

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 333

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

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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.

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.