



SLOVENSKI STANDARD
SIST ISO 13536:1996

01-oktober-1996

Kakovost tal - Ugotavljanje potencialne kationske izmenjalne kapacitete in izmenljivih kationov z raztopino barijevega klorida s pH=8,1

Soil quality -- Determination of the potential cation exchange capacity and exchangeable cations using barium chloride solution buffered at pH = 8,1

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Qualité du sol -- Détermination de la capacité d'échange cationique potentielle et des teneurs en cations échangeables en utilisant une solution tampon de chlorure de baryum à pH = 8,1

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Ta slovenski standard je istoveten z: ISO 13536:1995

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INTERNATIONAL
STANDARD

ISO
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**Soil quality — Determination of the
potential cation exchange capacity and
exchangeable cations using barium
chloride solution buffered at pH = 8,1**
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*Qualité du sol — Détermination de la capacité d'échange cationique
potentielle et des teneurs en cations échangeables en utilisant une
solution tampon de chlorure de baryum à pH = 8,1*

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Reference number
ISO 13536:1995(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 13536 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 the potential cation exchange capacity and exchangeable cations using barium chloride solution buffered at pH = 8,1

WARNING — Barium is a very toxic element if ingested. The barium ion has health risks for laboratory personnel working with this chemical; it is also harmful for the environment.

1 Scope

This International Standard specifies a method for the determination of the potential cation exchange capacity (CEC) of soil buffered at pH = 8,1 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.

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 11464:1994, *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 Mehlich^{[1],[2]} and modified by

Bascomb^[3]. The CEC of soil samples is determined in barium chloride solution buffered at pH = 8,1 using triethanolamine.

The soil is first saturated with respect to barium by treating the soil three times with buffered barium chloride solution. Subsequently, a known excess of 0,02 mol/l magnesium sulfate solution is added. All the barium present, in solution as well as adsorbed, is precipitated in the form of highly insoluble barium sulfate and the sites with exchangeable ions are then readily occupied by magnesium. The excess magnesium is determined by either flame atomic absorption spectrometry (FAAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES).

It is also possible to determine sodium, potassium, calcium and magnesium (and other elements) in the barium chloride extract of the soil.

NOTES

1 Besides sodium, potassium, calcium and magnesium, it is also possible to determine other cations, for example manganese, in the same extract.

2 A yellowish-brown colour of the barium chloride extract indicates that some organic matter has been dissolved. Since organic matter contributes to the CEC, the measured value will be an underestimate of the potential CEC.

3 The sum of exchangeable cations may give a result that is greater than the CEC due to the dissolution of salts present in the soil. Preliminary washing of the soil with water to remove these salts should not be employed because it could 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 distilled water or deionized water for all solutions.

4.1.1.1 Water, with a specific conductivity not higher than 0,2 mS/m at 25 °C (water conforming to grade 2 of ISO 3696).

4.1.1.2 Barium chloride solution, $c(\text{BaCl}_2) = 1 \text{ mol/l}$.

Dissolve 244 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 000 ml with water.

4.1.1.3 Hydrochloric acid, $c(\text{HCl}) = 2 \text{ mol/l}$.

Dilute 166 ml of concentrated hydrochloric acid ($\rho = 1,19 \text{ g/ml}$) with water to 1 000 ml.

4.1.1.4 Triethanolamine solution, $\text{pH} = 8,1$.

Dilute 90 ml of triethanolamine ($\text{HOCH}_2\text{CH}_2)_3\text{N}$ with water to about 1 litre and adjust the pH to $8,1 \pm 0,02$ with about 140 ml to 150 ml of hydrochloric acid (4.1.1.3). Dilute with water to 2 litres.

4.1.1.5 Extraction solution.

Mix equal volumes of solutions 4.1.1.2 and 4.1.1.4. Protect this solution from exposure to carbon dioxide during storage.

4.1.1.6 Magnesium sulfate solution, $c(\text{MgSO}_4) = 0,020 0 \text{ mol/l}$.

Dissolve $4,930 \text{ g} \pm 0,01 \text{ g}$ of magnesium sulfate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, see note 4) in water and dilute to 1 000 ml.

NOTE 4 Magnesium sulfate heptahydrate may lose water of crystallization on standing. The reagent should be standardized by titration with ethylenediaminetetraacetic acid (EDTA) at $\text{pH} = 10$ using Eriochrome Black T as the indicator. Alternatively the reagent may be kept in a sealed polyethylene bag placed in a refrigerator.

4.1.2 Procedure

Transfer 2,50 g to 5,00 g of air-dried soil with a high content of clay and/or humus, or 10 g of sandy soil or soil with a low content of humus (particle size

$\leq 2 \text{ mm}$), pretreated according to ISO 11464, into a tightly stoppered polyethylene centrifuge tube of about 50 ml. Note the mass of the tube with cover and soil (m_1). Add 30 ml of extraction solution (4.1.1.5) to the soil and shake for 1 h. Balance the tubes and centrifuge at 3 000g for 10 min.

Transfer the supernatant liquid to a 100 ml volumetric flask. Repeat the addition of extraction solution, shaking and centrifuging twice and adding the supernatant liquid to the 100 ml volumetric flask on each occasion. Dilute to the volume of the volumetric flask with extraction solution (4.1.1.5).

Mix, filter through a coarse filter paper (diameter 7 cm) and store the filtrate A for the determination of sodium, potassium, calcium and magnesium following the procedure described in 4.3 and 4.4. Add approximately 40 ml of water (4.1.1.1) to the soil cake and shake manually for 1 min to 2 min to break up the soil cake. Balance the tubes and centrifuge at about 3 000g for 10 min. Decant the supernatant to waste. Weigh the tube with its contents and cover (m_2). Add 30 ml of magnesium sulfate solution (4.1.1.6) to the soil cake and shake overnight. Balance the tubes and centrifuge at about 3 000g for 10 min.

Decant the supernatant solution through a coarse filter paper (diameter 7 cm) into a conical flask and store the filtrate B for the determination of magnesium following the procedure described in 4.2.4.

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

4.2 Determination of the CEC

4.2.1 Principle

An acidified lanthanum solution is added to the filtrate B obtained according to the procedure described in 4.1.2 and magnesium is determined by FAAS using an oxidizing (blue) air/acetylene flame. Lanthanum is added to prevent the formation of refractory compounds of magnesium with phosphate, aluminium etc. in the flame.

4.2.2 Reagents

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

4.2.2.1 Water, with a specific conductivity not higher than 0,2 mS/m at 25 °C (water conforming to grade 2 of ISO 3696).

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

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

Pipette 50,0 ml of the 0,020 0 mol/l magnesium sulfate solution (4.1.1.6) into a 1 000 ml volumetric flask and dilute to the mark with water.

4.2.2.4 Acidified lanthanum solution, $\rho(\text{La}) = 10\ \text{g/l}$.

Dissolve 15,6 g of lanthanum nitrate hexahydrate $[\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}]$ in water in a 500 ml volumetric flask, add 42 ml of hydrochloric acid (4.2.2.2) and dilute 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 (4.2.2.3) into a series of 100 ml volumetric flasks. Add 10 ml of acidified lanthanum solution (4.2.2.4), to each flask, dilute 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 Procedure

Pipette 0,20 ml of filtrate B of the soil samples (see 4.1.2) and of the blank (see 4.1.2) into individual 100 ml volumetric flasks. Add 10 ml of acidified lanthanum solution (4.2.2.4) to each flask, dilute to the mark with water and mix.

Determine the magnesium concentration in the diluted filtrate B (c_1) and the diluted blank (c_{b1}) using the calibration solutions (4.2.3) and FAAS at wavelength 285,2 nm, with the instrument set according to the manufacturer's instructions for optimum performance with an oxidizing (blue) air/acetylene flame.

4.2.5 Calculation

Correct the concentration of magnesium in filtrate B for dilution by the liquid retained in the centrifuged soil after being treated with water using the formula:

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

where

- c_1 is the magnesium concentration in the diluted filtrate B, in millimoles per litre;
- c_2 is the corrected magnesium concentration in the diluted filtrate B, 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:

$$\text{CEC} = \frac{3\ 000(c_{b1} - c_2)}{m}$$

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 diluted filtrate B solution, in millimoles per litre;

c_{b1} is the magnesium concentration in the diluted blank solution, 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, amending the calculation accordingly.

NOTE 5 The unit "centimoles positive charge per kilogram", written in abbreviated form as cmol+/kg, is numerically equal to the formerly used unit of milli-equivalents per hundred grams.

4.3 Determination of exchangeable sodium and potassium

4.3.1 Principle

Sodium and potassium are measured in an acidified barium chloride-triethanolamine extract of soil samples by flame emission spectrometry.

NOTE 6 Because of the high barium concentration in the medium, ionization interferences are eliminated.

4.3.2 Reagents

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

4.3.2.1 Water, with a specific conductivity not higher than 0,2 mS/m at 25 °C (water conforming to grade 2 of ISO 3696).

4.3.2.2 Potassium and sodium stock solution,

$\rho(\text{K}) = 1\,000\text{ mg/l}$ and $\rho(\text{Na}) = 400\text{ mg/l}$.

Dissolve 1,906 8 g of potassium chloride and 1,016 8 g of sodium chloride in water. Transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

NOTE 7 Pulverize the potassium chloride and sodium chloride, heat the powders obtained either at 400 °C to 500 °C for at least 8 h or at about 200 °C for 24 h, and cool the powders in a desiccator before use.

4.3.2.3 Diluted stock solution, $\rho(\text{K}) = 100\text{ mg/l}$ and

$\rho(\text{Na}) = 40\text{ mg/l}$.

Pipette 25,0 ml of the stock solution (4.3.2.2) into a 250 ml volumetric flask and dilute to the mark with water.

4.3.2.4 Hydrochloric acid, $c(\text{HCl}) = 1\text{ mol/l}$.

Dilute 83 ml of concentrated hydrochloric acid ($\rho = 1,19\text{ g/ml}$) to 1 000 ml with water.

4.3.3 Calibration series

Pipette 0 ml, 5 ml, 10 ml, 15 ml 20 ml and 25 ml of the diluted stock solution (4.3.2.3) into individual 50 ml volumetric flasks. Add 10,0 ml of extraction solution (4.1.1.5) and 5,0 ml of hydrochloric acid (4.3.2.4). Dilute to the mark with water. These calibration solutions have potassium concentrations of 0 mg/l, 10 mg/l, 20 mg/l, 30 mg/l, 40 mg/l and 50 mg/l and sodium concentrations of 0 mg/l, 4 mg/l, 8 mg/l, 12 mg/l, 16 mg/l and 20 mg/l, respectively.

4.3.4 Procedure

Pipette 2,0 ml of filtrate A (see 4.1.2) and of the blank extract (see 4.1.2) into individual test tubes. Add 1,0 ml of hydrochloric acid (4.3.2.4) followed by 7,0 ml of water to each test tube and mix. Determine the concentrations of sodium and potassium in the diluted filtrate A, blank and calibration solutions by flame emission spectrometry at wavelengths of 589 nm and 766 nm, respectively, using an air/propane flame.

NOTES

8 To prevent contamination with sodium, clean the glassware by soaking it overnight in 4 mol/l nitric acid, technical grade.

9 Recheck the standard with the highest concentration frequently (e.g. after every five measurements).

4.3.5 Calculations

Calculate the exchangeable sodium and potassium contents in the soil samples using the equations:

$$b(\text{Na, exch}) = \frac{2,174\,9(\rho_3 - \rho_{b2})}{m}$$

$$b(\text{K, exch}) = \frac{1,278\,8(\rho_3 - \rho_{b2})}{m}$$

where

$b(\text{Na, exch})$ is the content of exchangeable sodium in the soil, in centimoles positive charge per kilogram;

$b(\text{K, exch})$ is the content of exchangeable potassium in the soil, in centimoles positive charge per kilogram;

ρ_3 is the concentration of sodium or potassium in the diluted filtrate A, in milligrams per litre;

ρ_{b2} is the concentration of sodium or potassium in the diluted blank solutions, in milligrams per litre;

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

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4.4 Determination of exchangeable calcium and magnesium**4.4.1 Principle**

Magnesium and calcium are determined in the acidified barium chloride triethanolamine extract by FAAS.

NOTE 10 Due to the high barium concentration in this medium, there is no formation of refractory compounds of magnesium or calcium with phosphate, aluminium, etc. in the flame.

4.4.2 Reagents

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

4.4.2.1 Water, with a specific conductivity not higher than 0,2 mS/m at 25 °C (water conforming to grade 2 of ISO 3696).

4.4.2.2 Hydrochloric acid, $c(\text{HCl}) = 4\text{ mol/l}$.

Dilute 330 ml of concentrated hydrochloric acid ($\rho = 1,19\text{ g/ml}$) to 1 000 ml with water.

4.4.2.3 Magnesium stock solution, $\rho(\text{Mg}) = 100 \text{ mg/l}$.

Dissolve 0,837 g of magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in water. Transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

NOTES

11 Magnesium chloride hexahydrate may lose water of crystallization on standing. The reagent should be standardized by titration with EDTA buffered at $\text{pH} = 10$ using Eriochrome Black T as the indicator.

12 The use of commercial standard solutions and the dissolution of a weighed amount of metal is also possible.

4.4.2.4 Calcium stock solution, $\rho(\text{Ca}) = 1\ 000 \text{ mg/l}$.

Weigh 2,497 g of calcium carbonate (CaCO_3) into a 100 ml beaker. Dissolve the calcium carbonate in 12,5 ml of 4 mol/l hydrochloric acid (4.4.2.2). Boil the solution to expel carbon dioxide, cool to room temperature, transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

NOTE 13 Calcium carbonate should be heated for 2 h at 400°C , then cooled and stored in a desiccator, before it can be used as a standard.

4.4.2.5 Mixed stock solution, $\rho(\text{Mg}) = 5 \text{ mg/l}$ and $\rho(\text{Ca}) = 50 \text{ mg/l}$.

Pipette 5,0 ml of the magnesium stock solution (4.4.2.3) and 5,0 ml of the calcium stock solution (4.4.2.4) into a 100 ml volumetric flask and dilute to the mark with water.

4.4.3 Calibration series

Pipette 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of the mixed stock solution (4.4.2.5) into individual 100 ml volumetric flasks. Add 10,0 ml of extraction solution (4.1.1.5) and 10,0 ml of hydrochloric acid (4.3.2.4). Dilute to the mark with water. These calibration solutions have magnesium concentrations of 0 mg/l, 0,1 mg/l, 0,2 mg/l, 0,3 mg/l, 0,4 mg/l and 0,5 mg/l and calcium concentrations of 0 mg/l, 1 mg/l, 2 mg/l, 3 mg/l, 4 mg/l and 5 mg/l respectively.

4.4.4 Procedure

Pipette 1,0 ml of filtrate A (see 4.1.2) and of the blank (see 4.1.2) into individual test tubes. Add 1,0 ml of hydrochloric acid (4.3.2.4) followed by 8,0 ml of water to each test tube and mix. Determine the magnesium and calcium concentrations in the diluted filtrate A,

blank and calibration solutions by FAAS at wavelengths of 285,2 nm for magnesium and 422,7 nm for calcium, using an oxidizing (blue) air/acetylene flame for magnesium and a reduced (just bright) air/acetylene flame for calcium.

4.4.5 Calculations

Calculate the exchangeable magnesium and calcium contents in the soil samples using the equations:

$$b(\text{Mg, exch}) = \frac{8,228\ 8(\rho_4 - \rho_{b3})}{m}$$

$$b(\text{Ca, exch}) = \frac{4,990\ 3(\rho_4 - \rho_{b3})}{m}$$

where

$b(\text{Mg, exch})$ is the content of exchangeable magnesium in the soil, in centimoles positive charge per kilogram;

$b(\text{Ca, exch})$ is the content of exchangeable calcium in the soil, in centimoles positive charge per kilogram;

ρ_4 is the concentration of magnesium or calcium in the diluted filtrate A, in milligrams per litre;

ρ_{b3} is the concentration of magnesium or calcium in the diluted blank solutions, in milligrams per litre;

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

5 Test report

The test report shall include the following information:

- a reference to this International Standard;
- a precise identification of the sample;
- details of storage of the laboratory sample before analysis;
- the results of the determinations:
 - CEC, in centimoles positive charge per kilogram;
 - $b(\text{Na, exch})$, in centimoles positive charge per kilogram;
 - $b(\text{K, exch})$, in centimoles positive charge per kilogram;