
**Soil quality — Extraction of trace
elements from soil using ammonium
nitrate solution**

*Qualité du sol — Extraction des éléments traces du sol à l'aide d'une
solution de nitrate d'ammonium*

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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.org
Web www.iso.org

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 19730 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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Introduction

0.1 General

The displacement of trace elements in soil cannot be described solely in terms of their solubility in water, since solid and dissolved compounds interact with one another in a large number of processes. Processes that proceed in one direction (e.g. weathering, mineralization) are accompanied by equilibrium reactions between solid and dissolved forms (e.g. adsorption/desorption, precipitation/dissolution).

Factors that determine whether substances dissolve include the pH value, the redox potential, the concentration of ions in a soil solution and its complexing agent content, all of which result in the soil solution varying with location and time.

To be able to compare increases in easily soluble trace element concentrations in soil by a standardized method, soil extraction with ammonium nitrate is used, which is applicable to all those compounds of elements in a soil that may contribute to the concentration of dissolved elements under the prevailing site conditions (water-soluble compounds, exchangeably bound ions, readily soluble metal complexes).

Extraction with ammonium nitrate solution fulfils the criteria to be satisfied by an extractant for routine analyses.

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- a) It can be applied to a wide spectrum of soils and elements in a wide concentration range.
 - b) Disposal of the spent ammonium nitrate solution is environmentally friendly.
 - c) The determination of the elements in the solution has low susceptibility to interference.

The concentrations of elements in extracts obtained with other unbuffered salt solutions (such as calcium chloride or sodium nitrate solution) often correlate well with those in ammonium nitrate extracts, but the absolute concentrations are different and, at some sites, there may be concentrations deviating substantially from average values. In addition, critical concentrations may be in the region of the limit of determination in the case of some extraction methods.

In conjunction with extraction methods for determining the reserves of elements in soil (e.g. extraction with aqua regia), the proportions by mass determined by extraction with ammonium nitrate solution can serve as a basis for site investigations and for making recommendations with regard to use and restoration of soil containing critical concentrations of elements.

0.2 Prediction of impact

0.2.1 General

The concentrations determined by extraction with ammonium nitrate solution only provide an indication of the effects of trace elements, since it is necessary also to consider numerous site factors and sometimes the amounts of elements present in the soil in order to predict impact. Thus, designations such as “biologically available”, “available to plants” or “components capable of migration” are not appropriate.

In soil that is sometimes poorly aerated (e.g. water-logged soil), variations in the redox potential need to be considered when assessing the potential of arsenic and manganese to migrate.

0.2.2 Migration of elements

Extraction with ammonium nitrate solution has been found to be a suitable method of assessing easily soluble trace element fractions of a soil, whose element concentrations should be determined from samples of undisturbed soil taken from adequately distant sites and at different times.

Major factors governing the transport of elements via seepage water are:

- their chemical and physical properties;
- the thickness of the unsaturated zone;
- the distribution of easily soluble elements over the depth of the soil;
- and the actual speed with which the seepage water travels.

The easily soluble trace element fractions in soil vary with time and location. Increased variability can generally be expected with varying soil acidity (e.g. in soil covered by forest vegetation) or hydromorphic modification (e.g. in warp soil), and can also be expected where the amounts of elements in the soil vary (e.g. due to mineralization). This should be considered when designing a sampling programme.

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Soil quality — Extraction of trace elements from soil using ammonium nitrate solution

1 Scope

This International Standard specifies a method of extracting trace elements from soil using a 1 mol/l NH_4NO_3 solution (see the Introduction).

NOTE Information on determining selected elements and treatment of particular types of soil is given in Annex A.

2 Normative references

The following referenced documents are indispensable for the application 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 11464:1994, *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 with particle size < 2 mm is extracted with 1 mol/l ammonium nitrate solution using a soil to solution extraction ratio of 1:2,5 (m/V) for 120 min at (20 ± 2) °C under precise shaking conditions using an end-over-end shaker. The concentrations of elements in the extract solution are determined by appropriate analytical methods.

NOTE 1 The determination of trace elements in extracts can be performed using flame, hydride generation or electrothermal atomic absorption spectrometry, inductively coupled plasma atomic emission spectrometry, inductively coupled plasma mass spectrometry or any other relevant technique.

NOTE 2 In the case of soils with high absorption capacities, due to the relatively high solid-to-liquid ratio, it can be difficult to obtain a sufficient amount of extract solution.

4 Reagents

Reagents used shall be of analytical or higher grade. The blank value of the reagents shall be much smaller than the lowest element concentration to be determined.

4.1 Water, complying with the requirements for ISO 3696, grade 2 (electrical conductivity less than $0,1 \text{ mS}\cdot\text{m}^{-1}$ equivalent to resistivity greater than $0,01 \text{ M}\Omega\cdot\text{m}$ at 25 °C). It is recommended that the water be obtained from a water purification system that delivers high purity water having a resistivity greater than $0,18 \text{ M}\Omega\cdot\text{m}$.

4.2 Ammonium nitrate solution, $c_{\text{NH}_4\text{NO}_3} = 1 \text{ mol/l}$.

Dissolve 80,04 g of ammonium nitrate ($w_{\text{NH}_4\text{NO}_3} > 99 \%$, whose purity has been established in preliminary tests) in water and make the solution up to 1 l with water. As an alternative, ready-to-use 1 mol/l high purity ammonium nitrate solution may be used.

4.3 Nitric acid, $w_{\text{HNO}_3} = 65 \%$, $\rho_{\text{HNO}_3} \approx 1,4 \text{ g/ml}$.

4.4 Nitric acid, diluted, $\varphi = 5 \%$.

Dilute 50 ml of nitric acid (4.3) up to 1 l with water.

5 Apparatus

The following equipment shall be used.

5.1 Forced-circulation drying oven, capable of being maintained at a temperature of $(30 \pm 2) \text{ }^\circ\text{C}$.

5.2 Pestle and mortar, made of porcelain or sintered corundum.

5.3 Screens, having an aperture size of 2 mm for air-dried samples and of 5 mm to 8 mm for freshly collected samples, and which do not cause contamination by the elements to be determined.

5.4 Balance, of accuracy 0,01 g.

5.5 Conical test tubes with screw caps, of nominal capacity 50 ml, made of polypropylene (or high density polyethylene, polytetrafluoroethylene, perfluoroalkoxyalkane, or tetrafluoroethylene/hexafluoropropylene copolymer). Before use, check cleanliness of tubes and caps. If cleaning is necessary, fill the test tubes with dilute nitric acid (4.4) and allow to stand for 24 h, then wash with water and dry.

5.6 End-over-end shaker, capable of $(20 \pm 2) \text{ r/min}$, placed in a thermostatically controlled room $[(20 \pm 2) \text{ }^\circ\text{C}]$.

5.7 In-line membrane filter, for connection with a disposable syringe.

Connect an in-line membrane filter of pore size $0,45 \text{ }\mu\text{m}$ to a disposable syringe with Luer-lock joint. If necessary, clean the membrane filter and the syringe by rinsing with dilute nitric acid (4.4).

If colloids are present in the filtrate, use a $0,2 \text{ }\mu\text{m}$ membrane filter. High density filter papers can be used if they are tested and proved to be free of the elements to be determined.

5.8 Disposable plastic syringe, of nominal volume 10 ml or 20 ml, with Luer-lock joint.

5.9 Centrifuge, able to centrifuge the test tubes (5.5) at 1 000 g to 3 000 g.

6 Procedure

6.1 Sample preparation

The soil sample should be pretreated in accordance with ISO 11464. Use the soil fraction with grain size $< 2 \text{ mm}$. Extract soil samples from organic horizons (having more than 30 % by mass of organic matter, such as bog soil or topsoil and samples from reductomorphic horizons — horizons affected by backwater or ground water such as coastal marsh soil or gley soil) in the freshly collected state and homogenize as described in Annex C.

6.2 Determining water content

Determine the water content in accordance with ISO 11465.

6.3 Extraction

Extract the soil at $(20 \pm 2) ^\circ\text{C}$ with 1 mol/l NH_4NO_3 solution, using a mass-to-volume ratio of 1:2,5.

Using a pipette, add 25,0 ml of ammonium nitrate solution (4.2) to $(10,00 \pm 0,01)$ g of air-dried or freshly collected soil (6.1) into a 50 ml conical test tube with screw caps (5.5). Close the test tube with a screw cap, mount it to an end-over-end shaker (5.6) and extract the soil at (20 ± 2) r/min for (120 ± 5) min.

See Annex C for instructions on extracting organic or reductomorphic horizons.

6.4 Phase separation

Place the closed test tubes into a centrifuge and separate the solid phase by centrifugation with about 1 000 *g* for 10 min. Rinse the membrane filter and the syringe with 1 ml of extract solution and discard the rinsing solution. Filter 10 ml of the supernatant solution directly from the sample test tube into a 10 ml syringe equipped with a 0,45 μm disposable in-line membrane filter. Store the filtered ammonium nitrate extract in a clean conical test tube.

6.5 Stabilization of extract

After filtration, stabilize the extract with nitric acid (4.3) by adding about 1 % of its volume of nitric acid (e.g. 0,1 ml of nitric acid to 10 ml of extract).

This can also be done in connection with a dilution step (especially necessary if ICP techniques are applied for analysis). Transfer a fixed volume of the filtrate (e.g. 10,0 ml) using a pipette into a 50 ml volumetric flask, add 0,5 ml nitric acid (4.3) and fill up to the mark with water. Dilutions can be made also by mass.

6.6 Blank test

Subject at least one blank to the same extraction procedure.

7 Calculation

After subtracting the blank concentration of an element from the concentration of the sample solution, calculate the extractable element fraction, w_x , expressed in $\mu\text{g}/\text{kg}$ of dry matter, determined in accordance with ISO 11465. For air-dried soil samples, use Equation (1) and for wet, freshly collected samples use Equation (2)

$$\text{air-dried sample: } w_x = \frac{\rho_B V F (100 + w_{\text{H}_2\text{O}})}{m_0 \times 100} \quad (1)$$

$$\text{wet, fresh sample: } w_x = \rho_B F \left[\frac{V}{m_d} + \frac{w_{\text{H}_2\text{O}}}{\rho_{\text{H}_2\text{O}} \times 100} \right] \quad (2)$$

$$\text{with water content, in \%: } w_{\text{H}_2\text{O}} = \frac{m_0 - m_d}{m_d} \times 100 \quad (3)$$

where

w_x is the ammonium nitrate extractable mass fraction of an element (x) in soil, in $\mu\text{g}/\text{kg}$;

ρ_B is the blank-corrected concentration of the element in the analysis solution, in $\mu\text{g}/\text{l}$;

V is the volume of ammonium nitrate solution added to the soil sample, in litres (nominal 0,025 l);