, but using 1,0 g of the sucrose (4.5) instead of the sample.

8 Expression of results

The nitrogen (N) content of the sample as analysed[1], expressed as a percentage by mass, is given by the formula

$$\frac{1{,}4\, c\, (V_2 - V_1)}{m}$$

where

V_1 is the volume, in millilitres, of the sodium hydroxide solution (4.10) or potassium hydroxide solution (4.11) used in the determination (7.2);

V_2 is the volume, in millilitres, of the sodium hydroxide solution (4.10) or the potassium hydroxide solution (4.11) used in the blank test (7.3);

c is the actual concentration, expressed in moles per litre, of the sodium hydroxide solution (4.10) or the potassium hydroxide solution (4.11);

m is the mass, in grams, of sample taken.

The result (preferably the mean of duplicate determinations, see clause 9) shall be reported to the nearest 0,01 %.

1) Calculation of the results to other bases is dealt with in ISO 1170.