SLOVENSKI STANDARD
SIST EN 15909:2010

01-december-2010

Gnojila - Določevanje kalcija in formiata v kalcijevih foliarnih (listnih) gnojilih

Fertilizers - Determination of calcium and formate in calcium foliar fertilizers

Düngemittel - Bestimmung von Calcium und Formiat in Calcium-Blattdüngemitteln

Engrais - Dosage du calcium et du formate dans les engrais calcium foliaire

Ta slovenski standard je istoveten z: **EN 15909:2010**

ICS:

65.080	Gnojila	Fertilizers

SIST EN 15909:2010 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15909

June 2010

ICS 65.080

English Version

Fertilizers - Determination of calcium and formate in calcium foliar fertilizers

Engrais - Dosage du calcium et du formiate dans les engrais calcium pour pulvérisation foliaire

Düngemittel - Bestimmung von Calcium und Formiat in Calcium-Blattdüngemitteln

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 15909:2010: E

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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of an identical text or by endorsement, at the latest by December 2010, and conflicting national standards shall be withdrawn at the latest by December 2010.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:

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

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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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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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:

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

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)