



**SLOVENSKI STANDARD**  
**SIST EN 12945:2014+A1:2016**

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**Nadomešča:**  
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**Sredstva za apnjenje - Določevanje nevtralizacijske vrednosti - Titrimetrijske metode (vključno z dopnilom A1)**

Liming materials - Determination of neutralizing value - Titrimetric methods

Kalkdünger - Bestimmung des Neutralisationswertes - Titrimetrische Verfahren

Amendements minéraux basiques - Détermination de la valeur neutralisante - Méthodes par titrimétrie

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EUROPEAN STANDARD

EN 12945:2014+A1

NORME EUROPÉENNE

EUROPÄISCHE NORM

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ICS 65.080

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English Version

## Liming materials - Determination of neutralizing value - Titrimetric methods

Amendements minéraux basiques - Détermination de  
la valeur neutralisante - Méthode par titrimétrie

Kalkdünger - Bestimmung des Neutralisationswertes -  
Titrimetrische Verfahren

This European Standard was approved by CEN on 8 February 2014 and includes Amendment 1 approved by CEN on 11 June 2016.

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

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## European foreword

This document (EN 12945:2014+A1:2016) 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of an identical text or by endorsement, at the latest by April 2017 and conflicting national standards shall be withdrawn at the latest by April 2017.

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 includes Amendment 1 approved by CEN on 11 June 2016.

This document supersedes A1 EN 12945:2014 A1.

The start and finish of text introduced or altered by amendment is indicated in the text by tags A1 A1.

The following changes A1 had been made to the former edition EN 12945:2008 A1:

- a) scope modified taking into account that method A is not applicable to liming materials with more than 3 % P<sub>2</sub>O<sub>5</sub> and that method B is applicable to all liming materials;
- b) reference to EN 14984 added to the scope and the Bibliography;
- c) 9.2 amended by addition of an instruction concerning the use of correction factors;
- d) optional requirement concerning correction factors added to Clause 11 Test report;
- e) corrigendum EN 12945:2008/AC:2009 included;
- f) editorially revised.

A1 The following changes were made to the previous edition EN 12945:2014:

- g) Clause 9 modified taking into account that the neutralizing value may be expressed as CaO-equivalents or alternatively as HO--equivalents;
- h) Annex B with conversion factors between CaO and HO- added;
- i) Bibliography revised;
- j) document editorially revised. A1

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## Introduction

Two different procedures are described (method A and method B) because the titration to pH 7,0 is not applicable to silicate liming materials due to the precipitation of compounds at this pH value.

In method B the turning point at pH 4,8 on the titration curve is taken as the end-point of the titration. For carbonaceous liming materials the difference in the consumption of sodium hydroxide solution for back titration between the titration end-points of pH 4,8 and pH 7,0 is negligible.

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## 1 Scope

This European Standard specifies two methods for the determination of the neutralizing value (NV) of liming materials.

Method A is applicable to all liming materials except silicate liming materials.

Method B is applicable to all liming materials.

Both methods do not correctly take into account the potential neutralizing value of material containing more than 3 % P<sub>2</sub>O<sub>5</sub>. For a more accurate agronomic assessment of products containing more than 3 % P<sub>2</sub>O<sub>5</sub> determine the liming efficiency according to EN 14984.

NOTE The methods described in ISO 6598 [1] and ISO 7497 [2] can be used for the determination of P<sub>2</sub>O<sub>5</sub> content. Further information on P analyses is given in [3] and [4].

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 12944-3:2001, *Fertilizers and liming materials — Vocabulary — Part 3: Terms relating to liming materials*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-3:2001 apply.

## 4 Principle

Dissolution of the sample in a specified quantity of hydrochloric acid standard solution. Determination of the excess acid by back titration with a sodium hydroxide standard solution.

## 5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade.

NOTE Commercially available solutions can be used.

**5.1 Water**, according to EN ISO 3696, grade 3.

**5.2 Hydrochloric acid standard solution**,  $c(\text{HCl}) = 0,5 \text{ mol/l}$ .

Determine the exact concentration of the solution by titration with sodium hydroxide standard solution (5.3) using phenolphthalein solution (5.4) as indicator. Apply the appropriate correction factor in the calculation of the results (see Clause 9).

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**5.3 Sodium hydroxide standard solution**,  $c(\text{NaOH}) = 0,25 \text{ mol/l}$ .

Determine the exact concentration of the standard solution by titration against approximately 2 g of dried potassium hydrogen phthalate ( $\text{KHC}_8\text{H}_4\text{O}_4$ ), weighed to the nearest 0,001 g.

The solution shall be stored in a polyethylene bottle and absorption of carbon dioxide during storage should be avoided.

NOTE 1 ml of 0,25 mol/l sodium hydroxide solution is equivalent to 51,055 mg of potassium hydrogen phthalate.

Apply the appropriate correction factor in the calculation of the results (see Clause 9).

**5.4 Phenolphthalein indicator solution.**

Dissolve 0,25 g of phenolphthalein in 150 ml of ethanol with a mass fraction of 93 % and dilute with water to 250 ml.

Use the phenolphthalein solution (5.4) as an indicator.

**5.5 Hydrogen peroxide solution.**

Dilute one volume of hydrogen peroxide [ $\rho(\text{H}_2\text{O}_2) = 30 \text{ g/100 ml}$ ] with four volumes of water.

**6 Apparatus**

Usual laboratory apparatus and, in particular, the following:

**6.1 Test sieve**, conforming to the requirements of ISO 3310-1, of nominal aperture size 250  $\mu\text{m}$ .

**6.2 pH meter**, minimum sensitivity 0,05 pH units, with a suitable glass electrode and a calomel or other reference electrode or a combined electrode, calibrated using two buffer solutions whose pH values cover the range pH 4 to pH 7.

**6.3 Mechanical stirrer**, e.g. magnetic stirrer.

**6.4 Desiccator.**

**7 Sampling**

Sampling is not part of the method specified in this European Standard.  $\square_{A1}$  Recommended sampling methods are described in EN 1482-1 [5] and EN 1482-3 [6].  $\square_{A1}$

Prepare the sample of the liming materials in accordance with EN 1482-2.

**8 Procedure****8.1 Preparation of the test sample**

Dry the test sample at  $(105 \pm 2) \text{ }^\circ\text{C}$  to constant mass. Record the as-received ( $m_w$ ) and dry ( $m_d$ ) masses. Grind the sample so that it passes the 250  $\mu\text{m}$  test sieve (6.1). Mix thoroughly and store the sample in a desiccator (6.4).



## 8.2 Determination

### 8.2.1 Method A

#### 8.2.1.1 Test portion

Weigh about 0,5 g, to the nearest 0,001 g, of burnt or hydrated lime or 1 g of ground limestone or ground marl (prepared according to 8.1) into a 250 ml conical flask.

#### 8.2.1.2 Titration

Add 50 ml of the hydrochloric acid standard solution (5.2) with continuous shaking and boil gently for 10 min, using boiling granules.

Cool to ambient temperature. Transfer quantitatively into a 250 ml beaker and insert the electrodes of the pH meter (6.2) and a stirrer (6.3).

Titrate with the sodium hydroxide standard solution (5.3) with moderate stirring (avoid splashing) until a pH of 7,0 is stable for 1 min whilst stirring is maintained.

### 8.2.2 Method B

#### 8.2.2.1 Test portion

Weigh about 0,5 g, to the nearest 0,001 g, of the prepared test sample (8.1) into a 250 ml conical flask.

#### 8.2.2.2 Titration

Rinse the inside walls of the flask with 10 ml of water.

Add 35 ml of the hydrochloric acid standard solution (5.2) with continuous shaking.

Heat and boil gently for 10 min to dissolve the sample using boiling granules. Stir continuously. Cool to ambient temperature, then dilute with water to about 100 ml and add 5 ml of hydrogen peroxide solution (5.5).

NOTE Ferrous ions in silicate liming materials are oxidized by hydrogen peroxide to ferric ions before titration, because ferrous ions otherwise would consume hydrogen ions during titration.

Transfer quantitatively into a 200 ml graduated flask; make up the volume with water and mix. Pass through a dry filter into a dry container, discarding the initial portion. Pipette an aliquot portion of 100 ml of the solution into a 250 ml beaker.

Insert the electrodes of the pH meter (6.2) and a stirrer (6.3).

Titrate with the sodium hydroxide standard solution (5.3) with moderate stirring (avoid splashing) until a pH of 4,8 is stable for 1 min (whilst stirring is maintained).