भारतीय मानक

मृदा में नत्रजन एवं नत्रजनीय यौगिकों को ज्ञात करना

Indian Standard

DETERMINATION OF NITROGEN AND NITROGENOUS COMPOUNDS IN SOILS

ICS 65.080

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

April 1999

Price Group 1

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Soil Quality and Improvement Sectional Committee had been approved by the Food and Agriculture Division Council.

Nitrogen is one of the most essential nutrients required for successful crop production. It promotes vegetative growth of the plants. Several Indian soils are deficient in nitrogen content and no standard method is available for testing of nitrogen in soils. Hence, need was felt to provide appropriate analytical procedure for determination of nitrogen and nitrogenous compounds in soils. This standard may help various research organizations and those engaged in organized farming including plantation and forestry crops.

In reporting the result of a test made in accordance with the standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

DETERMINATION OF NITROGEN AND NITROGENOUS COMPOUNDS IN SOILS

1 SCOPE

This standrd prescribes the methods for the determination of total nitrogen and nitrogen compounds in soils.

2 REFERENCES

The Indian Standard listed below contains provisions which through reference in this text, constitutes provision of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)

3 DETERMINATION OF NITROGEN IN SOIL (EXCLUDING NITRATE)

3.1 Apparatus

3.1.1 *Macro-Kjeldahl Digestion Flask,* 500 or 800 ml.

3.1.2 *Macro-Kjedahl Digestion Unit*

3.1.3 *Macro-Kjeldahl Distillation Apparatus*

3.1.4 *Vacuum Pump*

3.1.5 *Mechanical Shaker*

3.1.6 *Hot Air Oven*

3.2 Reagents

3.2.1 *Sulphuric Acid,* Concentrated.

3.2.2 *Potassium Sulphate,* Analytical reagent grade.

3.2.3 *Copper Sulphate* ($CuSO_4 5H_2O$), Analytical reagent grade.

3.2.4 *Sodium Hydroxide Solution*

40 percent (*m/v*) solution in reagent grade water, kept in a tightly stoppered pyrex glass bottle.

3.2.5 *Mixed Indicator Solution*

Dissolve 0.5 g bromocresol green and 0.1 g methyl red in 95 percent (*m/v*) methanol.

3.2.6 *Boric Acid*

2 percent (*m/v*) aqueous solution containing 5 ml of mixed indicator solution per litre. The *p*H of the solution shall be adjusted to 5.0.

3.2.7 *Standard Sulphuric Acid Solution,* 0.1 N.

3.2.8 *Salicylic Acid: Sulphuric Acid Mixture*

Dissolve 25 g salicylic acid in one litre of concentrated sulphuric acid.

3.2.9 *Sodium Thiosulphate* ($Na_2S_2O_3$, $5H_2O$), Analytical reagent grade.

3.2.10 *Potassium Chloride Solution (IN)*

Dissolve 74.5 g potassium chloride analytical reagent grade, in one litre distilled water.

3.2.11 *Magnesium Oxide* — Analytical reagent grade (Heavy grade).

3.2.12 *Devarda's Alloy*

It is a mixture of Copper 50 percent (*m/m*), Aluminium 45 percent (*m/m*) and Zinc 5 percent (*m/m*) and is available commercially (Lab grade or Reagent grade).

3.3 General

The Kjeldahl procedure is generally employed for determination of total nitrogen and Kjeldahl's wet digestion distillation procedure for determination of total nitrogen involves two steps, namely:

a) *Digestion* — Heating the sample with sulphuric acid containing potassium sulphate, and copper sulphate as catalyst, thus oxidising the organic matter and converting organic nitrogen into ammonium nitrogen; and

b) *Distillation* — Distilling the digest with excess of above 40 percent solution of sodium hydroxide to recover ammonia in excess of standard boric acid which is thereafter titrated against standard acid.

3.3.1 *Procedure*

Take 10 g of dry soil sample passed through 2 mm sieve into a 500 ml or 1 000 ml Kjeldahl flask and add 10 g of digestion mixture comprising of potassium sulphate: copper sulphate 10:1 or 10.5; (*m/m*). Add

30 ml (6 ml/g of organic matter in soil) of concentrated sulphuric acid and heat the flask continuously avoiding excessive frothing by controlling heat. Raise the temperature to about 380°C and digest till the contents of the flask are clear. Continue digestion for about 15 min and after complete digestion allow the flask to cool and add 100 ml reagent grade water.

To determine ammonical nitrogen liberated by digestion, place a 500 ml Erlenmeyer flask containing 50 ml of boric acid under the condenser of distillation apparatus so that the end of the condenser is below the surface of the boric acid. Hold the distillation flask. Cool, dilute and digest at 45° angle. Add glass beads and pour about 150 ml of sodium hydroxide solution (*see* **3.2.4**), down the neck of the flask so that the alkali reaches the bottom of the flask without mixing appreciably with the digest. Attach the flask quickly to the distillation apparatus, mix the contents throughly by gentle swirling and heat. Control the heating to prevent sucking by back of boric acid and to minimize frothing during distillation. Check the flow of cold water through the condenser is sufficient to keep the temperature of the distillate less than 35°C. Collect about 150 ml of distillate, rinse the condenser with water and stop distillation. Determine total ammonical nitrogen in the distillate by titration with standard sulphuric acid. The colour change at end point of titration is from green to pink.

3.3.2 *Calculation*

Calculate total nitrogen in ammonical form, percent by mass of soil taken for the test by using the following formula:

1 ml of 0.1 N standard sulphuric acid = 0.001 4 g of nitrogen.

4 ESTIMATION OF TOTAL NITROGEN INCLUDING NITRATE FORM

Take 10 g of soil sample in a Kjeldahl flask, add 35 ml of salicylic acid sulphuric acid mixture (*see* **3.2.7** and **3.2.8**) and mix well. allow the mixture to stand overnight, add 5 g of sodium thiosulphate (*see* **3.2.9**) and heat the flask till frothing subsides. Cool the flask, add 10 g of digestion mixture comprising of potassium sulphate: copper sulphate (*see* **3.2.2** and **3.2.3**) 10:1 or 10 : 0.5 (*m/m*) and complete the digestion. Determine ammonical nitrogen including nitrate by distillation as described in **3.3.1** and **3.3.2**.

5 DETERMINATION OF AMMONICAL, NITRITE AND NITRATE NITROGEN IN SOIL EXTRACTS

5.1 General

Ammonical nitrogen in solutions containing glutamine or other alkali labile organic nitrogen compounds may be estimated quantitatively from the ammonical nitrogen liberated by distillation of these solutions with a small amount of magnesium oxide and determining ammonical nitrogen titrimetrically. The solution is then cooled, powdered Devarda's alloy is added and redistilled to recover ammonical nitrogen formed as a result of reduction of nitrite and nitrate forms of nitrogen.

5.2 Procedure

5.2.1 *Extraction of Inorganic Nitrogen from Soils*

Take about 50 g of fresh, wet soil sample in an Erlenmeyer flask, add 250 ml IN acidified potassium chloride solution, stopper the flask and shake on a mechanical shaker for one hour. Filter the contents of the flask using a vacuum pump or using a funnel and Whatman No.1 filter paper or equivalent.

Transfer the filtrate to a 1 000 ml Kjeldahl digestion flask and add about 2 to 4 g magnesium oxide carefully. Place a 250 ml Erlenmeyer flask containing 20 ml of 2 percent boric acid (*see* **3.2.6**) indicator solution under the condenser, the tip being placed below the boric acid level in the flask. Connect the Kjeldahl flask to the condenser and distill by heating. Collect about 40 to 45 ml of distillate and stop distillation. Titrate the ammonia against standard sulphuric acid. 1 ml of 0.02 N standard sulphuric acid is equivalent to 0.000 28 g of nitrogen.

5.2.2 *Determination of Nitrite and Nitrate Nitrogen*

Cool the flask after distillation (*see* **5.2**) carefully add 1 to 2 g of powdered Devarda's alloy and immediately connect the flask to the condenser. Distill ammonia by heating the flask as described in (*see* **5.2**). Titrate the ammonia against standard sulphuric acid. 1 ml of 0.02 N standard acid is equivalent to 0.000 28 g of nitrogen.

NOTE — To determine ammonical, nitrite and nitrate forms of nitrogen, fresh and moist sample is used. Determine the moisture content of the soil by drying a suitable aliquot in an air-oven at 105°C for 6 hours. Cool, weigh and calculate moisture content, percent by mass. Correct the result of inorganic nitrogen content by compensating for the moisture.

Bureau of Indian Standards

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Review of Indian Standards

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Amendments Issued Since Publication

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