



**SLOVENSKI STANDARD**  
**SIST-TS CEN/TS 15561:2008**  
**01-marec-2008**

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Fertilizers - Determination of total nitrogen in calcium cyanamide containing nitrates

Düngemittel - Bestimmung von Gesamtstickstoff in nitrathaltigem Kalkstickstoff

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

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**Ta slovenski standard je istoveten z: CEN/TS 15561:2007**

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**ICS:**

65.080

**SIST-TS CEN/TS 15561:2008**

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ICS 65.080

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 von Gesamtstickstoff in nitrathaltigem Kalkstickstoff

This Technical Specification (CEN/TS) was approved by CEN on 1 January 2007 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

CEN members are required to announce the existence of this CEN/TS in the same way as for an EN and to make the CEN/TS available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the CEN/TS) until the final decision about the possible conversion of the CEN/TS into an EN is reached.

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EUROPÄISCHES KOMITEE FÜR NORMUNG

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## Foreword

This document (CEN/TS 15561:2007) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

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

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this Technical Specification: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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## 1 Scope

This Technical Specification 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)*

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## 3 Terms and definitions

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 tin(II)chloride. 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

$\rho = 1,84 \text{ g/ml}$

### 5.3 Powdered iron

reduced in hydrogen

**5.4 Potassium sulfate**

p.a., finely pulverized

**5.5 Sulfuric acid** (for variant a)

$c = 0,05 \text{ mol/l}$

**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 2)

$c = 0,1 \text{ mol/l}$

**5.8 Sodium or potassium hydroxide standard solution** (for variant b, see NOTE 2)

carbonate free,  $c = 0,2 \text{ mol/l}$

**5.9 Sulfuric acid** (for variant c, see NOTE 2)

$c = 0,25 \text{ mol/l}$

**5.10 Sodium or potassium hydroxide standard solution** (for variant c, see NOTE 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**

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 Tin(II)chloride solution**

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

NOTE It is essential to check the reducing power of the tin(II)chloride, using the following procedure. 0,5 g of  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$  are dissolved in 2 ml of concentrated hydrochloric acid ( $\rho = 1,18 \text{ g/ml}$ ) and made up to 50 ml with water. Then

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5 g of Rochelle salt (potassium sodium tartrate) are added and a sufficient quantity of sodium bicarbonate for the solution to show an alkaline reaction to a litmus paper test.

It is titrated with an iodine solution of  $c = 0,1 \text{ mol/l}$  in the presence of a starch solution as an indicator.

1 ml of iodine solution  $c = 0,1 \text{ mol/l}$  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 needs to be in a bivalent form. For the titration at least 35 ml of the  $c = 0,1 \text{ mol/l}$  iodine solution 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.

### 5.15 Anti-bump granules of pumice stone

washed in hydrochloric acid and calcined

## 6 Apparatus

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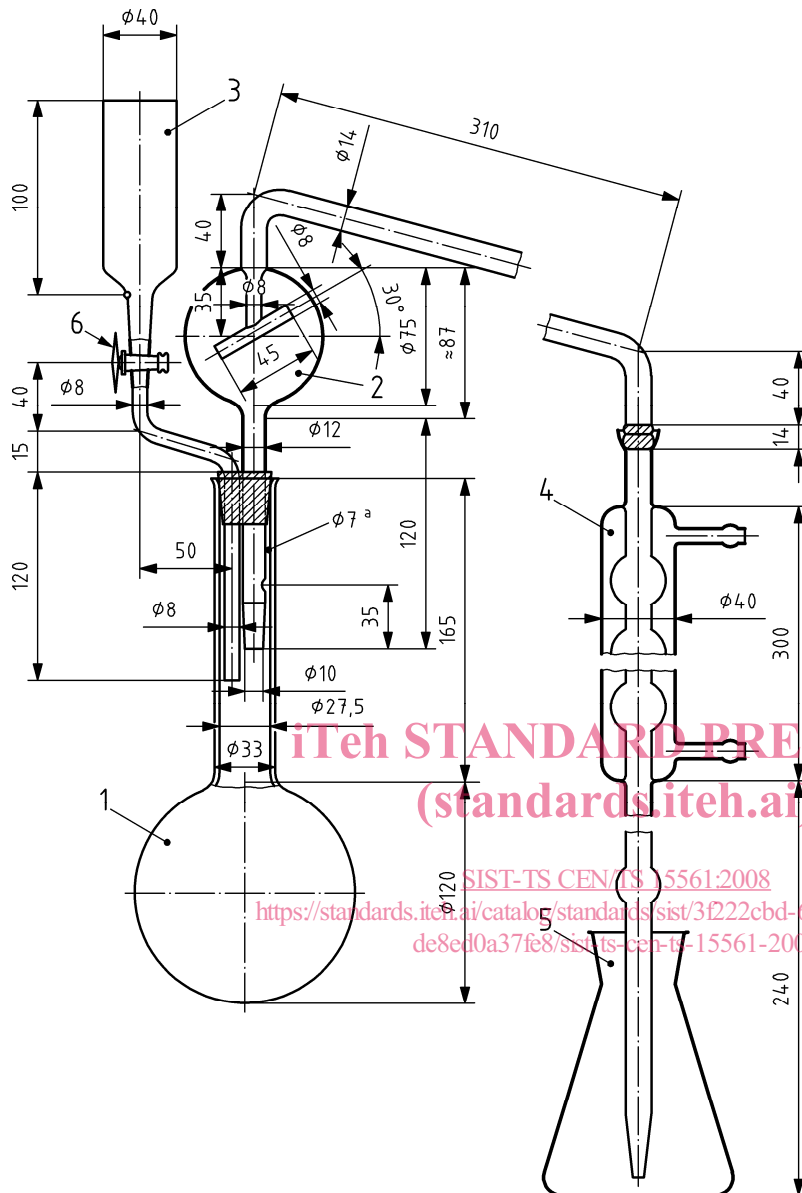
### 6.1 Distillation apparatus

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.



**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**