



SLOVENSKI STANDARD
SIST EN 16319:2013/oprA1:2015
01-januar-2015

Gnojila - Določevanje elementov v sledovih - Določevanje kadmija, kroma, svınca in niklja z atomsko emisijsko spektrometrijo z induktivno sklopljeno plazmo (ICP-AES) po raztapljanju v zlatotopki

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

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

Engrais et amendements minéraux basiques - 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

Ta slovenski standard je istoveten z: **EN 16319:2013/prA1**

ICS:

65.080 Gnojila Fertilizers

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EUROPEAN STANDARD
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English Version

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

Engrais et amendements minéraux basiques -
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 und Kalkdünger - Bestimmung von Cadmium,
Chrom, Blei und Nickel mit Atomemissionsspektrometrie mit
induktiv gekoppeltem Plasma (ICP-AES) nach
Königswasseraufschluss

This draft amendment is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 260.

This draft amendment A1, if approved, will modify the European Standard EN 16319:2013. If this draft becomes an amendment, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for inclusion of this amendment into the relevant national standard without any alteration.

This draft amendment was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Contents

	Page
Foreword.....	3
1 Modification to the Title	4
2 Modifications to Clause 1, Scope.....	4
3 Modification to Clause 2	4
4 Modification to Clause 3, Terms and definitions	4
5 Modification to Clause 10, Precision, Table 3.....	4
6 Modification to Clause 10, Precision, Table 4.....	5
7 Modification to Clause 10, Precision, Table 5.....	5
8 Modification to Clause 10, Precision, Table 6.....	5
9 Modification to Annex A.1.....	5
10 Modification to Annex A.2.....	6
11 Modification to Annex A.....	6

Foreword

This document (EN 16319:2013/prA1:2014) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

EN 16319:2013/prA1:2014 (E)**1 Modification to the Title**

Delete "Determination of trace elements" and insert "liming materials" after fertilizers to read the title as follows:

"Fertilizers and liming materials — Determination of cadmium, chromium, lead and nickel by inductively coupled plasma-atomic emission spectrometry (ICP-AES) after aqua regia dissolution".

2 Modifications to Clause 1, Scope

Replace the first sentence by the following sentence:

"This European Standard specifies a method for the determination of the content of cadmium, chromium, nickel and lead in fertilizers and liming materials using inductively coupled plasma-atomic emission spectrometry (ICP-AES) after aqua regia dissolution."

Insert the following NOTE 2 after NOTE 1:

"NOTE 2 The term fertilizer is used throughout the body of this European Standard and should be taken to include liming materials unless otherwise indicated."

3 Modification to Clause 2

Insert the following reference after EN 12944-2:1999:

"EN 12944-3:2001, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 3: Terms relating to liming materials*"

4 Modification to Clause 3, Terms and definitions

Modify the 1st sentence as follows:

"For the purposes of this document, the terms and definitions given in EN 12944-1:1999, EN 12944-2:1999 and EN 12944-3:2001 apply."

5 Modification to Clause 10, Precision, Table 3

Insert after the 3rd line (data for rock phosphate) the data for liming material samples:

Sample	\bar{x} mg/kg	r mg/kg	R mg/kg
Cadmium (Cd)			
Dolomitic limestone	0,967	0,039	0,250
Burnt lime	0,374	0,039	0,099
Converter lime	not detectable ^a		

^a Eight laboratories determined Cd in converter lime. For six laboratories the Cd contents were below the detection limit (< 0,05 - < 0,25). One laboratory found 0,4 mg/kg Cd, another one 0,83 mg/kg Cd in the mean.

6 Modification to Clause 10, Precision, Table 4

Insert after the 3rd line (data for rock phosphate) the data for liming materials samples:

Sample	\bar{x}	r	R
Chromium (Cr)	mg/kg	mg/kg	mg/kg
Dolomitic limestone	6,417	0,569	1,231
Burnt lime	6,534	0,377	1,390
Converter lime	1136	21	111

7 Modification to Clause 10, Precision, Table 5

Insert after the 3rd line (data for rock phosphate) the data for liming material samples:

Sample	\bar{x}	r	R
Nickel (Ni)	mg/kg	mg/kg	mg/kg
Dolomitic limestone	5,93	0,44	1,67
Burnt lime	2,500	0,168	0,767
Converter lime	10,83	1,28	4,83

8 Modification to Clause 10, Precision, Table 6

Insert after the 2nd line (data for rock phosphate) the data for liming material samples:

Sample	\bar{x}	r	R
Lead (Pb)	mg/kg	mg/kg	mg/kg
Dolomitic limestone	87,23	2,88	17,22
Burnt lime	2,509	0,153	0,741
Converter lime	10,20	0,47	6,69

9 Modification to Annex A.1

Modify the 1st sentence to read as follows:

"The precision of the method has been determined in an inter-laboratory trial in the year 2009 analysing samples of mineral fertilizer matrices and in the year 2013 analysing samples of liming material matrices. The statistical results are given in Table A.1 to Table A.4 for fertilizer samples and in Table A.5 to Table A.8 for liming material samples."

EN 16319:2013/prA1:2014 (E)

10 Modification to Annex A.2

Insert in the head line after nickel: "for fertilizers"

11 Modification to Annex A

Insert A.3 with the following headline:

"A.3 Statistical results for the determination of cadmium, chromium, lead and nickel for liming materials"

Insert after the headline A.3 the new Tables A.5 to A.8 as follows:

Table A.5 — Statistical results for cadmium

Parameter	Dolomitic limestone	Burnt lime
Number of laboratories	11	9
Number of laboratories retained after elimination of outliers	10	8
Number of outliers	1	1
Mean value, \bar{x} (mg/kg)	0,967	0,374
Repeatability standard deviation s_r (mg/kg)	0,039	0,039
Relative repeatability standard deviation RSD_r %	4,03	10,4
Repeatability limit r [$r = 2,8 \times s_r$] (mg/kg)	0,1092	0,1092
Reproducibility standard deviation s_R (mg/kg)	0,25	0,099
Relative reproducibility standard deviation RSD_R %	25,8	26,5
Reproducibility limit R [$R = 2,8 \times s_R$] (mg/kg)	0,700	0,277

Table A.6 — Statistical results for total chromium

Parameter	Dolomitic limestone	Burnt lime	Converter lime
Number of laboratories	13	13	13
Number of laboratories retained after elimination of outliers	13	13	12
Number of outliers	0	0	1
Mean value, \bar{x} (mg/kg)	6,42	6,53	1136,3
Repeatability standard deviation s_r (mg/kg)	0,57	0,38	21,5
Relative repeatability standard deviation RSD_r %	8,87	5,77	1,89
Repeatability limit r [$r = 2,8 \times s_r$] (mg/kg)	1,59	1,06	60,2
Reproducibility standard deviation s_R (mg/kg)	1,23	1,39	111
Relative reproducibility standard deviation RSD_R %	19,2	21,3	9,78
Reproducibility limit R [$R = 2,8 \times s_R$] (mg/kg)	3,45	3,89	311