



**SLOVENSKI STANDARD**  
**oSIST prEN 15561:2021**

**01-december-2021**

---

**Gnojila - Določevanje celotnega dušika v kalcijevem cianoamidu, ki vsebuje nitrates**

Fertilizers - Determination of total nitrogen in calcium cyanamide containing nitrates

Düngemittel - Bestimmung des Gesamtstickstoffs in nitrathaltigem Kalkstickstoff

Engrais - Détermination de l'azote total dans la cyanamide calcique nitraté

**Ta slovenski standard je istoveten z: prEN 15561**

[oSIST prEN 15561:2021](https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-3c2b606d461a/osist-pren-15561-2021)

<https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-3c2b606d461a/osist-pren-15561-2021>

**ICS:**

65.080

Gnojila

Fertilizers

**oSIST prEN 15561:2021**

**en,fr,de**

**iTeh STANDARD PREVIEW**  
**(standards.iteh.ai)**

[oSIST prEN 15561:2021](#)

<https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-3c2b606d461a/osist-pren-15561-2021>

EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**DRAFT**  
**prEN 15561**

November 2021

ICS 65.080

Will supersede EN 15561:2009

English Version

## Fertilizers - Determination of total nitrogen in calcium cyanamide containing nitrates

Engrais - Détermination de l'azote total dans la cyanamide calcique nitraté

Düngemittel - Bestimmung des Gesamtstickstoffs in nitrathaltigem Kalkstickstoff

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

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

[https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-](https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-3c2b6061461a/osist-pr-en-15561-2021)

Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

**Warning** : This document is not a European Standard. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a European Standard.



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

**CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels**

<b>Contents</b>	<b>Page</b>
European foreword .....	3
<b>1</b> Scope.....	4
<b>2</b> Normative references.....	4
<b>3</b> Terms and definitions.....	4
<b>4</b> Principle.....	4
<b>5</b> Reagents.....	4
<b>6</b> Apparatus and equipment.....	5
Figure 1 — Distillation apparatus 1 .....	6
Figure 2 — Distillation apparatus 2 .....	7
Figure 3 — Distillation apparatus 3 .....	8
Figure 4 — Distillation apparatus 4 .....	9
<b>7</b> Sampling and sample preparation .....	10
<b>8</b> Procedure .....	10
<b>8.1</b> Preparation of the solution .....	10
<b>8.2</b> Analysis of the solution.....	10
<b>Table 1</b> — Weighing, dilution and calculation variants a, b and c.....	11
<b>8.3</b> Blank.....	11
<b>8.4</b> Control test.....	11
<b>9</b> Calculation and expression of the result.....	11
<b>10</b> Test report.....	12
<b>Bibliography</b> .....	13

## European foreword

This document (prEN 15561:2021) 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 CEN Enquiry.

This document will supersede EN 15561:2009.

In comparison with the previous edition, the following technical modifications have been made:

- technical change to the preparation of the solution, i.e. deletion of the last paragraph of 8.1;
- deletion of former tables and addition of a technically revised Table (8.2).

## iTeh STANDARD PREVIEW (standards.iteh.ai)

[oSIST prEN 15561:2021](https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-3c2b606d461a/osist-pren-15561-2021)

<https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-3c2b606d461a/osist-pren-15561-2021>

**prEN 15561:2021 (E)****1 Scope**

This document specifies a method for the determination of total nitrogen in calcium cyanamide.

The method is applicable to calcium cyanamide containing nitrates.

**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*

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

**4 Principle**

<https://standards.iteh.ai/catalog/standards/sist/db71ee7b-7297-464d-99b8-3c2b606d461a/osist-pren-15561-2021>

Reduction of nitrate nitrogen to ammonia with metallic iron and stannous chloride solution. Digestion in sulfuric acid. Distillation of the ammonia from an alkaline solution, absorption in an excess of standard sulfuric acid solution and back-titration with standard sodium or potassium hydroxide solution.

**5 Reagents**

Use only reagents of recognized analytical grade and distilled or demineralized water, free from carbon dioxide and all nitrogenous compounds (grade 3 according to EN ISO 3696:1995).

- 5.1 Sulfuric acid**, mass concentration  $\rho = 1,84$  g/ml.
- 5.2 Powdered iron**, reduced in hydrogen.
- 5.3 Potassium sulfate**, analytical grade, finely pulverized.
- 5.4 Sulfuric acid** (for variant a), substance concentration  $c = 0,05$  mol/l.
- 5.5 Sodium or potassium hydroxide standard solution** (for variant a), carbonate free,  $c = 0,1$  mol/l.
- 5.6 Sulfuric acid** (for variant b),  $c = 0,1$  mol/l.
- 5.7 Sodium or potassium hydroxide standard solution** (for variant b), carbonate free,  $c = 0,2$  mol/l.
- 5.8 Sulfuric acid** (for variant c),  $c = 0,25$  mol/l.

**5.9 Sodium or potassium hydroxide standard solution** (for variant c), carbonate free,  $c = 0,5 \text{ mol/l}$ .

**5.10 Indicator solutions.**

**5.10.1 Mixed indicator.**

Solution A: Dissolve 1 g of methyl red in 37 ml of sodium hydroxide solution  $c = 0,1 \text{ mol/l}$  (5.6) and make up to 1 l with water.

Solution B: Dissolve 1 g of methylene blue in water and make up to 1 l.

Mix one volume of solution A with two volumes of solution B.

This indicator is violet in acid solution, grey in neutral solution and green in alkaline solution. Use 0,5 ml (10 drops) of this indicator solution.

**5.10.2 Methyl red indicator solution**

Dissolve 0,1 g of methyl red in 50 ml of 95 % ethanol. Make up to 100 ml with water and filter if necessary. This indicator may be used (4 to 5 drops) instead of that specified in 5.10.1.

**5.11 Stannous chloride solution**

Dissolve 120 g of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  in 400 ml of concentrated hydrochloric acid (density at  $20^\circ\text{C}$   $\rho_{20} = 1,18 \text{ g/ml}$ ) and make up to 1 l with water. The solution shall be completely clear and prepared immediately before use.

It is essential to check the reducing power of the stannous chloride: dissolve 0,5 g of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  in 2 ml of concentrated hydrochloric acid ( $\rho_{20} = 1,18 \text{ g/ml}$ ) and make up to 50 ml with water. Then add 5 g of Rochelle salt (potassium sodium tartrate), then a sufficient quantity of sodium bicarbonate for the solution to be alkaline to litmus paper.

Titrate with an iodine solution ( $\text{I}_2$ ) of  $c = 0,05 \text{ mol/l}$  in the presence of a starch solution as an indicator.

1 ml of iodine solution ( $\text{I}_2$ ) of  $c = 0,05 \text{ mol/l}$  corresponds to 0,011 28 g of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ .

At least 80 % of the total tin present in the solution thus prepared shall be in bivalent form. For the titration at least 35 ml of the  $c = 0,1 \text{ mol/l}$  iodine solution ( $\text{I}_2$ ) shall be used.

**5.12 Sodium hydroxide solution**, containing about 30 % NaOH ( $\rho = 1,33 \text{ g/ml}$ ), ammonia free.

**5.13 Standard nitrate-ammoniacal solution.**

Weigh 2,5 g of potassium nitrate and 10,16 g of ammonium sulfate and place them in a 250 ml graduated flask (6.4). Dissolve in water and make up to 250 ml. One ml of this solution contains 0,01 g of nitrogen.

**5.14 Anti-bump granules of pumice stone**, washed in hydrochloric acid and calcined.

## 6 Apparatus and equipment

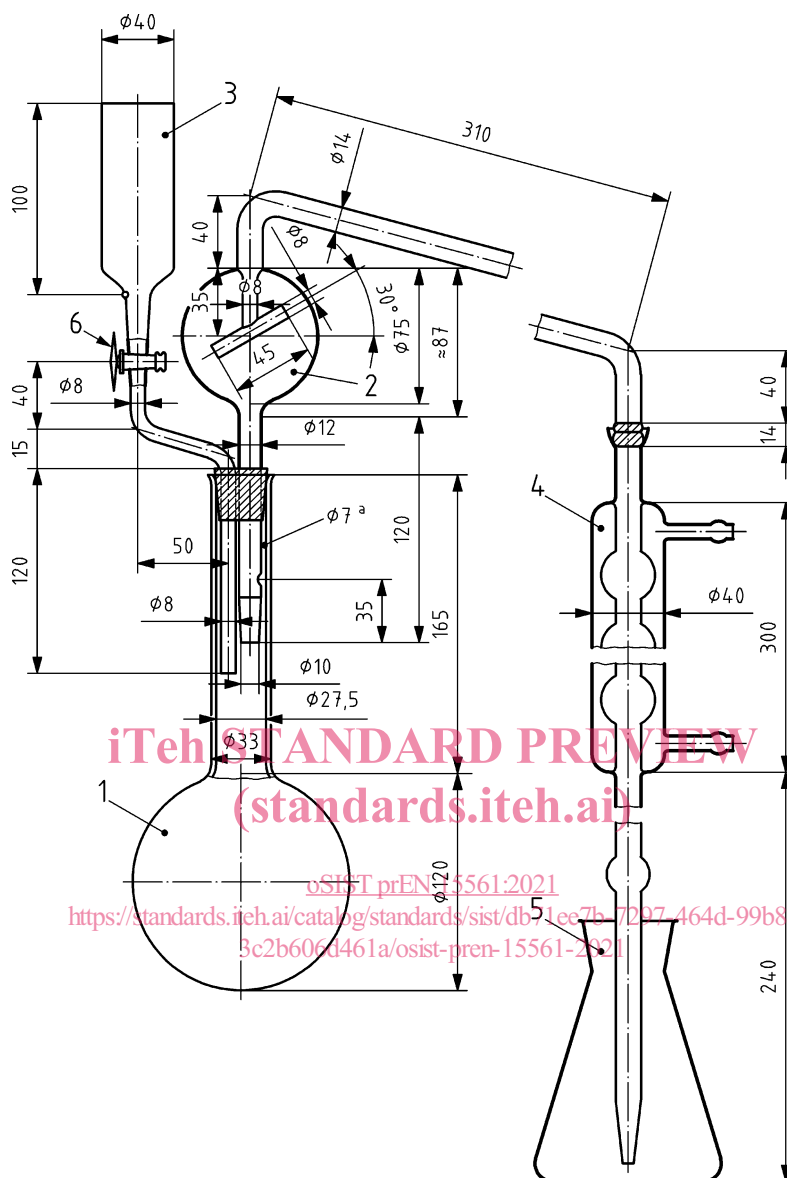
Usual laboratory glassware and equipment and, in particular, the following.

**6.1 Distillation apparatus**, consisting of a round-bottomed flask of suitable capacity connected to a condenser by means of a splash head. The equipment is made of borosilicate glass.

NOTE The different types of equipment recommended for this determination are reproduced, showing all the features of construction, in Figures 1, 2, 3, and 4.

Automatic distillation apparatus may be used also, provided that the results are statistically equivalent.

Dimensions in millimetres

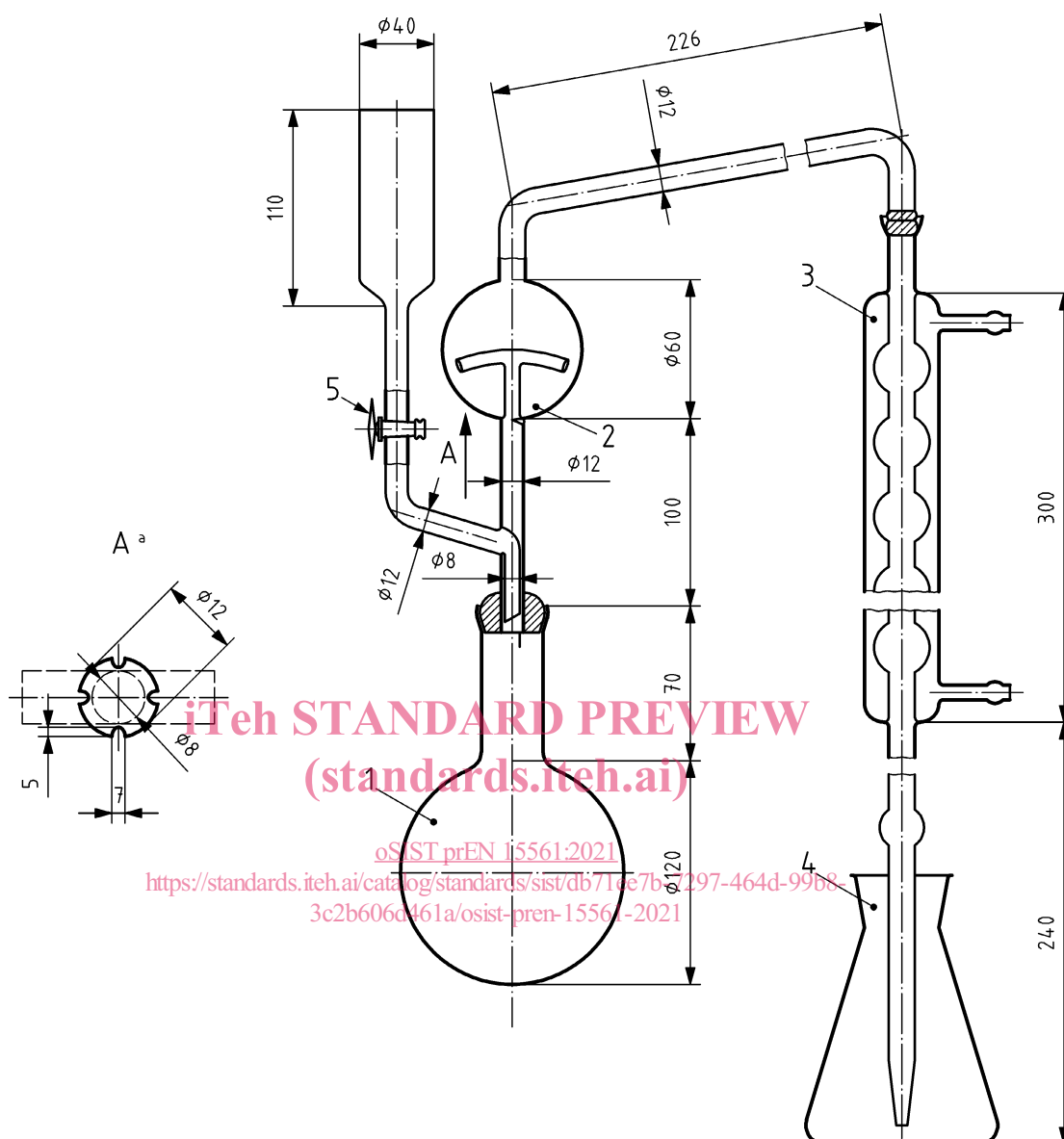
**Key**

- 1 round-bottomed, long-necked flask of 1 000 ml capacity
- 2 distillation tube with a splash head, connected to the condenser by means of a spherical joint (No 18) (the spherical joint for the connection to the condenser may be replaced by an appropriate rubber connection)
- 3 funnel with a polytetrafluoroethylene (PTFE) tap (6) for the addition of sodium hydroxide (the tap may likewise be replaced by a rubber connection with a clip)
- 4 six-bulb condenser with spherical joint (No 18) at the entrance, and joined at the issue to a glass extension tube by means of a small rubber connection (when the connection to the distillation tube is effected by means of a rubber tube, the spherical joint may be replaced by a suitable rubber bung)
- 5 500 ml flask in which the distillate is collected
- 6 PTFE-tap
- <sup>a</sup> hole

**Figure 1 — Distillation apparatus 1**



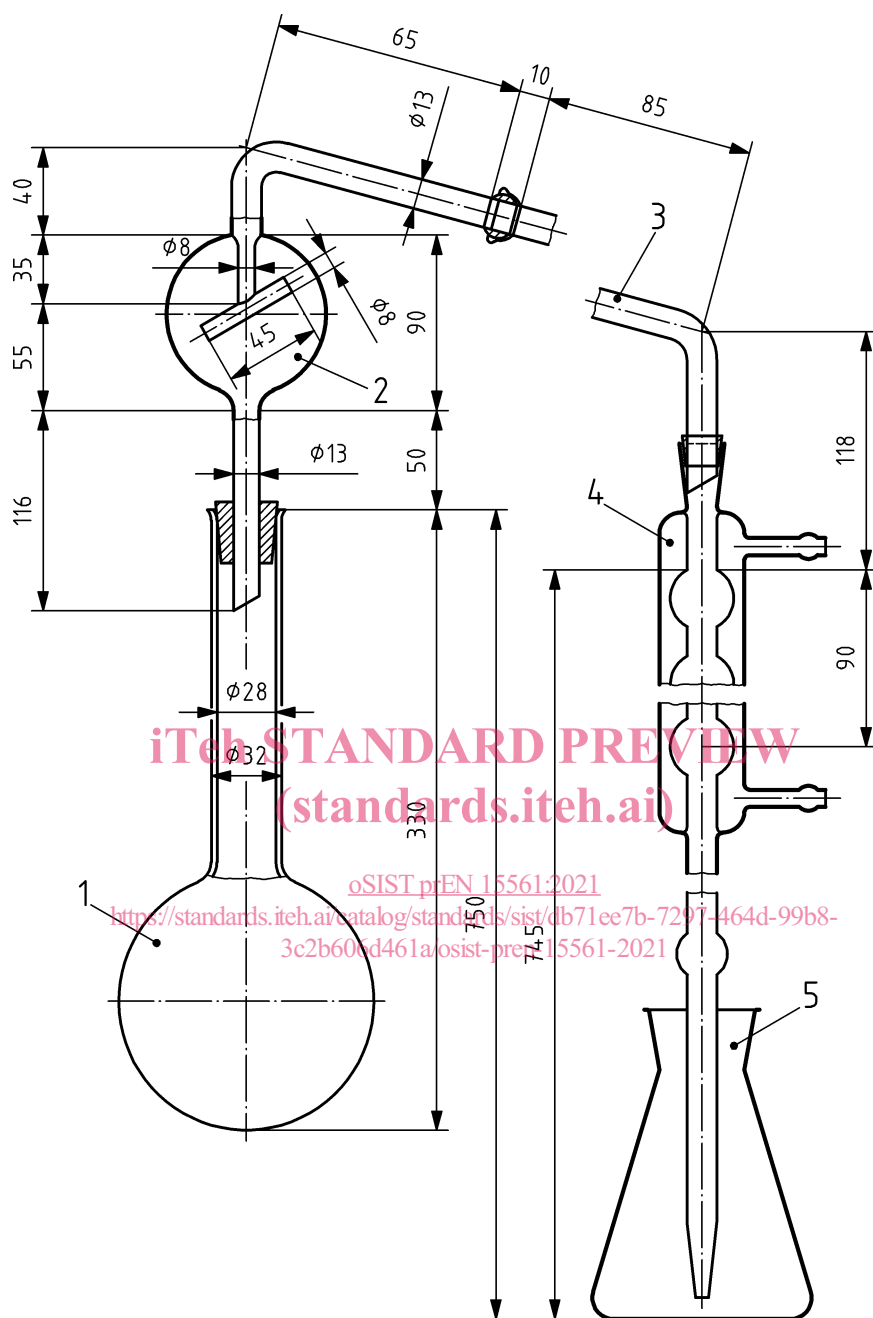
Dimensions in millimetres

**Key**

- 1 round-bottomed, short-necked flask of 1 000 ml capacity with a spherical joint (No 35)
  - 2 distillation tube with a splash head, equipped with a spherical joint (No 35) at the entrance and a spherical joint (No 18) at the issue, connected at the side to a funnel with a polytetrafluoroethylene (PTFE) tap (5) for the addition of sodium hydroxide
  - 3 six-bulb condenser with a spherical joint (No 18) at the entrance and joined at the issue to a glass extension tube by means of a small rubber connection
  - 4 500 ml flask in which the distillate is collected
  - 5 PTFE-tap
- <sup>a</sup> enlarged description

**Figure 2 — Distillation apparatus 2**

Dimensions in millimetres

**Key**

- 1 round-bottomed, long-necked flask of 750 ml or 1 000 ml capacity with a bell mouth
- 2 distillation tube with a splash head and a spherical joint (No 18) at the issue
- 3 elbow tube with a spherical joint (No 18) at the entrance and a drip cone (the connection to the distillation tube may be effected by means of a rubber tube instead of a spherical joint)
- 4 six-bulb condenser joined at the issue to a glass extension tube by means of a small rubber connection
- 5 500 ml flask in which the distillate is collected

**Figure 3 — Distillation apparatus 3**