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**Soil quality — Determination of  
mercury — Cold vapour atomic  
fluorescence spectrometry (CVAFS)**

*Qualité du sol — Dosage du mercure — Spectrométrie de fluorescence  
atomique à vapeur froide (CVAFS)*

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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](http://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](http://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*.

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## Introduction

ISO/TS 16727 is based upon CEN/TS 16175-2, *Sludge, treated biowaste and soil — Determination of mercury — Part 2: Cold vapour atomic fluorescence spectrometry (CV-AFS)*, which was developed by CEN/TC 400, *Project Committee — Horizontal standards in the fields of sludge, biowaste and soil*.

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# Soil quality — Determination of mercury — Cold vapour atomic fluorescence spectrometry (CVAFS)

**WARNING** — Persons using this Technical Specification should be familiar with usual laboratory practice. This Technical Specification does not purport to 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 Technical Specification be carried out by suitably trained staff.

## 1 Scope

This Technical Specification specifies a method for the determination of mercury in *aqua regia* or nitric acid digests of sludge, treated biowaste and soil, obtained according to ISO 11466 or ISO 16729 using cold vapour atomic fluorescence spectrometry. The lower working range limit is 0,003 mg/kg (dry matter).

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

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

ISO 16729, *Soil quality — Digestion of nitric acid soluble fractions of elements*

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

## 3 Principle

Monovalent and divalent mercury is reduced to the elemental form by tin(II)-chloride solution or sodium borohydride in acid medium. Elemental mercury is stripped off from the solution in a closed system, by means of a stream of argon or nitrogen. 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, usually about 254 nm. The intensity of the fluorescence radiation is a function of mercury concentration. The concentrations are calculated using a calibration curve.

**NOTE** The matrix of the solution analysed is dominated by the acids used in the digestion step. Tin(II)-chloride as a reduction substance is recommended in this Technical Specification, because sodium borohydride reduces many elements commonly found in soil, sludge and waste extract solutions, to the elemental state, which may cause matrix problems under particular circumstances. However, it is still possible to use sodium borohydride as reduction agent. The concentration range 0,1 µg/l to 10 µg/l in the digested solution, corresponding to 0,003 µg/g to 0,3 µg/g of mercury, when a 3,0 g of sample has been digested, can be determined directly. Higher concentrations can be determined if the digested solution is diluted. Sensitivity can be increased by the amalgamation technique.

## 4 Interferences

The presence of water vapour or aerosol in the fluorescence cell may cause suppression due to quenching. Water vapour should be removed from the carrier gas stream using a hygroscopic membrane before entering the detector. The noble metals, such as gold and silver, amalgamate with mercury and,

therefore, may cause suppression. Also anions, for instance sulfide, iodide and bromide, which complex strongly with mercury, can cause suppression.

Less interferences arise from heavy metals when tin(II)chloride is used rather than sodium borohydride. When flow systems are used, interference effects due to heavy metals may be less than indicated in [Table 1](#).

**Table 1 — Tolerable concentrations of some matrix elements**

Element	Acceptable concentration
	mg/l
Cu(II)	500
Ni(II)	500
Ag(I)	1

## 5 Reagents

For the determination of mercury at trace and ultra-trace level, the reagents shall be of adequate purity. The concentration of mercury or interfering substances in the reagents and the water should be negligible compared to the lowest concentration to be determined.

**5.1 Water** quality 2 according to ISO 3696 for all sample preparations and dilutions.

**5.2 Hydrochloric acid**, HCl,  $\rho(\text{HCl}) \approx 1,17 \text{ g/ml}$ ,  $c(\text{HCl}) \approx 12 \text{ mol/l}$ ,  $w(\text{HCl}) \approx 370 \text{ g/kg}$ .

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

**5.3 Nitric acid**,  $\text{HNO}_3$ ,  $\rho(\text{HNO}_3) \approx 1,4 \text{ g/ml}$ ,  $c(\text{HNO}_3) \approx 15 \text{ mol/l}$ ,  $w(\text{HNO}_3) \approx 650 \text{ g/kg}$ .

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

**5.4 Nitric acid**, diluted solution (1 + 9).

Add 100 ml of nitric acid ([5.3](#)) to 500 ml of water in a 1 000 ml volumetric flask, mix and fill to the mark with water

**5.5 Nitric acid**, rinsing solution for glassware,  $\approx 2 \text{ mol/l}$ .

Add 150 ml of nitric acid ([5.3](#)) to about 500 ml of water, and dilute with water to 1 000 ml.

**5.6 Aqua regia**, diluted solution (1 + 9).

Add 21 ml of hydrochloric acid ([5.2](#)) and 7 ml of nitric acid ([5.3](#)) to 500 ml of water in a 1 000 ml volumetric flask, mix and fill to the mark with water.

**5.7 Tin(II)chloride solution**,  $\rho(\text{SnCl}_2 \cdot 2 \text{ H}_2 \text{ O}) = 100 \text{ g/l}$ .

Dissolve 10 g of  $\text{SnCl}_2 \cdot 2 \text{ H}_2 \text{ O}$  in 30 ml of hydrochloric acid ([5.2](#)), 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.

A solution of lower concentration, e.g. 0,5 g in 100 ml, may be used with flow systems. Prepare this latter solution freshly on the day of use from the more concentrated solution by diluting with water.



**5.8 Sodium borohydride solution, NaBH<sub>4</sub>, 30 g/l.**

1 g of sodium hydroxide, NaOH, is weighed into a 100 ml volumetric flask and dissolved in water. 3 g of sodium borohydride, NaBH<sub>4</sub>, is weighed and dissolved in the sodium hydroxide solution, then diluted to the mark with water.

**5.9 Mercury standard stock solution, 1 000 mg/l**

Use a commercially available quantitative stock solution with a mercury concentration of  $(1\ 000 \pm 2)$  mg/l. This solution is considered to be stable for at least one year, but in reference to guaranteed stability, see the recommendations of the manufacturer.

**5.10 Mercury, standard solution I, 100 mg/l.**

Pipette 10 ml of the stock mercury solution (5.9) into a 100 ml volumetric flask, add 10 ml of nitric acid (5.3), mix and fill to the mark with water.

This solution is stable for one month.

**5.11 Mercury, standard solution II, 1 mg/l.**

Pipette 1 ml of the mercury standard solution I (5.10) into a 100 ml volumetric flask, add 10 ml of nitric acid (5.3), mix and fill to the mark with water.

This solution is stable for 7 days.

**5.12 Mercury, standard solution III, 100 µg/l.**

Pipette 10 ml of the mercury standard solution II (5.11) into a 100 ml volumetric flask, add 10 ml of nitric acid (5.3), mix and fill to the mark with water.

This solution shall be freshly prepared on the day of use.

**5.13 Argon, with a purity of  $\geq 99,99\%$** **6 Apparatus****6.1 Usual laboratory apparatus**

All glassware shall be carefully cleaned as usual for low trace element determinations, e.g. by immersion in nitric acid rinsing solution (5.5) for a minimum of 6 h, followed by rinsing with water before use. The nitric acid shall be replaced each week.

**6.2 Atomic fluorescence spectrometer (AFS)**

Equipped with a specific Hg lamp, a fixed 254 nm filter, a photomultiplier tube for the detection of fluorescence radiation and a suitable software for processing the output signal. Operate at a current recommended by the lamp or the instrument manufacturer. The gas supply (argon or nitrogen) should be equipped with a two stage regulator. The use of a gas purifier consisting of activated carbon is recommended. Nitrogen gas will cause reduced sensitivity compared to argon.

**6.3 Automated sample introduction system**

Automated mercury flow systems (flow injection systems or continuous flow systems) are very common for atomic fluorescence spectrometry. They allow a concentration range lower by about one order of magnitude. Manually operated systems or semi-automated batch systems are adequate as well.