CEN/TC 223/WG 4 "Analytical methods"
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Document type	Related content	Document date	Expected action
Project / Draft		2022-05-13	**INFO**

Description

Please find enclosed the 1st working draft of WI 00223xxx, Soil improvers and growing media — Determination of the chloride content.

As agreed during the last meeting of CEN/TC 223/WG 4 on 2022-04-27, the 1st working draft and the forms for the activation of the work item will be sent to CEN/TC 223 as soon as the call for tender for the project leaders is completed and the project leaders are known.

CEN/TC 223

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Soil improvers and growing media — Determination of the chloride content

Einführendes Element — Haupt-Element — Ergänzendes Element

Élément introductif — Élément central — Élément complémentaire

ICS:

CCMC will prepare and attach the official title page.

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European foreword

This document (prEN 00223xxx:2022) has been prepared by Technical Committee CEN/TC 223 “Soil improvers and growing media”, the secretariat of which is held by NEN, in accordance with CEN/TC 260 “Fertilizers and liming materials”.

This document is a working document.

This document has been prepared under a standardization request given to CEN by the European Commission and the European Free Trade Association.

1 Scope

This document specifies a method for the determination of the chloride content in growing media and soil improvers by potentiometric titration.

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 13040-1:—[1], *Soil improvers and growing media – Sample preparation – Part 1: Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory bulk density*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TS 00223137:—[2] apply.

4 Principle

The chlorides, dissolved in water, are precipitated in an acidified medium by an excess of standard solution of silver nitrate. The chloride concentration in the samples is quantified by potentiometric determination. To detect the end point, the voltage between a reference electrode on the one hand and the silver electrode or an ion selective electrode on the other hand, is tracked. This voltage depends on the logarithm of the chloride ion activity. If it is plotted, in mV, on the ordinate and the silver nitrate solution, in ml, on the abscissa, the point of inflection of the curve obtained in this manner is the equivalence point. Equivalent methods of evaluation are permitted [2, 4].

Other determination methods for chloride e.g. ion chromatography, discrete analyser, continuous flow analyser may be used once verification of the chosen analytical method has been performed and the applicability has been proven.

5 Reagents

All reagents used shall be of recognised analytical quality and water with a specific conductivity not higher than 0,2 mS/m at 25 °C, free from the elements to be determined.

5.1 Nitric acid, substance concentration c = 16 mol/l.

5.2 Nitric acid solution, c = 10 mol/l.

Mix 630 ml of nitric acid (5.1) with 370 ml of water.

5.3 Silver nitrate standard solution, c = 0,1 mol/l.

Stock solution or preparation from AgNO3 salt; since this salt is hygroscopic and cannot be dried without risk of decomposition, it is advisable to weigh out approximately 17 g, dissolve in water and fill up the volume to 1 l.

A commercially available silver nitrate standard solution might be used.

[1] Under preparation. Stage at the time of publication: prEN 13040-1:20**.
[2] Under preparation. Stage at the time of publication: prCEN/TS 00223137:20**.

5.4 Polyvinyl alcohol (PVA) solution, acidified with nitric acid**.**

Mix 2 g of polyvinyl alcohol with 400 ml of hot water (approx. 90 °C) in a 1 000 ml graduated flask and let it cool to room temperature. Add 10 ml of nitric acid (5.2) and fill up with water to 1 000 ml.

It is recommended to use an acidified protector colloid medium for the potentiometric titration.

5.5 Sodium chloride standard solution, c = 0,1 mol/l.

5.6 Sodium chloride control test sample.

Prepare a control test sample by dissolving 0,989 1 g ± 0,000 1 g of sodium chloride in 1 000 ml demineralized water and homogenize the solution. This control test sample contains 0,6 g chloride.

6 Apparatus and equipment

Usual laboratory glassware and equipment, in particular, the following.

6.1 Magnetic stirrer.

6.2 Pipettes.

6.3 Graduated flask, capacity 500 ml.

6.4 Plastic bottles or containers, sufficiently large (500 ml to 2000 ml) with screw cap to accommodate the volume of the sample, extractant and at least 10 % air volume.

6.5 Potentiometric titration system, incl. millivolt meter and burette or other liquid handling/dosing unit; optional incl. motoric stirrer.

6.6 Silver electrode or ion selective electrode for chloride.

6.7 Reference electrode with chloride-free salt bridge.

6.8 Analytical balance with an accuracy of 10 mg.

6.9 Filter paper, cellulose-based ashless types, with a medium pore size of approximately 8 µm and diameter of 150 mm.

NOTE Centrifugation is an acceptable alternative.

6.10 pH testing device, e.g. pH meter, pH indicator paper, or pH rapid test.

6.11 Shaking or mixing machine, capable of holding the plastic bottles or containers (6.2) and maintaining the sample in suspension without damaging the structure of the sample. The use of a horizontal table shaker is recommended.

7 Test Sample

Prepare the laboratory sample in accordance with EN 13040-1:—, clause 9, and determine the laboratory bulk density of the sample in accordance with EN 13040-1:—, Annex A.

8 Procedure

8.1 Preparation of the test portion and the sample solution

8.1.1 Extraction of materials in solid form

8.1.1.1 Test samples passing through a 20 mm sieve

Take a weight equivalent 60 ml of the sample volume to the nearest 1 g and transfer to the container (6.4). Add 300 ml of water, secure the cap and shake for 1 h on the shaking machine (6.11) at (22 ± 3) °C.

8.1.1.2 Test samples passing through a 40 mm sieve

Take a weight equivalent 250 ml of the sample volume to the nearest 1 g and transfer to the container (see 6.4). Add 1250 ml of water, secure the cap, and shake for 1 h on the shaking machine (see 6.11) at (22 ± 3) °C.

8.1.1.3 Test samples > 40 mm

Take a weight equivalent to 250 ml of the sample volume to the nearest 1 g and transfer to a 2000 ml container (6.4). Add 1250 ml of water, secure the cap and shake for 1 h on the shaking machine (6.11) at (22 ± 3) °C.

8.1.2 Extraction of material in pre-shaped form

Take a weight equivalent to 150 ml of the sample volume to the nearest 1 g and transfer to a 1000 ml container (6.4). Add 750 ml of water, secure the cap and shake for 1 h on the shaking machine (6.11) at (22 ± 3) °C.

Before filtration pour out the fluid and the pre-shaped sample in a beaker. Squeeze the pre-shaped sample in the beaker as much as possible by applying a pressure to the sample with help of a plastic masher or pestle.

8.1.3 Filtration

Filter through filter paper (see 6.9) discarding the first 10 ml. In some cases, paper filtration is too slow or even impossible. In such cases alternative procedures, (e.g. centrifugation, glass fibre filter papers) may be used to obtain a clear supernatant.

8.2 Calibration of the system (titer factor)

The titer factor of the silver nitrate standard solution (5.3) is determined by titrating 5 ml of sodium chloride standard solution (5.5), diluted with 60 ml of water in a beaker. Then add 5 ml of PVA solution (5.4). The titration is repeated three times. By the mean value of the used volume of the silver nitrate standard solution (5.3) calculate the titer factor.

The titer factor shall be calculated according to the manual of the potentiometric titration system (6.5).

NOTE The volumes of the sodium chloride standard solution and the water can be varied, provided that the volume ratio stays constant.

8.3 Determination

Take an aliquot part of the sample solution (see 8.1) containing not more than 0,150 g of chloride, e.g. 100 ml. Then add 5 ml of PVA solution (5.4). Test for the pH (6.10) and if the pH of the sample solution is > 2, adjust the pH to < 2 by adding dropwise nitric acid (5.2) to the sample solution (see 8.1).

Then add silver nitrate standard solution (5.3) with a burette or comparable liquid dosing unit (6.5). The silver nitrate consumptions depends on the chloride content in the sample solution (see 8.1) and shall be between 2 ml and 20 ml, ideally between 5 ml and 10 ml.

The pH shall be adjusted just under pH 2. Adding too much acid can bias the measurement.

If the sample solution (see 8.1) is not filtrated before determination, it is recommended to use a motoric stirrer within the potentiometric titration system (6.5) to prevent a clogging of the electrodes (6.6, 6.7).

8.4 Control test

Before carrying out the estimations check the accuracy of the method by using an aliquot part of 150 ml of a freshly prepared solution of sodium chloride (5.6). This should result in a mass fraction of chlorides equal to 30 g/kg.

9 Calculation and expression of the results

Calculate the mass fraction of chlorides (Cl), w_{Cl}, in g/l substrate according to equation (1).

$$w_{Cl} = \frac{M_{Cl} \times c_z \times V_z \times F \times V_a \times L_D}{m \times V_b \times 1000} \quad (1)$$

where

V_z is the amount of the silver nitrate standard solution (5.3), in ml;

F is the titer factor (8.2) of the silver nitrate standard solution (5.3);

V_a is the volume of the graduated flask used for the sample preparation (6.3), in ml;

m is the mass of the test portion taken (8.1), in g;

V_b is the volume of the sample aliquot taken for determination (8.3), in ml;

M_{Cl} is the molar mass of chlorine, 35,453 2 g/mol;

c_z is the substance concentration of the silver nitrate standard solution (5.3), in mol/l;

L_D is the laboratory bulk density in g/l according to EN 13040-1.

10 Test report

The test report shall contain the following information:

a) a confirmation note, confirming that the tested sample was taken according to EN 12579 or according to another sampling plan or that no information on how the sample was taken was received by the laboratory;

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

c) the results of the determination, expressed as mg/l sample;

d) details of any operations not specified in the European Standard or regarded as optional, as well as any factor which may have affected the results;

e) the laboratory bulk density.

Bibliography

[1] CEN/TS 00223137, *Soil improvers and growing media Terminology*

[2] DIN 38405-1, *Deutsche Einheitsverfahren zur Wasser-, Abwasser- und Schlammuntersuchung; Anionen (Gruppe D); Bestimmung der Chlorid-Ionen (D 1)*

[3] EN 12579, *Soil improvers and growing media – sampling*

[4] EN 16195, *Fertilizers — Determination of chlorides in the absence of organic material*