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**Soil quality — Determination of effective  
cation exchange capacity (CEC) and  
exchangeable cations using a  
hexamminecobalt trichloride solution**

*Qualité du sol — Détermination de la capacité d'échange cationique  
(CEC) effective et des cations échangeables à l'aide d'une solution de  
trichlorure de cobaltihexammine*

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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.

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The main task of technical committees is to prepare International Standards. 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.

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.

ISO 23470 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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# Soil quality — Determination of effective cation exchange capacity (CEC) and exchangeable cations using a hexamminecobalt trichloride solution

## 1 Scope

This International Standard specifies a method for the determination of the cation exchange capacity (CEC) and the content of exchangeable cations ( $\text{Al}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{K}^{+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Na}^{+}$ ) in soils using a hexamminecobalt trichloride solution as extractant.

**NOTE** As the pH of a soil suspension in the hexamminecobalt trichloride solution is close to the pH of the suspension in water, this method is considered to give the effective CEC, i.e. the CEC at the soil pH.

This International Standard is applicable to all types of air-dried soil samples which have been prepared in accordance with ISO 11464.

References and results of the comparison with other methods (barium chloride, ammonium acetate) are given in Annex A.

## 2 Normative references

The following referenced documents are indispensable for the application 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 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

## 3 Principle

Cations retained by a soil sample are exchanged with the hexamminecobalt ions of an aqueous solution, with shaking for  $60 \text{ min} \pm 5 \text{ min}$  at a temperature of  $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . The CEC is obtained by difference between the initial quantity of hexamminecobalt in solution and the quantity remaining in the extract after the exchange reaction. The measurement of hexamminecobalt concentration in the extract can be performed by direct spectrophotometric measurement, or by the determination of total ammonium nitrogen or total cobalt concentrations.

The quantities of exchanged cations are determined on the same extract using spectrometric methods, such as inductively coupled plasma atomic emission spectrometry (ICP-AES).

**NOTE 1** Exchangeable acidity can also be measured in the hexamminecobalt extract.

**NOTE 2** When exchangeable cations are held in micropores into which ammonium ions but not hexamminecobalt ions can enter, the CEC and exchangeable cation values determined with this method may be smaller than those determined by the ammonium acetate method. This has been observed in some soils containing allophane and imogolite, for example soils developed on volcanic rocks.

## 4 Reagents

Only reagents of recognized analytical quality shall be used.

**4.1 Distilled or demineralized water**, exempt from the elements under analysis and having a conductivity of less than  $0,5 \mu\text{S}\cdot\text{cm}^{-1}$ .

**4.2 Hexamminecobalt trichloride**  $[\text{Co}(\text{NH}_3)_6\text{Cl}_3]$ ,  $0,0166 \text{ mol}\cdot\text{l}^{-1}$  solution.

Dissolve, to within a milligram, 4,458 g of hexamminecobalt trichloride in a volumetric flask containing 700 ml of distilled water (4.1). Make up to volume and mix.

NOTE Hexamminecobalt trichloride can be purchased from Sigma-Aldrich or Fluka. <sup>1)</sup>

**4.3 Anti-bumping granules** (e.g. pumice stone in grain form, glass beads).

**4.4 Boric acid** ( $40 \text{ g}\cdot\text{l}^{-1}$  solution).

**4.5 Tashiro indicator**.

Dissolve 2,0 g of methyl red and 1,0 g of methylene blue in 1 000 ml of 95° ethanol.

**4.6 Phenolphthalein**.

Dissolve 1 g of phenolphthalein in 1 000 ml of 95° ethanol.

**4.7 Sodium hydroxide solution**, 33 % (ratio of mass to volume).

**4.8 Sulfuric acid** ( $\text{H}_2\text{SO}_4$ ),  $0,025 \text{ mol}\cdot\text{l}^{-1}$  standard volumetric solution.

**4.9 Ammonium sulfate**, for analysis. <http://www.iso.org/iso/catalog/standards/sist/0d9e409-c8b1-4262-a5c2-5c5e65f70a2d/iso-23470-2007>

**4.10 Stock solutions** of  $\text{Al}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{K}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Na}^+$ ,  $1,000 \text{ g}\cdot\text{l}^{-1}$ .

These solutions are supplied with a certified composition from a reputable source and are checked on a regular basis.

## 5 Apparatus

Usual laboratory apparatus and glassware, and in particular:

**5.1 Analytical balance**, the weighing uncertainty range of which will not exceed  $\pm 0,1 \%$  of the test portion mass.

**5.2 Containers for shaking**, tightly stoppered, rigid or non-rigid wall, having a volume between 75 ml and 100 ml.

**5.3 50 ml distributor**, set at  $\pm 0,25 \text{ ml}$  of the delivered volume.

**5.4 End-over-end shaker**, allowing the permanent suspension of the soil/extraction solution mixture, placed in ambient air at  $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ .

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1) Sigma-Aldrich and Fluka are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

**5.5 Slow-filtration paper filters**, allowing the checking of the adsorption level of the hexamminecobalt ions by the filter. If it cannot be neglected with respect to the measured CEC values, it should be taken into account (see 6.3).

**5.6 Centrifuge and relevant centrifuge vials**, of which the centrifugation time and speed depend on the type of centrifuge and are selected with a view to securing a clear supernatant. 4 000 r/min for 20 min are generally sufficient, but special care shall be taken due to the high content of fixed cobalt-hexammine ions on colloidal particles.

**5.7 Distillation apparatus**, of which the different parts shall be assembled to ensure tightness in order to prevent any loss of ammonia or entrainment of sodium hydroxide.

**5.8 Microburette.**

**5.9 Spectrophotometer**, allowing measurements to be performed at wavelengths of 380 nm and 475 nm, equipped with a 10 mm path-length measuring cell.

**5.10 Inductively coupled plasma atomic emission spectrometer (ICP-AES) or flame atomic absorption spectrometer.**

## 6 Procedure

### 6.1 Test portion

Pretreat soil in accordance with ISO 11464.

If  $Q$  is the number of centimoles of positive charges provided by a given volume of hexamminecobalt solution, the centimoles of positive charges carried by the test portion shall lie between  $Q/10$  and  $Q/3$  inclusive, see Table 1.

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**Table 1 — Test portions** (in 50 ml of solution 4.2)

Measured CEC, $\text{cmol}^+ \cdot \text{kg}^{-1}$	< 2,5	2,5 to 5	5 to 10	10 to 32	32 to 64
Test portion, g	10	10	5	2,5	1,25

Weigh, to within 0,1 %, the selected mass of the test portion (see ISO 11464) and transfer it to a container for shaking (5.2).

### 6.2 Exchange reaction

Add 50 ml of the hexamminecobalt trichloride solution (4.2) and shake for  $60 \text{ min} \pm 5 \text{ min}$  by means of the shaker (5.4). Filter immediately through the filter (5.5) or centrifuge (5.6). Collect the clear filtrate or supernatant. Carry out the determinations within 24 h after extraction at the latest; otherwise ascertain the neutrality of the storage conditions.

Produce an extraction “blank” under the same conditions, but without the test portion.

### 6.3 Determination of CEC

#### 6.3.1 Determination of ammonia nitrogen by distillation

##### 6.3.1.1 Procedure

Pipette 10 ml of the extract (6.2) into the distilling apparatus flask. Make up to the volume, to about 200 ml, with water (4.1). Add the anti-bumping granules (4.3).

Add to the distillate recovery container 40 ml of boric acid solution (4.4) and a few drops of Tashiro indicator (4.5). Immerse the end of the cooling apparatus to a depth of at least 1 cm in the recovery liquid. Add a few drops of phenolphthalein (4.6) to the distilling apparatus flask.

Assemble the apparatus, taking care to ensure its tightness.

Add approximately 10 ml of the sodium hydroxide solution (4.7) and progressively heat the flask so as to distil about 150 ml of liquid within 30 min. After this lapse of time, verify the neutrality of the distillate which flows from the end of the cooling apparatus by means of phenolphthalein paper. If the reaction is alkaline, resume the distillation.

Titrate the ammonia by the standard volumetric solution of sulfuric acid (4.8).

Perform a titration blank test with water using the same reagents. The poured acid volume in this case will not exceed 0,1 ml.

Verify periodically the efficiency of the apparatus by distilling a synthetic sample containing 5 mg of nitrogen provided in the form ammonium sulfate (4.9). The poured volume, corrected for the blank, shall be between 6,95 ml and 7,35 ml inclusive (for a theoretical value of 7,15).

Then sample 10 ml of the extraction blank and determine the ammonia in the same manner. In the absence of absorption of the hexaminecobalt by the filter in particular, use the solution (4.2) to perform this test.

### 6.3.1.2 Calculation of the CEC

The cation exchange capacity, expressed in centimoles of positive charges per kilogram, is given by the following equation:

$$T = \frac{(V_2 - V_1) \times 2 \times c \times 50 \times 100}{2 \times V \times m} \quad (1)$$

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where

$V_2$  is the volume of the sulfuric acid solution used for the blank test, in millilitres;

$V_1$  is the volume of the sulfuric acid solution used for the test, in millilitres;

$c$  is the concentration of the sulfuric acid solution, in moles per litre;

$V$  is the distilled volume of extract and blank test, in millilitres;

$m$  is the mass of the test portion, in grams.

Verify periodically the efficiency of the apparatus by distilling:

- 10 ml of the hexaminecobalt solution (4.2);
- 5 ml of this solution.

The difference between the two volumes of sulfuric acid (4.8) added in the titration step shall be between 9,7 ml and 10,3 ml inclusive (for a theoretical value of 10,0).

### 6.3.2 Spectrophotometric determination

#### 6.3.2.1 Calibration and measurement

The spectrophotometric determination of the dissolved hexaminecobalt is carried out at a wavelength of 475 nm without any pretreatment of the analysed solution. The calibration function shall be linear, especially in



the highest range of concentrations. Check it regularly using the following solutions: water, solution 4.2, solution 4.2 diluted with water at ratios 1/5, 2/5, 3/5, 4/5.

In such a case, for each series of measurements, calculate the coefficients of the straight calibration line using two standard solutions: water (4.1) and hexamminecobalt trichloride solution (4.2).

Transfer the test solution into the spectrophotometer and record the optical density value; deduce the quantity of dissolved hexamminecobalt. Subtract this concentration from that contained in solution 4.2 in order to obtain the fixed quantity ( $q$ ) in moles per litre ( $\text{mol}\cdot\text{l}^{-1}$ ).

### 6.3.2.2 Correction of the adsorption by the filter

Transfer the extraction "blank" into the spectrophotometer and record the optical density value, then deduce the quantity of dissolved hexamminecobalt. Subtract this concentration from that contained in solution 4.2, in order to obtain the quantity trapped by the filter ( $q_b$ ) in moles per litre ( $\text{mol}\cdot\text{l}^{-1}$ ).

### 6.3.2.3 Correction of the influence of the soluble organic matter

By colouring the extract, the organic matter solubilized during the extraction absorbs the radiations at 475 nm. When the solubilized quantities are high, the measurement of the CEC is affected by this phenomenon. This interference can be corrected by conducting a measurement at 475 nm and another one at 380 nm. The ratio between the optical densities (OD) of the pure hexamminecobalt solutions, measured at the two previous wavelengths, is  $R_1 = [\text{OD}(475)/\text{OD}(380)] = 8,13$ .

The ratio between the optical densities of the solutions containing soluble organic matter, measured at the two previous wavelengths, is  $R_2 = [\text{OD}(475)/\text{OD}(380)] = 0,2$ . This value has been estimated by conducting extractions of soils containing variable organic matter contents in the presence of a  $0,05 \text{ mol}\cdot\text{l}^{-1}$  solution of ammonium chloride.

Considering  $X_1$  and  $X_2$  as the optical densities of the test solution measured respectively at 475 nm and 380 nm, the specific optical density for hexamminecobalt at 475 nm is given by:

$$\text{OD} = (X_1 - R_2 X_2) R_1 / (R_1 - R_2) \quad (2)$$

Once this value has been obtained, deduce the real quantity of remaining hexamminecobalt with reference to the coefficient determined by calibration. Subtract this concentration from that contained in solution 4.2, in order to obtain the exchanged quantity ( $q'$ ) in moles per litre ( $\text{mol}\cdot\text{l}^{-1}$ ).

### 6.3.2.4 Calculation of the CEC

If  $m$  is the mass of the test portion, in grams, and  $V$  the volume of hexamminecobalt solution used, in millilitres, one obtains:

$$\text{CEC} = (300qV)/m, \text{ in } \text{cmol}^+\cdot\text{kg}^{-1} \quad (3)$$

If a correction of soluble organic matter is necessary:

$$\text{CEC} = (300q'V)/m, \text{ in } \text{cmol}^+\cdot\text{kg}^{-1} \quad (4)$$

If the quantity of hexamminecobalt ions adsorbed by the filter is not negligible:

$$\text{CEC} = [300(q - q_b)V]/m, \text{ in } \text{cmol}^+\cdot\text{kg}^{-1} \quad (5)$$

or

$$\text{CEC} = [300(q' - q_b)V]/m, \text{ in } \text{cmol}^+\cdot\text{kg}^{-1} \quad (6)$$

### 6.3.3 Spectrometric determination of cobalt

#### 6.3.3.1 General

Cobalt concentrations are assumed to be quantitatively linked to hexamminecobalt concentrations. CEC is given as the difference between the hexamminecobalt content of the original extracting solution and the hexamminecobalt content remaining in the extract after the exchange reaction with the soil.

#### 6.3.3.2 Experimental conditions

See the details given in 6.4.5.

#### 6.3.3.3 Calculation of the CEC

In a first step, concentrations  $c$  of cobalt have to be converted in terms of moles per litre ( $\text{mol}\cdot\text{l}^{-1}$ ) of hexamminecobalt ( $c'$ ), for example, if  $c$  is given in  $\text{mg}\cdot\text{l}^{-1}$ ,  $c' = c/58\,933$ .

If  $c'_0$  is the concentration of hexamminecobalt, in  $\text{mol}\cdot\text{l}^{-1}$ , in the original extracting solution, the exchange quantity is given by:

$$q = (c'_0 - c') \quad (7)$$

If  $c'_b$  is the concentration of hexamminecobalt, in  $\text{mol}\cdot\text{l}^{-1}$  in the extraction “blank” (see 6.3.2.1), the quantity trapped by the filter is given by:

$$q_b = (c'_0 - c'_b) \quad (8)$$

If  $m$  is the mass of the test portion, in grams, and  $V$  is the volume of hexamminecobalt solution used, in millilitres, see Equations (3) and (5).

### 6.4 Determination of cobalt and exchangeable cations

#### 6.4.1 General

The following conditions for the determination of cations  $\text{Al}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{K}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$  and  $\text{Na}^+$  by inductively coupled plasma atomic emission spectrometry (ICP-AES) in hexamminecobalt extracts are given as an example.

Due to the large variety of available instruments, and the sensitivity of measurement signals to operating conditions, each adaptation of this example shall take into account the instructions provided by the manufacturer, the range of linearity, and the possibility of spectral and non-spectral interferences.

#### 6.4.2 Standard solutions for exchangeable cations

Solution (4.2) is the blank solution (standard 0) for the determination of exchangeable cations. Note that, for the determination of CEC through the measurement of cobalt content, this solution constitutes the highest calibration point (see 6.4.3).

For each element, transfer the volumes of the stock solution (4.10) given in Table 2 into a 500 ml volumetric flask. Add and dissolve 2,229 g of hexamminecobalt trichloride, adjust the volume to the mark with water (4.1) and homogenize. This solution constitutes the highest calibration point for the determination of exchangeable cations (standard 3, Table 2).

**Table 2 — Standard solutions for exchangeable cations (standard 3)**

Element	Volume of stock solution (4.10) ml	Final concentration mg·l <sup>-1</sup>	Final concentration cmol <sup>+</sup> ·kg <sup>-1</sup>
Al <sup>3+</sup>	20	40	8,895
Ca <sup>2+</sup>	200	400	39,94
Fe <sup>2+</sup>	10	20	1,432
K <sup>+</sup>	20	40	2,046
Mg <sup>2+</sup>	10	20	3,292
Mn <sup>2+</sup>	10	20	1,456
Na <sup>+</sup>	20	40	3,480

NOTE 1 Final concentrations, in cmol<sup>+</sup>·kg<sup>-1</sup>, are calculated for a ratio extractant/soil ( $V/m$ ) of 20.

NOTE 2 A high range of concentrations for calcium is useful when both acid and calcareous soils are analysed. When only acid soils are envisaged, the highest level of concentration can be reduced.

Transfer 50 ml of standard 3 into a 100 ml volumetric flask, adjust to the mark with standard 0, and homogenize to obtain standard 2.

Transfer 25 ml of standard 3 into a 100 ml volumetric flask, adjust to the mark with standard 0, and homogenize to obtain standard 1.

#### 6.4.3 Standard solutions for cobalt

Determination of CEC is based on the difference of cobalt concentrations between standard 0 and test sample solution. The concentration range of interest can be reduced to only the highest-half part of the full range.

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Dissolve the quantities given in Table 3 in water (4.1) in 4 000 ml volumetric flasks.

**Table 3 — Standard solutions for cobalt**

Standard	Hexaminecobalt trichloride g	Final concentration cmol <sup>+</sup> ·kg <sup>-1</sup>
0	4,458	100,00
1	3,789	85,00
2	2,898	65,00
3	2,229	50,00

NOTE Final concentrations, in cmol<sup>+</sup>·kg<sup>-1</sup>, are expressed here in terms of CEC for the theoretical case of a total adsorption by soil, considering a ratio of 20 (2,5 g of soil in 50 ml of extractant).

#### 6.4.4 Spectrometric determination of exchangeable cations

Calibration standards (standards 0 to 3), extraction blank and test sample extracts can be injected after or without a dilution step. Dilution reduces the risk of non-linearity and disturbing effects due to non-spectral interferences but increases detection and quantification limits; this last effect may be of some importance for the determination of Fe<sup>2+</sup>, K<sup>+</sup>, Mn<sup>2+</sup> and Na<sup>+</sup>. Whatever the envisaged option, linearity and absences of spectral and non-spectral interferences have to be checked and background corrections have to be set up, when necessary.

Table 4 gives the wavelengths of some suitable spectral lines.