International Standard 7408

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Fertilizers — Determination of ammoniacal nitrogen content in the presence of other substances which release ammonia when treated with sodium hydroxide — Titrimetric method

Engrais — Dosage de l'azote ammoniacal en présence d'autres substances libérant de l'ammoniac sous l'effet d'hydroxyde de sodium — Méthode titrimétrique

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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 7408 was developed by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners,* and was circulated to the member bodies in July 1981.

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

Austria	Israel	Poland
Brazil	Italy	Portugal
China	Kenya	Romania
Czechoslovakia	Korea, Rep. of	South Africa, Rep. of
Egypt, Arab Rep. of	Mexico	Sri Lanka
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Germany, F.R.	New Zealand	USA
Hungary	Norway	USSR

The member body of the following country expressed disapproval of the document on technical grounds:

India

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INTERNATIONAL STANDARD ISO 7408-1983 (E)

Fertilizers — Determination of ammoniacal nitrogen content in the presence of other substances which release ammonia when treated with sodium hydroxide — Titrimetric method

1 Scope and field of application

This International Standard specifies a method for the determination of the ammoniacal nitrogen content of fertilizers containing other substances, such as urea or urea-aldehyde condensates, which release ammonia in the presence of sodium hydroxide.

2 Principle

Entrainment, by means of a strong current of air, of the ammonia from a moderately alkaline test mixture at ambient temperature into standard volumetric sulfuric acid solution. Back-titration of the excess sulfuric acid with standard volumetric sodium hydroxide solution.

3 Reagents

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

3.1 Nonyl alcohol (nonanol).

3.2 Potassium carbonate, saturated solution at room temperature.

3.3 Sulfuric acid, approximately 590 g/l solution.

3.4 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0{,}25$ mol/l.[1)]

3.5 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0{,}05$ mol/l.[2)]

3.6 Sodium hydroxide, approximately 120 g/l solution.

3.7 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0{,}50$ mol/l.[1)]

3.8 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0{,}10$ mol/l.[2)]

1) Hitherto expressed as "0,50 N standard volumetric solution".

2) Hitherto expressed as "0,10 N standard volumetric solution".

3.9 Mixed indicator solution: screened ethanolic methyl red solution.

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

The colour of this indicator changes from lilac in acid medium, via grey at pH 5,4, to green in alkaline medium.

3.10 Ammonium sulfate, dried to constant mass at 100 °C.

3.11 Urea.

4 Apparatus

Ordinary laboratory apparatus and

4.1 Entrainment 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 should be replaced when they begin to perish or show signs of wear.

A suitable apparatus is illustrated in figures 1 and 2 and comprises the following components:

4.1.1 Double-necked bottle, of capacity 350 to 400 ml.

4.1.2 Inlet tube, fitted with a mushroom-shaped air distributor.

This distributor has an external diameter of 20 mm and has six openings in the rim, each 1 ± 0,2 mm in diameter.

NOTE — The shape of the distributor and the number of holes and their diameters are important in ensuring adequate distribution of air bubbles despite the strong flow of air.

4.1.3 Outlet tube, with a single bulb splash head.

If ground glass joints are used, the inlet tube (4.1.2) and the outlet tube with its splash head (4.1.3) form one rigid glass assembly. If a rubber bung is used, the inlet and outlet tubes shall be separate and the inlet tube shall be straight and shall be of uniform diameter at the top end.

4.1.4 Inlet tube, similar to that described in 4.1.2.

4.1.5 Conical flask, of capacity 500 ml.

4.1.6 Three-necked flask (or conical flask of capacity 500 ml), fitted with an inlet tube, an outlet tube and a stopcock (only required when suction is used instead of compressed air).

4.2 Washing bottles.

4.2.1 Two washing bottles, of capacity 500 ml, with the usual inlet and outlet tubes of diameter 6 mm without bulb splash head.

4.2.2 Washing bottle, similar to those described in 4.2.1, but with a shorter inlet tube.

4.3 Four pipettes, of capacities 50 — 25 — 20 and 10 ml.

4.4 Three burettes, of capacities 50 — 25 and 10 ml.

4.5 Compressed air source, fitted with a regulator capable of controlling the air flow rate at about 3 000 ml/min, or

4.6 Suction device, for example a water jet pump, capable of maintaining an air flow rate through the apparatus at about 3 000 ml/min.

4.7 Flowmeter, to measure the flow of air.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the laboratory sample.[1)]

Transfer the test portion to the double-necked flask (4.1.1).

5.2 Assembly of the apparatus

Assemble the entrainment apparatus (4.1).

Connect in series with the double-necked bottle (4.1.1), in the following order (see figure 1):

a) one of the bottles (4.2.1) in which has been placed 250 ml of the sodium hydroxide solution (3.6);

b) the other bottle (4.2.1) in which has been placed 250 ml of the sulfuric acid solution (3.3);

c) the empty bottle (4.2.2).

Connect the flowmeter (4.7) to the empty bottle (4.2.2).

If compressed air is used, connect the compressed air source (4.5) to the flowmeter (4.7). If suction is used, connect the suction device (4.6), via the flask (4.1.6) to the conical flask (4.1.5), as shown in figure 2.

NOTE — The flask (4.1.6) prevents water entering the apparatus in the event of pressure fluctuations.

Ensure that all the connections are airtight.

5.3 Determination

5.3.1 Fertilizers of known composition

Depending on the expected ammoniacal nitrogen content of the sample, measure into the flask (4.1.5), by means of a pipette, the appropriate volume of the appropriate sulfuric acid solution (3.4 or 3.5) specified in the table. Add water until the level of the liquid is about 50 mm above the orifice of the inlet tube (4.1.4) and then add 5 drops of the mixed indicator solution (3.9).

Add, through the side-neck of the double-necked bottle (4.1.1), 50 ml of water, a few drops of the nonyl alcohol (3.1) and render the solution alkaline by adding 50 ml of the saturated potassium carbonate solution (3.2).

NOTE — The addition of nonyl alcohol prevents foaming when the air is passed.

Immediately close the side-neck of the bottle and, using compression or suction as appropriate, start the air flow. Adjust the air flow rate to about 3 000 ml/min and pass air through the apparatus for 2 h.

Lower the conical flask so that its lip supports the end of the inlet tube (4.1.4). Rinse the outside of the tube with water, collecting the rinsings in the flask, and stop the air flow.

Titrate the residual acid in the conical flask with the appropriate sodium hydroxide solution (see the table) to the neutral colour (grey) of the mixed indicator solution (3.9).

Table — Reagents required for titration

Expected ammoniacal nitrogen content, % (*m*/*m*)	Sulfuric acid solution added to the conical flask (4.1.5)		Sodium hydroxide solution used for the titration
	Identity	Volume, ml	Identity (concentration)
> 3,5	3.4	40,0	3.7 (0,5 mol/l)
⩾ 1,0 and < 3,5	3.5	40,0	3.8 (0,1 mol/l)
< 1,0	3.5	10,0	3.8 (0,1 mol/l)

1) The preparation of samples of fertilizers will form the subject of a future International Standard.

5.3.2 Fertilizers of unknown composition

Carry out the procedure specified in 5.3.1 for samples having ammoniacal nitrogen contents greater than 3,5 % (m/m) and then continue as follows.

After removing the conical flask (4.1.5), substitute another similar flask containing 10,0 ml of the standard volumetric sulfuric acid solution (3.4), 5 drops of the mixed indicator solution (3.9) and sufficient water for the level of liquid to be 50 mm above the orifice of the inlet tube. Restart the air flow, adjust it to about 3 000 ml/min and pass the air for 1 h. Lower the conical flask so that its lip supports the end of the inlet tube (4.1.4). Rinse the outside of the tube with water, collecting the rinsings in the flask and stop the air flow.

Titrate the residual acid in the conical flask with the sodium hydroxide solution (3.7) to the neutral colour (grey) of the mixed indicator solution (3.9). Combine the results of each determination.

5.4 Blank test

At the same time as the determination, carry out a blank test using the same reagents but omitting the test portion and using the sulfuric acid solution (3.5) and the sodium hydroxide solution (3.8).

5.5 Check test

Periodically, carry out a check test on the efficiency of the apparatus and the accuracy of the test method, as follows.

Weigh 4,716 g of the ammonium sulfate (3.10) and 25 g of the urea (3.11) into a 250 ml one-mark volumetric flask. Dissolve in 200 ml of water and dilute to the mark with water.

Use as the test portion 25,0 ml of this solution, equivalent to 100 mg of ammoniacal nitrogen, and use the same conditions as for the determination and the blank test and with the same indicator.

6 Expression of results

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

$$\left[(2\,V_1c_1 - V_2c_2) - (2\,V_1c_1 - V_3c_2)\right] \times 14{,}006 \times \frac{100}{m_0 \times 10^3}$$

where

c_1 is the concentration, in moles per litre, of the sulfuric acid solution used for the determination and for the blank test;

c_2 is the concentration, in moles per litre, of the sodium hydroxide solution used for the determination and for the blank test;

V_1 is the volume, in millilitres, of the sulfuric acid solution used for the determination and for the blank test;

V_2 is the volume, in millilitres, of the sodium hydroxide solution used for the determination;

V_3 is the volume, in millilitres, of the sodium hydroxide solution used for the blank test;

m_0 is the mass, in grams, of the test portion (5.1).

7 Precision

Precision data have been analysed statistically from an interlaboratory study in which 16 laboratories (6 countries) participated with 7 levels. No statistical relationship between repeatability (r) or reproducibility (R) and the mean value of the ammoniacal nitrogen content of the samples was found.

7.1 Repeatability, r

The difference between two individual test results, obtained simultaneously or in rapid succession by the same analyst, using the same apparatus, on identical test material, under the same operating conditions, should not exceed 0,16 % (m/m), expressed as nitrogen (N) content, at a confidence level of 95 %.

7.2 Reproducibility, R

The difference between two individual and independent test results, obtained by different analysts in different laboratories, on identical test material, should not exceed 0,71 % (m/m), expressed as nitrogen (N) content, at a confidence level of 95 %.

8 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 7408;

b) the results and the method of expression used;

c) details of any unusual features noted during the determination;

d) details of any operations not included in this International Standard, or regarded as optional.

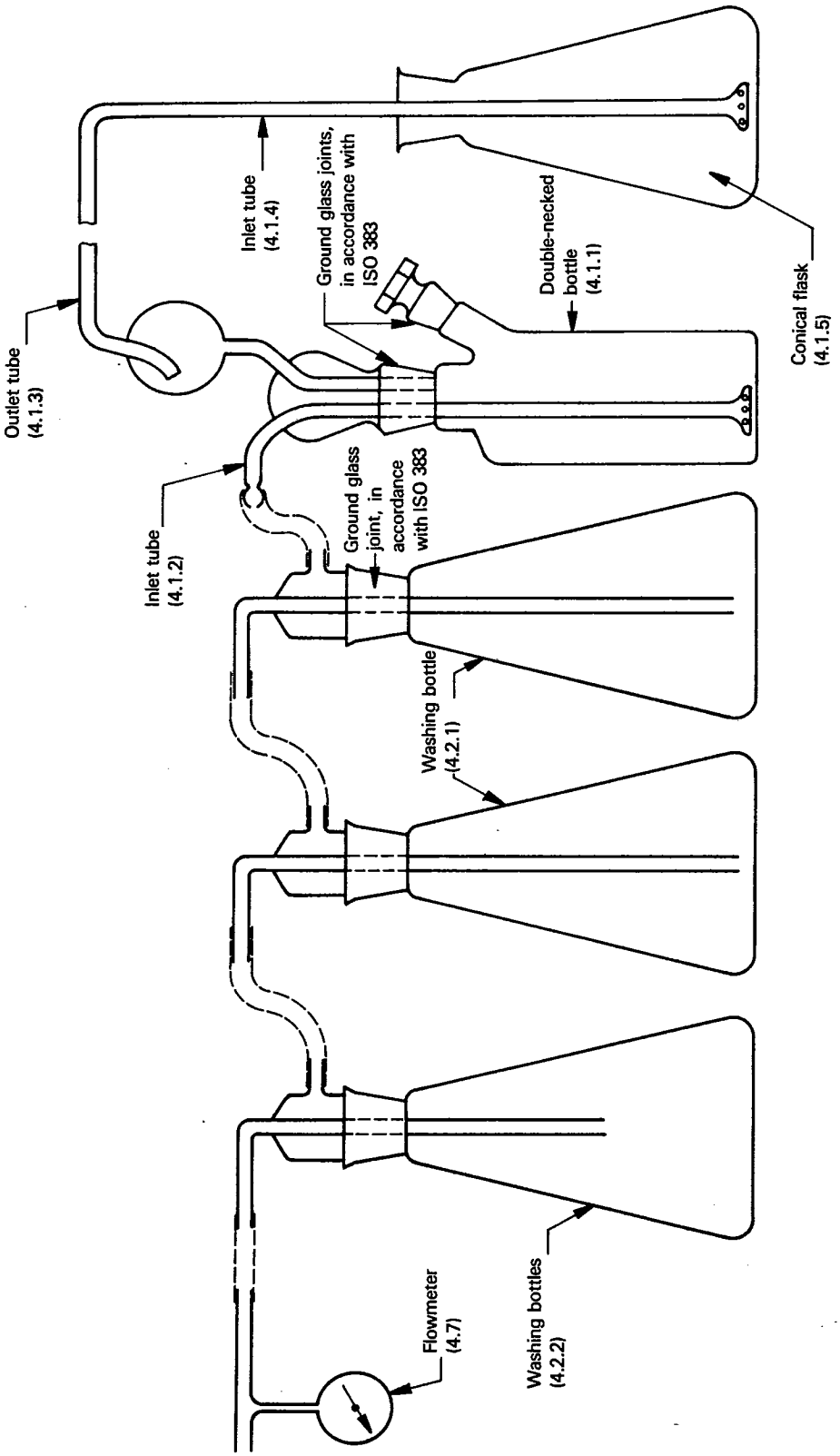

Figure 1 — Typical apparatus for the determination of ammoniacal nitrogen content using compressed air

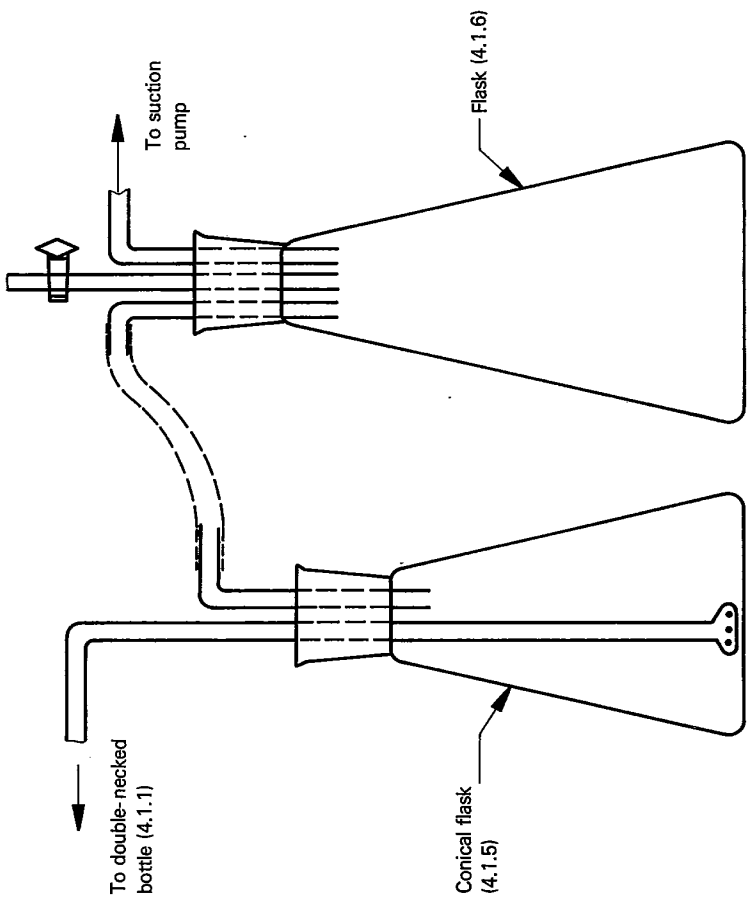

Figure 2 — Typical apparatus for the determination of ammoniacal nitrogen content using suction