UDC 662.6

Ref. No.: ISO/R 332 - 1963 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 332

DETERMINATION OF NITROGEN IN COAL BY THE KJELDAHL METHOD

1st EDITION

August 1963

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

Printed in Switzerland

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

BRIEF HISTORY

The ISO Recommendation R 332, *Determination of Nitrogen in Coal by the Kjeldahl Method*, was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels*, the Secretariat of which is held by the British Standards Institution (B.S.I.).

Work on this question by the Technical Committee began in 1950 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In April 1958, this Draft ISO Recommendation (No. 222) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in August 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 332 August 1963

DETERMINATION OF NITROGEN IN COAL BY THE KJELDAHL METHOD

1. 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.

2. APPARATUS

All volumetric apparatus should be of the best analytical quality obtainable, and the balance used should be sensitive to 0.1 mg.

2.1 **Digestion flask.** A Kjeldahl flask of borosilicate glass having a pear-shaped bulb of 200 to 500 ml effective capacity and a neck about 20 cm long and 2.3 cm in internal diameter. A suitable device for closing the mouth of the flask is provided, e.g. a light blown-glass stopper which fits loosely in the neck of the flask.

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

3. REAGENTS

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

3.1 *Potassium sulphate*, anhydrous.

3.2 *Selenium powder*, or

3.3 *Mercuric sulphate*, or

3.4 *Mixed catalyst*, containing by mass — 32 parts of potassium sulphate (3.1),
1 part of selenium powder (3.2), and
5 parts of mercuric sulphate (3.3).

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

3.5 *Sucrose*.

3.6 *Sulphuric acid*, relative density *d* 1.84.

3.7 *Sodium hydroxide solution*, 40 per cent (mass/volume). Dissolve 400 g of sodium hydroxide in water and dilute to 1 litre.

3.8 *Alkaline sodium sulphide solution.* Dissolve 20 g of sodium sulphide ($Na_2S \cdot 9H_2O$) 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 solution*, 0.1 N, or

3.11 *Potassium hydroxide solution*, 0.1 N.

3.12 *Methyl red indicator solution.* Dissolve 0.125 g of o-carboxybenzene-azo-dimethyl aniline (methyl red) in 50 ml of ethanol.

4. PROCEDURE

Before commencing the determination, mix the air-dried sample of coal, ground to pass a sieve of 0.2 mm aperture, for at least 1 minute, preferably by mechanical means.

Weigh to the nearest 0.1 mg about 1 g of the sample, and transfer it to the dry digestion flask. Add 10 g of the potassium sulphate (3.1), followed by 0.2 g of the selenium powder (3.2) *or* 1 g of the mercuric sulphate (3.3) and 30 ml of the sulphuric acid (3.6); (when the mixed catalyst is used, add 10 g of the mixture (3.4) followed by 30 ml of the sulphuric acid (3.6)). Place the digestion flask on the heating arrangement, close the open end of the neck, e.g. 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 two hours.

Determine the quantity of ammonia present in the liquid by liberating it with sodium hydroxide (or alkaline sodium sulphide solution, where mercury has been used) and distilling it into an excess of standard acid.

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 (3.9) contained in a conical flask.

Add through a funnel 125 ml of the sodium hydroxide solution (3.7) (or the alkaline sodium sulphide solution (3.8) where mercury has been used) and distil 150 ml to 200 ml of the liquid into the conical flask. Titrate the excess of sulphuric acid with the sodium hydroxide solution (3.10) or the potassium hydroxide solution (3.11), using the methyl red indicator solution (3.12).

4.1 Blank determination. Carry out a blank determination in exactly the same manner, but using 1.0 g of the sucrose (3.5) instead of the coal.

5. CALCULATION AND EXPRESSION OF RESULTS

If m = mass of coal taken, expressed in grammes,

a = volume of 0.1 N sodium or potassium hydroxide solution used in the test, expressed in millilitres,

b = volume of 0.1 N sodium or potassium hydroxide solution used in the blank determination, expressed in millilitres,

F = normality of sodium or potassium hydroxide solution (i.e. 0.1000, if the solution is exactly decinormal),

N = nitrogen in the coal as analysed, expressed in per cent,

$$N = \text{nitrogen per cent in the coal} = \frac{1.4F\,(b - a)}{m}$$