



BSI Standards Publication

**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**

National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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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 traces - Détermination du cadmium, chromium, 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

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Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (CEN/TS 16319:2012) 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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1 Scope

This Technical Specification 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.

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 methods specified in this Technical Specification. 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, $\rho \approx 1,42 \text{ g/ml}$.

6.4 Mixed solution of 0,8 mol/l nitric acid and 1,8 mol/l hydrochloric acid

Mix 150 ml of hydrochloric acid (6.2) and 50 ml nitric acid (6.3) to 1,0 l of water (6.1).

6.5 Standard stock solutions cadmium, chromium, nickel and lead standard stock solutions, e.g. $\rho = 1\,000 \text{ mg/l}$ for each element.

Use suitable stock solutions. Both single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. It is recommended to use commercially available standard stock solutions for cadmium, chromium, nickel and lead.

6.6 Working standard solutions

Depending on the scope, different working standard solutions may be necessary.

In general, when combining elements in working standard solutions, their chemical compatibility shall be regarded. Spectral interferences from other elements present in working standard solutions also need to be considered. Various combinations of elements at different concentrations may be used, provided that the standard stock solutions (6.5) are diluted with the same acid and in equal concentration as the acid in the test solution.

NOTE In equal concentrations (in mg/l), cadmium, chromium, nickel and lead are compatible in a multi-element standard solution for the determination by ICP-AES for this application.

6.6.1 Working standard solution I $\rho = 100 \text{ mg/l}$ for cadmium, chromium, nickel and lead.

Dilute 10,0 ml of each standard stock solution of cadmium, chromium, nickel and lead (6.5) to 100,0 ml with the mixed acid solution (6.4) in the same 100 ml flask. If non-equal concentrations of cadmium, chromium, nickel and lead are needed, dilute the required volumes into 100,0 ml. This solution is used to prepare spiked test solutions and standard and calibration solutions.

6.6.2 Working standard solution II $\rho = 10 \text{ mg/l}$ for cadmium, chromium, nickel and lead.

Dilute 10,0 ml of the working standard solution I of cadmium, chromium, nickel and lead (6.6.1) to 100,0 ml with the mixed acid solution (6.4) in a 100 ml flask. If non-equal concentrations of cadmium, chromium, nickel and lead are needed, dilute the required volume from the standard stock solutions (6.5) into 100,0 ml. This solution is used to prepare spiked test solutions and calibration solutions.

7 Apparatus

7.1 Common laboratory glassware

7.2 Analytical balance capable of weighing to an accuracy of 1 mg.