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Sludge, treated biowaste and soil — Determination of elements in aqua regia and nitric acid digests — Flame atomic absorption spectrometry

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Foreword

This document (BT/TF 151 WI CSS99028) has been prepared by Technical Committee CEN/BT TF 151 "Horizontal standards in the Field of Sludge, Biowaste and Soil", the secretariat of which is held by Danish Standards.

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).

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

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Material	Validated for (type of sample, e.g. municipal sludge, compost)	Document
Sludge	#	(reference)
Soil	#	
Treated biowaste	#	

Not enough data received for validation.

Introduction

This document is developed in the project 'Horizontal'. It is the result of a desk study "Horizontal no 20 AAS" and aims at evaluation of the latest developments in assessing cobalt, copper, iron, manganese, nickel and zinc in sludge, treated biowaste and soil and neighbouring fields. After an evaluation study, in which e.g. the ruggedness of the method was studied, a European wide validation of the draft standard has taken place. The results of the desk studies as well as the evaluation and validation studies have been subject to discussions with all parties concerned in CEN. The standard is part of a modular horizontal approach in which the standard belongs to the analytical step.

Until now test methods determining properties of materials were often prepared in Technical Committees (TCs) working on specific products or specific sectors. In those test methods often steps as sampling, extraction, release or other processing, analyses, etc were included. In this approach it was necessary to develop, edit and validate similar procedural steps over and over again for every material or product. Consequently this has resulted in duplication of work. To avoid such duplication of work for parts of a testing procedure references to parts of test methods from other TCs were introduced. However the following problems are often encountered while using references in this way: 1) The referenced parts are often not edited in a way that they could easily be referred to, 2) the referenced parts are often not validated for the other type of material and 3) the updates of such test standards on products might lead to inadequate references.

In the growing amount of product and sector oriented test methods it was recognised that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

Based on this hypothesis a horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole "chain of measurement" (from sampling to analyses). **A beneficial feature of this approach is that "modules" can be replaced by better ones without jeopardizing the standard "chain".**

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

The modules that relates to this standard are specified in section XX Normative references.

An overview of modules and the manner, in which modules are selected will be worked out later, at which time proper reference in this standard will be provided.

1 Scope

This European Standard describes principles and procedures for the determination of metals in aqua regia and nitric acid digests of sludge, soil and treated biowaste and neighbouring samples, using flame atomic absorption.

The method detection limit for each element depends on the sample matrix as well as on the instrument. For water samples with a simple matrix (i.e. low concentration of dissolved solids and particles), the method detection limits will be close to instrument detection limits. In digests containing higher concentrations of dissolved solids, interference effects may lead to an increase in the method detection limit.

Table 1 — Widely used detection limits and measurement ranges for direct determination.

Element	Wavelength (nm)	Detection limit (mg/l)	Measurement range (mg/l)
Cobalt	240,7	0,02	0,06 – 10
Copper	324,8	0,005	0,015 – 10
Iron	248,3	0,03	0,1 – 10
Manganese	279,5	0,01	0,3 – 5
Nickel	232,0	0,02	0,06– 10
Zinc	213,9	0,005	0,015 – 2

The optimum measurement range is from the limit of quantification (LOQ) (which is three times the limit of detection, LOD), and the upper limit for direct determination is corresponding to a concentration giving an absorbance about 1 abs/cm. Dilute the sample for the determination of higher concentrations.

NOTE This Horizontal Standard refers specifically to the use of atomic absorption spectrometry. Users of this Horizontal Standard are advised to operate their laboratories to accepted quality control procedures. Certified Reference Materials (CRM) should be used to establish the amounts of the relevant elements in in-house reference materials. The latter can be used for routine quality control of the procedures given in this Horizontal Standard. Results should be established with control charts, for each element, within the laboratory. No results should be accepted which falls outside an agreed limit. Quality control procedures based on a widely accepted statistical technique should be used to establish such limit, to ensure that these are stable and that no long-term drift is occurring. CRMs should be used regularly to maintain the integrity of the in-house reference materials and, thereby, the quality control system.

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.

EN ISO 3696:1997, *Water for analytical laboratory use – Specification and test methods*.

CSS99031 *Sludge, treated biowaste, and soils in the landscape – Sampling – Framework for the preparation and application of a sampling plan*

CSS99058 *Sludge, treated biowaste, and soils in the landscape – Sampling – Part 1: Guidance on selection and application of criteria for sampling under various conditions*

CSS99057 *Sludge, treated biowaste, and soils in the landscape – Sampling – Part 2: Guidance on sampling techniques*

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CSS99032 *Sludge, treated biowaste, and soils in the landscape – Sampling - Part 3: Guidance on sub-sampling in the field*

CSS99059 *Sludge, treated biowaste, and soils in the landscape – Sampling – Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery*

CSS99060 *Sludge, treated biowaste, and soils in the landscape – Sampling – Part 5: Guidance on the process of defining the sampling plan*

CSS99034 Soil, sludge and treated biowaste – Guidance for sample pre-treatment

CSS99025A Soil, sludge and treated biowaste – Microwave digestion for the extraction of nitric acid soluble fraction of trace elements

CSS99025B Soil, sludge and treated biowaste – Digestion for the extraction of aqua regia soluble fraction of trace elements.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
term
text of the definition

3.2
term
text of the definition

4 Principle

The method is based on the atomic absorption spectrometric measurement of the concentration of the element in an aqua regia or nitric acid extract of the sample, prepared in accordance with the standards given under clause 2, using the instrumental conditions given in table 2.

Table 2 — General conditions for flame atomic absorption spectrometry

Element	Wavelength (nm)	Flame type	Lanthanum chloride	Main interference	Background correction
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 ₂ O	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. Interferences are generally lower if the nitrous oxide 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 especially if background correction is not used. Users should, therefore, familiarize themselves with these aspects of their instrument's performance.

5 Interferences and sources of errors

Sample solutions of waste waters and digestions of sediments and 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.

Nickel is affected a little by a high concentration of cobalt in aqua regia solution. However, the signal was increased by 10 % at a cobalt concentration of 1 000 mg/l.

6 Reagents

Use only reagents of recognised analytical grade and water grade 1 in accordance with ISO 3696.

The metal contribution from water, reagents and gases should be significantly less than the lowest metal content to be determined. The overall metal content of water, chemicals, and gases shall be checked by measuring the total blank (see 9.2)

6.1 Nitric acid, HNO_3 , $c = 1,42 \text{ g/ml}$, 65 % (m/m) solution.

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

6.2 Nitric acid, diluted 1 + 3 (V/V)

Add 250 ml of nitric acid (6.1) to 500 ml of water in a 1 000 ml volumetric flask and fill to the mark with water.

6.3 Hydrochloric acid, 37 %; $\rho \sim 1,18 \text{ g/ml}$

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

6.4 Aqua regia, diluted $\sim 1 + 3$

Dilute 210 ml of hydrochloric acid (6.3) and 70 ml of nitric acid (6.1) with about 500 ml of water in a 1 000 ml volumetric flask, and dilute to the mark.

6.5 Stock solutions

6.5.1 General information

Both single-element stock solutions and multi-element 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 stock solutions may be prepared as indicated in Table A.1 of Annex A.

6.5.2 Standard solutions

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

6.5.3 Standard solution corresponding to 100 mg/l of element

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Pipette 10,00 ml of the actual element stock solution (6.5) into a 100 ml volumetric flask. Add 20 ml of nitric acid (6.2) or 20 ml of aqua regia (6.4), fill to the mark with water and mix well.

6.5.4 Standard solution corresponding to 10 mg/l of element

Pipette 10,00 ml of the element standard solution (6.5.2) into a 100 ml volumetric flask. Add 2 ml of nitric acid (6.2) or 2 ml of aqua regia (6.4), fill to the mark with water and mix well. Prepare this solution on the day of use.

6.6 Calibration solutions

6.6.1 General information

Before each batch of determinations, prepare, from the standard solutions of each element (6.5.2 or 6.5.3), at least four calibration solutions covering the range of concentrations to be determined, the optimum working range being indicated in Table 1. Prepare the calibration solutions on the day of use. Use the set of standard solutions containing the same acid as the digested samples.

6.6.2 Blank calibration solutions

Prepare a blank calibration solution in the same way as the calibration solutions, but add no standard solution. Use a 100 ml volumetric flask. Add acid in correspondence to the samples that the solution will be analysed together with. Cool if necessary and dilute to volume with water.

6.6.3 Lanthanum chloride solution, 37 g/l lanthanum

Dissolve 100 g lanthanum(III) chloride, $\text{LaCl}_3 \cdot 7\text{H}_2\text{O}$, in 700 ml water. Then quantitatively transfer it to a 1 000 ml volumetric flask and fill to the mark with water.

6.6.4 Blank solution without lanthanum, aqua regia

Dilute 210 ml of hydrochloric acid (6.3) and 70 ml of nitric acid (6.1) with 500 ml water in a 1 000 ml volumetric flask and fill to the mark with water.

6.6.5 Blank solution without lanthanum, nitric acid

Dilute 200 ml of nitric acid (6.1) with 500 ml water in a 1 000 ml volumetric flask and fill to the mark with water.

6.6.6 Blank solution with lanthanum, aqua regia

Dilute 210 ml of hydrochloric acid (6.3) and 70 ml of nitric acid (6.1) with 500 ml water in a 1 000 ml volumetric flask. Add 100 ml lanthanum chloride solution (6.6.3) and fill to the mark with water.

6.6.7 Blank solution with lanthanum, nitric acid

Dilute 200 ml of nitric acid (6.1) with 500 ml water in a 1 000 ml volumetric flask. Add 100 ml lanthanum chloride solution (6.6.3) and fill to the mark with water.

7 Apparatus

7.1 Usual laboratory equipment

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

NOTE 1 It is possible to use normal acid wash, if the control tests of blank is proving that this washing procedure is giving glass and plastic ware clean 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 Horizontal Standard, in order to reduce the possibility of within-laboratory contamination. Similarly, it can be convenient to carry out the acid cleaning step overnight.

7.2 Atomic absorption spectrometer

WARNING – It is essential that the manufacturer’s safety recommendations are strictly observed when using these flames.

The atomic absorption spectrometer shall be equipped with:

- a hollow cathod 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 and a halogen lamp for measurement wavelengths above 350 nm. Other systems (e.g. Zeeman polarization, Smith-Hieftje) are equally acceptable and, in certain circumstances, can be superior.

8 Sampling and sample pre-treatment

8.1 Sampling

Sampling shall be carried out in accordance with sampling standards CSS99031-32 and 99057-60.

Store samples in suitable containers with an appropriate closure material such as PTFE. Samples to be frozen may be stored in aluminium containers pre-cleaned by heating to 450°C for minimum 4 hours or by rinsing with a non-chlorinated solvent.

Samples should be kept cold (< 8°C) and in the dark. The sample pre-treatment should take place within 24 hours of sampling. Alternatively, samples may be frozen (-18 °C) directly after sampling and kept frozen for a maximum of one month before sample pre-treatment.

8.2 Sample pre-treatment

Transfer a sub-sample, homogenized according to CSS99034 (Horizontal standard module(s) for pre-treatment of solid materials) of approximately 100 g to a porcelain dish.

Store the ground material in a desiccators or a tightly closed glass container.

9 Procedure

9.1 Test solution

Extract the solid samples with aqua regia or nitric acid in accordance with CSS99025A or B, respectively. Transfer the nitric acid or aqua regia digested samples to 100 ml volumetric flasks and fill to the mark with water, thus giving the test solutions.

9.2 Blank test

Carry out a blank test at the same time as the extraction with aqua regia or nitric acid following the same procedure, using the same quantities of all reagents for the determination, but omitting the test sample. Transfer the digested blank solution to a 100 ml volumetric flask and fill to the mark with water.

9.3 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 with the suitable background correction system in operation. Aspirate a calibration solution (6.6) 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 (6.6.4 or 6.6.5; alternatively 6.6.6 or 6.6.7 when lanthanum is added to the test solution). After a delay of more than 10 sec, read the absorbance of each solution at least twice and, if the values fall within 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 1 Nickel shows severe curvature above about 0,5 absorbance units even with a spectral bandwidth of 0,2 nm.

NOTE 2 The definition of an accepted range is outside the scope of this Horizontal Standard. However, users are reminded of the NOTE in Clause 1, concerning quality control procedures. Whatever the basis for the latter in the laboratory, it should conform to well-established practices, such as those based on control charts, confidence limits, and the statistics of normal and non-normal distributions.

9.4 Plotting the graph

Plot a graph for each element with the concentrations of the 6.6.4 or 6.6.5, (alternatively 6.6.6 or 6.6.7), in mg/l, as abscissa, and the corresponding absorbance values as ordinate, alternatively calculate the correlation coefficients and slope for each element (or determine the calibration function).

9.5 Determination of the element content of the test portion

Aspirate the blank test solution (9.2) and the test portion (9.1) 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 (see NOTE 2 in 9.3). 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 range, dilute the test solution with the blank solution (6.6.4 or 6.6.5, alternatively 6.6.6 or 6.6.7) accordingly. Particular elements might need special precautions (9.6).

If an unknown type of sample is to be handled, determine the concentration of each element by the standard addition method. If the analytical result according to the standard addition method and the calibration curve are equal, the calibration curve method can be applied.

NOTE The temperature of all calibration and test solutions should be within 1 °C of each other at the time of atomic absorption measurement.

9.6 Special precautions

9.6.1 Chromium

For measurement with an air/acetylene flame, add 10 ml of lanthanum chloride solution (6.6.6 or 6.6.7) to the blank, each standard and sample flask.

NOTE The efficiency of the extraction of chromium from soils by aqua regia or nitric acid depends strongly on the nature of chromium compounds present, and the analytical signal is strongly affected by matrix elements (see Table 1) in the extract.

9.6.2 Manganese

For measurement with an air/acetylene flame, add 10 ml of lanthanum chloride solution (6.6.6 or 6.6.7) to the blank, each standard and sample flask.

9.6.3 Nickel

For wavelength $\lambda = 232,0$ nm, select a spectral bandwidth of 0,2 nm to separate the analytical line from adjacent non-absorbing lines.

9.7 Quality Assurance of the overall procedure

9.7.1 Analysis of CRM

Analyse a test sample of a certified reference material with matrix comparable to the samples to be analysed.

9.7.2 Analysis of spiked natural samples

Analyse at least one spiked natural sample for each 20 samples in each series of samples.

Spike 50 μl or 100 μl of the metal stock solution (6,5) to an aliquot of a test sample. Establish a control limit for recovery of the spike based for example on precision data in Annex D or based on laboratory precision data.

NOTE If the recovery is outside the control limits, the whole series of analyses should be repeated.

10 Expression of results

10.1 Method of calculation

By reference to the calibration graph obtained, determine the concentration of the element corresponding to the absorbance of the test portion (9.1) and of the blank test solution (9.2). Calculate the content (w) of the element in the sample using the following equation:

$$w_M = \frac{(\rho_1 - \rho_2) \times f \times V}{m}$$

where

w_M is the mass fraction of the element M in the sample, in milligrams per kilogram;

ρ_1 is the concentration of the analyte, in milligrams per litre, in the diluted test sample solution;

ρ_0 is the concentration of the analyte, in milligrams per liter, in the blank test solution with the same dilution factor as for the test sample solution;

f is the dilution factor of the diluted test portion (9.1), if applicable;

V is the volume, in litres, of the test portion taken for the analysis;

m is the mass of the sample, in kilograms, corrected for water content;

10.2 Expression of results

The measurement uncertainty reported for the results should reflect the results from the quality control measures and incorporate the deviation between the individual readings for the sample in question. In general, values shall not be expressed to a greater degree of accuracy than two significant figures. The rounding of values will depend on the statistics of the quality control procedures mentioned earlier, and the requirements of the analysis.

Example: $w_{(\text{Co})} = 8,5$ mg/kg

$w_{(\text{Co})} = 0,3$ mg/kg

11 Precision data

No data available

12 Test report

The test report shall contain the following information:

- a) a reference to this European Standard including its date of publication;
- b) precise identification of the sample;
- c) expression of results, according to EN xxxxx;
- d) any deviation from this standard, and any facts which may have influenced the result.

When the test is not carried out in accordance with this standard, reference may only be made to EN xxx:2003 in the report in case all deviations from the procedures prescribed in this standard are indicated in the report stating the reason for deviation.

13 Performance characteristics

An interlaboratory test has to be performed to yield precision data. Results are given in Annex D.

Annex A (informative)

Preparation of stock solutions, 1 000 mg/l

A.1 General information

The following procedures for preparation of stock solutions are taken from [1] and [2].

All salts should be dried for one hour at 105 °C, unless otherwise is specified. It is also possible to use commercial standard stock solutions as an alternative.

Table A.1 — Amount of metals and metal salts for preparation of stock solutions

Element	Compound	Formulae	Amount, g
Co	Metal ^a	Co	1,000
Cu	Metal ^b	Cu	1,000
Fe	Metal	Fe	1,000
Mn	Electrolytic metal	Mn	1,000
Ni	Metal	Ni	1,000
Zn	Metal	Zn	1,000

^a Acid cleaned with (1 + 9) HNO₃. ^b Acid cleaned with (1 + 1) HCl.

A.2 Cobalt (Co) and Copper (Cu) stock solution

Dissolve the metal in 50 ml (1 + 1) HNO₃ with heating if necessary to effect dissolution. Cool and dilute to volume with water in a 1 000 ml volumetric flask.

A.3 Iron (Fe) stock solution

Weigh, to the nearest ± 0,0002 g, approximately 1,0000 g iron metal (minimum purity 99,5 %) and dissolve it in a covered 250 ml glass beaker with 10 ml nitric acid. Then add 100 ml of water. Boil to expel nitrous fumes, cool, transfer to a 1 000 ml volumetric flask and fill to the mark with water.

A.4 Manganese (Mn) stock solution

Clean manganese metal by transferring several grams of electrolytic manganese (minimum purity 99,5 %) and dissolve it in a covered 250 glass beaker containing about 150 ml dilute sulfuric acid. Stir and allow the manganese to settle for several minutes. Decant, wash several times with water and finally with acetone. Decant the surplus acetone, dry the metal for 2 minutes at 105 °C and cool in a desiccator.

Weigh, to the nearest ± 0,0002 g, approximately 1,0000 g of such cleaned manganese metal and dissolve it in a covered 250 ml glass beaker with 20 ml hydrochloric acid and 20 ml nitric acid. Then add 100 ml of water. Boil to expel nitrous fumes, cool, transfer to a 1 000 ml volumetric flask and fill to the mark with water.

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A.5 Nickel (Ni) stock solution

Dissolve the metal in 20 ml hot concentrated HNO₃. Cool and dilute to volume with water in a 1 000 ml volumetric flask.

A.6 Zinc (Zn) stock solution

Weigh, to the nearest $\pm 0,0002$ g, approximately 1,0000 g zinc metal (minimum purity 99,5 %) and dilute it in a covered 250 ml glass beaker with 40 ml nitric acid. Then add 100 ml of water. Boil to expel nitrous fumes, cool, transfer to a 1 000 ml volumetric flask and fill to the mark with water.

Annex B
(informative)

Repeatability and reproducibility data

Bibliography

- [1] U.S. Environmental Protection Agency, Method 200.9 – Determination of Trace Elements by Stabilized Temperature Graphite Furnace Atomic Absorption, Rev. 2.2, 1994.
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- [3] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results – Part 1: general principles and definitions.*
- [4] ISO 11047:1998, *Soil quality – Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc in aqua regia extracts of soil – Flame and electrothermal atomic absorption spectrometric methods.*