



SLOVENSKI STANDARD
SIST-TS CEN/TS 15475:2006

01-december-2006

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Fertilizers - Determination of ammoniacal nitrogen

Düngemittel - Bestimmung von Ammoniumstickstoff

Engrais - Détermination de l'azote ammoniacal

Ta slovenski standard je istoveten z: **CEN/TS 15475:2006**

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ICS:

65.080 Gnojila Fertilizers

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ICS 65.080

English Version

Fertilizers - Determination of ammoniacal nitrogen

Engrais - Détermination de l'azote ammoniacal

Düngemittel - Bestimmung von Ammoniumstickstoff

This Technical Specification (CEN/TS) was approved by CEN on 24 June 2006 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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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (CEN/TS 15475:2006) 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 CEN Technical Specification: Austria, Belgium, 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 the United Kingdom.

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1 Scope

This Technical Specification specifies a method for the determination of the ammoniacal nitrogen content in fertilizers. The method is applicable to all nitrogenous fertilizers including compound fertilizers, in which nitrogen is found exclusively either in the form of ammonium salts or ammonium salts together with nitrates.

The document is not applicable to fertilizers containing urea, cyanamide or other organic nitrogenous compounds.

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.

prEN 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

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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

Displacement of ammonia by means of an excess of sodium hydroxide, distillation and determining the yield of ammonia in a given volume of a standard sulfuric acid and titration of the excess acid by means of a standard solution of sodium or potassium hydroxide.

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 hydrochloric acid

Mix one volume of $\rho(\text{HCl}) = 1,18 \text{ g/ml}$ with one volume of water.

5.3 Sulfuric acid (for variant a)

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

5.4 Sodium or potassium hydroxide solution (for variant a)

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

5.5 Sulfuric acid (for variant b, see NOTE in 8.2)

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

5.6 Sodium or potassium hydroxide solution (for variant b, see NOTE in 8.2)

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

5.7 Sulfuric acid (for variant c, see NOTE in 8.2)

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

5.8 Sodium or potassium hydroxide solution (for variant c, see NOTE in 8.2)

carbonate free, $c = 0,5 \text{ mol/l}$

5.9 Sodium hydroxide

30 %, of approximately $\rho(\text{NaOH}) = 1,33 \text{ g/ml}$, ammonia free

5.10 Indicator solutions

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5.10.1 Mixed indicator

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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 A with two volumes of 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.

5.10.2 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 the preceding one. This indicator is red in acid solution and yellow in alkaline solution.

5.11 Anti-bump granules (i. e. pumice stone, glass pearls)

washed in hydrochloric acid and calcined

5.12 Ammonium sulfate

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.

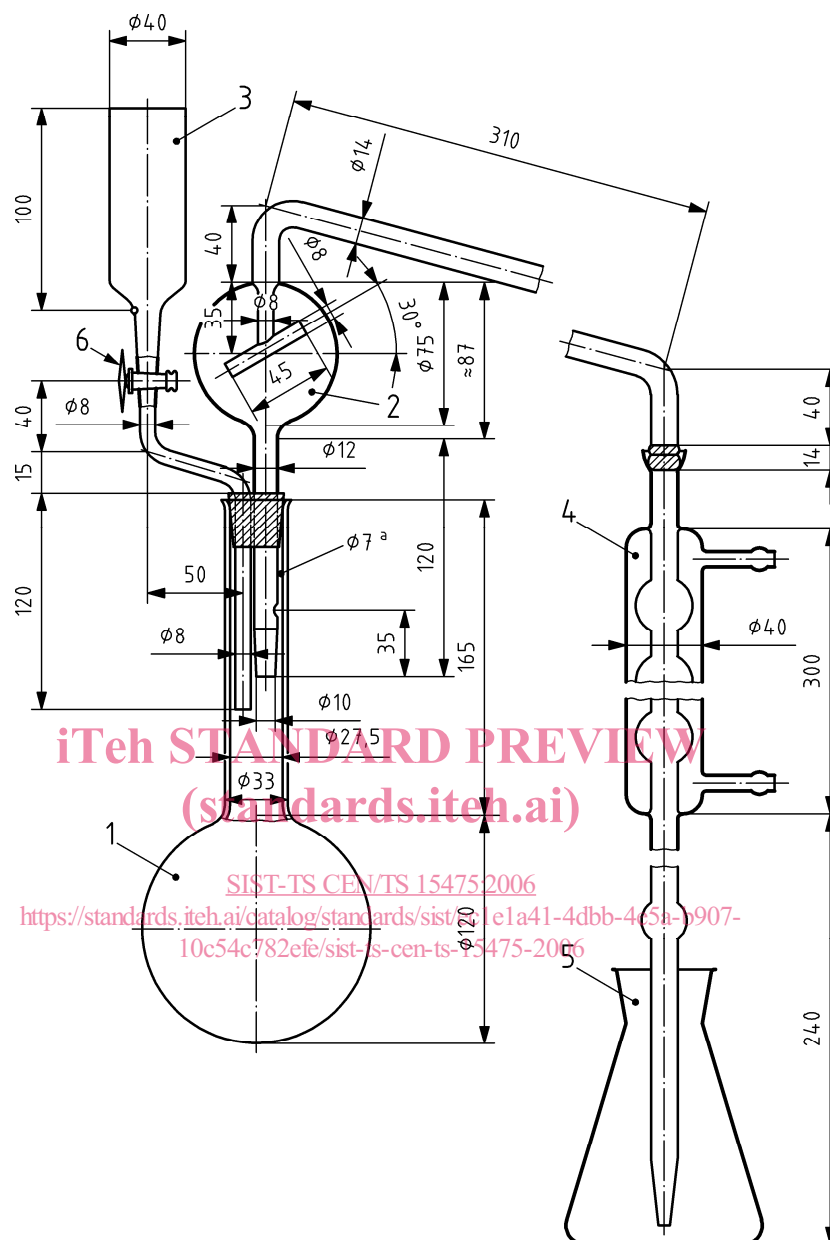
NOTE The different types of equipment recommended for this determination are reproduced, showing all the features of construction in Figures 1, 2, 3 and 4.

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

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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
- 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 (the tap may likewise be replaced by a rubber connection with a clip)

Figure 1 — Distillation apparatus 1