

ICS 65.080

English Version

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

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

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

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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 Fe³⁺/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 Fe³⁺/EDDHSA.

5 Reagents

5.1 General

- All reagents shall be of recognized analytical grade.
- All water used for the preparation of eluent, standards, and sample solutions should be water type 1 according to EN ISO 3696.
- 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

$c(\text{NaOH}) = 0,5 \text{ mol/l}$

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

$c(\text{NaOH}) = 0,1 \text{ mol/l}$