International Standard 5314

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Fertilizers — Determination of ammoniacal nitrogen content — Titrimetric method after distillation

Engrais — Dosage de l'azote ammoniacal — Méthode titrimétrique après distillation

First edition — 1981-05-15

UDC 631.8 : 543.24 : 546.171.1 **Ref. No. ISO 5314-1981 (E)**

Descriptors : fertilizers, chemical analysis, determination of content, nitrogen, ammoniacal nitrogen, volumetric analysis.

Price based on 4 pages

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5314 was developed by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*, and was circulated to the member bodies in March 1978.

It has been approved by the member bodies of the following countries:

Australia	Ireland	Portugal
Brazil	Israel	Romania
Canada	Italy	South Africa, Rep. of
Czechoslovakia	Kenya	Spain
Egypt, Arab Rep. of	Mexico	Thailand
France	Netherlands	Turkey
Germany, F. R.	New Zealand	United Kingdom
Hungary	Norway	USSR
India	Philippines	Venezuela
Iran	Poland	Yugoslavia

No member body expressed disapproval of the document.

Printed in Switzerland

Fertilizers — Determination of 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, $\varrho \approx 1{,}18$ g/ml, 1 + 1 with water.

4.3 Sodium hydroxide, approximately 400 g/l solution.

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

4.5 Sulphuric acid, standard volumetric solution, $c(H_2SO_4) = 0{,}10$ 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 385, 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 648, 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 × ϕ 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 ammoniacal nitrogen content, expressed as nitrogen (N) as a percentage by mass, is given by the formula

$$\frac{[(V_1 - V_2) - (V_3 - V_4)] \times 0{,}002\,801 \times 100}{m}$$

$$= \frac{V_4 - V_2}{m} \times 0{,}280\,1$$

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.

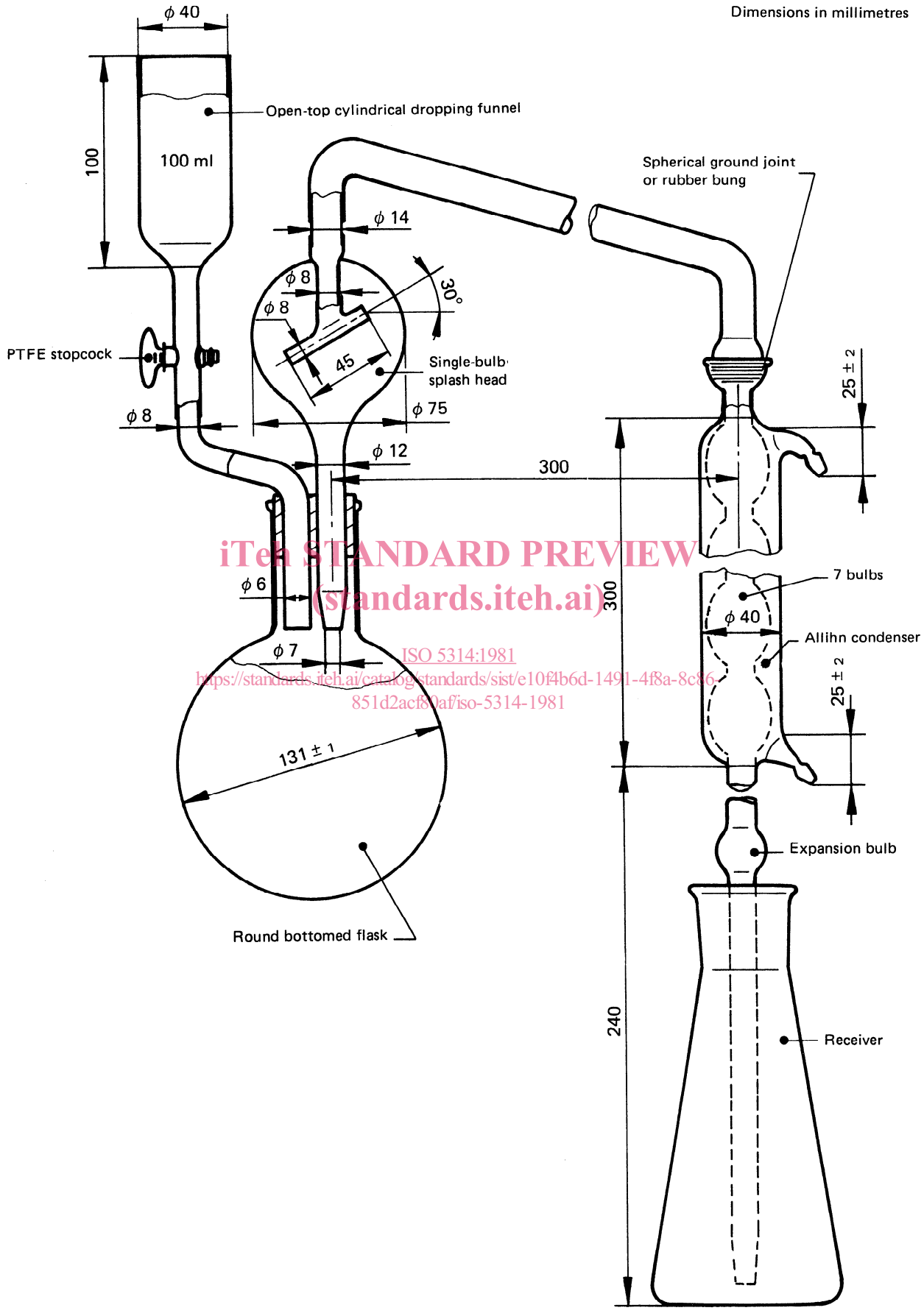

Figure — Typical distillation apparatus

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