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R 333

DETERMINATION OF NITROGEN IN COAL BY THE SEMI-MICRO KJELDAHL METHOD

1st EDITION

August 1963

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

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BRIEF HISTORY

The ISO Recommendation R 333, *Determination of Nitrogen in Coal by the Semi-micro 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 1951 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In April 1958, this Draft ISO Recommendation (No. 223) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

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

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 333 August 1963

DETERMINATION OF NITROGEN IN COAL BY THE SEMI-MICRO KJELDAHL METHOD

1. PRINCIPLE

Coal (see Note below) 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 steam distillation from alkaline solution, is absorbed in boric acid and determined by titration with sulphuric acid.

NOTE.- In bituminous coals and anthracite, the nitrogen is fairly uniformly distributed, even when the seam is banded; in some lignites the nitrogen varies in the different bands of the seam, and sampling difficulties make it undesirable to employ a semi-micro method using only a 0.1 g sample, so that the Kjeldahl method using a 1 g sample should be used for such banded lignites.

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 flask of borosilicate glass, of bulb capacity 50 ml, preferably pistol-shaped, with a light borosilicate blown-glass bulb which is a loose fit in the neck of the flask, to prevent loss of acid.

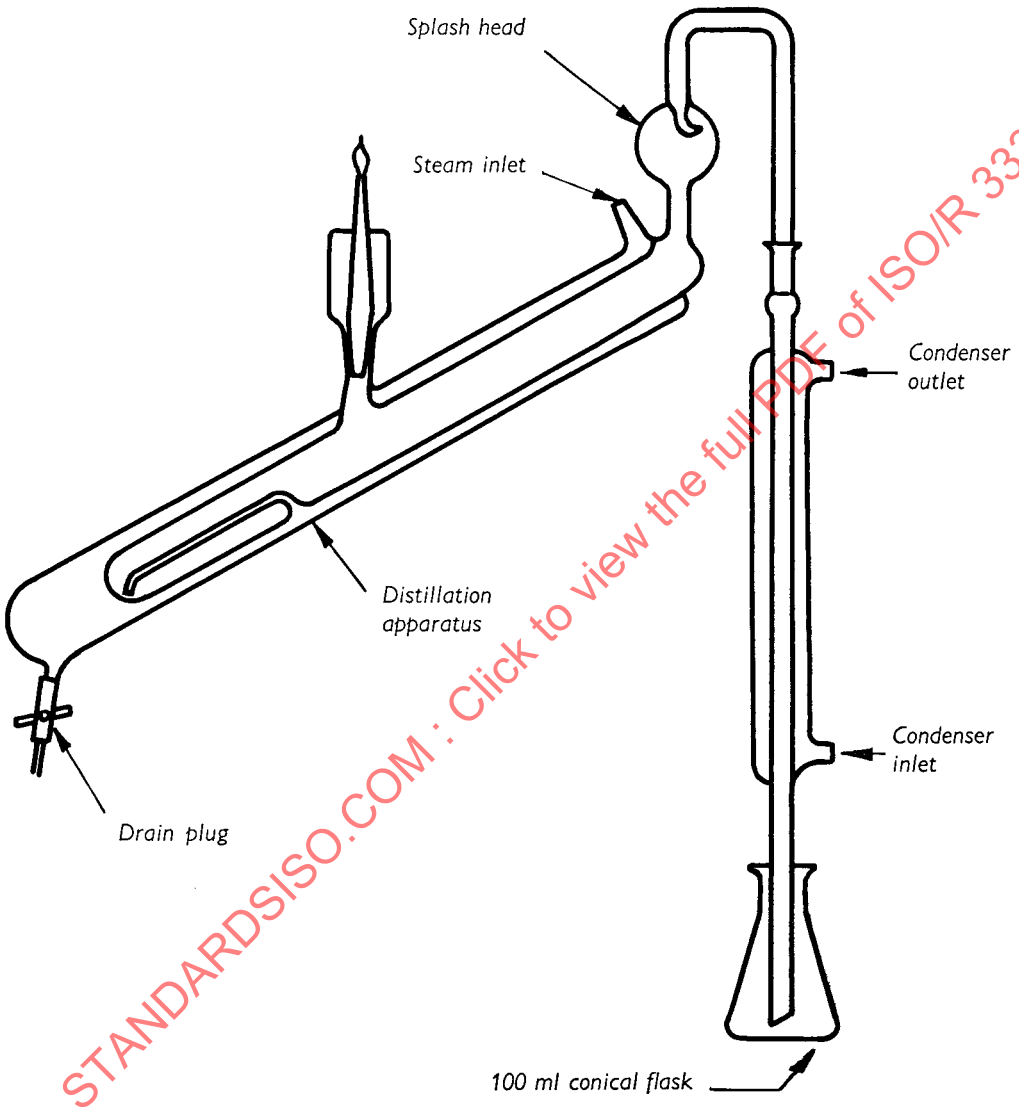
2.2 **Distillation apparatus.** A suitable water steam distillation apparatus of borosilicate glass, fitted with a splash head to arrest the passage of any entrained sodium hydroxide from the distillation flask (see Fig. 1, page 4).

2.3 **Receiver.** A wide-necked, flat-bottomed conical flask, of 100 ml capacity.

2.4 **Burette,** of 25 ml capacity.

2.5 **Heating arrangement**, to heat simultaneously one or more flasks inclined at about 35° from the vertical. A suitable arrangement is illustrated in Figure 2 and in the Table, page 6.

FIG. 1. — **Suitable distillation apparatus**



3. REAGENTS

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

3.1 *Mixed catalyst*, containing by mass —

32 parts of anhydrous potassium sulphate,

1 part of selenium powder, and

5 parts of mercuric sulphate.

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

3.2 *Sucrose.*

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

3.4 *Boric acid solution, saturated.* Dissolve 60 g of boric acid in 1 litre of hot water, cool and allow to mature for three days before decanting the clear liquid.

3.5 *Alkaline sodium sulphide solution.* Dissolve 20 g of sodium sulphide ($Na_2S \cdot 9H_2O$) in water, and dilute to 50 ml; add a solution of 240 g of sodium hydroxide in 600 ml of water, and mix well.

3.6 *Sulphuric acid*, 0.01 N.

3.7 *Mixed indicator solution:*

SOLUTION A. — Dissolve 0.125 g of o-carboxybenzene-azo-dimethyl aniline (methyl red) in 60 ml of ethanol or industrial spirit, and dilute to 100 ml with water.

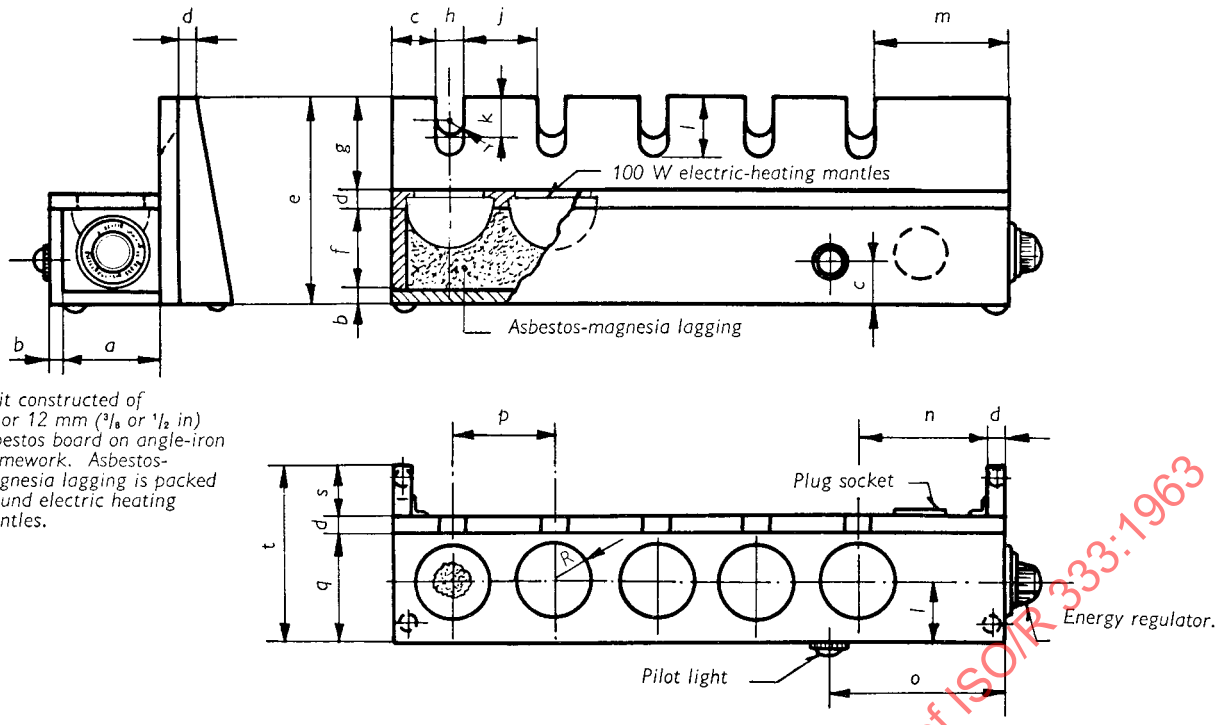
SOLUTION B. — Dissolve 0.083 g of 3: 7 bisdimethylaminophenothiazinium chloride (methylene blue) in 100 ml of ethanol or industrial spirit. Store in a dark glass bottle.

Mix equal volumes of solutions A and B. Do not use the mixed solution after more than one week.

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 0.10 g of the sample, transfer carefully to the digestion flask, add 2.0 g of the mixed catalyst (3.1) and shake to mix. Add 4 ml of the sulphuric acid (3.3) and mix again.



Unit constructed of 10 or 12 mm (3/8 or 1/2 in) asbestos board on angle-iron framework. Asbestos-magnesia lagging is packed around electric heating mantles.

FIG. 2. — **Electric digestion unit suitable for semi-micro Kjeldahl method**

TABLE. — **Dimensions for electric digestion unit**

Dimensions	Millimetres *	Inches	Dimensions	Millimetres *	Inches
a	73	2 7/8	*l*	44.5	1 3/4
b	9.5	3/8	*m*	98	3 7/8
c	31.8	1 1/4	*n*	95	3 3/4
d	12.7	1/2	*o*	130	5 1/8
e	152	6	*p*	76	3
f	60	2 3/8	*q*	82	3 1/4
g	69	2 3/4	*r*	9.5	3/8
h	19	3/4	*R*	28.5	1 1/8
j	57	2 1/4	*s*	38	1 1/2
k	28.5	1 1/8	*t*	133	5 1/4

* The Metric dimensions have been rounded off.