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English version

Fertilizers - Determination of chelating agents EDDHA and EDDHMA by ion pair chromatography - Comparison of non-standardized Lucena method with EN 13368-2:2001

Engrais - Détermination des agents chélatants EDDHA et EDDHMA par chromatographie ionique - Comparaison entre la méthode Lucena non normalisée et la norme EN 13368-2:2001

Düngemittel - Bestimmung der Chelatbildner EDDHA und EDDHMA mit Ionenpaarchromatographie - Vergleich der nicht standardisierten Lucena-Methode mit EN 13368-2:2001

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Foreword

This document (CEN/TR 15106:2005) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

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Introduction

European Regulation (EC) No 2003/2003 of the European parliament and of the Council of 13th October 2003 relating to fertilizers states that the synthetic commercial iron chelates shall be water soluble products obtained by the chemical combination of iron with a chelating agent. The iron (Fe) chelated by each chelating agent shall be declared on the label.

EN 13368-2:2001 specifies a method for the determination of EDDHA and EDDHMA by ion chromatography. This method determines the amount of chelated iron in Fe-EDDHA and Fe-EDDHMA commercial products. A ring test performed by CEN/TC 260, showed that reproducibility data were poor, possibly due to the low number of participating laboratories.

Some laboratories used a non-standardized method [1], [2], based on ion-pair chromatography. This can be performed using common chromatography equipment and some experiences [3] suggested that it can give adequate reproducibility and repeatability data.

CEN/TC 260/WG 5 agreed to launch a new work item in order to compare the reproducibility, repeatability and accuracy of the two methods (standardized and non-standardized). The aim being to develop a European Standard for the non-standardized method if better results were obtained. The results of the first ring test (ring test A) were not significant enough to confirm that the non-standardized method is better than the method standardized in EN 13368-2:2001.

The comments of the participating laboratories (see Annex B) suggested that the non-standardized method could be improved with some modifications which could then result in significant differences with respect to EN 13368-2:2001.

For this reason, WG 5 decided to carry out a second ring test (ring test B) using the method with the suggested modifications.

This Technical Report includes the results and conclusions from the two ring tests.

1 Scope

This Technical Report covers the results from two ring tests (A and B, see Introduction) to compare the repeatability and reproducibility of the standardized method for the determination of EDDHA and EDDHMA by ion chromatography (EN 13368-2:2001) with a non-standardized alternative method using ion-pair chromatography ("Lucena-method", given in Annex A). This Technical Report also provides the results of an univariate multifactorial ANOVA test and F-test for ring test A and F-test for ring test B to verify the significance of the reproducibility and the repeatability data.

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 13368-2:2001, *Fertilizers - Determination of chelating agents in fertilizers by ion chromatography - Part 2: EDDHA and EDDHMA*.

3 Test procedure for ring test A

3.1 Methods used in ring test A for the determination of chelating agents EDDHA and EDDHMA by chromatography.

Both methods tested for the determination of EDDHA and EDDHMA are based on high performance liquid chromatography.

The standardized method, based on ion chromatography is given in EN 13368-2:2001.

The non-standardized method, based on ion-pair chromatography is given in Annex A (without the modifications presented in italics, which were only used for ring test B).

3.2 Test Samples

Four different samples were provided to all the participants. Two samples were commercial iron chelates in solid form, one containing Fe-EDDHA and the other containing Fe-EDDHMA. The other two samples were solutions prepared from commercially available standards.

3.3 Ring test A procedure

The test samples were sent to 15 private or official laboratories from seven countries of which only 14 provided results.

The participating laboratories were requested to carry out 2 replicate analyses of each sample according to each method.

Table 1 shows the samples analysed by each participating laboratory.