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SIST EN 15560:2009

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EUROPEAN STANDARD

EN 15560

NORME EUROPÉENNE

EUROPÄISCHE NORM

January 2009

ICS 65.080

Supersedes CEN/TS 15560:2007

English Version

Fertilizers - Determination of total nitrogen in calcium cyanamide nitrate free

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

Düngemittel - Bestimmung von Gesamtstickstoff in nitratfreiem Kalkstickstoff

This European Standard was approved by CEN on 6 December 2008.

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

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Foreword

This document (EN 15560:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 15560:2007.

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

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EN 15560:2009 (E)**1 Scope**

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

2 Normative references

The following referenced documents are indispensable for the application 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 12944-1:1999, *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 ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

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For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

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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**5.1 General**

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).

5.2 Diluted sulfuric acid, mix one volume of sulfuric acid, $\rho = 1,84$ g/ml, with one volume of water.

5.3 Potassium sulphate, p.a.

5.4 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.

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

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

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

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

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

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

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

5.12 Indicator solutions

5.12.1 Mixed indicator

Solution A: Dissolve 1 g of methyl red in 37 ml of sodium hydroxide solution $c = 0,1 \text{ mol/l}$ 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.

5.12.2 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 5.12.1.

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

5.14 Potassium thiocyanate, p.a.

6 Apparatus

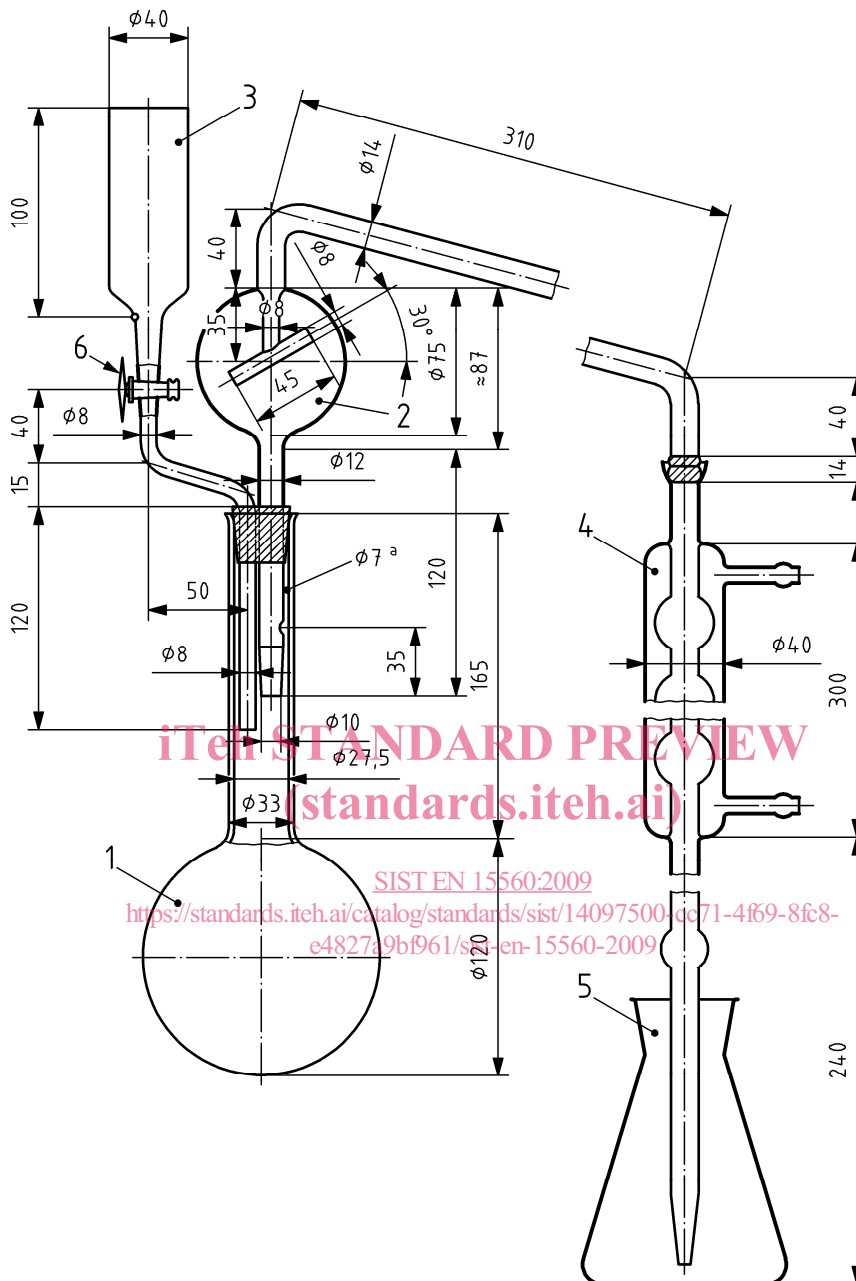
6.1 Distillation apparatus

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

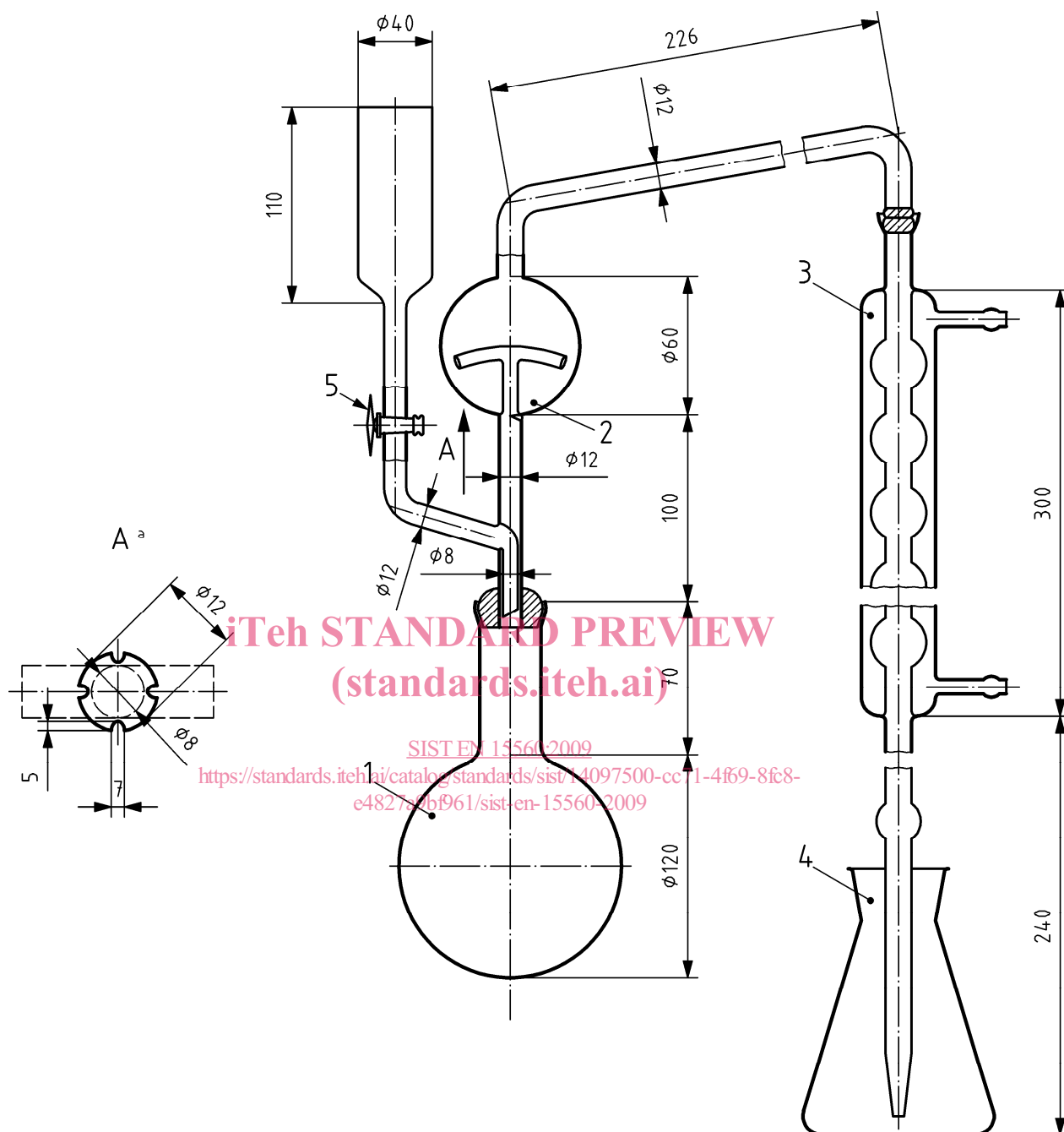


Key

- 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