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# 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*

First edition — 1983-12-15

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## Foreword

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India

# 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,  $c(\text{H}_2\text{SO}_4) = 0,25 \text{ mol/l.}^{1)}$

**3.5 Sulfuric acid,** standard volumetric solution,  $c(\text{H}_2\text{SO}_4) = 0,05 \text{ mol/l.}^{2)}$

**3.6 Sodium hydroxide,** approximately 120 g/l solution.

**3.7 Sodium hydroxide,** standard volumetric solution,  $c(\text{NaOH}) = 0,50 \text{ mol/l.}^{1)}$

**3.8 Sodium hydroxide,** standard volumetric solution,  $c(\text{NaOH}) = 0,10 \text{ mol/l.}^{2)}$

**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  $1 \pm 0,2 \text{ mm}$  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.

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

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

**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.<sup>1)</sup>

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.

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