INTERNATIONAL STANDARD

ISO 11260

Second edition
2018-05

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 et du taux de saturation en bases échangeables à l'aide d'une solution de chlorure de baryum

Reference number
ISO 11260:2018(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190 *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*.

This second edition cancels and replaces the first edition (ISO 11260:1994), which has been technically revised. It also incorporates the Technical Corrigendum ISO 11260:1994/Cor.1:1996.

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

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

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

This document is applicable to all types of air-dried soil samples. ISO 11464 can be used for pre-treatment.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 11265, *Soil quality — Determination of the specific electrical conductivity*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The determination of CEC as specified in this document is a modification of the method proposed by Gillman (see Reference [6]). The CEC of soil samples is determined at the pH of the soil and at a low total ionic strength.

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 inductively coupled plasma atomic emission spectrometry ICP-AES. Alternative methods with a comparable precision and detection limit may also be used.

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 mol/l barium chloride extract of the soil.

If the barium chloride extract has a yellowish-brown colour, this indicates that some organic matter has been dissolved. If this occurs, record it in the test report.

NOTE 1 Since organic matter contributes to the CEC, its presence will result in a measured CEC value which is an underestimation of the actual CEC.

NOTE 2 The sum of exchangeable cations can 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 can change the relative proportions of cations in the CEC.

5 Interferences

The method described suffers from interference from calcium which may be present 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 (see References [4] and [5]).

Measurement of the specific electrical conductivity of the soil samples in accordance with ISO 11265 will indicate if the soil samples are affected by salt.

6 Procedures

6.1 Sample pretreatment

Pretreat and air-dry the sample, e.g. in accordance with ISO 11464.

6.2 Leaching

6.2.1 Reagents

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

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

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

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

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

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

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

$MgSO_4 \cdot 7H_2O$ 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.

6.2.2 Leaching procedure

Transfer 2,50 g of air-dried soil (sieved to a particle size < 2 mm) 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 ± 0,1 ml of barium chloride solution (6.2.1.1) to the soil and shake for 1 h. Centrifuge the tubes 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 (6.2.1.1).

Mix, filter and store the extract for the determination of the concentration of sodium, potassium, calcium and magnesium in accordance with 6.4 and 6.5. Add 30 ml of barium chloride solution (6.2.1.2) 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 ± 0,1 ml of magnesium sulfate solution (6.2.1.3) to the soil cake and shake overnight. Centrifuge at 3 000*g* for 10 min. Decant the supernatant solution through a coarse filter paper into a conical flask and store for the determination of the concentration of excess of magnesium in accordance with 6.3.4.

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

6.3 Determination of CEC

6.3.1 Principle

The cations are determined by ICP-AES.

6.3.2 Reagents

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

6.3.2.1 Hydrochloric acid, c(HCl) = 12 mol/l (ρ = 1,19 g/ml).

6.3.2.2 Magnesium standard solution, c(Mg) = 0,001 0 mol/l.

Pipette 50,0 ml of the 0,020 0 mol/l magnesium sulfate solution (6.2.1.3) into a volumetric flask of 1 000 ml and make up to the mark with water.

6.3.2.3 Calibration check solution.

Prepare the calibration check solution by using an independent standard of the same chemical matrix as the calibration solutions. The concentration of the standard shall be in the centre of the calibration curve.

6.3.3 Calibration series

Pipette 0 ml, 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of magnesium standard solution (6.3.2.2) into a series of 100 ml volumetric flasks. 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.

6.3.4 Spectrometric procedure

Pipette 0,200 ml of each of the final filtrates of the soil samples (see 6.2.2) and of the blanks (see 6.2.2) into individual 100 ml volumetric flasks. 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 ICP-AES (see ISO 22036), with the instrument set according to the manufacturer's instructions for optimum performance.

6.3.5 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 Formula (1):

$$c_2 = \frac{c_1\left(30 + m_2 - m_1\right)}{30} \tag{1}$$

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 Formula (2):

$$\text{CEC} = \frac{\left(c_{b1} - c_2\right) \cdot 2 \cdot 500 \cdot V}{m \cdot 10} \cdot \frac{100 + w}{100} \tag{2}$$

where

- CEC is the cation exchange capacity of the soil, in centimoles positive charge per kilogram;
- c_{b1} is the magnesium concentration in the blank (see 6.2.2), in millimoles per litre, mmol/l;
- c_2 is the corrected magnesium concentration in the sample, in millimoles per litre;
- 2 is the valency of magnesium (2 positive charges, Mg^{2+});
- V is the volume of the magnesium sulfate solution (6.2.1.3) in millilitres, ml;
- m is the mass of the air-dried sample, in grams;
- 10 is the factor to convert millimoles to centimoles: 10 mmol/cmol;
- 500 is the factor of dilution during the measuring step (6.3.4);
- w is the percentage of water content by mass on the basis of oven dried soil, determined in accordance with ISO 11465.

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

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

6.4 Determination of exchangeable sodium and potassium

6.4.1 Principle

Sodium and potassium are measured by ICP-AES on an acidified 0,1 mol/l barium chloride extract of soil samples.

6.4.2 Reagents

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

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

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.

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.

Commercially available potassium and sodium stock solutions may be used.

6.4.2.2 Diluted stock solution, $\rho(\text{K})$ = 100 mg/l and $\rho(\text{Na})$ = 40 mg/l.

Pipette 25,0 ml of the stock solution (6.4.2.1) into a 250 ml volumetric flask and make up to the mark with water.

6.4.2.3 Calibration check solution.

Prepare the calibration check solution by using an independent standard of the same chemical matrix as the calibration solutions. The concentration of the standard shall be in the centre of the calibration curve.

6.4.3 Calibration series

Pipette 0 ml, 5 ml, 10 ml, 15 ml, 20 ml and 25 ml of the diluted stock solution (6.4.2.2) into individual 50 ml volumetric flasks. Add 10,0 ml of 0,1 mol/l barium chloride solution (6.2.1.1). 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.

6.4.4 Spectrometric procedure

Pipette 2,0 ml of each of the soil extracts and the blank (see 6.2.2) into test tubes. Add 8,0 ml of water to each tube and mix. Determine the concentrations of sodium and potassium in the calibration solutions, samples and blank by ICP-AES (see ISO 22036).

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

6.4.5 Calculations

Calculate the exchangeable sodium and potassium contents in the soil samples using Formulae (3) and (4):

$$b(\text{Na, exch}) = \frac{2{,}174\,9\left(\rho_3 - \rho_{\text{b2}}\right)}{m} \times \frac{100 + w}{100} \qquad (3)$$

$$b(\text{K, exch}) = \frac{1{,}278\,8\left(\rho_3 - \rho_{\text{b2}}\right)}{m} \times \frac{100 + w}{100} \qquad (4)$$

where

b(Na, exch)	is the content of exchangeable sodium in the soil, in centimoles positive charge per kilogram;
b(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;
w	is the percentage of water content by mass on the basis of oven dried soil, determined in accordance with ISO 11465.

6.5 Determination of exchangeable calcium and magnesium

6.5.1 Principle

Calcium and magnesium are determined in the acidified barium chloride soil extract by ICP-AES.

6.5.2 Reagents

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

6.5.2.1 Hydrochloric acid, $c(HCl) = 4$ mol/l.

Dilute 330 ml of hydrochloric acid (6.3.2.1) to 1 000 ml with water.

6.5.2.2 Magnesium stock solution, $\rho(Mg) = 100$ mg/l.

Dissolve 0,836 g of magnesium chloride hexahydrate ($MgCl_2 \cdot 6\,H_2O$) in a small amount of water. Transfer into a 1 000 ml volumetric flask and make up to the mark with water.

$MgCl_2 \cdot 6\,H_2O$ 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.

Commercially available magnesium stock solution may be used.

6.5.2.3 Calcium stock solution, $\rho(Ca) = 1\,000$ mg/l.

Weigh 2,497 g of calcium carbonate ($CaCO_3$) into a 1 000 ml volumetric flask. Dissolve the calcium carbonate in 12,5 ml of 4 mol/l hydrochloric acid (6.5.2.1). Boil the solution for 2 min to 5 min to expel carbon dioxide, cool to room temperature, transfer into a 1 000 ml volumetric flask and make up to the mark with water.

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

Commercially available calcium stock solution may be used.

6.5.2.4 Mixed stock solution, $\rho(Mg) = 5$ mg/l and $\rho(Ca) = 50$ mg/l.

Pipette 5,0 ml of the magnesium stock solution (6.5.2.2) and 5,0 ml of the calcium stock solution (6.5.2.3) into a 100 ml volumetric flask an make up to the mark with water.

6.5.2.5 Calibration check solution.

Prepare the calibration check solution by using an independent standard of the same chemical matrix as the calibration solutions. The concentration of the standard shall be in the centre of the calibration curve.

6.5.3 Calibration series

Pipette 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml of the mixed stock solution (6.5.2.4) into individual 100 ml volumetric flasks. Add 10,0 ml of 0,1 mol/l barium chloride solution (6.2.1.1). 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.

6.5.4 Spectrometric procedure

Pipette 1,0 ml of the soil extracts (see 6.2.2) and of the blank (see 6.2.2) into individual test tubes. Add 1,0 ml of barium chloride solution (6.2.1.1) 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 using ICP-AES (see ISO 22036).

6.5.5 Calculation

Calculate the exchangeable magnesium and calcium contents in the soil samples using Formulae (5) and (6):

$$b(\text{Mg, exch}) = \frac{8{,}228\,8(\rho_4 - \rho_{b3})}{m} \times \frac{100 + w}{100} \tag{5}$$

$$b(\text{Ca, exch}) = \frac{4{,}990\,3(\rho_4 - \rho_{b3})}{m} \times \frac{100 + w}{100} \tag{6}$$

where

b(Mg, exch)	is the content of exchangeable magnesium in the soil, in centimoles positive charge per a kilogram, cmol+/kg;
b(Ca, exch)	is the content of exchangeable calcium in the soil, in centimoles positive charge per a kilogram, cmol+/kg;
ρ_4	is the concentration of magnesium or calcium in the diluted extracts, in milligrams per litre, mg/l;
ρ_{b3}	is the concentration of magnesium or calcium in the diluted blank, in milligrams per litre, mg/l;
m	is the mass of air-dried soil, in grams, g;
w	is the percentage of water content by mass on the basis of oven dried soil, determined in accordance with ISO 11465.

7 Performance characterization

7.1 Calibration check

The result of the calibration check solution shall not deviate more than 10 %, or within 10 % of the theoretical concentration, otherwise recalibrate.

7.2 Repeatability and reproducibility

Annex A presents the performance data for the determination of CEC and of the exchangeable sodium, potassium, calcium and magnesium contents in four types of soils.

8 Test report

This test report shall contain at least the following information:

a) the test method used, together with a reference to this document, i.e. ISO 11260:2018;

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 determination:

    1) CEC, in centimoles positive charge per kilogram (cmol+/kg);

    2) *b*(Na, exch), in centimoles positive charge per kilogram (cmol+/kg);

    3) *b*(K, exch), in centimoles positive charge per kilogram (cmol+/kg);

    4) *b*(Ca, exch), in centimoles positive charge per kilogram (cmol+/kg);

    5) *b*(Mg, exch), in centimoles positive charge per kilogram (cmol+/kg).

f) details of any operations not specified in this document or regarded as optional, as well as any other factor which may have affected the results.

Annex A
(informative)

Performance data

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

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 in accordance with ISO 5725-2.

Table A.1 — Performance data on characteristics of soils used for the 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

Table A.2 — Performance data for the 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)	0	1	0	0
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
Coefficient of variation 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
Coefficient of variation 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

Table A.3 — Performance data for the determination of the exchangeable sodium content

Parameter	Results			
	Soil No.			
	100	200	300	400
Number of laboratories retained after eliminating outliers	10	7	10	10
Number of outliers (laboratories)	0	1	0	0
Number of accepted results	20	14	20	20
Mean value (cmol+/kg)	0,115	0,066	0,054	0,098
Standard deviation of the repeatability (S_r)	0,051	0,029	0,012	0,029
Coefficient of variation of the repeatability (%)	44,510	43,424	21,911	29,750
Repeatability limit (r = 2,8 × S_r)	0,143	0,080	0,033	0,082
Standard deviation of the reproducibility (S_R)	0,051	0,038	0,031	0,099
Coefficient of variation of the reproducibility (%)	44,510	58,491	54,477	100,519
Reproducibility limit (R = 2,8 × S_R)	0,143	0,108	0,087	0,276

Table A.4 — Performance data for the determination of exchangeable potassium content

Parameter	Results			
	Soil No.			
	100	200	300	400
Number of laboratories retained after eliminating outliers	10	7	9	10
Number of outliers (laboratories)	0	1	1	0
Number of accepted results	20	14	18	20
Mean value (cmol+/kg)	0,679	0,626	0,288	0,400
Standard deviation of the repeatability (S_r)	0,103	0,112	0,033	0,076
Coefficient of variation of the repeatability (%)	15,105	17,904	11,357	19,105
Repeatability limit (r = 2,8 × S_r)	0,287	0,314	0,092	0,214
Standard deviation of the reproducibility (S_R)	0,472	0,394	0,124	0,152
Coefficient of variation of the reproducibility (%)	69,519	62,922	43,177	38,041
Reproducibility limit (R = 2,8 × S_R)	1,323	1,104	0,349	0,426