SLOVENSKI STANDARD
SIST ISO 13878:1999

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Kakovost tal - Določ evanje skupnega dušika po suhem sežigu (elementna analiza)

Soil quality -- Determination of total nitrogen content by dry combustion ("elemental analysis")

Qualité du sol -- Détermination de la teneur totale en azote par combustion sèche ("analyse élémentaire")

Ta slovenski standard je istoveten z: **ISO 13878:1998**

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13.080.10	Kemijske znaļ ilnosti tal	Chemical characteristics of soils

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INTERNATIONAL STANDARD

ISO 13878

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Soil quality — Determination of total nitrogen content by dry combustion ("elemental analysis")

Qualité du sol — Détermination de la teneur totale en azote par combustion sèche («analyse élémentaire»)

Reference number
ISO 13878:1998(E)

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 13878 was prepared by Technical Committee ISO/TC 190, *Soil quality,* Subcommittee SC 3, *Chemical methods and soil characteristics.*

Annexes A and B of this International Standard are for information only.

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Soil quality — Determination of total nitrogen content by dry combustion ("elemental analysis")

1 Scope

This International Standard describes a method for the determination of the total nitrogen of soil after dry combustion.

It is applicable to all types of soil.

NOTE — The method was developed originally as a manual method by Dumas [3]. Its applicability is improved greatly due to the use of modern automated equipment.

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 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 nitrogen content of a soil pretreated in accordance with ISO 11464 is determined by heating to a temperature of at least 900 °C in the presence of oxygen gas. Mineral and organic nitrogen compounds are oxidized and/or volatilized. The combustion products are oxides of nitrogen (NO_x) and molecular nitrogen (N_2). After transforming all nitrogen forms into N_2, the content of total nitrogen is measured using thermal conductivity.

4 Reagents

All reagents shall be of recognized analytical grade.

4.1 Combustion gas (oxygen)

For special requirements, see the instruction manual of the apparatus used.

4.2 Chemicals and/or catalysts

For reduction, oxidation, removal and/or fixing of combustion gases which interfere with the analysis.

4.3 Calibration substances

For example, acetanilide (C_8H_9NO), L-aspartic acid ($C_4H_7NO_4$), amino acids of known composition or soil samples with certified nitrogen content.

NOTE — The nitrogen content of the calibration substance should be as similar to the soil nitrogen content as possible.

5 Apparatus

5.1 Analytical balance, capable of weighing accurately to 0,1 mg, or **microbalance,** capable of weighing accurately to 0,01 mg.

5.2 Combustion apparatus used to determine total nitrogen content at a temperature of at least 900 °C, including a detector for measuring the nitrogen gas formed. Consult the manufacturer's manual for the apparatus.

5.3 Crucibles of various sizes, e.g. 10 ml or 20 ml nominal volume. Special requirements are given in the manual of the apparatus used.

6 Laboratory sample

Pretreat the soil in accordance with ISO 11464. Use the fraction of particles of diameter < 2 mm to determine the content of total nitrogen. The same fraction is used to determine the water content in accordance with ISO 11465.

If a soil mass of less than 2 g is required for nitrogen analysis, mill a representative subsample further, to pass a sieve of an aperture specified in the manufacturer's manual to ensure sufficient test reproducibility. Ensure sufficient milling of the soil concerned by comparing the results of multiple analyses (see clause 9).

NOTE — The appropriate size of particles of the test sample depends primarily on the more or less homogeneous distribution and composition of soil organic matter.

7 Procedure

7.1 Calibration of the apparatus

Calibrate the apparatus as described in the relevant manual, using one of the calibration substances mentioned in 4.3. The substance should have a nitrogen content similar to that of the sample.

7.2 Determination of total nitrogen content

The amount of test sample for analysis depends on the expected total nitrogen content and on the apparatus used. Weigh out m_1 g of the air-dried sample or subsample into a crucible (5.3). Carry out the analyses in accordance with the manufacturer's manual for the apparatus.

Normally the primary results are given as milligrams nitrogen (X_1) or a mass fraction of nitrogen (X_2), expressed as a percentage, referred to the mass of air-dry soil used (m_1).

NOTE — Soil pores are filled with air and, therefore, with nitrogen gas. Nitrogen gas can also enter the combustion cell when it is opened for sample exchange. Both facts can lead to an overestimation of the total nitrogen content of soil. Therefore, sufficient purging should be carried out by oxygen gas flow before the combustion step.

8 Calculation and expression of results

Calculate the total content of nitrogen (w_N), in milligrams per gram, on the basis of the dried soil according to the following equations:

a) For primary results given in milligrams of nitrogen:

$$w_N = X_1/m_1 \cdot (100 + w)/100$$

b) For primary results, given as percent mass fraction of nitrogen:

$$w_N = X_2 \cdot 10 \cdot (100 + w)/100$$

where

w_N is the content of N in milligrams per gram of oven-dry soil;

X_1 is the primary result in milligrams N;

X_2 is the primary result in percentage N (mass fraction);

m_1 is the mass of air-dried soil for analysis, in grams;

w is the percentage water content (mass fraction) on the basis of oven-dried soil, determined in accordance with ISO 11465.

Round the results to two significant figures.

9 Repeatability

The repeatability of the determination of the total nitrogen content, obtained from two separate consecutively executed measurements, shall satisfy the conditions given in table 1.

Table 1 — Repeatability

Total nitrogen content mg/g	**Acceptable variation**
⩽ 2,0	10 % relative
> 2,0	5 % relative

10 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

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

c) the results of the determination, in milligrams nitrogen per gram sample;

d) details of any operation not specified in this International Standard or which are optional, as well as any factor which may have affected the results.

Annex A
(informative)

Results of interlaboratory trials

Data on repeatability and reproducibility were calculated in accordance with ISO 5725-2 [1] from the results of two interlaboratory trials conducted in 1992 (samples No. 2 and 3) and 1993 (sample No. 1) involving respectively 12 and 11 laboratories in Germany. These laboratories worked with equipment from different manufacturers. The following precision data were obtained (table A.1).

The difference in nitrogen contents between the present method of dry combustion and the method of wet digestion in accordance with ISO 11261 (see [2] annex B), which were tested together, was statistically not significant.

Table A.1 — Precision data for the proposed method

Sample No.	N content m mg/g	Repeatability standard deviation s_r mg/g	Repeatability r mg/g	Reproducibility standard deviation s_R mg/g	Reproducibility R mg/g
1	1,457	0,067	0,188	0,205	0,574
2*)	2,054	0,073	0,204	0,333	0,932
3*)	11,16	0,334	0,935	0,982	2,750
*) One laboratory was identified as an outlier due to the high variance of the values reported from it.					