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DRAFT
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English Version

Organo-mineral fertilizers - Determination of the N-(n-butyl)thiophosphoric triamide (NBPT) urease inhibitor content

Engrais organo-minéraux - Détermination de la teneur en inhibiteur d'uréase N-(n-butyl) triamide thiophosphorique (NBPT)

Organisch-mineralische Düngemittel - Bestimmung des Gehaltes an Ureasehemmstoff

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European foreword

This document (prEN 18317:2026) 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 CEN Enquiry.

This document has been prepared under a standardization request addressed to CEN by the European Commission. The Standing Committee of the EFTA States subsequently approves these requests for its Member States.

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prEN 18317:2026 (E)

1 Scope

This document specifies a method for the determination of the urease inhibitor N-(n-butyl)thiophosphoric triamide (NBPT) and its oxidate form N-(n-butyl)phosphoric triamide (NBPTO) in urea based organo-mineral fertilizers, using the liquid chromatography coupled with triple quadrupole mass spectrometry (LC-MS/MS).

This document is applicable to organo-mineral fertilizers.

NOTE It is possible to apply this method to inorganic fertilizers; in this case a validation is carried out by the laboratory for the procedure and data generated.

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, liming materials and inhibitors — Sampling and sample preparation — Part 2: General sample preparation provisions*

prEN 12944-1, *Fertilizers, liming materials and inhibitors — Vocabulary — Part 1: General terms*

prEN 12944-2, *Fertilizers, liming materials and inhibitors — Vocabulary — Part 2: Terms relating to fertilizers*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in prEN 12944-1 and prEN 12944-2 and the following shall be applied.

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

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

4 Principle

This analytical method is based on the principles of liquid chromatography coupled with triple quadrupole mass spectrometry (LC-MS/MS) for determination of the separated inhibiting compounds NBPT and NBPTO.

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this document be carried out by suitably trained staff.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade and distilled water or ultrapure water for LC-MS.

5.2 Reagents for liquid chromatography

5.2.1 **Methanol**, LC-MS grade.

5.2.2 **Formic acid**, LC-MS grade.

5.2.3 **Ammonium formate**, p.a. high purity.

5.2.4 **Water**, ultrapure LC-MS grade or distilled water.

5.2.5 **N-(n-butyl)thiophosphoric triamide (NBPT)**, CAS n. 94317-64-3, minimum mass fraction of 98 %.

5.2.6 **N-(n-butyl)phosphoric triamide (NBPTO)**, CAS n. 25316-39-6, minimum mass fraction of 80 %.

5.2.7 **Urea**, p.a. high purity.

5.3 Calibration standards

5.3.1 **Stock solution**, mass concentration $\rho_{\text{NBPT}} = 0,20$ mg/ml.

Weigh 50 mg NBPT (5.2.5) into a 250 ml volumetric glass flask and dissolve to volume with water (5.2.4). Store at $+4$ °C \pm 1 °C for no more than 2 days.

5.3.2 **Stock solution**, $\rho_{\text{NBPTO}} = 0,20$ mg/ml.

Weigh 50 mg NBPTO (5.2.6) into a 250 ml volumetric glass flask and dissolve to volume with water (5.2.4). Store at $+4$ °C \pm 1 °C for no more than 2 days.

5.3.3 **Blank solution**, water (5.2.4).

6 Equipment and consumables

Disposable equipment is acceptable in the same way as reusable glassware if the specifications are similar. Ordinary laboratory equipment, and particularly the following shall be used.

6.1 **Analytical scale**, capable of weighing to the nearest 0,000 1 g.

6.2 **Graduated pipettes**, for volumes 5 ml, 10 ml, 15 ml, 20 ml, 25 ml with an accuracy of 0,01 ml.

6.3 **One-mark volumetric glass flasks Class A** [3], capacity 100 ml and 250 ml.

6.4 **Horizontal shaker agitator**.

6.5 **Qualitative filter paper**.

6.6 **LC-MSQQQ**.

6.6.1 **Operative conditions**