



SLOVENSKI STANDARD
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Liming materials - Determination of neutralizing value - Titrimetric methods

Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung des
Neutralisationswertes - Titrimetrische Verfahren

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

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English version

Liming materials - Determination of neutralizing value - Titrimetric methods

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

Calcium-/Magnesium-Bodenverbesserungsmittel -
Bestimmung des Neutralisationswertes - Titrimetrische
Verfahren

This European Standard was approved by CEN on 6 March 2002.

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.

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Contents

	page
Foreword.....	3
Introduction	4
1 Scope	4
2 Normative references	4
3 Terms and definitions.....	4
4 Principle	4
5 Reagents	5
6 Apparatus	5
7 Sampling	5
8 Procedure	6
9 Expression of results	7
10 Precision	8
11 Test report	8
Annex A (informative) Results of an interlaboratory trial to determine the neutralizing value.....	9
Bibliography	10

<https://standards.iteh.ai/catalog/standards/sist/858235ff-501f-4dd0-82cd-6f5bac212abf/sist-en-12945-2003>

Foreword

This document EN 12945:2002 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of an identical text or by endorsement, at the latest by November 2002, and conflicting national standards shall be withdrawn at the latest by November 2002.

Annex A is 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, Malta, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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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.

1 Scope

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

Method A is applicable to liming materials except silicate liming materials and liming materials with more than 3 % P₂O₅.

Method B is applicable to all liming materials except those with more than 3 % P₂O₅.

NOTE The methods described in ISO 6598 and ISO 7497 can be used for the determination of P₂O₅ content.

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2 Normative references

SIST EN 12945:2003

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 1482, *Sampling of solid fertilizers and liming materials*.

EN 12944-3, *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 3696:1987)*.

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 European Standard, the terms and definitions given in EN 12944-3 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

5.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and water conforming to grade 3 in accordance with EN ISO 3696.

NOTE Commercially available solutions can be used.

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).

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.

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

NOTE 2 The solution should be stored in a polyethylene bottle and absorption of carbon dioxide during the storage should be avoided.

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

Use the phenolphthalein solution (5.4) as an indicator.

[SIST EN 12945:2003](#)

5.4 Phenolphthalein indicator solution

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

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 μm .

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

7 Sampling

Sample the liming materials in accordance with EN 1482.

8 Procedure

8.1 Preparation of the test sample

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

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.

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8.2.2 Method B

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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 to dissolve the sample and boil gently for 10 min, 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).

9 Expression of results

9.1 Method A

The neutralizing value of the dried product, N_d , is given by equation (1):

$$N_d = \frac{0,014 \times (V_1 - 0,5 \times V_2) \times 100}{m_t} \quad (1)$$

where

- V_1 is the volume of hydrochloric acid standard solution (5.2), in millilitres;
- V_2 is the volume of sodium hydroxide standard solution (5.3), in millilitres;
- m_t is the mass of the test portion, in grams.

The neutralizing value of the "as-received" product, N_{ar} , is given by equation (2):

$$N_{ar} = \frac{N_d m_d}{m_w} \quad (2)$$

where

- N_d is the neutralizing value of the dried sample;
- m_d is the mass of the sample after drying, in grams;
- m_w is the mass of the sample before drying, in grams.

The result should be taken as the arithmetic mean of at least two determinations.

9.2 Method B

The neutralizing value of the dried product, N_d , is given by equation (3):

$$N_d = \frac{0,014 \times (0,5 \times V_1 - 0,5 \times V_2) \times 100}{m_t} \quad (3)$$

where

- V_1 is the volume of hydrochloric acid standard solution (5.2), in millilitres;
- V_2 is the volume of sodium hydroxide standard solution (5.3), in millilitres;
- m_t is the mass of the test portion in the aliquot portion taken, in grams.

The neutralizing value of the "as-received" product, N_{ar} , is given by equation (4):

$$N_{ar} = \frac{N_d m_d}{m_w} \quad (4)$$

where

- N_d is the neutralizing value of the dried sample;
- m_d is the mass of the sample after drying, in grams;