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Soil quality — Determination of effective cation exchange capacity (CEC) and exchangeable cations using a hexamminecobalt(III)chloride 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

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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).

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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 23470:2007), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the scope has been broadened for soils with a pH > 6,5;

— a new Annex C has been added;

— a new Annex D has been added;

— a new Annex E has been added;

— the document has been editorially revised.

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.

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Introduction

The cation exchange capacity (CEC) of soils and clays as well as the exchangeable cation population are essential features of soil fertility. Various attempts have been made in the literature to measure these parameters accurately and efficiently. The completeness of cation exchange on the other hand is not absolute but should be comparable between different methods. Traditional methods used ammonium or barium as exchangeable cations which require repeated treatments to ensure complete cation exchange. The oldest one-step CEC method is based on hexamminecobalt(III)chloride solution which has a much stronger affinity to soil clay minerals than the typical cations of the soil solution (usually Ca, Mg, Na and K). The principle of this method was published by Morel (1958)[11] and has been modified by Ciesielski and Sterckeman (1997)[7]. This method, as described in this document, is very efficient and comparable to the established CEC methods. It determines the effective CEC when used for soils with pH value of <6,5.

All CEC methods including hexamminecobalt(III)chloride have typical limitations such as inflation of exchangeable cations caused by dissolution of carbonates, sulfates or other soluble minerals (compare also ISO 13536). Carbonate dissolution is one of the most frequently occurring source of error (e.g. in the procedure described in ISO 13536), hence, many studies focused on minimization of their dissolution or correction of the dissolved fraction. Reference [13] summarized the discussion and presented solutions for this analytical problem. For calcareous soils or clays, the authors used exchange solutions that were previously equilibrated with calcite. In the course of the extraction, dissolution of carbonates present in the samples was minimized largely and resulting exchangeable Ca values were nearly free of errors. It was a great success compared to methods used in the past (Annex E). This method using calcite-saturated hexamminecobalt(III)chloride exchange solutions was published as VDLUFA-method[6]. The resulting exchangeable cation values agreed well with the total CEC when tested in a round robin (Annex C) which is a good measure for the plausibility of the results. Using different solution/solid ratios, identical exchangeable cation values were measured indicating absence of systematic errors caused by mineral dissolution (compare the model described in Reference [12], for detection of inflated exchangeable Ca values as described in Annex D). This calcite-saturated hexamminecobalt(III)chloride exchange solution should be used for calcareous soils and clays only, practically for soils with pH values of ≥6,5 in which only Ca, Mg, Na and K are present as “exchangeable bases”. Thus, the results are comparable to the determination of potential CEC (for example according to ISO 13563). This method was introduced to avoid erroneous (inflated) Ca values.

Hexamminecobalt(III)chloride is recommended as extractant for non-calcareous soils with a pH value of ≤6,5. As the pH value of a soil suspension in the hexamminecobalt(III)chloride solution is close to the pH value of the suspension in water, this method is considered to give the effective CEC, i.e. the CEC at the soil pH value (e.g. according to ISO 11260).

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WARNING — Persons using this document should be familiar with usual 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 cation exchange capacity (CEC) and the content of exchangeable cations (Al, Ca, Fe, K, Mg Mn, Na) in soils using a hexamminecobalt(III)chloride solution as extractant. For soils containing calcium carbonate a calcite saturated hexamminecobalt(III) chloride solution is specified particularly for determination of exchangeable Ca. This document is applicable to all types of air-dry soil samples which have been prepared according to ISO 11464.

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 11464, *Soil quality — Pretreatment of samples for physico-chemical analysis*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

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

Cations adsorbed to a soil sample are exchanged with the hexamminecobalt ions of an aqueous solution, with a (60 ± 5) min shaking at a temperature of (20 ± 2) °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 determination of total ammonium nitrogen (see 7.3.2), direct spectrophotometric measurement (see 7.3.3) or total cobalt concentrations (see 7.3.4).

The quantities of exchanged cations are determined on the same extract using spectrometric methods such as inductively coupled plasma atomic emission spectrometry (ICP-AES). In case the calcite saturated hexamminecobalt(III)chloride solution is used, the initial Ca concentration of the pure

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