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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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Foreword

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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.

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.

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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 $mS \cdot m^{-1}$ equivalent to resistivity greater than 0,01 $M\Omega \cdot m$ 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 $M\Omega \cdot m$.

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4.2 Ammonium nitrate solution, $c_{NH_4NO_3} = 1$ mol/l.

Dissolve 80,04 g of ammonium nitrate ($w_{NH_4NO_3} > 99$ %, 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, $w_{HNO_3} = 65$ %, $\rho_{HNO_3} \approx 1,4$ 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 ± 2) °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 ± 2) r/min, placed in a thermostatically controlled room [(20 ± 2) °C].

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

Connect an in-line membrane filter of pore size 0,45 µm 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 µm 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.

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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 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.

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6.3 Extraction

Extract the soil at (20 ± 2) °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 ± 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 µg/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)

$$\text{air-dried sample:}\quad w_x = \frac{\rho_B\, V\, F\left(100 + w_{H_2O}\right)}{m_0 \times 100} \tag{1}$$

$$\text{wet, fresh sample:}\quad w_x = \rho_B\, F\left[\frac{V}{m_d} + \frac{w_{H_2O}}{\rho_{H_2O} \times 100}\right] \tag{2}$$

$$\text{with water content, in \%:}\quad w_{H_2O} = \frac{m_0 - m_d}{m_d} \times 100 \tag{3}$$

where

w_x is the ammonium nitrate extractable mass fraction of an element (x) in soil, in µg/kg;

ρ_B is the blank-corrected concentration of the element in the analysis solution, in µg/l;

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

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F dilution factor, V_{end}/V_i: V_i is the volume of the aliquot take of NH_4NO_3 solution, diluted to volume V_{end} for use in the analysis;

m_0 is the initial mass of the air-dried or freshly collected soil, in kg;

m_d is the mass of soil, dried in accordance with ISO 11465, in kg;

w_{H_2O} is the percentage of water in the soil sample, determined in accordance with ISO 11465;

ρ_{H_2O} is the density of water, usually taken as 1 kg/l.

Additionally, the concentration of the extracted element in the dry residue, ρ_M, in µg/dm^3, determined in accordance with ISO 11465, can be calculated using Equation (4):

$$\rho_M = w_x \ \rho_d \tag{4}$$

where ρ_d is the bulk density of the dry soil, determined in a separate analysis, in kg/dm^3.

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8 Expression of results

Report the mass fraction of an element extracted with ammonium nitrate solution from soil, w_x, in µg/kg or µg/dm^3.

EXAMPLES w_{Pb} = 20,3 µg/kg

w_{Cd} = 1,3 µg/kg

The number of significant places reported generally depends on the precision of the method used, or on the limits of determination. As a rule the contents are reported to three significant places, but to only two or even one in the vicinity of the limit of determination.

Precision data are reported in Annex B.

9 Test report

The test report shall refer to this International Standard and include the following details:

a) description of soil sample;

b) details of sample pretreatment, indicating whether the sample was air-dried or was freshly collected from the field;

c) any details not specified in this International Standard or regarded as optional, and any factors that may have affected the result.

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Annex A
(informative)

Information on method of determining selected elements and treatment of particular types of soil

A.1 Test procedure

Concentrations listed in Table A.1 are within the ranges of concentration frequently encountered in soil, critical concentrations being higher.

Table A.1 — Trace element concentrations and mass fractions of soil frequently encountered in ammonium nitrate solutions

Element	Concentrations in extracts of 1 mol/l NH_4NO_3 solution µg/l	Mass fractions from NH_4NO_3 extraction of soil µg/kg
Arsenic	10	25
Antimony	10	25
Beryllium	1	2,5
Cadmium	2	5
Chromium	4	10
Cobalt	20	50
Copper	100	250
Lead	8	20
Manganese	2 000	5 000
Mercury	< 0,4	< 1
Molybdenum	10	25
Nickel	100	250
Uranium	1	2,5
Vanadium	10	25
Zinc	100	250

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A.2 Analytical methods

To determine the element fraction extractable with 1 mol/l ammonium nitrate solution, the extract solution should be analysed with a sufficiently sensitive analytical method capable of determining concentrations in the µg/l region. Use atomic absorption methods described in ISO 11047 and ISO 20280, inductively coupled plasma atomic emission spectrometry in ISO 22036 or inductively coupled plasma mass spectrometry in ISO 17294-2 or any other relevant technique. Carefully designed temperature programmes for NH_4NO_3 destruction above 300 °C in connection with appropriate matrix modifiers should be used with electrothermal atomic absorption spectrometry determination. Matrix matching should be applied in calibration solutions, use of internal standards, dilution of extracts to reduce matrix load when using inductively coupled plasma methods to determine trace element concentrations in NH_4NO_3 extracts. Common techniques should be used for trace element determinations to avoid contamination. Non-neglectable blank concentrations can always be expected, which need to be taken into account.

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Annex B
(informative)

Precision

An interlaboratory comparison on the determination of trace elements by extraction with ammonium nitrate solution yielded the results given in Table B.1. The test was carried out in accordance with the ISO 5725 series, using the analytical methods described in A.2. The interlaboratory comparison was conducted in 2007. Four samples were prepared and distributed to 14 participating laboratories. Each soil sample was extracted twice and two blanks of the 1 mol/l ammonium nitrate extraction solution used were sent to BAM for measurements.

Four polluted soil samples were prepared:

- **Sample A**: soil from INRA (France); real total (HF) contents: Zn = 3 960 mg/kg; Pb = 970 mg/kg and Cd = 20 mg/kg.
- **Sample B**: soil from VDLUFA (Germany).
- **Sample C**: soil from UKZUZ, NRL (Czech Republic); aqua regia extractable contents: As = 13 mg/kg; Hg = 0,8 mg/kg; Ni = 57 mg/kg; Cr = 51 mg/kg.
- **Sample D**: soil from UKZUZ, NRL (Czech Republic); aqua regia extractable contents: As = 8,5 mg/kg; Hg = 0,6 mg/kg; Ni = 30 mg/kg and Cr = 51 mg/kg.

The materials were sent to the laboratory of the Federal Institute for Materials Research and Testing (BAM), sieved over a 1 mm sieve, homogenized by the cross-riffling procedure and bottled with 80 g soil in brown glass bottles with screw caps.

Measurements were carried out using inductively coupled plasma mass spectrometry (ICP-MS), Agilent 7 500cs[1], quadrupole, collision cell with He or H_2 as collision gases, Babington nebuliser, Scott-type spray chamber, cooled to 2 °C by a Peltier element, quartz-shielded plasma torch with 15 l/min Ar, Pt-sample and skimmer cones.

Due to the high salt concentration of the 1 mol/l ammonium nitrate solutions (80 g/l), the samples could not be measured directly with ICP-MS. It was necessary to dilute the samples 1:10 with 1 % nitric acid and add internal standards Ge and Ho to achieve concentrations of 10 µg/l. To 1,0 ml sample, 100 µl internal standard solution and 8,9 ml nitric acid (1 %) were added using a Hamilton Microlab[1] diluter.

Samples A and B were additionally diluted 1:100 or 1:1 000 for determination of high Zn and Pb contents. All blank solutions were diluted 1:10 before measurements as described above. The stability of low blank concentrations in a laboratory is a prerequisite for the trace element determinations.

Chromium in samples A, B and D and mercury in all samples could not be validated, due to very low extractable contents.

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[1] These proprietary names are given for the convenience of users of this International Standard and do not constitute endorsements of these products by ISO.

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Table B.1 — Results of an interlaboratory comparison based on ISO 19730

Sample	**Element**	l	n	x μg/kg	S_R μg/kg	S_r μg/kg	CV(R) %	CV(r) %	R μ/kg	r μg/kg
A	Sb	12	24	18,57	0,569	0,195	3,07	1,05	1,58	0,54
B	Sb	12	24	11,75	0,491	0,099	4,18	0,84	1,36	0,28
C	Sb	13	26	9,15	1,15	0,117	12,57	1,28	3,19	0,32
D	Sb	11	22	13,19	0,912	0,174	6,91	1,32	2,53	0,48
A	As	13	26	3,99	0,438	0,112	10,99	2,81	1,21	0,31
B	As	13	26	62,56	4,584	0,44	7,33	0,70	12,70	1,22
C	As	14	28	18,05	0,948	0,185	5,25	1,02	2,63	0,51
D	As	13	26	26,49	1,189	1,061	4,49	4,01	3,29	2,94
A	Cd	13	26	5 605,15	222,38	67,629	3,97	1,21	615,99	187,33
B	Cd	11	22	138,76	7,562	0,878	5,45	0,63	20,95	2,43
C	Cd	11	22	75,39	4,423	0,639	5,87	0,85	12,25	1,77
D	Cd	10	20	16,08	0,929	0,235	5,78	1,46	2,57	0,65
A	Co	14	28	175,82	9,013	3,75	5,13	2,13	24,97	10,39
B	Co	13	26	44,29	2,011	0,405	4,54	0,92	5,57	1,12
C	Co	13	26	45,62	1,19	0,47	2,61	1,03	3,30	1,30
D	Co	13	26	13,89	0,914	0,615	6,58	4,42	2,53	1,70
C	Cr	14	28	11,68	2,925	1,529	25,03	13,09	8,10	4,24
A	Cu	12	24	250,18	25,969	3,543	10,38	1,42	71,94	9,82
B	Cu	13	26	287,03	25,556	2,713	8,90	0,95	70,79	7,52
C	Cu	13	26	384,41	45,203	6,095	11,76	1,59	125,21	16,88
D	Cu	13	26	587,75	48,057	23,773	8,18	4,04	133,12	65,85
A	Ni	13	26	905,7	40,74	16,18	4,50	1,79	112,85	44,82
B	Ni	14	28	211,0	16,08	2,11	7,62	1,00	44,55	5,85
C	Ni	13	26	999,2	45,66	7,52	4,57	0,75	126,47	20,83
D	Ni	13	26	141,6	9,49	6,091	6,70	4,30	26,29	16,87
A	Pb	14	28	13 302,4	665,702	205,771	5,00	1,55	1 844,00	569,99
B	Pb	14	28	74,14	16,667	6,454	22,48	8,70	46,17	17,88
A	Se	14	28	1,80	0,10	0,086	5,82	4,78	0,29	0,24
B	Se	14	28	4,16	0,21	0,105	5,02	2,54	0,58	0,29
C	Se	14	28	6,21	0,42	0,204	6,81	3,29	1,17	0,57
D	Se	14	28	4,79	0,41	0,081	8,59	1,69	1,14	0,22

l is the number of laboratories after elimination of outliers;
n is the number of results;
x is the mean value, in micrograms per kilogram;
S_r is the repeatability standard deviation, in micrograms per kilogram;
CV(r) is the coefficient of variation of repeatability, in percent;
S_R is the reproducibility standard deviation, in micrograms per kilogram;
CV(R) is the coefficient of variation of reproducibility, in percent;
R is the reproducibility limit (2,77);
r is the repeatability limit (2,77).

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Annex C
(informative)

Notes on homogenization, initial sample mass and extraction

C.1 Homogenization of freshly collected samples

Depending on their cohesion, freshly collected samples should be forced by hand, using gloves, through a 2 mm, 5 mm or 8 mm screen. For samples of mineral soils, particles exceeding about 2 mm in diameter can be picked out by hand. If homogenization is inadequate, larger sample masses may be extracted (e.g. 100 g of soil with 250 ml of ammonium nitrate solution), but the ratio of air-dried or freshly collected soil to solution should be kept constant in order to obtain reproducible results.

C.2 Extraction of organic horizons

Samples from organic horizons shall be weighed out in the freshly collected state, since dried samples are frequently hydrophobic or absorb only some of the ammonium nitrate solution. The mixing of the sample can be improved by increasing the amounts extracted (e.g. 40 g of soil with 100 ml of ammonium nitrate solution) and adding glass beads (e.g. 20 g of beads with a diameter of 3 mm). The ratio of air-dried or freshly collected soil to solution should be kept constant in order to obtain reproducible results.

C.3 Extraction of reductomorphic horizons

Samples from reductomorphic horizons should be processed with oxygen excluded, from the beginning of sampling to the extraction. Extraction should be carried out as soon as possible after sample preparation (in particular the reduction in size of large aggregates).

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