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**Gnojila - Določevanje perklorata v mineralnih gnojilih s tekočinsko kromatografijo in tandemsko masno spektrometrijo (LC-MS/MS)**

Fertilizers - Determination of perchlorate in mineral fertilizers by liquid chromatography and tandem mass spectrometry detection (LC-MS/MS)

Düngemittel - Bestimmung von Perchlorat in mineralischen Düngemitteln - Bestimmung mittels Flüssig-Chromatographie und Tandem-Massenspektrometrischer Detektion (LC-MS/MS)

Engrais - Dosage du perchlorate dans les engrais minéraux par chromatographie liquide couplée à la spectrométrie de masse (LCMS/MS)

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**Ta slovenski standard je istoveten z: EN 17411:2020**

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**ICS:**

65.080	Gnojila	Fertilizers
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

**SIST EN 17411:2021****en,fr,de**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 17411**

November 2020

ICS 65.080

English Version

**Fertilizers - Determination of perchlorate in mineral fertilizers by liquid chromatography and tandem mass spectrometry detection (LC-MS/MS)**

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
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## Contents

	Page
European foreword.....	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions .....	4
4 Principle .....	4
5 Reagents .....	4
6 Apparatus and equipment .....	5
7 Procedure.....	5
7.1 Sampling and sample preparation.....	5
7.2 Matrix calibration curve.....	5
7.3 Determination of perchlorate in test solutions.....	6
8 Calculation .....	7
9 Precision.....	7
9.1 Inter-laboratory test.....	7
9.2 Repeatability.....	7
9.3 Reproducibility.....	7
10 Additional remarks .....	8
11 Test report.....	8
Annex A (informative) Results of the inter-laboratory test.....	9
Bibliography.....	10

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## European foreword

This document (EN 17411:2020) 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 May 2021, and conflicting national standards shall be withdrawn at the latest by May 2021.

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## EN 17411:2020 (E)

## 1 Scope

This document specifies a method for the determination of traces of perchlorate by liquid chromatography and tandem mass spectrometry detection (LC-MS/MS). The method is applicable to mineral fertilizers.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

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

EN 12944-2, *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 and EN 12944-2 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

## 4 Principle

The perchlorate content of the fertilizer sample is determined by liquid chromatography with tandem mass spectrometry detection after water extraction. The sample is dissolved, filtered and directly analysed by LC-MS/MS. Quantification is performed using matrix calibration curve.

## 5 Reagents

Use only reagents of recognized analytical grade.

**5.1 Water**, grade 3 according to EN ISO 3696.

**5.2 Blank solution**, free of perchlorate similar to the samples.

Weigh 5 g of potassium nitrate in 250 ml eluent (5.4).

**5.3 Perchlorate standard stock solutions**,  $\rho(\text{ClO}_4^-) = 1 \text{ mg/l}$  and  $10 \text{ mg/l}$ .

Prepare the standard stock solutions using commercially available standard stock solutions.

**5.4 Eluent**, used for LC-MS/MS (mobile phase).

Prepare 500 ml of mobile phase with 250 ml of water (5.1), 250 ml of acetonitrile and 500 µl of (concentrated) acetic acid in order to have a final concentration of acetic acid in the mobile phase equal to 0,1 % (v+v).

## 6 Apparatus and equipment

Usual laboratory equipment, glassware, and the following:

### 6.1 LC-MS/MS instrumentation:

#### 6.1.1 Column.

For perchlorate determination, it is recommended to use a KP-RPX 250 × 4.6, 5 µm column, from K' (PRIME) Technologies<sup>1</sup>. Similarly performing columns may also be used.

#### 6.1.2 Equipment.

Use a mass spectrometer equipped with electrospray ionization (ESI) in the negative mode. The mass transitions for the perchlorate ion are: (-) 99/83, (-) 101/85.

### 6.2 Analytical balance, with an accuracy of ±0,1 mg.

### 6.3 Graduated flasks, capacity 1 000 ml, 250 ml, 50 ml and 10 ml volumetric flasks.

### 6.4 Nylon filters, with 0,22 µm filter membrane.

### 6.5 Autosampler vials, suitable for LC auto-samplers.

## 7 Procedure

### 7.1 Sampling and sample preparation

Sampling is not part of this document. A recommended sampling method is given in EN 1482-1 [1].

Sample preparation shall be performed according to EN 1482-2.

### 7.2 Matrix calibration curve

The calibration curve is prepared in matrix to correct for matrix interferences.

For the calibration solution, take 5 ml of acetonitrile and transfer quantitatively to a 10 ml volumetric flask (6.3). Add an adequate volume of perchlorate standard solution (5.3) as given in Table 1 and bring to volume with the blank solution.

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<sup>1</sup> This column – KP-RPX 250 × 4.6, 5 µm column, from K' (PRIME) Technologies is an example of a suitable instrument available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

### Table 1 — Preparation of calibration solutions

<b>Calibration solution</b>	<b>Standard solution</b>	<b>Standard solution</b>	<b>Mass concentration of calibration solution</b>
	$\rho(\text{ClO}_4^-) = 1 \text{ mg/l}$ (5.3) $\mu\text{l}$	$\rho(\text{ClO}_4^-) = 10 \text{ mg/l}$ (5.3) $\mu\text{l}$	$\text{mg/l}$
1	50	-	0,005
2	100	-	0,01
3	-	20	0,02
4	-	50	0,05
5	-	100	0,1
6	-	200	0,2

Prepare a calibration curve by injecting the six calibration solutions (see Table 1) according to the operation procedure of the available instrument (6.1) at the beginning of every day of the analysis.

The calibration curve is described by the following Formula (1):

$$y = mx + b$$

SIST EN 17411:2021

(1)

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Calibration levels lower than 0,005 mg/l may result in not linear response when matrix calibration is used.

### 7.3 Determination of perchlorate in test solutions

**7.3.1** With the analytical balance (6.2) weigh 5 g of ground sample.

**7.3.2** Transfer the sample to a 250 ml volumetric flask (6.3) and add 150 ml 0,1 % acetic acid (5.4) to dissolve the sample. Bring to volume with 0,1 % acetic acid (5.4). Stir manually to homogenize (solution 1).

**7.3.3** Take an aliquot of 25 ml of solution 1 and transfer quantitatively to a 50 ml volumetric flask (6.3). Add acetonitrile to the mark and stir manually to homogenize (solution 2).

NOTE      Solution 1 (sample diluted with 1 % acetic acid) is diluted with acetonitrile to obtain solution 2 (solution 2 = 25 ml solution 1 + 25 ml acetonitrile).

**7.3.4** Depending on the concentration of perchlorates in the sample, solution 2 may be diluted again using blank solution (5.2).

**7.3.5** Filter the solution using a nylon filter (6.4). Discard the first filtrate and then collect the filtrate in a vial (6.5).

**7.3.6** Proceed with the analysis according to the operation procedure of the available instrument (6.1) using the same instrument and the same conditions as used for the preparation of the calibration curve.



## 8 Calculation

Calculate the the mass fraction of  $\text{ClO}_4^-$  in the fertilizer, in mg/kg, by using the following Formula (2):

$$w(\text{ClO}_4^-) = \frac{\rho \times D \times V}{m} \quad (2)$$

where

$w(\text{ClO}_4^-)$  is the mass fraction of  $\text{ClO}_4^-$  in the fertilizer, in mg/kg;

$\rho$  is the mass concentration of the reading of the sample from the calibration curve, in mg/l;

$D$  is the dilution used (here: 2);

$V$  is the volume of the sample, in ml (here: 250 ml);

$m$  is the mass of the test portion, in g (here: 5 g).

## 9 Precision

### 9.1 Inter-laboratory test

An inter-laboratory test was carried out in 2018 with seven participating laboratories analysing four different samples. Repeatability and reproducibility were calculated according to ISO 5725-2 [2]. Each laboratory carried out three determinations on each sample. The statistical results are explained in Table A.1.

The values derived from the inter-laboratory test might not be applicable to concentration ranges and matrices other than those given in Annex A.

### 9.2 Repeatability

The absolute difference between two independent test results obtained with the same method on identical test material, in the same laboratory, by the same operator, using the same equipment within a short interval of time, will in no more than 5 % of the cases exceed the values of  $r$  given in Table 2.

### 9.3 Reproducibility

The absolute difference between two single test results obtained with the same method on identical test material, in different laboratories, by different operators, using different equipment, will in no more than 5 % of the cases exceed the values of  $R$  given in Table 2.

**Table 2 — Mean values, repeatability and reproducibility limits**

Sample	Mean value mg/kg	$r$ mg/kg	$R$ mg/kg
1	21,0	3,9	6,6
2	5,8	0,7	4,1
3	34,7	4,4	18,3
4	13,7	1,1	6,6