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Sludge, treated biowaste and soil – Determination of elements in aqua regia and nitric acid digests – Flame atomic absorption spectrometry method (FAAS)

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TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 16188

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English Version

Sludge, treated biowaste and soil - Determination of elements in aqua regia and nitric acid digests - Flame atomic absorption spectrometry method (FAAS)

Boues, biodéchets traités et sols - Détermination des éléments solubles dans l'eau régale et l'acide nitrique - Spectrométrie d'absorption atomique dans la flamme (SAA)

Schlamm, behandelter Bioabfall und Boden - Bestimmung von Elementen in Königswasser- und Salpetersäure-Aufschlusslösungen - Flammen-Atomabsorptionsspektrometrie (FAAS)

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Contents

Page

Foreword

This document (CEN/TS 16188:2012) 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.

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.

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.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, 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.

1 Scope

This Technical Specification specifies the determination of metals in *aqua regia* and nitric acid digests of sludge, treated biowaste and soil samples, using flame atomic absorption spectrometry. The method is applicable for the determination of the following elements:

Chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), manganese (Mn), nickel (Ni), zinc (Zn).

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 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

The method is based on the atomic absorption spectrometric measurement of the concentration of the elements in an *aqua regia* or nitric acid extract of the sample, prepared in accordance with EN 16173 or EN 16174, using the instrumental conditions given in Table 1.

Table 1 — General conditions for flame atomic absorption spectrometry

Element	Wavelength nm	Flame type	Lanthanum chloride	Main interference	Background correction
Chromium	357,9	reduced or neutral air/acetylene or acetylene/N_2O (recommended)	Yes No	Fe, Si	Deuterium
Cobalt	240,7	Oxidizing air/acetylene	No		Deuterium
Copper	324,8	Oxidizing air/acetylene	No		Deuterium
Iron	248,3	Oxidizing air/acetylene	No	Co, Ni, Si	Deuterium
Manganese	279,5	Oxidizing air/acetylene or acetylene/N_2O	Yes No	Fe, Si	Deuterium
Nickel	232,0	Oxidizing air/acetylene	No	Fe	Deuterium
Zinc	213,9	Oxidizing air/acetylene	No		Deuterium

NOTE The wavelengths given are the most sensitive. The use of less sensitive lines to avoid the dilution step before measurement is suitable especially for determination of Fe (e. g. 372,0 nm) and Mn (e. g. 403,1 nm) in soil extracts. Interferences are generally lower if the nitrous oxide (N_2O) flame is used for the determination of chromium and manganese. Users should be aware that small changes in gas volume ratios can have significant effects on the intensity of the analytical signal, and can also change the linearity of the instrument response. Also difference in acid strength,

which will vary slightly from digest to digest, can have a measurable effect on some elements. Users should, therefore, familiarize themselves with these aspects of their instrument's performance.

4 Interferences

Digests of soils may contain large amounts of substances that may affect the results. Matrix effects may be overcome, partially or completely, by the use of a chemical modifier like lanthanum, the standard addition technique, and the use of background correction.

5 Reagents

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 Water quality 1 according to EN ISO 3696 for all sample preparations and dilutions.

5.2 Nitric acid, HNO_3, $\rho(HNO_3)$ = 1,4 g/ml, $c(HNO_3)$ = 15 mol/l, $w(HNO_3)$ = 650 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 Hydrochloric acid, HCl, $\rho(HCl)$ = 1,17 g/ml, $c(HCl)$ = 12 mol/l, $w(HCl)$ = 370 g/kg.

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

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

Add 210 ml of hydrochloric acid (5.4) and 70 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.

5.6 Standard stock solutions

5.6.1 General information

Both single-element standard stock solutions and multi-element standard stock solutions with adequate specification, 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 of Annex A.

5.6.2 Standard solutions

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

5.6.3 Standard solution corresponding to 100 mg/l of element

Pipette 10,00 ml of the actual element stock solution (5.6) into a 100 ml volumetric flask. Add 20 ml of nitric acid (5.3) or 20 ml of *aqua regia* (5.5), fill to the mark with water and mix well.

5.6.4 Standard solution corresponding to 10 mg/l of element

Pipette 10,00 ml of the element standard solution (5.6.2) into a 100 ml volumetric flask. Add 2 ml of nitric acid (5.3) or 2 ml of *aqua regia* (5.5), fill to the mark with water and mix well. Prepare this solution on the day of use.

5.7 Calibration solutions

5.7.1 General information

Before each batch of determinations, prepare, from the standard solutions of each element (5.6.3 or 5.6.4), at least four calibration solutions covering the range of concentrations to be determined. Prepare the calibration solutions on the day of use. Use the set of standard solutions containing the same acid as the digested samples.

5.7.2 Blank calibration solutions

Prepare a blank calibration solution in the same way as the calibration solutions without standard solution. Use a 100 ml volumetric flask. Add the appropriate acid that is also used for the preparation and analysis of the sample solutions. Cool if necessary and dilute to volume with water.

5.7.3 Lanthanum chloride solution, $\rho(LaCl_3 \cdot 7\ H_2O)$ = 37 g/l.

Dissolve 100 g lanthanum(III)chloride in 700 ml water. Then quantitatively transfer it to a 1 000 ml volumetric flask and fill to the mark with water.

5.7.4 Blank solution without lanthanum, *aqua regia.*

For preparation see 5.5.

5.7.5 Blank solution without lanthanum, nitric acid.

For preparation see 5.3.

6 Apparatus

6.1 Usual laboratory equipment

Clean all glass or plastic ware carefully before trace element determinations, e. g. by immersion in warm 5 % (volume fraction) aqueous nitric acid solution for a minimum of 6 h, followed by rinsing with water before use. Replace the nitric acid each week.

NOTE 1 It is possible to use usual acid wash if the control blank tests are proving that this washing procedure cleans glass and plastic ware enough for flame atomic absorption.

NOTE 2 It has been found convenient to keep separate sets of glass or plastic ware for the determinations given in this Technical Specification, in order to reduce the possibility of within-laboratory contamination. Similarly, it can be convenient to carry out the acid cleaning step overnight.

6.2 Atomic absorption spectrometer

WARNING — It is essential that the manufacturer's safety instructions are strictly observed when using the atomic absorption spectrometer.

The atomic absorption spectrometer shall be equipped with:

— a hollow cathode lamp or electrodeless discharge lamp appropriate to the element of interest and operated at a current recommended for the lamp by the instrument manufacturer;

— a background correction system;

— a burner suitable for an air/acetylene or nitrous oxide/acetylene flame (operated according to the manufacturer's instructions).

Deuterium background correction is the minimum technical specification acceptable for background correction for measurement wavelengths below 350 nm. Other systems (e. g. Zeeman polarization, Smith-Hieftje) are equally acceptable.

7 Procedure

7.1 Test sample solution

The test sample solution is an aliquot of the particle-free digest solution prepared according to EN 16173 or EN 16174 or other extraction procedures.

7.2 Test blank solution

Prepare the test blank solution at the same time as the extraction with *aqua regia* or nitric acid or other extraction procedure is performed. Follow the same procedure, using the same quantities of all reagents for the determination, but omit the test sample. The dilution of the test blank solution shall be performed in the same manner as the test sample solutions.

If manganese or chromium are measured using a N_2O-acetylene flame, the addition of lanthanum to a test blank solution is not necessary. Only if an air-acetylene flame is applied for manganese or chromium measurement, the addition of lanthanum solution (5.7.3) is necessary as a release agent. Use of sample and blank solutions should be selected accordingly.

7.3 Calibration and determination

Set up the atomic absorption spectrometer according to the manufacturer's instructions at the appropriate wavelength using appropriate conditions (see Table 1), and operate with a suitable background correction system. Aspirate a calibration solution (5.7) and optimize the aspiration conditions, burner height and flame conditions. Adjust the response of the instrument to zero absorbance whilst aspirating water.

Aspirate the set of calibration solutions in ascending order and, as a zero member, the blank solution without lanthanum (5.7.4) or (5.7.5). After a delay of more than 10 s, read the absorbance of each solution at least twice and, if the values fall with an accepted range, average the values. Care should be taken to ensure that, when using the more concentrated standards, the absorbance is < 1, and preferably not more than 0,7.

NOTE Nickel shows severe curvature above about 0,5 absorbance units even with a spectral bandwidth of 0,2 nm. Reduction of bandwidth may reduce curvature.

Establish the formula for the linear calibration from the series of results obtained (see ISO 8466-1). Usually this step is carried out with the aid of the integrated instrument software of the AA system.

Non-linear calibration functions according to ISO 8466-2 are also allowed.

7.4 Determination of the element content of the test sample solution

Aspirate the test blank solution (7.2) and the test sample solution (7.1) separately into the flame, and measure the absorbance for that element. Read the solutions at least twice and, if the values fall within an accepted range, average the values. After each measurement, aspirate water and re-adjust the zero if necessary. In case of re-adjusting the zero, re-check the calibration, e. g. by measuring a standard solution with intermediate element content. If the concentration of the element in the test portion exceeds the calibration