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Description

Please find enclosed the 1st working draft on WI 00223107, Soil improvers and growing media — Extraction of calcium chloride/DTPA (CAT) soluble nutrients and elements for your information.

As agreed during the meeting of CEN/TC 223/WG 4 on 2021-11-09, the enclosed 1st working draft and the forms for the adoption of the new work item will be circulated 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

Date: 2021-11

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Soil improvers and growing media — Extraction of calcium chloride/DTPA (CAT) soluble nutrients and elements

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 13651:2021) has been prepared by Technical Committee CEN/TC 223 "Soil improvers and growing media", the secretariat of which is held by NEN.

This document is a working document.

This document will supersede EN 13651:2001.

In comparison with the previous edition, the following technical modifications have been made:

- The title has been changed.
- Principle (4) has been completed;
- Procedure (8) has been updated with new type of test samples;
- Expression of results (10) has been completed inserting conversion factors;
- Methods of analysis (Annex B) have been updated.

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

SAFETY PRECAUTIONS — Care should be taken when handling substances of caustic nature or samples that may contain sharps or is of a dusty nature.

1 Scope

This European Standard specifies an extraction method for the routine determination of calcium chloride/DTPA (CAT-method) extractable nutrients and elements (as listed in annex B) in soil improvers or growing media.

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

A sample is extracted with calcium chloride/DTPA at 22 °C ± 3 °C in an extraction volume ratio of 1+5. The extracted nutrients and elements are determined by various methods as appropriate (see annex B). If soluble calcium is to be determined, a barium chloride/DTPA solution with the same molar concentration can be used.

5 Reagents

5.1 General

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.

5.2 Dihydrated calcium chloride, $CaCl_2.2H_2O$.

5.3 Diethylenetriaminepentaacetic acid (DTPA), $C_{14}H_{23}N_3O_{10}$.

5.4 Hydrocloride acid, *c*(HCl) = 1:1 vol/vol.

5.5 Concentrated extracting solution $CaCl_2$/DTPA, (CAT), dissolve 14,7 g $CaCl_2.2H_2O$ (5.2) and 7,88 g DTPA (5.3) in 800 ml hot water (temperature approximately 80 °C) with stirring on a magnetic stirrer in a 1000 ml beaker. At 75 °C ± 10 °C the reagents will dissolve within 2 h. Allow to cool to ambient temperature. Transfer the solution to a 1000 ml flask and dilute to the mark with water. The solution is stable at room temperature for several weeks. Any precipitation that occurs will disappear with warming and stirring.

5.6 Extracting solution $CaCl_2$/DTPA, (CAT), dilute the concentrated extracting solution (5.4) with water in the proportions one part concentrated solution with nine parts water. The final concentration

[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**.

of the extracting solution should be 0,01 mol/l $CaCl_2$ and 0,002 mol/l DTPA. The pH of the extracting solution should be adjusted if necessary to be between 2,6 and 2,65 by using HCl (5.4).

5.7 Nitric acid, $c(HNO_3)$ = 15 mol/l: ρ ≈ 1,42 g/ml; not less than 65 % mass/volume.

5.8 Nitric acid, $c(HNO_3)$ = 0,5 mol/l, dilute 35 ml nitric acid (5.6) to one litre with water.

6 Apparatus

6.1 General

NOTE It has been found convenient to carefully clean the equipment (e.g. using nitric acid) and keep separate sets of glassware for the determination of Boron, in order to reduce the possibility of within-laboratory contamination.

All glass apparatus and plastic vessels used in the procedure should be subject to an appropriate preparation procedure in order to keep the risk of contamination as low as possible. It is recommended that all vessels (glass and plastic) are cleansed by carefully immersing in warm nitric acid (5.6) for a minimum of 6 h and then rinsed in water.

Rubber stoppers, which may contain traces of metals shall not be used. It is recommended to use plastic or any other stopper free of all substances to be analyzed.

Borosilicate glassware shall not be used if boron is to be determined.

The apparatus consists of the usual laboratory apparatus, and in particular the following:

6.2 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.

6.3 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.

6.4 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.

6.5 Analytical balance with an accuracy of 10 mg.

7 Test sample

Prepare the laboratory sample in accordance with EN 13040-1:—, clause 8, and determine the laboratory bulk density of the sample in accordance with EN 13040-1:—, Annex A.

8 Procedure

8.1 Extraction of material in solid form

8.1.1 Test samples passing through a 20 mm sieve

Take a weight equivalent to 60 ml of the sample volume (EN 13040-1:—, clause 9.2.5) to the nearest 1 g and transfer to the container (6.2). Add 300 ml of extracting solution (5.5), secure the cap and shake for 1 h on the shaking machine (6.3) at 22 °C ± 3 °C.

8.1.2 Test samples passing through a 40 mm sieve

Take a weight equivalent to 250 ml of the sample volume (EN 13040-1:—, clause 9.2.3) to the nearest 1 g and transfer to the container (6.2). Add 1250 ml of extracting solution (5.5) secure the cap and shake for 1 h on the shaking machine (6.3) at 22 °C ± 3 °C.

8.1.3 Test samples > 40 mm

Take a weight equivalent to 300 ml of the sample volume (EN 13040-1:—, clause 9.2.1) to the nearest 1 g and transfer to the container (6.2). Add 1500 ml of extracting solution (5.5), secure the cap and shake for 1 h on the shaking machine (6.3) at 22 °C ± 3 °C.

8.2 Extraction of material in pre-shaped form

Take a weight equivalent to 150 ml of the sample volume (EN 13040-1:—, clause 9.5.3) to the nearest 1 g and transfer to the 1000 ml container (6.2). Add 750 ml of extracting solution (5.5), secure the cap and shake for 1 h on the shaking machine (6.3) at 22 °C ± 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.

8.3 Filtration

Filter through filter paper (6.4) discarding at least the first 10 ml. In some cases, paper filtration is too slow or even impossible. In such cases alternative procedures for obtaining a clear supernatant are acceptable and the technique used shall be reported. The filtered extract is stable for three days in a hermetically closed polyethylene bottle if stored in a refrigerator at 0 °C to 5 °C. The filtrate can be stored for longer periods in a deep freezer at about -18 °C.

NOTE Before using a solution that has been frozen, the thawed solution shall be thoroughly mixed to eliminate gradient separation that occurs on freezing and subsequent thawing.

NOTE The measurement of a blank can be used to check possible contaminations of the extracting solution, glassware and filter paper used.

9 Determination of the extracted nutrients and elements

See Annex B.

10 Expression of results

Subtract the values determined for the reagent blank from those obtained for the samples. All results shall be calculated using the determined laboratory bulk density and expressed in mg/l substrate as received basis.

Phosphorus pentoxide (P_2O_5) shall be calculated by multiplying phosphorus (P) with the factor 2,29. Potassium oxide (K_2O) shall be calculated by multiplying potassium (K) with the factor 1,20. The total extractable nitrogen shall be calculated as the sum of ammonium nitrogen and nitrate nitrogen.

11 Precision

The repeatability and reproducibility of the calcium chloride DTPA soluble nutrient content in at least two separately prepared samples should be in accordance with Tables in Annex A.

A summary of the results of an interlaboratory trial to determine the precision of the method, in accordance with ISO 5725 [2], is given in Annex A.

NOTE The values derived from this interlaboratory trial may not be applicable to concentrations and matrices other than those tested.

12 Test report

The test report shall contain the following information:

a) a reference to this European Standard;

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.

Annex A
(informative)

Results of an interlaboratory trial to determine calcium chloride/DTPA (CAT) soluble nutrients and elements

A.1 Materials used in the interlaboratory comparison study

The validation study of determination of nutrients in growing media were carried out with *** to *** European laboratories on *** materials. The materials selected for the interlaboratory comparison were chosen to represent growing media as broadly as possible.

Table. A.1 provides a list of the material chosen for testing and the selected components.

Table A.1 — Materials tested in the interlaboratory comparison of the method for the determination of NPK in GM

Material	Particle size
1: Green compost	unsieved
2: Perlite, peat	<40 mm
3: Green compost, digestate, peat	< 20 mm
4: Coir	< 40 mm
5: Bark	>40 mm

Annex B
(informative)

Methods of analysis used in the interlaboratory trial

ELEMENT	METHOD
Ammonium-N	5, 6, 7
Nitrate-N	5, 7, 8
Phosphorus	10
Potassium	1, 2, 3, 9
Magnesium	1, 2,9
Sulfate S	1, 9
Sodium	1, 2, 3, 9
Boron	1, 4, 9, 12,13
Copper	1, 2, 9,13
Iron	1, 2,9,13
Manganese	1, 2,9,13
Molybdenum	1, 2, 9, 11,13
Zinc	1, 2, 9,13
Cadmium	1, 2, 9
Nickel	1, 2, 9
Lead	1, 2, 9

Method

1 ISO 11885:1996 Inductively coupled plasma – atomic emission spectrometry [9]

2 ISO 11047:1998 Flame or furnace atomic absorption spectrometry [7]

3 ISO 9964-3:1993 Flame emission spectrometry [6]

4 Hoffmann 1997 Dianthrimide method [10]

5 ISO 5664:1984 Distillation [1]

6 ISO 7150-1:1984 Manual spectrophotometric [3]

7 ISO 11732:1997 Flow spectrophotometric [8]

8 ISO 7890-3:1988 Spectrometric - sulphosalic acid [5]

9 ISO 17294-2:2016 Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of selected elements including uranium isotopes [4]

10 Hoffmann 1966 Ammonium molybdate ascorbic acid/stannous chloride reduction [11]

11 Reference Book 427 1986 spectrophotometric -iron/thiocyanate [12]

12 John M.K. et al., 1975, azomethine-H [13]

13 CEN/TS 00260237:—, Organic and organo-mineral fertilizers - Determination of the content of specific elements by ICP-AES after extraction by water [17]

Alternative methods may be suitable for the concentration range and extract used. The user is to confirm that the method chosen gives results equivalent to those obtained by the methods listed above.

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[17] CEN/TS 00260237:—, *Organic and organo-mineral fertilizers - Determination of the content of specific elements by ICP-AES after extraction by water*