# BS EN 13971:2012



# **BSI Standards Publication**

Carbonate and silicate liming materials — Determination of reactivity — Potentiometric titration method with hydrochloric acid



BS EN 13971:2012 BRITISH STANDARD

#### **National foreword**

This British Standard is the UK implementation of EN 13971:2012. It supersedes BS EN 13971:2008 which is withdrawn.

BSI, as a member of CEN, is obliged to publish EN 13971:2012 as a British Standard. However, attention is drawn to the fact that during the development of this European Standard, the UK committee voted against its approval as a European Standard.

The UK committee submitted a negative vote with respect to this standard due to concerns over reproducibility limits as described in Table 1. In two cases out of eight the reproducibility limit was 60 % and in all but three cases it exceeded 30 %. The UK committee advises that users validate their performance.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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ISBN 978 0 580 72251 6

ICS 65.080; 91.100.10

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 January 2013.

Amendments/corrigenda issued since publication

Date Text affected

# EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN 13971

December 2012

ICS 65.080

Supersedes EN 13971:2008

#### **English Version**

# Carbonate and silicate liming materials - Determination of reactivity - Potentiometric titration method with hydrochloric acid

Amendements minéraux basiques carbonatés et silicatés -Détermination de la réactivité - Méthode par titrage potentiométrique à l'acide chlorhydrique Carbonatische und silikatische Kalke - Bestimmung der Reaktivität - Potentiometrisches Titrationsverfahren mit Salzsäure

This European Standard was approved by CEN on 3 November 2012.

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## **Foreword**

This document (EN 13971:2012) 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 June 2013, and conflicting national standards shall be withdrawn at the latest by June 2013.

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 EN 13971:2008.

The following has been added to the former edition of the European Standard:

- a) silicate liming materials added to the scope and to the title;
- b) EN 12947 and EN 13475 added to the normative references;
- c) Clause 3 reaction formula for silicates added;
- d) subclauses 7.1.2 and 7.2.2 enlarged concerning determination of silicate liming materials;
- e) subclause 8.2 and formulas (2), (3) and (4) on expression of results for silicate liming materials added;
- f) subclause 9.2 and Table 2 on the precision data for silicate liming materials added;
- g) Bibliography revised.

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# Introduction

The results obtained by this method can be used to estimate the behaviour of the liming material in the soil. The results show a good correlation with the results obtained by a soil incubation method (see [1] to [5]). Regarding the precision of the method, the results are not used to declare a value, but to classify the different product groups.

# 1 Scope

This European Standard specifies a method for the determination of the speed and effectiveness of the neutralising potential of calcium carbonate, calcium magnesium carbonate and calcium magnesium silicate liming materials by potentiometric titration with hydrochloric acid.

This method is applicable only to liming materials with a maximum particle size of 6,3 mm.

The type of liming material should be identified according to EN 14069 and the particle size should be determined according to EN 12948.

## 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 12048, Solid fertilizers and liming materials — Determination of moisture content — Gravimetric method by drying at  $(105 \pm 2)$  °C (ISO 8190:1992 modified)

EN 12945, Liming materials — Determination of neutralizing value — Titrimetric methods

EN 12947, Liming materials — Determination of magnesium content — Atomic absorption spectrometric method

EN 12948, Liming materials — Determination of size distribution by dry and wet sieving

EN 13475, Liming materials — Determination of calcium content — Oxalate method

# 3 Principle

Decomposition of carbonates and silicates with acids according to the following reactions:

$$MeCO_3 + 2 H^+ \rightarrow Me^{2+} + H_2O + CO_2$$

$$MeSiO_4 + 2 H^+ \rightarrow Me(OH)_2 + SiO_2$$

Titration under stable pH conditions either with an automatic titration apparatus or a manual method. The acid consumption during a given time is a direct measure of the reaction rate of the liming materials being tested.

# 4 Apparatus

Usual laboratory apparatus and, in particular, the following:

# **4.1 pH meter**, with electrode.

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#### 4.2 Burette.

NOTE Used only for 5,0 mol/l hydrochloric acid solution (5.2).

- **4.2.1 50 ml motor driven burette** (for automatic titration),a pH stat function is recommended, for example Metrohm 716 DMS Titrino<sup>®</sup>1).
- 4.2.2 50 ml burette (for manual titration).
- **4.3 250 ml glass beaker**, with an inner diameter of 65 mm.
- **4.4** Magnetic stirrer, with centring mark for the 250 ml glass beaker.
- **4.5** Magnetic stirrer rod,  $(9 \pm 1)$  mm diameter times  $(50 \pm 1)$  mm length, with central ring.
- 4.6 Stop-watch.
- **4.7** Filter paper, acid-proof, medium filtration speed, average retention capacity about 5 μm to 12 μm.

## 5 Reagents

All reagents shall be of recognised analytical grade.

- **5.1 Hydrochloric acid solution**, mass fraction, w(HCI) = 25 %.
- **5.2 Hydrochloric acid**, standard volumetric solution, c(HCI) = 5.0 mol/l.
- **5.3** Calcium carbonate, precipitated, mass fraction, w(CaCO<sub>3</sub>) = of at least 99 %.

Precipitated calcium carbonate is from crystalline origin. Commercial PCC for analysis is granted for its chemical characteristics. However, physical characteristics are not granted. The use of a highly reactive PCC, such as commercial PCC from VWR / Prolabo / BDH, reference GPR, Rectapur, Ref 22296.294, Molar mass 100,09<sup>2</sup>) which will consume 15 ml after 15 min, is recommended.

- 5.4 Silicone defoamer.
- **5.5 Standard buffer solution**, pH = 2,0 (commercial solution).

NOTE This has a limited life.

**5.6** Standard buffer solution, pH = 4,0, (commercial solution).

NOTE This has a limited life.

# 6 Preparation of the test sample

**6.1** Prepare the sample of the liming material in accordance with EN 1482-2.

<sup>1)</sup> Metrohm 716 DMS Titrino<sup>®</sup> is an example of a suitable product available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

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- **6.2** Dry the test sample in accordance with EN 12048. Record the result for information only.
- **6.3** Use the dried test sample without further preparation, e.g. grinding.
- **6.4** Weigh a 5,0 g test portion of the dried sample to the nearest 0,01 g. For liming materials coarser than 1 mm, the test portion shall be prepared in accordance with Annex A.

#### 7 Procedure

#### 7.1 Automatic titration

#### 7.1.1 Calibration

**7.1.1.1** Calibrate the pH meter (4.1) with two standard buffer solutions, pH 2,0 (5.5) and pH 4,0 (5.6). The pH electrode shall react quickly.

Check the sluggishness of the electrode and if necessary, clean it carefully with hydrochloric acid (5.1) and recalibrate with the standard buffer solutions.

Checking and cleaning is more frequently required for liming materials containing clay.

It is recommended to flush the acid introduction pipe between each sample for slow reacting samples.

- **7.1.1.2** Adjust the motor driven burette (4.2.1) to the position "continuous working" and a standard flow rate of 35 ml/min to 38 ml/min.
- NOTE Wide deviations in the flow rate adversely affect the results obtained.
- **7.1.1.3** Place the glass beaker (4.3) centrally on the magnetic stirrer (4.4). Add 100 ml water and the magnetic stirrer rod (4.5). The test apparatus shall be arranged according to Figure B.1.
- **7.1.1.4** Set the magnetic stirrer speed control to between 500 min<sup>-1</sup> and 600 min<sup>-1</sup>. Fill the burette (4.2.1) with 5,0 mol/l hydrochloric acid (5.2).
- **7.1.1.5** Adjust the titration control so that the stepwise run of the titration only starts below pH 2,5. For apparatus with a step length adjustment, set the adjustment at the middle position.
- **7.1.1.6** Set up the electrode (4.1) and the burette (4.2.1) in the glass beaker (4.3) according to Figure B.1. This is to ensure that the added hydrochloric acid (5.2) is mixed with the contents of the glass beaker before reaching the electrode (4.1). Avoid contact with the walls of the glass beaker.
- **7.1.1.7** For the exact adjustment of the operating conditions, start the magnetic stirrer (4.4) and add  $(5,00\pm0,01)$  g of calcium carbonate (5.3) to the stirred water in the glass beaker (4.3). The solution should be stirred for 30 s. Start the stop-watch (4.6) and commence the titration, adding the hydrochloric acid (5.2) in a fast sequence of drops, aiming at a pH value of 2,0.

When pH 2,5 is reached, add the hydrochloric acid more slowly. Use  $(16 \pm 0.2)$  ml of hydrochloric acid in the first 30 s and then continue with stepwise additions to dissolve the remaining carbonate within 60 s. Check the acid consumption is  $(20.0 \pm 0.2)$  ml of 5,0 mol/l hydrochloric acid (5.2). During the procedure the solution shall not be allowed to fall below pH 2,0 by more than 0,2 pH units, even for a short time.

If the material being tested foams very strongly, one drop of silicon defoamer (5.4) should be added to the solution.

When operating conditions are correctly adjusted, about 80 % of the mass of calcium carbonate is dissolved by the first 16 ml of hydrochloric acid (5.2) which should run almost uninterrupted. The remaining amount of calcium carbonate should be dissolved by a further 4 ml of hydrochloric acid, added stepwise, within the next

60 s. Any fine adjustment that might be necessary can be carried out by a slight alteration to the start of the stepwise titration or by changing the length of the titration.

A pH stat function is recommended for the titration.

#### 7.1.2 Determination

- **7.1.2.1** Set up the apparatus as described in 7.1.1.1 to 7.1.1.6.
- **7.1.2.2** Add the weighed test portion (6.4) to the stirred water in the glass beaker (4.3) and immediately start the stop-watch (4.6) and the titration procedure. The possible abrasive effect of stirring is reduced by commencing the titration immediately the magnetic stirrer (4.4) is switched on.
- **7.1.2.3** Stop the titration after 10 min and record the amount of acid consumed. During the procedure the pH value shall not be allowed to fall below pH 2,0 by more than 0,2 pH units, even for a short time. For carbonate liming materials continue with 8.1.
- **7.1.2.4** For silicate liming materials immediately after end of titration pass the suspension through a dry filter (4.7) into a dry container without rinsing the pH electrode. Discard the initial portion. Take an aliquot portion and dilute it with water to a measurable concentration (e. g. 1 : 100). Determine the concentration of calcium and in the solution according to EN 13475 and magnesium according to EN 12947. Repeat the titration three times. Take the mean acid consumption of the four titrations and record this amount.

#### 7.2 Manual titration

## 7.2.1 Calibration

**7.2.1.1** Calibrate the pH meter (4.1) with two standard buffer solutions, pH 2,0 (5.5) and pH 4,0 (5.6). The pH electrode shall react guickly.

Check the sluggishness of the electrode and if necessary, clean it carefully with hydrochloric acid solution (5.1) and re-calibrate with the standard buffer solutions (see NOTE in 7.1.1.1).

- **7.2.1.2** Place the glass beaker (4.3) centrally on the magnetic stirrer (4.4). Add 100 ml water and the magnetic stirrer rod (4.5).
- **7.2.1.3** Set the magnetic stirrer speed control to between 500 min<sup>-1</sup> and 600 min<sup>-1</sup>. Fill the burette (4.2.2) with 5,0 mol/l hydrochloric acid (5.2).
- **7.2.1.4** Set up the electrode (4.1) and the burette (4.2.2) in the glass beaker (4.3) according to Figure B.1. This is to ensure that the added hydrochloric acid (5.2) is mixed with the contents of the glass beaker before reaching the electrode (4.1). Avoid contact with the walls of the glass beaker.
- **7.2.1.5** For the exact adjustment of the operating conditions, start the magnetic stirrer (4.4) and add  $(5,00\pm0,01)$  g of calcium carbonate (5.3) to the stirred water in the glass beaker (4.3). Start the stop-watch (4.6) and commence the titration, adding the hydrochloric acid (5.2) in a fast sequence of drops, aiming at a pH value of 2,0.

When pH 2,5 is reached, add the hydrochloric acid more slowly. Use  $(16 \pm 0,2)$  ml of the hydrochloric acid in the first 30 s. and then continue stepwise additions to dissolve the remaining carbonate within 60 s. Check that the acid consumption is  $(20,0 \pm 0,2)$  ml of 5,0 mol/l hydrochloric acid (5.2). During the procedure the solution shall not be allowed to fall below pH 2,0 by more than 0,2 pH units, even for a short time.

#### 7.2.2 Determination

**7.2.2.1** Set up the apparatus as described in 7.2.1.1 to 7.2.1.3.

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- **7.2.2.2** Add the weighed test portion (6.4) to the stirred water in the glass beaker (4.3) and immediately start the stop-watch (4.6) and the titration procedure. The possible abrasive effect of stirring is reduced by commencing the titration immediately the magnetic stirrer (4.4) is switched on.
- **7.2.2.3** Add the hydrochloric acid (5.2) in a fast sequence of drops, aiming at a pH of 2,0. Do not allow the pH to drop below 2,0.
- **7.2.2.4** When pH 5 is reached, the hydrochloric acid shall be added more slowly. After a while, the pH value increases very slowly, and the hydrochloric acid shall be added carefully drop by drop, always aiming to maintain a pH of 2,0.

For example, when the pH rises above pH 2,05, a drop of hydrochloric acid is added. This may drop the pH value to just below 2,0 for a short time, but never allow the pH value to fall below pH 2,0 by more than 0,2 pH units, even for a short time.

- **7.2.2.5** Stop the titration after 10 min and record the amount of acid consumed. For carbonate liming materials continue with 7.2.2.7.
- **7.2.2.6** For silicate liming materials immediately after end of titration pass the suspension through a dry filter (4.7) into a dry container without rinsing the pH electrode. Discard the initial portion. Take an aliquot portion and dilute it with water to a measurable concentration (e. g. 1 : 100). Determine the concentration of calcium in the solution according to of EN 13475 and magnesium according to EN 12947.
- **7.2.2.7** Repeat the titration three times. Take the mean acid consumption of the four titrations and record this amount.

# 7.3 Determination of neutralising value

Determine the neutralising value of the test sample in accordance with EN 12945 and record the result.

# 8 Expression of results

## 8.1 Carbonate liming materials

The reactivity,  $A_{ij}$  of the liming material being tested, in percent, is given by Formula (1):

$$A = \frac{C \times 14,0 \times 100}{m_{\rm t} \times N_{\rm d}} \tag{1}$$

where

- *C* is the mean consumption of 5,0 mol/l hydrochloric acid (5.2), in millilitres;
- $m_{\rm t}$  is the mass of the dried test portion (6.4) of liming material, in grams;
- $N_{
  m d}$  is the neutralising value of the test sample, expressed on a dry matter basis;
- 14,0 is taken as one tenth of the mass of CaO, in milligrams, neutralised by 1 ml of 5,0 mol/l hydrochloric acid.

NOTE 20 ml of hydrochloric acid with an amount-of-substance concentration of 5,0 mol/l is sufficient to neutralise 5 g of pure calcium carbonate. The  $H^{+}$  ionic concentration in the reaction mixture caused by the adjustment of the apparatus is, however, to be found at  $10^{-2}$  mol/l which ensures a delayed liming material reaction in spite of a high concentration of the titration medium.

# 8.2 Silicate liming materials

The reactivity, B of the liming material being tested, in percent, is given by Formula (2):

$$B = \frac{(D + E \times F_{MgO}) \times 100}{N_{d}}$$
 (2)

Where

 $F_{MqO}$  is the conversion factor for the conversion of MgO to CaO = 1,391

D is the within 10 min dissolved CaO given by Formula (3), in percent:

$$D = \frac{\mathsf{CaO} \times V_{\mathsf{c}} \times F}{m_{\mathsf{t}} \times 10} \tag{3}$$

E is the within 10 min dissolved MgO given by Formula (4), in percent:

$$E = \frac{\text{MgO} \times V_{\text{c}} \times F_{\text{D}}}{m_{\text{t}} \times 10}$$
 (4)

CaO is the concentration of Calcium in the final dilution, in milligrams CaO per litre;

MgO is the concentration of Magnesium in the final dilution, in milligrams MgO per litre;

 $F_{\mathsf{D}}$  is the dilution factor;

 $m_{\rm t}$  is the mass of the dried test portion (6.4) of liming material, in milligrams;

 $N_{\rm d}$  is the neutralising value of the test sample, expressed on a dry matter basis;

 $V_c$  is 100 ml + the mean consumption of 5,0 mol/l hydrochloric acid (5.2), in millilitres.

## 9 Precision

## 9.1 Precision data for carbonate liming materials

The precision data were derived from an inter-laboratory test involving 15 participants carried out in 1996 and using a range of liming materials including limestone, dolomitic limestone, magnesium limestone and chalk of varying fineness.

The repeatability limits, r, and reproducibility limits, R, expressed as relative percent values of the mean reactivity, are given in Table 1.

Table 1 — Precision data for carbonate liming materials

Liming material tested	Fineness	Mean reactivity %	Repeatability limit r % (relative)	Reproducibility limit R % (relative)
Limestone	98 % < 1 mm 90 % < 0,16 mm	74,93	10,37	24,31
Limestone	98 % < 3,35 mm 35 % < 1,16 mm	59,58	16,68	32,86
Magnesium limestone	98 % < 1 mm 90 % < 0,16 mm	72,02	7,74	14,97
Magnesium limestone	98 % < 3,35 mm 35 % < 1,16 mm	47,82	23,19	40,94
Dolomitic limestone	98 % < 1 mm 90 % < 0,16 mm	44,14	7,84	17,49
Dolomitic limestone	98 % < 3,35 mm 35 % < 1,16 mm	22,89	22,56	60,64
Chalk	98 % < 1 mm 90 % < 0,16 mm	93,19	6,35	61,18
Chalk	60 % < 10 mm 40 % < 1 mm	75,96	21,43	35,16

The precision data shows the precision of different liming materials tested in this inter-laboratory test. The statistical evaluation has been carried out in accordance with ISO 5725:1986.

# 9.2 Precision data for silicate liming materials

The precision data for converter lime were derived from an inter-laboratory test involving 12 German laboratories carried out in 1998 using a converter lime from converter slag. The repeatability limits, r, and reproducibility limits, R, expressed as relative percent values of the mean reactivity, are given in Table 2.

Table 2 — Precision data of silicate liming materials

Liming material tested	Fineness	Mean reactivity %	Repeatability limit r % (relative)	Reproducibility limit R % (relative)
Converter lime	97 % < 3,15 mm 40 % < 0,315 mm	50,1	4,39	37,32

# 10 Test report

The test report shall contain at least the following information:

- a) all details necessary for the identification of the sample;
- b) a reference to this document, i.e. EN 13971;
- c) the results and units in which the results have been expressed;
- d) any particular points observed in the course of the test;
- e) any operations not specified in the method or regarded as optional which might have affected the results.

# Annex A

(normative)

# Preparation of the test portion of liming materials coarser than 1 mm

## A.1 General

For liming materials coarser than 1 mm determine the size distribution by dry sieving according to EN 12948 and store the different fractions. Select a maximum of three test sieves with different principal sizes to cover the range of the particle size expected. Compose the test portion of 5,00 g from the individual sieving fractions according to their proportion of the total test sample.

# A.2 Calculation of mass fractions retained on the test sieves

The individual mass fractions of the total test sample,  $w_n$ , (n = 1 to 3), expressed in percent, shall be calculated using Formula (A.1):

$$w_n = \frac{m_n \times 100}{\sum m_n} \tag{A.1}$$

where

 $m_{\rm n}$  is the mass of the sieving fraction n in grams.

# A.3 Calculation of the mass of each sieving fraction to be incorporated in the test portion

The mass of the individual sieving fractions to be incorporated in the test portion  $t_n$  (n = 1 to 3) expressed in grams shall be calculated using Formula (A.2):

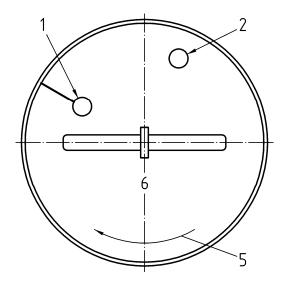
$$t_n = \frac{w_n \times m_t}{100} \tag{A.2}$$

where

 $m_{\rm t}$  is the mass of the test portion, in grams.

# **Annex B** (normative)

# Arrangement of the test apparatus

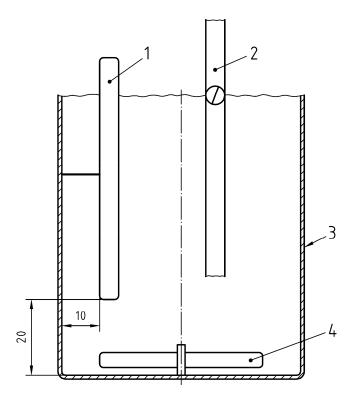


# Key

- 1 electrode
- 2 burette
- 5 direction of stir
- 6 magnetic stirrer

Figure B.1 — Test apparatus – Plan view

Dimensions in millimetres



# Key

- 1 electrode
- 2 burette
- 3 250 ml glass beaker (65 mm diameter)
- 4 magnetic stirrer rod

Figure B.2 — Test apparatus – Section view

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<sup>3)</sup> ISO 5725:1986 (now withdrawn) was used to obtain the precision data.



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