

# DRAFT INTERNATIONAL STANDARD

## ISO/DIS 17586

ISO/TC 190/SC 3

Secretariat: DIN

Voting begins on:  
2013-09-04

Voting terminates on:  
2013-12-04

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## Soil quality — Extraction of trace elements using dilute nitric acid

*Qualité du sol —*

ICS: 13.080.10

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Reference number  
ISO/DIS 17586:2013(E)

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## Foreword

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ISO 17586 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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## Soil quality — Extraction of trace elements using dilute nitric acid

### 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.

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

### 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 3696:1991, *Water for analytical laboratory use — Specification and test methods*

ISO 11074, *Soil quality — Vocabulary*

ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analyses*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

### 3 Principle

The soil sample with particle size of < 2 mm is extracted with  $(0,43 \pm 0,02)$  mol/l nitric acid solution at a soil:solution ratio of 1:10 (*m/V*) for 120 min at  $(20 \pm 2)$  °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** 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, which is required to extract the potential environmental availability of metals as defined in ISO 17402. 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.

### 4 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.

## 5 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  $0,1 \text{ mS m}^{-1}$  equivalent to resistivity greater than  $0,01 \text{ M}\Omega \text{ m}$  at  $25 \text{ }^\circ\text{C}$ ). It is recommended that the water be obtained from a water purification system that delivers high purity water having a resistivity greater than  $0,18 \text{ M}\Omega \text{ m}$ .

**6.2 Nitric acid**,  $w(\text{HNO}_3) = 63 \text{ \%}$  to  $68 \text{ \%}$ .

**6.3 Nitric acid**, diluted,  $5 \text{ mol/l}$ .

Pour  $347 \text{ ml}$  of nitric acid (6.2) into  $500 \text{ ml}$  of water, cool to room temperature and fill up in a volumetric flask to  $1 \text{ litre}$  with water.

**6.4 Nitric acid**, diluted,  $0,5 \text{ mol/l}$ .

Pipette  $100,0 \text{ ml}$  nitric acid (6.3) into a volumetric flask and fill up to  $1 \text{ litre}$  with water (6.1).

## 6 Apparatus

All glassware used shall be thoroughly cleaned, e. g. with  $5 \text{ \%}$  nitric acid to remove contaminants.

**7.1 Balance**, accuracy  $0,01 \text{ g}$ .

**7.2 Conical test tubes with screwing caps**, of nominal capacity  $150 \text{ ml}$  to  $200 \text{ ml}$ , 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  $(25 \pm 10)$  per min, placed in a room with a constant temperature of  $(20 \pm 2) \text{ }^\circ\text{C}$ .

**7.4 In-line membrane filter for connection with a disposable syringe**

Connect an in-line membrane filter of  $0,45 \text{ }\mu\text{m}$  pore size with luer-lock joint to a disposable syringe. If necessary, clean the membrane filter and the syringe by rinsing with  $5 \text{ \%}$  nitric acid.

If the supernatant after filtration is turbid, repeat the filtration using a  $0,2 \text{ }\mu\text{m}$  membrane filter.

**7.5 Disposable plastic syringe**, of nominal volume  $10 \text{ ml}$  or  $20 \text{ ml}$  with luer-lock joint.

**7.6 Centrifuge**, capable to centrifuge the test tubes (7.2) at least at  $1\ 000 \text{ g}$ .

**7.7 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.

**7.8 Sieving equipment** with  $2 \text{ mm}$  or  $4 \text{ mm}$  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.

## 7 Procedure

### 7.1 Sample preparation

Pretreat the sample according to ISO 11464. The 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 (7.8) and crushed with suitable crushing equipment (7.7) to grain size < 2 mm.

Oversized material of natural origin (e. g. stones, pebbles, twigs) in the sample shall be removed, the total mass determined and then discarded. The oversized fraction, which has been reduced in a grain size < 2 mm, shall be mixed and homogenized with the test sample. If the laboratory sample cannot be sieved because of high water content, it is allowed, in this case only, to reduce the water content until the laboratory sample can be sieved.

The drying temperature shall not exceed 25 °C.

On no account the material shall be finely ground.

### 7.2 Determining water content

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

### 7.3 Extraction

Using a pipette or dispenser add 100,0 ml of  $(0,43 \pm 0,02)$  mol/l nitric acid solution (6.4) to  $(10 \pm 0,1)$  g of the soil test portion (8.1) into a suitable test tube (7.2). Add, with a pipette, 0,50 ml of 5 mol/l nitric acid (6.4) for each mass fraction of calcium carbonate in percent which is above 5 %. 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  $(20 \pm 2)$  °C and  $(25 \pm 10)$  revolutions per min for  $(120 \pm 5)$  min.

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

### 7.4 Phase separation

Place the closed test tubes into a centrifuge and separate the solid phase by centrifugation at 1 000 g or higher for 10 min. Rinse the membrane filter and the syringe with at least 1 ml of extract solution and discard the rinsing solution. 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. Store the acid extract in a clean conical test tube.

Note Because the extract is already acid, no extra addition for stabilization is necessary.

### 7.5 Blank test

Subject at least one blank to the same extraction procedure.

### 7.6 Measurement of trace elements

To determine the element fraction extracted with 0,5 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-neglectable blank concentrations which shall be taken into account.

## 8 Calculation

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

Air-dried sample:

$$w_{x_{\text{air}}} = \frac{\rho_B \cdot V \cdot F \cdot (100 + w_{\text{H}_2\text{O}})}{m_o \cdot 100} \quad (1)$$

Wet field sample:

$$w_{x_{\text{wet}}} = \rho_B \cdot F \cdot \left[ \frac{V}{m_d} + \frac{w_{\text{H}_2\text{O}}}{\rho_{\text{H}_2\text{O}} \cdot 100} \right] \quad (2)$$

Determine the dry matter fraction according to ISO 11465 and calculate according to Equation (3).

Water content in %:

$$w_{\text{H}_2\text{O}} = \frac{m_o - m_d}{m_d} \cdot 100 \quad (3)$$

Where:

$w_{x_{\text{air}}}$ or $w_{x_{\text{wet}}}$	is the nitric acid extractable mass fraction of an element (x) in soil, in micrograms per kilogram, $\mu\text{g}/\text{kg}$ ;
$\rho_B$	is the blank-corrected concentration of the element (x), in micrograms per litre, $\mu\text{g}/\text{l}$ ;
$V$	is the volume of nitric acid added to the soil sample, in litres (nominal 0,1 l);
$F$	is the dilution factor, $V_{\text{end}}/V_i$ ; $V_i$ aliquot taken of nitric acid, diluted to volume $V_{\text{end}}$ of analysis solution;
$m_o$	is the initial mass or air-dried or fresh-field sample, in kilograms, kg;
$m_d$	is the mass of soil, dried according to ISO 11465, in kilograms, kg;
$w_{\text{H}_2\text{O}}$	is the percentage of water in soil sample, determined according to ISO 11465;
$\rho_{\text{H}_2\text{O}}$	is the density of water, usually taken as 1 kg/l.

Additionally, the concentration of the extracted element  $\rho_M$ , in  $\mu\text{g}/\text{dm}^3$ , determined as specified in ISO 11465 may be calculated according to Equation (4).

$$\rho_M = w_x \cdot \rho_d \quad (4)$$

where

$\rho_d$	is the bulk density of dry soil, determined in a separate analysis, in kilograms per decimetre, $\text{kg}/\text{dm}^3$ .
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## 9 Expression of results

Report the results expressed in  $\mu\text{g}/\text{kg}$ .

EXAMPLE 1  $w_{\text{Pb}} = 20,3 \mu\text{g}/\text{kg}$ .

EXAMPLE 2  $w_{\text{Cd}} = 1,3 \mu\text{g}/\text{kg}$ .

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.

## 10 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard (ISO 17586);
- b) the identification of the sample;
- c) details of sample pretreatment, 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.