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BSI Standards Publication

Soil quality — Extraction of trace elements using dilute nitric acid

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National foreword

This British Standard is the UK implementation of ISO 17586:2016.

The UK participation in its preparation was entrusted to Technical Committee EH/4, Soil quality.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Soil quality — Extraction of trace elements using dilute nitric acid

Qualité du sol — Extraction d'éléments traces à l'aide d'acide nitrique dilué

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

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

Soil quality — Extraction of trace elements using dilute nitric acid

WARNING — Users of this International Standard should be familiar with usual laboratory practice. This International Standard does not 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 according to this International Standard be carried out by suitably trained staff.

1 Scope

This International Standard specifies a method of extracting trace elements from soil at approximately pH 0,5 using a dilute nitric acid solution. Using this method the potential environmental available trace elements as defined in ISO 17402 is extracted.

The method is applicable for all soils and soil like materials.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
test portion
analytical portion
quantity of material, of proper size, for measurement of the concentration or other property of interest, removed from the test sample

Note 1 to entry: The test portion may be taken from the field sample or from the laboratory sample directly if no preparation of the sample is required (e.g. with liquids), but usually it is taken from the prepared test sample.

Note 2 to entry: A unit or increment of proper homogeneity, size, and fineness, needing no further preparation, may be a test portion.

[SOURCE: ISO 11074:2005, 4.3.13]

4 Principle

The soil sample with particle size of <2 mm is extracted with (0,43 ± 0,02) mol/l nitric acid solution at a soil:solution ratio of 1:10 (m/V) for four hours at (20 ± 2) °C. After centrifugation of the suspension, the liquid phase is filtered through an in-line membrane filter. The solution is then ready for the determination of elements 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 sensitive technique.

The pH after extraction is usually in the range of pH 0,5 to 1. For highly calcareous soils, the final pH shall be less than 1,0. In this case, the pH should be adjusted accordingly or the method may not work appropriately.

NOTE 2 The range of pH 0,5 to 1 is required to extract the potential environmental availability of trace elements as defined in ISO 17402.

5 Interferences

Sulfide minerals will interfere, e.g. if pyrite in soil or sediment change from anaerobic to aerobic conditions and hydrogen sulfide is evolved by contact with nitric acid. Heavy metal sulfides will precipitate.

The extraction is directly applicable to all types of soil, with less than 5 % carbonate. In order to ensure that soils with more carbonate are extracted at the proper pH, the amount of acid neutralised by the soil carbonate has to be accounted by adding extra acid.

6 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.

6.1 Water.

Use only water complying with the requirements for ISO 3696 grade 2 water (electrical conductivity less than 0,1 mS m^{-1} equivalent to resistivity greater than 0,01 MΩ 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 MΩ m.

6.2 Nitric acid, pro analysis, $w(HNO_3)$ = 63 % to 68 %.

6.3 Nitric acid, diluted, 5 mol/l.

Put 350 ml of nitric acid (6.2) into 500 ml of water, cool to room temperature and fill up in a volumetric flask to 1 l with water.

6.4 Nitric acid, diluted, 0,43 mol/l.

Dilute 30 ml of nitric acid (65 %) to 1 l water (6.1). Add nitric acid (6.2) to the water.

NOTE Using another percentage of nitric acid (6.2), adjust the amount of nitric acid to obtain 0,43 ± 0,02 mol/l.

7 Apparatus

All glassware used shall be thoroughly cleaned, e.g. with 5 % nitric acid to remove contaminants.

7.1 Balance, accuracy 0,01 g.

7.2 Conical test tubes with screwing caps, of nominal capacity 150 ml to 200 ml, made of polypropylene or another suitable material. Before use check cleanliness of tubes and caps.

7.3 Shaking machine, end-over-end shaker, capable for revolutions of (25 ± 10) per min, placed in a room with a constant temperature of (20 ± 2) °C.

7.4 Membrane filter for connection with a disposable syringe of 0,45 µm pore size.

7.5 Membrane filter for connection with a disposable syringe of 0,2 µm. If necessary, clean the membrane filter and the syringe by rinsing with 5 % nitric acid.

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

7.7 Centrifuge, capable to centrifuge the test tubes (7.2) at least at 1 000 *g*.

7.8 Crushing equipment: jaw crusher or cutting device.

NOTE Due to crushing, contamination of the sample may occur to an extent which affects the concentration of some constituents of concern e.g. cobalt and tungsten from tungsten carbide equipment or chromium, nickel and molybdenum from stainless steel equipment.

7.9 Sieving equipment, with 2 mm nominal screen size.

NOTE Due to sieving, contamination of the sample may occur to an extent which affects the concentration of some constituents of concern, e.g. chromium, nickel and molybdenum from stainless steel or copper and zinc from brass equipment.

8 Procedure

8.1 Sample preparation

The method can be applied to wet field samples (see also B.2) and samples pre-treated according to ISO 11464. The pre-treated test sample shall have a grain size less than or equal to 2 mm. Oversized material which is not of natural origin shall be removed and if the fraction > 2 mm exceeds 5 % (mass fraction), the entire oversized fraction shall be separated by sieving (7.9) and crushed with suitable crushing equipment (7.8) to grain size <2 mm.

The drying temperature shall not exceed 40 °C.

On no account the material shall be finely ground.

8.2 Determining water content

Determine the water content as specified in ISO 11465 using a separate test sample portion.

8.3 Extraction

8.3.1 Using a pipette or dispenser, add 100,0 ml of (0,43 ± 0,02) mol/l nitric acid solution (6.4) to (10 ± 0,1) g of the soil test portion (8.1) into a suitable test tube (7.2).

8.3.2 Add, with a pipette, drop by drop, 0,50 ml of 5 mol/l nitric acid (6.4) for each mass fraction of calcium carbonate in percent which is above 5 %.

8.3.3 Close the test tube with a screw cap, mount it to an end-over-end shaker (7.3) and extract the soil at room temperature (20 ± 2) °C and (25 ± 10) revolutions per min for (240 ± 10) min.

NOTE After the addition of nitric acid, in case of effervescence, allow the foaming to stop; then continue.

8.4 Phase separation

8.4.1 Place the closed test tubes into a centrifuge and separate the solid phase by centrifugation at 1 000 *g* or higher for 10 min.

8.4.2 Rinse the membrane filter and the syringe with at least 1 ml of extract solution and discard the rinsing solution.

8.4.3 Filter a suitable amount for following analysis (e.g. 10 ml) of the supernatant solution directly from the sample test tube by pushing it through a syringe equipped with a 0,45 µm disposable in-line membrane filter.

8.4.4 Store the acid extract in a clean conical test tube.

NOTE Because the extract is already acid, extra addition for stabilization is not necessary.

8.5 Blank test

Subject at least one blank to the same extraction procedure.

8.6 Measurement of trace elements

To determine the element fraction extracted with 0,43 mol/l nitric acid solution, the extract solution should be analysed with a sufficiently sensitive analytical method capable to determine concentrations down to the µg/l level. Use inductively coupled plasma atomic emission spectrometry described in ISO 22036 or inductively coupled plasma mass spectrometry in ISO 17294-2, atomic absorption methods in ISO 11047 or ISO 20280. Carefully designed temperature programmes in connection with appropriate matrix modifier should be used with ET-AAS determination. Apply matrix matching in calibration solutions. Always expect non-negligible blank concentrations which shall be taken into account.

9 Calculation

Subtract the blank concentration of an element from the concentration in the sample solution. Calculate the extractable element mass fraction according to Formula (1) for air-dried samples or Formula (2) for wet-field sample.

Air-dried sample:

$$w_{x_{\mathrm{air}}} = \frac{\rho_{\mathrm{B}} \cdot V \cdot F \cdot \left(100 + w_{\mathrm{H_2O}}\right)}{m_{\mathrm{o}} \cdot 100} \tag{1}$$

Wet-field sample. The equation corrects for the dilution by the water present in the sample:

$$w_{x_{\mathrm{wet}}} = \rho_{\mathrm{B}} \cdot F \cdot \left[\frac{V}{m_{\mathrm{d}}} + \frac{w_{\mathrm{H_2O}}}{\rho_{\mathrm{H_2O}} \cdot 100}\right] \tag{2}$$

Determine the water content according to ISO 11465 and calculate using Formula (3).

Water content in %:

$$w_{H_2O} = \frac{m_o - m_d}{m_d} \cdot 100 \tag{3}$$

where

$w_{x_{air}}$ or $w_{x_{wet}}$	is the nitric acid extractable mass fraction of an element (x) in soil, in micrograms per kilogram, µg/kg;
ρ_B	is the blank-corrected concentration of the element (x), in micrograms per litre, µg/l;
V	is the volume of nitric acid added to the soil sample, in litres (nominal 0,1 l);
F	is the dilution factor, V_{end}/V_i: V_i aliquot taken of nitric acid, diluted to volume V_{end} of analysis solution;
m_o	is the initial mass of air-dried or fresh-field sample, in kilograms, kg;
m_d	is the mass of soil, dried according to ISO 11465, in kilograms, kg;
w_{H_2O}	is the percentage of water in soil sample, determined according to ISO 11465;
ρ_{H_2O}	is the density of water, usually taken as 1 kg/l.

Additionally, the concentration of the extracted element ρ_M, in µg/dm^3, determined as specified in ISO 11465 may be calculated according to Formula (4).

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

where ρ_d is the bulk density of dry soil, determined in a separate analysis, in kilograms per cubic decimetre (kg/dm^3).

10 Expression of results

Report the results expressed in µg/kg.

EXAMPLE 1 w_{Pb} = 20,3 µg/kg.

EXAMPLE 2 w_{Cd} = 1,3 µg/kg.

The number of decimal places reported will generally depend on the precision of the analytical method used or on the limits of determination. As a rule, the contents should be reported with three significant decimal places, but only in two or even one in the vicinity of the limit of determination.

11 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard, i.e. ISO 17586;

b) the identification of the sample;

c) details of sample pre-treatment, indicating whether the sample was used as collected from the field (wet or air-dried);

d) the dry matter content according to ISO 11465;

e) the result of the determination of trace elements according to the analytical method used;

f) any details not specified in this standard or that are optional, and any circumstances that may have affected the result.

Annex A
(informative)

Precision

An inter-laboratory comparison on the determination of trace elements by extraction with diluted nitric acid according to this International Standard has been carried out in the beginning of 2015. The extracts were measured using the method applied in the individual laboratories. Most elements were analysed using ICP with AES or MS detection. One laboratory has used AAS including deuterium background correction and for arsenic the hydride technique. The final results are given in Tables A.2 and A.3. A more detailed report is available on www.wepal.nl.

The statistical test was carried out according to the ISO 5725 series. Six samples were used that are part of the WEPAL proficiency scheme and distributed to 17 participating laboratories. Each soil sample should be extracted four times according to ISO 17586 and measured according the analytical procedures available in the laboratory.

Six soil samples have been used with different soil properties (Table A.1) and content of trace elements (Tables A.2 and A.3):

Table A.1 — Consensus values of general soil parameters in the analysed soils

Parameter	**1**	**2**	**3**	**4**	**5**	**6**
Clay (%)	13,3	25,2	22,0	26,7	13,6	8,9
Organic matter (LOI) (%)	5,6	5,3	5,3	12,0	3,0	2,1
$CaCO_3$ (%)	9,0	8,4	2,8	8,4	0	1,8

All materials used were homogenized and grinded materials. They were sieved over 0,5 mm and distributed in pots to contain 100 g of soil. Before sending, it was checked if the soils were homogenous distributed in the pots using previous test results and specific analysis of total concentrations in a random selection of 10 pots. Participants have submitted their results electronically by www.wepal.nl.

Outliers were identified using Cochran's test and Grubb's test described in ISO 5725-2. Although the laboratories were asked to analyse four replicates, two laboratories measured two replicates and one laboratory analysed three replicates. The results of the validation are summarized in Tables A.2 and A.3. In these tables, we have added the total concentration measured using *aqua regia*. The concentration measured after extraction using ISO 17586 is a part of the total concentration.

Table A.2 describes the results with more than four participating laboratories. For most elements, the repeatability and reproducibility are as expected for such an analysis, and the method is validated for these elements (all elements in Table A.2 with exception of B, Mo and Se). Higher values are obtained for B, Mo and Se. For Mo and Se this is caused by the low concentrations and the higher values for B shows that the method is less suitable for this element.

Table A.3 summarizes the results for elements with four or less than four participating laboratories. Repeatability and reproducibility of the elements in Table A.3 are indicative. High values of repeatability and reproducibility are obtained for Sn.

For all elements presented in Tables A.2 and A.3, it has to be considered that they are the results of a chemical method and more precisely, the amount that can be extracted with a mild acid extraction (approximately pH 0,5). If this amount is representative for the bioavailable, fraction has to be proven using calibration studies (e.g. uptake by crops, effect on organisms, leaching to groundwater). The guideline on bioavailability, ISO 17402, stated that it is necessary to correlate the results of a chemical method and biologically measured effects.

Table A.2 — Results of validation for elements with more than four participating laboratories

Sample	Element	*Aqua regia* extractable amount mg/kg	l	n	Outliers %	x	S_R	S_r	VC_R	VC_r
1	Al[a]	9,87	5	20	0,0	0,640	0,045	0,025	7,0	3,9
2	Al[a]	18,9	5	20	0,0	0,940	0,040	0,026	4,2	2,7
3	Al[a]	19,7	5	20	0,0	1,32	0,05	0,02	3,6	1,6
4	Al[a]	26,4	5	20	0,0	2,29	0,18	0,05	7,8	2,4
5	Al[a]	13,6	5	20	20,0	0,849	0,086	0,010	10,2	1,2
6	Al[a]	9,15	5	20	0,0	0,481	0,053	0,015	11,1	3,1
1	As	15,8	16	61	6,25	6,68	0,91	0,32	13,7	4,8
2	As	10,4	16	61	0,0	4,19	0,48	0,16	11,4	3,9
3	As	9,83	16	61	25,0	0,963	0,155	0,025	16,1	2,6
4	As	42,4	16	61	12,5	19,11	1,57	0,49	8,2	2,5
5	As	7,63	15	57	13,3	0,421	0,081	0,013	19,3	3,0
6	As	3,09	15	57	13,3	0,611	0,055	0,033	9,0	5,5
1	B	24,0	7	25	14,3	6,51	2,49	0,34	38,3	5,3
2	B	28,3	7	25	28,6	5,05	1,95	0,13	38,7	2,6
3	B	17,1	7	25	28,6	2,73	0,96	0,07	35,4	2,7
4	B	73,0	7	25	14,3	4,54	1,82	0,24	40,0	5,3
5	B	9,45	5	17	20,0	0,511	0,107	0,073	21,0	14,3
6	B	10,3	6	21	33,3	1,26	0,15	0,08	11,7	6,3
1	Ba	108	13	47	23,1	25,2	1,5	1,0	6,0	4,0
2	Ba	40,9	12	46	8,3	15,9	0,9	0,3	5,5	2,1
3	Ba	86,2	13	47	15,4	51,6	2,8	0,8	5,4	1,6
4	Ba	826	13	47	7,7	345	34	10	9,9	2,8
5	Ba	76,3	12	43	16,7	48,6	2,4	0,8	5,0	1,6
6	Ba	52,3	12	43	8,3	32,5	2,5	0,9	7,7	2,7
1	Be	0,601	7	28	14,3	0,184	0,033	0,008	17,7	4,5
2	Be	0,882	8	32	12,5	0,271	0,034	0,006	12,6	2,2
3	Be	0,876	8	32	0,0	0,331	0,026	0,013	8,0	3,8
4	Be	1,58	8	32	0,0	0,689	0,078	0,027	11,3	3,9
5	Be	0,624	6	24	0,0	0,216	0,019	0,006	9,0	2,6
6	Be	0,538	6	24	0,0	0,115	0,009	0,004	7,5	3,8
1	Cd	1,28	17	63	11,8	1,14	0,06	0,04	5,5	3,3
2	Cd	0,222	15	57	13,3	0,180	0,043	0,006	23,8	3,3
3	Cd	0,343	16	61	0,0	0,264	0,042	0,007	15,9	2,8
4	Cd	8,35	17	63	0,0	7,44	1,06	0,13	14,2	1,8
5	Cd	0,635	16	59	12,5	0,531	0,054	0,008	10,1	1,6
6	Cd	0,178	14	53	14,3	0,138	0,011	0,008	7,7	5,9
1	Co	5,86	13	47	7,7	2,22	0,35	0,14	15,6	6,4
2	Co	7,15	13	47	7,7	2,48	0,27	0,06	10,9	2,5

Table A.2 *(continued)*

Sample	Element	*Aqua regia* extractable amount mg/kg	l	n	Outliers %	x	S_R	S_r	VC_R	VC_r
3	Co	8,86	13	47	15,4	1,81	0,30	0,03	16,3	1,5
4	Co	18,6	13	47	15,4	8,61	0,98	0,22	11,4	2,5
5	Co	7,72	12	43	25,0	2,26	0,19	0,08	8,4	3,4
6	Co	3,88	12	43	8,3	0,97	0,09	0,04	9,0	3,8
1	Cr	34,9	17	63	11,8	4,62	1,11	0,20	24,1	4,2
2	Cr	35,1	16	59	6,3	2,07	0,55	0,05	26,6	2,6
3	Cr	60,1	17	63	11,8	3,15	0,60	0,05	19,1	1,5
4	Cr	185	17	63	17,6	65,8	13,2	1,4	20,0	2,2
5	Cr	28,6	15	59	0,0	1,11	0,17	0,04	15,1	3,5
6	Cr	13,2	15	51	7,1	0,786	0,117	0,024	14,9	3,0
1	Cu	48,7	17	63	5,9	35,01	5,88	4,42	16,8	12,6
2	Cu	10,6	17	63	23,5	4,81	0,54	0,09	11,2	1,9
3	Cu	22,8	17	63	29,4	9,37	1,19	0,09	12,7	1,0
4	Cu	153	17	63	17,6	109,2	8,9	2,4	8,2	2,2
5	Cu	11,4	16	59	25,0	4,27	0,50	0,10	11,7	2,3
6	Cu	28,4	16	59	0,0	18,67	1,96	0,74	10,5	3,9
1	Fe[a]	22,4	6	22	33,3	5,74	1,28	0,12	22,2	2,1
2	Fe[a]	21,7	6	22	33,3	2,96	0,59	0,07	19,	2,2
3	Fe[a]	21,7	6	22	16,7	0,774	0,097	0,018	12,5	2,4
4	Fe[a]	35,7	6	22	0,0	5,94	1,13	0,21	19,1	3,6
5	Fe[a]	17,3	6	22	16,7	0,794	0,076	0,017	9,5	2,2
6	Fe[a]	9,14	6	22	0,0	0,397	0,085	0,015	21,3	3,8
1	Mn	239	7	26	14,3	160	24,0	5,05	15,1	3,2
2	Mn	470	7	26	0,0	338	59,9	14,2	17,7	4,2
3	Mn	1 010	7	26	14,3	557	57,4	16,6	10,3	3,0
4	Mn	1 040	7	26	0,0	763	141	25,2	18,5	3,3
5	Mn	554	7	26	14,3	249	19,4	8,6	7,8	3,5
6	Mn	304	7	26	0,0	184	18,3	6,41	9,9	3,5
1	Mo	1,23	9	36	22,2	0,096	0,042	0,010	44,1	10,4
2	Mo	0,322	9	36	11,1	0,023	0,017	0,003	72,3	11,9
3	Mo	1,02	9	36	11,1	0,013	0,006	0,002	49,0	16,2
4	Mo	1,41	9	36	0,0	0,077	0,030	0,007	38,8	8,7
5	Mo	0,720	7	28	0,0	0,009	0,003	0,003	38,8	32,5
6	Mo	0,222	7	27	28,6	0,011	0,002	0,001	15,3	14,1
1	Ni	16,8	17	62	23,5	4,57	0,67	0,15	14,7	3,3
2	Ni	21,4	17	62	17,6	4,06	0,62	0,09	15,3	2,2
3	Ni	40,4	17	62	17,6	4,57	0,75	0,10	16,4	2,2
4	Ni	55,0	17	62	23,5	17,2	1,6	0,3	9,1	1,9

Table A.2 *(continued)*

Sample	Element	*Aqua regia* extractable amount mg/kg	l	n	Outliers %	x	S_R	S_r	VC_R	VC_r
5	Ni	19,0	16	58	18,8	2,12	0,27	0,04	12,8	2,1
6	Ni	10,3	15	54	6,7	1,17	0,14	0,05	12,0	3,9
1	P	558	7	28	0	391	24	9	6,2	2,4
2	P	571	7	28	0,0	371	36	12	9,8	3,1
3	P	962	7	28	14,3	366	13	6	3,4	1,7
4	P	2 500	7	28	0,0	1623	61	27	3,8	1,7
5	P	368	7	28	0,0	50,4	5,3	1,4	10,5	2,7
6	P	573	7	28	0,0	412	37	18	9,0	4,3
1	Pb	239	17	63	5,9	218	23	9	10,5	4,1
2	Pb	31,4	17	63	23,5	25,1	4,2	0,7	16,9	2,8
3	Pb	25,9	17	63	5,9	15,7	2,9	0,7	18,6	4,6
4	Pb	285	17	63	29,4	240	14	4	6,0	1,5
5	Pb	30,5	16	59	12,5	17,7	2,3	0,4	12,8	2,4
6	Pb	18,5	16	58	12,5	12,3	1,3	0,9	10,8	7,3
1	Se	0,437	7	28	42,9	0,068 2	0,077 2	0,002 3	113,3	3,4
2	Se	0,275	7	28	28,6	0,059 9	0,061 1	0,002 7	102,0	4,4
3	Se	0,155	7	28	28,6	0,049 4	0,059 8	0,002 5	121,1	5,1
4	Se	1,78	7	28	28,6	0,249	0,205 5	0,006 0	82,5	2,4
5	Se	0,190	7	28	14,3	0,068 6	0,097 3	0,004 2	141,8	6,1
6	Se	0,270	6	24	33,3	0,005 7	0,004 2	0,003 0	73,9	52,3
1	Sr	120	8	30	0	109,9	4,6	1,9	4,2	1,7
2	Sr	89,4	8	30	0,0	77,8	4,3	1,3	5,6	1,6
3	Sr	22,7	8	30	0,0	16,2	0,7	0,4	4,1	2,2
4	Sr	133	8	30	12,5	106	5	2	4,6	1,5
5	Sr	15,8	7	26	0,0	6,05	0,35	0,12	5,9	2,0
6	Sr	26,7	7	26	14,3	22,5	1,8	1,1	8,1	5,1
1	Ti	235	5	20	40	10,61	1,59	0,11	15,0	1,0
2	Ti	346	5	20	20,0	5,48	0,66	0,14	12,1	2,5
3	Ti	251	4	16	0,0	1,15	0,07	0,04	6,4	3,3
4	Ti	416	5	20	0,0	29,8	5,3	0,8	17,7	2,7
5	Ti	375	5	20	20,0	2,53	0,18	0,04	7,2	1,7
6	Ti	-	5	20	0,0	3,11	0,18	0,12	5,7	3,8
1	V	31,2	10	40	0	9,63	1,62	0,33	16,8	3,4
2	V	35,8	10	40	0,0	8,45	1,37	0,24	16,3	2,9
3	V	54,6	10	40	10,0	16,0	2,7	0,3	16,8	2,0
4	V	56,1	10	40	0,0	16,0	3,2	0,3	19,9	2,1
5	V	28,1	8	32	0,0	2,52	0,44	0,07	17,3	2,8
6	V	15,4	9	36	0,0	2,73	0,28	0,12	10,3	4,4

Table A.2 *(continued)*

Sample	**Element**	***Aqua regia* extractable amount** mg/kg	***l***	***n***	**Outliers** %	***x***	S_R	S_r	VC_R	VC_r
1	Zn	378	17	63	11,8	282	22	9	7,9	3,3
2	Zn	83,0	17	63	17,6	33,0	3,8	0,7	11,6	2,3
3	Zn	67,8	17	63	17,6	12,6	1,8	0,2	14,3	1,6
4	Zn	1 030	17	63	11,8	624	90	13	14,4	2,1
5	Zn	97,1	16	59	6,3	29,4	4,1	1,1	14,1	3,8
6	Zn	63,8	16	59	0,0	29,8	3,1	1,4	10,4	4,6

Explanation of symbols

l is the number of laboratories after elimination of outliers;

n is the number of results;

x is the mean value, in milligrams per kilogram;

S_r is the repeatability standard deviation, in milligrams per kilogram;

VC_r is the relative repeatability standard deviation, in percent;

S_R is the reproducibility standard deviation, in milligrams per kilogram;

VC_R is the relative reproducibility standard deviation, in percent;

[a] Concentration in g/kg instead of mg/kg.

Table A.3 — Results of validation for elements with four or less than four participating laboratories (indicative values)

Sample	**Element**	***Aqua regia* extractable amount** mg/kg	***l***	***n***	**Outliers** %	***x***	S_R	S_r	VC_R	VC_r
1	Ca[a]	36,3	4	16	0,0	37,6	3,4	1,5	8,9	4,0
2	Ca[a]	35,1	4	16	0,0	35,0	2,6	1,4	7,3	4,0
3	Ca[a]	10,9	4	16	0,0	10,8	0,6	0,3	5,3	3,1
4	Ca[a]	36,2	4	16	25,0	36,4	2,6	0,7	7,1	1,9
5	Ca[a]	2,19	4	16	0,0	1,75	0,19	0,03	10,6	1,9
6	Ca[a]	8,27	4	16	0,0	8,32	1,16	0,50	13,9	6,0
1	K	2 770	4	16	0,0	277	24,5	6,27	8,8	2,3
2	K	4 710	4	16	25,0	412	41,6	8,34	10,1	2,0
3	K	3 730	4	16	0,0	340	22,9	9,63	6,7	2,8
4	K	5 570	4	16	0,0	276	16,3	10,2	5,9	3,7
5	K	2 200	4	16	25,0	98,9	7,09	1,97	7,2	2,0
6	K	3 190	4	16	0,0	279	19,5	10,4	7,0	3,7
1	Mg	3 800	4	16	0,0	1486	101	28,0	6,8	1,9
2	Mg	7 070	4	15	0,0	2 575	77,2	35,1	3,0	1,4
3	Mg	8 510	4	16	0,0	2 990	122	32,9	4,1	1,1
4	Mg	9 520	4	16	0,0	3 101	104	75,3	3,4	2,4
5	Mg	2 470	4	16	0,0	168	26,3	2,52	15,7	1,5

Table A.3 *(continued)*

Sample	Element	*Aqua regia* extractable amount mg/kg	*l*	*n*	Outliers %	*x*	s_R	s_r	VC_R	VC_r
6	Mg	4 050	4	16	0,0	299	55,4	13,7	18,5	4,6
1	Na	248	4	16	0,0	150	9,77	7,22	6,5	4,8
2	Na	181	4	16	0,0	70,0	3,95	1,34	5,6	1,9
3	Na	85,4	4	16	0,0	20,6	1,14	0,64	5,5	3,1
4	Na	324	4	16	0,0	132	5,44	3,93	4,1	3,0
5	Na	93,1	4	16	25,0	22,9	0,73	0,42	3,2	1,8
6	Na	105	4	16	25,0	22,1	1,91	1,99	8,6	9,0
1	Sb	2,14	4	16	25	0,122	0,006 9	0,003 4	5,6	2,8
2	Sb	0,332	4	16	0	0,020 8	0,003 2	0,001 0	15,2	4,7
3	Sb	0,575	4	16	0	0,033 8	0,001 8	0,001 1	5,2	3,3
4	Sb	3,03	4	16	0	0,23 3	0,037 3	0,007 2	16,0	3,1
5	Sb	0,430	4	16	0	0,023 5	0,001 3	0,001 2	5,4	4,9
6	Sb	0,391	4	16	0	0,030 7	0,002 5	0,002 7	8,3	8,8
1	Sn	14,4	5	18	0	1,08	0,35	0,30	32,4	28,0
2	Sn	1,88	3	12	0,0	0,020	0,004	0,002	21,9	8,1
3	Sn	2,1	2	8	0,0	0,016	0,008	0,005	48,1	31,8
4	Sn	23,7	5	18	20,0	2,21	0,69	0,03	31,3	1,3
5	Sn	1,07	2	8	0,0	0,014	0,009	0,003	62,1	22,9
6	Sn	1,78	3	12	0,0	0,016	0,008	0,004	49,0	22,6
For an explanation of symbols see Table A.2.										

Annex B
(informative)

Notes on homogenization, initial sample mass and extraction

B.1 Homogenization of freshly collected samples

Depending on their cohesion, freshly collected samples should be forced by hand through a 2 mm, 5 mm or 8 mm screen using gloves, 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 1 000 ml of extraction solution (0,43 mol/l nitric acid)), but the ratio of air-dried or freshly collected soil to solution shall be kept constant in order to obtain reproducible results,

B.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 extraction solution (0,43 mol/l nitric acid), The mixing of the sample can be improved by increasing the amounts extracted [e.g. 40 g of soil with 400 ml of extraction solution (0,43 mol/l nitric acid)] 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 shall be kept constant in order to obtain reproducible results.

B.3 Extraction of reductomorphic horizons

Samples from reductomorphic horizons shall 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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