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Carbonate liming materials — Determination of reactivity — Automatic titration method with citric acid

Amendements minéraux basiques carbonatés — Détermination de la réactivité — Méthode par titration automatique à l'acide citrique

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Foreword

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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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This document was prepared by the European Committee for Standardization (CEN) (as EN 16357/2013) and was adopted, without modification apart from editorial corrections, by Technical Committee ISO/TC 134, *Fertilizers, soil conditioners and beneficial substances*.

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.

Introduction

This document has been prepared to improve the existing agricultural reactivity methods (see References [8], [9], [10], [11] and [12]) for carbonate liming materials: duration, accuracy, representativeness, closer from soil conditions, automation.

Carbonate liming materials — Determination of reactivity — Automatic titration method with citric acid

1 Scope

This document specifies a method for determining the reactivity of calcium carbonate and calcium magnesium carbonate liming materials. It assesses the speed and effectiveness of their neutralising potential by automatic titration with citric acid.

This method is applicable only to liming materials with a maximum particle size of 6,3 mm determined in accordance with ISO 20977.

NOTE For marble dolomite (BET procedure as defined in ISO 9277 below 500 m^2/kg), see EN 14984.

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

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

ISO 20977, *Liming materials — Determination of size distribution by dry and wet sieving*

ISO 20978, *Liming materials — Determination of neutralizing value — Titrimetric methods*

EN 12048, *Solid fertilizers and liming materials — Determination of moisture content — Gravimetric method by drying at (105 ± 2) °C*

3 Terms and definitions

No terms and definitions are listed in this document.

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

The principle of the method is based on the following points:

— Limited decomposition of carbonates in a given time with acid according to the following reaction:

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

where Me represents Ca, Mg, etc;

— Titration under stable pH conditions (pH = 4,5) with an automatic titration apparatus. The citric acid consumption during a given time (15 min) is a direct measure for the reaction of the liming materials being tested.

Attention is drawn to the following critical steps:

— identification of the liming material type (influence on precision data);

— size distribution (influence on test portion preparation and amount);

— calibration of pH electrode (influence on titrator's pH adjustments);

— pH stat programme setting (influence on the accuracy of added amounts of citric acid solution);

— suitability of precipitated calcium carbonate (PCC) used to check calibration;

— stirring device (provides homogeneousness without grinding);

— additional uncertainty with neutralising value and MgO content determination.

5 Apparatus

The usual laboratory apparatus and, in particular, the following:

5.1 pH meter with electrode.

This instrument is generally included in the automatic motor-driven burette device.

5.2 Automatic motor-driven burette, with a capacity of 20 ml.

This kind of burette is generally equipped with all necessary accessories such as pH regulation programme (pH stat), automatic refilling device, pH electrode, continuous pH measurement and propeller stirring device.

Though a propeller stirring device is preferred, a magnetic stirring device (5.7) may be used, provided the central ring of the stirring rod is thick enough and does not lead to the grinding of the tested material. Make sure the rotation speed of the stirring rod is fast enough to make a homogeneous dispersion in the beaker. If not, increase the speed up to the appropriate value.

The burette shall deliver at least 0,05 ml/s of citric acid solution (6.3). This is to ensure the first part of the reaction [pH dropping from initial pH value to target pH value (4,5) is not a limiting factor for liming material dissolution speed. This figure is higher than the flow rate obtained with the fastest reaction observed in preliminary tests.

The burette shall deliver its whole content in at least 4 000 steps to ensure accuracy for small amounts of citric acid solution (6.3).

NOTE This condition is always fulfilled with modern titrators. All contemporary (less than 10 years old) titrators allow such accuracy, with a minimum step amount of 0,002 5 ml for a 10 ml burette or 0,012 5 ml for a 50 ml burette. This is sufficient, even for small amounts. However, this accuracy is obtained only if the correct (minimal) step volume is specified in the titrator setup. If not, the precision of the method is altered.

Use the burette only for the citric acid solution (6.3).

For liming materials coarser than 1 mm, use a 50 ml burette.

For most products, a 10 ml burette is sufficient. However, a 20 ml burette is necessary for highly reactive chalks and precipitated calcium carbonate (PCC). Because refilling takes a significant amount of time, this can alter the results. If volumes higher than 10 ml are expected, do not use the automatic refilling possibility and use a 20 ml or a 50 ml burette.

5.3 Glass beaker, with a capacity of 100 ml.

For liming materials coarser than 1 mm, use a 200 ml beaker.

Minimum diameter in case of magnetic stirring device (5.7): 50 mm.

5.4 Stop-watch.

5.5 Balance, capable of weighing 10 g to the nearest 0,01 g.

5.6 Sample changer, optional.

If a sample changer is utilized, a beaker of water (6.1) shall be inserted between two samples.

5.7 Magnetic stirring device, optional, (see 5.2).

Capable of a minimum rotational speed of 500 min^{-1}.

Stirring rod minimum length: 40 mm.

6 Reagents

All reagents shall be of recognized analytical grade.

6.1 Water, meeting the requirements of ISO 3696, grade 2.

6.2 Citric acid monohydrate, $C_6H_8O_7\ H_2O$, crystallized or powdered, molar mass: 210,14 g/mol.

Do not use anhydrous citric acid having a different molar mass and can partially hydrate when stored.

6.3 Citric acid solution, ρ = 457,17 g/l.

Preferably, use a fresh home-made solution as described below. Its pure citric acid concentration is conventionally supposed to be the required one, i.e. ρ_{ca} = 457,17 g/l.

The solution may be used for up to one month if stored in a closed, dark glass vessel. If the solution has been stored for more than one week, check its concentration by any means, for example by titration with a strong base (NaOH) solution of known concentration, and report the result in Formula (2).

Weigh 500 g of citric acid monohydrate (6.2) to the nearest 0,1 g. Pour it quantitatively into a 1 l measuring vessel. Rinse the weighing material and pour the rinsing water into the vessel in a way that it takes any acid stuck on the edge or on the bottom. Add about 500 ml of water (6.1) to the measuring vessel. Heat the vessel until full dissolution (at a temperature of about 80 °C). Let the vessel cool to ambient temperature. Make up the volume with water to 1 l. Stir to get a homogeneous solution.

The pure citric acid concentration of this solution, ρ_{ca} ($C_6H_8O_7$), in grams per litre, is calculated according to Formula (1) as follows.

$$\rho_{ca} = \frac{500 \times 192,14}{210,14} = 457,17 \quad (1)$$

where

500 is the added mass of citric acid monohydrate (6.2), in grams (g);

192,14 is the molar mass of anhydrous citric acid, in grams per mole (g/mol);

210,14 is the molar mass of citric acid monohydrate, in grams per mole (g/mol).

6.4 Calcium carbonate, precipitated (PCC), with a mass fraction $w(CaCO_3)$ of at least 99 %.

Commercial PCC for analysis is certified for its chemical characteristics. However, its physical characteristics are not certified. As reactivity depends on fineness, even for PCC, it is essential to take a highly reactive PCC[1)] as a reference, which consumes 15 ml in 15 min. This type of PCC was used in the ring test before launching measurements. By experience, some types of PCC do not meet this requirement. See also the note in 8.2.6.

6.5 Silicone defoamer.

6.6 Standard buffer solution, pH = 4 (commercial solution, pH = 4,01).

NOTE This solution has a limited lifetime.

6.7 Standard buffer solution, pH = 7 (commercial solution, pH = 6,98).

NOTE This solution has a limited lifetime.

7 Sampling and sample preparation

7.1 General

Sampling is not part of the methods specified in this document. A recommended sampling method is given in ISO 14820-1.

Sample preparation shall be carried out in accordance with ISO 14820-2.

This document specifies that samples are tested "as received" in order to allow immediate starting of all the necessary measurements (Neutralizing Value, MgO). No preliminary determination is required to calculate the mass of the tested samples. However, make sure that sample moisture is the same in the reactivity test portion as in the neutralising value measurement.

Correcting factors are to be applied later on in the expression of results: actual citric acid concentration, exact mass of sample, as received neutralising value, MgO content. Such a procedure shortens the total necessary time for analysis because required measurements are made simultaneously instead of successively. However, the additional uncertainty introduced by the correction factors is neither described in this document nor taken into account for the precision data mentioned in Clause 10. All implemented correction factors should be evaluated to quantify the additional uncertainty they introduce to the expression of the reactivity.

NOTE This procedure is also better than drying the sample before titrating, because drying can modify fineness or physical presentation and consequently have an impact on the reactivity of some products.

1) Such as commercial PCC from VWR / BDH Prolabo ®, GPR, Rectapur®, Ref 22296.294, Molar mass 100,09. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7.2 Preparation of the test sample

Measure the moisture content of the tested material in accordance with EN 12048 and record the result for information.

Make sure there is no oxide or hydroxide in the material to be tested (pH with 1/10 dilution shall be below 10). An oxide or hydroxide fraction in the product alters the result. Oxide or hydroxide chemical forms are not included in the scope of this method.

Use the test sample without any further preparation, e.g. grinding or drying.

NOTE The procedure (8.3.2) includes a fixed time of preliminary stirring.

7.3 Preparation of the test portion

Weigh (5,0 ± 0,2) g of the test sample as received to the nearest 0,01 g and record the weight.

For liming materials coarser than 1 mm, or which appear to be heterogeneous, the test portion shall be prepared by fraction sampling in accordance with Annex A, and a test portion of 10 g shall be taken instead of 5 g. This can happen even if the material particle size is finer than 1 mm. The representativeness of the product as delivered in the 5 g or 10 g test portion is the most important aspect.

Note that fraction sampling involves additional uncertainty. Consequently, it should be used only if the coarse particles are higher than 4 mm. Otherwise, increase the test portion up to 10 g.

8 Procedure

8.1 General

The determination shall be made under usual laboratory conditions, i.e. ambient temperature [(20 ± 2) °C].

Reagents shall be at the same room temperature.

As this method does not use a total reaction but a partial one in a limited amount of time, the accuracy of results is highly dependent on trained and skilled staff. Do not proceed to perform measurements until the analyst becomes familiar with the method.

8.2 Calibrations

8.2.1 Calibrate the pH meter (5.1 or 5.2) with two buffer solutions, pH = 4 (6.6) and pH = 7 (6.7), to exactly the indicated values before each series of measurement. The pH electrode shall react quickly. Check that it can change from pH 4 to pH 7 within 5 s.

Check the sluggishness of the electrode and, if necessary, clean it carefully with the citric acid solution (6.3) and re-calibrate with the standard buffer solutions.

If the pH regulation system only accepts mV instead of pH for the target value, register mV values during calibration at pH 4 and pH 7 and calculate by interpolation the mV value corresponding to a pH target value of 4,5.

This mV value can vary over time for the same pH electrode.

This value shall be calculated each time a new series is being processed, just after calibration.

Checking and cleaning shall be more frequent for liming materials containing clay.

8.2.2 Select the pH regulation programme (generally called “pH stat”) and run set up adjustments.

A titration includes two successive steps that both consume citric acid solution:

— The pH lowers from pH ≈ 9 to the target value (pH = 4,5).

— Then, the regulation allows small solution flushes to keep the target pH.

Some titrators break the titration into these two steps, called “pre-titration” (reach target pH) and “titration” or “pH stat” (maintain target pH). In this method, pre-titration is part of the reaction and shall not be skipped. Choose the “no pre-titration” option for a total consumption of the solution (step 1 + step 2).

Generally, during titration, the target pH is obtained within 30 s to 2 min, depending on tested materials.

Let the system add citric acid solution as soon as deviations are higher than 0,01 pH (or 0,01 mV if the system only accepts target values expressed in mV).

The system shall be able to deliver at least an amount of 0,5 ml within 10 s. This is to ensure that, even with the most reactive products, the flow rate is not a limiting factor for the reaction.

Adjust the titration control so that the stepwise run of the titration is 0,001 ml. Set Ph = 4,5 as the target pH value. Set the stirring device speed control at 500 min^{-1} to 600 min^{-1}.

With an automatic sample changer (5.6), programme a 30 s stirring time before beginning titration.

Set the end of reaction at 15 min. Even when the target pH is reached, the reaction can continue, so the measurement shall be taken after precisely 15 min.

When using an automatic sample changer, insert a blank water (6.1) sample between two measurements, and programme the device so that the stirrer runs during 30 s to rinse the pH electrode. If necessary (dolomites), flush 1 ml or 2 ml of solution to clean the capillary pipe. If the samples contain clay (chalks), make sure the electrode remains clean.

NOTE Very reactive products can cause small projections and particles can stick to the electrode or to the top part of the beaker. This can alter the results.

8.2.3 Fill the burette (5.2) with citric acid solution (6.3).

8.2.4 If required (see 6.3), check the effective concentration of the citric acid solution (6.3) and record it.

An old solution can be more concentrated and modify results. Using a fresh solution is recommended.

8.2.5 Set up the electrode (5.1 or 5.2) and the capillary pipe in the glass beaker (5.3) according to Figure B.1. In the reaction beaker, the stirring device shall be located in the middle. The pH electrode shall be placed away from the acid introduction pipe such as 3/4 of the way around the edge of the beaker (see Figure B.1). This is to make sure that the added citric acid solution (6.3) is mixed with the content of the glass beaker before reaching the electrode (5.1 or 5.2). Avoid contact with the edge of the glass beaker.

8.2.6 For the exact adjustment of the operating conditions, start the stirring device (5.2) and add (5,00 ± 0,01) g of precipitated calcium carbonate (6.4) to the stirred water in the glass beaker (5.3). Start the stop-watch (5.6) and begin the titration with the automatic addition of the citric acid solution (6.3) until the pH target value is reached (8.2.2).

Check that the citric acid solution consumption is (15,0 ± 0,5) ml of citric acid (6.3) after 15 min. If the consumption of citric acid solution is below 14 ml, it can mean either:

a) the PCC (6.4) is not of the correct analytical grade for its physical characteristics. This can happen because only chemical values are certified. Consumption below 10 ml always means that the used PCC is not suitable; or

b) the settings of operating conditions are not correct and give lower values than expected.

In both cases, first make sure that operating conditions are as described in this document. Then test a highly reactive fine soft chalk. Generally, it approximately consumes 10 ml of citric acid and PCC is assumed to reach much higher values (about 15 ml).

If chalk gives higher results than PCC, the relative results are not as expected, and the reference PCC shall be changed for another one with higher reactivity. Test several types of PCC until 15 ml consumption is obtained.

If chalk gives lower results than PCC (good relative results), check the operating conditions again.

NOTE Citric acid consumption for PCC is only an additional way of checking. The volume consumed with PCC is only used to check calibration. It does not have any influence on the results themselves. If the operating conditions are properly set, the results can be trusted, whatever the PCC.

If the material being tested foams very strongly, one drop of silicon defoamer (6.5) should be added to the solution.

8.3 Measurement

8.3.1 Set up the apparatus as described in 8.2.1 to 8.2.6.

8.3.2 Pour the weighed test portion (see 7.3) into the glass beaker (5.3). Pour water (6.1) in the 100 ml beaker (5.3) and adjust the level to 80 ml. For liming materials coarser than 1 mm, use a 200 ml beaker and adjust to 160 ml. Let the stirring device run for 30 s. Start the stop-watch (5.6) and the titration procedure. If samples contain clay (chalks), clean the pH electrode after each titration.

NOTE Very reactive products can cause small projections and particles can stick to the electrode or to top edge of the beaker. This can alter results.

8.3.3 Stop the titration after 15 min and record the amount of citric acid solution consumed with three significant figures.

8.3.4 Rinse and clean the pH electrode with water (6.1). Do not take a rinsing beaker previously used. Cleaning the pH electrode can require stronger cleaning solutions than water, depending on the tested material. It is recommended to clean the pH electrode with a specific cleaning solution once every day.

For low reactive materials, i.e. those that require less than 2 ml of solution, such as dolomites, it is necessary to flush 1 ml or 2 ml of citric acid solution (6.3) after rinsing and cleaning to avoid capillary pipe contamination by the sample.

NOTE With an automatic sample changer, these different steps can be pre-set and are automatically processed once beakers with samples and water have been prepared. As carbonates are nearly insoluble in water, the preliminary duration of contact between carbonate and water has no influence.

8.3.5 Carry out the titration three times for each product to be tested. Take the mean acid consumption of the three titrations and record the average amount of citric acid solution (6.3) used.

8.4 Determination of neutralising value

Determine the neutralising value of the test sample as received in accordance with ISO 20978, and record the result to the nearest 0,1.

8.5 Determination of MgO content

Determine the MgO content of the test sample as received in accordance with a suitable method, such as EN 12946 or EN 12947, and record it.

9 Calculation and expression of the results

Calculate the citric acid reactivity, R_{CA}, expressed in percentages of the liming material being tested, using Formula (2).

$$R_{CA} = 100 \times \left[\frac{V_{CA}}{12}\right] \times \left[\frac{C_{CA}}{457{,}17}\right] \times \left[\frac{5}{m_t}\right] \times \left[\frac{56}{NV_{AR}}\right] \times \left[\frac{\left((\ 4-1\ ) \times \mathrm{MgO}\right)}{21} + 1\right] \quad (2)$$

where

V_{CA} is the mean volume consumption (8.3.5) of citric acid solution (6.3), in millilitres (ml);

12 is the conventional reference amount to which the mean consumption is compared, in millilitres (ml);

C_{CA} is the actual concentration of the citric acid solution (6.3 and 8.2.4), in grams per litre (g/l);

457,17 is the nominal concentration of the citric acid solution (6.3), in grams per litre (g/l);

5 is the dried standardized amount to be tested, in grams (g);

m_t is the mass of the test portion of liming material as received (see 7.3), in grams (g);

56 is the neutralising value of pure dry calcium carbonate;

NV_{AR} is the neutralising value of the test sample, expressed on an "as received" basis (see 8.4);

MgO is the MgO content expressed in per cent of "as received" product (see 8.5).

NOTE 1 MgO content is used to define a multiplying coefficient linked to MgO content that is equal to 1 when the sample contains no dolomite (0 % MgO) and to 4 when sample contains only dolomite (21 % MgO). This is made to balance the low rate of dissolution of dolomite in this conventional test, where dolomite is known to be more efficient in field conditions, although less than calcium carbonates.

NOTE 2 For materials coarser than 1 mm, as both V_{CA} and m_t are multiplied, Formula (2) remains valid.

NOTE 3 For PCC or for exceptionally reactive materials, R_{CA} can exceed 100 %. This is normal and due to the conventional consumption reference (12 ml, where PCC gives 15 ml).

NOTE 4 R_{CA} is not a percentage of the total possible dissolution (31,48 ml) (see Reference [13]). It is only a percentage of the conventional consumption reference (12 ml) used to express results in a traditional range from 0 % to about 100 % for almost all traditional liming materials.

NOTE 5 The reaction is stopped early before total dissolution. It allows a quick assessment of the material reactivity. Waiting for a total dissolution is not necessary, because citric acid is added in unlimited amounts. In such conditions, all products have the same reactivity.

NOTE 6 The reactivity percentage, wrongly considered as a dissolution rate, does not mean that the undissolved part never reacts in soil. Field conditions of dissolution can bring different dissolution rates. It only gives an idea of the reaction speed of the material.

NOTE 7 For wet products, which are tested as received, moisture is taken into account through the "as received" neutralising value correction factor used in Formula (2).

10 Precision

10.1 Inter-laboratory tests

The precision data were derived from an inter-laboratory test involving 16 participants, which was carried out in 2010 and analysed a range of liming materials including limestone, dolomitic limestone, magnesian limestone and chalk of varying fineness or presentation. As some deviations resulted from

a lack of experience or training, which is not expected in routine measurement, all the data from laboratories giving a mean value out of (100 ± 10) % of the median value of the level were taken out before calculation.

Repeatability and reproducibility were calculated using ISO 5725-1 and ISO 5725-2.

The values derived from this test might not be applicable to concentration ranges and matrices other than those given.

In order to assess the uncertainty of the method itself, and to avoid a cumulative uncertainty of correction factors, the statistical treatment of the ring test was made on the basis of the consumption of citric acid solution (V_{CA}). However, to get the final precision of results, expressed as reactivity (R_{CA}) instead of millilitres of citric acid solution (V_{CA}), laboratories shall consider the precision data of all the methods mentioned in Formula (2). Otherwise, values in Table 1 underestimate the true uncertainty of the method.

10.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, shall in no more than 5 % of the cases be greater than the repeatability limit r given in Table 1.

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, shall in no more than 5 % of the cases be greater than the reproducibility limit R given in Table 1.

The repeatability limits, r, and reproducibility limits, R, expressed as absolute and relative per cent values, are given in Table 1.

Table 1 — Mean values, repeatability and reproducibility limits

Liming material tested	Fineness 80 % passing µm	Number of laboratories	$\bar{x}$ citric acid consumption ml	Repeatability limit r (absolute)	Reproducibility limit R (absolute)	Repeatability limit r % (relative)	Reproducibility limit R % (relative)
Limestone fine	56	12	6,668	0,693	1,280	10,4	19,2
Limestone coarse	691	14	4,919	0,617	0,816	12,5	16,6
Limestone granulated	3 860	8	6,981	1,120	1,080	16,0	15,5
Dolomite + limestone granulated	5 030	16	2,524	0,212	0,365	8,4	14,5
Magnesian limestone fine	138	11	4,415	0,510	0,721	11,6	16,3

Table 1 *(continued)*

Liming material tested	Fineness 80 % passing µm	Number of laboratories	$\bar{x}$ citric acid consumption ml	Repeatability limit r (absolute)	Reproducibility limit R (absolute)	Repeatability limit r % (relative)	Reproducibility limit R % (relative)
Magnesian limestone coarse	712	14	2,527	0,356	0,453	14,1	17,9
Dolomite fine	113	6	0,627	0,106	0,107	16,9	17,0
Dolomite coarse	340	5	0,299	0,074	0,071	24,7	23,9
Chalk fine	851	10	8,617	0,919	1,200	10,7	13,9
Soft chalk coarse	1 080	11	9,508	1,127	1,404	11,9	14,8

NOTE 1 The analytical data used to calculate r and R is the volume of citric acid solution, in millilitres, poured for exactly 5 g of dry material within 15 min.

NOTE 2 For dolomite, which had higher r and R values in the ring test, the method has been improved to get better repeatability and reproducibility.

11 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: ISO 22146:2018;

c) the results and units in which the results are expressed;

d) any particular points observed in the course of the test;

e) all operating details not specified in this document, or regarded as optional, together with details of any incidents that occurred when performing the method and that might have influenced the test result(s).