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Soil quality — Determination of total nitrogen — Modified Kjeldahl method

Qualité du sol — Dosage de l'azote total — Méthode de Kjeldahl modifiée

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11261 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Annex A of this International Standard is for information only.

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Soil quality — Determination of total nitrogen — Modified Kjeldahl method

1 Scope

This International Standard specifies a method for the determination of the total nitrogen (ammonium-N, nitrate-N, nitrite-N and organic N) content of a soil. Nitrogen in N-N-linkages, N-O-linkages and some heterocyclics (especially pyridine) is only partially determined. This International Standard is applicable to all types of soils.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analyses.*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method.*

3 Principle

The method is based on the Kjeldahl-digestion, but titanium dioxide (TiO_2) is used as the catalyst instead of selenium.

NOTE 1 Titanium dioxide is ecotoxicologically less harmfull than selenium.

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4 Reagents

All reagents shall be of recognized analytical grade. Use water of grade 2 complying with ISO 3696.

4.1 Salicylic acid/sulfuric acid.

Dissolve 25 g of salicylic acid in 1 litre of concentrated sulfuric acid $\left(\varrho = 1{,}84\ \text{g/cm}^3\right)$.

4.2 Potassium sulfate catalyst mixture.

Grind and thoroughly mix 200 g of potassium sulfate, 6 g of copper(II) sulfate pentahydrate and 6 g of titanium dioxide, with the crystal structure of anatase.

4.3 Sodium thiosulfate pentahydrate.

Crush the crystals to form a powder that passes through a sieve with an aperture of 0,25 mm.

4.4 Sodium hydroxide, $c(\text{NaOH}) = 10$ mol/l.

4.5 Boric acid solution, $\rho(H_3BO_3) = 20$ g/l.

4.6 Mixed indicator.

Dissolve 0,1 g of bromocresol green and 0,02 g of methyl red in 100 ml of ethanol.

4.7 Sulfuric acid, $c(H^+) = 0{,}01$ mol/l.

5 Apparatus

Usual laboratory equipment and

5.1 Digestion flasks or **tubes**, of nominal volume 50 ml, suitable for the digestion stand.

5.2 Digestion stand.

NOTE 2 Glass tubes placed in holes drilled into an aluminium block are also suitable.

5.3 Distillation apparatus, preferably of the Parnas-Wagner type.

5.4 Burette, graduated in intervals of 0,01 ml or smaller.

6 Pretreatment of soil samples

Samples shall be pretreated according to ISO 11464.

NOTE 3 Losses of nitrogen can occur with samples of high ammonium-N and nitrate-N content. Therefore, excessive drying (105 °C) should be avoided.

7 Procedure

Place a test portion of the air-dried soil sample, of about 0,2 g (expected nitrogen content ≈ 0,5 %) to 1 g (expected nitrogen content ≈ 0,1 %), in the digestion flask (5.1). Add 4 ml of salicylic/sulfuric acid (4.1) and swirl the flask until the acid is thoroughly mixed with the soil. Allow the mixture to stand for at least several hours (or overnight). Add 0,5 g of sodium thiosulfate (4.3) through a dry funnel with a long stem that reaches down into the bulb of the digestion flask, and heat the mixture cautiously on the digestion stand (5.2) until frothing has ceased.

Then cool the flask, add 1,1 g of the catalyst mixture (4.2) and heat until the digestion mixture becomes clear. Boil the mixture gently for up to 5 h so that the sulfuric acid condenses about 1/3 of the way up to the neck of the flask. Ensure that the temperature of the solution does not exceed 400 °C.

NOTE 4 In most cases a boiling period of 2 h is sufficient.

After completion of the digestion step, allow the flask to cool and add about 20 ml of water slowly while shaking. Then swirl the flask to bring any insoluble material into suspension and transfer the contents to the distillation apparatus (5.3). Rinse three times with water to complete the transfer. Add 5 ml of boric acid (4.5) to a 100 ml conical flask and place the flask under the condenser of the distillation apparatus in such a way that the end of the condenser dips into the solution. Add 20 ml of sodium hydroxide (4.4) to the funnel of the apparatus and run the alkali slowly into the distillation chamber. Distil about 40 ml of condensate (the amount for quantitative results depends on the dimensions of the apparatus), rinse the end of the condenser, add a few drops of indicator (4.6) to the distillate and titrate with sulfuric acid (4.7) to a violet endpoint.

NOTE 5 A potentiometric titration is also possible. The endpoint of the titration should be pH = 5,0.

NOTE 6 If steam distillation is used, a distillation rate of up to about 25 ml/min is applicable. Stop the distillation when about 100 ml of distillate have been collected.

Carry out a blank test in which the same procedure is performed without soil. Notify the consumption of sulfuric acid in the blank test and in the tests of the soil samples.

8 Calculation of the result

The total content of nitrogen, (w_N), in milligrams per gram, is calculated using the formula:

$$w_N = \frac{(V_1 - V_0) \times c(\mathrm{H}^+) \times M_N}{m} \times \frac{100 + w_{\mathrm{H_2O}}}{100}$$

where

V_1 is the volume, in millilitres, of the sulfuric acid (4.7) used in the titration of the sample [indicator (4.6)];

V_0 is the volume, in millilitres, of the sulfuric acid (4.7) used in the blank test [indicator (4.6)];

$c(\mathrm{H}^+)$ is the concentration of H^+ in the sulfuric acid (4.7), in moles per litre [e.g. if 0,01 mol/l sulfuric acid is used, $c(\mathrm{H}^+)$ = 0,02 mol/l];

M_N is the molar mass of nitrogen, in grams per mole (= 14);

m is the mass, in grams, of the air-dried sample of soil;

$w_{\mathrm{H_2O}}$ is the water content, expressed as a percentage by mass, on the basis of oven-dried soil, determined according to ISO 11465.

Round the result to two significant figures.

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