International Standard ISO 4176

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Fertilizers — Determination of nitrate nitrogen content — Nitron gravimetric method

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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 4176 was developed by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*, and was circulated to the member bodies in October 1978.

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

Australia
Bulgaria
Canada
Czechoslovakia
Egypt, Arab Rep. of
Ethiopia
France
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Poland
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United Kingdom
USSR

No member body expressed disapproval of the document.

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Fertilizers — Determination of nitrate nitrogen content — Nitron gravimetric method

1 Scope and field of application

This International Standard specifies the nitron 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 % of the result obtained.

2 References

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

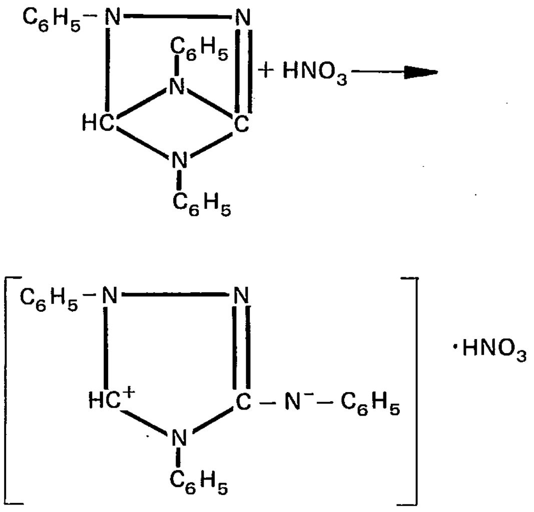
ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

3 Principle

Precipitation of nitrate ions in acid solution as a complex with nitron reagent.

Filtration of the precipitate, drying and weighing.

4 Reaction



5 Reagents

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

5.1 Acetic acid, 28,5 % (V/V) solution.

Dilute 285 ml of glacial acetic acid to 1 000 ml with water.

5.2 Sulphuric acid solution.

Dilute one volume of sulphuric acid (ϱ_{20} = 1,84 g/ml) with three volumes of water.

5.3 Nitron, 100 g/l solution.

Dissolve 10 g of nitron {(3,5,6-triphenyl-2,3,5,6-tetraazabicyclo [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.

New reagent shall be used to avoid a high result in the blank test.

6 Apparatus

Ordinary laboratory apparatus and

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

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

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

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

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

6.6 Ice-bath, capable of being maintained at a temperature of 0 to 0,5 °C.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the laboratory sample[1] and transfer to the volumetric flask (6.1).

7.2 Preparation of test solution

7.2.1 Products soluble in water

Add about 400 ml of water at 20 °C to the test portion (7.1) and, using the mechanical flask shaker (6.5), shake the flask continuously for 30 min. Dilute to the mark with water and mix.

7.2.2 Products containing water-insoluble material likely to retain nitrate

Add 50 ml of water and 50 ml of the acetic acid solution (5.1) to the test portion (7.1). 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 (6.5), shake the flask continuously for 30 min. Dilute to the mark with water and mix.

7.3 Determination

7.3.1 Filter the test portion (7.2) 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 (6.2), transfer an aliquot portion of the filtrate containing 11 to 23 mg (preferably 17 mg) of nitrate nitrogen, to a 250 ml beaker. Dilute with water to 100 ml.

7.3.2 Add 10 to 12 drops of the sulphuric acid solution (5.2). Check that the pH is between 1 and 1,5. Heat rapidly to the boiling point and, without allowing the solution to boil[2], remove the flask from the source of heat. Check that no calcium sulphate has precipitated; if it has, dissolve it by adding a few more drops of the sulphuric acid solution. Add 10 to 12 ml of the nitron solution (5.3) in one addition. Place the flask in the ice-bath (6.6) and stir the contents for 2 min. Leave the flask in the ice-bath for 2 h. Add, from time to time, sufficient ice to the ice-bath to ensure that the temperature of the contents of the flask remains between 0 and 0,5 °C.[3]

Weigh the filter crucible (6.3) 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 to 12 ml of water at between 0 and 0,5 °C. Dry the crucible and precipitate in the oven (6.4), 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.

7.4 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 100 ml with water. Proceed as described in (7.3.2). The mass of precipitate shall not exceed 1 mg. 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.

8 Expression of results

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 7.3.1;

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;

375,3 is the relative molecular mass of the complex.

9 Precision

The statistical information given below was obtained from the analysis of 30 sets of results, from laboratories in seven different countries.

9.1 Repeatability

Successive results obtained using this method on identical test material and under the same conditions (same operator, same laboratory and same time) shall be considered suspect if they differ by more than 0,4 % of the mean of their values.

9.2 Reproducibility

Individual results obtained using this method on identical test material, but under different conditions (different operators, different laboratories, different apparatus and/or different times) shall be considered suspect if they differ by more than 1,8 % of the mean of their values.

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

2) Urea and urea aldehyde condensates are decomposed by boiling acid.

3) Temperatures lower than 0 °C will lead to high results, temperatures higher than 0,5 °C to low results.

10 Test report

The test report shall include the following particulars :

a) the reference of the method used, i.e. ISO 4176;

b) the results and the method of expression used;

c) all information necessary for the complete identification of the sample;

d) any operation not specified in this International Standard or regarded as optional, together with any factors likely to have influenced the result.