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Plant biostimulants - Sampling and sample preparation - Part 1: Sampling

Pflanzen-Biostimulanzien - Probenahme und Probenvorbereitung - Teil 1: Probenahme

Biostimulants des végétaux - Échantillonnage et préparation des échantillons - Partie 1 : Échantillonnage

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (CEN/TS 17702-1:2022) has been prepared by Technical Committee CEN/TC 455 "Plant Biostimulants", the secretariat of which is held by AFNOR.

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 has been prepared under a Standardization Request given to CEN by the European Commission and the European Free Trade Association.

The CEN/TS 17702 series, *Plant biostimulants* — *Sampling and sample preparation*, consists of the following parts:

- Part 1: Sampling;
- Part 2: Sample preparation.

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 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, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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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 fertilizing products ("FPR" or "Fertilising Products Regulation").

This standardization request, presented as M/564, also contributes to the Communication on "Innovating for Sustainable Growth: A Bio economy for Europe". The Working Group 1 "Sampling", 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.

This document covers the following aspects of sampling, derived from EN 1482-1:2007, *Fertilizers and liming materials* — *Sampling and sample preparation* — *Part 1: Sampling* and documents indicated. This document is presented in a form adapted to the specificity of plant biostimulants. The titles of the standards are given in the Bibliography.

From a technical point of view, sampling is generally defined as the withdrawal operation, of the part of a "mass", of such dimensions that the properties found in the sample taken are, within the limits of statistical acceptability, the same as those of the mass of origin (representativeness of the sample). In other words, the ultimate purpose of sampling is to allow the collection of representative portions of plant biostimulants to be subject to analysis. Therefore, it fundamentally affects the significance and reliability of the analytical results themselves.

The final results, in fact, must as far as possible refer to the state and conditions in where the material is found at the time of collection, therefore, care must be taken to avoid or minimize possible modifications to the chemical, physical and biological properties of the sample during or after sampling.

In conclusion, for a correct sampling, it is necessary that the sampling and collection of samples take place quickly, if possible, taking necessary precautions to ensure that they are representative of the plant biostimulants to be analysed and that the samples taken are stored in appropriate way. The surfaces, containers and instruments used must be clean and dry.

Furthermore, remember the protection of health and safety in places of work, and that every intervention must be carried out in compliance with the defined prevention and protection measures (including the use of any suitable personal protective equipment (PPE)), in particular a careful reading of the labels on the product and where available on the safety data sheet.

Figure 1 gives a schematic diagram of the sampling and sample preparation process.

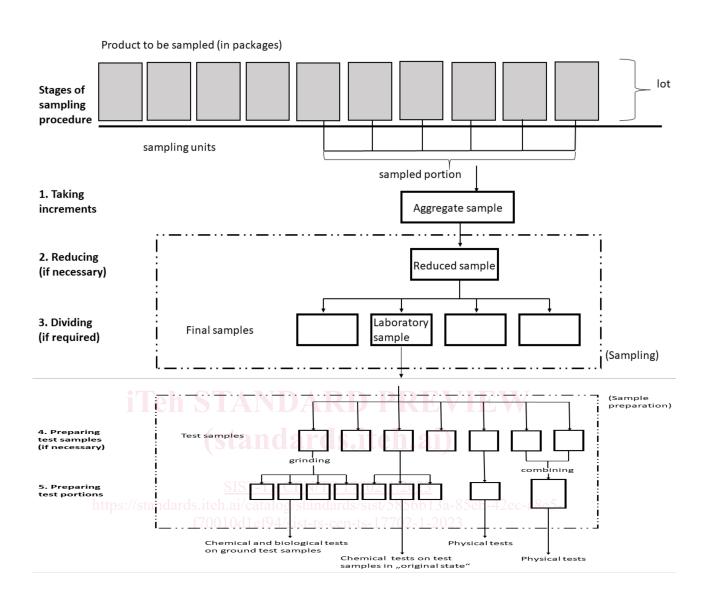


Figure 1 — Schematic diagram of sampling and sample preparation process for solid plant biostimulants

1 Scope

This document specifies sampling plans and methods of representative sampling of plant biostimulants to obtain samples for physical, chemical and biological analysis.

It is applicable to the sampling of lots of plant biostimulants supplied or ready for supply to third parties, as such, or in smaller lots.

It is also applicable to the sampling of blends of fertilizing products where plant biostimulants are main part of the blend. Otherwise, deliverables of sampling relevant for the main part of the blend apply.

This document is intended to be used by manufacturers, buyers and competent authorities to obtain samples prior to transport and supply it to a laboratory for testing.

NOTE This document is applicable to the category of EU fertilizing product (plant biostimulants) in the meaning of the Regulation (EU) 2019/1009.

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

aggregate sample

combination of all increments from the lot

3.2

division

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process of producing a number of representative smaller portions, approximately equal in mass to each other, from a larger mass 70010d1ef94/sist-ts-cen-ts-17702-1-2023

3.3

final sample

representative part of the reduced sample or, where no intermediate reduction is required, of the aggregate sample

Note 1 to entry: Often, more than one sample is prepared, at the same time, from the reduced sample (or from the aggregate sample). One or more of these final samples will be used as a laboratory sample or as laboratory samples, while others may be stored for reference purposes.

3.4

increment

representative quantity of material taken from a sampling unit

Note 1 to entry: This may be constituted from a number of sub samples.

3.5

laboratory sample

final sample intended for laboratory inspection or testing

3.6

lot

total quantity of material, assumed to have the same characteristics, to be sampled using a sampling plan

3.7

reduced sample

representative part of the aggregate sample obtained by a process of reduction in such a manner that the mass is at least the mass of the required final samples

3.8

reduction

process of producing a representative smaller mass of plant biostimulant from a larger mass, with the remainder being discarded

3.9

sampling unit

defined quantity of material having a boundary (e.g. a container)

3.10

sampled portion

quantity of a material consisting of all the sampling units from which increments are to be taken and having characteristics presumed to be uniform

4 Sampling plans and quantitative data

4.1 Principle

The sampling plans given in this document are not based on strict statistical principles, but samples obtained by following the procedures described in this clause shall be considered to be representative of the original lot or sampled portion.

This clause specifies sampling plans for the evaluation of deliveries of plant biostimulants as well as statutory control plans which have to be followed in certain circumstances.

According to available resources, the plant biostimulants are not supplied in other than packaged form (up to 1 000 kg or 1 000 l). Therefore this document specifies principles for those cases. Nevertheless, if plant biostimulants were delivered in larger packages and containers or in bulk, the principles of EN 1482-1 should be applied accordingly.

For statutory control and the simple commercial evaluation of a small quantity of plant biostimulant, one final sample is sufficient, but this may subsequently be divided into a number of identical samples.

The number of sampling units from which increments are to be taken depends on the size of the lot.

No incremental samples are taken at microbial plant biostimulants – in order to preserve the sensitive content and maintain its properties intact avoiding possible contamination. Thus, the original package or container itself shall be considered a final sample.

4.2 Sampling plans

4.2.1 Determination of the number of sampling units which form the sampled portion

4.2.1.1 General

The number of sampling units from which increments are to be taken depends on the size of the lot.

4.2.1.2 Plant biostimulant in packages or containers up to 50 kg or 50 l

The sampling unit is a package or container and the number of individual packages (containers) from which incremental samples are to be taken should be in accordance with Table 1. For packages smaller than 1 kg (1 l) each, it might be necessary to increase the number taken to ensure a sufficiently large aggregate sample.

Lot size	Minimum number of sampling units
4 or fewer packages	All packages
More than 4 up to 10 packages	4 packages
More than 10 up to 400 packages	The nearest whole number above the square root of the number of packages
More than 400 packages	20

Table 1 — Number of individual packages (containers) from which incremental samples are to be taken

4.2.1.3 Plant biostimulant in packages or containers of more than 50 kg or 50 l and up to 1 000 kg or 1 000 l

Sampling units are mostly larger containers such as Intermediate Bulk Containers (IBC's). The number of sampling units from which incremental samples should be taken depends on the total mass present. The number of sampling units to be sampled should be in accordance with Table 2.

Table 2 — Number of sampling units from which incremental samples are to be taken

Lot size	Minimum number of sampling units
25 t (25 m ³) or less (Sta	indards.iteh ^{.10}
More than 25 t (25 m ³) and up to 400 t (400 m ³) <u>SIS</u>	The nearest whole number above the square root of <u>I-TS CEN/TS</u> the number of packages
More than 400 t (400 m ³)	catalog/standards/sist/585(40 /3a-85eb-42ec-a8e5- lef94/sist-ts-cen-ts-17702-1-2023

4.2.2 Identification of the sampling units to be sampled

Identify the packages in the lot or sampled portion consecutively and, by using a source of random numbers, select the packages from which incremental samples are to be taken and mark them.

For microbial plant biostimulants – identify the packages in the lot or sampled portion consecutively and, using a source of random numbers, select five packages which are to be taken as final samples.

4.2.3 Collection of increments

4.2.3.1 General

All incremental samples shall be of approximately the same mass or volume.

4.2.3.2 Solid plant biostimulants

Collect the relevant number of increments from each of the selected packages (sampling units – 4.2.2), by the use of a divider (5.2 or 5.3), by the use of a spear (5.4) or by the manual method (5.5).

4.2.3.3 Fluid plant biostimulants

Follow the appropriate procedure described in 5.6.

4.3 Quantitative data

4.3.1 Mass of increments

Increments should normally be of at least 250 g (250 ml) each. For packages of 1 kg (1 l) or smaller, the entire contents is taken as the increment.

4.3.2 Mass of aggregate/reduced samples

Combine and mix all the collected increments. When necessary, reduce the aggregate sample as described in Clause 5, so that the final mass for chemical or biological testing is at least 2 kg (2 l) and for physical testing at least 4 times the maximum amount required for the physical test method.

4.3.3 Mass of final sample

The mass of each final sample for chemical or biological analysis shall be at least 500 g. For physical testing the mass is dependent on the test(s) to be carried out.

5 Incremental sampling methods

5.1 General

Packages of solid plant biostimulants may be sampled by a process of reduction (see 5.2 and 5.3), starting with the total contents of the package, or by spear sampling (see 5.4.) from the selected packages but the latter only when the product is homogenous. The packages (including IBC's) may be sampled by emptying the contents as in the method described in 5.5.

Containers up to 20 l of fluid plant biostimulants may be sampled by a process of pouring into collecting vessel immediately after homogenization. All containers may be sampled by a process of filling of tube, sucking by sampling pump or by sampling at source.

The sampling apparatus shall be clean, dry and inert (i.e. fabricated of materials that will not affect the characteristics of the plant biostimulant to be sampled).

All sampling operations should be carried out in such a way as to minimize changes to sample properties, e.g. moisture content.

5.2 Solid plant biostimulants in packages – Reduction method using a rotary mechanical sample divider

5.2.1 General

This subclause specifies a method suitable for the reduction of a mass of a solid plant biostimulant to a smaller quantity which forms the incremental sample from the package.

The method may also be used to prepare reduced samples, final samples or laboratory samples.

By choosing suitable equipment, the method is applicable to the reduction of a sample of any mass above a minimum defined by the size and number of particles.

5.2.2 Principle

Passage of the material through a rotary mechanical sample divider. Collection of the fractions followed by rejection or recombination of some of the fractions to give the desired quantity for the incremental sample.

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5.2.3 Apparatus

5.2.3.1 General

Rotary mechanical sample dividers are of several basic types. They can operate by collecting subsamples from a falling stream (cutter type) or by extracting a helical ribbon from a falling cylindrical curtain, such as is created by allowing the plant biostimulant to fall onto the apex of a cone distributor. In the case of the cutter type, each sub-sample consists of a complete cross-section of the stream.

The sample divider is fed from a hopper fitted with one of a series of interchangeable orifices so that the criteria below can be met.

A standard divider operates at a rotational frequency of about 60 rounds \min^{-1} but this rotational frequency can be increased up to about 360 rounds \min^{-1} , the variance of the sample division being reduced as a larger number of sub-samples are taken. However, care is needed to ensure that there is no bias because of larger particles bouncing on the rapidly moving edges of the sample receiver or because particles are shattered.

The hopper can be on the vertical axis of the receiver, feeding via the distributing cone, or off-centre when no such cone is needed.

Examples of rotary sample dividers are shown in Annex A, Figures A.1, A.2 and A.3.

All sample dividers shall conform to the following basic requirements.

- a) The effective opening of the cutter or slot shall be at least three times, but preferably five times, the maximum particle size of the plant biostimulant to be divided. In practice, this means a minimum dimension of at least 15 mm.
- b) The divider shall be constructed and operated in such a manner that every particle has an equal opportunity of being included in the sub-sample. Provided that all parts of the stream are sampled in due proportion, an unbiased sample should be obtained.

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c) During reduction, there shall be at least 50 rotations of the cup(s) so that at least 50 increments are taken from the gross sample at each stage of division.

5.2.3.2 Test for bias (referred to previous clause: Rotary mechanical sample dividers)

A suitable test for bias is given in Annex B.

5.2.4 Procedure

5.2.4.1 General

Follow the procedure specified in 5.2.4.2, or 5.2.4.3 depending on the mass of the bulk sample.