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Soil quality — Determination of exchangeable acidity using barium chloride solution as extractant

Qualité du sol — Détermination de l'acidité échangeable dans des extraits au chlorure de baryum

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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

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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.

The committee responsible for this document is ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

This second edition cancels and replaces the first edition (ISO 14254:2001), which has been technically revised.

Soil quality — Determination of exchangeable acidity using barium chloride solution as extractant

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 and to ensure compliance with any national regulatory conditions.

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 exchangeable acidity in barium chloride extracts of soil samples obtained according to ISO 11260.

The procedure described herein mainly concerns the determination of total exchangeable acidity by means of a fixed pH end point titration.

Two additional and optional procedures are given, describing respectively, determinations of free H^+ acidity and aluminium in the extracts.

This document is applicable to all types of air dry soil samples.

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 11260, *Soil quality — Determination of effective cation exchange capacity and base saturation level using barium chloride solution*

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*

ISO 22036, *Soil quality — Determination of trace elements in extracts of soil by inductively coupled plasma — Atomic emission spectrometry (ICP-AES)*

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 https://www.electropedia.org/

4 Principle

The air-dried soil sample is extracted in accordance with ISO 11260. The 0,1 mol/l barium chloride extract is titrated with a 0,05 mol/l NaOH solution up to pH = 7,8 or against phenolphthalein as indicator.

An alternative method of determination of the free H^+ acidity is proposed. Sodium fluoride is added to the soil extract before the titration (BaF_2 precipitates but an excess of NaF is not necessary). Aluminium ions are complexed and only the H^+ acidity is detected during the titration process.

NOTE Titration of exchangeable acidity by means of a fixed pH end point cannot be specific to given acid species. The end point pH value retained is pH 7,8 corresponding to complete precipitation of Al^{3+} ions in synthetic solutions. Titration of soil extracts to this pH can also include some more weakly ionized species or weak acid organic compounds.

Although pH = 7,8 is the basic parameter, the use of phenolphthalein as indicator is allowed but shall be specified in the test report.

Two other optional methods for the determination of aluminium are proposed using respectively flame atomic absorption spectrometry and inductively coupled plasma emission spectrometry.

5 Reagents

Use only reagents of recognized analytical grade and distilled or deionized water for all solutions which shall be stoved in suitable plastic bottles.

5.1 **Water**, having an electrical conductivity not higher than 0,2 mS/m at 25 °C.

5.2 **Buffer solutions**, for pH-meter calibration.

5.2.1 **Buffer solution**, pH = 4,00 (20 °C).

Dissolve (10,21 ± 0,01) g of potassium hydrogen phthalate, $C_8H_5KO_4$, in water (5.1) and dilute to 1 000 ml.

5.2.2 **Buffer solution**, pH = 7,00 (20 °C).

Dissolve (3,800 ± 0,005) g of potassium dihydrogen phosphate, KH_2PO_4, and (3,415 ± 0,005) g of disodium hydrogen phosphate, Na_2HPO_4, in water (5.1) and dilute to 1 000 ml.

5.2.3 **Buffer solution**, pH = 9,22 (20 °C).

Dissolve (3,800 ± 0,005) g of disodium tetraborate decahydrate, $Na_2B_4O_7 \cdot 10\ H_2O$, in water (5.1) and dilute to 1 000 ml.

Commercial buffer solutions with other pH values can be used according to the manufacturer's instructions.

5.3 **Indicators** (optional).

5.3.1 **Methyl orange** (colour change between pH 3,0 and pH 4,4): (0,100 ± 0,005) g in 100 ml of water.

5.3.2 **Methyl red** (colour change between pH 4,2 and pH 6,2): (0,200 ± 0,005) g in 100 ml of ethanol.

5.3.3 **Phenolphthalein** (colour change between pH 8,2 and pH 10,0): (1,00 ± 0,01) g in 100 ml of ethanol.

5.4 Hydrochloric acid, $c(HCl) = 1$ mol/l.

A commercial standard solution can be used according to the manufacturer's instructions.

5.5 Sodium hydroxide solution, $c(NaOH) = (1{,}000 \pm 0{,}025)$ mol/l.

Dissolve (40,0 ± 0,5) g of carbonate free sodium hydroxide in water (5.1) and dilute to 1 000 ml. This solution will be standardized by titration with the hydrochloric acid solution (5.4) (end point: pH = 3,5 or methyl orange as indicator). Its concentration shall be tested every week at least and shall be contained between 0,975 mol/l and 1,025 mol/l.

With time, this solution can absorb carbon dioxide from atmosphere. The presence of carbonate can be detected by carrying out another titration as described above but down to pH 8,5 instead of pH 3,5 (or phenolphthalein as indicator instead of methyl orange). When a difference of volume is observed between the two end points then a new solution (5.5) shall be prepared.

A commercial standard solution can be used according to the manufacturer's instructions.

5.6 Sodium hydroxide solution, $c(NaOH) = (50{,}00 \pm 1{,}25) \times 10^{-3}$ mol/l.

Pipette 50 ml of the solution (5.5) in a 1 000 ml volumetric flask and make up to volume with water (5.1). Store in a bottle with on top a container filled with sodium lime, regularly renewed, as CO_2 trap. This solution shall be prepared every day.

5.7 Sodium fluoride solution, $c(NaF) = 1$ mol/l.

Dissolve (42,0 ± 0,2) g of sodium fluoride in water (5.1) and dilute to approximately 900 ml. Adjust the pH to 7,0 with hydrochloric acid (5.4) and the volume to 1 000 ml with water (5.1).

5.8 Barium chloride dihydrate, $BaCl_2 \cdot 2H_2O$.

5.9 Stock solution of aluminium, 1 000 g/l.

Dissolve (1,000 ± 0,001) g of aluminium metal (purity > 99,9 %) in 20 ml of concentrated hydrochloric acid ($d = 1{,}19$ g/ml) and dilute to 1 000 ml with water (5.1). A commercial standard solution can be used according to the manufacturer's instructions.

5.10 Working solution of aluminium, 0,100 g/l.

Transfer 10 ml of the aluminium stock solution (5.9) into a volumetric flask of 100 ml and adjust to the mark with water (5.1).

5.11 Barium chloride solution, $c(BaCl_2) = 0{,}5$ mol/l.

Dissolve (12,21 ± 0,01) g of barium chloride (5.8) in approximately 60 ml of water (5.1). Transfer the solution into a 100 ml volumetric flask and adjust to the mark with water (5.1).

5.12 Calibrations solutions.

Transfer respectively 5 ml, 10 ml and 20 ml aliquots of the working solution (5.10) in 100 ml volumetric flasks, dilute with approximately 20 ml of water (5.1) and add 20 ml of barium chloride solution (5.11). Finally adjust the volume with water (5.1). A fourth solution is prepared in the same way but without aluminium so as to constitute the blank of calibration.

6 Apparatus and glassware

6.1 Usual laboratory glassware, including a burette graduated in intervals of 0,05 ml or smaller.

6.2 pH-meter with automatic temperature compensation.

Set up following the manufacturer's instructions and calibrated with buffer solutions (5.2).

6.3 Magnetic stirrer.

6.4 Automatic titrimeter (optional).

Set up following the manufacturer's instructions and calibrated with buffer solutions (5.2). Results shall be expressed relatively to an end point of pH = 7,8.

6.5 Flame atomic absorption spectrometer.

Equipped with a nitrous oxide/acetylene burner and set up following the manufacturer's instructions. Determination of aluminium is commonly carried out at a wavelength of 396,2 nm.

6.6 Inductively coupled plasma optical emission spectrometer.

Determine the cation concentration according to ISO 22036.

6.7 Balance, accuracy 0,001 g.

7 Samples

7.1 Sample pretreatment

Pretreat and air-dry the sample e.g. according to ISO 11464.

7.2 Determination of water content

Determine the water content in accordance with ISO 11465.

7.3 Barium chloride extract, obtained according to ISO 11260.

7.4 Blank extract, obtained by following the complete procedure except for the addition of soil.

8 Procedure

8.1 Total exchangeable acidity

Pipette 50 ml of the extract (7.3) into a container of sufficient capacity to also receive the electrodes of the pH-meter (6.2). Introduce a magnet bar and set up the speed of the magnetic stirrer (6.3). Insert the electrodes and titrate with the sodium hydroxide solution (5.6) until a pH value of 7,8 is reached and remains stable for 30 s.

When phenolphthaleine is used titrate until the colour turns just permanently pink (in practice wait about 30 s). 50 ml of the blank solution (7.4) are titrated in the same way in order to obtain a blank value.

8.2 Free H⁺ acidity (optional)

Pipette 50 ml of the extract (7.3). Add 2,5 ml of the sodium fluoride solution (5.7). Titrate with the sodium hydroxide solution (5.6) to a pH value of 7,8.

When phenolphthaleine is used, titrate until the colour turns just permanently pink (in practice wait about 30 s). Titrate a blank (7.4) in the same way.

The total volume extracted according to ISO 11260 is 100 ml. If determinations of exchangeable cations, total acidity and free H^+ acidity are needed, pipetted volumes in 8.1 and 8.2 shall be adapted.

8.3 Spectrometric determination of aluminium (optional)

For both proposed spectrometric methods the calibration curves are practically linear for the standard solutions (5.12). Samples (7.3) exceeding the range of calibration shall be diluted with extracting solution prepared according to ISO 11260.

Contaminations by aluminium are unusual and easily avoided, so differences between the signals given by the calibration blank and by the extraction blank should not be accepted.

Barium serves as an ionization suppressant for aluminium, thus addition of potassium or caesium, which is generally recommended for determination of aluminium in nitrous oxide/acetylene flame, is not necessary.

NOTE When using ICP spectrometric method, if sensitivity remains sufficient, standard solutions (5.12) and samples (7.3 and 7.4) can be diluted in the same way with water (5.1) prior to the measurements.

Alternative spectroscopic methods with a comparable precision and detection limit may also be used.

9 Calculations

Calculate the total exchangeable acidity on basis of the oven-dried soil according to Formula (1):

$$EA = \frac{(a-b)\times T\times 100\times V}{v\times m}\times\frac{100+w}{100} \tag{1}$$

where

EA is the total exchangeable acidity of the soil in cmol+/kg on the basis of oven-dried soil;

a is the amount of sodium hydroxide used for the test portion, in ml;

b is the amount of sodium hydroxide used for the blank, in ml;

T is the concentration of the sodium hydroxide solution in mol/l;

v is the volume of test sample pipetted, in ml;

m is the mass of test sample weighed according to ISO 11260, in g;

V is the final volume of the extract according to ISO 11260, in ml;

w is the percentage of water content by mass on the basis of oven-dried soil, determined according to ISO 11465.

For free H^+ acidity use Formula (1), too. Then, a and b are the volumes added in 8.2.