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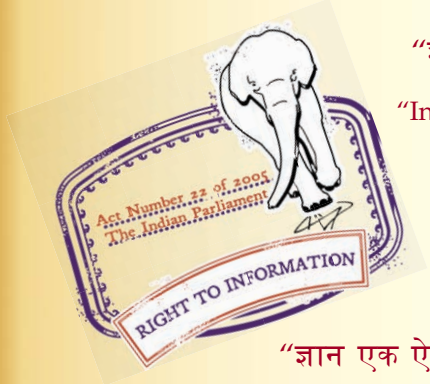

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IS : 5194 - 1969
(Reaffirmed 2002)

Indian Standard

METHOD FOR DETERMINATION OF NITROGEN—KJELDAHL METHOD

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 2 *October* 1969

Indian Standard

METHOD FOR DETERMINATION OF NITROGEN—KJELDAHL METHOD

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Indian Standard

METHOD FOR DETERMINATION OF NITROGEN—KJELDAHL METHOD

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 June 1969, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The Kjeldahl method for the determination of nitrogen is convenient for determining ammonia producing nitrogen, especially in organic compounds. This method has been increasingly adopted in Indian Standards on chemicals. This standard is intended to assist the various technical committees of the Indian Standards Institution in preparing chemical standards by avoiding unnecessary variations in the details of the method. It is also intended to be of use to testing laboratories.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the Kjeldahl method for the determination of amino nitrogen in organic materials.

2. PRINCIPLE OF THE METHOD

2.1 The sample is digested with concentrated sulphuric acid in the presence of a catalyst to convert the organic nitrogen into ammonium sulphate from which the ammonia is liberated by distillation with concentrated alkali solution. The ammonia so evolved is absorbed in standard sulphuric acid and the excess acid is titrated with standard alkali solution. Alternatively, in the modified method, the ammonia evolved is absorbed in boric acid and titrated against standard acid.

*Rules for rounding off numerical values (*revised*).

2.1.1 No single digestion procedure which gives good results with all nitrogen containing compounds can be recommended. As a general guide, however, the use of potassium sulphate and a mercury catalyst as the most reliable mixture, particularly when prolonged digestion is required, is suggested. The mercury-selenium catalyst is more effective, but prolonged digestion should be avoided. Copper sulphate and selenium have been effectively used as catalyst for the analysis of biological materials. This mixture is probably not as efficient as the mercury-selenium catalyst but its use obviates the necessity of precipitating mercury before distillation of the ammonia. The time of digestion is reduced when selenium is used as a catalyst. The use of oxidizing agents, such as potassium permanganate or hydrogen peroxide, may be advantageous, particularly when a large amount of carbonaceous matter is to be destroyed. The organic nitrogen is not always completely converted into ammonium sulphate when the digest has become 'charfree', since some compounds, for example, pyridine carboxylic acids, do not char when heated with concentrated sulphuric acid. It is, therefore, particularly important not to confuse 'charring time' with 'digestion time'. In many cases, a considerable 'after boil' may be necessary to obtain complete conversion to ammonia.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. APPARATUS

4.1 Kjeldahl Flask — 500 ml capacity.

4.2 Distillation Assembly — The assembly shown in Fig. 1 consists of a round-bottom flask *A* of 1 000 ml capacity fitted with a rubber stopper having two holes, through one of which passes one end of the connecting bulb tube *B* and through the other the end of the tap or separating funnel *F* which dips into the contents of the flask. The other end of the bulb tube is connected to the condenser *C*. The lower end of the condenser is attached by means of a rubber tube to a dip tube *D* which dips into a known quantity of acid (sulphuric or boric), contained in a beaker *E* of 500 ml capacity, to which 3 to 4 drops of indicator solution has been added.

5. REAGENTS

5.1 Potassium Sulphate or Anhydrous Sodium Sulphate

*Specification for water, distilled quality (*revised*).

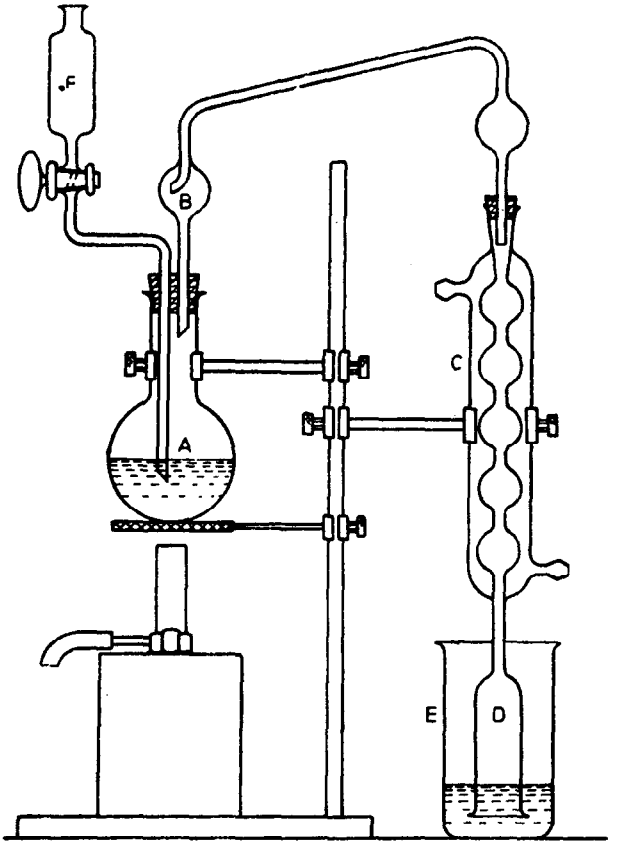

FIG. 1 DISTILLING ASSEMBLY FOR DETERMINATION OF NITROGEN

5.2 Copper Sulphate or Selenium Powder or Mercury or any Other Suitable Mixed Catalyst — *See* 2.1.1.

5.3 Concentrated Sulphuric Acid — conforming to IS:266-1961*.

5.4 Sodium Hydroxide Solution — Dissolve about 450 g of sodium hydroxide (pellets, flakes, sticks or lumps) in 1 000 ml of water.

5.5 Standard Sulphuric Acid — 0·5 N.

5.6 Standard Sodium Hydroxide Solution — 0·25 N.

*Specification for sulphuric acid (*revised*).

5.7 Alkaline Sodium Sulphide Solution — Dissolve 20 g of sodium sulphide ($Na_2S.9H_2O$) in water, dilute to 50 ml, add 600 ml of sodium hydroxide solution (*see* **5.4**) and mix well.

5.8 Methyl Red Indicator Solution — *See* IS:2263-1962*.

5.9 Boric Acid Solution — saturated. Dissolve 60 g of boric acid in 1 litre of hot water, cool and allow to mature for 3 days before decanting the clear liquid.

5.10 Mixed Indicator Solution — methyl red and methyl blue prepared as prescribed in Table III of IS:2263-1962*.

6. PROCEDURE

6.1 Weigh accurately a suitable quantity of the finely ground sample into the Kjeldahl flask. The quantity of the sample taken shall be such that the ammonia liberated neutralizes not more than 40 ml of standard sulphuric acid or boric acid taken in the beaker *E*. Add 10 g of potassium sulphate or anhydrous sodium sulphate, 0·5 to 1 g of the catalyst and 30 ml, or more if necessary, of concentrated sulphuric acid. Place the digestion flask in an inclined position and close the flask with a loosely fitting, pear shaped, hollow glass stopper to prevent loss of sulphuric acid or entry of dust. Heat the mixture gently in a fume cupboard until the initial frothing has ceased. If the sample tends to foam or froth, heat very gently in the initial stages; a small piece of paraffin or zinc may also be added to reduce frothing, if necessary. Heat the liquid to boiling point. Continue boiling freely until the solution becomes clear and then boil for a further period of about two hours. Cool the contents of the flask.

6.1.1 Transfer completely the contents of the digestion flask into the round-bottom flask of the distillation assembly, using water. Add a few pieces of pumice stone. Place a measured volume (normally 50 ml is sufficient) of standard sulphuric acid in the beaker *E* and add 3 drops of methyl red indicator. Fit up the distillation assembly. Add an excess of sodium hydroxide solution (or alkaline sodium sulphide solution where mercury is used as catalyst), through the separating funnel, and mix with the contents of the flask by mild shaking, so as to make the solution alkaline. Distil about one-third of the total volume of the solution in the flask. Cool and dismantle the distillation assembly. Wash the condenser and the dip tube with water, collecting the washings in the beaker *E*. Titrate the excess of sulphuric acid in beaker *E* with standard sodium hydroxide solution. Carry out a blank determination in the same manner using the same quantities of all the reagents but without the sample.

*Methods of preparation of indicator solutions for volumetric analysis.

6.1.1.1 *Calculation*

$$\text{Nitrogen, percent by weight} = \frac{1{\cdot}4\ (V_2 - V_1)\ N}{W}$$

where

V_1 = volume in ml of standard sodium hydroxide solution used to neutralize the excess acid in the determination with the sample,

V_2 = volume in ml of standard sodium hydroxide solution used to neutralize the excess acid in the blank determination,

N = normality of the standard sodium hydroxide solution, and

W = weight in g of the sample taken for the test.

6.1.2 Alternatively, the ammonia evolved by distillation shall be absorbed in boric acid. Carry out digestion as prescribed in **6.1**. Transfer completely the contents of the digestion flask into the round-bottom flask through the separating funnel. Rinse the separating funnel with water. The total volume of liquid in the distillation flask should not exceed half the capacity of the flask otherwise frothing may occur. Add excess of sodium hydroxide solution (or alkaline sodium sulphide solution when mercury is used as catalyst) to make the solution alkaline. Connect immediately the round-bottom flask to steam trap and condenser. The condenser should be arranged to dip the dip tube in 50 ml of boric acid which is kept cool in beaker *E*. Add 2-3 drops of the mixed indicator. Distil about one third of the total volume of the solution in the flask. Cool and dismantle the distillation assembly. Rinse the tip of the condenser and the dip tube with water, collecting the washings in the beaker *E*. Titrate the ammonia present in the distillate with sulphuric acid until the grass green colour changes to steel grey, a further drop then giving the purple colour.

6.1.2.1 *Calculation*

$$\text{Nitrogen, precent by weight} = \frac{1{\cdot}4 \times V \times N}{W}$$

where

V = volume in ml of standard sulphuric acid used in titration,

N = normality of standard sulphuric acid, and

W = weight in g of the sample taken for the test.

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Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 APRIL 1979

TO

IS:5194-1969 METHOD FOR DETERMINATION OF NITROGEN-KJELDAHL METHOD

Addendum

(*Page 4, clause 4.2*) - Add the following note after 4.2:

'Note - In order to avoid back suction of the liquid in the beaker, presence of positive pressure by introduction of gas (nitrogen gas or air free from carbon dioxide) would make the operation smoother.'

(CDC 1)

Reprography Unit, BIS, New Delhi, India