

INTERNATIONAL
STANDARD

ISO
333

Fourth edition
1996-03-15

**Coal — Determination of nitrogen —
Semi-micro Kjeldahl method**

iTeh STANDARD PREVIEW
(standards.iteh.ai)

*Charbon — Dosage de l'azote — Méthode semi-micrométrique de
Kjeldahl*

ISO 333:1996

<https://standards.iteh.ai/catalog/standards/sist/1e009f24-d5f4-46eb-b11d-ced2ee92b50a/iso-333-1996>



Reference number
ISO 333:1996(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 333 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*, Subcommittee SC 5, *Methods of analysis*.

This fourth edition cancels and replaces the third edition (ISO 333:1983), which has been technically revised.

Annex A of this International Standard is for information only.

Coal — Determination of nitrogen — Semi-micro Kjeldahl method

1 Scope

This International Standard specifies a method of determining the nitrogen content of hard coal, brown coal and lignite by the semi-micro Kjeldahl method.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method.*

ISO 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method.*

ISO 1170:1977, *Coal and coke — Calculation of analyses to different bases.*

ISO 1988:1975, *Hard coal — Sampling.*

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method.*

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis.*

3 Principle

A known mass of the sample is heated with concentrated sulfuric acid in the presence of a mixed catalyst to convert the nitrogen into ammonium sulfate, from which the ammonia, released by steam distillation from alkaline solution, is absorbed in boric acid and determined by titration with sulfuric acid.

4 Reagents

WARNING — Care should be exercised when handling reagents, many of which are toxic and corrosive.

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

4.1 Mixed catalyst, containing by mass:

90 parts of anhydrous potassium sulfate;

2 parts of selenium powder;

5 parts of vanadium pentoxide.

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

4.2 Sucrose.

4.3 Sulfuric acid, concentrated, approximately 98 % (m/m).

4.4 Boric acid solution, saturated.

Dissolve 60 g of boric acid in 1 litre of hot water, cool and allow to stand for 3 d before decanting the clear solution.

4.5 Sodium hydroxide, 400 g/l solution.

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

NOTE 1 It is advisable to stir the mixture frequently while dissolving in order to dissipate the heat of solution.

4.6 Sulfuric acid, standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0,005 \text{ mol/l}$.**4.7 Mixed indicator solution.****4.7.1 Solution A.**

Dissolve 0,125 g of 2-(4-dimethylaminophenylazo) benzoic acid, sodium salt (methyl red) in 60 ml of ethanol and dilute to 100 ml with water.

4.7.2 Solution B.

Dissolve 0,083 g of 3,7-bis(dimethylamino)phenthiazin-5-ylum chloride (methylene blue) in 100 ml of water. Store in a dark bottle.

4.7.3 Mixed solution.

Mix equal volumes of solution A and solution B. Store in a dark bottle. Discard the mixed solution after 1 week.

5 Apparatus

5.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

5.2 Graduated glassware, conforming to the requirements for Grade "A" in the International Standards prepared by ISO/TC 48, *Laboratory glassware and related apparatus*.

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

5.4 Distillation apparatus, comprising a suitable water/steam distillation flask of borosilicate glass, fitted with a splash head to arrest the passage of any entrained sodium hydroxide from the distillation flask and a condenser.

An example of a suitable distillation apparatus is illustrated in figure 1.

5.5 Receiver, a wide-necked, flat-bottomed conical flask, of capacity 100 ml.

5.6 Heating apparatus, capable of heating one or more digestion flasks while supporting it at an angle of 35° from the vertical. An electric heating unit or a gas microburner may be used. An example of a suitable electric heating unit, capable of heating several flasks simultaneously, is shown in figure 2.

6 Preparation of the test sample

Prepare the test sample for general analysis in accordance with ISO 1988, or ISO 5069-2, as appropriate. Ensure that the moisture content of the sample is in equilibrium with the laboratory atmosphere, exposing it, if necessary, in a thin layer for the minimum time required to achieve equilibrium.

Before commencing the determination, thoroughly mix the test sample for at least 1 min, preferably by mechanical means.

If the results are to be calculated other than on an "air-dried" basis (see clause 9), after weighing the test portion, determine the moisture content using a further portion of the test sample by the method described in ISO 331, ISO 1015 or ISO 5068, as appropriate.

7 Procedure**7.1 Digestion**

Weigh, to the nearest 0,1 mg, about 0,1 g of the test sample and transfer it quantitatively to the digestion flask (5.3). Add 2 g of the mixed catalyst (4.1) and shake to mix. Add 4 ml of the sulfuric acid (4.3) and mix again.

Switch on the electric heating unit (5.6) at approximately half load for 20 min, and then at maximum load for 10 min before placing the flask on the unit. Alternatively, with gas heating, place the digestion flask over the fully aerated flame of a microburner and adjust the flame so that clearing of the solution is effected in 7 min to 10 min, for example by using a flame 50 mm high with the lowest part of the bulb of the flask 40 mm above the top of the burner. After the solution become clear (see note 2), continue boiling for a further 20 min, in order to complete the conversion of the nitrogenous compounds into ammonium sulfate.

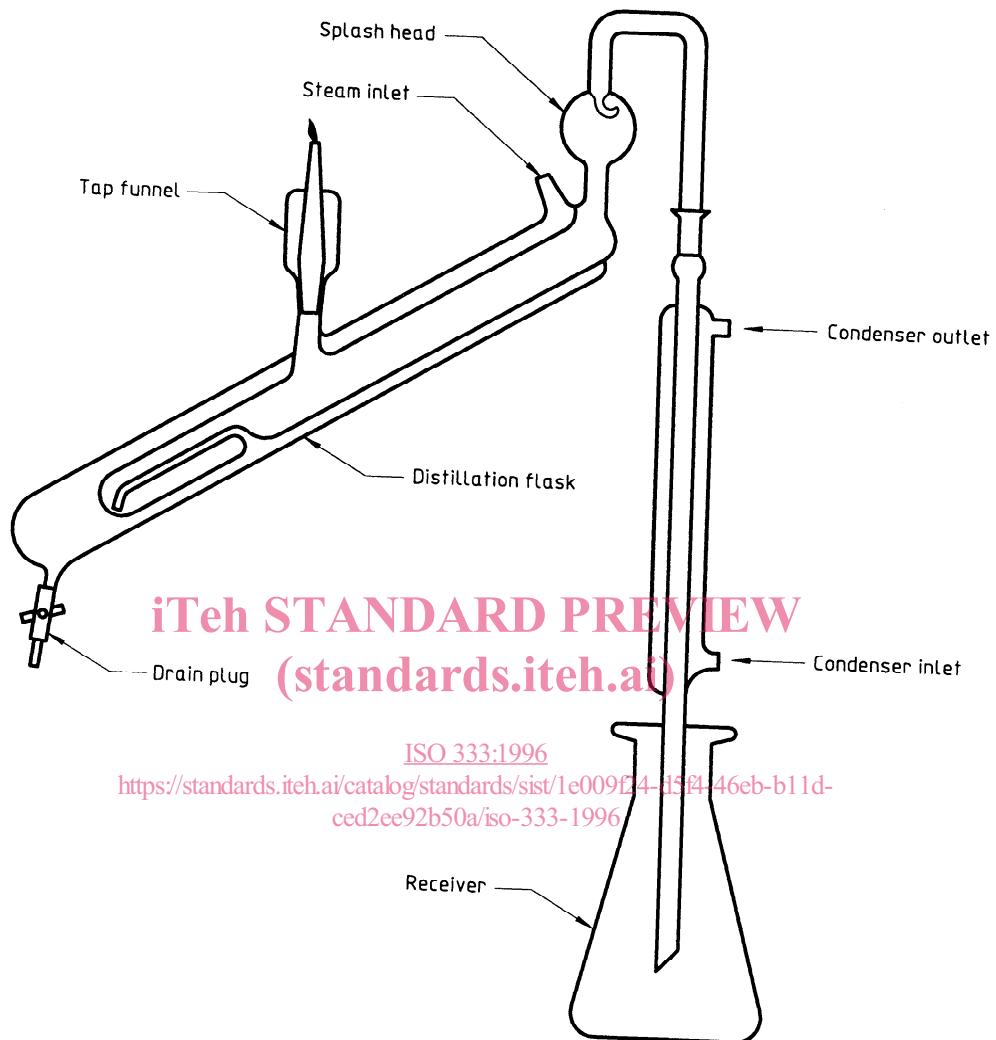
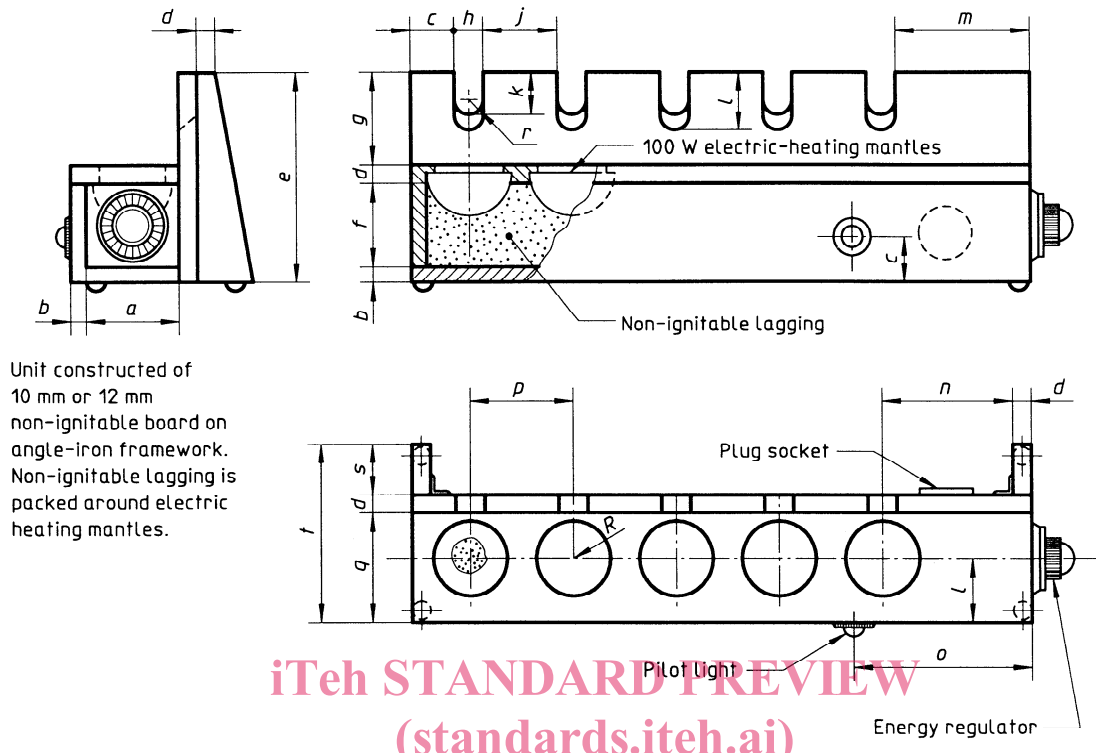


Figure 1 — Suitable distillation apparatus



Unit constructed of 10 mm or 12 mm non-ignitable board on angle-iron framework. Non-ignitable lagging is packed around electric heating mantles.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

Dimensions	ISO 333:1996	mm
a	73	
b	9,56	
c	31,8	
d	12,7	
e	152	
f	60	
g	69	
h	19	
j	57	
k	28,5	
l	44,5	
m	98	
n	95	
o	130	
p	76	
q	82	
r	9,5	
R	28,5	
s	38	
t	133	

Figure 2 — Electric digestion unit suitable for semi-micro Kjeldahl method

NOTE 2 If it is impossible to obtain a clear solution in the time specified, the digestion period may be extended, providing this deviation in the procedure is mentioned in the test report. The use of a long digestion period is likely to affect the accuracy of the results.

7.2 Distillation

While the digestion is progressing, condition the distillation apparatus (5.4) (see note 3), close the connection to the distillation flask, and blow out the waste from the previous distillation. Pour 20 ml of the sodium hydroxide solution (4.5) into the distillation flask through the funnel, and rinse with water. Measure 2 ml of the boric acid solution (4.4) into the receiver (5.5), and add 4 drops of the mixed indicator solution (4.7). Adjust the receiver, so that the end of the condenser tube dips below the surface of the liquid.

Allow the digest to cool for 5 min, and dilute by adding 10 ml of distilled water, in drops at first, and shaking until the reaction is less violent. Pour the diluted digest through the tap funnel into the distillation flask; rinse the digestion flask into the tap funnel with water, then rinse the tap funnel with water (see note 4). The total volume of liquid in the distillation flask should not exceed half the capacity of the flask, or frothing and priming may occur.

Heat the distillation flask with a small flame and pass steam into the apparatus. Distil for 5 min from the time the steam enters the condenser, at a rate of about 4 ml of distillate per minute. Lower the receiver so that the condenser end is about 25 mm above the surface of the liquid, and continue distillation for 1 min longer. The contents of the receiver should remain cold. Rinse the end of the condenser tube with water.

Add a few drops of the mixed indicator solution (4.7) to the distillate and titrate the ammonia present in the distillate with the sulfuric acid solution (4.6) until the grass-green colour changes to steel-grey, a further drop then giving the purple (acid) colour.

NOTES

3 The distillation apparatus should be conditioned by steaming through for 30 min before commencing a batch of distillations. There is no need, however, to do this between individual distillations in a batch.

4 A small volume of water left in the funnel will help to seal the inlet against the escape of gases.

8 Blank test

Carry out a blank test as described in clause 7, but using 0,1 g of the sucrose (4.2) instead of the test portion.

NOTE 5 If the splash head removes all entrained sodium hydroxide, the results of the blank test should not exceed 0,1 ml of 0,005 mol/l sulfuric acid (4.6). Steam removes alkali compounds from some glasses and, if higher blank results are found, the central tube of the condenser should be replaced by a fused silica or borosilicate glass tube.

9 Expression of results

The nitrogen content, w_N , of the sample as analysed, expressed as a percentage by mass, is given by the formula

$$w_N = \frac{2,8c(V_1 - V_2)}{m}$$

V_1 is the volume, in millilitres, of the sulfuric acid solution (4.6) used in the determination;

V_2 is the volume, in millilitres, of the sulfuric acid solution used in the blank test;

c is the concentration, in moles per litre, of the sulfuric acid solution;

m is the mass, in grams, of the test portion.

NOTE 6 The derivation of the factor used in the above calculation is detailed in annex A.

Report the results as the mean of duplicate determinations, to the nearest 0,01 %.

The results of the determination described in this International Standard are reported on the "air-dried" basis. The calculation of results to other bases is dealt with in ISO 1170.

10 Precision

10.1 Repeatability limit

The results of duplicate determinations, carried out at different times, in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same test sample, should not differ by more than 0,05 % absolute.

10.2 Reproducibility critical difference

The means of the results of duplicate determinations, carried out in each of two laboratories, on representative portions taken from the same sample after the last stage of sample preparation, should not differ by more than 0,10 % absolute, at the 95 % confidence level.

11 Test report

The test report shall include the following information:

- a) The method used by reference to this International Standard;
- b) an identification of the sample tested;
- c) the results and the method of expression used;
- d) any unusual features noted during the determination;
- e) any operation not included in this International Standard, or regarded as optional;
- f) the date of the test.

iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO 333:1996

<https://standards.iteh.ai/catalog/standards/sist/1e009f24-d5f4-46eb-b11d-ced2ee92b50a/iso-333-1996>

Annex A (informative)

Derivation of factor used in the calculation in clause 9

The percentage, w_N , of nitrogen in the test sample is given by the formula

$$w_N = \frac{(V_1 - V_2) \times 2 \times 14,006 \times c \times 100}{1\,000 \times m}$$

$$= \frac{2,8c(V_1 - V_2)}{m}$$

where

m is the mass, in grams, of the test portion;

V_1 is the volume, in millilitres, of the sulfuric acid solution (4.6) used in the determination;

V_2 is the volume, in millilitres, of the sulfuric acid solution used in the blank test;

c is the concentration, in moles per litre, of the sulfuric acid solution;

14,006 is the relative atomic mass of nitrogen.

1 ml of the sulfuric acid

$$\cong \frac{2w_N \times c}{1\,000}$$

$$= \frac{2 \times 14,006 \times c}{1\,000} \text{ g of nitrogen}$$

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 333:1996

<https://standards.iteh.ai/catalog/standards/sist/1e009f24-d5f4-46eb-b11d-ced2ee92b50a/iso-333-1996>