

**Markundersökningar – Extraktion och
bestämning av tallium med elektrotermisk
atomabsorptionspektrometri**
(ISO 20279:2005, IDT)

**Soil quality – Extraction of thallium and
determination by electrothermal atomic
absorption spectrometry**
(ISO 20279:2005, IDT)

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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.

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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 20279 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Soil quality — Extraction of thallium and determination by electrothermal atomic absorption spectrometry

WARNING — Thallium solutions are highly toxic. Appropriate measures shall be taken to avoid ingestion. Care should be taken in the disposal of these solutions.

Certain procedures, reagents and apparatus used in this International Standard pose potential hazards, especially in connection with concentrated acids, toxic solutions of thallium, and high-pressure gases. Users should ensure that they are familiar with the safety procedures necessary in such situations, and with any legal requirements (including waste disposal). If in any doubt, seek advice from the competent authorities.

1 Scope

This International Standard specifies methods for extraction of thallium from soil by nitric acid and hydrogen peroxide and its determination in the extract by electrothermal atomic absorption spectrometry (ETAAS).

NOTE The relevant working range of this method is approximately from 0,05 mg/kg to 100 mg/kg.

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

ASTM E11:2004, *Standard Specification for Wire Cloth and Sieves for Testing Purposes*

3 Principle

Thallium is extracted from soil by a mixture of nitric acid and hydrogen peroxide, and determined in solution by electrothermal atomic absorption spectrometry, in which discrete volumes of sample solution are dispensed into a graphite tube. By increasing the temperature of this tube step by step, the processes of drying, thermal decomposition of the matrix and thermal dissociation into free atoms occur. The resulting absorption signal should (under optimum conditions) be a sharp symmetrical peak with a height proportional to the element concentration in solution. See Table 1.

Table 1 — Measurement wavelength for thallium

Element	Wavelength nm	Spectral bandwidth nm	Measurement method
Thallium	276,8	0,2	ETAAS

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NOTE Thallium contents in non-polluted soils are low. Therefore, a highly sensitive analytical method like ETAAS is necessary for the determination of trace concentrations. Unfortunately, severe interferences in aqua regia extracts of soils are observed using ETAAS for thallium determination. Due to a high chloride concentration in aqua regia extracts, thallium will be volatilised and lost during the pretreatment step. Therefore, in this International Standard, a mixture of nitric acid and hydrogen peroxide will be used for the extraction of thallium from soil.

Aqua regia can be used with approximately the same extraction efficiency to extract thallium from soil. However, for the determination of the element in the extract solution, other analytical methods, like inductively coupled plasma optical emission spectrometry (ICP-OES) or inductively coupled plasma mass spectrometry (ICP-MS), shall be used.

4 Reagents

All reagents shall be of recognised analytical grade.

4.1 Water, demineralised or distilled, from an all-glass apparatus, conforming to Grade 2 of ISO 3696. The water used for blank determinations, and for preparing reagents and standard solutions, shall have element concentrations that are negligible compared with the lowest concentration to be determined in the sample solutions.

4.2 Nitric acid, $w(\text{HNO}_3) = 65 \%$; $\rho \sim 1,40 \text{ g/ml}$.

The same batch of nitric acid shall be used throughout the procedure.

4.3 Nitric acid, $c(\text{HNO}_3) = 0,5 \text{ mol/l}$.

Add 32 ml of nitric acid (4.2) to 500 ml of water in a 1 000 ml volumetric flask, fill to the mark with water and mix well.

4.4 Hydrochloric acid, $w(\text{HCl}) = 37 \%$; $\rho \sim 1,185 \text{ g/ml}$.

The same batch of hydrochloric acid shall be used throughout the procedure.

4.5 Hydrogen peroxide, $w(\text{H}_2\text{O}_2) = 30 \%$.

The same batch of hydrogen peroxide solution shall be used throughout the procedure.

4.6 Palladium/magnesium nitrate modifier solution.

Dissolve 0,3 g of palladium nitrate $[\text{Pd}(\text{NO}_3)_2]$ and 0,36 g of magnesium nitrate $[\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}]$ in a 100 ml volumetric flask, fill to the mark with 0,5 mol/l nitric acid (4.3) and mix.

Alternatively, prepare the palladium/magnesium nitrate modifier solution as follows. Dissolve 0,14 g of palladium powder in a 250 ml beaker with 3,5 ml of nitric acid (4.2), add 10 μl of hydrochloric acid (4.4), evaporate the solution to near dryness on a water bath or hot-plate, then add 0,36 g of magnesium nitrate $[\text{Mg}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}]$. Dissolve this solid residue in 50 ml of nitric acid (4.3), transfer the solution into a 100 ml volumetric flask, fill to the mark with nitric acid (4.3) and mix. 10 μl of this solution are equal to 14 μg of Pd and 36 μg of $\text{Mg}(\text{NO}_3)_2$.

4.7 Preparation of stock and standard solutions of individual elements.

Two sources of stock solutions are available:

- commercially available stock solutions;
- stock solutions prepared in the laboratory from pure elements or stoichiometrically defined salts or oxides.