

# SLOVENSKI STANDARD SIST EN 17701-2:2025

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# Rastlinski biostimulanti - Določanje specifičnih elementov - 2. del: Določanje celotnega Cd, Pb, Ni, As, Cr, Cu in Zn

Plant biostimulants - Determination of specific elements - Part 2: Determination of total content of Cd, Pb, Ni, As, Cr, Cu and Zn

Pflanzen-Biostimulanzien - Bestimmung spezifischer Elemente - Teil 2: Bestimmung des Gesamtgehaltes an Cd, Pb, Ni, As, Cr, Cu und Zn

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Biostimulants des végétaux - Dosage des éléments spécifiques - Partie 2 : Détermination de la teneur totale en Cd, Pb, Ni, As, Cr, Cu et Zn

Ta slovenski standard je istoveten z: EN 17701-2:2024

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Gnojila

Fertilizers

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# EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

# EN 17701-2

November 2024

ICS 65.080

Supersedes CEN/TS 17701-2:2022

**English Version** 

# Plant biostimulants - Determination of specific elements -Part 2: Determination of total content of Cd, Pb, Ni, As, Cr, Cu and Zn

Biostimulants des végétaux - Dosage des éléments spécifiques - Partie 2 : Détermination de la teneur totale en Cd, Pb, Ni, As, Cr, Cu et Zn Pflanzen-Biostimulanzien - Bestimmung spezifischer Elemente - Teil 2: Bestimmung des Gesamtgehaltes an Cd, Pb, Ni, As, Cr, Cu und Zn

This European Standard was approved by CEN on 26 August 2024.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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# EN 17701-2:2024 (E)

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# **European foreword**

This document (EN 17701-2:2024) has been prepared by Technical Committee CEN/TC 455 "Plant biostimulants", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2025, and conflicting national standards shall be withdrawn at the latest by May 2025.

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

This document supersedes CEN/TS 17701-2:2022.

EN 17701-2:2024 includes the following significant technical changes with respect to CEN/TS 17701-2:2022:

- scope text regarding blends has been changed;
- Annex A "Results of the inter-laboratory study" has been added;
- Annex ZA has been added.

This document has been prepared under a standardization request addressed to CEN by the European Commission. The Standing Committee of the EFTA States subsequently approves these requests for its Member States.

For the relationship with EU Legislation, see informative Annex ZA, which is an integral part of this document.

EN 17701 series, *Plant biostimulants — Determination of specific elements*, consists of the following parts:

- Part 1: Digestion by aqua regia for subsequent determination of elements;
- Part 2: Determination of total content of Cd, Pb, Ni, As, Cr, Cu and Zn;
- Part 3: Determination of mercury.

Any feedback and questions on this document should be directed to the users' national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.

#### EN 17701-2:2024 (E)

# Introduction

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 [1] laying down rules on the making available on the market of EU fertilising products ("FPR" or "Fertilising Products Regulation").

This standardization request, presented as SR M/564 and relevant amendments, also contributes to the Communication on "Innovating for Sustainable Growth: A Bio economy for Europe". The interest in plant 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

This document concerns the analytical measurement step. Aqua regia digestion followed by inductively coupled plasma atomic emission spectrometry (ICP-AES) is widely used for determination of many elements, for example, a multi-matrix standard for aqua regia extraction of soils, sludges and biowaste [2] and soil [3]. 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 [4, 5] and for determination of micronutrients in mineral fertilizers [6]. Inductively coupled plasma atomic emission spectrometry (ICP-AES) is nowadays widely used and a well-established method in many laboratories.

The inter-laboratory study reflects the final statistical characteristics of the method for the determination of the specific elements in aqua regia digests including both, the digestion and the measurement steps.

**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.

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# 1 Scope

This document specifies a method for the determination of total contents of arsenic (As), cadmium (Cd), copper (Cu), chromium (Cr), lead (Pb), nickel (Ni) and zinc (Zn) in aqua regia plant biostimulant digests using inductively coupled plasma-atomic emission spectrometry (ICP-AES).

This document is applicable to the blends of fertilizing products where a blend is a mix of at least two of the following component EU fertilising products categories: Fertilizers, Liming Materials, Soil Improvers, Growing Media, Inhibitors, Plant Biostimulants, and where the following category Plant Biostimulants is the highest % in the blend by mass or volume, or in the case of liquid form by dry mass. If Plant Biostimulants is not the highest % in the blend, the European Standard for the highest % of the blend applies. In case a blend of fertilizing products is composed of components in equal quantity or in case the component EU fertilising products used for the blend have identical formulations<sup>1</sup>, the user decides which standard to apply.

This method is applicable to aqua regia digests prepared according to EN 17701-1:2024. The method can be used for the determination of other elements, provided the user has verified the applicability.

NOTE Alternatively, inductively coupled plasma mass spectrometry (ICP-MS) can be used for the determination of the elements in the aqua regia digests if the user proves that the method gives the same results.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 17701-1:2024, Plant biostimulants — Determination of specific elements — Part 1: Digestion by aqua regia for subsequent determination of elements

EN 17704:2024, Plant biostimulants — Determination of dry matter

## 3 Terms and definitions SIST EN 17701-2:2025

://standards.iteh.ai/catalog/standards/sist/5a7f855c-0d64-4e58-b7a8-2032f54f1e63/sist-en-17701-2-2025 For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <u>https://www.iso.org/obp/</u>
- IEC Electropedia: available at <u>https://www.electropedia.org/</u>

#### 3.1

**analyte** parameter to be determined

#### 3.2

blank calibration solution

solution prepared in the same way as the calibration solution but leaving out the analytes

#### 3.3

#### blank test solution

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

<sup>&</sup>lt;sup>1</sup> An example of such a blend is a product with two claimed functions consisting of a non-microbial plant biostimulant and an organic fertilizer composed of 1kg/kg of plant biostimulant from seaweed.

#### 3.4

#### calibration solution

solution used to calibrate the instrument, prepared from stock solutions by adding acids, buffer, reference element and salts as needed

#### 3.5

#### digest

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

#### 3.6

#### stock solution

solution with accurately known analyte concentration(s), prepared from pure chemicals (6.5)

#### 3.7

#### test sample solution

solution prepared after extraction or digestion of the test sample according to appropriate specifications

# 4 Principle

The method is based on the inductively coupled plasma atomic emission spectrometry (ICP-AES) measurement of the concentration of arsenic, cadmium, copper, chromium, lead, nickel and zinc in plant biostimulant aqua regia digests. The elements are determined after an appropriate dilution of the digest, if necessary. The solution is dispersed by a suitable nebulizer of the ICP-AES instrument and the resulting aerosol is transported into the plasma torch. Element specific emission spectra are produced by a radiofrequency inductively coupled argon plasma where atoms or ions are excited at high temperature. The emission spectra are dispersed by a spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Multi-element determinations using sequential or simultaneous optical systems and axial, radial or dual viewing of the plasma may be used.

#### **5** Interferences

<u>ST EN 17701-2:2025</u>

ttps://standards.iteh.ai/catalog/standards/sist/5a7f855c-0d64-4e58-b7a8-2032f54f1e63/sist-en-17701-2-2025 5.1 General

Interferences and matrix effects shall be recognized and appropriate measures to minimize them shall be made. There are several types of interferences – see 5.2 to 5.6.

## **5.2 Spectral interferences**

Spectral interferences are due to incomplete isolation of the radiation emitted by the analyte from other detected radiation sources. Spectral interferences are caused by the background emission from continuous or recombination phenomena, by stray light which causes background increase or overlap of a spectral line from another element, or by the unresolved overlap of molecular band spectra. Background emission and stray light can usually be compensated for by subtracting the background emission measured adjacent to the analyte wavelength peak. To correct a sloping background, shift background correction points on each side of the peak are used. The increase of background is more intensive with axial-view instruments. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. A spectral line overlap usually leads to the choice of an alternative line. If this is not possible, mathematical correction procedures (e.g. inter-element correction technique or multi-component spectral fitting) can be used to compensate for the interference. These correction procedures are usually a part of the instrument software.

#### **5.3 Transport interferences**

Transport interferences are caused by differences in the properties between the sample solutions and the calibration solutions (viscosity, surface tension, density, dissolved solid content, type and concentration of acids). As a consequence, the supply of solution to the nebulizer, the efficiency of nebulization and the droplet size distribution of the aerosol is altered, resulting in a change of sensitivity. Errors due to these interferences can be overcome by dilution of the solutions, by matrix matching, by standard addition or by internal standard.

#### **5.4 Excitation interferences**

Excitation interferences are attributed to a change in the excitation conditions in the plasma, especially by the presence of easily ionisable elements. The interferences depend on the operating conditions of the plasma (e.g. power, sample introduction, gas flowrate or observation height) and differs from element to element. Improvement of the plasma conditions can therefore reduce excitation interferences. Other possibilities are dilution of the solutions, matrix matching or the standard addition technique.

#### **5.5 Chemical interferences**

Chemical interferences are not significant with the ICP-AES technique, but if observed, they can be minimized by a careful selection of operating conditions (e.g. radio frequency power, observation position, gas flow rate and so forth).

#### **5.6 Memory interferences**

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. This type of interference can be caused by sample deposits or the accumulation in pump tubing, nebulizer, spray chamber or plasma torch. The possibility of memory interferences should be recognized within an analytical run and suitable rinse steps and rinse times should be used.

## 6 Reagents

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.

**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** Nitric acid,  $c(HNO_3) \approx 14.3 \text{ mol/l}, \rho \approx 1.4 \text{ g/ml}.$ 

#### **6.2.1 Diluted nitric acid solution,** *c*(HNO<sub>3</sub>) = 5 mol/l.

Add 350 ml of nitric acid (6.2) to 650 ml of water (6.1).

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

**6.4 Mixed acid solution,** 0,8 mol/l nitric acid and 1,8 mol/l hydrochloric acid.

Add 150 ml of hydrochloric acid (6.3) and 56 ml nitric acid (6.2) consecutively to 600 ml of water (6.1) and fill up to 1 000 ml with water (6.1).

**6.5 Standard stock solutions,** arsenic, cadmium, copper, chromium, lead, nickel and zinc standard stock solutions,  $\rho = 1\,000$  mg/l for each element.

Both single-element stock solutions and multi-element stock solutions with adequate specification, stating the acid used and the preparation technique, are commercially available. Multi-element stock solutions are usually available at the individual mass concentration  $\rho = 100 \text{ mg/l}$  for each element. 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 by the dissolution of high purity metals.

#### **6.5.1 Standard solution**, $\rho = 100$ mg/l of element.

Use commercially available solution of this concentration for each element or pipette, 10 ml of the appropriate element stock solution (6.5) into a 100 ml volumetric flask. Add 10 ml of diluted nitric acid (6.2.1), fill to the mark with water (6.1) and mix well. This solution is used to prepare spiked test solutions and calibration solutions.

#### **6.5.2** Standard solution, $\rho = 10 \text{ mg/l of cadmium}$ .

Pipette 10 ml of cadmium standard solution (6.5.1) into a 100 ml volumetric flask. Add 10 ml of diluted nitric acid (6.2.1), fill to the mark with water (6.1) and mix well. This solution is used to prepare calibration solutions for cadmium.

#### **6.6 Argon**, purity 99,995 % or better.

- 7 Apparatus
- 7.1 Common laboratory glassware. //standards.iteh.ai)

## 7.2 Inductively coupled plasma atomic emission spectrometer.

**WARNING** — It is essential that the manufacturer's safety instructions are strictly observed when using this apparatus.

The inductively coupled plasma atomic-emission spectrometer consists of a sample introduction system, the plasma as an excitation source, an optical system, a detector and a computer with suitable software. The sample is transported by the introduction system (rotation tube pump, nebulizer and a spray chamber) to the plasma torch. Around the torch a water-cooled radio frequency (RF) coil is placed. A frequency of 27 MHz to 56 MHz with a power of 600 W to 2 000 W is usually used. The emission from the plasma can be observed either from the side (radial) or from the torch central symmetrical axis (axial). Axial viewing gives more signal intensity due to the increased observation path length of the normal analytical zone of the plasma, but an increase of interference is also commonly observed. Spectral lines are measured and registered either in a sequential or a simultaneous manner.

# 8 Procedure

## 8.1 Preparation of test sample solution and blank test solution

Aqua regia digests shall be prepared according to EN 17701-1:2024. The digests are usually measured using ICP-AES directly with calibration solutions of the same final concentration of aqua regia. The aqua regia digests may also be used for the determination of other elements.

A blank test solution is prepared for the measurement following the same procedure as for test sample solutions.