Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

"जानने का अधिकार, जीने का अधिकार"
Mazdoor Kisan Shakti Sangathan
“The Right to Information, The Right to Live”

"पुराने को छोड कर नये के तरफ"
Jawaharlal Nehru
“Step Out From the Old to the New”

Indian Standard

METHODS OF SAMPLING AND TEST FOR FERTILIZERS

PART 2 DETERMINATION OF NITROGEN

Section 4 Ammoniacal Nitrogen Content — Titrimetric Method After Distillation
NATIONAL FOREWORD

This Indian Standard (Part 2/Sec 4) which is identical with ISO 5314 : 1981 'Fertilizers — Determination of ammoniacal nitrogen content — Titrimetric method after distillation' issued by the International Organization for Standardization was adopted by the Bureau of Indian Standards on the recommendations of the Fertilizers Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO Standard has been proposed to be approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (,) has been used as a decimal marker, while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their places, are listed below along with their degree of equivalence for the editions indicated. However, that International Standard cross-referred in this adopted ISO Standard, which has subsequently been revised, position in respect of that latest ISO Standard has been given:

<table>
<thead>
<tr>
<th>International Standard</th>
<th>Corresponding Indian Standard</th>
<th>Degree of Equivalence</th>
</tr>
</thead>
<tbody>
<tr>
<td>ISO/R 385 Burettes</td>
<td>Nil</td>
<td></td>
</tr>
<tr>
<td>ISO 641 : 1975 Laboratory glassware — Interchangeable spherical ground joints</td>
<td>IS 14868 : 2000 Laboratory interchangeable spherical ground glass joints — Specification</td>
<td>Technically equivalent</td>
</tr>
<tr>
<td>ISO 1042 : 1983 Laboratory glassware — One-mark volumetric flasks</td>
<td>IS 915 : 1975 One-mark volumetric flasks (first revision)</td>
<td>do</td>
</tr>
</tbody>
</table>

The Technical Committee responsible for the preparation of this standard has reviewed the provision of the ISO 648 and ISO 1042 and decided that they are acceptable for use in conjunction with this standard.
Indian Standard

METHODS OF SAMPLING AND TEST
FOR FERTILIZERS

PART 2 DETERMINATION OF NITROGEN

Section 4 Ammoniacal Nitrogen Content — Titrimetric Method After Distillation

1 Scope and field of application

This International 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.

2 References

ISO/R 385, "Burettes".

ISO 641, "Laboratory glassware — Interchangeable spherical ground joints".

ISO 648, "Laboratory glassware — One-mark pipettes".

ISO 1042, "Laboratory glassware — One-mark volumetric flasks".

3 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.

4 Reagents

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

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

4.2 Hydrochloric acid solution.

Dilute concentrated hydrochloric acid, $q = 1,18$ g/ml, 1 + 1
with water.

4.3 Sodium hydroxide, approximately 400 g/l solution.

4.4 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 0,20$ mol/l.\(^1\)

4.5 Sulphuric acid, standard volumetric solution, $c(\text{H}_2\text{SO}_4) = 0,10$ mol/l.\(^1\)

4.6 Indicator solution.

4.6.1 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

4.6.2 Methyl red indicator, ethanolic solution.

Dissolve 0,1 g of methyl red in 50 ml of 95 % (V/V) ethanol.

4.7 pH indicator paper, wide range.

5 Apparatus

5.1 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 the figure and comprises:

5.1.1 Round bottomed flask, of nominal capacity 1 litre.

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

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

5.1.4 Receiver (conical flask or conical beaker), of capacity 500 ml.

\(^{1}\) Hitherto expressed as "0,20 N standard volumetric solution".
5.2 Two burettes, of capacity 50 ml, complying with the requirements of ISO/R 365, class A.

5.3 One-mark volumetric flask, of capacity 500 ml, complying with the requirements of ISO 1042, class A.

5.4 One-mark pipettes, of capacities 10 – 25 – 50 and 100 ml, complying with the requirements of ISO 646, class A.

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

5.6 Anti-bumping granules or an anti-bumping device consisting of a 100 mm × 5 mm glass rod connected to a 25 mm length of polyethylene tubing.

6 Procedure

6.1 Test portion

Weigh, to the nearest 0,001 g, about 10 g of the analytical sample and transfer to the one-mark volumetric flask (5.3).

NOTE – Procedures for the preparation of analytical samples will form the subject of a future International Standard.

6.2 Preparation of test solution

6.2.1 Products soluble in water

Add about 400 ml of water at 20 °C and shake the flask continuously for 30 min using the mechanical flask shaker (5.5).

6.2.2 Products containing water-insoluble material likely to retain ammonia

Add 50 ml of water and 20 ml of the hydrochloric acid solution (4.2) to the test portion (6.1). 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 at 20 °C and shake the flask continuously for 30 min using the mechanical flask shaker.

NOTE – Complete dissolution of the test portion is not necessary. The procedure described extracts all the ammoniacal nitrogen.

6.3 Determination

Dilute the contents of the flask to the mark with water, mix well and filter through a dry medium rate and retention low ash grade of filter paper into a dry beaker. Discard the first 50 ml of filtrate and then transfer an aliquot portion of the filtrate, by means of a pipette (5.4), into the flask (5.1.1). The aliquot portion shall contain preferably between 75 and 100 mg of ammoniacal nitrogen but, in any case, shall be in the range 25 to 100 mg.

Dilute the contents of the flask to about 200 ml with water and add a few anti-bumping granules or the anti-bumping device (5.6) to prevent bumping during the distillation. Add a few drops of the indicator solution (4.6). Assemble the apparatus as shown in the figure.

Measure 50,0 ml of the standard volumetric sulphuric acid solution (4.5) with a burette (5.2) into the receiver (5.1.4) and add 4 or 5 drops of the indicator solution (4.6). Place the receiver so that the end of the delivery tube (see 5.1.3) is below the surface of the acid, adding water to the flask if necessary.

Pour 15 ml of the sodium hydroxide solution (4.3) into the dropping funnel. If 20 ml of the hydrochloric acid solution (4.2) has been added to dissolve the test portion (see 6.2), use 25 ml of the sodium hydroxide solution (4.3).

Cool the contents of the distillation flask to room temperature and add the sodium hydroxide solution (4.3). When nearly all the sodium hydroxide solution has been added, close the stopcock, 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 (4.7) to ensure that all the ammonia has completely distilled. Remove the source of heat.

Detach the splash head from the condenser 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 (4.4) to the neutral colour of the indicator.

6.4 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 should not exceed 0,25 ml of 0,10 mol/l sulphuric acid solution.

6.5 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 (4.1) 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.

7 Expression of results

7.1 Calculation

The ammonical nitrogen content, expressed as nitrogen (N) as a percentage by mass, is given by the formula:

\[
\frac{(V_1 - V_2) - (V_3 - V_4)) \times 0.002801 \times 100}{m}
\]

or

\[
\frac{V_4 - V_2}{m} \times 0.2801
\]

where

- \(V_1\) is the volume of the standard sulphuric acid solution
- \(V_2\) is the volume of the acid added
- \(V_3\) is the volume of the acid remaining
- \(V_4\) is the volume of the standard sodium hydroxide solution
- \(m\) is the mass of the sample

The result of the check test should be within 10% of the result of the determination.
where

\[ V_1 \] is the volume, in millilitres, of the standard volumetric sulphuric acid solution (4.5) used for the determination (50.0 ml);

\[ V_2 \] is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (4.4) used for the determination;

\[ V_3 \] is the volume, in millilitres, of the standard volumetric sulphuric acid solution (4.5) used for the blank test (50.0 ml);

\[ V_4 \] is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (4.4) used for the blank test;

\[ m \] is the mass, in grams, of sample in the aliquot portion taken for the determination.

NOTE — If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, appropriate corrections should be made.

### 7.2 Precision

The statistical information given below was obtained from analysis of 22 sets of results (two operations in each case, each operator carrying out two determinations) from laboratories in seven different countries.

#### 7.2.1 Repeatability

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material should not, in the long run, in the normal and correct operation of the test method, exceed the value of 0.03 % (m/m) at a confidence level of 95 %.

#### 7.2.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material should not, in the long run, in the normal and correct operation of the test method, exceed the value of 0.08 % (m/m) at a confidence level of 95 %.

### 8 Test report

The test report shall include the following particulars:

a) the reference of the method used, i.e. ISO 5314;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.
Figure — Typical distillation apparatus
Bureau of Indian Standards

BIS is a statutory institution established under the Bureau of Indian Standards Act, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publication), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. PCD 20 (2005).

Amendments Issued Since Publication

<table>
<thead>
<tr>
<th>Amend No.</th>
<th>Date of Issue</th>
<th>Text Affected</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

BUREAU OF INDIAN STANDARDS

Headquarters:
Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402

Regional Offices:

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002
{ 2323 7617
2323 3841

Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi
KOLKATA 700054
{ 2337 8499, 2337 8561
2337 8626, 2337 9120

Northern : SCO 335-336, Sector 34-A, CHANDIGARH 160022
{ 60 3843
60 9285

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113
{ 2254 1216, 2254 1442
2254 2519, 2254 2315

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
MUMBAI 400093
{ 2832 9295, 2832 7858
2832 7891, 2832 7692

Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COMBATORE. FARIDABAD.
GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR.
NALAGARH. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.

Printed at Simco Printing Press, Delhi