
Liming material — Determination of neutralizing value — Titrimetric methods

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

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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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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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.

This document 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.

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

- expression of results is allowed as CaCO_3 equivalent, not only as CaO or HO^- 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.

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Liming material — Determination of neutralizing value — Titrimetric methods

1 Scope

This document 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.

NOTE 1 Examples of hard liming materials are limestone and dolomite. Examples of soft liming materials are chalk, marl and burnt lime.

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_2O_5 . For a more accurate agronomic assessment of products containing more than 3 % P_2O_5 , EN 14984 [8] is used to determine the liming efficiency.

NOTE 2 The methods described in ISO 6598 and ISO 7497 can be used for the determination of P_2O_5 content. Further information on P analyses is given in References [5] and [6].

NOTE 3 Carbonate consumes H^+ and removes acidity in solution with subsequent dissociation to H_2O and CO_2 . Forms of orthophosphate can consume H^+ 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 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

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

ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8157 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

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.

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

5 Reagents

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

NOTE Commercially available solutions can be used.

5.1 Water, according to 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 M 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 precipitated calcium carbonate (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 (0,249 5 M to 0,250 5 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 precipitated calcium carbonate (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 colour indicator to determine when the end point pH is reached and is only suitable in Method A with an end point pH of 7. The indicator is not required if a pH electrode is used to monitor end point pH.

NOTE If there is a buffering capacity in the sample, a deviation can 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 recognized analytical grade, minimum 99 % CaCO_3 .

Quality control check by using precipitated calcium carbonate and blank solution should be used in each series of measurements.

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 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.

A pH meter is not required if using phenolphthalein to determine end point 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 document. Recommended sampling methods are described in ISO 14820-1 and ISO 14820-3.

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 μm test sieve (6.1).

NOTE A smaller sieve size depending on material type can be used such as 150 μm /Mesh 100 for slags.

Mix thoroughly.

Perform the following 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 min (10 min for hard materials) using boiling granules.

Cool down to 20 °C to 25 °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 colour of phenolphthalein are stable for 1 min while 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 down to between 20 °C to 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⁻ during titration. To avoid OH⁻ 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 (while 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):

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