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Soil quality — Determination of potential cation exchange capacity (CEC) and exchangeable cations buffered at pH 7, using a molar ammonium acetate solution

Qualité du sol — Détermination de la capacité d'échange cationique (CEC) potentielle et de la teneur en cations échangeables, à l'aide d'une solution molaire d'acétate d'ammonium tamponnée à pH 7

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Foreword

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

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Cation exchange capacity (CEC) is an intrinsic property of soil defining the concentration of negatively charged sites on soil colloids that can adsorb exchangeable cations. Cation exchange capacity can be a good indicator of soil productivity and is useful for making recommendations of phosphorus (P), potassium (K), and magnesium (Mg) if testing soils of different textures. Cation exchange capacity is also used for regulatory purposes in monitoring land application of biosolids.

Cation exchange capacity is a measure of exchangeable bases and soil acidity at some specific soil pH. The exchangeable bases and acidity neutralize negative charges arising from permanent charges due to isomorphic substitution in clays, or pH-dependent charges from hydroxyl groups on clay and oxides or carboxyl groups on soil organic matter. A common method for determining CEC uses 1 M ammonium acetate at pH 7 (neutral NH_4OAc) and is a standard method used for soil surveys by the Natural Resource Conservation Service.[6],[7] An advantage of CEC measured at a constant pH of 7 is elimination of CEC variability due to differences in soil pH. Thus, comparisons of CEC can occur across varied soil types and lime applications. A disadvantage of the neutral NH_4OAc method is that it may not provide a realistic depiction of the actual CEC at the natural pH of the soil, particularly with soils having considerable pH-dependent charge and a soil pH that is significantly different from 7. An unbuffered salt extract can be used to determine CEC at the natural pH of soil, for example, by using a hexamminecobalt(III)-chloride solution (see ISO 23470, References [6] and [7]).

The method described here determines potential cation exchange capacity (CEC) buffered at pH 7 and exchangeable cations Ca, Mg, K, and Na. Molar ammonium acetate is added to soil to saturate exchange sites with NH_4^+ and release exchangeable cations in a leachate which are measured. The exchanged NH_4^+ is then released either with 1 M KCl or 1 M NaCl and measured to quantify the potential cation exchange capacity at pH 7.

Ammonium acetate, due to its complexing effect, can contribute to the dissolution of part of soil carbonates and other salts present in the soil. Calcium concentrations (or even magnesium) are thus no longer limited to exchangeable quantities. Presence of other soluble salts such as gypsum, sodium chloride or else would also inflate exchangeable cation quantities.

Soil quality — Determination of potential cation exchange capacity (CEC) and exchangeable cations buffered at pH 7, using a molar ammonium acetate solution

1 Scope

This document specifies a method for the determination of potential cation exchange capacity (CEC) and the content of exchangeable cations (Ca, K, Mg, Na) in soils using a molar ammonium acetate solution buffered at pH 7 as extractant.

This document is applicable to all types of air-dry soil samples which have been prepared, for example, according to ISO 11464.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Negatively charged sites on soil are saturated by ammonium ion, in leaching conditions, using a molar ammonium acetate solution. The leaching solution can be used to determine exchangeable cations by AAS or ICP AES measurements. An alcoholic solution is used to eliminate any remaining soluble ammonium ion. The soil test sample is then back extracted by a molar potassium chloride or sodium chloride solution, in order to liberate all ammonium cations fixed in the previous step. For practical reason, a single back extraction is performed. This is generally sufficient to assess the whole CEC as the molarity of the back-extraction solution is strong (molar solution of KCl), Ammonium cations are finally determined in the extract by a continuous flow spectrophotometric method.

5 Reagents

5.1 Water, with a specific conductivity not higher than 0,2 mS/m at 25 °C.

5.2 Ammonium acetate molar solution at pH 7,00.

Two ways are available to prepare ammonium acetate solution: 5.2.1 or 5.2.2.

5.2.1 From acetate ammonium solid

Dissolve 77,0 g ± 0,1 g of ammonium acetate in a 1 000 ml flask, containing approximately 900 ml of water. Adjust the pH to 7,00 ± 0,05 using ammonia solution or acetic acid solution depending on pH initial. Make up to the volume with water and mix.

5.2.2 From acetic acid and ammonium hydroxide

Make the solution in a fume hood to avoid breathing vapours of ammonia and acetic acid. Add 58 ml of concentrated glacial acetic acid (CH_3COOH) to approximately 500 ml of water. Add 68 ml of concentrated ammonium hydroxide (NH_4OH, 29 % mass fraction NH_3). Add water to yield a volume of approximately 900 ml. Adjust pH to 7,00 with dropwise additions of either ammonium hydroxide or acetic acid. Transfer into a 1 000 ml flask and make up to volume with water. Thoroughly stir contents to ensure complete mixing.

5.3 Ethyl alcohol, 95 % (free from amines)

5.4 Molar potassium chloride solution (KCl 1 mol·l^{-1})

Dissolve 74,5 g ± 0,1 g of potassium chloride in a 1 000 ml volumetric flask. Make up to the volume with water and mix.

5.5 Sodium nitroprusside solution

Weigh 0,50 g ± 0,05 g of sodium nitroprusside. Transfer into a 500 ml volumetric flask. Add 400 ml of water. Shake with a magnetic stirrer until complete dissolution. Then, adjust to volume.

This solution shall be kept in a dark coloured bottle.

5.6 Sodium salicylate solution

Weigh 12,5 g ± 0,1 g of sodium hydroxide pellets. Transfer into a 500 ml volumetric flask. Add 300 ml of water. Stir with a magnetic stirrer until complete dissolution.

Weigh 40,00 g ± 0,02 g of sodium salicylate. Add to previous sodium hydroxide solution. Stir until complete dissolution. Bring to volume with water.

This solution shall be kept in a dark coloured bottle.

5.7 Sodium dichloroisocyanurate solution

Weigh 0,50 g ± 0,05 g of sodium dichloroisocyanurate. Transfer into a 500 ml volumetric flask. Add 400 ml of water. Shake with a magnetic stirrer until complete dissolution. Then, adjust to volume.

This solution shall be kept in a dark coloured bottle.

5.8 Brij®-35[1] solution 25 % (m/V) polyoxyethylene lauryl ether

Dissolve 250 g ± 2 g of Brij-35 in a 1 000 ml beaker placed in a water bath at 60 °C and containing 800 ml of water. After cooling, transfer into a 1 000 ml flask. Adjust to the volume. Homogenize.

5.9 Buffer solution

Dissolve 33,0 g ± 0,2 g of potassium sodium tartrate tetrahydrate and 24,0 g ± 0,2 g of trisodium citrate in a 1 000 ml volumetric flask. Adjust to the volume with water. Homogenize. Add 2,5 g ± 0,1 g of Brij-35 solution (5.8). Homogenize again.

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1) Brij®-35 is the trademark of a product supplied by ICI America Inc. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.10 Standard solution

5.10.1 Solution containing 1,400 g·l^{-1} of N/NH_4^+

Weigh 6,620 g ± 0,005 g of ammonium sulphate. Quantitatively transfer into 1 000 ml volumetric flask. Adjust to volume with molar potassium chloride solution (5.4). This solution contains 0,1 mol of positive charge (0,1 mol$^+$·l^{-1}).

5.10.2 Calibration series

See Table 1.

Table 1 — Calibration series

Concentration NH4$^+$ mmol·l^{-1}	Volume of solution 5.10.1 per 1 l ml	Concentrations with reference to soil cmol$^+$·kg^{-1}	Concentration of KCl in final medium mol·l^{-1}
0	0	0	1
5	50	10	1
10	100	20	1
15	150	30	1
20	200	40	1

6 Apparatus

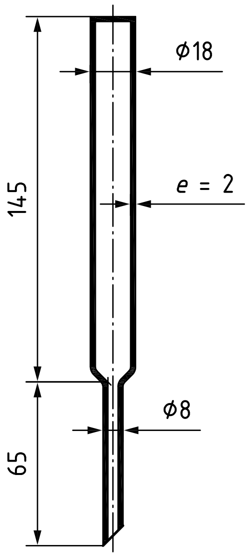
Standard laboratory glassware and equipment, in particular:

6.1 Analytical balance

The weighing uncertainty range shall not exceed ±0,1 % of the mass of the test portion.

6.2 Percolation extension, packed with a non-contaminant cellulose fibre filter mass over a height of approximately 15 mm (see Figure 1).

Dimensions in millimetres



Volume around 37 ml

Figure 1 — Percolation extension

6.3 Receptacles for shaking, with air-tight seal, optionally rigid walls, between 75 ml and 100 ml in volume. A plastic bag, sealed with heat sealing machine, can also be used.

6.4 Dispenser 50 ml, set to 50 ml ± 0,25 ml or automated system providing the same precision.

6.5 Tipping shaker, enabling the permanent suspension of the soil/extraction solution mixture, placed in an ambient air at (20 °C ± 3 °C).

6.6 Filters, ashless, free from the items under assay.

6.7 Spectrocolorimeter, continuous flow (see Figure 2).

7 Laboratory samples

Use the fraction of particles of air-dried soil samples, or soil samples dried at a temperature not higher than 40 °C, which passes through a sieve with a 2 mm mesh size. For example, soil samples pretreated according to ISO 11464 or EN 16179 can be used.

8 Procedure

8.1 Test portion

Weigh 2,500 g ± 0,002 5 g of the soil sample prepared for testing and put it into the percolation extension (6.2).

8.2 Extraction step

Add approximately 20 ml molar ammonium acetate solution (5.2). Wait until almost (around 80 %) all the liquid has filtered. Then, repeat this operation a further 3 times (approximately final volume 80 ml). This step may be done by using an automated dispensing system.

If exchangeable cations have to be determined, collect all leaching solutions in a 100 ml flask. Make up to volume with molar ammonium acetate solution (5.2). This solution can be analysed using an inductively coupled plasma emission spectrometry, if exchangeable cations (Ca, Mg, K, Na) are needed, see, for example, ISO 22036 for details on analyses.

Leach the soil with ethyl alcohol (5.3) to remove any excess ammonium acetate solution. Leach the soil with a 20 ml portion of ethyl alcohol five times for a total volume of about 100 ml. Discard the leachate.

Allow to dry for 24 h at ambient temperature.

Quantitatively transfer the test portion and the filter mass into a shaking receptacle or a plastic bag (6.3). Add 50 ml of the potassium chloride solution (5.4). Close the shaking receptacle or seal the plastic bag. Shake for 1 h ± 10 min on the shaker (6.5). Filter the suspension on a filter (6.6). Retrieve the filtrate in a clean and dry vial. It is also possible to centrifuge.

Any storage of the filtrate for more than 24 h shall be subject to checking of the neutrality of the storage conditions.

If using an automated system, it is possible to realize the back extraction by percolating the KCl N solution in four portions of 20 ml. Collect all percolated solution in a 100 ml flask and adjust to the mark. Then analyse for N-NH_4^+. Take care to use the correct value for V (volume used for back extraction) in Formulae (1) and (2).

8.3 Ammoniacal nitrogen assay by continuous flow spectrophotometry

8.3.1 Apparatus set-up

Determination of ammonium ions is based on the Berthelot reaction during which a phenol derivative (here sodium salicylate), forms an indophenol in the presence of ammonium and sodium dichloroisocyanurate under the catalytic action of sodium nitroprusside. In alkaline medium, the indophenol thus formed is blue-green; its absorbance is measured at a wavelength of 660 nm. Figure 2 shows an example of a device for assaying ammoniacal nitrogen by continuous flow spectrophotometry. Follow the manufacturer's instructions for the set-up of the apparatus.

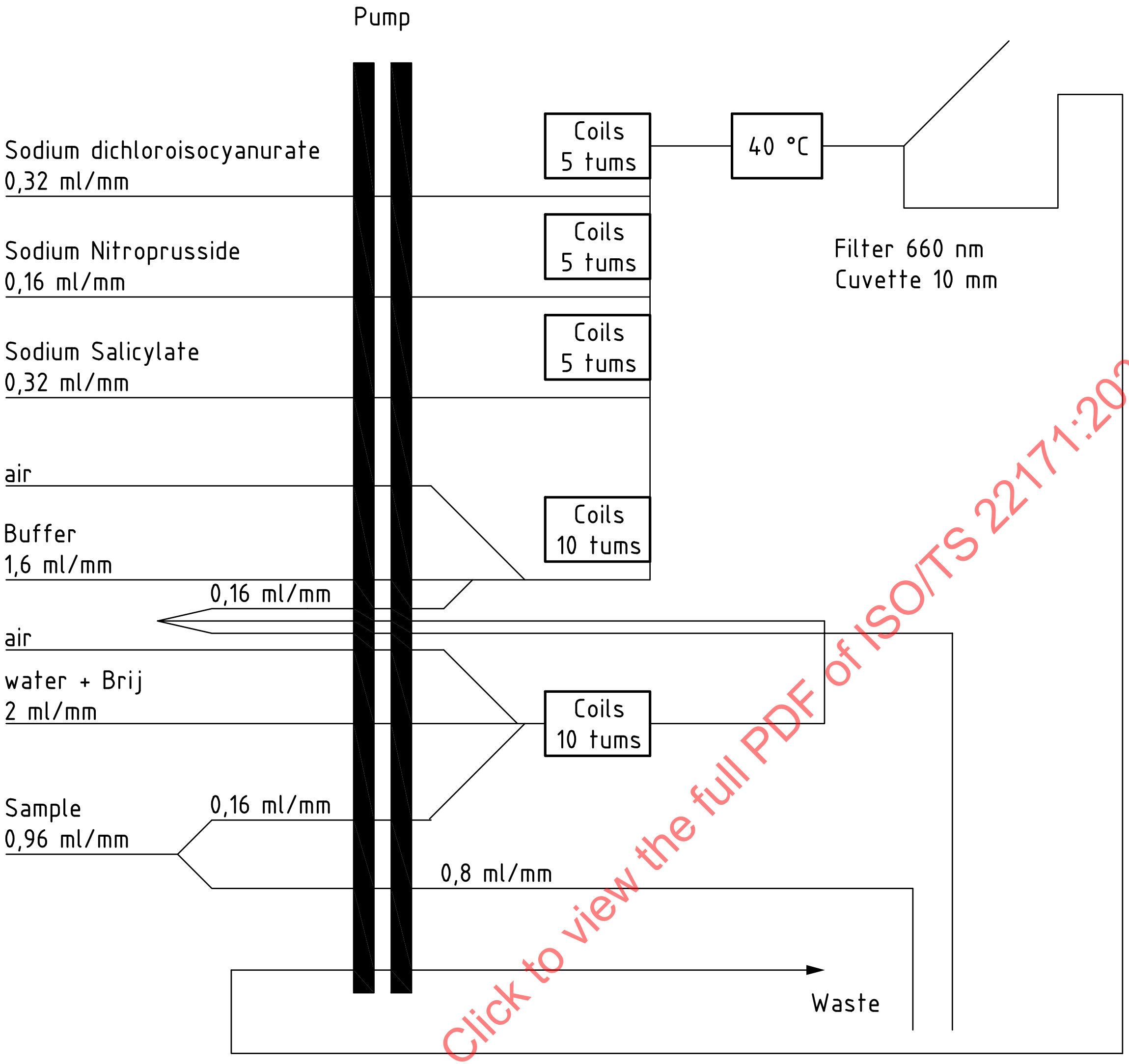


Figure 2 — Example of device for assaying ammoniacal nitrogen by continuous flow spectrophotometry

8.3.2 Assays

Determine the calibration function using the standard solutions (5.10). The calibration function coefficients may be tested and readjusted if required within predefined variation limits with a lower number of standard solutions, greater than or equal to two however, according to a clearly defined procedure. The frequency of calibration function coefficient testing, their readjustment shall be dependent on the stability of the measurement signal over time.

Present the test solutions, carrying out decontamination between each sample. Dilute with the potassium chloride solution (5.4) the solutions exceeding, in concentration, the value of the most concentrated standard solution.

In the event of a chemical modification of the ammoniacal nitrogen assay or alternative colorimetric techniques, the user shall provide validation data.