

SRI LANKA STANDARD 645 : PART 1 : 2009
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METHODS OF TEST FOR FERTILIZERS
PART 1 : DETERMINATION OF NITROGEN CONTENT
(First Revision)

SRI LANKA STANDARDS INSTITUTION

Sri Lanka Standard
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SLS 645 : Part 1 : 2009

Gr. 9

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Sri Lanka Standard
METHODS OF TEST FOR FERTILIZERS
PART 1 : DETERMINATION OF NITROGEN CONTENT
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FOREWORD

This standard was approved by the Sectoral Committee on Chemicals and Polymer Technology and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2009-11-30.

This standard was first published in 1984. In this First Revision, test methods specified in IS 6092 :Part 2: Sec 3, IS 6092 :Part 2:Sec 4 and AOAC 978.02 : 2007 had been considered to identify the test methods for the determination of nitrogen content of fertilizers.

This Part of the standard consists of 3 Sections as follows :

- Section A Determination of nitrate nitrogen content – Nitron gravimetric method
- Section B Determination of ammoniacal nitrogen content – Titrimetric method after distillation
- Section C Determination of total nitrogen content-Titrimetric method after distillation

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 **SLS 102**.

In the preparation of this standard the assistance obtained from the following publications is gratefully acknowledged:

- IS 6092 :Part 2: 2004 (2006) Methods of sampling and test for fertilizers
 - Part 2 : Determination of Nitrogen
 - Section 3 : Nitrate nitrogen content-Nitron gravimetric method
 - Section 4 : Ammoniacal nitrogen content-Titrimetric method after distillation
- AOAC 978.02 : 2007 AOAC Official Methods of Analysis
 Nitrogen (Total) in Fertilizers

1 SCOPE

This part of the standard prescribes the methods for the determination of nitrogen in its various forms in fertilizers including fertilizer mixtures.

2 REFERENCES

- ISO 385 Laboratory glassware – Burettes
- ISO 641 Laboratory glassware – Interchangeable spherical glass joints
- ISO 648 Laboratory glassware – One- mark pipettes
- ISO 1042 Laboratory glassware – One-mark volumetric flasks
- SLS 102 Rules for rounding off numerical values
- SLS 559 Method for sampling fertilizers

3 QUALITY OF REAGENTS

Unless specified otherwise, chemicals of a recognized analytical grade and distilled water shall be employed in tests.

4 PREPARATION OF TEST SAMPLE

Reduce the test sample specified as in Clause 6 of **SLS 559 : 1982** to a quantity sufficient for analysis and grind not less than 225 g of the reduced sample without previously sieving. For fertilizer materials and moist fertilizer mixtures, grind to pass through a sieve of 1-mm aperture size. For dry mixtures that tend to segregate, grind to pass through a sieve of 350- μ m aperture size. Grind as rapidly as possible to avoid loss or gain of moisture during the operation. Mix thoroughly and store in tightly stoppered bottles.

SECTION A – DETERMINATION OF NITRATE NITROGEN CONTENT – NITRON GRAVIMETRIC METHOD

A.1 SCOPE AND FIELD OF APPLICATION

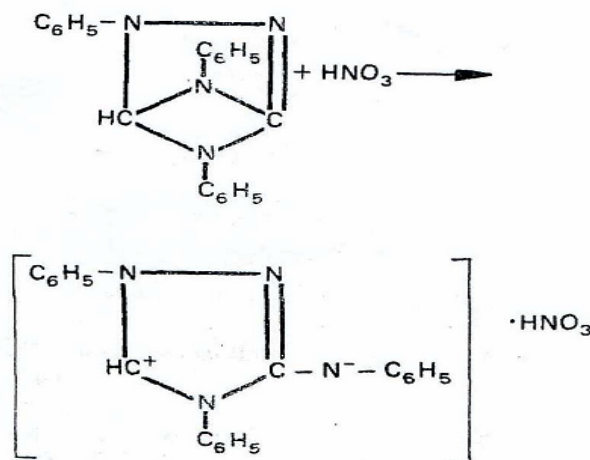
This section of the standard specifies the nitrogen gravimetric method for the determination of the nitrate nitrogen content of fertilizers. It is suitable for use as a reference method and is applicable to all fertilizers.

NOTE : *In the presence of a 9 – fold mass excess of chloride, the result will be too high by 0.4 per cent of the result obtained.*

A.2 PRINCIPLE

Precipitation of nitrate ions in acid solution as a complex with nitron reagent. Filtration of the precipitate, drying and weighing.

A.3 REACTION



A.4 REAGENTS

A.4.1 Acetic acid, 28.5 per cent (v/v) solution. Dilute 285 ml of glacial acetic acid to 1 000 ml with water.

A.4.2 Sulphuric acid, solution. Dilute one volume of sulphuric acid (d = 1.84 g/ml) with three volumes of water.

A.4.3 Nitron, 100 g/l solution. Dissolve 10 g of nitron {(3, 5, 6-triphenyl-2, 3, 5, 6-tatrazabicyclo (2.1.1) hex-1-ene)} in a mixture of 95 ml of water and 5 ml of glacial acetic acid. Filter the solution through a dry filter paper and store in a dark glass bottle.

NOTE : *New reagent should be used to avoid a high result in the blank test.*

A.5 APPARATUS

A.5.1 One-mark volumetric flask, of capacity 500 ml complying with the requirements of **ISO 1042**, Class A.

A.5.2 One-mark pipettes, of capacity in the range 5 ml to 20 ml, complying with the requirements of **ISO 648**, Class A.

A.5.3 Glass filter crucible, of porosity in the range 4 μm to 16 μm.

A.5.4 Oven, capable of being maintained at 110 ± 2 °C.

A.5.5 Mechanical flask shaker, with a rotary or reciprocating action.

A.5.6 Ice –bath, capable of being maintained at a temperature of 0 °C to 0.5 °C ; and

A.5.7 Ordinary laboratory apparatus.

A.6 PROCEDURE

A.6.1 Test portion

Weigh, to the nearest 0.001 g, about 5 g of the laboratory sample and transfer to the volumetric flask (A.5.1).

A.6.2 Preparation of test solution

A.6.2.1 Products soluble in water

Add about 400 ml of water at 20 °C to the test portion (A.6.1) and, using the mechanical flask shaker (A.5.5), shake the flask continuously for 30 min. Dilute to the mark with water and mix.

A.6.2.2 Products containing water-insoluble material likely to retain nitrate.

Add 50 ml of water and 50 ml of the acetic acid solution (A.4.1) to the test portion (A.6.1). Mix the contents of the flask and allow to stand undisturbed until any liberation of carbon dioxide has ceased. Add about 300 ml of water at 20 °C and, using the mechanical flask shaker (A.5.5), shake the flask continuously for 30 minutes. Dilute to the mark with water and mix.

A.6.3 Determination

A.6.3.1 Filter the test portion (A.6.2) through a medium retention paper into a clean, dry conical flask and discard the first 50 ml of filtrate. Using one of the one-mark pipettes (A.5.2), transfer an aliquot portion of the filtrate containing 11 mg to 23 mg (preferably 17 mg) of nitrate nitrogen, to a 250-ml beaker. Dilute with water to 100 ml.

A.6.3.2 Add 10 to 12 drops of the sulphuric acid solution (A.4.2). Check that the pH is between 1 and 1.5. heat rapidly to the boiling point and, without allowing the solution to boil (see Note 1), remove the flask from the source of heat. Check that no calcium sulphate has precipitated; if it has, dissolve it by adding a few drops of the sulphuric acid solution. Add 10 ml to 12 ml of the nitron solution (A.4.3) in one addition. Place the flask in the ice-bath (A.5.6) and stir the contents for 2 minutes. Leave the flask in the ice –bath to ensure that the temperature of the contents of the flask remains between 0 °C and 0.5 °C (see Note 2).

NOTES :

1. *Urea and urea aldehyde condensates are decomposed by boiling acid.*
2. *Temperatures lower than 0 °C will lead to high results, temperatures higher than 0.5 °C to low results.*

Weigh the filter crucible (A.5.3) to the nearest 0.001 g and cool it in the ice-bath. With the aid of suction, collect the precipitate quantitatively in the filter, using the filtrate to transfer the last traces of precipitate from the beaker to the filter crucible. Finally, wash the precipitate with 10 ml to 12 ml of water at between 0 °C and 0.5 °C. Dry the crucible and precipitate in the oven (A.5.4), maintained at 110 ± 2 °C, for 1 h. Cool in a desiccator and weigh. Repeat the operations of heating, cooling and weighing until two successive weighings do not differ by more than 0.001 g.

A.6.4 Blank test

Take 100 ml of water, or, if acetic acid solution has been used to dissolve the test portion, the same amount of acid as in the aliquot portion diluted to 10 ml with water. Proceed as described in (A.6.3.2). The mass of precipitate shall not exceed one milligram. If this limit is exceeded, repeat the blank test and the determination using new reagent. Old reagent is known to give high results in the blank test.

A.7 CALCULATION

The nitrate nitrogen content, W , expressed as nitrogen (N) as a percentage by mass, is given by the formula.

$$W = \frac{14.01}{375.3} \times \frac{m_1}{m_0} \times \frac{500}{V} \times 100 = \frac{1.866 \times m_1}{m_0 \times V}$$

where,

- V is the volume, in millilitres, of the aliquot portion of the test solution taken in A.6.3.1 ;
- m_0 is the mass, in grams, of the test portion ;
- m_1 is the mass, in grams, of the precipitated nitron nitrate ;
- 14.01 is the relative molecular mass of nitrogen ; and
- 375.3 is the relative molecular mass of the complex.

SECTION B - DETERMINATION OF AMMONIACAL NITROGEN CONTENT-TITRIMETRIC METHOD AFTER DISTILLATION

B.1 SCOPE AND FIELD OF APPLICATION

This section of the standard specifies a titrimetric method, after distillation, for the determination of the ammoniacal nitrogen content of fertilizers.

The method is applicable only in the absence of urea or its derivatives, of cyanamide and of organic nitrogenous compounds.

B.2 PRINCIPLE

Distillation of the ammonia from an alkaline solution, absorption in an excess of standard volumetric sulphuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of methyl red or screened methyl red as indicator.

B.3 REAGENTS

B.3.1 Ammonium sulphate, dried at 105 °C to constant mass.

B.3.2 Hydrochloric acid, solution. Dilute concentrated hydrochloric acid $d = 1.18$ g/ml, 1 + 1 with water.

B.3.3 Sodium hydroxide, 400 g/l solution

B.3.4 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0.20$ mol/l

B.3.5 Sulphuric acid, standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0.10$ mol/l

B.3.6 Indicator solution

B.3.6.1 *Screened methyl red indicator*, ethanolic solution

Mix 50 ml of a 2 g/l ethanolic solution of methyl red with 50 ml of 1 g/l ethanolic solution of methylene blue, or

B.3.6.2 *Methyl red indicator*, ethanolic solution

Dissolve 0.1 g of methyl red in 50 ml of 95 per cent (V/V) ethanol.

B.3.7 pH indicator paper, wide range

B.4 APPARATUS

B.4.1 Distillation apparatus

The components of the apparatus may be connected by means of rubber bungs and tubing or by the use of ground glass joints.

Ground glass joints should be held by spring clamps to ensure that they are leak –tight. Rubber bungs and tubing shall be replaced when they begin to perish or show signs of wear.

A suitable apparatus is illustrated in Figure 1 and comprises :

B.4.1.1 *Round bottomed flask*, of nominal capacity one litre

B.4.1.2 *Single-bulb splash head*, and separate open-top cylindrical dropping funnel, of capacity 100 ml.

B.4.1.3 *Allihn condenser*, seven-bulb, with an expansion bulb, of approximate capacity 100 ml, followed by a delivery tube at the outlet.

B.4.1.4 *Receiver*, (*conical flask or conical beaker*), of capacity 500 ml.

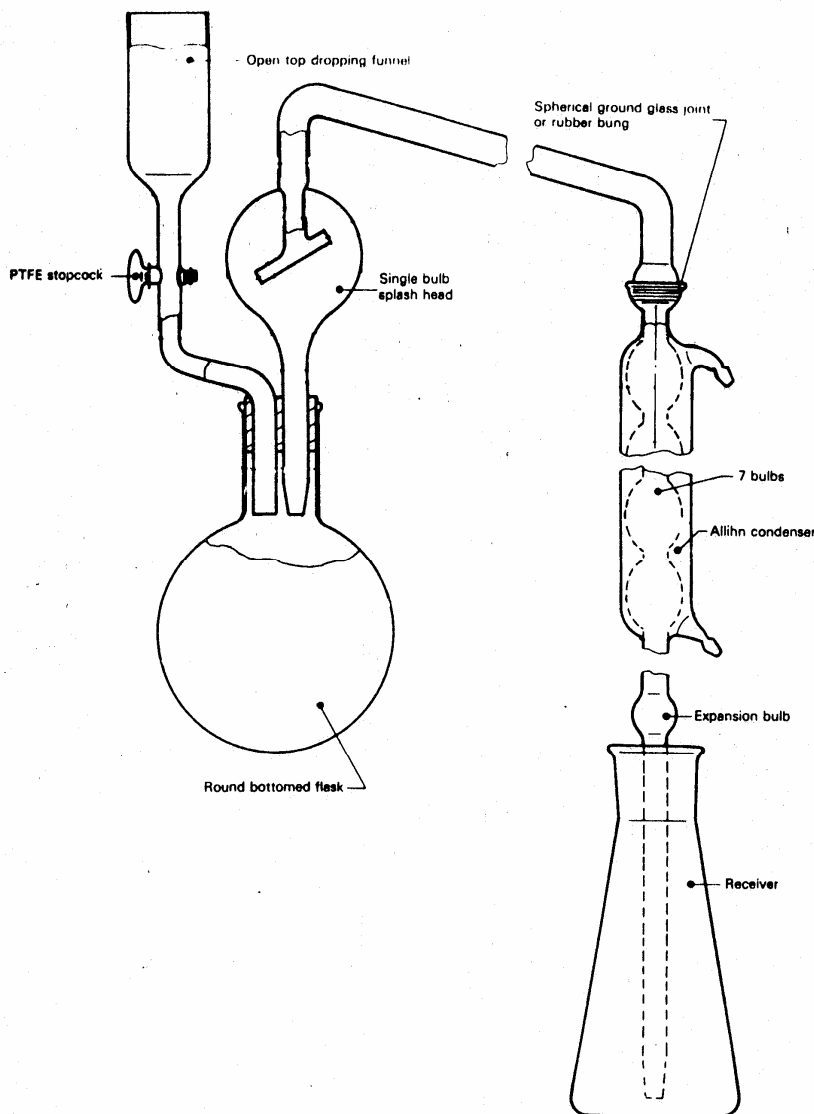


Figure 1 – Typical distillation apparatus

B.4.2 Two burettes, of capacity 50 ml, complying with the requirements to **ISO/R 385**, Class A.

B.4.3 One-mark volumetric flask, of capacity 500 ml, complying with the requirements of **ISO 1042** Class A.

B.4.4 One-mark pipette, of capacities 10, 25 50 and 100 ml, complying with the requirements of **ISO 648**, Class A.

B.4.5 Mechanical flask shaker, with a rotary or reciprocating action.

B.4.6 Anti-bumping granules, or an anti-bumping device, consisting of a 100 mm x 5-mm glass rod connected to a 25-mm length of polyethylene tubing.

B.5 PROCEDURE

B.5.1 Test portion

Weigh, to the nearest 0.001 g, about 10 g (*m*) of the analytical sample and transfer to the one-mark 500-ml volumetric flask (**B.4.3**).

B.5.2 Preparation of test solution

B.5.2.1 *Products soluble in water*

Add about 400 ml of water and shake the flask continuously for 30 minutes using the mechanical flask shaker (**B.4.5**).

B.5.2.2 *Products containing water-insoluble material likely to retain ammonia*

Add 50 ml of water and 20 ml of the hydrochloric acid solution (**B.3.2**) to the test portion (**B.5.1**). Mix the contents of the flask and allow to stand undisturbed until any liberation of carbon dioxide has ceased. Add about 400 ml of water and, shake the flask continuously for 30 minutes using the mechanical flask shaker.

NOTE : *Complete dissolution of the test portion is not necessary. The procedure described extract all the ammoniacal nitrogen.*

B.5.3 Determination

Dilute the contents of the flask to the mark with water, mix well and filter through a dry medium filter paper into a dry beaker. Discard the first 50 ml of filtrate and then transfer an aliquot portion (*v*) of the filtrate, by means of a pipette (**B.4.4**) into the flask (**B.4.1.1**). The aliquot portion shall contain between 75 mg and 100 mg of ammoniacal nitrogen.

Dilute the contents of the flask to about 200 ml with water and add a few anti-bumping granules or the anti-bumping device (**B.4.6**) to prevent bumping during the distillation. Add a few drops of the indicator solution (**B.3.6**). Assemble the apparatus as shown in Figure 1.

Measure 50.0 ml of the standard volumetric sulphuric acid solution (**B.3.5**) with a burette (**B.4.2**) into the receiver (**B.4.1.4**) and add 4 or 5 drops of the indicator solution (**B.3.6**). Place the receiver so that the end of the delivery tube (see **B.4.1.3**) is below the surface of as the acid, adding water to the flask if necessary.

Pour 15 ml of the sodium hydroxide solution (**B.3.3**) into the dropping funnel. If 20 ml of the hydrochloric acid solution (**B.3.2**) has been added to dissolve the test portion (see **B.5.2**), use 25 ml of the sodium hydroxide solution (**B.3.3**).

Add the sodium hydroxide solution (**B.3.3**). When nearly all the sodium hydroxide solution has been added, close the stop-cock, leaving about 2 ml in the dropping funnel.

Bring the contents of the flask to the boil, increasing the rate of heating progressively until the contents of the flask are boiling briskly. The contents of the flask shall remain alkaline during the distillation period. When at least 150 ml of distillate has collected, partially withdraw the receiver so that the delivery tube rests on its rim. Test the subsequent distillate with the pH indicator paper (**B.3.7**) to ensure that all the ammonia has been completely distilled.

Detach the splash head from the condenser, remove the source of heat and wash the condenser and expansion bulb through with water, collecting the washings in the receiver. The outside of the delivery tube shall also be rinsed into the flask.

Back-titrate the excess of acid with the standard volumetric sodium hydroxide solution (**B.3.4**) to the neutral colour of the indicator.

B.5.4 Blank test

Carry out a blank test at the same time as the determination, using the same reagents but omitting the test solution.

The result of the blank test shall not exceed 0.25 ml of 0.10 mol/l sulphuric acid solution.

B.5.5 Check test

Carry out a periodic check on the efficiency of the apparatus and the accuracy of the method using an aliquot portion of a freshly prepared solution of the ammonium sulphate (**B.3.1**) containing 100 mg of nitrogen, the check shall be made using the same conditions as for the sample and blank determinations and with the same indicator.

B.6 CALCULATION

The ammoniacal nitrogen content, expressed as nitrogen (N) as a percentage by mass;

$$\frac{(2 M_1 V_1 - M_2 V_2) - (2 M_1 V_3 - M_2 V_4) \times 1.4007 \times 500}{mV} = \frac{M_2 (V_4 - V_2) \times 700.35}{mV}$$

where,

V_1 is the volume used, in millilitres, of the standard volumetric sulphuric acid solution (**B.3.5**) for the determination (50.0 ml) ;

- V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (B.3.4) used for the determination ;
- V_3 is the volume, in millilitres, of the standard volumetric sulphuric acid solution (B.3.5) used for the blank test (50.0 ml) ;
- V_4 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (B.3.4) used for the blank test ;
- m is the mass, in gram, of sample in the aliquot portion taken for the determination.
- M_1 is the molarity of the sulphuric acid solution (B.3.5);
- M_2 is the molarity of the sodium hydroxide solution (B.3.4) ; and
- V is the volume, in millilitres, of the aliquot portion of filtrate.

SECTION C - DETERMINATION OF TOTAL NITROGEN CONTENT - TITRIMETRIC METHOD AFTER DISTILLATION

C.1 SCOPE AND FIELD OF APPLICATION

This section of the standard specifies a titrimetric method, after distillation, for the determination of the total nitrogen content of fertilizers in all forms, including those which have to be digested.

The method is not recommended for fertilizers containing large amounts (above 7 per cent) of organic matter.

C.2 PRINCIPLE

Reduction of nitrate to ammonia by chromium powder in acid medium. Conversion of organic and urea nitrogen into ammonium sulphate by digestion with concentrated sulphuric acid in the presence of a catalyst. Distillation of the ammonia from an alkaline solution, absorption in an excess of standard volumetric sulphuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of methyl red or screened methyl red as indicator.

C.3 REAGENTS

C.3.1 Chromium metal powder

C.3.2 Aluminium oxide, fused. Pumice is suitable.

C.3.3 Anti – foaming agent, for example paraffin wax of melting point not lower than 100 °C.

C.3.4 Ammonium nitrate, *dried at* 100 °C to constant mass.

C.3.5 Potassium Sulphate or anhydrous sodium sulphate

C.3.6 Anhydrous copper sulphate or copper (II) sulphate pentahydrate (CuSO₄.5H₂O)

C.3.7 Sulphuric acid, concentrated, $d = 1.85$ g/ml.

C.3.8 Hydrochloric acid, concentrated, $d = 1.18$ g/ml.

C.3.9 Sodium hydroxide, $d = 400$ g/l solution.

C.3.10 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0.10$ mol/l

C.3.11 Sulphuric acid, standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0.25$ mol/l.

C.3.12 Sulphuric acid, standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0.10$ mol/l.

C.3.13 Sulphuric acid, standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0.05$ mol/l.

C.3.14 Indicator solution

Use either the screened methyl red solution (**C.3.12.1**) or the methyl red solution (**C.3.12.2**)

C.3.14.1 *Screened methyl red*, ethanolic indicator, solution

Mix 50 ml of a 2 g/l ethanolic solution of methyl red with 50 ml of 1g/l ethanolic solution of methylene blue.

C.3.14.2 *Methyl red*, ethanolic indicator, solution

Dissolve 0.1 g of methyl red in 50 ml of 95 per cent (V/V) ethanol.

C.3.15 pH indicator paper, wide range

C.3.16 *Phenolphthalein*, indicator solution

Dissolve 1 g phenolphthalein in 100 ml of ethanol and add 100 ml of water while stirring. Filter if a precipitation forms.

C.4 APPARATUS

C.4.1 Usual laboratory equipment, and digestion apparatus, comprising an 800-ml Kjeldahl flask and a pear-shaped hollow glass stopper.

C.4.2 Distillation apparatus (see **B.4.1** of Section **B**, Figure 1)

C.4.3 Anti-bumping granules, or an anti-bumping device, consisting of a 100-mm x 5-mm glass rod connected to a 25-mm length of polyethylene tubing.

C.4.4 Two burette, of capacity 50 ml, complying with the requirements to **ISO 385/1**, Class A.

C.4.5 Glass beads, of diameter 2 mm to 3 mm.

C.5 PROCEDURE

C.5.1 Test portion

Weigh, to the nearest 0.001 g, between 0.5 g and 2.0 g of the test sample, containing not more than 60 mg of nitrate nitrogen and 235 mg of total nitrogen.

C.5.2 Determination

C.5.2.1 Reduction (this step is not required if nitrate nitrogen is known to be absent).

Transfer the test portion (**C.5.1**) to the Kjeldhal flask of the digestion apparatus (**C.4.1**) and add sufficient water to make up the total volume to 35 ml. Allow the flask to stand for 10 minutes with occasional gentle swirling to ensure dissolution of all nitrate salts.

Add 1.2 g of the chromium powder (**C.3.1**) and 7 ml of the hydrochloric acid solution (**C.3.8**). Allow the flask to stand for at least 5 minutes but not more than 10 minutes, at ambient temperature.

Place the flask on a heating device in a fume cupboard with the heat input regulated to pass a 7-minute to 7.5-minute boil test (see Note) and heat the flask for 4.5 minutes. Remove from the heat and allow to cool.

C.5.2.2 Hydrolysis (this step can be used instead of the digestion (**C.5.2.3**) if it is known that the only forms of organic nitrogen present are urea and Cyanamide forms).

Stand the flask in a fume cupboard and add about 1 g of the fused aluminum oxide (**C.3.2**). Carefully add 25 ml of the sulphuric acid (**C.3.7**) to the flask. Insert the pear – shaped hollow glass stopper into the neck of the flask and place on a heating device and initially, heat until gently boiling. Then adjust the heat input to give a 7-minute to 7.5 minute boil test (see Note **1**).

Continue to heat the flask and contents until dense white fumes of sulphuric acid have been evolving for at least 15 minutes. Allow the flask to cool to room temperature.

C.5.2.3 Digestion (this step is necessary only if organic forms of nitrogen other than urea or Cyanamide forms are present (see **C.5.2.2**) or in the case of fertilizers of unknown composition).

Place the flask in a fume cupboard and add 15 g powdered K_2SO_4 or 12 g anhydrous Na_2SO_4 (**C.3.5**), 0.4 g anhydrous $CuSO_4$ or 0.6 g $CuSO_4 \cdot 5H_2O$ (**C.3.6**) and about 1.5 g of the fused aluminum oxide (**C.3.2**). Carefully add 20 ml of the sulphuric acid (**C.3.7**) to the flask and add 0.5 g of the anti-foaming agent (**C.3.3**) to reduce foaming (see Note 2). Insert the pear-shaped hollow glass stopper into the neck of the flask and place on a heating device with the heat input adjusted to give a 7-minute to 7.5-minute boil test (see Note 1).

NOTES :

1 The heat input required to bring 350 ml of water at $25^{\circ}C$ to a "rolling" boil in 7 minutes to 7.5 minutes.

2 If organic matter other than urea exceeds 1.0 g, add additional 1.0 ml sulphuric acid for each 0.1 g fat or -2 g other organic matter in excess of 1.0 g.

If considerable foaming occurs, reduce the heat input until this phase is over. Continue to heat the flask and contents and increase the heat input progressively until dense white fumes are clear from the bulb of the flask. Gently swirl the flask and continue digestion for a further 60 minutes or until the solution is clear, whichever is the longer. Allow the flask to cool to room temperature.

C.5.2.4 Distillation

Place the test portion or retain the solution in the Kjeldahl flask. If distillation from the round bottom flask is preferred, transfer the hydrolyzed (**C.5.2.2**) or digested (**C.5.2.3**) solution quantitatively into a round bottom flask. (Alternatively transfer the hydrolyzed or digested solution quantitatively into a 500-ml volumetric flask and dilute up to the mark with water. Take V ml portions of this solution.)

Add a few anti-bumping granules or glass beads and assemble the apparatus as shown in Figure 1 (See Section B).

Measure into the receiver (**C.4.2**), according to the expected mass of nitrogen in the test portion, appropriate volume shown in Table 1 of one of the sulphuric acid solution (**C.3.11**, **C.3.12** or **C.3.13**).

TABLE 1 - Appropriate volume of sulphuric acid solution

Expected mass of nitrogen in the test portion mg (1)	Concentration of sulphuric acid solution mol/l (2)	Volume of sulphuric acid solution ml (3)
0 to 30	0.05	25.0
30 to 50		40.0
50 to 65	(solution 3.14)	50.0
65 to 80	0.10	35.0
80 to 100		40.0
100 to 125	(solution 3.11)	50.0
125 to 170	0.25	25.0
170 to 200		30.0
200 to 235	(solution 3.10)	35.0

Add 4 or 5 drops of the indicator solution (**C.3.12**) and place the receiver so that the end of the delivery tube is below the surface of the acid, adding water to the receiver if necessary.

Pour sodium hydroxide solution (**C.3.9**), through the dropping funnel until the solution in the distillation flask is neutralized to phenolphthalein indicator (**C.3.16**). Close the stop-cock, leaving the 2 ml remaining in the dropping funnel. Bring the contents of the flask to the boil, increase the rate of heating progressively until, finally, the contents of the flask are boiling briskly. The contents of the flask shall remain alkaline during the distillation period.

When at least 150 ml of distillate have been collected, partially withdraw the receiver so that the delivery tube rests on the rim of the receiver. Test the subsequent distillate with the pH indicator paper (**C.3.15**) to ensure that all the ammonia is completely distilled.

Detach the splash head from the condenser, remove the source of heat, and wash the insides of the condenser and expansion bulb with water. Collect the rinsings in the receiver. Also rinse the outside of the delivery tube and collect the rinsings in the receiver.

C.5.2.5 Titration

Back-titrate the excess of acid with the standard volumetric sodium hydroxide solution (**C.3.10**) to the neutral colour of the indicator.

C.5.3 Blank test

Carry out a blank test at the same time as the determination, using the same reagents but omitting the test portion, and using the 0.05 mol/l standard volumetric sulphuric acid solution (**C.3.13**). The result of the blank test shall not exceed 1.0 mol/l. If the result is greater than 1.0 mol/l, check the reagents, especially the chromium powder (**C.3.1**).

C.5.4 Check test

Carry out a periodic check on the efficiency of the apparatus and the accuracy of the method using an aliquot portion of the solution of freshly prepared ammonium nitrate (C.3.4) containing 100 mg of nitrogen. The check shall be made using the same conditions as for the determinations and blank test and using the same indicator.

C.6 CALCULATION

C.6.1 The total nitrogen content, expressed as nitrogen (N) as a percentage by mass, is given by the following.

$$\frac{((2 M_1 V_1 - M_2 V_2) - (2 M_3 V_3 - M_2 V_4)) \times 1.4007}{m} = \frac{M_2 (V_4 - V_2) \times 1.4007}{m}$$

where,

V_1 is the volume, in millilitres, of the sulphuric acid solution (C.3.11, C.3.12 or C.3.13), (as appropriate) used for the determination ;

V_2 is the volume, in millilitres, of the sodium hydroxide solution (C.3.10) used for the determination ;

V_3 is the volume, in millilitres, of the sulphuric acid solution (C.3.13) used for the blank test ;

V_4 is the volume, in millilitres, of the sodium hydroxide solution (C.3.10) used for the blank test ;

m is the mass, in grams, of the test portion (C.5.1) ;

M_1 is the molarity of the sulphuric acid solution (C.3.11, C.3.12 or C.3.13), (as appropriate) used for the determination ;

M_2 is the molarity of the sodium hydroxide solution (C.3.10) ; and

M_3 is the molarity of the sulphuric acid solution (C.3.13) used for the blank test.

C.6.2 If a portion (V in ml) of the digested or hydrolyzed solution transferred to 500 ml volumetric flask is taken for distillation, The total nitrogen content, expressed as nitrogen (N) as a percentage by mass, is given by the following.

$$\frac{((2 M_1 V_1 - M_2 V_2) - (2 M_3 V_3 - M_2 V_4)) \times 1.4007 \times 500}{mV} = \frac{M_2 (V_4 - V_2) \times 700.35}{mV}$$

SLS CERTIFICATION MARK

The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI which includes surveillance inspection of the factory, testing of both factory and market samples.

Further particulars of the terms and conditions of the permit may be obtained from the Sri Lanka Standards Institution, 17, Victoria Place, Elvitigala Mawatha, Colombo 08.



SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the National Standards Organization of Sri Lanka established under the Sri Lanka Standards Institution Act No. 6 of 1984 which repealed and replaced the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Science & Technology.

The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and other services offered for Industry and Business Sector. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The development and formulation of National Standards is carried out by Technical Experts and representatives of other interest groups, assisted by the permanent officers of the Institution. These Technical Committees are appointed under the purview of the Sectoral Committees which in turn are appointed by the Council. The Sectoral Committees give the final Technical approval for the Draft National Standards prior to the approval by the Council of the SLSI.

All members of the Technical and Sectoral Committees render their services in an honorary capacity. In this process the Institution endeavours to ensure adequate representation of all view points.

In the International field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.