# SLOVENSKI STANDARD
# kSIST-TS FprCEN/TS 17771:2021

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**Organska in organsko-mineralna gnojila - Določevanje dušika**

Organic and organo-mineral fertilizers - Determination of the nitrogen content

Organische und organisch-mineralische Düngemittel - Bestimmung des Stickstoffgehaltes

Engrais organiques et organo-minéraux - Détermination de la teneur en azote



**Ta slovenski standard je istoveten z:** **FprCEN/TS 17771**



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65.080 Gnojila Fertilizers

**kSIST-TS FprCEN/TS 17771:2021** **en,fr,de**

TECHNICAL SPECIFICATION

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# FINAL DRAFT FprCEN/TS 17771

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English Version

# Organic and organo-mineral fertilizers - Determination of the nitrogen content

Organische und organisch-mineralische Düngemittel - Bestimmung des Stickstoffgehaltes

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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## European foreword

This document (FprCEN/TS 17771:2021) 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 Vote on TSnt.

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

# Introduction

Regulation (EU) 2019/1009 [5] lays down the rules on the making available on the market of EU fertilizing products and the specific safety and quality requirements for the defined product function categories (PFCs). Organic and organo-mineral fertilizers have been classified as PFC 1 (A) and PFC 1 (B).

This document defines test methods for the determination of the nitrogen content to be used for organic and organo-mineral fertilizers in order to measure the compliance with the related requirement in the Regulation (EU) 2019/1009 [5].

# 1 Scope

This document is applicable to fertilizing products, which are classified as PFC 1(A) or PFC 1(B) of Regulation (EU) 2019/1009 [5]. However, the present method was not validated for blends.

This document specifies a method for the determination of the total nitrogen content and the content of ammoniacal, nitric, ureic and organic nitrogen in organic and organo-mineral fertilizers. This method is based on EN 15604:2009 and adapted to be applicable to organic and organo-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 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*

EN 15604:2009, *Fertilizers - Determination of different forms of nitrogen in the same sample, containing nitrogen as nitric, ammoniacal, urea and cyanamide nitrogen*

# 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 and the following apply.

ISO and IEC maintain terminological 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

The fertilizer sample shall be analyzed according to four different analytical pathways to quantify the different forms of nitrogen in the sample. The nitrogen content determined by the four analytical pathways is representative for:

1) Total nitrogen content;

2) Total nitrogen content with exception of nitric nitrogen;

3) Ammoniacal nitrogen content;

4) Ammoniacal nitrogen and ureic nitrogen content.

The analysis results that are obtained by the determination of nitrogen according to these four pathways shall be used to calculate the content of ammoniacal, nitric, ureic, and organic nitrogen in the sample (see 9.1).

All four analytical pathways are based on or derived from the Kjeldahl principle. First, the operator shall perform a pretreatment and/or digestion to convert the nitrogen of a certain fraction of nitrogenous compounds to ammonia. Next, the operator shall distil the ammoniacal nitrogen into a known volume and concentration of hydrochloric acid. Finally, the operator shall quantify the ammoniacal nitrogen by titration of excess amount of acid in the receiving flask with sodium hydroxide.

The procedures that shall be performed for the pretreatment, digestion and distillation depend on the analytical pathway, as schematically shown in Figure 1.

When performing analytical pathway 1, the total nitrogen content shall be determined by a reduction with the aid of reduced iron and stannous chloride to convert nitric nitrogen to ammonia, followed by Kjeldahl digestion of the sample. Subsequently, the ammonia shall be distilled after addition of sodium hydroxide.

NOTE For the determination of the total nitrogen content, the Dumas method can also be applied if it is proven to be as accurate as the modified Kjeldahl method.

When performing analytical pathway 2, the total nitrogen with the exception of nitric nitrogen shall be determined by a Kjeldahl digestion with ferrous sulfate. Then, the ammonia shall be distilled after addition of sodium hydroxide.

When performing analytical pathway 3, the ammoniacal nitrogen content shall be determined by a mild distillation after addition of magnesium oxide.

Performing pathway 4, the ureic and ammoniacal nitrogen content shall be determined by a pretreatment with urease. Subsequently, the ammonia shall be distilled by a mild distillation after addition of magnesium oxide.

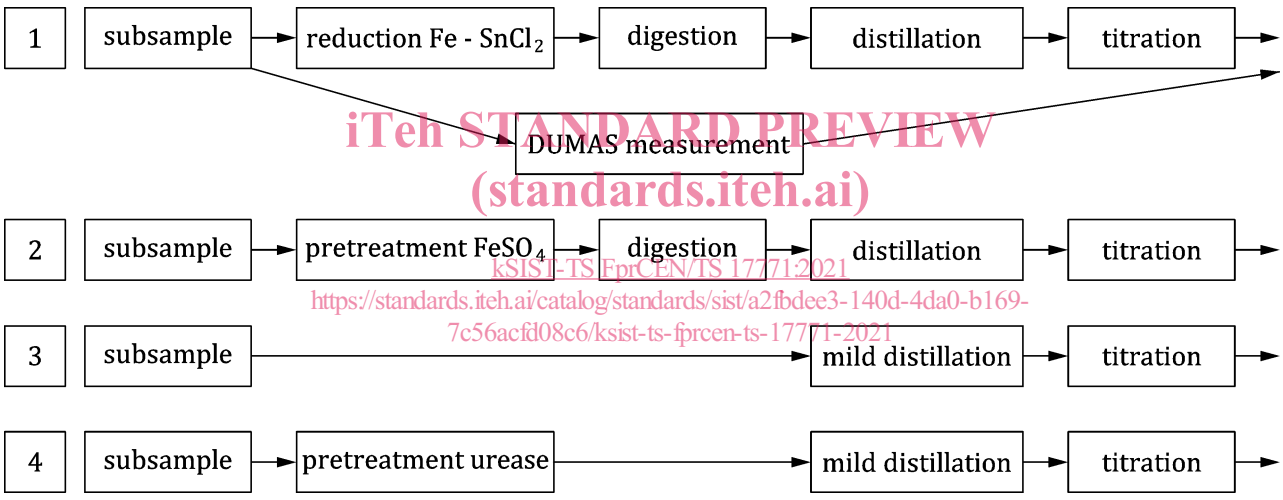




**Figure 1 — The four analytical pathways for the determination of the different nitrogen fractions**

## 5 Reagents

All water used should be of grade 3 according to EN ISO 3696:1995. All reagents should be of recognized analytical grade.

The following reagents shall be used.

### 5.1 Reduced iron

Iron powder reduced by hydrogen.

### 5.2 Standard iodine solution ($I_2$, substance concentration $c$ = 0,05 mol/l), certified.

### 5.3 Stannous chloride solution ($SnCl_2$, $c$ = 0,532 mol/l)

For the preparation of the stannous chloride solution, the following tasks shall be performed:

- Weigh 120 g of $SnCl_2$ • 2 $H_2O$ and transfer it into a 1 l volumetric flask;

- Add 400 ml concentrated hydrochloric acid (density at 20 °C $\rho_{20}$ = 1,18 g/ml) to the flask;
- Bring to volume with water and shake the flask until all $SnCl_2$ is dissolved.

The solution shall be clear and prepared immediately before use.

It is essential to check the reducing power of stannous chloride dihydrate. In order to check the reducing power, the following steps shall be performed:

- Weigh 0,5 g of $SnCl_2$ • 2 $H_2O$ and transfer it into a 50 ml volumetric flask,
- Add 2 ml concentrated hydrochloric acid to the flask,
- Bring to volume with water and shake the flask until all $SnCl_2$ is dissolved,
- Transfer the content of the volumetric flask into a glass beaker,
- Weigh 5 g of Rochelle salt (potassium sodium tartrate) and transfer into the glass beaker,
- Add sufficient quantity of sodium bicarbonate for the solution to be alkaline to litmus paper.

Titrate this solution with an iodine solution ($I_2$) (5.2) in the presence of a starch solution as an indicator. 1 ml of the iodine solution corresponds to 0,011 28 g of $SnCl_2$ • 2 $H_2O$. At least 80 % of the total tin present in the solution thus prepared shall be in bivalent form.

Therefore, at least 35 ml of the iodine solution should be required for the titration.



### 5.4 Defoaming agent

Octyl alcohol shall be used as defoaming agent, or any other non-nitrogen containing agent.



### 5.5 Ferrous sulfate

Crystalline ferrous sulfate heptahydrate ($FeSO_4 \cdot 7\ H_2O$).

### 5.6 Sulfuric acid ($c$ = 18 mol/l)

Concentrated sulfuric acid ($H_2SO_4$), $\rho_{20}$ = 1,84 g/ml.

### 5.7 Hydrochloric acid solution ($c$ = 6 mol/l)

For the preparation of this hydrochloric acid solution gradually add one volume of concentrated hydrochloric acid ($\rho_{20}$ = 1,18 g/ml) to one volume of water.

### 5.8 Hydrochloric acid solution ($c$ = 0,1 mol/l)

For the preparation of this hydrochloric acid solution of, the following tasks shall be performed:

- Fill a 1 l volumetric flask about three-quarters with water,
- Add 8,5 ml of concentrated hydrochloric acid),
- Bring to a volume of 1 l.

### 5.9 Sodium hydroxide solution ($c$ = 7,5 mol/l)

For the preparation of the sodium hydroxide solution of about 30 % (mass concentration), the following tasks shall be performed:

- Fill a 1 l flask about three-quarters with water,

- Weigh 300 g sodium hydroxide and gradually add it to the flask,
- Stir the solution until all sodium hydroxide is dissolved,
- Bring to a volume of 1 l.

### 5.10 Sodium hydroxide solution (*c* =0,1 mol/l)

Certified standard solution of sodium hydroxide 0,1 mol/l.

### 5.11 Potassium hydrogen phthalate buffer (*c* = 0,1 mol/l)

A potassium hydrogen phthalate buffer set to pH 5,5. For the preparation of this solution the following steps shall be performed:

- Weigh 20,42 g of potassium hydrogen phthalate and transfer into a 1 l volumetric flask,
- Weigh 2,9 g of sodium hydroxide and transfer into the volumetric flask,
- Bring to volume with water and shake the flask until all potassium hydrogen phthalate and sodium hydroxide are dissolved,
- Adjust the pH to 5,5 by adding hydrochloric acid solution (5.8) or sodium hydroxide solution (5.10).

### 5.12 Urease solution



For the preparation of the urease solution the following tasks shall be performed:

- Suspend 5 g of active urease, with an activity of at least 5 U/mg in 1 l of potassium hydrogen phthalate buffer (5.11).



- Adjust the pH to 5,5 by adding hydrochloric acid (5.8) or sodium hydroxide solution (5.10).

### 5.13 Catalyst

A mixture of: (a) 10 g of potassium sulfate ($K_2SO_4$) and 0,3 g of copper oxide (CuO) or (b) 10 g of potassium sulfate and 1 g of copper sulfate pentahydrate ($CuSO_4 . 5 H_2O$) shall be used as catalyst for the digestion, possibly in the form of tablets containing similar amount of the chemicals mentioned.

### 5.14 Boiling chips.

### 5.15 Indicator solutions

The following tasks shall be performed to prepare the indicator solutions:

#### 5.15.1 Indicator solution I

- Weigh 1 g of methyl red and transfer it into a 1 l volumetric flask;
- Add 0,5 ml of sodium hydroxide solution 7,5 mol/l (5.9);
- Bring to volume with water and shake the flask until all methyl red is dissolved.

#### 5.15.2 Indicator solution II

- Weigh 1 g of methylene blue and transfer into a 1 l volumetric flask,