SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17701-1:2021

01-november-2021

Rastlinski biostimulansi - Določevanje določenih elementov - 1. del: Razklop z zlatotopko za določevanje elementov

Plant biostimulants - Determination of specific elements - Part 1: Digestion by aqua regia for subsequent determination of elements

Biostimulanzien für die pflanzliche Anwendung - Bestimmung spezifischer Elemente - Teil 1: Aufschluss durch Königswasser zur anschließenden Bestimmung der Elemente

Biostimulants des plantes - Détermination des éléments spécifiques - Partie 1 : Digestion par eau régale pour la détermination subséquente des éléments

Ta slovenski standard je istoveten z: **FprCEN/TS 17701-1**

ICS:

65.080	Gnojila	Fertilizers

kSIST-TS FprCEN/TS 17701-1:2021 **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

FINAL DRAFT
FprCEN/TS 17701-1

September 2021

ICS 65.080

English Version

Plant biostimulants - Determination of specific elements - Part 1: Digestion by aqua regia for subsequent determination of elements

Biostimulants des plantes - Détermination des éléments spécifiques - Partie 1 : Digestion par eau régale pour la détermination subséquente des éléments

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This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 455.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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Ref. No. FprCEN/TS 17701-1:2021 E

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European foreword

This document (FprCEN/TS 17701-1:2021) has been prepared by the Technical Committee CEN/TC 455 “Plant Biostimulants”, the secretariat of which is held by AFNOR.

This document is currently submitted to the Vote on TS.

The CEN/TS 17701 series, *Plant biostimulants - Determination of specific elements*, consists of the following parts:

- CEN/TS 17701-1, *Plant biostimulants - Determination of specific elements - Part 1: Digestion by aqua regia for subsequent determination of elements*;
- CEN/TS 17701-2, *Plant biostimulants - Determination of specific elements - Part 2: Determination of total content of Cd, Pb, Ni, As, Cr, Cu and Zn*;
- CEN/TS 17701-3, *Plant biostimulants - Determination of specific elements - Part 3: Determination of mercury*.

Introduction

This document was prepared by the experts of CEN/TC 455 ‘Plant Biostimulants’. The European Committee for Standardization (CEN) was requested by the European Commission (EC) to draft European standards or European standardization deliverables to support the implementation of Regulation (EU) 2019/1009 of 5 June 2019 laying down rules on the making available on the market of EU fertilising products (“FPR” or “Fertilising Products Regulation”).

This request, presented as SR M/564, also contributes to the Communication on “Innovating for Sustainable Growth: A Bio economy for Europe”. The Working Group 4 “Other safety parameters”, was created to develop a work program as part of this Request. The technical committee CEN/TC 455 ‘Plant Biostimulants’ was established to carry out the work program that will prepare a series of standards. The interest in biostimulants has increased significantly in Europe as a valuable tool to use in agriculture. Standardization was identified as having an important role in order to promote the use of biostimulants. The work of CEN/TC 455 seeks to improve the reliability of the supply chain, thereby improving the confidence of farmers, industry, and consumers in biostimulants, and will promote and support commercialisation of the European biostimulant industry.

The preparation of this document is based on a standardization request to CEN by the European Commission and the European Free Trade Association (Mandate M/564) concerning the modernization of methods of analysis of fertilizers in the framework of Regulation (EU) 2019/1009 of the European Parliament and of the Council.

Aqua regia is applied to digest different matrices for the subsequent determination of many elements. For example, a multi-matrix standard for aqua regia extraction of soils, sludges and biowaste was prepared by CEN/TC 444. A similar procedure was applied for determination of aqua regia extractable contents of arsenic, mercury, cadmium, chromium, nickel and lead in fertilizers and liming materials (standard prepared by CEN/TC 260). CEN/TC 260 also prepared a standard for extraction of a total micronutrients content in fertilizers using aqua regia. CEN/TC 223 published a standard for a similar procedure for soil improvers and growing media. Wide use of the aqua regia digestion, availability of the instruments and the possibility to merge the standards for different matrices in future, were the main reasons for also applying this method of digestion for plant biostimulants. A simple pre-concentration procedure is a part of this document to also cover the digestion of liquid samples with low dry matter content in the case that the measurement method is not sufficiently sensitive. An Annex A describes in detail the calculation of the initial sample weight to be taken for the pre-concentration step to achieve concentrations of the individual elements above the limit of quantification.

WARNING – Persons using this document should be familiar with usual laboratory practice. This document does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and to ensure compliance with any national regulatory conditions.

IMPORTANT – It is absolutely essential that tests conducted according to this document are carried out by suitably trained staff.

1 Scope

This document specifies the procedure for the digestion of different plant biostimulants with aqua regia to enable a subsequent determination of arsenic (As), cadmium (Cd), copper (Cu), chromium (Cr), mercury (Hg), nickel (Ni), lead (Pb) and zinc (Zn). The procedure can be also applied for determination of other elements. The procedure is applicable for all solid and/or liquid plant biostimulants.

This document is also applicable to the blends of fertilizing products where plant biostimulants are the main part of the blend. Otherwise, the Technical Specification for the main part of the blend applies.

The extracts are suitable for analysis using CEN/TS 17701-2 (ICP-AES) and CEN/TS 17701-3 (Hg analysis).

NOTE Alternatively, inductively coupled plasma mass spectrometry (ICP-MS) can be used for the measurement if the user proves that the method gives the same results.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

3.1
digestion
mineralization of the organic matter of a sample and dissolution of its mineral part, more or less completely, when reacting with a reagent mixture

3.2
blank test solution
solution prepared in the same way as the test sample solution but omitting the test portion

4 Principle

The samples are digested by boiling in aqua regia for two hours under reflux conditions.

5 Sampling and sample preparation

Sampling and sample preparation are not part of this document. A recommended sampling and a sample preparation method is given in:

CEN/TS 17702-1, *Plant biostimulants - Sampling and sample preparation - Part 1: Sampling*;

CEN/TS 17702-2, *Plant biostimulants - Sampling and sample preparation - Part 2: Sample preparation*.

6 Reagents

6.1 Water with a specific conductivity not higher than 0,2 mS/m at 25 °C, free from the elements to be determined.

6.2 Hydrochloric acid 37 % HCl, $c(\text{HCl}) \approx 12$ mol/l, $\rho \approx 1{,}18$ g/ml.

6.3 Nitric acid 65 %, $c(\text{HNO}_3) \approx 14{,}3$ mol/l; $\rho \approx 1{,}4$ g/ml.

6.4 Antifoaming agent, e.g. *n*-dodecane ($C_{12}H_{26}$) or octanol ($C_8H_{18}O$) are suitable.

7 Apparatus

7.1 Common laboratory glassware. All glassware and plastic ware shall be adequately cleaned and stored to avoid any contamination.

7.2 Apparatus for thermal heating digestion, temperature controlled with reaction vessel and reflux condenser. The capacity of the reaction vessel should be at least 5 times of the volume of the aqua regia used.

7.3 Filter paper, ash free and of recognized and tested quality.

7.4 Analytical balance capable of weighing to an accuracy of 1 mg or better.

8 Procedure

CAUTION – Suitable precautions shall be taken to avoid any contact of laboratory staff with acid fumes. The digestion procedure shall be carried out in a well-ventilated fume cupboard.

8.1 Sample digestion

This procedure can be used for all samples of plant biostimulants if a sufficiently sensitive method for determination is used. For some liquid samples with a low dry matter content, a pre-concentration step (8.2) may be necessary.

Weigh 2,5 to 5,0 g of the solid, or liquid sample representing approximately1,5 to 3,0 g of dry sample, to the nearest 0,001 g. Transfer the sample quantitatively to the reaction vessel (7.2). Liquid samples are preferably weighed directly into the reaction vessel (7.2). Moisten the solid sample with about 0,5 ml to 1,0 ml of water and add, while gently mixing, 21 ml of hydrochloric acid (6.2) followed by 7 ml of nitric acid (6.3) drop wise if necessary. Add one drop of the antifoaming agent (6.4) in the case of excessive foaming. Connect condenser to the reaction vessel and let the mixture stand at laboratory temperature until any effervescence almost ceases. Transfer to the heating device and slowly raise the temperature of the reaction mixture to reflux conditions and maintain for 2 h ensuring that the condensation zone is lower than ⅓ of the height of the condenser. Then allow to cool and rinse the condenser with 10 ml of water (6.1).

Transfer the content of the reaction vessel quantitatively into a 150ml volumetric flask, dilute to the mark with water (6.1) and mix well.

Test solutions may be filtered or centrifuged if necessary. Use ash-free filter paper of recognized and tested quality (7.3) and discard the first portion of the filtrate (approximately 20 ml).

Carry out the measurement immediately or store the extracts in tightly closed vessels for up to 15 days.

The procedure may be modified for the use of 100 ml or 50 ml volumetric flasks or graduated plastic tubes. In this case the weight of the sample and volumes of the acids should be changed accordingly. For more information see Annex A.

NOTE 1 Weigh the samples for dry matter determination at the same time and from the same sub-sample to ensure that the dry matter is determined on the samples identical to those used for determination of parameters that relates to dry matter.

NOTE 2 If excessive foaming occurs despite the addition of an anti-foaming reagent, it is possible to leave the samples overnight after the addition of the acids to allow a slow oxidation or to use a digestion block with two independent heating zones.

NOTE 3 The presence of small quantities of organic matter after digestion will not affect determination of the elements by ICP-AES and it is not necessary to apply additional organic matter removal.

8.2 Sample pre-concentration

This procedure is necessary for liquid samples with low dry matter content in the case that the measurement method is not sufficiently sensitive. See Annex A for calculation of the initial sample weight to be taken for the pre-concentration step to achieve concentrations of the individual elements above the limit of quantification.

Weigh approximately 10 to 50 g of the liquid sample representing 1 to 3 g of dry sample to the nearest 0,001 g directly into the reaction vessel (7.2) and heat the vessel to approx. 75 °C to evaporate excessive water. After evaporation to approx. 5 ml and cooling, follow procedure 8.1. Adjustment of the pre-concentration step for different final volumes is given in Annex A.

Evaporation of a large volume of sample usually takes several hours. Higher temperature (up to 95 °C) for faster evaporation may be used if the final digest is not used for the determination of mercury. The fastest evaporation can be achieved by using a 250 ml beaker placed on a hot plate for evaporation and consequently also for digestion under a watch glass.

8.3 Blank

Prepare a blank test solution following the same procedure as for samples. The measurement of a blank is necessary to determine the contribution of the extracting solution, glassware and filter paper used.

9 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) test method used with reference to this document;

c) date of sampling and sampling procedure (if known);

d) date when the extraction was finished;

e) all operating details not specified in this document, or regarded as optional, together with details of any incidents that occurred when performing the method, which might have influenced the test result(s).

Annex A
(informative)

Adjustment to different digestion procedures and measurement apparatuses

A.1 Adjustment of the procedure to different final volumes

Volumetric flasks or graduated plastic tubes for 150, 100, or 50 ml may be used for the final volume adjustment. In Table A.1 there are given recalculated parameters for the different final volumes.

Table A.1 — Recalculated parameters for different final volumes

VF [ml]	HNO_3 [ml]	HCl [ml]	W [g]	VM [ml]
150	7	21	1,5 - 3	6
100	4,7	14	1 - 2	4
50	2,3	7	0,5 - 1	2
VF – final volume of a volumetric flask or a graduated plastic tube W – weight of the sample recalculated to dry matter VM – maximum volume after pre-concentration step (approx.)				

A.2 Calculation of the sample weight

A.2.1 General

According to the given legislative limits and the limits of quantification for the individual elements and the measurement apparatus, the weight of the sample should be adjusted to ensure that the concentration of the element in the final digest is above the limit of quantification. The calculation of the weight of the sample is necessary only for the elements with a low legislation limit and/or very high LOQ. Usually only for cadmium, in some cases also for arsenic and nickel. The highest volume calculated for different elements will be applied.

Table A.2 — Legislative limits and typical limits of the quantitative determination

Element	Cd	Pb	Hg	As	Cr	Cu	Ni	Zn
Legislative limit [mg/kg]	1,5	120	1	40	200	600	50	1500
Legislative limit [µg/l]	30	2400	20	800	4000	12000	1000	30000
LOQ [µg/l]	7,5	100	1,0	75	75	75	75	250
Legislative limits are from Regulation (EU) 2019/1009 of the European Parliament and of the Council Legislative limit in [µ/l] was calculated for 1 g sample (dry matter) in 50 ml LOQ – example of typical values for an ICP-AES instrument, radial view LOQ – example of a limit for Hg is given for a direct amalgamation technique								