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SIST EN 15561:2009

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EUROPEAN STANDARD

EN 15561

NORME EUROPÉENNE

EUROPÄISCHE NORM

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English Version

Fertilizers - Determination of total nitrogen in calcium cyanamide containing nitrates

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

Düngemittel - Bestimmung von Gesamtstickstoff in nitrathaltigem 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 15561:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.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 15561:2007.

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 15561:2009 (E)**1 Scope**

This European Standard 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 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

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**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 Sulfuric acid, $\rho = 1,84$ g/ml.

5.3 Powdered iron, reduced in hydrogen.

5.4 Potassium sulfate, p.a., finely pulverized.

5.5 Sulfuric acid (for variant a), $c = 0,05$ mol/l.

5.6 Sodium or potassium hydroxide standard solution (for variant a), carbonate free, $c = 0,1 \text{ mol/l}$.

5.7 Sulfuric acid (for variant b, see NOTE in 8.2), $c = 0,1 \text{ mol/l}$.

5.8 Sodium or potassium hydroxide standard solution (for variant b, see NOTE in 8.2), carbonate free, $c = 0,2 \text{ mol/l}$.

5.9 Sulfuric acid (for variant c, see NOTE in 8.2), $c = 0,25 \text{ mol/l}$.

5.10 Sodium or potassium hydroxide standard solution (for variant c, see NOTE in 8.2), 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 $c = 0,1 \text{ mol/l}$ 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

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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 Stannous chloride solution

Dissolve 120 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 400 ml of concentrated hydrochloric acid ($\rho_{20} = 1,18 \text{ g/ml}$) 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 (I_2) of $c = 0,05 \text{ mol/l}$ in the presence of a starch solution as an indicator.

1 ml of iodine solution (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 (I_2) should be used.

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

5.14 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. Dissolve in water and make up to 250 ml. 1 ml of this solution contains 0,01 g of nitrogen.

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

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.

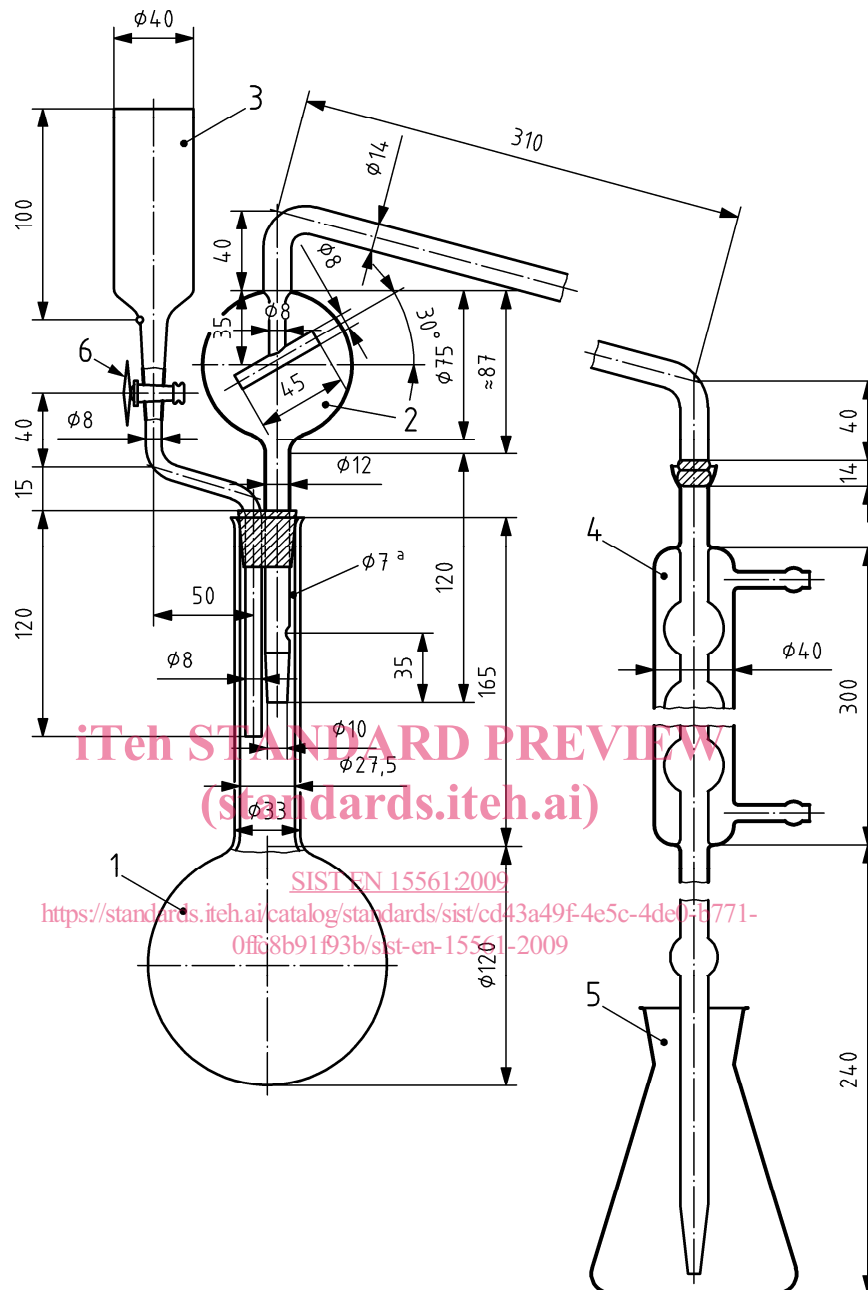
Automatic distillation apparatus may be used also, provided that the results are statistically equivalent.

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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