SLOVENSKI STANDARD
oSIST prEN 17723:2023

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Rastlinski biostimulanti - Določevanje klorida

Plant biostimulants - Determination of chloride

Pflanzen-Biostimulanzien - Chloridbestimmung

Biostimulants des végétaux - Dosage des chlorures

Ta slovenski standard je istoveten z: **prEN 17723**

ICS:

65.080 Gnojila Fertilizers

oSIST prEN 17723:2023 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 17723

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ICS

Will supersede

English Version

Plant biostimulants - Determination of chloride

Biostimulants des végétaux - Dosage des chlorures

Pflanzen-Biostimulanzien - Chloridbestimmung

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

If this draft becomes a European Standard, 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.

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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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Contents

European foreword

This document (prEN 17723:2023) has been prepared by Technical Committee CEN/TC 455 “Plant Biostimulants”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN enquiry.

This document will supersede CEN/TS 17723:2022.

This document has been prepared under a Standardization Request given to CENELEC by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s) / Regulation(s).

For relationship with EU Directive(s) / Regulation(s), see informative Annex ZA, which is an integral part of this document.

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 the European Parliament and of the Council of 5 June 2019 laying down rules on the making available on the market of EU fertilising products (“FPR” or “Fertilising Products Regulation”). This standardization request, presented as M/564 and M/564/Amd1, also contributes to the Communication on “Innovating for Sustainable Growth: A Bio economy for Europe”. Working Group 5 “Labelling and denominations”, was created to develop a work program as part of this standardization request.

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 commercialization of the European biostimulant industry.

To improve the market of EU Plant Biostimulants products, they should be labelled in accordance with the labelling requirements set out in Annex III of Regulation 2019/1009 [5], which prescribes that the phrase “poor in chloride” or similar may only be used if the chloride (Cl^-) content is below 30 g/kg of dry matter.

This means that the manufacturer has the possibility to note on the label that the plant biostimulant is “poor in chloride”, only if the manufacturer has done the analysis and the chloride content is well below 30 g/kg of dry matter.

The trend of the last few years is to reduce the use of harmful chemicals both for operators and the environment, favouring the use of the most advanced technologies that have a lower impact; therefore, the potentiometric is preferable to other techniques.

Furthermore, the potentiometric method is not influenced by interferences due to colour or turbidity of the sample solution.

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 a potentiometric method for the determination of chloride (Cl^-) content in the presence or in the absence of organic material. This method is applicable to plant biostimulants.

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.

prEN 17702-2:—,[1] *Plant biostimulants - Sampling and sample preparation - Part 2: Sample preparation*

prEN 17724:—,[2] *Plant biostimulants - Terminology*

EN ISO 3696:1995, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in prEN 17724:—[2] and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org

3.1
chloride content of plant biostimulants
mass fraction of chlorides, expressed in g/kg of dry matter, determined in accordance with the procedure described in this document

4 Sampling

Sampling is not part of this method specified in this document. A recommended sampling method is given in prEN 17702-1:—[3].

Laboratory sample preparation shall be carried out in accordance with EN 17702-2:—[1]. Homogeneous samples are recommended.

[1] Under preparation
[2] Under preparation
[3] Under preparation

5 Principle

5.1 General

Soluble chlorides are extracted from the sample with water containing nitric acid. The chloride content of the solution is titrated with silver nitrate standard solution and the end point is determined potentiometrically by using either a silver ion selective electrode plus a reference electrode or a combined electrode.

5.2 Interferences

Maintain the sample at a lower pH can avoid the silver ion interact with OH^- to form silver oxide (Ag_2O) precipitation, minimize other anionic ions interference with the AgCl formation, and improve the binding between the titrant and the analyte.

6 Reagents

Use only reagents of recognized analytical grade.

6.1 Water

Water free from chlorides in compliance with quality grade 2, according to EN ISO 3696:1995 shall be used.

6.2 Nitric acid solution

Nitric acid solution: dilute 1:1 concentrated nitric acid (70 %) with water (6.1).

6.3 Silver nitrate standard solution, *c* = 0,1 mol/l solution

Silver nitrate solution, 0,1 mol/l in water (6.1). Use commercially available silver nitrate solution with certified concentration or prepare and standardize a solution according to the instructions in Annex A.

7 Apparatus

Normally, automatic titrators are used. This method describes manual titrations.

Ordinary laboratory equipment and the following are required:

7.1 Electromagnetic stirrer and polytetrafluoroethylene (PTFE) coated magnetic bars.

7.2 Glass burettes class A reading with accuracy of 0,02 ml or automatic pipette with accuracy of 0,002 ml.

7.3 One-mark volumetric glass flask Class A, capacity 150 ml, 500 ml and 1 000 ml and graduated glass flask Class A, capacity 100 ml.

7.4 Glass beaker, capacity 300 ml.

7.5 Glass conical (Erlenmeyer) flask, capacity 250 ml.

7.6 Potentiometer, with reading accuracy of 0,1 mV, equipped with an electrode suitable for the measurement of chloride (e.g. a silver electrode) and a reference electrode (e.g. a mercury(I) sulfate electrode). Alternatively, use a suitable combination electrode.

8 Procedure

8.1 Measurement temperature

In order to reduce the effects of thermal and electric hysteresis, take care that the temperatures of the electrodes, the water used for washings, the standard solutions and the test solution are as close to each other as possible. The temperatures of the standard solutions and the test solution shall not differ by more than 1 °C. The measurement temperature should be 25 °C whenever possible.

8.2 Preparation of the test portion

Homogenize solid samples without addition of water. Mix liquid samples well.

Depending on the expected chloride content, with an accuracy of 0,1 mg, weigh 1 g to 10 g of sample into a beaker (7.4). Add 50 ml of water (6.1) and 5 ml of nitric acid solution (6.2). Mix for 30 min with magnetic stirrer (7.1).

8.3 Blank test

Prepare a blank test in the same manner (8.2) containing the reagents but omitting the sample.

8.4 Titration

8.4.1 Test portion (sample)

Immerse the electrode(s) (7.6) in the sample solution (8.2). Mix the solution with a magnetic stirrer (7.1) and, after having verified the zero of the apparatus, note the value of the starting potential. Titrate using an automatic pipette or manually (7.2) by adding 0,1 ml portions of silver nitrate standard solution (6.3) and read the potential after each addition.

Allow the potentiometer to stabilize 1 min to 2 min between each addition. If the potentiometer, at the beginning of the titration, indicates a high chloride level (low reading), the silver nitrate standard solution may be added in larger portions (1 ml to 2 ml); close to the equivalence point, titrate using 0,1 ml portions.

At the equivalence point the difference between two successive voltage readings is largest.

If the experiment is done by an automatic titrator, the instrument will automatically display the volume value at the equivalent point after the end point is reached.

8.4.2 Blank test

Titrate the blank test (8.3) in the same manner (8.4.1).

8.5 Recovery test

Before carrying out the estimations, check the accuracy of the method by adding a known amount of sodium chloride at the expected chloride level to the sample and proceed from (8.1).

Titrate the recovery test in the same manner (8.4.1).

9 Expression of results

Many titrators automatically calculate the equivalence point and the chloride concentration.

In manual titrations, it is usually sufficient to regard as the consumption v_s (samples) and v_0 (blank test) of the titrating solution at the equivalence point the volume immediately before the largest voltage difference.

If a more exact equivalence point is needed, calculate the equivalence point from the more sophisticated formula in Annex B.

Calculate the chloride content *Cl*, expressed as g/kg of fresh matter, of the sample as received for analysis using the formula:

$$Cl_{fm} = \frac{\left[\left(v_s - v_0\right)*a*M_{Cl}\right]}{m}$$

where

Cl_{fm} is the chloride content expressed as g/kg of fresh matter;

v_s is volume consumed (ml) of the titrating solution at the equivalence point, the sample (8.4.1);

v_0 is volume consumed (ml) of the titrating solution at the equivalence point, the blank test (8.4.2);

a is concentration of the titrating solution (mol/l);

M_{Cl} is molar mass of chlorine = 35,453 g/mol;

m is the mass, in grams, of the test portion (g).

Give the result as the average of two replicated determinations done on two different test portions from two different samples, with an accuracy of two figures, e.g. 12 g/kg, 1,2 g/kg, 0,12 g/kg.

To express the result in g/kg of dry matter, use the following formula:

$$Cl_{dr} = \frac{Cl_{fm} \cdot 100}{w_{dr}}$$

where

Cl_{dr} is the chloride content expressed as g/kg of dry matter;

w_{dr} is the dry matter content of the sample, expressed as mass fraction in percent (%).

If a recovery test (8.5) was carried out, calculate the result for the sample to which chloride (Cl^-) had been added (*Cl^-added*) and calculate the recovery from the formula:

$$Recovery\left(\%\right) = \frac{\left[\left(Cl^-_{added} - Cl^-_{sample}\right)\right]}{\mathrm{Cl}^-_{amount}} * 100$$

where

Cl^-_{added} is the amount found in the sample to which chloride had been added;

Cl^-_{sample} is the amount found in the sample without addition;

Cl^-_{amount} is the amount of chloride added to the sample.

Consider correcting the result of the sample with the recovery factor if the recovery is less than 95 %.