



SLOVENSKI STANDARD
SIST-TS CEN/TS 16172:2013
01-julij-2013

Blato, obdelani biološki odpadki in tla - Določevanje elementov z metodo atomske absorpcijske spektrometrije z grafitno pečjo (GF-AAS)

Sludge, treated biowaste and soil - Determination of elements using graphite furnace atomic absorption spectrometry (GF-AAS)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von Elementen mittels Graphitofen-Atomabsorptionsspektrometrie (GF-AAS)

Boue, biodéchets traités et sols - Détermination des éléments par spectrométrie d'absorption atomique avec four en graphite (GF-AAS)

<https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fc4b4e84/sist-ts-cen-ts-16172-2013>

Ta slovenski standard je istoveten z: CEN/TS 16172:2013

ICS:

13.030.20	Tekoči odpadki. Blato	Liquid wastes. Sludge
13.080.10	Kemijske značilnosti tal	Chemical characteristics of soils

SIST-TS CEN/TS 16172:2013

en,fr,de

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST-TS CEN/TS 16172:2013](https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fccb4b4e84/sist-ts-cen-ts-16172-2013)

<https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fccb4b4e84/sist-ts-cen-ts-16172-2013>

TECHNICAL SPECIFICATION
SPÉCIFICATION TECHNIQUE
TECHNISCHE SPEZIFIKATION

CEN/TS 16172

January 2013

ICS 13.080.10; 13.030.01

English Version

**Sludge, treated biowaste and soil - Determination of elements
using graphite furnace atomic absorption spectrometry (GF-
AAS)**

Boues, bio-déchets traités et sols - Détermination des
éléments par spectrométrie d'absorption atomique avec
four en graphite (GF-AAS)

Schlamm, behandelter Bioabfall und Boden - Bestimmung
von Elementen mittels Graphitrohrofen-
Atomabsorptionsspektrometrie (GF-AAS)

This Technical Specification (CEN/TS) was approved by CEN on 16 June 2012 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

<https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fcb4b4e84/sist-ts-cen-ts-16172-2013>



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents	Page
Foreword.....	3
Introduction	4
1 Scope	5
2 Normative references	5
3 Principle.....	5
4 Interferences and sources of error	6
5 Reagents.....	6
6 Apparatus	8
7 Procedure	9
7.1 Graphite furnace programme	9
7.2 Test solution.....	9
7.3 Test blank solution	10
7.4 Determination.....	10
7.5 Calibration	10
7.5.1 Standard calibration technique	10
7.5.2 Standard addition technique	10
8 Calculation.....	11
9 Expression of results	11
10 Test report	12
Annex A (informative) Examples of instrumental parameter settings.....	13
Bibliography.....	14

STANDARD PREVIEW
(standards.iteh.ai)

[SIST-TS CEN/TS 16172:2013](https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fcb4b4e84/sist-ts-cen-ts-16172-2013)

[https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-](https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fcb4b4e84/sist-ts-cen-ts-16172-2013)

[79fcb4b4e84/sist-ts-cen-ts-16172-2013](https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fcb4b4e84/sist-ts-cen-ts-16172-2013)

Foreword

This document (CEN/TS 16172:2013) has been prepared by Technical Committee CEN/TC 400 "Project Committee - Horizontal standards in the fields of sludge, biowaste and soil", the secretariat of which is held by DIN.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

(standards.iteh.ai)

SIST-TS CEN/TS 16172:2013

<https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fccb4b4e84/sist-ts-cen-ts-16172-2013>

Introduction

This Technical Specification is applicable for several types of matrices as indicated in Table 1.

Table 1 — Matrices for which this Technical Specification is applicable

Matrix	Materials tested
Sludge	Municipal sludge
Biowaste	Compost Fresh compost
Soil	Agricultural soil

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.

<https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fccb4b4e84/sist-ts-cen-ts-16172-2013>

1 Scope

This Technical Specification specifies the determination of trace elements in *aqua regia* or nitric acid digest solutions of sludge, treated biowaste and soil, using atomic absorption spectrometry with electrothermal atomisation 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 0,1 mg/kg to 0,01 mg/kg, depending on the element to be determined.

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.

EN 15934, *Sludge, treated biowaste, soil and waste — Calculation of dry matter fraction after determination of dry residue or water content*

EN 16173, *Sludge, treated biowaste and soil — Digestion of nitric acid soluble fractions of elements*

EN 16174, *Sludge, treated biowaste and soil — Digestion of aqua regia soluble fractions of elements*

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

3 Principle

[SIST-TS CEN/TS 16172:2013](https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fcb4b4e84/sist-ts-cen-ts-16172-2013)

[https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-](https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fcb4b4e84/sist-ts-cen-ts-16172-2013)

[79fcb4b4e84/sist-ts-cen-ts-16172-2013](https://standards.iteh.ai/catalog/standards/sist/c9a99a0e-fa1a-4a83-97d6-79fcb4b4e84/sist-ts-cen-ts-16172-2013)

In graphite furnace atomic absorption spectrometry (GF-AAS) (also known as electrothermal atomic absorption spectrometry, ET-AAS), 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 material.

4 Interferences and sources of error

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. This effect is particularly pronounced for thallium, so for determination of this analyte avoid *aqua regia* digestions and use only nitric acid digestion according to EN 16173. Matrix effects may be overcome, partially or completely, by optimisation of the temperature programme, the use of pyrolytically coated tubes or platforms, the use of chemical modifiers, the standard addition technique and/or the use of background correction.

5 Reagents

Use only reagents of recognised analytical grade and water grade 1 as specified in EN 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, $\rho(\text{HCl}) = 1,18 \text{ g/ml}$, $c(\text{HCl}) = 12 \text{ mol/l}$, $w(\text{HCl}) = 370 \text{ g/kg}$.

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

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

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

5.3 Nitric acid, diluted.

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.

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 a concentration of 1 000 mg/l, stating the acid used, 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 in the laboratory using pure metals or salts of known stoichiometry and purity.

5.6 Standard solutions

When preparing the standard and the calibration solutions, use the same acids as for the digestion procedures according to EN 16173 or EN 16174.

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 the digest sample solutions to be analysed. Calibration solutions shall be prepared on the day of use.

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

Use a commercially available palladium nitrate solution ($c(\text{Pd}(\text{NO}_3)_2) = 10 \text{ g/l}$). This solution is stable for more than one month.

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 with two times the volume of magnesium nitrate solution. 10 μl of the mixed solution is equal to 15 μg Pd and 10 μg $\text{Mg}(\text{NO}_3)_2$. Prepare the solution freshly every month.

5.10 Magnesium nitrate modifier

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