

**Fertilizers - Determination of trace elements -
Determination of cadmium, chromium, lead and nickel
by inductively coupled plasma-atomic emission
spectrometry (ICP-AES) after aqua regia dissolution**

EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

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English Version

Fertilizers - Determination of trace elements - Determination of cadmium, chromium, lead and nickel by inductively coupled plasma-atomic emission spectrometry (ICP-AES) after aqua regia dissolution

Engrais - Dosage des éléments trace - Détermination du cadmium, chrome, plomb et nickel par spectrométrie d'émission atomique avec plasma induit par haute fréquence (ICP-AES) après digestion à l'eau régale

Düngemittel - Bestimmung von Elementspuren - Bestimmung von Cadmium, Chrom, Blei und Nickel mit Atomemissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-AES) nach Königswasseraufschluss

This European Standard was approved by CEN on 15 September 2013.

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Foreword

This document (EN 16319:2013) 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of an identical text or by endorsement, at the latest by April 2014, and conflicting national standards shall be withdrawn at the latest by April 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 16319:2012.

The following changes have been made to the former edition:

- the CEN Technical Specification has been adopted as a European Standard;
- an explanation concerning possible interferences when using this method for the determination of Cd, Cr, Ni and Pb in micro-nutrient fertilizers has been added as a NOTE to Clause 1;
- the document has been editorially revised.

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 implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the content of cadmium, chromium, nickel and lead in fertilizers with inductively coupled plasma-atomic emission spectrometry (ICP-AES) after extraction with aqua regia. Limits of quantification are dependent on the sample matrix as well as on the instrument, but can roughly be expected to be 0,3 mg/kg for Cd and 1 mg/kg for Cr, Ni and Pb.

NOTE Due to significant interference from Cu, Fe and Mn, no valid results can be reported using this method for fertilizer matrices containing high concentrations ($\geq 10\%$) of these micro-nutrients.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

Cadmium, chromium, nickel and lead are extracted from the sample with aqua regia and conventional boiling. The concentrations in the extract are measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES), with axial or radial viewing.

5 Sampling and sample preparation

Sampling is not part of the method specified in this European Standard. A recommended sampling method is given in EN 1482-1.

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

6 Reagents

Use only reagents of recognized analytical grade.

Commercially available stock solutions shall be replaced according to the specifications from the supplier or after one year if prepared in the laboratory from available salts. Standard solutions shall be renewed monthly as a general rule.

6.1 Water, conforming to grade 2 according to EN ISO 3696.

6.2 Hydrochloric acid, $c(\text{HCl}) = 12 \text{ mol/l}$; 37 % volume fraction; $\rho \approx 1,18 \text{ g/ml}$.

6.3 Nitric acid, $c(\text{HNO}_3) = 16 \text{ mol/l}$; not less than 65 % volume fraction; $\rho \approx 1,42 \text{ g/ml}$.