

Fertilizers and liming materials — Determination of cadmium content

The European Standard EN 14888:2005 has the status of a
British Standard

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National foreword

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Summary of pages

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English version

Fertilizers and liming materials - Determination of cadmium content

Engrais et amendements minéraux basiques - Dosage du cadmium

Düngemittel und Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung des Cadmiumgehaltes

This European Standard was approved by CEN on 27 June 2005.

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Foreword

This document (EN 14888:2005) 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of an identical text or by endorsement, at the latest by February 2006, and conflicting national standards shall be withdrawn at the latest by February 2006.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association to provide a means of conforming to Essential Requirements of the Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilizers.

Once this standard is cited in the Official Journal of the European Communities under that Regulation and has been implemented as a national standard in at least one Member State, compliance with the normative clauses of this standard confers, within the limits of the scope of this standard, a presumption of conformity with the relevant Essential Requirements of that Regulation and associated EFTA regulations.

WARNING — Other requirements and other EU Directives may be applicable to the product(s) falling within the scope of this standard.

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

Introduction

Because flame atomic absorption spectrometry (flame AAS) and inductively coupled plasma-optical emission spectrometry (ICP-OES) are widely used techniques for the determination of cadmium, both are described in this European Standard (as method A and method B respectively). Much of the procedure and many of the reagents are common to both methods.

1 Scope

This European Standard specifies two methods for the determination of the cadmium content, after extraction with nitric acid, of solid mineral fertilizers and rock phosphates. It is not applicable to organic and organo-mineral fertilizers. It is applicable to the determination of cadmium contents greater than 1 mg/kg.

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 1482, *Sampling of solid fertilizers and liming materials*

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 following terms and definitions apply.

3.1

limit of detection

smallest measured content from which it is possible to deduce the presence of the analyte with reasonable statistical certainty

NOTE The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations ($n > 10$, where n is the number of measurements).

3.2

limit of quantification

lowest content of the analyte which can be measured with reasonable statistical certainty

NOTE If both accuracy and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to ten times the standard deviation of the mean of blank determinations ($n > 10$, where n is the number of measurements).

4 Principle

The cadmium is extracted from the fertilizer by means of a nitric acid digestion. Its concentration is measured either by flame atomic absorption spectrometry (Flame-AAS) (Method A) or by inductively coupled plasma-optical emission spectrometry (ICP-OES) (Method B). Both methods are relative techniques. Cadmium concentrations are determined by the standard addition method.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade, and water conforming to grade 2 of EN ISO 3696:1995.

Stock solutions shall be replaced after a maximum of one year, but the standard solution shall be renewed monthly as a minimum. If a stock solution is prepared directly from a metal, care needs to be taken to ensure that the metal used is free of surface oxide layers.

WARNING — Cadmium is highly toxic. It is essential that all necessary measures are taken to avoid ingestion.

5.2 Nitric acid, 65 %, ρ approximately 1,42 g/ml

5.3 Diluted nitric acid, diluted to a concentration of about 7 mol/l (1 + 1 volume fraction).

Mix 1 volume of nitric acid (5.2) with 1 volume of water.

5.4 Diluted nitric acid, diluted to a concentration of about 4,3 mol/l (1 + 3 volume fraction).

Mix 1 volume of nitric acid (5.2) with 3 volumes of water.

5.5 Diluted nitric acid, diluted to a concentration of 2 % (volume fraction).

Pipette 20,00 ml nitric acid (5.2) in a 1 000 ml volumetric flask and fill to the mark with water.

5.6 Cadmium stock solution, corresponding to 1 000 mg/l.

Weigh to the nearest 0,1 mg, approximately 1,000 g of metallic cadmium (minimum purity 99,5 %) and dilute in a covered 250 ml glass beaker with 40 ml of nitric acid (5.4). Then add 100 ml of water. Boil to expel nitrous fumes, cool, transfer to a 1 000 ml volumetric flask and fill to the mark with water.

NOTE Cadmium stock solution of 1 000 mg/l is also readily available commercially, and may be used instead.

5.7 Cadmium standard solution, corresponding to 50 mg/l.

Pipette 50,00 ml of the cadmium stock solution (5.6) into a 1 000 ml volumetric flask and fill to the mark with the diluted 2 % nitric acid (5.5).