
**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 14254:2001

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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.

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*.

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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

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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)

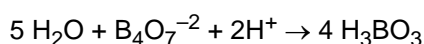
4.4.1 Methyl orange (colour change between pH = 3,0 and pH = 4,4): $0,100 \text{ g} \pm 0,005 \text{ g}$ in 100 ml of water.

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.

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

4.7 Sodium hydroxide solution, $c(\text{NaOH}) = 50 \times 10^{-3} \text{ mol} \cdot \text{l}^{-1} \pm 1,25 \times 10^{-3} \text{ mol} \cdot \text{l}^{-1}$

Pipette 50 ml of the solution (4.6) in a 1 000 ml volumetric flask and make up to volume with water (4.1). Store in a bottle with a container on top filled with soda lime, regularly renewed, as CO_2 trap. This solution shall be prepared every day.

4.8 Sodium fluoride solution, $c(\text{NaF}) = 1 \text{ mol} \cdot \text{l}^{-1}$

Dissolve $42,0 \text{ g} \pm 0,2 \text{ g}$ of sodium fluoride in water (4.1) and dilute to approximately 900 ml. Adjust the pH to 7,0 with hydrochloric acid (4.5) and the volume to 1 000 ml with water (4.1).

4.9 Barium chloride dihydrate, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$

4.10 Stock solution of aluminium, $1,000 \text{ g} \cdot \text{l}^{-1}$

Dissolve $1,000 \text{ g} \pm 0,001 \text{ g}$ of aluminium metal (purity $> 99,9 \%$) in 20 ml of concentrated hydrochloric acid ($d = 1,19 \text{ g} \cdot \text{ml}^{-1}$) and dilute to 1 000 ml with water (4.1). A commercial standard solution can be used in accordance with the manufacturer's instructions.

4.11 Working solution of aluminium, $0,100 \text{ g} \cdot \text{l}^{-1}$

Transfer 10 ml of the aluminium stock solution (4.10) into a volumetric flask of 100 ml and adjust to the mark with water (4.1).

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4.12 Barium chloride solution, $c(\text{BaCl}_2) = 0,5 \text{ mol} \cdot \text{l}^{-1}$

Dissolve $12,21 \text{ g} \pm 0,01 \text{ g}$ of barium chloride (4.9) in approximately 60 ml of water (4.1). Transfer the solution into a 100 ml volumetric flask and adjust to the mark with water (4.1).

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4.13 Calibrations solutions

Transfer respectively 5 ml, 10 ml and 20 ml aliquots of the working solution (4.11) in 100 ml volumetric flasks, dilute with approximately 20 ml of water (4.1) and add 20 ml of barium chloride solution (4.12). Finally adjust the volume with water (4.1). A fourth solution is prepared in the same way but without aluminium so as to constitute the calibration blank.

5 Apparatus

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

5.2 pH-meter

Set up following the manufacturer's instructions and calibrate with buffer solutions (4.3).

5.3 Magnetic stirrer

5.4 Automatic titrimer (optional)

Set up following the manufacturer's instructions and calibrate with buffer solutions (4.3). Results shall be expressed relatively to an end-point of $\text{pH} = 7,8$.

5.5 Flame atomic absorption spectrometer

Equip 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.

5.6 Inductively coupled plasma spectrometer

Set up following the manufacturer's instructions. Determination of aluminium is commonly carried out with background correction at various wavelengths such as 309,28 nm or 396,15 nm.

6 Sample

6.1 Barium chloride extract, obtained in accordance with ISO 11260.

6.2 Blank extract, obtained in the same way.

7 Procedure

7.1 Total exchangeable acidity

Pipette 50 ml of the extract (6.1) into a container of sufficient capacity to also receive the electrodes of the pH-meter (5.2). Introduce a bar magnet and set up the speed of the magnetic stirrer (5.3). Insert the electrodes and titrate with the sodium hydroxide solution (4.7) until a pH value of 7,8 is reached and remains stable for 30 s.

When phenolphthalein is used, titrate until the colour just turns permanently pink (in practice wait about 30 s). Titrate 50 ml of the blank solution (6.2) in the same way in order to obtain a blank value.

7.2 Free H⁺ acidity (optional)

Pipette 50 ml of the extract (6.1). Add 2,5 ml of the sodium fluoride solution (4.8). Titrate with the sodium hydroxide solution (4.7) to a pH value of 7,8.

When phenolphthalein is used, titrate until the colour just turns permanently pink (in practice wait about 30 s). Titrate a blank (6.2) in the same way.

NOTE The total volume extracted in accordance with ISO 11260 is 100 ml. If determinations of exchangeable cations, total acidity and free H⁺ acidity are needed, volumes pipetted in 7.1 and 7.2 need to be adapted.

7.3 Spectrometric determination of aluminium (optional)

For both proposed spectrometric methods, the calibration curves are practically linear for the standard solutions (4.13). Samples (6.1) with aluminium concentrations exceeding the range of calibration need to be diluted with extracting solution prepared according to ISO 11260.

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

NOTE 1 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 2 When using the ICP spectrometric method, if sensitivity remains sufficient, standard solutions (4.13) and samples (6.1 and 6.2) can be diluted in the same way with water (4.1) prior to measurement.

8 Calculations

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

$$E_A = \frac{(V_a - V_b) \cdot c_{\text{NaOH}} \cdot 100 \cdot V}{V_s \cdot m} \cdot \frac{100 + w}{100} \quad (1)$$

where

E_A is the total exchangeable acidity of the soil, in $\text{cmol} \cdot \text{kg}^{-1}$, on the basis of oven-dried soil;

V_a is the volume of sodium hydroxide used for the test portion, in millilitres;

V_b is the volume of sodium hydroxide used for the blank, in millilitres;

c_{NaOH} is the concentration of the sodium hydroxide solution, in $\text{mol} \cdot \text{l}^{-1}$;

V_s is the volume of test sample pipetted, in millilitres;

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

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

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

For free H^+ acidity use equation (1), where a and b are the volumes added in 7.2.

Calculate the exchangeable aluminium concentration on the basis of the oven-dried soil according to equation (2):

$$E_{\text{Al}} = \frac{\rho_{\text{Al}} \cdot V}{90 \cdot m} \cdot \frac{100 + w}{100} \quad (2)$$

where

E_{Al} is the concentration of exchangeable aluminium, in $\text{cmol} \cdot \text{kg}^{-1}$;

ρ_{Al} is the concentration of aluminium found in the extract, in $\text{mg} \cdot \text{l}^{-1}$;

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

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

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

9 Test report

The test report shall contain the following information:

- a reference to this International Standard;
- all information necessary for complete identification of the sample;
- the result of the determination, expressed in $\text{cmol} \cdot \text{kg}^{-1}$ to one decimal point;
- the use of phenolphthalein instead of a pH-meter;
- details of any operation not specified in this International Standard or which are optional, as well as any factor which may have affected the results.