



SLOVENSKI STANDARD
oSIST prEN 15560:2021

01-december-2021

Gnojila - Določevanje celotnega dušika v kalcijevem cianoamidu, ki ne vsebuje nitratov

Fertilizers - Determination of total nitrogen in calcium cyanamide nitrate free

Düngemittel - Bestimmung des Gesamtstickstoffs in nitratfreiem Kalkstickstoff

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

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ICS:

65.080

Gnojila

Fertilizers

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en,fr,de

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

DRAFT
prEN 15560

November 2021

ICS 65.080

Will supersede EN 15560:2009

English Version

Fertilizers - Determination of total nitrogen in calcium cyanamide nitrate free

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

Düngemittel - Bestimmung des Gesamtstickstoffs in nitratfreiem 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.

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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.

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

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European foreword

This document (prEN 15560:2021) 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 15560:2009.

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

- deletion of former tables and addition of a technically revised Table (8.2);
- rewording of the control test (8.4).

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1 Scope

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

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

Digestion in sulfuric acid with the aid of a catalyst. 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 Diluted sulfuric acid, mix one volume of sulfuric acid, mass concentration $\rho = 1,84$ g/ml, with one volume of water.

5.2 Potassium sulphate, analytical grade.

5.3 Catalyst

Use 0,3 g to 0,4 g of copper(II)oxide or 0,95 g to 1,25 g of copper(II)sulfate pentahydrate for each determination.

5.4 Sodium hydroxide solution, 30 % mass concentration, of approximately $\rho(\text{NaOH}) = 1,33 \text{ g/ml}$, ammonia free.

5.5 Sulfuric acid, substance concentration $c = 0,05 \text{ mol/l}$.

5.6 Sodium or potassium hydroxide solution, carbonate free, $c = 0,1 \text{ mol/l}$.

5.7 Sulfuric acid, $c = 0,1 \text{ mol/l}$.

5.8 Sodium or potassium hydroxide solution, carbonate free, $c = 0,2 \text{ mol/l}$.

5.9 Sulfuric acid, $c = 0,25 \text{ mol/l}$.

5.10 Sodium or potassium hydroxide solution, carbonate free, $c = 0,5 \text{ mol/l}$.

5.11 Indicator solutions

5.11.1 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.

5.11.2 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 5.11.1.

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

5.13 Potassium thiocyanate, analytical grade.

6 Apparatus

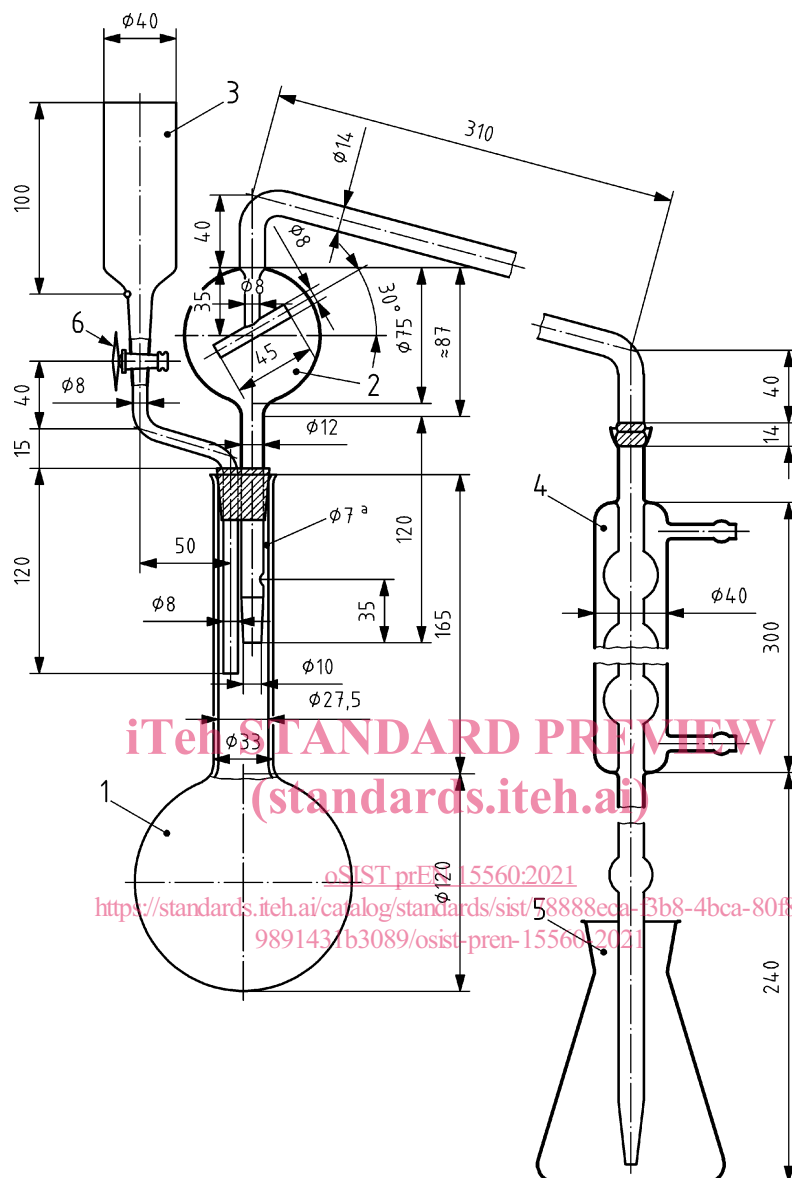
Usual laboratory glassware and equipment and, in particular, the following.

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.

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

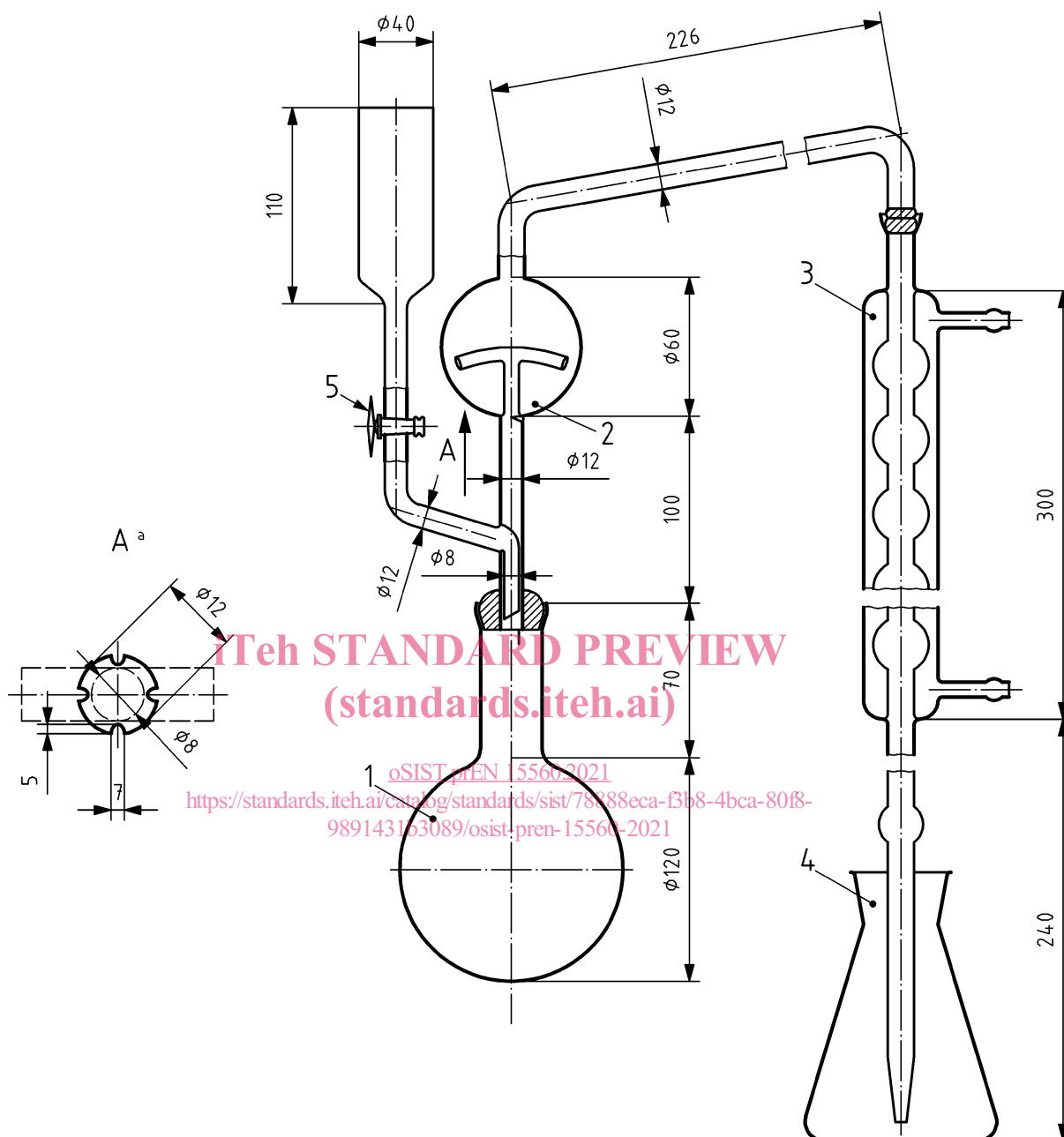
Dimensions in millimetres

**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
- ^a hole

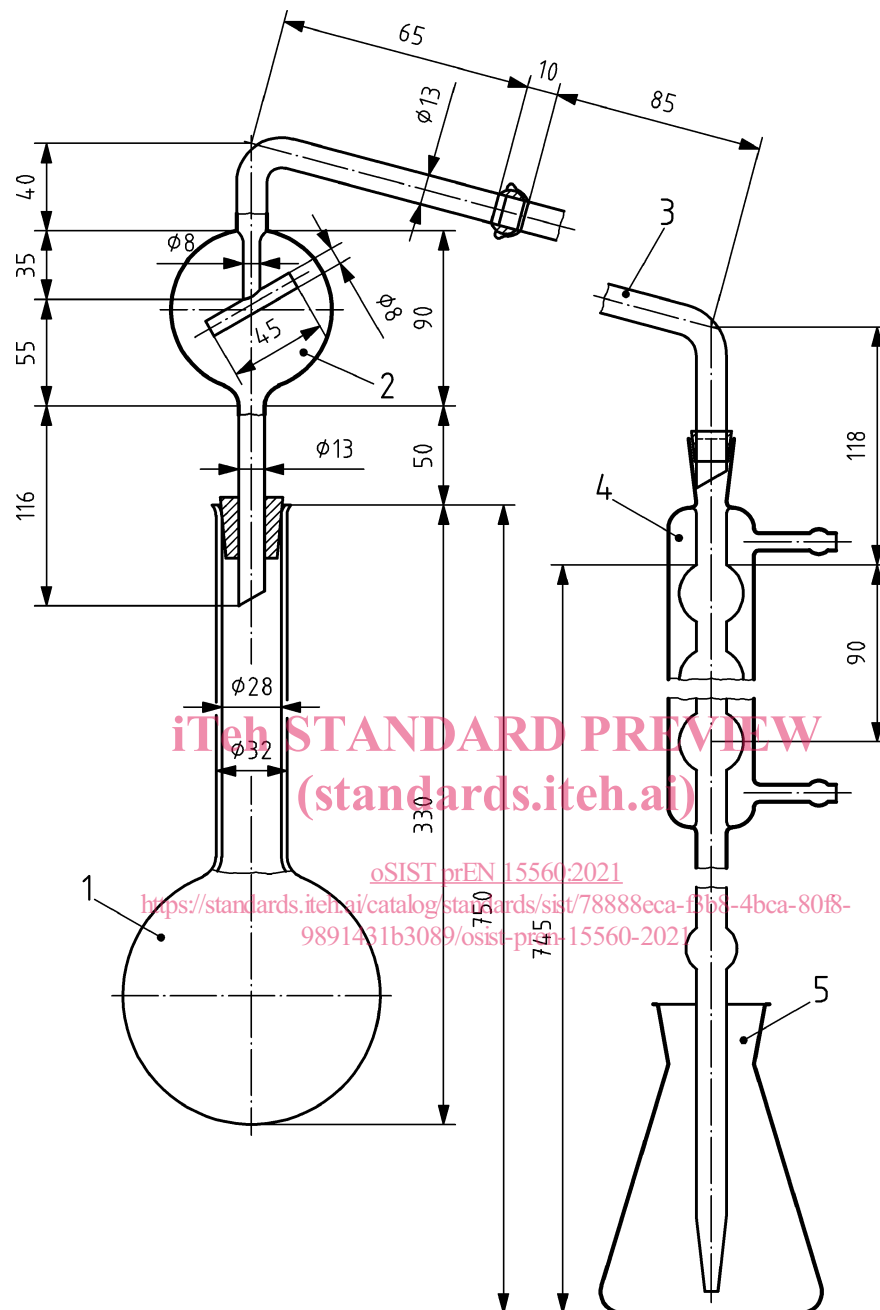
Figure 1 — Distillation apparatus 1

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
- ^a enlarged description

Figure 2 — Distillation apparatus 2

**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