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English Version

Fertilizers - Determination of chelating agents - Determination of iron chelated by o,p-EDDHA by reversed phase HPLC

Engrais - Dosage des agents chélatants - Dosage du fer chélaté par o,p-EDDHA par chromatographie liquide à haute performance à polarité de phase inversée

Düngemittel - Bestimmung von Chelatbildnern -Bestimmung von Eisen-chelatisiertem o,p-EDDHA mit Umkehrphasen HPLC

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Foreword

This document (CEN/TS 15452:2006) 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.

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1 Scope

This Technical Specification specifies a method for the chromatographic determination of the amount of iron chelated by each of the individual isomers of the chelating agent ortho-para EDDHA (o,p-EDDHA) in fertilizers. The method allows the identification of this chelating agent and the determination of the water soluble fraction of iron chelated by this chelating agent. The method is not applicable for the determination of the amount of free chelating agent.

NOTE 1 This method has been shown to be also suitable for the determination of the amount of iron chelated by each of the individual isomers of the chelating agent ortho-ortho EDDHA (o,o-EDDHA) in fertilizers.

NOTE 2 o,o-EDDHA and o,p-EDDHA are abbreviations used in this standard for the sake of simplicity. For complete names see Annex C.

NOTE 3 The substances o,o-EDDHA and o,p-EDDHA both exist as different stereoisomers. For o,o-EDDHA a meso form and a d/l pair (the racemic isomers) exist, for o,p-EDDHA two different d/l pairs exist. All four stereoisomers are observed separately in this method.

NOTE 4 Presently, an analytically pure standard only exists for o,o-EDDHA. The method for o,p-EDDHA has been developed with an o,p-EDDHA standard containing an uncertain concentration of o,p-EDDHA.

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 3696:1987)

3 Principle

The technique used is reversed phase HPLC with UV detection at 277 nm. The sample is separated on a silica-based reversed phase column using sodium formate, c = 0,015 mol/l, pH = 3,0, and acetonitrile as mobile phase.

For both o,p-EDDHA and o,o-EDDHA, two stereoisomer peaks are observed.

The concentration of iron chelated by o,p-EDDHA (o,p-Fe) is determined according to the external standard method.

4 Interferences

No interferences have been detected. Iron chelates with EDTA, HEDTA, DTPA and EDDHMA do not interfere.

5 Reagents

5.1 General

a) all reagents shall be of recognized analytical grade.