SLOVENSKI STANDARD
SIST EN 15957:2011

01-november-2011

Nadomešča:
SIST-TS CEN/TS 15957:2010

Gnojila - Ekstrakcija fosforja, topnega v amonijevem citratu v nevtralnem mediju

Fertilizers - Extraction of phosphorus which is soluble in neutral ammonium citrate

Düngemittel - Extraktion des in neutralem Ammoniumcitrat löslichen Phosphors

Engrais - Extraction du phosphore soluble dans le citrate d'ammonium neutre

Ta slovenski standard je istoveten z: EN 15957:2011

ICS:

65.080	Gnojila	Fertilizers

SIST EN 15957:2011 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15957

September 2011

ICS 65.080

Supersedes CEN/TS 15957:2009

English Version

Fertilizers - Extraction of phosphorus which is soluble in neutral ammonium citrate

Engrais - Extraction du phosphore soluble dans le citrate d'ammonium neutre

Düngemittel - Extraktion des in neutralem Ammoniumcitrat löslichen Phosphors

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

Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 15957:2011: E

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Foreword

This document (EN 15957:2011) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by March 2012, and conflicting national standards shall be withdrawn at the latest by March 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 15957:2009.

The following changes have been made to the former edition:

a) the CEN Technical Specification has been adopted as a European Standard;

b) the document has been editorially revised.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: 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, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This document specifies a method for the extraction of phosphorus soluble in neutral ammonium citrate.

The method is applicable to all fertilizers in respect of which solubility in neutral ammonium citrate is laid down in Regulation (EC) 2003/2003, Annex I (see [2]).

2 Normative references

The following referenced documents are indispensable for the application 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 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

Extraction of phosphorus at a temperature of 65 °C using a neutral ammonium citrate solution of pH = 7 under the specified conditions.

5 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2. Grinding of the laboratory sample is recommended for homogeneity reasons.

6 Reagents

6.1 Water, distilled or demineralized.

6.2 Neutral ammonium citrate solution, pH = 7, containing 185 g crystallized citric acid per litre, specific gravity 1,09 at 20 °C.

Prepare the reagent as follows:

Dissolve 370 g of crystalline citric acid ($C_6H_8O_7 \cdot H_2O$) in about 1,5 l of water and make an approximately neutral solution by adding 345 ml of ammonium hydroxide solution (28 % to 29 % of NH_3). If the NH_3 concentration is lower than 28 % add a correspondingly larger quantity of ammonium hydroxide solution and dilute the citric acid in correspondingly smaller quantities of water.

Cool and make exactly neutral by keeping the electrodes of a pH-meter immersed in the solution. Add the ammonia, at 28 % to 29 % of NH_3, drop by drop, stirring continuously (with a mechanical stirrer) until obtaining exactly a pH of 7 at a temperature of 20 °C. At this point make up the volume to 2 l and check the pH again. Keep the reagent in a closed container and check the pH at regular intervals.

7 Apparatus

7.1 Beaker, capacity 2 l.

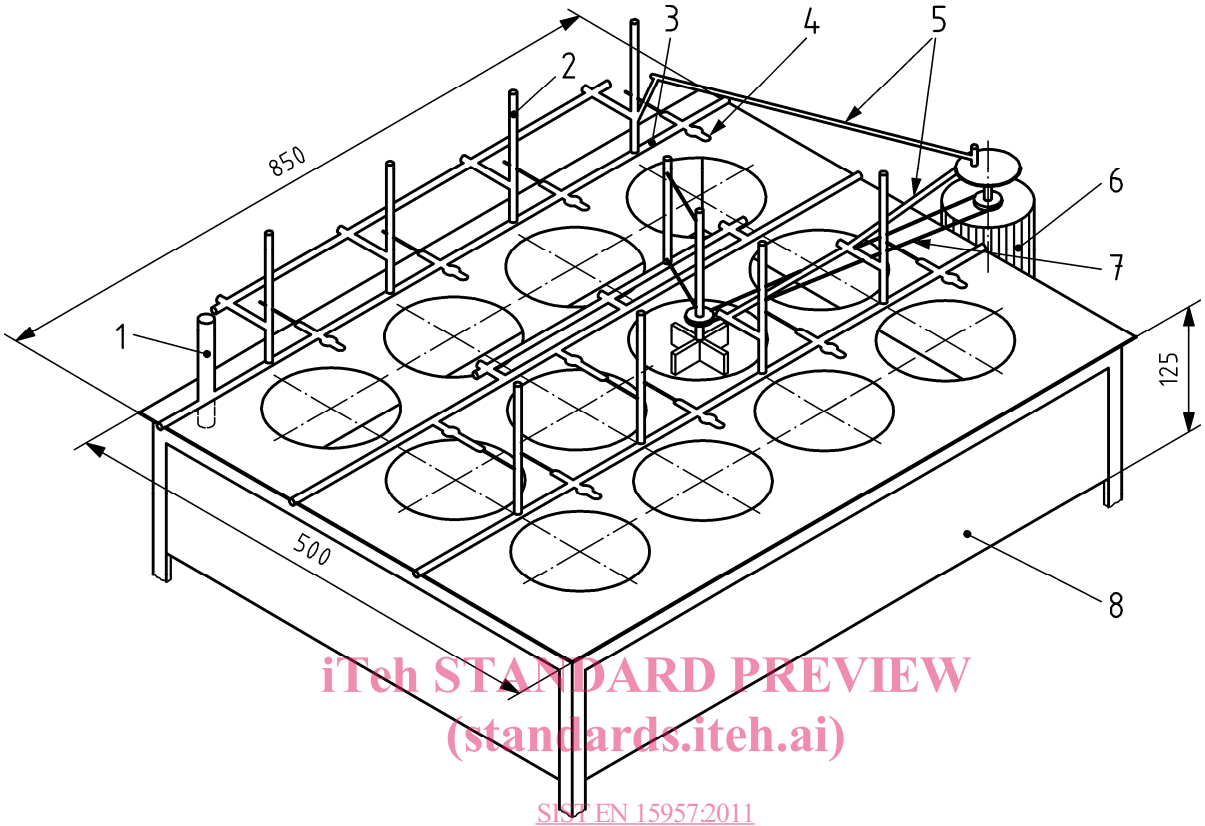
7.2 pH-meter.

7.3 Erlenmeyer flask, capacity 200 ml or 250 ml.

7.4 Graduated flasks, capacity 500 ml and 2 000 ml.

7.5 Water bath, to be set thermostatically at 65 °C, equipped with a suitable stirrer (see Figure 1).

Dimensions in millimetres



Key

1 contact thermometer
2 support for the fixing of clips
3 support for pivot
4 clip
5 arm actuated from eccentric
6 motor with reduction gear
7 belt drive
8 copper bath

Figure 1 — Water bath

7.6 Dry pleated filter, medium speed, phosphate free.

8 Procedure

8.1 Test portion

Transfer 1 g or 3 g of the laboratory sample to be analyzed (see Annex I A and B to the Regulation (see [2]) into a 200 ml or 250 ml Erlenmeyer flask (7.3) containing 100 ml of ammonium citrate solution (6.2) previously heated to 65 °C.

8.2 Extraction

Stopper the Erlenmeyer flask (7.3) and shake in order to suspend the test portion without forming lumps. Remove the stopper for an instant in order to balance the pressure and close the Erlenmeyer flask again. Place the flask in a water bath (7.5) set to maintain the contents of the flask at exactly 65 °C and connect it to the stirrer (see Figure 1). During stirring, the level of the suspension in the flask shall stay constantly below the level of the water in the water bath. If a mechanical stirrer is not available, the flask may be shaken by hand every 5 min.

Regulate mechanical stirring to ensure complete suspension.

After stirring for exactly 1 h, remove the Erlenmeyer flask from the water bath.

Cool immediately under running water to ambient temperature and, immediately, quantitatively transfer the contents from the Erlenmeyer flask into a graduated 500 ml flask (7.4) with a jet of water (wash bottle). Make up the volume with water. Mix thoroughly. Filter through a dry pleated filter (7.6) into a dry container, discarding the first part of the filtrate (about 50 ml).

Collect about 100 ml of clear filtrate.

9 Test report

The test report shall contain at least the following information:

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

b) test method used with reference to this document;

c) test results obtained;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method, which might have influenced the test result(s).