SLOVENSKI STANDARD
SIST EN ISO 11260:2011

01-november-2011

Kakovost tal - Določevanje efektivne kationske izmenjalne kapacitete in stopnje nasičenosti z bazičnimi kationi z uporabo raztopine barijevega klorida (ISO 11260:1994+Cor 1:1996)

Soil quality - Determination of effective cation exchange capacity and base saturation level using barium chloride solution (ISO 11260:1994+Cor 1:1996)

Bodenbeschaffenheit - Bestimmung der effektiven Kationenaustauschkapazität und der Basensättigung unter Verwendung von Bariumchloridlösung (ISO 11260:1994+Cor 1:1996)

Qualité du sol - Détermination de la capacité d'échange cationique effective et du taux de saturation en bases échangeables à l'aide d'une solution de chlorure de baryum (ISO 11260:1994+Cor 1:1996)

Ta slovenski standard je istoveten z: **EN ISO 11260:2011**

ICS:

13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

SIST EN ISO 11260:2011 **en,fr,de**

EUROPEAN STANDARD
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EN ISO 11260

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ICS 13.080.10

English Version

Soil quality - Determination of effective cation exchange capacity and base saturation level using barium chloride solution (ISO 11260:1994+Cor 1:1996)

Qualité du sol - Détermination de la capacité d'échange cationique effective et du taux de saturation en bases échangeables à l'aide d'une solution de chlorure de baryum (ISO 11260:1994+Cor 1:1996)

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

The text of ISO 11260:1994+Cor 1:1996 has been prepared by Technical Committee ISO/TC 190 “Soil quality” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 11260:2011 by Technical Committee CEN/TC 345 “Characterization of soils” the secretariat of which is held by NEN.

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Endorsement notice

The text of ISO 11260:1994+Cor 1:1996 has been approved by CEN as a EN ISO 11260:2011 without any modification.

INTERNATIONAL
STANDARD

ISO
11260

First edition
1994-08-15

Soil quality — Determination of effective cation exchange capacity and base saturation level using barium chloride solution

Qualité du sol — Détermination de la capacité d'échange cationique effective et du taux de saturation en bases échangeables à l'aide d'une solution de chlorure de baryum

Reference number
ISO 11260:1994(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 11260 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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INTERNATIONAL STANDARD © ISO **ISO 11260:1994(E)**

Soil quality — Determination of effective cation exchange capacity and base saturation level using barium chloride solution

1 Scope

This International Standard specifies a method for the determination of the cation exchange capacity (CEC) at the pH of the soil and of the determination of the content of exchangeable sodium, potassium, calcium and magnesium in soil.

This International Standard is applicable to all types of air-dried soil samples; pretreatment according to ISO 11464 is recommended.

NOTES

1 The method described suffers from interference from calcium as calcite or gypsum in the sample. Also, the presence of any soluble salts gives values for the exchangeable cations that are higher than the actual exchangeable amounts [3], [4].

2 Measurement of the specific electrical conductivity of the soil samples according to ISO 11265 will indicate if the soil samples are affected by salt.

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 11265:—1), *Soil quality — Determination of the specific electrical conductivity.*

ISO 11464:—1), *Soil quality — Pretreatment of samples for physico-chemical analyses.*

3 Principle

The determination of CEC as specified in this International Standard is a modification of the method proposed by Gillman [5]. The CEC of soil samples is determined at the pH of the soil and at a low total ionic strength (about 0,01 mol/l).

The soil is first saturated with respect to barium by treating the soil three times with a 0,1 mol/l barium chloride solution. Thereafter, the soil is equilibrated with a 0,01 mol/l barium chloride solution. Subsequently, a known excess of 0,02 mol/l magnesium sulfate is added. All the barium present, in solution as well as adsorbed, is precipitated in the form of highly insoluble barium sulfate and, consequently, the sites with exchangeable ions are readily occupied by magnesium. The excess magnesium is determined by flame atomic absorption spectrometry (FAAS).

It is also possible to determine the concentrations of sodium, potassium, calcium and magnesium (and other elements such as iron, manganese and aluminium) in the 0,1 ml/l barium chloride extract of the soil.

If the barium chloride extract has a yellowish-brown colour, this indicates that some organic matter has

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1) To be published.

been dissolved. If this occurs, record it in the test report.

NOTES

3 Since organic matter contributes to the CEC, its presence will result in a measured CEC value which is an underestimation of the actual CEC.

4 Inductively coupled plasma atomic emission spectrometry (ICP-AES) may be used as an alternative method for the measurement of sodium, potassium, calcium and magnesium.

5 The sum of exchangeable cations may give a result that is greater than the actual CEC due to the dissolution of salts present in the soil. However, preliminary washing of the soil with water to remove these salts should not be employed because it will change the relative proportions of cations in the CEC.

4 Procedures

4.1 Leaching

4.1.1 Reagents

Use only reagents of recognized analytical grade and water complying with grade 2 of ISO 3696.

4.1.1.1 Barium chloride solution, $c(BaCl_2) = 0,1$ mol/l.

Dissolve 24,43 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in water and make up to 1 000 ml with water at 20 °C.

4.1.1.2 Barium chloride solution, $c(BaCl_2) = 0,002\ 5$ mol/l.

Dilute 25 ml of the 0,1 ml/l barium chloride solution to 1 000 ml at 20 °C.

4.1.1.3 Magnesium sulfate solution, $c(MgSO_4) = 0,020\ 0$ mol/l.

Dissolve 4,93 g ± 0,01 g of magnesium sulfate heptahydrate ($MgSO_4.7H_2O$) (see note 6) in water and make up to 1 000 ml at 20 °C.

NOTE 6 $MgSO_4.7H_2O$ may lose water of crystallization on standing. The reagent should be standardized by titration with EDTA at pH 10 using Eriochrome Black T indicator or be kept in a bottle in a sealed polyethylene bag placed in a refrigerator.

4.1.2 Leaching procedure

Transfer 2,50 g of air-dried soil (particle size ⩽ 2 mm), for example, pretreated according to ISO 11464, to a tightly stoppered polyethylene centrifuge tube of about 50 ml capacity. Note the combined mass of tube and soil (m_1). Add 30 ml of barium chloride solution (4.1.1.1) to the soil and shake for 1 h. Balance the tubes and centrifuge at 3 000 *g* for 10 min.

Transfer the supernatant liquid to a 100 ml volumetric flask. Repeat the addition of 30 ml of the barium chloride solution, the shaking and centrifugation twice more, adding the supernatant liquid to the 100 ml volumetric flask each time. Make up to the volume of the volumetric flask with barium chloride solution (4.1.1.1).

Mix, filter and store the extract for the determination of the concentration of sodium, potassium, calcium and magnesium in accordance with 4.3 and 4.4. Add 30 ml of barium chloride solution (4.1.1.2) to the soil cake and shake overnight. (The barium concentration in the equilibrium solution will be about 0,01 mol/l when 2,5 ml of solution is left in the soil cake.) Balance the tubes and centrifuge at about 3 000 g for 10 min. Decant the supernatant liquid.

Weigh the tube with its contents and cover (m_2). Add 30 ml of magnesium sulfate solution (4.1.1.3) to the soil cake and shake overnight. Balance the tubes and centrifuge at 3 000 *g* for 10 min. Decant the supernatant solution through a coarse filter paper (7 cm diameter) into a conical flask and store for the determination of the concentration of excess of magnesium in accordance with 4.2.4.

Prepare a blank by following the above described procedure completely without the addition of soil.

4.2 Determination of CEC

4.2.1 Principle

To prevent the formation of refractory compounds of magnesium with phosphate, aluminium, etc. in the flame, an acidified lanthanum solution is added to the solution obtained in accordance with 4.1.2, and magnesium is then determined by FAAS.

4.2.2 Reagents

Use only reagents of recognized analytical grade and distilled or deionized water for all solutions.

4.2.2.1 Hydrochloric acid, $c(HCl) = 12$ mol/l ($\rho = 1,19$ g/ml).

4.2.2.2 Magnesium standard solution, $c(\mathrm{Mg}) = 0{,}001\ 0$ mol/l.

Pipette 50,0 ml of the 0,020 0 mol/l magnesium sulfate solution (4.1.1.3) into a volumetric flask of 1 000 ml and make up to the mark with water.

4.2.2.3 Acidified lanthanum solution, $\rho(\mathrm{La}) = 10$ mg/l.

Dissolve 15,6 mg of lanthanum nitrate hexahydrate $[\mathrm{La(NO_3)_3.6H_2O}]$ in water in a 500 ml volumetric flask, add 42 ml of hydrochloric acid (4.2.2.1) and make up to the mark with water.

4.2.3 Calibration series

Pipette 0 ml, 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of magnesium standard solution (4.2.2.2) into a series of 100 ml volumetric flasks. Add 10 ml of acidified lanthanum solution (4.2.2.3), make each flask up to the mark with water and mix. These calibration solutions have magnesium concentrations of 0 mmol/l, 0,01 mmol/l, 0,02 mmol/l, 0,03 mmol/l, 0,04 mmol/l and 0,05 mmol/l, respectively.

4.2.4 Spectrometric procedure

Pipette 0,200 ml of each of the final filtrates of the soil samples (see 4.1.2) and of the blanks (see 4.1.2) into individual 100 ml volumetric flasks. Add 0,3 ml of the barium chloride solution (4.1.1.1) to the filtrates of the soil samples and the blanks. Next, add 10 ml of acidified lanthanum solution (4.2.2.3) to each flask, make up to the mark with water and mix.

Determine the magnesium concentration in the diluted sample extracts (c_1), the diluted blank (c_{b1}) and in the calibration solutions by FAAS at wavelength 285,2 nm, with the instrument set according to the manufacturer's instructions for optimum performance.

4.2.5 Calculation

Correct the concentrations of magnesium in the sample solutions for the volume of the liquid retained by the centrifuged soil after being treated with 0,002 5 mol/l barium chloride solution using the formula:

$$c_2 = \frac{c_1(30 + m_2 - m_1)}{30}$$

where

c_2 is the corrected magnesium concentration in the sample, in millimoles per litre;

c_1 is the magnesium concentration in the sample, in millimoles per litre;

m_1 is the mass of the centrifuge tube with air-dried soil, in grams;

m_2 is the mass of the centrifuge tube with wet soil, in grams.

Calculate the cation exchange capacity (CEC) of the soil using the formula:

$$\mathrm{CEC} = (c_{\mathrm{b1}} - c_2)3\ 000/m$$

where

CEC is the cation exchange capacity of the soil, in centimoles positive charge per kilogram;

c_2 is the corrected magnesium concentration in the sample, in millimoles per litre;

c_{b1} is the magnesium concentration in the blank, in millimoles per litre;

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

If the CEC exceeds 40 cmol+/kg, repeat the determination using less soil, adjusting the calculation accordingly.

NOTE 7 The unit "centimoles positive charge per a kilogram", written in abbreviated form as cmol+/kg, is an absolute amount equivalent to the formerly used unit millielectrons per hundred grams.

4.3 Determination of exchangeable sodium and potassium

4.3.1 Principle

Sodium and potassium are measured by FAAS on an acidified 0,1 mol/l barium chloride extract of soil samples. A caesium solution is added to the test solution to eliminate ionization interference.

4.3.2 Reagents

Use only reagents of recognized analytical grade and distilled or deionized water for all solutions.

4.3.2.1 Acidified caesium chloride solution.

Dissolve 10 g of caesium chloride in a small amount of water. Add 83 ml of hydrochloric acid (4.2.2.1) and make up to 1 000 ml with water.