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SIST EN 15562:2009

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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 15562

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Supersedes CEN/TS 15562:2007

English Version

Fertilizers - Determination of cyanamide nitrogen

Engrais - Détermination de l'azote cyanamidé

Düngemittel - Bestimmung von Cyanamidstickstoff

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

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Foreword

This document (EN 15562:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.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 15562:2007.

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EN 15562:2009 (E)

1 Scope

This European Standard specifies a method for the determination of cyanamide nitrogen in fertilizers. The method is applicable to calcium cyanamide and calcium cyanamide/nitrate mixtures.

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

Precipitation of cyanamide nitrogen as a silver complex. 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 Glacial acetic acid

5.3 Ammonia solution, containing 10 % of ammonia gas by mass ($\rho_{20} = 0,96$ g/ml).

5.4 Ammoniacal silver solution, according to Tollens.

Mix 500 ml of 10 % silver nitrate (AgNO_3) solution in water with 500 ml of 10 % ammonia solution (5.3).

Do not expose unnecessarily to light, heat or air. The solution normally keeps for years. As long as the solution remains clear, the reagent is of good quality.

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

5.6 Potassium sulfate, p.a.

5.7 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.

5.8 Sodium hydroxide solution, approximately 30 % NaOH ($\rho_{20} = 1,33$ g/ml), ammonia free.

5.9 Sulfuric acid, $c = 0,05$ mol/l.

5.10 Sodium or potassium hydroxide solution, $c = 0,1$ 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 $c = 0,1$ 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.

5.11.2 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 (i.e. pumice stone, glass pearls), washed in hydrochloric acid and calcined.

5.13 Potassium thiocyanate, for the control test.

6 Apparatus

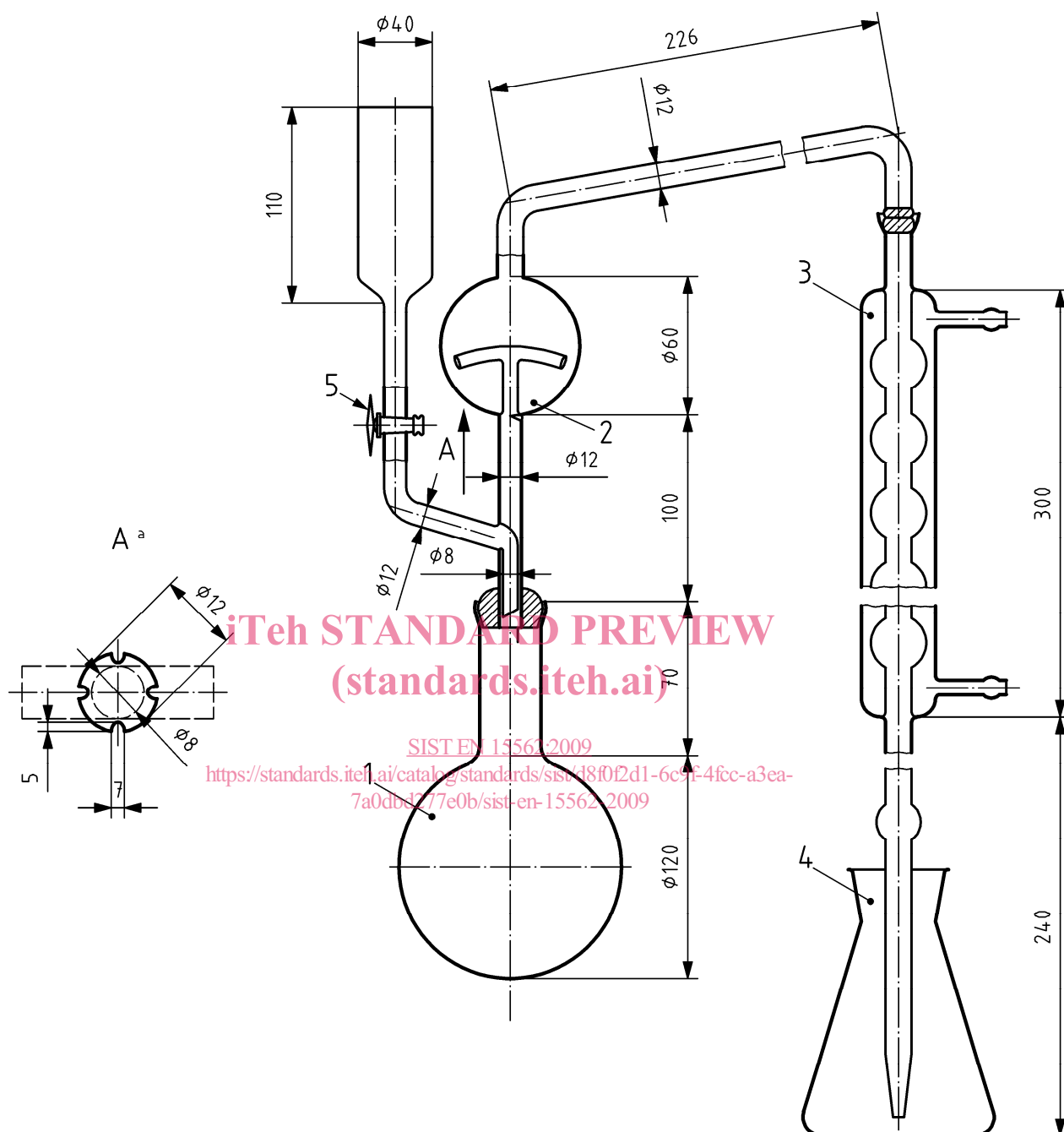
6.1 Distillation apparatus

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.

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

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