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SIST EN 13651:2002

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EUROPEAN STANDARD

EN 13651

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

Amendements du sol et supports de culture - Extraction des éléments nutritifs solubles dans le chlorure de calcium/DTPA (CAT)

Bodenverbesserungsmittel und Kultursubstrate - Extraktion von in Calciumchlorid/DTPA (CAT) löslichen Nährstoffen

This European Standard was approved by CEN on 11 August 2001.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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## Foreword

This European Standard has been prepared by Technical Committee CEN/TC 223 "Soil improvers and growing media", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2002, and conflicting national standards shall be withdrawn at the latest by March 2002.

The annexes A and B are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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**EN 13651:2001 (E)**

**SAFETY PRECAUTIONS** — Care should be taken when handling 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.

The method is not applicable to liming materials and preformed materials such as mineral wool slabs and foam slabs.

NOTE The requirements of the standard may differ from the national legal requirements for the declaration of the products concerned.

**2 Normative references**

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

EN 13040:1999, *Soil improvers and growing media - Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density*.

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**3 Terms and definitions**

For the purposes of this standard the terms and definitions given in EN 13040 apply.

**4 Principle**

A sample is extracted with calcium chloride/DTPA at  $22\text{ °C} \pm 3\text{ °C}$  in an extraction volume ratio of 1+5. The extracted nutrients are determined by various methods as appropriate (see annex B).

**5 Reagents****5.1 General**

All reagents used shall be of recognised analytical quality and water of grade 2 according to EN ISO 3696.

**5.2 Dihydrated calcium chloride,  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ .**

**5.3 Diethylenetriaminepentaacetic acid (DTPA),  $\text{C}_{14}\text{H}_{23}\text{N}_3\text{O}_{10}$ .**

**5.4 Concentrated extracting solution  $\text{CaCl}_2/\text{DTPA}$ , (CAT),** dissolve 14,7 g  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  (5.2) and 7,88 g DTPA (5.3) in 800 ml hot water (temperature approximately  $80\text{ °C}$ ) with stirring on a magnetic stirrer in a 1000 ml beaker. At  $75\text{ °C} \pm 10\text{ °C}$  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.

**5.5 Extracting solution CaCl<sub>2</sub>/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 of the extracting solution should be 0,01 mol/l CaCl<sub>2</sub> 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.

**5.6 Nitric acid**,  $c(\text{HNO}_3) = 15 \text{ mol/l}$ ;  $\rho \approx 1,42 \text{ g/ml}$ ; not less than 65 % mass/volume.

**5.7 Nitric acid**,  $c(\text{HNO}_3) = 0,5 \text{ mol/l}$ , dilute 35 ml nitric acid (5.6) to one litre with water.

## 6 Apparatus

### 6.1 General

NOTE It has been found convenient to keep separate sets of glassware for the determinations given in annex B, 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 1500 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  $\mu\text{m}$  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 13040:1999, clause 8, and determine the laboratory compacted bulk density of the sample in accordance with EN 13040:1999, annex A.

**EN 13651:2001 (E)****8 Procedure****8.1 Extraction****8.1.1 Test samples passing through a 20 mm sieve**

Take a weight equivalent to 60 ml of the sample volume (EN 13040:1999, clause 8.5) 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  $22\text{ °C} \pm 3\text{ °C}$ .

**8.1.2 Test samples passing through a 40 mm sieve**

Take a weight equivalent to 250 ml of the sample volume (EN 13040:1999, clause 8.3) 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  $22\text{ °C} \pm 3\text{ °C}$ .

**8.1.3 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  $0\text{ °C}$  to  $5\text{ °C}$ . The filtrate can be stored for longer periods in a deep freezer at about  $-18\text{ °C}$ .

**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.

**8.2 Blank**

The reagent blank test shall be carried out in parallel with the determination, by the same procedure as outlined in 8.1.1 or 8.1.2 and 8.1.3, using the same quantities of all the reagents as in the determination but omitting the test portion.

**NOTE** The measurement of a blank is introduced to determine the contribution of the extracting solution, glassware and filter paper used.

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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 compacted laboratory bulk density and expressed in mg/l substrate as received basis.

**11 Precision**

The repeatability and reproducibility of the calcium chloride DTPA soluble nutrient content in separately prepared samples should be in accordance with Tables A.1 to A.6.

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) all the analytical methods used;
- d) the results of the determination, expressed as mg/l fresh sample;
- e) 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;
- f) the laboratory compacted bulk density result.

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