SLOVENSKI STANDARD
SIST EN 17090:2018

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Gnojila - Določevanje inhibitorja nitrifikacije DMPSA v gnojilih - Metoda s tekočinsko kromatografijo visoke ločljivosti (HPLC)

Fertilizers - Determination of nitrification inhibitor DMPSA in fertilizers - Method using high-performance liquid chromatography (HPLC)

Düngemittel - Bestimmung des Nitrifikationshemmstoffs DMPSA in Düngemitteln - Verfahren mit Hochleistungs-Flüssigchromatographie (HPLC)

Engrais - Dosage de l'inhibiteur de nitrification DMPSA - Méthode par chromatographie liquide à haute performance (HPLC)

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English Version

Fertilizers - Determination of nitrification inhibitor DMPSA in fertilizers - Method using high-performance liquid chromatography (HPLC)

Engrais - Dosage de l'inhibiteur de nitrification DMPSA dans les engrais - Méthode par chromatographie liquide haute performance (HPLC)

Düngemittel - Bestimmung des Nitrifikationshemmstoffs DMPSA in Düngemitteln - Verfahren mit Hochleistungs-Flüssigchromatographie (HPLC)

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COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Page

European foreword

This document (EN 17090:2018) 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of an identical text or by endorsement, at the latest by May 2019, and conflicting national standards shall be withdrawn at the latest by May 2019.

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1 Scope

This document specifies a method for the determination of nitrification inhibitor 2-(3,4-dimethyl-pyrazol-1-yl)-succinic acid (DMPSA) using high-performance liquid chromatography (HPLC), which is applicable to all 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 http://www.iso.org/obp

4 Principle

The fertilizer sample is solved in water or extracted with water. The solved DMPSA is determined with HPLC/UV-detector.

5 Reagents

5.1 Water, grade 3 according to EN ISO 3696.

5.2 Acetonitrile, HPLC grade.

5.3 Phosphoric acid, w = 85 % for analysis.

5.4 DMPSA, CAS-Nr 940-877-5, purity > 99 %.

6 Apparatus

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

6.2 Volumetric flasks, capacity 100 ml and 1 000 ml.

6.3 Micro filter, pore size 0,45 µm.

6.4 HPLC unit, for isocratic operation with UV detector.

6.5 Reverse phase HPLC-column, e.g. C18, 7 µm, 250 mm × 4 mm.

Other types of columns equivalent to the one mentioned may be used.

7 Sampling and sample preparation

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

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

8 Procedure

8.1 Preparation of the test solution

Weigh to the nearest 0,001 g an amount of 30 g of the test sample and transfer it into a 1 000 ml volumetric flask (6.2). Add approximately 900 ml of water (5.1) and dissolve the sample while stirring until the solution is homogeneous. Fill up the flask with water (5.1) to 1 000 ml.

Filtrate a part of this solution in a small flask through a membrane filter (6.3) in order to remove eventually existing conditioning agents.

8.2 Preparation of the calibration solutions

Weigh (100 ± 0,1) mg DMPSA (5.4) and transfer into a 1 000 ml volumetric flask (6.2). Dissolve the sample with some water (5.1), homogenize well and fill up to 1 000 ml. Pipette 5 ml, 10 ml, 25 ml and 50 ml of this solution, each into a 100 ml volumetric flask (6.2) and fill up with water (5.1) to 100 ml. Use the solutions to determine the calibration curve.

8.3 Preparation of the eluents

Mix 900 ml of water (5.1) with 100 ml acetonitrile (5.2) and add 1 ml phosphoric acid (5.3) and micro filtrate (6.3).

8.4 HPLC conditions

— Column: e.g. C18, 7 µm, 250 mm × 4 mm;

— column temperature: room temperature;

— eluent: water/acetonitrile 90/10 with 1 ml phosphoric acid (see 8.3);

— flow rate: 1 ml/min;

— injection volume: 20 µl;

— wavelength: 232 nm;

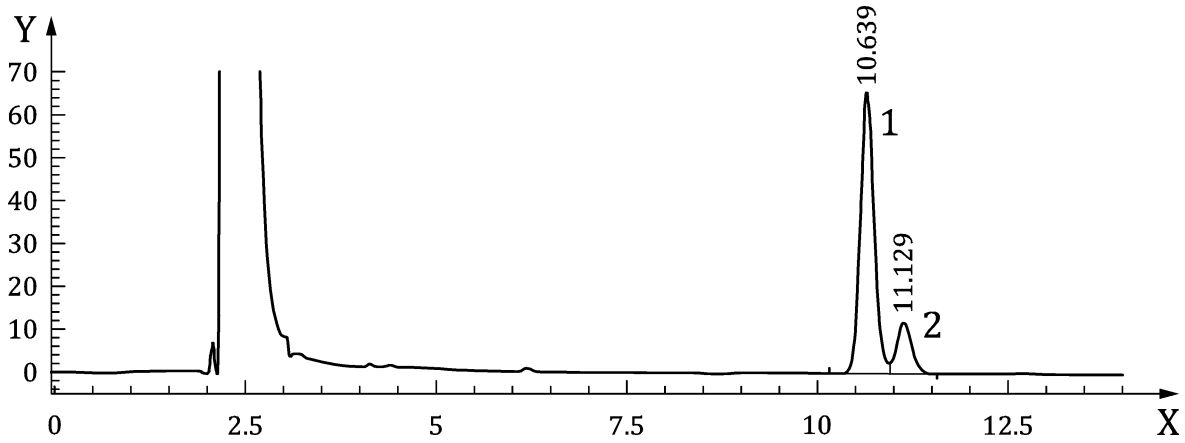
— retention time of DMPSA (at 1 ml/min flow rate): approximately 10 min to 12 min.

8.5 HPLC determination

To prepare a calibration curve, inject each calibration solution (8.2) two times consecutively. Calculate best-fit line and correlation coefficient r_{cc} by least-square method. The calibration curve can be used for assay when $r_{cc} \geq 0{,}99$.

Inject the test solution (8.1) two times consecutively.

DMPSA exists of two isomers of the 2-(dimethyl-pyrazol-1-yl)-succinic acid in a fixed ratio. Both isomers are determined by this method and the sum of the area of both chromatographic peaks shall be used for calculation of the results. Depending on the column used, the separation of both peaks might change (see Figure 1).



Key

X	time in minutes	1	chromatographic peak isomer 1
Y	absorption units	2	chromatographic peak isomer 2

Figure 1 — Chromatogram of DMPSA

9 Calculation and expression of the results

Carry out the evaluation on the basis of the calibration line over the peak areas. Calculate the content of 2-(3,4-dimethyl-pyrazol-1-yl)-succinic acid, w_{DMPSA}, in mg/kg according to Formula (1).

$$w_{DMPSA} = \frac{(A_{pk} - b) \times F_d \times 100}{a \times m} \quad (1)$$

where

A_{pk} is the peak area;

b is the y-axis section of straight calibration line;

a is the slope of straight calibration line;

F_d is the dilution factor;

m is the mass of the test portion, in milligrams.

Calculate the arithmetic mean from both values obtained. Report the result to the nearest 0,001 g/kg.

10 Precision

10.1 Inter-laboratory test

An inter-laboratory test was carried out in 2015 with eleven participating laboratories and three different samples of mineral fertilizers (see Table 1). Details and statistical results are summarized in Annex A.

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

10.2 Repeatability

The absolute difference between two independent single 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 repeatability limit *r* given in Table 1.

10.3 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 reproducibility limit *R* given in Table 1.

Table 1 — Mean values, repeatability and reproducibility limits

Sample	Mean value mg/kg	*r* mg/kg	*R* mg/kg
Sample 1: NPK 15+15+15	834	27,1	112,0
Sample 2: ASN	1772	98,5	171,8
Sample 3: Urea	4203	85,7	421,9

11 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the test method used with reference to this document, i.e. EN 17090;

c) the test results obtained;

d) date of the sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method which might have influenced the test result(s).