
Soil quality — Determination of trace elements in aqua regia and nitric acid digests — Graphite furnace atomic absorption spectrometry method (GFAAS)

Qualité du sol — Détermination des éléments en traces solubles dans l'eau régale et l'acide nitrique — Spectrométrie d'absorption atomique avec four graphite

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO/TS 17073:2013](https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013)

<https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013>



iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO/TS 17073:2013

<https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2013

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

	Page
Foreword.....	iv
Introduction.....	v
1 Scope	1
2 Normative references	1
3 Principle	1
4 Interferences and sources of errors	2
5 Reagents	2
6 Apparatus	4
6.1 Usual laboratory apparatus.....	4
6.2 Atomic absorption spectrometer.....	4
6.3 Automated sample introduction system.....	4
6.4 Graphite tubes.....	4
6.5 Chemical modification.....	4
7 Procedure	5
7.1 Graphite furnace programme.....	5
7.2 Test sample solution.....	5
7.3 Test blank solution.....	6
7.4 Determination.....	6
7.5 Calibration.....	6
8 Calculation	7
9 Expression of results	7
10 Test report	7
Annex A (informative) Examples of instrumental parameter settings	8
Bibliography	9

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).

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).

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*.

[ISO/TS 17073:2013](https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013)

<https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013>

Introduction

ISO/TS 17073 is based upon CEN/TS 16172 *Sludge, treated biowaste and soil — Determination of elements using graphite furnace atomic absorption spectrometry (GF-AAS)*, which was developed by CEN/TC 400, *Project Committee — Horizontal standards in the fields of sludge, biowaste and soil*.

This Technical Specification is applicable and validated for several types of matrices as indicated in [Table 1](#).

Table 1 — Matrices for which this Technical Specification is applicable and validated

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Compost Fresh compost
Soil	Agricultural soil

iTeh STANDARD PREVIEW (standards.iteh.ai)

[ISO/TS 17073:2013](#)

<https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO/TS 17073:2013

<https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013>

Soil quality — Determination of trace elements in aqua regia and nitric acid digests — Graphite furnace atomic absorption spectrometry method (GFAAS)

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 the determination of trace elements in *aqua regia* or nitric acid digests or other extraction procedures of sludge, treated biowaste and soil, using atomic absorption spectrometry with electrothermal atomization in a graphite furnace. The method is applicable for the determination of the following elements:

Arsenic (As), cadmium (Cd), cobalt (Co), lead (Pb), antimony (Sb), thallium (Tl), vanadium (V).

This method may be applied to other elements. The lower working range is approximately 0,01 mg/kg to 0,001 mg/kg, depending on the element to be determined.

2 Normative references

[ISO/TS 17073:2013](https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013)

<https://standards.iteh.ai/catalog/standards/sist/c4cb0929-0fc8-432e-9c22-5e858d73b6e7/iso-ts-17073-2013>

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

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 16965, *Soil quality — Determination of trace elements using inductively coupled plasma mass spectrometry (ICP-MS)*

3 Principle

Graphite furnace atomic absorption spectrometry (GFAAS) (also known as Electrothermal Atomic Absorption Spectrometry (ETAAS)), discrete sample aliquots are dispensed into a graphite tube (of which there are several types), which can be heated to over 2 500 °C very rapidly and in a controlled manner. By increasing the temperature stepwise, the processes of drying, thermal decomposition of the matrix and thermal dissociation into free atoms occurs. Atomic absorption spectrometry is based on the ability of free atoms to absorb light. A light source emits light specific for a certain element (or elements). When the light beam passes through the atom cloud in the heated graphite furnace, the light is selectively absorbed by atoms of the chosen element(s). The decrease in light intensity is measured with a detector at a specific wavelength. The concentration of an element in the sample is determined by comparing the absorbance of the sample with the absorbance of calibration solutions. The signal-peak produced is, under optimum conditions, sharp and symmetrical, and of narrow half-width. The peak area is for most elements proportional to the concentration of the element in solution. The measurements are made at the wavelengths given in [Table 2](#).

Table 2 — Wavelengths of the elements

Element	Wavelength nm
Arsenic	193,7
Cadmium	228,8
Cobalt	240,7
Lead	217,0
Antimony	217,6
Thallium	276,8
Vanadium	318,4

Zeeman background correction and platform use is common practice.

If necessary, interferences may be overcome by adding a matrix modifier to the samples before analysis, or by performing the calibration with standard addition technique.

The results are given as the mass of analyte (micrograms or milligrams) per kilogram of dried sample materials.

4 Interferences and sources of errors

The sample solutions prepared from digestion of samples may contain large amounts of substances that may affect the results. High concentrations of chloride may cause low results, because the volatility of many elements is increased and analyte loss may occur during the pyrolysis step. High chloride concentrations interfere with TI determinations and result in severe losses of the analyte, avoid *aqua regia* digestions and use only nitric acid digestion according to ISO 16729. Matrix effects may be overcome, partially or completely, by optimization of the temperature programme, the use of pyrolytically coated tubes or platforms, the use of chemical modifiers, the standard addition technique and the use of background correction.

5 Reagents

Use only reagents of recognized analytical grade and water grade 1 as specified in ISO 3696.

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

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

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

5.2 Nitric acid, HNO₃, $\rho(\text{HNO}_3) \sim 1,4 \text{ g/ml}$, $c(\text{HNO}_3) \sim 15 \text{ mol/l}$, $w(\text{HNO}_3) \sim 650 \text{ g/kg}$.

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

5.3 Nitric acid, diluted 1 + 3 (volume fraction).

Add 250 ml of nitric acid (5.2) to 500 ml of water in a 1 000 ml volumetric flask and fill to the mark with water.

5.4 Aqua regia, diluted 1 + 3 (volume fraction).

Dilute 210 ml of hydrochloric acid (5.1) and 70 ml of nitric acid (5.2) with about 500 ml of water in a 1 000 ml volumetric flask, and dilute to the mark.

5.5 Standard stock solutions

Both single-element standard stock solutions and multi-element standard stock solutions with concentration of 1 000 mg/l, stating the acid used and the preparation technique, are commercially available. These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

Alternatively, the standard stock solutions may be prepared as indicated in [Table A.1](#).

5.6 Standard solutions

Use the same acid as the digested samples when preparing the standard and the calibration solutions.

5.6.1 Standard solution corresponding to 10 mg/l of element

Pipette 10 ml of the actual standard stock solution ([5.5](#)) into a 1 000 ml volumetric flask. Add 20 ml of nitric acid ([5.3](#)) or 20 ml of *aqua regia* ([5.4](#)), fill to the mark with water and mix well.

5.6.2 Standard solution corresponding to 0,1 mg/l of element

Pipette 5 ml of the standard solution ([5.6.1](#)) into a 500 ml volumetric flask. Add 10 ml of nitric acid ([5.3](#)) or 10 ml of *aqua regia* ([5.4](#)), fill to the mark with water and mix well. Prepare this solution on the day of use.

5.7 Calibration solutions

Before each batch of determinations, prepare, from the standard solutions of each element ([5.6.1](#) or [5.6.2](#)), at least four calibration solutions covering the linear range of the calibration curve for the element to be determined. Use nitric acid ([5.3](#)) or *aqua regia* ([5.4](#)) to adapt the acid concentration in calibration solutions to digest sample solution used. Calibration solutions shall be prepared on the day of use. Use the set of standard solutions containing the same acid as the digested samples.

5.8 Calibration blank solutions

Prepare a calibration blank solution in the same way as the calibration solutions, but add no standard solution. Use a 100 ml volumetric flask. Use nitric acid ([5.3](#)) or *aqua regia* ([5.4](#)) to adapt the acid concentration to that in calibration solutions ([5.7](#)). Cool if necessary and dilute to volume with water.

5.9 Palladium nitrate/magnesium nitrate modifier

Dissolve 0,259 g of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 100 ml of water. Mix the palladium nitrate solution [10 g/l $\text{Pd}(\text{NO}_3)_2$] with twice the volume of magnesium nitrate solution. Observe the manufacturer's recommendations. 10 μl of the mixed solution is equal to 15 μg of Pd and 10 μg of $\text{Mg}(\text{NO}_3)_2$. $\text{Pd}(\text{NO}_3)_2$ solution is commercially available (10 g/l). Prepare the solution freshly every month. The palladium solution is stable for more than one month.

5.10 Magnesium nitrate modifier

Dissolve 0,865 g of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 100 ml of water. 10 μl of this solution is equal to 50 μg of $\text{Mg}(\text{NO}_3)_2$. Observe the manufacturer's recommendations.

5.11 Ammonium dihydrogen phosphate modifier

Dissolve 2,0 g of $\text{NH}_4\text{H}_2\text{PO}_4$ in 100 ml of water. 10 μl of this solution is equal to 200 μg of $\text{NH}_4\text{H}_2\text{PO}_4$. Observe the manufacturer's recommendations.