BRITISH STANDARD

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Fertilizers and liming materials — Determination of carbon dioxide —

Part 2: Method for liming materials

The European Standard EN 14397-2:2004 has the status of a British Standard

ICS 65.080

National foreword

This British Standard is the official English language version of EN 14397-2:2004.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilizers and related chemicals, which has the responsibility to:

— aid enquirers to understand the text;

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English version

Fertilizers and liming materials - Determination of carbon dioxide - Part 2: Method for liming materials

Engrais et amendements minéraux basiques - Détermination de la teneur en dioxide de carbone - Partie 2: Méthode applicable aux amendements minéraux basiques

Düngemittel und Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung von Kohlenstoffdioxid - Teil 2: Verfahren für Calcium-/Magnesium-Bodenverbesserungsmittel

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Contents

Foreword

This document (EN 14397-2:2004) 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of an identical text or by endorsement, at the latest by January 2005, and conflicting national standards shall be withdrawn at the latest by January 2005.

EN 14397 "Fertilizers and liming materials — Determination of carbon dioxide" consists of two parts:

- *Part 1: Method for solid fertilizers (prCEN/TS 14397-1)*

- *Part 2: Method for liming materials*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This document specifies a method for the determination of carbon dioxide in all liming materials.

2 Normative references

The following referenced documents are indispensable for the application 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.

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

EN 12944-3:2001, *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).*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-3:2001 apply.

4 Principle

The carbon dioxide contained in liming materials in the form of carbonates is liberated by reaction with hydrochloric acid and determined volumetrically.

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.

5.2 Concentrated hydrochloric acid, ρ_{20} (HCl) = 1,18 g/ml to 1,19 g/ml

5.3 Diluted hydrochloric acid, ρ_{20} (HCl) = 1,09 g/ml

Dilute one part by volume of concentrated hydrochloric acid (5.2) with one part by volume of water.

5.4 Concentrated sulfuric acid, ρ_{20} (H_2SO_4) = 1,84 g/ml

5.5 Sodium sulfate, Na_2SO_4

5.6 Sealing liquid

Add 20 g of sodium sulfate (5.5) and 5 ml of concentrated sulfuric acid (5.4) to water and make up to 100 ml. Colour with a few drops of methyl red solution (5.8). The sealing liquid shall be saturated with carbon dioxide.

5.7 Ethanol, w (C_2H_5OH) = 95,6 % (mass fraction)

5.8 Methyl red solution

Dissolve 0,1 mg of methyl red in 50 ml of ethanol (5.7). Dilute with water up to 100 ml and homogenize.

5.9 Potassium hydroxide solution, w (KOH) = 50 % (mass fraction)

5.10 Calcium carbonate, $CaCO_3$, pre-dried to constant mass at 250 °C

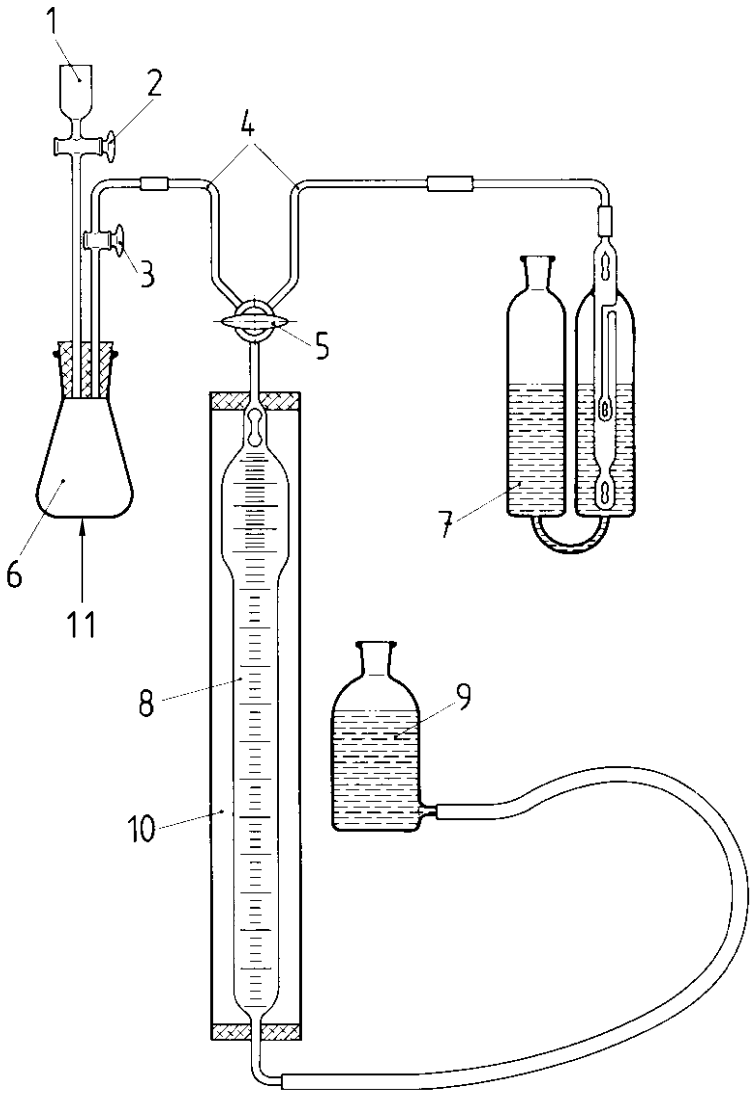
5.11 Copper sulfate, $CuSO_4 \cdot 5\ H_2O$

6 Apparatus

6.1 Apparatus for liberation and determination of carbon dioxide as shown in Figure 1

6.2 Laboratory balance capable of weighing to the nearest 0,0001 g

6.3 Heating device, electrical or gas-burning



Key

1 Dropping funnel with a volume of 25 ml
2 Stopcock 1
3 Stopcock 2
4 Capillary tubes
5 Three-way tap
6 Heated decomposition flask with a volume of 50 ml
7 Absorption vessel with a volume of 100 ml containing KOH solution
8 Measuring burette with a volume of 100 ml
9 Levelling bottle with a volume of 200 ml containing sealing liquid
10 Tube filled with water
11 Heating device

Figure 1 — Apparatus for liberation and determination of carbon dioxide

7 Sampling

Sample the liming material in accordance with EN 1482.

8 Procedure

8.1 Test portion

The test portion used depends on the expected carbon dioxide content and shall be chosen according to Table 1.

Table 1 — Test portion

Expected carbon dioxide content mass fraction	**Test portion** g
> 15 % ≤ 50 %	0,1
> 10 % ≤ 15 %	0,3
> 5 % ≤ 10 %	0,5
> 2 % ≤ 5 %	1,0
0 % ≤ 2 %	2,0

8.2 Determination

Weigh the test portion (8.1) to the nearest 0,1 mg into the decomposition flask. Bond any hydrogen sulfide formed by adding a spatula-tip of copper sulfate (5.11) and suspend in a little water.

Connect the decomposition flask to the rest of the apparatus (see Figure 1) with a double-bored stopper. A dropping funnel and the feed tube to the measuring burette pass through the stopper. Open the stopcocks in these two lines. Bring the three-way tap to the position such that it connects the flask and measuring burette with one another. Fill the burette with sealing liquid (5.6) up to the three-way tap by raising the levelling bottle. Now close stopcock 1 and fill the funnel with diluted hydrochloric acid (5.3). Add diluted hydrochloric acid (5.3) to the flask through the dropping funnel until the flask is half full. With stopcock 1 closed a little acid remains as sealing liquid in the funnel.

Allow the mixture to react for about 3 min and then heat it to the boiling point and boil for about 3 min. Fill the decomposition flask completely with diluted hydrochloric acid (5.3) up to stopcock 2 through the dropping funnel to transfer the remaining gas mixture into the burette. Take care that no diluted hydrochloric acid overflows. Close the burette with the three-way tap. After about 5 min, bring the sealing liquid (5.6) in the burette and in the levelling bottle to the same level and record the gas volume V_1.

Turn the three-way tap to connect the measuring burette with the absorption vessel and wash out the air-carbon dioxide-mixture collected. For this, raise the levelling bottle so that all the gas is forced through the potassium hydroxide solution (5.9) in the absorption vessel. The carbon dioxide is thereby absorbed. Repeat the absorption operation by raising the levelling bottle seven or eight times until, finally, the measuring burette contains only the residual gas. Close the three-way tap, bring the sealing liquid (5.6) in the burette and in the levelling bottle to the same level and record the gas volume V_2.

NOTE The volume difference $V_1 - V_2$ corresponds to the carbon dioxide content of the sample.

8.3 Calibration of the apparatus

Weigh 0,1 g of calcium carbonate (5.10) to the nearest 0,0001 g into the decomposition flask (see Figure 1). Carry out the determination as described in 8.2.

NOTE The volume difference $V_3 - V_4$ corresponds to the carbon dioxide content of the calibration material.

Calculate the correction factor F_1 of the absorption apparatus from equation (1):

$$F_1 = \frac{82{,}96\,T}{(V_3 - V_4)\,p} \tag{1}$$

where

T is the measurement temperature, in Kelvins;

V_3 is the volume of the gas before absorption related to the calibration material, in millilitres;

V_4 is the volume of the gas after absorption related to the calibration material, in millilitres;

p is the corrected barometer reading, in hectopascals.

F_1 shall be in the range from 1,00 to 1,04. Otherwise the apparatus shall be checked for gas-tightness and proper functioning and the calibration repeated.

9 Expression of results

Calculate the carbon dioxide content w_{CO_2}, given as mass fraction in percent, from equation (2):

$$w_{CO2} = 0{,}053 F_1 \frac{(V_1 - V_2)p}{T\,m} \tag{2}$$

where

F_1 is the correction factor in accordance with equation (1);

V_1 is the volume of the gas before absorption related to the test portion, in millilitres;

V_2 is the volume of the gas after absorption related to the test portion, in millilitres;

p is the corrected barometer reading, in hectopascals.

T is the measurement temperature, in Kelvins;

m is the mass of the test portion, in grams.

If the calibration and determination are carried out directly after one another, the temperature and atmospheric pressure need not be taken into consideration. For this case, equation (2) is simplified to

$$w_{CO2} = \frac{4{,}397(V_1 - V_2)}{(V_3 - V_4)\,m} \tag{3}$$

10 Precision

10.1 General

The values of the repeatability limit *r* and the reproducibility limit *R* are derived from inter-laboratory tests with building lime products in accordance with EN 459-2.

10.2 Repeatability

The absolute difference between two single test results found on identical test material by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit r in not more than 5 % of cases. The repeatability limit r is calculated from $r = 2{,}8\ s_r$, where s_r is the repeatability standard deviation.

The value of s_r is given by the following equation:

$s_r = 0{,}065\ w_{CO_2}$

10.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories with different operators using different equipment, will exceed the reproducibility limit R in not more than 5 % of the cases. The reproducibility limit R is calculated from $R = 2{,}8\ s_R$, where s_R is the reproducibility standard deviation.

The value of s_R is given by the following equation:

$s_R = 0{,}09\ w_{CO_2}$

11 Test report

The test report shall contain at least the following information:

a) all data necessary for the complete identification of the sample;

b) a reference to this document;

c) the results and the 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.

Bibliography

EN 196-21, *Methods of testing cement — Part 21: Determination of the chloride, carbon dioxide and alkali content of cement.*

EN 459-2, *Building lime — Part 2: Test methods*

VDLUFA, Manual II of analysing methods for fertilisers. (VDLUFA-Verlag, Bismarckstraße 41 A, D-64293 Darmstadt).

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