SLOVENSKI STANDARD
SIST EN 16328:2013

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Gnojila - Določevanje 3,4-dimetil-1H-pirazol fosfata (DMPP) - Metoda tekočinske kromatografije visoke ločljivosti (HPLC)

Fertilizers - Determination of 3,4-dimethyl-1H-pyrazole phosphate (DMPP) - Method using high-performance liquid chromatography (HPLC)

Düngemittel - Bestimmung von 3,4-dimethyl-1H-pyrazolphosphat (DMPP) - Verfahren mit Hochleistungs-Flüssigchromatographie (HPLC)

Engrais - Dosage de la 3,4-diméthyle-1H-pyrazole phosphate (DMPP) - Méthode par chromatographie liquide à haute performance (CLHP)

Ta slovenski standard je istoveten z: **EN 16328:2012**

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65.080	Gnojila	Fertilizers

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Fertilizers - Determination of 3,4-dimethyl-1H-pyrazole phosphate (DMPP) - Method using high-performance liquid chromatography (HPLC)

Engrais - Dosage du 3,4-diméthyl-1H-pyrazole phosphate (DMPP) - Méthode par chromatographie liquide à haute performance (HPLC)

Düngemittel - Bestimmung von 3,4-Dimethyl-1H-pyrazolphosphat (DMPP) - Verfahren mit Hochleistungs-Flüssigchromatographie (HPLC)

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Foreword

This document (EN 16328:2012) 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of an identical text or by endorsement, at the latest by June 2013, and conflicting national standards shall be withdrawn at the latest by June 2013.

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1 Scope

This European Standard specifies a method for the determination of 3,4-dimethyl-1H-pyrazole phosphate (DMPP, CAS-No: 202842-98-6) in mineral N containing fertilizers using high-performance liquid chromatography (HPLC).

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*

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

The fertilizer sample is dissolved in or extracted with water. The concentration of 3,4-dimethyl-1H-pyrazole phosphate is determined in diluted aqueous solution by high performance liquid chromatography (HPLC) at a reversed phase using a UV detector.

5 Reagents

WARNING — The described method includes the handling with hazardous materials. Attention must be paid to technical, organizational and personal safety measures.

Use only reagents of recognized analytical grade.

5.1 **Water**, de-ionized.

5.2 **Acetonitrile**, HPLC grade.

5.3 **Sodiumdihydrogenphosphate-monohydrate**, $NaH_2PO_4 \cdot H_2O$.

5.4 **3,4-dimethyl-1H-pyrazole**, > 99 % purity.

6 Apparatus

6.1 **Ultrasonic bath and magnetic stirrer**, respectively.

6.2 Filtration unit, manual or high-pressure filtration with membrane filter 0,45 µm or comparable filter.

NOTE High pressure filtration is advantageous for difficult samples.

6.3 HPLC unit, for isocratic operation with UV detector.

6.4 Evaluation unit.

6.5 Reverted phase-HPLC separation column, e.g. C18, 5 µm, 150 mm × 4,6 mm.

NOTE With satisfactory separation performance, a shorter separation column can be used.

6.6 Analytical balance, capable of weighing to an accuracy of 0,001 g.

7 Sampling

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

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

8 Procedure

8.1 Preparation of the test solution

Weigh (6.6) (30 ± 0,001) g of sample (m) into a 1 000 ml volumetric flask. Add approximately 900 ml water (5.1) and dissolve the sample in the ultrasonic bath (6.1) for about 15 min. Fill up the flask with water (5.1) to capacity. Filter part of the sample solution through a membrane filter (6.2) to remove possible conditioners. If necessary, dilute the sample for the calibration range.

8.2 Preparation of calibration solution

Prepare from at least two weighted samples (stock solutions) made with the present 3,4-dimethyl-1H-pyrazole (5.4) at least four concentrations for calibration. The concentration range should be between 3 mg/l and 100 mg/l.

8.3 HPLC conditions

— Separation column: filled for reversed phase-HPLC (6.5);

— column temperature: room temperature;

— elution agent: dissolve 1,38 g $NaH_2PO_4 \cdot H_2O$ (5.3) in 1 l water and add 175 ml acetonitrile (5.2);

— the elution agent shall be de-gassed, e.g. in an ultrasonic bath (6.1);

— flow rate: 1,5 ml/min;

— injection volume: 10 µl;

— wave length: 224 nm;

— retention time DMPP (at 1,5 ml/min flow rate): approximately 5,3 min.

8.4 HPLC determination

To prepare a calibration curve, inject 10 µl of each calibration solution three times consecutively. Calculate the compensation curve and correlation coefficient p by least-square method. The calibration curve can be used for concentration determination when $p \geq 0{,}99$.

Inject the test solution two times consecutively.

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 3,4-dimethyl-1H-pyrazole phosphate, w_{DMPP}, in mg/kg according to formula (1):

$$w_{DMPP} = \frac{(A_{pk} - b) \cdot F_d \cdot 100 \cdot 194{,}2}{a \cdot m \cdot 96{,}1} \times 10\,000 \quad (1)$$

where

A_{pk} is the peak height or 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 sample weight, in milligrams.

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

10 Precision

10.1 Inter-laboratory test

Details of inter laboratory tests performed in 2010 on the precision of the method are summarized in Annex A.

The values derived from this test may not be applicable to concentration ranges and matrices other than those given.

10.2 Repeatability

The absolute difference between two independent single test results, obtained using 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 be greater than the repeatability limit r given in Table 1.

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in no more than 5 % of the cases be greater than the reproducibility limit R given in Table 1.

Table 1 — Mean values, repeatability and reproducibility limits for DMPP

Sample	$\bar{x}$ mg/kg	r mg/kg	R mg/kg
Sample 1 Ammonium sulfate nitrate (ASN)	1 700	90	170
Sample 2 NPK 24+8+7	1 270	70	410
Sample 3 NPK 13+10+20	706	56	210

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) test method used with reference to this document;

c) test results obtained;

d) date of 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).