
**Soil quality — Extraction of trace elements
by buffered DTPA solution**

*Qualité du sol — Extraction des éléments en traces par une solution
tamponnée de DTPA*



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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14870 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Soil quality — Extraction of trace elements by buffered DTPA solution

1 Scope

This International Standard specifies a method for the extraction of trace elements in soil samples using a buffered diethylenetriaminepentaacetic acid (DTPA) solution. This method mainly applies to the estimation of the availability of copper, iron, manganese and zinc to plants which grow in the soils. It is preferably applicable to soils having a pH greater than 6. Potentially toxic elements such as cadmium, chromium, nickel and lead can also be determined in the extracts. In soils containing large amounts of one or more of these elements, the efficiency of the extraction of any of these elements can be lessened by exceeding the complexation capacity of DTPA.

This International Standard does not specify the methods of measurement of the trace elements in the extracts.

2 Normative references

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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

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 is extracted at a temperature of 20 °C, under precise shaking conditions and with a soil:solution extraction ratio of 1:2 (m/V), of the soluble species of trace elements in a mixed buffered solution (pH = 7,3) of triethanolamine ($0,1 \text{ mol}\cdot\text{l}^{-1}$) with calcium chloride ($0,01 \text{ mol}\cdot\text{l}^{-1}$) and diethylenetriaminepentaacetic acid (DTPA, $0,005 \text{ mol}\cdot\text{l}^{-1}$).

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

NOTE 2 Due to the relatively high extraction ratio, it may be difficult to obtain a sufficient amount of extract solution in case of soils with high absorbing capacities.

4 Reagents

Use only reagents of recognized analytical quality and distilled or deionized water of grade 2 in accordance with ISO 3696.

- 4.1 **Triethanolamine (TEA)**, $C_6H_{15}NO_3$.
- 4.2 **Diethylenetriaminepentaacetic acid (DTPA)**, $C_{14}H_{23}N_3O_{10}$.
- 4.3 **Calcium chloride dihydrate**, $CaCl_2 \cdot 2H_2O$.
- 4.4 **Hydrochloric acid**, HCl , $6 \text{ mol} \cdot \text{l}^{-1}$.
- 4.5 **Extraction solution**, $c_{TEA} = 0,1 \text{ mol} \cdot \text{l}^{-1}$, $c_{CaCl_2} = 0,01 \text{ mol} \cdot \text{l}^{-1}$; $c_{DTPA} = 0,005 \text{ mol} \cdot \text{l}^{-1}$.

Into a beaker transfer successively 14,92 g of TEA (4.1), 1,967 g of DTPA (4.2), 1,470 g of calcium chloride dihydrate (4.3). Add water and mix to dissolve all the salts completely. Dilute the solution with water up to approximately 800 ml. Adjust the pH to $7,3 \pm 0,2$, using hydrochloric acid (4.4). Transfer quantitatively into a 1 000 ml volumetric flask. Make up to 1 000 ml and homogenize. The extraction solution should be stored at $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ and not used until it has reached that temperature.

5 Apparatus and glassware

Rubber stoppers, which may contain trace elements, are prohibited. It is recommended to use plastics stoppers or any other stopper free of the trace elements to be determined.

All laboratory glassware and plasticsware shall be soaked in $4 \text{ mol} \cdot \text{l}^{-1} \text{ HNO}_3$ for at least 0,5 h, rinsed three times with distilled water and once with the extraction solution (4.5).

Make systematic controls and trials to ensure that the apparatus and glassware do not release the trace elements to be determined.

- 5.1 **Containers of capacity 100 ml**, that can be hermetically sealed.
- 5.2 **End-over-end shaker** with rotational speed $(30 \pm 3) \text{ r/min}$, placed in a thermostatted enclosure at $20 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$.
- 5.3 **Centrifuge**.
- 5.4 **Centrifuge tube**.
- 5.5 **Polyethylene bottles** that can be hermetically sealed.
- 5.6 **Membrane filters**, pore size $0,45 \text{ } \mu\text{m}$, free of the trace elements to be determined.
- 5.7 **pH meter**.
- 5.8 **Plastics spatula**.
- 5.9 **Pipette or automatic dispenser** capable of dispensing 20 ml.

6 Procedure

6.1 Laboratory sample

Use the fraction of air-dry soil samples pretreated in accordance with ISO 11464, which has passed through a 2 mm aperture sieve. Use a separate part of the sample to determine the water content in accordance with ISO 11465.

6.2 Extraction procedure

Using a plastics spatula (5.8), weigh 10,00 g of the laboratory sample (6.1) and transfer into a 100 ml container (5.1). Add 20,0 ml of the extraction solution (4.5) using the pipette or the dispenser (5.9). Close the container tightly and shake for 2 h at $20\text{ °C} \pm 2\text{ °C}$ using the shaker (5.2). Decant a portion of the extract into a centrifuge tube (5.4) and centrifuge for 10 min at 3 000 g. Filter the supernatant using a filter (5.6). After separation of the phases, collect the extract in a polyethylene bottle (5.5) and analyse within 48 h.

Carry out blank extractions with each batch of analyses, using the same procedure but without the soil sample.

7 Test report

The test report can optionally be written together with the report of the analytical measurement.

The test report for the extraction shall contain the following information:

- a) a reference to this International Standard;
- b) all information necessary for the complete identification of the sample;
- c) 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.

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