SLOVENSKI STANDARD

SIST-TS CEN/TS 15451:2008

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Fertilizers - Determination of chelating agents - Determination of iron chelated by EDDHSA by ion pair chromatography

Düngemittel - Bestimmung von Chelatbildnern - Bestimmung von Eisen-chelatisiertem EDDHSA mit Ionen-Paarchromatographie

Engrais - Dosage des agents chélatants - Dosage du fer chélaté par EDDHSA par chromatographie d'appariement d'ions

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TECHNICAL SPECIFICATION

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CEN/TS 15451

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English Version

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This document (CEN/TS 15451:2006) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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

1 Scope

This Technical Specification specifies a method for the chromatographic determination of the total amount of iron chelated by EDDHSA in commercial products.

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.

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

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1995)*

3 Principle

The iron chelates are determined by ion-pair high-performance liquid chromatography. When an iron chelate (anion) is added to a polar fluid (eluent), containing a large cation (ion-pair reagent), an ion pair is formed. This ion pair is retained by an apolar solid phase (stationary phase). The strength of the retention depends on the molecular size and its acidity. Each iron chelate presents a characteristic retention time and a characteristic spectrum depending on the chelating agent, and it is separated from the other substances present in the sample. The separation is carried out on a reverse phase silica column and an aqueous solution of TBA^+ (tetrabutylammonium) and acetonitrile as eluent. The detection is based on photometry at 480 nm. For Fe^{3+}/EDDHSA a gradient method is required.

4 Interferences

No interferences have been detected. Iron chelates with o,o-EDDHA, o,p-EDDHA, o,o-EDDHMA, EDTA, DTPA, CDTA, HEDTA, as well as the chelating agents do not interfere since they are separated from Fe^{3+}/EDDHSA.

5 Reagents

5.1 General

a) All reagents shall be of recognized analytical grade.

b) All water used for the preparation of eluent, standards, and sample solutions should be water type 1 according to EN ISO 3696.

c) When products with a declared purity of less than 99 % are used for the preparation of standard solutions, a correction should be made in order to obtain the required concentration in the solution.

5.2 Sodium hydroxide solution

c(NaOH) = 0,5 mol/l

Dissolve 20 g of NaOH in pellet form in a 1 litre-volumetric flask with water free of carbon dioxide. Dilute to the mark and homogenize.

5.3 Sodium hydroxide solution

c(NaOH) = 0,1 mol/l

Dissolve 4 g of NaOH in pellet form in a 1 litre volumetric flask with water free of carbon dioxide. Dilute to the mark and homogenize.

5.4 Hydrochloric acid solution

c(HCl) = 1,0 mol/l

Dilute 88 ml of hydrochloric acid (35 % HCl) to 1 000 ml with water.

5.5 Hydrochloric acid solution

c(HCl) = 0,1 mol/l

Dilute 50 ml of hydrochloric acid (5.4) to 500 ml with water.

5.6 Iron (III) nitrate solution

ρ(Fe) = 1 050 mg/l

Dissolve 0,7594 g of iron (III) nitrate nonahydrate $[Fe(NO_3)_3 \cdot 9H_2O]$ in 100 ml of water. Transfer to a 100 ml-volumetric flask. Dilute to the mark with water and homogenize. Check (for example by AAS) that the Fe concentration of this solution is (1 050 ± 30) mg/l.

As the $Fe(NO_3)_3 \cdot 9H_2O$ is deliquescent, for preparation of the Fe^{3+}/EDDHSA solution (5.7) it shall be added in solution of a known concentration.

5.7 Fe^{3+}/EDDHSA solution

ρ(Fe) = 100 mg/l

Dissolve 0,233 0 g×100/P, where P is the complexometric purity of the standard in percentage of the acid form H_6EDDHSA obtained by photometric titration with a Fe(III)standard in a pH stat system, of ethylene diamine di(2-hydroxy-5-sulfophenyl) acetic acid and its condensation products (EDDHSA) in 150 ml of water and 2,7 ml of NaOH (5.2). After complete dissolution, add 25 ml of the Fe solution (5.6) to the chelating agent solution stirring about 5 min. The solution is adjusted to pH 7,0 with NaOH solution (5.3). Let the solution stand overnight in darkness to allow excess Fe to precipitate as oxide. Filter quantitatively through a cellulose filter and make up to volume (250 ml) with water.

5.8 Eluent for the determination

Two solutions are used to make a linear gradient eluent:

Solution A: Add 3,33 ml of TBAOH [40 % (mass fraction) Tetrabutylammonium hydroxide solution in water] to 500 ml of water. Adjust pH to 6,0 with HCl (5.4 and 5.5). Add 350 ml of acetonitrile (HPLC grade) and make up to volume in a 1 litre-volumetric flask with water. Filter through 0,2 µm membrane filter (6.3 b).

Solution B: Add 3,33 ml of TBAOH [40 % (mass fraction) Tetrabutylammonium hydroxide solution in water] to 150 ml of water. Adjust pH to 6,0 with HCl (5.4 and 5.5). Add 750 ml of acetonitrile (HPLC grade) and make up to volume in a 1 litre-volumetric flask with water. Filter through 0,2 µm-membrane filter (6.3 b).

NOTE 1   TBACl or TBABr may be used, providing that pH is adjusted to 6,0 with NaOH or HCl.

NOTE 2   Tetrabutylammonium bisulfate should be avoided because peak retention times and shape may significantly change.

6 Apparatus

Usual laboratory equipment, glassware, and:

6.1 Magnetic stirrer

Magnetic stirrer with magnets.

6.2 Chromatograph

Chromatograph equipped with:

a)   gradient pump delivering the eluent at a flow rate of 1,5 ml/min (see Table 1);

b)   injection valve with a 20 µl injection loop;

c)   C18 column; 150 mm×3,9 mm ID; dp = 5 µm[1)];

d)   use of a C18 guard column is recommended;

e)   UV/VIS-detector with a 480 nm-filter or diode array;

f)   integrator.

Table 1 — Gradient table

Time	Flow rate	A	B	Curve type
min	ml/min	%	%	
	1,5	100	0	Linear
5	1,5	100	0	Linear
6	1,5	0	100	Linear
11	1,5	0	100	Linear
12	1,5	100	0	Linear
20	1,5	100	0	Linear

6.3 Membrane filters

a)   Micromembrane filters resistant to aqueous solutions, with porosity of 0,45 µm;

b)   Micromembrane filters resistant to organic solutions, (e.g.: nylon micromembrane filters) with porosity of 0,2 µm.

7 Sampling and sample preparation

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

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1)   SYMMETRY[TM] C18, from WATERS cat.nº: WAT054205, or equivalent are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of these products.

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

NOTE   For the size reduction of samples with a high amount of chelating agents, it is not recommended to use a high speed laboratory mill. It is more convenient to grind the sample in a mortar to a particle size less than 1 mm.

8   Procedure

8.1 Preparation of the sample solution of iron chelates

Weigh approximately 125 mg within 0,1 mg, into a 100 ml-beaker (for samples with more than 4 % of chelated iron weigh 80 mg and for samples with less than 2 % of chelated iron weigh 250 mg). Add 80 ml of water. Stir on a magnetic stirrer for 30 min. Dilute to the mark in a 100 ml-volumetric flask with water and homogenize.

8.2 Preparation of the calibration solutions

Pipette a volume (V ml) (see Table 2) of the Fe^{3+}/EDDHSA standard solution (5.7) in six 100 ml-volumetric flasks respectively. Make up to volume with water and homogenize.

Table 2 — Preparation of the calibration curve of Fe^{3+}/EDDHSA

Solution	V ml	Concentration of Fe in the form of Fe^{3+}/EDDHSA mg Fe/l
1	1	1,0
2	5	5,0
3	10	10,0
4	25	25,0
5	50	50,0
6	100	100

8.3 Chromatographic analysis

Immediately before injection, all solutions should be filtered through a 0.45 μm-membrane filter (6.3 a). Inject the standard solutions (8.2.) into the chromatographic system (6.2). Measure the retention times and the areas of the Fe^{3+}/EDDHSA chromatographic peaks for all solutions. Two chromatographic peaks appear for Fe^{3+}/EDDHSA, corresponding with the different forms of Fe^{3+}/EDDHSA (see Annex A). Draw the calibration graph with the added values of the two chromatographic peaks of the standard solutions of Fe^{3+}/EDDHSA versus the chelated iron concentration (mg Fe/l) in the standards.

Inject the sample solution (8.1). Identify the chelating agent by the retention time of the obtained peaks (see Annex A) and if diode array detector is used, confirm with its UV-visible spectrum (see Annex B). Measure the areas of the peaks for the sample solution corresponding with the iron chelated by EDDHSA as chelating agent. Determine the concentration of the iron chelated (mg Fe/l) using the calibration graph.

NOTE   Normally the two chromatographic peaks in Fe^{3+}/EDDHSA appear overlapped. For integration use baseline correction as indicated in Annex A, Figure A.1.