BRITISH STANDARD

BS EN 15909:2010

Fertilizers — Determination of calcium and formate in calcium foliar fertilizers

ICS 65.080

British Standards

National foreword

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Düngemittel - Bestimmung von Calcium und Formiat in Calcium-Blattdüngemitteln

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Foreword

This document (EN 15909:2010) 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of an identical text or by endorsement, at the latest by December 2010, and conflicting national standards shall be withdrawn at the latest by December 2010.

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1 Scope

This European Standard specifies a method for the determination of the content of calcium and formate in calcium foliar fertilizers in the presence of calcium chloride. This is determined and calculated by individual analytical determination of the following components:

— Calcium (Ca^{2+}),

— Chloride (Cl^-),

— Formate ($HCOO^-$).

The method is applicable to calcium foliar fertilizers with a calcium content of approximately 30 %.

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 480-10, *Admixtures for concrete, mortar and grout — Test methods — Part 10: Determination of water soluble chloride content*

EN 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

EN ISO 3696:1995, *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 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

4.1 General

Three different analytical procedures are used for the determination of the components to be determined, calcium, formate and chloride, in calcium foliar fertilizers. The substances calcium formate and calcium chloride are calculated stoichiometrically from the analytical results determined in each case (4.2, 4.3 and 4.4) (see Clause 9).

4.2 Calcium

The calcium content is determined complexometrically with the diluted sample in strongly alkaline solution by means of adjusted 0,1 mol/l EDTA solution (5.1) and the calcium-specific indicator, calconcarboxylic acid (8.2).

4.3 Formate

The formate content is determined isocratically by reversed phase HPLC on the basis of the diluted sample (see 8.3). Evaluation is carried out with external standard.

4.4 Chloride

The chloride content is determined by potentiometric titration according to the following reaction (see 8.4); the sample is first diluted, acetone is added and then the sample is acidified with acetic acid:

$$Cl^- + Ag^+ \rightarrow AgCl\ (solid)$$

After each addition of $AgNO_3$ the measured potentials are measured and recorded using a titroprocessor. The endpoint is reached when the differential quotient $E/\Delta V$, i. e. the potential change E observed for each volume step ΔV, reaches its greatest value. This point can be determined using a titroprocessor or voltmeter. In this titration the chloride ions which are determined include other water-soluble halogen ions apart from fluorides. The total halogen content is designated chloride content. See EN 480-10 for the principles.

5 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696:1995).

5.1 EDTA solution, (ethylenedinitrilotetraacetic acid disodiumsalt dehydrate), c(EDTA) = 0,1mol/l.

5.2 Calcium carbonate, reference material for complexometry.

5.3 Calconcarboxylic acid, indicator for metal titration.

5.4 Methyl orange.

5.5 Sodium chloride, p. a.

5.6 Sodium hydroxide solution, p. a., ρ = 45 %.

5.7 Water, purified.

5.8 Ortho-phosphoric acid, p. a., w = 85 %.

5.9 Formate standard solution, for ion chromatography, ρ = 1 000 mg/l[1].

5.10 Silver nitrate solution, $c(AgNO_3)$ = 0,01 mol/l.

5.11 Diluted hydrochloric acid, p.a., dilute 1 volume of hydrochloric acid, w = 37 %, with 1 volume of water (5.7).

5.12 Sodium chloride, certified primary reference material.

5.13 Diluted acetic acid, dilute 1 volume of acetic acid, w = 98 % to 100 %, with 1 volume of water (5.7).

5.14 Acetone, p.a.

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1) Fluka Art. No: 49897-100 ML-F is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product.

5.15 Magnesium sulfate, anhydrous p.a.

6 Apparatus

6.1 Analytical balance, accuracy of ± 1 mg.

6.2 HPLC apparatus, equipped with an automatic sample injection system, UV detector and evaluation system.

6.3 Titroprocessor[2]**.**

6.3.1 Dosimat, 10 ml exchange unit, for 0,01 mol/l sodium chloride solution (8.4.4).

6.3.2 Dosimat, 20 ml exchange unit, for 0,01 mol/l silver nitrate solution (5.11).

6.3.3 Silver ring electrode[3]**.**

6.4 Volumetric flask, capacity 100 ml.

6.5 Volumetric flask, capacity 1 000 ml.

6.6 Bulb pipette, capacity 10 ml.

6.7 Bulb pipette, capacity 20 ml.

6.8 Bulb pipette, capacity 25 ml.

6.9 Bulb pipette, capacity 100 ml.

6.10 Burette, capacity 25 ml.

6.11 Agate mortar.

6.12 Magnetic stirrer.

6.13 Glass beaker, capacity 50 ml.

6.14 Glass beaker, capacity 250 ml.

6.15 Glass beaker, capacity 600 ml.

6.16 Weighing dishes.

7 Sampling and sample preparation

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2.

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[2] Metrohm DMS-Titrino 716 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product.

[3] Metrohm 6.0430.100 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product.

8 Procedure

8.1 Preparation of the test solution

Transfer approximately 1 000 mg ± 10 mg of the sample to be determined, accurately weighed to 0,1 mg, to a weighing dish (6.16) (weight m).

Transfer the test portion from the weighing dish to the volumetric flask (6.5) by rinsing with distilled water. Make the volumetric flask up to the mark with distilled water at 20°C, and dissolve (solution R1).

8.2 Determination of the calcium content (concentration C1)

8.2.1 Using a bulb pipette (6.9), transfer 100 ml of solution R1 (8.1) to a glass beaker (6.15), and make up to 400 ml with distilled water.

8.2.2 Render the solution alkaline (pH > 12) with 10 ml of sodium hydroxide solution (5.6).

8.2.3 Add approximately 0,3 g of indicator (8.2.6) to the alkaline solution and, immediately after dissolution, titrate with adjusted EDTA solution (5.1) until the colour changes from red to green (6.10 and 6.12). Consumption: ml 0,1 mol/l EDTA solution (V_1). The colour change can be improved significantly by adding a spatula tipful (approximately 20 mg to 40 mg) of magnesium sulfate (5.15).

8.2.4 Blank value: proceed as described in 8.2.2 and 8.2.3 with 400 ml of distilled water. Consumption: ml 0,1 mol/l EDTA solution (V_2).

8.2.5 Calculation

Calculate the calcium content, w_{Ca}, in percent (mass fraction) according to the following equation:

$$w_{\mathrm{Ca}} = \frac{(V_1 - V_2) \times 4,008 \times t \times 10 \times 100}{1000 \times m} \qquad (1)$$

where

V_1 is the consumption of 0,1 mol/l EDTA solution for sample, in millilitre;

V_2 is the consumption of 0,1 mol/l EDTA solution for blank value, in millilitre;

4,008 is 1/10 g/mol calcium;

t is the correction factor for 0,1 mol/l EDTA solution;

10 is the aliquot portion;

100 is the factor for conversion to percent;

1 000 is the 0,1 mol/l EDTA solution (stoichiometric to 1/10 g/mol calcium) in millilitre;

m is the mass of the test portion in grams (8.1).

8.2.6 Preparation of the indicator

Triturate 0,10 g of calconcarboxylic acid (5.3), 0,05 g of methyl orange (5.4) and 9,85 g of sodium chloride (5.5) in the agate mortar (6.11) to a homogenous powder.

8.2.7 Adjustment of correction factor (titre), for 0,1 mol/l EDTA solution (5.1)

Transfer 0,19 g to 0,21 g of calcium carbonate (5.2), accurately weighed to ± 0,1 mg, to a glass beaker (6.15), and dissolve in 50 ml of distilled water, if necessary adding a few drops of hydrochloric acid (5.11) until the solution is clear. Dry the calcium carbonate (5.2) at 120°C for at least 2 h before use.

Subsequently, make up to 400 ml with distilled water, and proceed as described in 8.2.2 to 8.2.4.

Calculate the correction factor, t, according to the following equation:

$$t = \frac{1000 \times m_1}{(V_1 - V_2) \times 10{,}009} \tag{2}$$

where

t	is the correction factor (titre) of 0,1 mol/l EDTA solution;
1 000	is the 0,1 mol/l EDTA solution (stoichiometric to 1/10 g/mol calcium carbonate), in millilitre;
V_1	is the consumption of 0,1 mol/l EDTA solution for calcium carbonate, in millititre;
V_2	is the consumption of 0,1 mol/l EDTA solution for blank value, in millilitre;
10,009	is 1/10 g/mol calcium carbonate;
m_1	is the weight of calcium carbonate (5.2), in grams.

8.3 Determination of formate content (concentration C2)

8.3.1 HPLC conditions

- Separation column[4], particle size 5 µm, 250 mm × 4 mm,
- Eluent: phosphoric acid 0,5 % (8.3.4.1).

8.3.2 Elution conditions

- Flow rate: 1,5 ml/min;
- Column temperature: 30 °C;
- Elution type: isocratic
- Injection volume: 20 µl;
- Run time: 5 min;
- Retention time of component to be determined: approximately 2 min;

8.3.3 Detection conditions

- Type: UV;
- Measurement wavelength: 214 nm.

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[4] LiChrospher 100 RP-8 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product.

8.3.4 Procedure

8.3.4.1 Preparation of eluent

Dissolve 5 g of ortho-phosphoric acid (5.8) in 1 000 ml of purified water (5.7).

8.3.4.2 Calibration

8.3.4.2.1 Use bulb pipette (6.7) to transfer 20 ml of the 1 000 mg/l formate standard solution m_0 (5.9) (refer to certificate for exact content $w0$ %) to a glass beaker (6.13). Add 10 ml of purified water (5.7) by pipette (6.6), and subsequently homogenize. Solution R01.

8.3.4.2.2 Chromatograph solution R01 under the conditions specified in 8.3.1 to 8.3.4.2, and determine the peak area for formate (A_0).

8.3.4.3 Determination of the content of the sample

Chromatograph solution R1 (8.1) under the conditions specified in 8.3.1 to 8.3.4.2, and determine the peak area for formate (A).

8.3.5 Calculation

Calculate the content of component to be determined in the sample, w_F, in percent (formate) according to the following equation:

$$w_F = \frac{A \times m_0 \times w0\%}{A_0 \times m} \times \frac{2}{3} \tag{3}$$

where

A	is the peak area of component to be determined in sample solution R1;
m_0	is the mass of calibration substance in formate standard solution (5.9), in grams;
$w0\%$	is the content of component to be determined in calibration substance for formate standard solution (5.9), in percent;
A_0	is the peak area of component to be determined in calibration substance for formate standard solution (R01);
m	is the mass of the test portion in grams (8.1);
⅔	is the aliquot portion, dilution of formate standard solution R01.

8.4 Determination of chloride content (concentration C3)

8.4.1 Instrument settings for the automatic titroprocessor (6.3)

Titration parameters:

- Density of measured points: 0,
- Min. increment: 50,0 µl,
- Signal drift: 20 mV/min,
- Start V: off.

8.4.2 Stop conditions

- Stop V: 15 ml,
- Stop U: off mV,
- Stop EP: off.

8.4.3 Evaluation

Using the titroprocessor (6.3), the following evaluation criteria are applicable:

- EP criterion: 5
- EP recog: largest
- Fix-EP1 U: off mV
- pK/HNP: off

Using an other equipment, different instrument settings may be applicable. See the appropriate user manual.

8.4.4 Preparation of sodium chloride solution 0,01 mol/l (for blank value determination)

Transfer (0,584 ± 0,001) g of sodium chloride (5.5) to a 50 ml glass beaker (6.13), transfer to a 1 000 ml volumetric flask (6.5) by rinsing with distilled water, and dissolve. Subsequently, make the volumetric flask up to the mark with distilled water at 20 °C, and transfer to the Dosimat stock flask (6.3.1) with 10 ml exchange unit.

NOTE In the case of samples with a very low chloride content, it is possible that during titration the highest potential change E is not clearly recognized if at all. The addition of a defined quantity of chloride ions to the sample to be tested has the effect of clearly indicating any potential change. This defined quantity of chloride ions is then determined separately as blank value, and allowance must be made in evaluation.

8.4.5 Preparation of methyl orange indicator solution

Transfer 0,1 g of methyl orange (5.4) to a 100 ml volumetric flask (6.4), and dissolve in distilled water. Subsequently, make the volumetric flask up to the mark with distilled water.

8.4.6 Procedure

8.4.6.1 Use bulb pipettes (6.8) to transfer 25 ml of solution R1 (8.1) and 25 ml of acetone (5.14) to a 250 ml glass beaker (6.14). Make up to 150 ml with distilled water. Acidify by adding 10 ml of diluted acetic acid (5.13) by bulb pipette (6.6). Subsequently, add 3 to 5 drops of methyl orange indicator solution (8.4.5) until there is a red coloration. The solution shall be coloured red. If this is not the case continue to add acetic acid (5.13) drop wise until there is a red coloration.

8.4.6.2 Use the titroprocessor (6.3) to add exactly 2,000 ml of 0,01 mol/l sodium chloride solution (8.4.4) from the exchange unit (6.3.1) to solution 8.4.6.1.

8.4.6.3 Titrate using the titroprocessor (6.3) with adjusted 0,01 mol/l silver nitrate solution (5.10) and exchange unit (6.3.2) under the conditions specified in 8.4.1 to 8.4.3, using a silver ring electrode (6.3.3). Consumption: ml 0,01 mol/l silver nitrate solution (V_2).

8.4.6.4 Determination of blank value of 0,01 mol/l sodium chloride solution (8.4.4).

Using a bulb pipette (6.8) transfer 25 ml of acetone (5.14) to a 250 ml glass beaker (6.14). Make up to 150 ml with distilled water. Acidify by adding 10 ml of diluted acetic acid (5.13) by bulb pipette (6.6). Subsequently add 3 to 5 drops of methyl orange indicator solution (8.4.5) until there is a red coloration. The solution shall be coloured red. If this is not the case continue to add acetic acid (5.13) drop wise until there is a red coloration.

Use the titroprocessor (6.3) to add exactly 2,000 ml of 0,01 mol/l sodium chloride solution (8.4.4) from the exchange unit (6.3.1).

8.4.6.5 Titrate using the titroprocessor (6.3) with adjusted 0,01 mol/l silver nitrate solution (5.10) and exchange unit (6.3.2) under the conditions specified in 8.4.1 to 8.4.3, using a silver ring electrode (6.3.3). Consumption: ml 0,01 mol/l silver nitrate solution blank value (V_1).

8.4.7 Calculation

Calculate the chloride content, w_{Ch}, in percent (mass fraction) according to the following equation:

$$w_{\mathrm{ch}} = \frac{0{,}3545 \times (V_2 - V_1) \times t \times 40 \times 100}{1000 \times m} \tag{4}$$

where

V_2	is the consumption of 0,01 mol/l $AgNO_3$ solution for sample, in millilitre;
V_1	is the consumption iof 0,01 mol/l $AgNO_3$ solution for blank value, in millilitre;
0,354 5	is 1/100 g/mol chloride;
t	is the correction factor for 0,01 mol/l $AgNO_3$ solution;
40	is the aliquot portion;
100	is the factor for conversion to percent:
1 000	is the 0,01 mol/l $AgNO_3$ solution (stoichiometric to 1/100 g/mol chloride), in millilitre;
m	is the mass of the test portion in grams (8.1).

8.4.8 Adjustment of correction factor (titre) of 0,01 mol/l silver nitrate solution with sodium chloride (5.12)

8.4.8.1 Dry the sodium chloride (5.12) at 110 °C for at least 4 h before use.

8.4.8.2 Transfer (0,584 ± 0,001) g of dried sodium chloride (5.12), accurately weighed to 0,000 1 g, to a 50 ml glass beaker (6.13) (weight m), and transfer to a 1 000 ml volumetric flask (6.5) with distilled water. Subsequently, make the volumetric flask up to the mark with distilled water at 20 °C (solution R2**)**.

8.4.8.3 Use bulb pipettes (6.6 and 6.8) to transfer 10 ml of solution R2 and 25 ml of acetone (5.14) to a 250 ml glass beaker (6.14), and make up to 150 ml with distilled water. Acidify by adding 10 ml of diluted acetic acid (5.13) by bulb pipette (6.6). Subsequently add 3 to 5 drops of methyl orange indicator solution (8.4.5) until there is a red coloration. The solution shall be coloured red. If this is not the case continue to add acetic acid (5.13) drop wise until there is a red coloration (solution R3).

8.4.8.4 Titrate solution R3 using the titroprocessor (6.3) with 0,01 mol/l silver nitrate solution to be adjusted (5.10) under the conditions specified in 8.4.1 to 8.4.3, using a silver ring electrode (6.3.3). Consumption: ml 0,01 mol/l silver nitrate solution (V).

8.4.8.5 Calculation of correction factor (titre) for 0,01 mol/l silver nitrate solution:

Calculate the correction factor (titre), t, for 0,01 mol/l $AgNO_3$ solution according to the following equation:

$$t = \frac{1000 \times m \times 10}{1000 \times V \times 0{,}5844} = \frac{m \times 10}{V \times 0{,}5844} \qquad (5)$$

where

10	is 1/100 ml solution R2;
V	is the consumption of 0,01 mol/l $AgNO_3$ solution, in millilitre;
0,584 4	is 1/100 g/mol NaCl;
m	is the weight of NaCl (5.12) in grams.

9 Calculation and expression of the result

9.1 Stoichiometric data

Calcium	[Ca = 40,08 g/mol]
Calcium formate	[$Ca(HCOO)_2$ = 130,11 g/mol]
Calcium chloride	[$Ca(Cl)_2$ = 110,99 g/mol]

1,445 18 = Factor for converting formate to calcium formate

1,565 22 = Factor for converting chloride to calcium chloride

3,246 26 = Factor for converting calcium to calcium formate

2,769 21 = Factor for converting calcium to calcium chloride

9.2 Expression of the result

9.2.1 C1 (8.2) = % Calcium

C2 (8.3) = % Formate

C3 (8.4) = % Chloride

9.2.2 % Calcium formate = concentration C2 (8.3) × 1,445 18

9.2.3 % Calcium chloride = concentration C3 (8.4) × 1,565 22

9.2.4 % Calcium (residual content) see 9.3

9.3 Determination of residual content of calcium (9.2.4)

9.3.1 % Calcium from calcium formate = (9.2.2): 3,246 26

9.3.2 % Calcium from calcium chloride = (9.2.3): 2,769 21

9.3.3 % Residual content of calcium (9.2.4) = C1 – (9.3.1 + 9.3.2)

10 Precision

10.1 Inter-laboratory test

An inter-laboratory test has been carried out in 2008 with 7 participating laboratories and 1 sample of fertilizer. This test yielded the data given in Annex A. Repeatability and reproducibility were calculated according to ISO 5725-1 and ISO 5725-2.

The values derived from this inter-laboratory test may not be applicable to concentration ranges and matrices other than those given in Annex A.

10.2 Repeatability

The absolute difference between two independent single test results, obtained with 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, will in not more than 5 % of the cases exceed the values of r given in Table 1.

10.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of the cases exceed the values of R given in Table 1.

Table 1 — Mean values, repeatability and reproducibility limits

Substance	$\bar{x}$	r	R
Calcium	30,093	0,241	0,685
Formate	54,693	1,056	3,354
Chloride	10,325	0,229	1,104

11 Test report

The test report shall contain at least the following information:

a) All information necessary for the complete identification of the sample;

b) The test method used with reference to this document;

c) The test results obtained;

d) Date of sampling and sampling procedure (if known);

e) Date when the analysis was finished;

f) Whether the requirement of the repeatability limit has been fulfilled;

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

Annex A
(informative)

Statistical results of the inter-laboratory tests

The precision of the method has been determined in the year 2008 in an inter-laboratory trial with 7 laboratories participating and carried out on 1 sample of fertilizer. The statistical results are given in Table A.1.

Table A.1 — Statistical results of the inter-laboratory trial

Parameter	Calcium	Formate	Chloride
Year of the test	2008	2008	2008
Number of participating laboratories	7	7	7
Number of laboratories after eliminating outliers	6	6	7
Mean value, $\bar{x}$, (%) (mass fraction)	30,093	54,693	10,325
Repeatability standard deviation s_r, (%) (mass fraction)	0,087	0,381	0,083
RSD_r (%) (mass fraction)	0,29	0,70	0,80
Repeatability limit r (2,83 s_r) (%) (mass fraction)	0,241	1,056	0,229
Reproducibility standard deviation, s_R (%) (mass fraction)	0,247	1,211	0,398
RSD_R (%) (mass fraction)	0,82	2,21	3,86
Reproducibility limit R (2,83 s_R) (%) (mass fraction)	0,685	3,354	1,104

Bibliography

[1] EN 1482-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4] *Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers*, Official Journal L 304, 21.11.2003, p. 1-194

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