
Kakovost tal - Določevanje živega srebra po razklopu v zlatotopki z atomsko absorpcijsko spektrometrijo s tehniko hladnih par (CV-AAS) ali atomsko fluorescentno spektrometrijo s tehniko hladnih par (CV-AFS)

Soil quality - Determination of mercury in aqua regia soil extracts with cold-vapour atomic absorption spectrometry or cold-vapour atomic fluorescence spectrometry

Qualité du sol - Dosage du mercure dans les extraits de sol à l'eau régale par spectrométrie d'absorption atomique de vapeur froide ou par spectrométrie de fluorescence atomique de vapeur froide

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INTERNATIONAL STANDARD

ISO 16772

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Soil quality — Determination of mercury in aqua regia soil extracts with cold- vapour atomic spectrometry or cold- vapour atomic fluorescence spectrometry

*Qualité du sol — Dosage du mercure dans les extraits de sol à l'eau
régale par spectrométrie d'absorption atomique de vapeur froide ou par
spectrométrie de fluorescence atomique de vapeur froide*

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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 16772 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

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Soil quality — Determination of mercury in aqua regia soil extracts with cold-vapour atomic spectrometry or cold-vapour atomic fluorescence spectrometry

WARNING — Mercury is highly toxic. Safety measures shall be taken in handling mercury and mercury solutions. Mercury compounds should not be introduced into the environment. The laboratory handling these compounds should be aware of the relevant international and national legislation regulating the handling of mercury and its compounds.

1 Scope

This International Standard specifies a method for the determination of mercury in an aqua regia extract of soil, obtained in accordance with ISO 11464 and ISO 11466, using cold-vapour atomic absorption spectrometry or cold-vapour atomic fluorescence spectrometry. The limit of determination of the method is at least 0,1 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 analysis*

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

ISO 11466:1995, *Soil quality — Extraction of trace elements soluble in aqua regia*

3 Principle

Mercury is reduced to the elemental state by tin(II) chloride solution and liberated from solution in a closed system. The mercury vapour passes through a cell positioned in the light path of an atomic absorption spectrometer. Its absorbance at a wavelength of 253,7 nm is measured.

The absorbance signal is a function of mercury concentration.

Alternatively, after the reduction step, the mercury vapour is injected into the cell of an atomic fluorescence spectrometer, where the mercury atoms are excited by radiation of a specific wavelength. The intensity of the fluorescence radiation is a function of mercury concentration.

NOTE Tin(II) chloride as a reduction substance is specified in this International Standard because sodium borohydride reduces many elements common in soil extract solutions to the elemental state, which cause matrix problems under particular circumstances.

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4 Reagents and gases

All reagents shall be of recognized analytical grade. Use deionized water or water distilled from an all-glass apparatus, complying with grade 2 as defined in ISO 3696. The water used for blank determinations, and for preparing reagents and standard solutions shall have a mercury concentration that is negligible compared with the lowest calibration concentration, e.g. 10 times of the determination limit of the method.

4.1 Hydrochloric acid, $w(\text{HCl}) = 37\%$; $c(\text{HCl}) \approx 12 \text{ mol/l}$, $\rho(\text{HCl}) \approx 1,18 \text{ g/ml}$.

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

4.2 Nitric acid, $w(\text{HNO}_3) = 65\%$, $c(\text{HNO}_3) \approx 14,5 \text{ mol/l}$, $\rho(\text{HNO}_3) \approx 1,40 \text{ g/ml}$.

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

When different batches of acids are used throughout the procedure, the blank shall be controlled for each batch.

4.3 Nitric acid, diluted solution (1+4), $c(\text{HNO}_3) \approx 4 \text{ mol/l}$.

Add slowly 250 ml of nitric acid (4.2) to 500 ml of water in a 1000 ml volumetric flask, mix and fill to the mark with water.

4.4 Aqua regia, diluted solution (1+9).

Add 21 ml hydrochloric acid (4.1) and 7 ml nitric acid (4.2) to 500 ml of water in a 1000 ml volumetric flask, mix and fill to the mark with water.

4.5 Tin(II) chloride solution, $\rho(\text{SnCl}_2 \cdot 2\text{H}_2\text{O}) = 100 \text{ g/l}$, $c(\text{Sn}) = 0,443 \text{ mol/l}$.

Dissolve 10 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 30 ml of hydrochloric acid (4.1), transfer to a 100 ml volumetric flask and fill to the mark with water. The blank concentration of mercury can be reduced by bubbling a stream of nitrogen through the solution for 30 min, if necessary. Prepare this solution on the day of use.

NOTE Other concentrations of tin(II) chloride may be necessary using other systems.

4.6 Mercury, stock solution corresponding to $\rho(\text{Hg}) = 1000 \text{ mg/l}$.

4.6.1 General

Two sources of stock solutions can be used:

- commercially available stock solutions (4.6.2);
- stock solutions prepared in the laboratory from elemental mercury (4.6.3).

4.6.2 Commercially available stock solutions

Certified commercial stock solutions should preferably be used.

Commercial as well as home-made stock solutions should be checked on a regular basis.

NOTE Commercially available stock solutions have the advantage that they limit the need to handle toxic mercury. However, special care needs to be taken that these solutions are supplied with a certified composition from a reputable source.

4.6.3 Stock solutions prepared in the laboratory from elemental mercury

In a beaker covered with a watch glass, dissolve $100 \text{ mg} \pm 0,4 \text{ mg}$ of mercury metal [minimum purity $w(\text{Hg}) = 99,99 \%$] with 17 ml nitric acid (4.2). Dilute with water, boil gently to expel nitrous oxides, cool and transfer quantitatively into a 100 ml volumetric flask and fill to the mark with water. This solution is stable for at least six months.

4.7 Mercury, standard solution corresponding to $\rho(\text{Hg}) = 20 \text{ mg/l}$.

Pipette 2 ml of the stock mercury solution (4.6) into a 100 ml volumetric flask, add 10 ml nitric acid (4.3), mix and fill to the mark with water.

4.8 Mercury, standard solution corresponding to $\rho(\text{Hg}) = 0,2 \text{ mg/l}$.

Pipette 1 ml of the standard mercury solution (4.7) into a 100 ml volumetric flask, add 10 ml of nitric acid (4.3), mix and fill to the mark with water. Prepare this solution on the day of use.

Low-level mercury standard solutions should be stored in suitable silica flasks or use PFA or FEP bottles as mercury vapour diffuses through low density polyethylene bottles.

4.9 Argon or nitrogen.

Argon or nitrogen with a purity of 99,99% should be used as carrier gases. For atomic fluorescence spectrometric techniques, argon is strongly recommended because the sensitivity is higher than with nitrogen.

5 Apparatus

5.1 Usual laboratory glassware.

All glassware or PFA, FEP bottles shall be carefully cleaned for determinations involving trace elements, e.g. by immersion in aqueous nitric acid solution of 5 % volume fraction for a minimum of 6 h, followed by rinsing with water before use. The nitric acid shall be replaced each week. Grade B volumetric glassware is adequate for this analysis (see ISO 648 and ISO 1042).

5.2 Atomic absorption spectrometer (AAS), equipped with a mercury hollow cathode or an electrodeless discharge lamp (which gives a greater light intensity) operated at a current recommended by the lamp and instrument manufacturer, and an automatic background correction device.

5.3 Atomic fluorescence spectrometer (AFS), equipped with a specific mercury lamp, a fixed 254 nm filter and a photomultiplier tube for the detection of fluorescence radiation.

Operate at a current recommended by the lamp and instrument manufacturer (see also EN 13506).

5.4 Cold-vapour generator, batch system or an automated flow injection analysis system (FIAS), adaptable either to the atomic absorption spectrometer (5.2) or to the atomic fluorescence spectrometer (5.3), according to the detection technique used for the determination of mercury.

A flow-controlled nitrogen or argon stream (4.9) is used as an inert carrier to transport mercury vapour into the cell. Time-controlled addition of tin(II)-chloride reducing solution (4.5) in combination with automatic start of the read signal of the spectrometer is required. Systems with a 10 cm or longer silica cell path length with silica windows adapted for atomic absorption spectrometry (5.2) are commercially available. In this case, the silica cell is electrically heated to a temperature of 50 °C to 100 °C to avoid condensation of water. For the atomic fluorescence spectrometer (5.3), condensation in the silica cell is avoided by inclusion of a high efficiency permeation drying tube in the detection system.

If an automated system is used, where the reaction occurs continuously (FIAS), the concentration of tin(II) chloride solution, reaction time and the gas-liquid separator configuration shall be optimized due to the slow reaction kinetics of the reducing system.