Open call for tenders for the validation of methods in CEN/TC 455/WG 4 "Other safety parameters" (Proficiency test providers for 6 inter-laboratory studies)

as part of the European Commission Standardisation Request M/564 to the European Committee for Standardisation referring to the EU fertilising products in support of Regulation (EU) 2019/1009 of the European Parliament and of the Council

1. Background

1.1 Regulation, standardization request and involved standardization bodies

The European Committee for Standardisation (CEN) has been requested to draft harmonised standards and European standardisation deliverables in support of Regulation (EU) 2019/1009 for EU fertilising products which lays down rules on the making available on the market of EU fertilising products and amending Regulations (EC) No 1069/2009 and (EC) No 1107/2009 and repealing Regulation (EC) No 2003/2003 (FPR).

The FPR aims at promoting an increased use of recycled nutrients to further aid the development of a circular economy and allow a more resource-efficient general use of nutrients, while reducing EU's dependency on nutrients from third countries.

Certain products are being used in combination with fertilisers for the purpose of improving nutritional efficiency, with the beneficial effect of reducing the amount of fertilising products used and hence their environmental impact. In order to facilitate their free movement in the internal market, not only fertilising products, i.e. products intended to provide plants with nutrients, but also products intended to improve plants' nutrition efficiency are covered by the harmonisation.

Different product functions warrant different product safety and quality requirements adapted to their different intended uses. EU fertilising products are therefore divided into product function categories (PFCs) and component material categories (CMCs).

In order to provide the market with the means to claim proof of compliance, Technical Specifications (TSs) followed by harmonised European standards (hENs) have to be developed under this Specific Agreement SA/CEN/564 (SA), related to the European Commission Standardisation Request M/564 ("SReq"). Three CEN/Technical Committees (TCs) will perform the work mandated under this SReq:

- CEN/TC 223 Soil Improvers and growing media,
- CEN/TC 260 Fertilizers and liming materials, and
- CEN/TC 455 Plant biostimulants.

The current open call for the recruitment of proficiency test providers concerns for every of the six inter-laboratory studies the following PFCs relevant to CEN/TC 455 *Plant biostimulants*:

PFC 6. Plant biostimulant
  A. Microbial plant biostimulant
  B. Non-microbial plant biostimulants, and
CMC 7: Micro-organisms.

The work programme listed out in the SReq (Annex 1) for CEN/TC 455 Plant biostimulants includes 33 CEN TSs and 33 hENs. Work is expected to start in 10/2020. The expected duration is 4 years.

CEN/TC 455 *Plant biostimulants* was created in 2017 to set up European Standards for all kinds of plant biostimulants. AFNOR, the French member of the National Standardisation Bodies represented at the European and international level, detains the Secretariat of CEN/TC 455 *Plant biostimulants* and will deal with the administrative management of

the standardisation work.

ÚNMZ, the Czech member of standards networking at European (CEN) and international (ISO) levels, holds the secretariat of CEN/TC 455/WG 4 *Other safety parameters* since September 2017.

Since 2018 ÚNMZ delegated in accordance with Czech law all tasks related to the CEN membership to the Czech Standardization Agency (ČAS). Therefore ČAS will be responsible for the administrative management of the standardisation work in WG 4.

The Czech Standardization Agency was established as a state contributory organization by the Czech Office for Standards, Metrology and Testing (ÚNMZ) pursuant to Act No. 265/2017 Coll., Amending Act No. 90/2016 Coll. their supply to the market, and Act No. 22/1997 Coll., on technical requirements for products and on amendments and supplements to certain acts, as amended.

Since 1 January 2018, the Czech Standardization Agency (ČAS) has taken over all activities related to the development, issue and distribution of technical standards from the ÚNMZ including the fulfilment of membership obligations in European and international standardization organizations. Therefore ČAS will be responsible for the administrative management of the standardisation work in WG 4.

1.2 Related standardization projects

Through M/564, the European Commission is requesting the development of 33 CEN Technical Specifications (TS) and 33 European Harmonised Standards (hEN). WG 4 *Other safety parameters* is in charge of developing nine TS and nine hEN. Six of them will contain new methods for the analysis of Plant Biostimulants.

These methods will be published in a first step as TS without validation, but will have to be validated before they will be in a second step published as harmonized European standards (hEN).

a) *Plant Biostimulants – Determination of specifics elements - Part 1 Digestion by aqua regia for subsequent determination of elements*

   This hEN specifies a method for digestion of plant biostimulants by the use of aqua regia as a digestion solution. This method is applicable for the subsequent determination of the following elements: cadmium (Cd), lead (Pb), nickel (Ni), mercury (Hg), copper (Cu), zinc (Zn), chromium (Cr) and may also be applicable for the digestion of other elements.

   The proposed hEN will be used for digestion of different plant biostimulants to enable a subsequent determination of total content of Cd, Pb, Ni, (As), Hg, Cu, Zn, Cr and other elements, if required in the future. Aqua regia digestion is well established in the analytical laboratories and it is used for a wide variety of matrices (soil, sludge, bio waste, mineral and organic fertilisers etc.). An individual standard for the digestion procedure will enable easier future development of the different measurement procedures using the same digestion. The proposal applies a modular aspect of standardisation that is widely used in the methodology applied to different environmental matrices (CEN/TC 444 Environmental characterization of solid matrices) and also for determination of micronutrients in mineral fertilisers. Existing CEN and ISO standards on this topic will be considered while developing this standard for plant biostimulants, although new approaches especially for liquid and low dry matter biostimulants will have to be adopted given the specific nature of plant biostimulants.

   The producers will be able to check the quality of their products for compliance with the required legislative limits and competent authorities will have an instrument for an effective control of the regulatory limits. Consumers and environmental stakeholders will profit from the well-established uniform and reliable control of the products applied to soil and to crops. The apparatus needed for the method are widely used in analytical laboratories and the application of this method will not have any excessive demands on the laboratory equipment.

b) *Plant biostimulants – Determination of specifics elements - Part 2 Determination of total content of Cd, Pb, Ni, As, Cr, Cu and Zn*

   This hEN specifies the method for determination of elements after digestion of plant biostimulants by aqua regia. This method is applicable for the simultaneous multi-element determination of the following elements: cadmium (Cd), lead (Pb), nickel (Ni), copper (Cu), zinc (Zn), chromium (Cr) and may also be applicable for the determination of many other elements.

   The proposed hEN will be used for determination of total content of Cd, Pb, Ni, (As), Cu, Zn, Cr after digestion of different plant biostimulants by aqua regia. The method covers also some possible future needs because it can be

used for determination of many other elements. Inductively coupled plasma atomic emission spectrometry (ICP-AES) is a multi-element method with a suitable sensitivity for all required elements. The method is well established in the analytical laboratories and it is used for a wide variety of matrices digested by aqua regia (soil, sludge, biowaste, mineral and organic fertilisers etc.). An individual standard for the measurement will enable easier future modification of the scope (more elements to be determined etc.). The proposal applies a modular aspect of standardisation used widely in the area of methods for different environmental matrices (CEN/TC 444) and also for determination of micronutrients in mineral fertilisers. Existing CEN and ISO standards will be considered in developing this standard for plant biostimulants.

The producers will be able to check the quality of their products for compliance with the legislative limits and competent authorities will have a suitable tool for an effective control of the regulatory limits. Consumers and environmental stakeholders will profit from the well-established uniform and reliable control of the products applied to soil and crops. The instruments needed for the method are widely used in the analytical laboratories and the application of this method will not have any excessive demands on the laboratory equipment and/or staff training.

c) *Plant biostimulants - Determination of specifics elements - Part 3 Determination of mercury*

This hEN specifies the method for determination of mercury (Hg) by cold vapour generation or by direct amalgamation technique after digestion of plant biostimulants by aqua regia.

The proposed standard will be used for determination of Hg after digestion of different plant biostimulants by aqua regia. The method covers all methods of cold vapour generation (batch, continuous flow, segmented flow) and two measurement methods coupled to the cold vapour apparatus – atomic absorption spectrophotometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES). There will be also a possibility to use instruments with an amalgamation technique. The method is well established in the analytical laboratories and it is used for a wide variety of different matrices and digestates. The proposal applies modular l aspect of standardisation used widely in the area of methods for different environmental matrices (CEN/TC 444). Available standards (e.g. EN 16320 and EN 16175-1) will be considered during development of the standard for plant biostimulants.

The producers will be able to check the quality of their products for compliance with the demanded legislative limits and competent authorities will have a suitable tool for an effective control of the regulatory limits. Consumers and environmental stakeholders will profit from the well-established uniform and reliable control of the products applied to soil and crops. The instruments needed for the method are widely used in the analytical laboratories and the application of this method will not have any excessive demands on the laboratory equipment and/or staff training.

d) *Plant biostimulants - Determination of chromium (VI)*

This hEN specifies the method for determination of hexavalent chromium after extraction of different plant biostimulants by ion chromatography.

The proposed hEN will be used for determination of hexavalent chromium – Cr(VI) after extraction of different plant biostimulants using:

A. Phosphate buffer, for organic and organic-based plant biostimulants;

B. Alkaline digestion, for inorganic plant biostimulants.

Both procedures use ion chromatography for speciation of different chromium ions in the extract followed by spectrophotometric determination (direct or after post-column reaction) or by ICP-MS determination. The methods are able to extract all species of Cr(VI), the adapted conditions of the extraction do not induce reduction of native Cr(VI) to Cr(III), and it does not cause oxidation of native Cr(III) contained in the sample to Cr(VI). For procedure B) alkaline conditions prevent reduction of Cr(VI) and the addition of magnesium and phosphate buffer prevents air oxidation of Cr(III).

The methods described in the available standards are relatively well established in control analytical laboratories and they are used for a wide variety of matrices (soil, sludge, biowaste, fertilizers etc.). A preliminary determination of chromium in aqua regia extracts by ICP-AES can reduce the number of the samples where determination of Cr(VI)

is necessary. (If the content of aqua regia extractable chromium is lower than the legislative limit for hexavalent chromium then the determination of this individual species is not necessary).

The method will be developed with respect to EN ISO 17075-2 (after improvement of LOQ) and after consideration of EN 15192 and EN 16318 (method B).

The producers will be able to check the quality of their products for compliance with the demanded legislative limits and competent authorities will have a suitable tool for an effective control of the regulatory limits. Consumers and environmental stakeholders will profit from the well-established uniform and reliable control of the products applied to soil and crops. However, the instruments needed for this method tend to be more prevalent in well-equipped analytical laboratories; in addition, very good staff training is required to conduct this method. Therefore, preliminary determination of total chromium simultaneously with the other elements by ICP-AES can give an indication of the necessity to determine Cr(VI) with this specific method. This stepwise approach can also further minimize the cost for the analyses.

e) Plant biostimulants – Determination of dry matter

This hEN specifies a method for determination of dry matter of plant biostimulants using gravimetric determination of the dry residue. This method is applicable for solid and liquid plant biostimulants with dry matter higher than 3 %.

In case of analysis of plant biostimulants, water is usually not considered as a part of the sample and results are generally related to dry matter, which can be calculated by determination of the dry residue (dry matter fraction). For determination of the dry matter content, an individual standard must be developed. This proposal applies modular aspect of standardisation that is used widely in the methodology applied to different environmental matrices. Standards EN 15934, ISO 11465 and methods described in literature (e.g. Robert L. Bradley, Jr.: Moisture and total solid analysis in: S. Suzanne Nielsen: Food Analysis. Springer, fourth edition, 2010) will be considered in developing this standard for plant biostimulants.

The producers will be able to check the quality of their products for compliance with the required legislative limits and competent authorities will have an instrument for an effective control of the regulatory limits. Consumers and environmental stakeholders will profit from the well-established uniform and reliable control of the products applied to soil and to crops. The apparatus needed for the method are widely used in analytical laboratories and the application of this method will not have any excessive demands on the laboratory equipment.

f) Plant biostimulants - Determination of phosphonates

This hEN specifies the method for determination of phosphonates in water extracts of plant biostimulants by ion chromatography.

The proposed hEN will be used for determination of phosphonates in different plant biostimulants. Sample is extracted by water at 20°C. Phosphonates are completely dissolved and determined. A new method based on a simultaneous determination of phosphonates and other ionic substances by ion chromatography with conductivity detector (IC-CD) will have to be developed. The analytical method –ion chromatography- is well established in analytical laboratories. Nevertheless, the method has to be developed because the available published methods are suitable only for inorganic matrices and they cannot be used in the presence of organic matter. A preliminary determination of total phosphorus in aqua regia extracts by ICP-AES can be used as a screening method. If the content of aqua regia extractable phosphorus is lower than the legislative limit for phosphonates, then the determination of individual species is not necessary.

The producers will be able to check the quality of their products for compliance with the demanded legislative limits and competent authorities will have a suitable tool for an effective control of the regulatory limits. Consumers and environmental stakeholders will profit from the well-established uniform and reliable control of the products applied to soil and crops.

g) Plant biostimulants - Determination of the inorganic arsenic

This hEN specifies the method for determination of inorganic arsenic (iAs) after a mild oxidative acid extraction. HPLC coupled to ICP-MS is used for determination.

The proposed hEN will be used for determination of inorganic arsenic (iAs) in different plant biostimulants. Sample is treated with a diluted nitric acid and hydrogen peroxide solution in a heated water bath. Arsenic species are

extracted into the solution in which As(III) is oxidized to As(V). The iAs is selectively separated from other arsenic compounds using anion exchange HPLC coupled to the ICP-MS for the determination of the mass fraction of iAs.

The methods described in the available standards are relatively well established in well-equipped analytical laboratories and they are used mainly for food and animal feeding stuff analysis.

A preliminary determination of total arsenic in aqua regia extracts by ICP-AES can reduce the number of the samples where determination of iAs is necessary. (If the content of aqua regia extractable arsenic is lower than the legislative limit for inorganic arsenic than the determination of individual species is not necessary).

Available standards (e.g. EN 16802, EN 15517, EN 16278) will be considered during the development of the standard for plant biostimulants.

The producers will be able to check the quality of their products for compliance with the demanded legislative limits and government will have a suitable tool for an effective control of the regulatory limits. Consumers and environmental stakeholders will profit from the well-established uniform and reliable control of the products applied to soil and crops. But the instruments needed for the method are used only in well-equipped analytical laboratories and the application of this method demands very good staff training. Therefore, preliminary determination of the total arsenic simultaneously with the other elements can give an indication of the necessity of using the more expensive iAs determination. This solution can further minimize the cost for the analyses.

2. Objective

The objective of the current open calls is the recruitment of proficiency test providers for six inter-laboratory studies:

Table 1 – Calls and objectives

Call no.	Validation of the methods in the standardization project	Project description	Project leader in WG 4
1	Plant biostimulants – Determination of specifics elements - Part 2: Determination of total content of Cd, Pb, Ni, As, Cr, Cu and Zn)	1.2 a and b[a)]	Jiří Zbíral (UKZUZ)
2	Plant biostimulants – Determination of specifics elements - Part 3: Determination of mercury	1.2 a and c[a)]	Jiří Zbíral (UKZUZ)
3	Plant biostimulants – Determination of chromium (VI)	1.2 d	Chiara Manoli (ILSA s.p.A.)
4	Plant biostimulants – Determination of dry matter	1.2 e	Jiří Zbíral (UKZUZ)
5	Plant biostimulants – Determination of phosphonates	1.2 f	Miroslav Florán (UKZUZ)
6	Plant biostimulants – Determination of inorganic arsenic	1.2 g	Eva Čižmárová (UKZUZ)

a) This project includes the digestion according to part 1 Digestion by aqua regia for subsequent determination of elements

The work of the proficiency test provider implies the coordination of the work of the participating laboratories, which participate in the ILS. Tight cooperation with the individual WG 4 project leader is required.

3. Execution

3.1 Tasks

The tasks of the proficiency test provider are:

a) Nomination of a project leader for the proficiency test. He coordinates the tests, is contact partner for the relevant WG4 project leader and ensures the involvement of the producers in the inter-laboratory tests.

b) Reception of a draft test method from the WG 4 project leader.
c) Identification of the participating laboratories[1].
d) Selection of common products on the market which can be used for sampling.
e) Preparation of a validation plan and confirmation in WG 4.
f) Preparation of samples, where necessary spiking of samples.
g) Instruction of the participating laboratories.
h) Distribution of samples to the participating laboratories for the inter-laboratory test.
i) If necessary assist participating laboratories in performing the tests.
j) Collect, review and evaluate results from the participating laboratories.
k) Proposal for an improvement of test method.
l) Compile the validation report.
m) Supply of data for the Annex of the hEN "Results of a validation study"
n) Presentation and discussion in WG 4.
o) Evaluation of the comments of WG 4 and the HAS-Consultant.
p) Support the relevant part of the Enquire draft and progress report.

3.2 Timeframe

The proficiency test provider must adhere to the following schedule for the milestones in the standardization project:

- S = 2. 6. 2020 Start of the standardization project
- S + 12 months Start of inter-laboratory studies
- S + 22 months Release of inter-laboratory studies
- S + 23 months Support WG 4 project leader and Convenor in the discussion with the HAS Consultant

3.3 Open calls for the validation of methods (proficiency test providers for six inter-laboratory studies)

The CEN/TC 455 *Plant biostimulants* WG 4 Secretariat launches the current open call for the recruitment of proficiency test providers for every of the six inter-laboratory studies to:

- validate the methods of the standardization projects according to Table 1 and subclause 1.2 a-g.
- perform the validation, management and evaluation of the inter-laboratory studies according to the tasks 3.1.
- identify and coordinate of the work of the participation laboratories, which participate in the ILS.
- cooperate trustfully with the SA project leader (ÚNMZ/ČAS), WG 4 project leaders (Table 1) and the HAS consultant.
- and supply the results timely according to the timeframe 3.2.

The proficiency test provider will be subcontracted by ČAS.

4. Financial support

There will be a financial support from the European Commission and EFTA for the services described in Table 1 and clause 2 and 3.

The financial support from the European Commission and EFTA is based on the Framework Partnership Agreement (FPA 2014). The subcontractor shall fulfil the conditions of the FPA 2014 (liability, ownership pf results, confidentiality…).

The assignment of the task and execution of the work will be dependent upon European Commission/EFTA funding.

The terms and schedule for payment by the EU/EFTA will be defined in the Specific grant agreement.

[1] The specific agreement with the EC states that a minimum of ten laboratories is required and foreseen for each method. In case this number of laboratories cannot be achieved for justified reasons (method is new, complicated instruments are demanded etc.), WG 4 will have to be asked to adopt a modified validation plan.

5. Criteria for selection

The selection of the applying proficiency test providers is based on the following criteria:

1. PROFILE

a) Institutional background

- Equipment and staff for sample pretreatment, spiking and homogenization available
- Methodology for spiking of plant biostimulants available
- Preliminary tests and tests for homogeneity provided by an accredited laboratory – EN ISO 17025
- Experience in ILS for at least 10 years
- Accredited provider of an ILS according to EN ISO 17043
- Software for results collection and evaluation of ILS available

b) Personal background

- Expertise knowledge of the validated method and validation procedures
- Competence in statistics – evaluation of the results according to ISO 5725-2

c) Management competences. Experience or ability to:

- present complex issues in the given context as a definition in an understandable way
- coordinate a group of experts, ensure the consolidation and integration of all contents provided by the participant of ILS

2. SUPPLY OF RESULTS OF ILS

a) Understanding of tasks and responsibilities

- Number of days of work
- Comprehension of the scope
- Quality of the proposal (clarity, match with description given…)

b) Ability to supply ILS results at specified target dates

- Calendar of the proposal vs expected

3. VALIDATION AND STANDARDIZATION OF THE METHODS

a) Experience in validation and development of methods

- Number of projects
- Number of years

b) Experience in European and/or International standardization

- Number of projects
- Number of years

These criteria will be evaluated by a scoring system. The scores are laid down in Annex 1.

Tenders must score minimum 65% in total. After evaluation, the tenders ill be ranked using the formula below to determine the tender offering best value for money. A weight of 70/30 is given to quality and price.

$$\text{Score for tender X} = \frac{\text{cheapest price}}{\text{price of tender X}} * 30 + \frac{\text{total quality score (out of 100) for all award criteria of tender X}}{100} * 70$$

The formula won't be used for the selection of the convenor. The price will represent 15% of the selection criteria (daily rates, number of work days, travel costs and other costs).

Applicants will be excluded from participating in the call for proposals procedure according to the following **exclusion criteria**:

- The tenders' score is lower than 65% in total

- The offer was received after thedeadline
- The offer is not complete (see the elements requested in section 6)
- The tenders are subject to a conflict ofinterest
- They are in any of the situations described in the exclusion criteria of the *Guide for tenderers Submitting bids in response to a call for tenders published by the Office for Infrastructure and Logistics – Brussels (OIB)*[2].

6. Replies to tender

Tenders to the calls in clause 3.3 should be sent by email to the Secretary of CEN/TC455/WG 4, Mr Stefan Krebs (see contact details below, krebs@agentura-cas.cz) as soon as possible.

The deadline for all the candidatures is April 20th, 2021. The call for tenders will start on March 16th, 2021 (35 days according to the CEN/CENELEC Management Centre):

Each applicant shall submit the completed form (Annex 2) and the following information in his tender:

a) A CV of the project leader of the validation(ILS).
b) A specified proposal how to supply the results of the validation in time, breakdown of tasks and responsibilities.
c) A signed declaration, by which the candidate certifies not to be in one of the situations described in the exclusion criteria[3].

Please consider that ČAS is not a VAT-payer, so you have to tax the payments[4].

Tenders must be clear and concise, with continuous page numbering, and must be written in English. They must be signed by the tenderers or their duly authorised representative. They also must be perfectly legible so that there can be no doubt as to words and figures.

Late delivery will lead to the non-admissibility of the tender and its rejection from the award procedure for this contract.

The selection and appointment of the tender will be conducted by Stefan Krebs (ÚNMZ/ČAS, Secretary of CEN/TC 455/WG WG 4), Samantha Gagnon (AFNOR, Secretary of CEN/TC 455), Benoît Planques (Chairperson of CEN/TC 455), and Alessia Gaetani (Technical Project Manager CEN/CENELEC Management Centre) and, if possible, a member from the EC.

Czech Standardization Agency (ČAS)
Stefan Krebs
Secretary of CEN/TC 455/WG 4 on behalf of
ÚNMZ krebs@agentura-cas.cz
Management of European Projects
Email:

Biskupský dvůr 1148/5, 110 00 Praha 1
Czech Republic
Tel.: +420 727 825 546
NUTS -Code: CZ010

Enclosure:
Annex 1 –Scoring system for the selection criteria for proficiency test providers
Annex 2 – Application form

[2] https://ec.europa.eu/oib/doc/tenders-submission-guide_en.pdf, 2.2.3.2
[3] https://ec.europa.eu/oib/doc/tenders-submission-guide_en.pdf , 2.2.3.2
[4] ÚNMZ/ČAS is a governmental organisation tax exempt. When signing a contract with ČAS, the subcontractor will need to cover the tax payments.