
Kakovost tal – Določevanje izmenljive kislosti v ekstraktih barijevega klorida

Soil quality - Determination of exchangeable acidity in barium chloride extracts

**Soil quality — Determination of
exchangeable acidity in barium chloride
extracts**

*Qualité du sol — Détermination de l'acidité échangeable dans un extrait au
chlorure de baryum*



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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14254 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Soil quality — Determination of exchangeable acidity in barium chloride extracts

1 Scope

This International Standard 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 (see note). Two optional procedures are also given, describing respectively, determinations of free H^+ acidity and of aluminium in the extracts.

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

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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 analyses*

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

3 Principle

The soil sample is extracted in accordance with ISO 11260. The $0,1 \text{ mol} \cdot \text{l}^{-1}$ extract is titrated with a $0,05 \text{ mol} \cdot \text{l}^{-1}$ NaOH solution up to $\text{pH} = 7,8$. The use of phenolphthalein as indicator is allowed, but shall be specified in the test report.

An alternative method for determination of the free H^+ acidity is proposed, in which 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.

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

4 Reagents

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

4.1 Water having an electrical conductivity not higher than $0,2 \text{ mS} \cdot \text{m}^{-1}$ at $25 \text{ }^\circ\text{C}$ (grade 2 water in accordance with ISO 3696).

4.2 Disodium tetraborate decahydrate (borax), $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ (molecular mass = 381,5) of a purity $> 99,5 \%$, which should be stored in a tightly closed bottle.

4.3 Buffer solutions for pH-meter calibration.

4.3.1 Buffer solution pH = 4,00 ($20 \text{ }^\circ\text{C}$): Dissolve $10,21 \text{ g} \pm 0,01 \text{ g}$ of potassium hydrogen phthalate, $\text{C}_8\text{H}_5\text{KO}_4$, in water (4.1) and dilute to 1 000 ml.

4.3.2 Buffer solution pH = 7,00 ($20 \text{ }^\circ\text{C}$): Dissolve $3,800 \text{ g} \pm 0,005 \text{ g}$ of potassium dihydrogen phosphate, KH_2PO_4 , and $3,415 \text{ g} \pm 0,005 \text{ g}$ of disodium hydrogen phosphate, Na_2HPO_4 , in water (4.1) and dilute to 1 000 ml.

4.3.3 Buffer solution pH = 9,22 ($20 \text{ }^\circ\text{C}$): Dissolve $3,800 \text{ g} \pm 0,005 \text{ g}$ of disodium tetraborate decahydrate, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, in water (4.1) and dilute to 1 000 ml.

Commercial buffer solutions can be used in accordance with the manufacturer's instructions.

4.4 Indicators (optional)

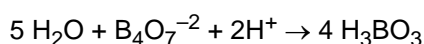
4.4.1 Methyl orange (colour change between pH = 3,0 and pH = 4,4): $0,100 \text{ g} \pm 0,005 \text{ g}$ in 100 ml of water.

4.4.2 Methyl red (colour change between pH = 4,2 and pH = 6,2): $0,200 \text{ g} \pm 0,005 \text{ g}$ in 100 ml of ethanol.

4.4.3 Phenolphthalein (colour change between pH = 8,2 and pH = 10,00): $1,00 \text{ g} \pm 0,01 \text{ g}$ in 100 ml of ethanol.

4.5 Hydrochloric acid, $c(\text{HCl}) = 1 \text{ mol} \cdot \text{l}^{-1}$

Dilute 83 ml of concentrated hydrochloric acid ($d = 1,19 \text{ g} \cdot \text{ml}^{-1} \sim 12 \text{ mol} \cdot \text{l}^{-1}$) with water to 1 000 ml. This solution shall be standardized by means of borax (4.2) following the reaction:



A mass of borax of about 1,928 g dissolved in about 100 ml of water (4.1) (heat slightly to accelerate dissolution) will need 10 ml of a solution of hydrochloric acid containing exactly $1 \text{ mol} \cdot \text{l}^{-1}$ to be neutralized.

An end-point at pH = 5 shall be considered, or methyl red used as indicator. The concentration of this solution shall be tested at least every month.

A commercial standard solution can be used in accordance with the manufacturer's instructions.

4.6 Sodium hydroxide solution, $c(\text{NaOH}) = (1,000 \pm 0,025) \text{ mol} \cdot \text{l}^{-1}$

Dissolve $40,0 \text{ g} \pm 0,5 \text{ g}$ of carbonate-free sodium hydroxide in water (4.1) and dilute to 1 000 ml. This solution shall be standardized by titration with the hydrochloric acid solution (4.5) (end point: pH = 3,5 or methyl orange as indicator).

Its concentration shall be tested at least every week and shall be within the range $0,975 \text{ mol} \cdot \text{l}^{-1}$ to $1,025 \text{ mol} \cdot \text{l}^{-1}$.

With time, this solution can absorb carbon dioxide from the 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 (4.6) shall be prepared.