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

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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

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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 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.

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

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,

$c(\text{BaCl}_2) = 0,1 \text{ mol/l}$.

Dissolve 24,43 g of barium chloride dihydrate ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and make up to 1 000 ml with water at 20 °C.

4.1.1.2 Barium chloride solution,

$c(\text{BaCl}_2) = 0,002 5 \text{ mol/l}$.

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,

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

Dissolve $4,93 \text{ g} \pm 0,01 \text{ g}$ of magnesium sulfate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) (see note 6) in water and make up to 1 000 ml at 20 °C.

NOTE 6 $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ 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 $\leq 2 \text{ mm}$), 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, $c(\text{HCl}) = 12 \text{ mol/l}$ ($\rho = 1,19 \text{ g/ml}$).

4.2.2.2 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 (4.1.1.3) into a volumetric flask of 1 000 ml and make up to the mark with water.

4.2.2.3 Acidified lanthanum solution,

$\rho(\text{La}) = 10\ \text{mg/l}$.

Dissolve 15,6 mg 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 (4.2.2.1) 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:

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

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.

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

Pulverize the potassium chloride and sodium chloride, heat the powders obtained either at $400\text{ }^{\circ}\text{C}$ to $500\text{ }^{\circ}\text{C}$ for at least 8 h or at about $200\text{ }^{\circ}\text{C}$ for 24 h, and cool the powders in a desiccator before use.

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

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 make up to the mark 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 0,1 mol/l barium chloride solution (4.1.1.1) and 5,0 ml of acidified caesium chloride solution (4.3.2.1). Make up 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 Spectrometric procedure

Pipette 2,0 ml of each of the soil extracts (see 4.1.2) and the blank (see 4.1.2) into test tubes. Add 1,0 ml of acidified caesium chloride solution (4.3.2.1) followed by 7,0 ml of water to each tube and mix. Determine the concentrations of sodium and potassium in the calibration solutions, samples and blank by FAAS at wavelengths of 589,0 nm and 766,0 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 formulas:

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

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

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 extracts, in milligrams per litre;

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

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

4.4 Determination of exchangeable calcium and magnesium

4.4.1 Principle

Calcium and magnesium are determined in the acidified barium chloride soil extract by FAAS. To prevent formation of refractory compounds of calcium and magnesium with phosphate, aluminium, etc. in the flame, an excess of lanthanum is added, which replaces calcium and magnesium in these compounds.

4.4.2 Reagents

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

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

Dilute 330 ml of hydrochloric acid (4.2.2.1) to 1 000 ml with water.

4.4.2.2 Magnesium stock solution,

$\rho(\text{Mg}) = 100\text{ mg/l}$.

Dissolve 0,836 g of magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in a small amount of water. Transfer

into a 1 000 ml volumetric flask and make up to the mark with water.

NOTE 10 $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ may lose water of crystallization on standing. The reagent should be standardized by titration with EDTA buffered at pH 10 using Eriochrome Black T as the indicator.

4.4.2.3 Calcium stock solution,

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

Weigh 2,497 g of calcium carbonate (CaCO_3) into a 1 000 ml flask. Dissolve the calcium carbonate in 12,5 ml of 4 mol/l hydrochloric acid (4.4.2.1). Boil the solution to expel carbon dioxide, cool to room temperature, transfer into a 1 000 ml volumetric flask and make up to the mark with water.

NOTE 11 CaCO_3 should be heated for 2 h at 400 °C before using it as a standard.

4.4.2.4 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 (4.4.2.2) and 5,0 ml of the calcium stock solution (4.4.2.3) into a 100 ml volumetric flask and make up 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 (4.4.2.4) into individual 100 ml volumetric flasks. Add 10,0 ml of 0,1 mol/l barium chloride solution (4.1.1.1) and 10,0 ml of acidified lanthanum solution (4.2.2.3). Make up 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 Spectrometric procedure

Pipette 1,0 ml of the soil extracts (see 4.1.2) and of the blank (see 4.1.2) into individual test tubes. Add 1,0 ml of acidified lanthanum solution (4.2.2.3) followed by 8,0 ml of water and mix. Determine the magnesium and calcium concentrations in the calibration solutions, the sample extracts and the blank by FAAS at wavelengths of 285,2 nm for magnesium and 422,7 nm for calcium, using a blue air/acetylene flame for magnesium and a just luminous air/acetylene flame for calcium.

4.4.5 Calculation

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

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

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

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 extracts, in milligrams per litre;

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

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

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5 Repeatability and reproducibility

Annex A presents the results of an interlaboratory trial for the determination of CEC and of the exchangeable sodium, potassium, calcium and magnesium contents in four types of soils.

6 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) a precise identification of the sample;
- c) details of storage of the laboratory sample before analysis;
- d) a statement of the repeatability achieved by the laboratory when using this method;
- e) the results of the determinations:
 - 1) CEC, in centimoles positive charge per kilogram;
 - 2) $b(\text{Na, exch})$, in centimoles positive charge per kilogram;

- 3) $b(\text{K, exch})$, in centimoles positive charge per kilogram;
- 4) $b(\text{Ca, exch})$, in centimoles positive charge per kilogram;
- 5) $b(\text{Mg, exch})$, in centimoles positive charge per kilogram.
- f) details of any operations not specified in this International Standard or regarded as optional, as well as any other factor which may have affected the results.

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Annex A

(informative)

Results of interlaboratory trials

In 1990 an interlaboratory trial was organized by the Wageningen Agricultural University to verify the procedures specified in this International Standard.

For this interlaboratory trial, the determination of CEC and the contents of exchangeable sodium, potassium, calcium and magnesium in four types of soil was carried out by eight to ten laboratories.

The characteristics of the soils analysed are given in table A.1.

In tables A.2 to A.6, the repeatability (r) and reproducibility (R) of the results of the analyses obtained by the laboratories are presented. The values have been calculated according to ISO 5725-2.

Table A.1 — Characteristics of soils used for the interlaboratory trial for determination of CEC

Soil No.	Soil type	Origin	pH (CaCl ₂)	Organic matter %	% < 2 µm (min. frac.)
100	organic	France	5,2	86,1	16,4
200	rhodic ferral	Tanzania	5,8	33,3	n.d.
300	sand	Netherlands	5,4	2,6	6,2
400	sea clay	Netherlands	7,4	2,0	16,7

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Table A.2 — Results of the interlaboratory trial for determination of CEC

Parameter	Results			
	Soil No.			
	100	200	300	400
Number of laboratories retained after eliminating outliers	10	7	10	10
Number of outliers (laboratories)	—	1	—	—
Number of accepted results	20	14	20	20
Mean value (cmol+/kg)	10,818	11,013	4,626	11,264
Standard deviation of the repeatability (S_r)	1,827	0,716	0,743	0,475
Relative standard deviation of the repeatability (%)	16,890	6,498	16,071	4,218
Repeatability limit ($r = 2,8 \times S_r$)	5,116	2,004	2,081	1,330
Standard deviation of the reproducibility (S_R)	2,006	1,530	2,401	1,078
Relative standard deviation of the reproducibility (%)	18,543	13,891	51,914	9,567
Reproducibility limit ($R = 2,8 \times S_R$)	5,617	4,284	6,724	3,017