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## Liming material — Determination of neutralizing value — Titrimetric methods

*Amendements minéraux basiques — Détermination de la valeur  
neutralisante — Méthodes par titrimétrie*

ICS: 65.080

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document was prepared by Technical Committee ISO/TC 134, Fertilizers and soil conditioners/WG 4, Mineral soil amendments.

This document (ISO 20978) derives from the European standard EN 12945:2014+A1. However, its principle is the same as in AOAC 955.01 and BNQ 0419-070: 2011.

This ISO Standard may be given the status of a national standard, either by publication of an identical text or by endorsement.

The following changes have been made to the EN 12945:2014+A1 edition:

- Expression of results is allowed as CaCO<sub>3</sub> equivalent, not only as CaO or HO<sup>-</sup> equivalents
- Annex B has been completed accordingly to present all the necessary conversion tables
- A new ISO international ring test was performed in 2017 to determine precision data (see clause 10)
- Annex A was revised accordingly
- Editorially revised.

## Introduction

This method has been developed to assess the ability of a product to deliver potential neutralization capacity, i.e. to neutralize protons or acidic cations in soils and consequently maintain or increase its pH. It relies on the measurement of Hydrochloric acid consumption when a liming material is mixed in solution with Hydrochloric acid in excess.

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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# Liming material — Determination of neutralizing value — Titrimetric methods

## 1 Scope

This ISO 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.

Neither method correctly takes 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 [8].

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 bibliography [3] [4].

NOTE Carbonate consumes H<sup>+</sup> and removes acidity in solution with subsequent dissociation to H<sub>2</sub>O and CO<sub>2</sub>. Forms of orthophosphate can consume H<sup>+</sup> but are not dissociated to molecular forms that remove acidity. The acidity is back titrated with alkali causing an underestimation of NV.

## 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.

ISO 14820-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

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

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 ISO 8157 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.

NOTE For products containing iron, a preliminary oxidization is necessary in method B

## 5 Reagents

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

NOTE Commercially available solutions can be used.

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

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

This solution can be purchased from a chemical supply company certified to have a concentration within a 0.4% variance (0.498 to 0.502 M).

If preparing from concentrated HCl, 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).

In case of any doubt about the concentration of the HCl solution, measuring the neutralizing value of a PCC (5.6) is recommended.

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

This solution can be purchased from a chemical supply company certified to have a concentration within a 0.2% variance (0.2495 to 0.2505 M).

If preparing from NaOH pellets, 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).

In case of any doubt about the concentration of the NaOH solution, measuring the neutralizing value of a PCC (5.6) is recommended.

**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.

The phenolphthalein solution (5.4) is a color indicator to determine when the endpoint pH is reached and is only suitable in Method A with an endpoint pH of 7. The indicator is not required if a pH electrode is used to monitor endpoint pH.

NOTE If there is a buffering capacity in the sample, a deviation may occur between Phenolphthalein indicator solution (changes at pH 8.3) and pH end point with pH meter (7.0).

**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.

Hydrogen peroxide is used in Method B to oxidize any reduced iron in slag liming materials.

**5.6 Precipitated calcium carbonate.**

Precipitated calcium carbonate of recognised analytical grade, minimum 99 %  $\text{CaCO}_3$ .

This reagent is to be used when HCl or NaOH solution concentration is subject to doubt or when the result of neutralizing value measurement does not look as expected. In such a situation, insert this reagent grade  $\text{CaCO}_3$  as a quality control check within a set of samples.



## 6 Apparatus

Usual laboratory apparatus and, in particular, the following:

**6.1 Test sieve**, conforming to the requirements of ISO 3310-1, of maximum 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.

A pH meter is not required if using phenolphthalein to determine endpoint pH of 7 in Method A.

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

**6.4 Desiccator**.

## 7 Sampling

Sampling is not part of the method specified in this ISO standard. Recommended sampling methods are described in ISO 14820-1 [5] and EN 1482-3 [6].

Prepare the sample of the liming materials in accordance with ISO 14820-2.

## 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  $\mu\text{m}$  test sieve (6.1).

NOTE A smaller sieve size depending on material type may be used such as 150  $\mu$  / Mesh 100 for slags.

Mix thoroughly.

When titration is not processed immediately:

- In case of burnt lime, store the prepared test sample in a desiccator (6.4) until the effective measurement.
- Otherwise, store the prepared test sample in a waterproof plastic bag.

### 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 suitable flask.

##### 8.2.1.2 Titration

Add 50 ml of the hydrochloric acid standard solution (5.2) with continuous shaking and boil gently for 5 minutes, 10 minutes for hard materials, using boiling granules.

Cool to 20 to 25  $^\circ\text{C}$ . Add a few drops of phenolphthalein solution or insert pH and reference electrodes (6.2) into the solution. Stir (6.3) the solution.

Titrate with the sodium hydroxide standard solution (5.3) with moderate stirring (avoid splashing) until a pH of 7.0 or pink color of phenolphthalein are 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 suitable 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 20-25 °C, then dilute with water to about 100 ml.

Add 5 ml of hydrogen peroxide solution (5.5)

NOTE Ferrous ions from silicate liming materials can oxidize and consume OH<sup>-</sup> during titration. To avoid OH<sup>-</sup> consumption by ferrous ions, hydrogen peroxide is added which forms ferric and ferrous ions in a neutral reaction.

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 Calculation and expression of results for method A and method B

9.1 Calculate the neutralizing value of the dried product,  $N_d$ , according to Formula (1)

$$N_d = \frac{c \times (M_1 \times V_1 \times f_1 \times A - M_2 \times V_2 \times f_2) \times 100}{m_t \times A} \quad (1)$$

where

$c$  is the factor to convert hydrochloric acid standard solution into CaO or CaCO<sub>3</sub> or HO<sup>-</sup> equivalent;

$c = 0,028$  when neutralizing value should be expressed as CaO

$c = 0,050$  when neutralizing value should be expressed as CaCO<sub>3</sub>

$c = 0,017$  when neutralizing value should be expressed as HO<sup>-</sup>

$M_1$  is the molarity of hydrochloric acid standard solution (5.2), in mol/l;

$V_1$  is the total volume of hydrochloric acid standard solution (5.2), in millilitres;

$f_1$  is the factor of hydrochloric acid standard solution (5.2);

$A$  is equal to 1 for method A, and the factor of the taken aliquot is 0,5 for method B;

$M_2$  is the molarity of sodium hydroxide standard solution (5.3), in mol/l;