SLOVENSKI STANDARD
SIST-TS CEN/TS 15478:2006

01-december-2006

Gnojila ï Določ evanje celotnega duġika v seļ nini

Fertilizers - Determination of total nitrogen in urea

Düngemittel - Bestimmung von Gesamtstickstoff in Harnstoff

Engrais - Détermination de l'azote total dans l'urée

Ta slovenski standard je istoveten z: **CEN/TS 15478:2006**

ICS:

65.080 Gnojila Fertilizers

SIST-TS CEN/TS 15478:2006 **en,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 15478

September 2006

ICS 65.080

English Version

Fertilizers - Determination of total nitrogen in urea

Engrais - Détermination de l'azote total dans l'urée

Düngemittel - Bestimmung von Gesamtstickstoff in Harnstoff

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Ref. No. CEN/TS 15478:2006: E

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Foreword

This document (CEN/TS 15478:2006) 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.

1 Scope

This Technical Specification specifies a method for the determination of total nitrogen in urea. This method is applied exclusively to urea fertilizers which are nitrate free.

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 — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials — Vocabulary — Part 2: Terms relating to fertilizers (including corrigendum AC:2000)*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

3 Terms and definitions

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

4 Principle

Urea is transformed quantitatively into ammonia by boiling in the presence of sulfuric acid. The ammonia thus obtained is distilled from an alkaline medium, the distillate being collected in an excess of standard sulfuric acid. The excess acid is titrated by means of a standard alkaline 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 Potassium sulfate

Kjeldahl tablets, 5 g/tablet containing 100 parts K_2SO_4 to 1 part selenium

5.3 Sulfuric acid

concentrated (ρ_{20} = 1,84 g/ml)

5.4 Sodium hydroxide solution

approximately NaOH 500g/l

5.5 Sulfuric acid

c = 0,05 mol/l, to use for the blank test.

5.6 Sodium or potassium hydroxide solution

carbonate free, *c* = 0,1 mol/l, to use for the blank test.

5.7 Sulfuric acid

c = 0,5 mol/l

5.8 Sodium or potassium hydroxide solution

carbonate free, *c* = 1,0 mol/l

5.9 Indicator solutions

5.9.1 Mixed indicator

Solution A: Dissolve 1 g of methyl red in 37 ml of 0,1 mol/l sodium hydroxide solution and make up to one litre with water.

Solution B: Dissolve 1 g of methylene blue in water and make up to one litre.

Mix one volume of A with two volumes of B.

This indicator is violet in an acid solution, grey in a neutral solution and green in an alkaline solution; use 0,5 ml (10 drops).

5.9.2 Methyl red indicator solution

Dissolve 0,1 g of methyl red in 50 ml of 95 % ethanol and make up to 100 ml with water. Filter if necessary. This indicator (4 or 5 drops) may be used instead of the preceding one. This indicator is red in acid solution and yellow in alkaline solution.

5.10 Anti-bump granules

pumice stone, washed in hydrochloric acid and calcined.

5.11 Urea

p. a.

6 Apparatus

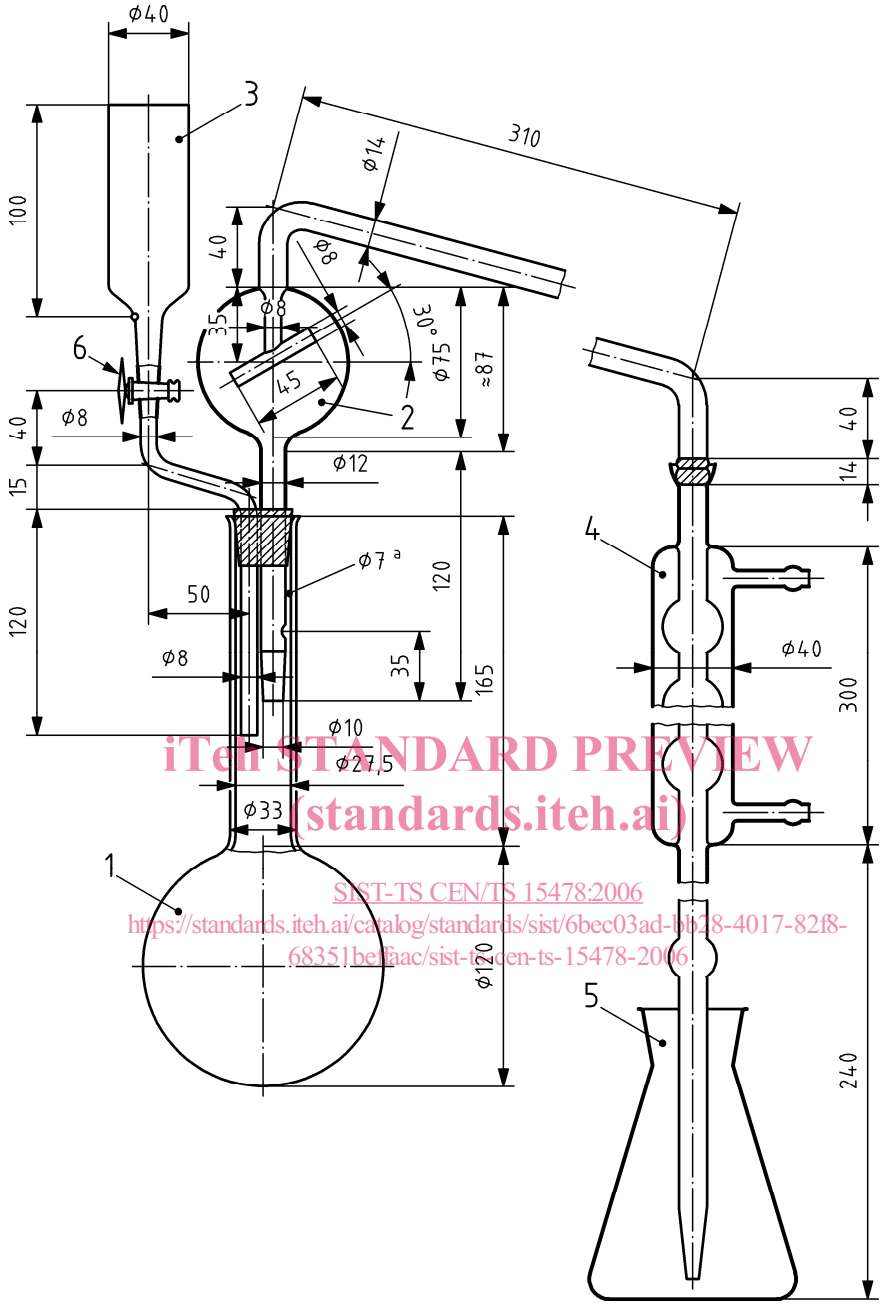
6.1 Distillation apparatus

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.

Automatic distillation apparatus may be used as well 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

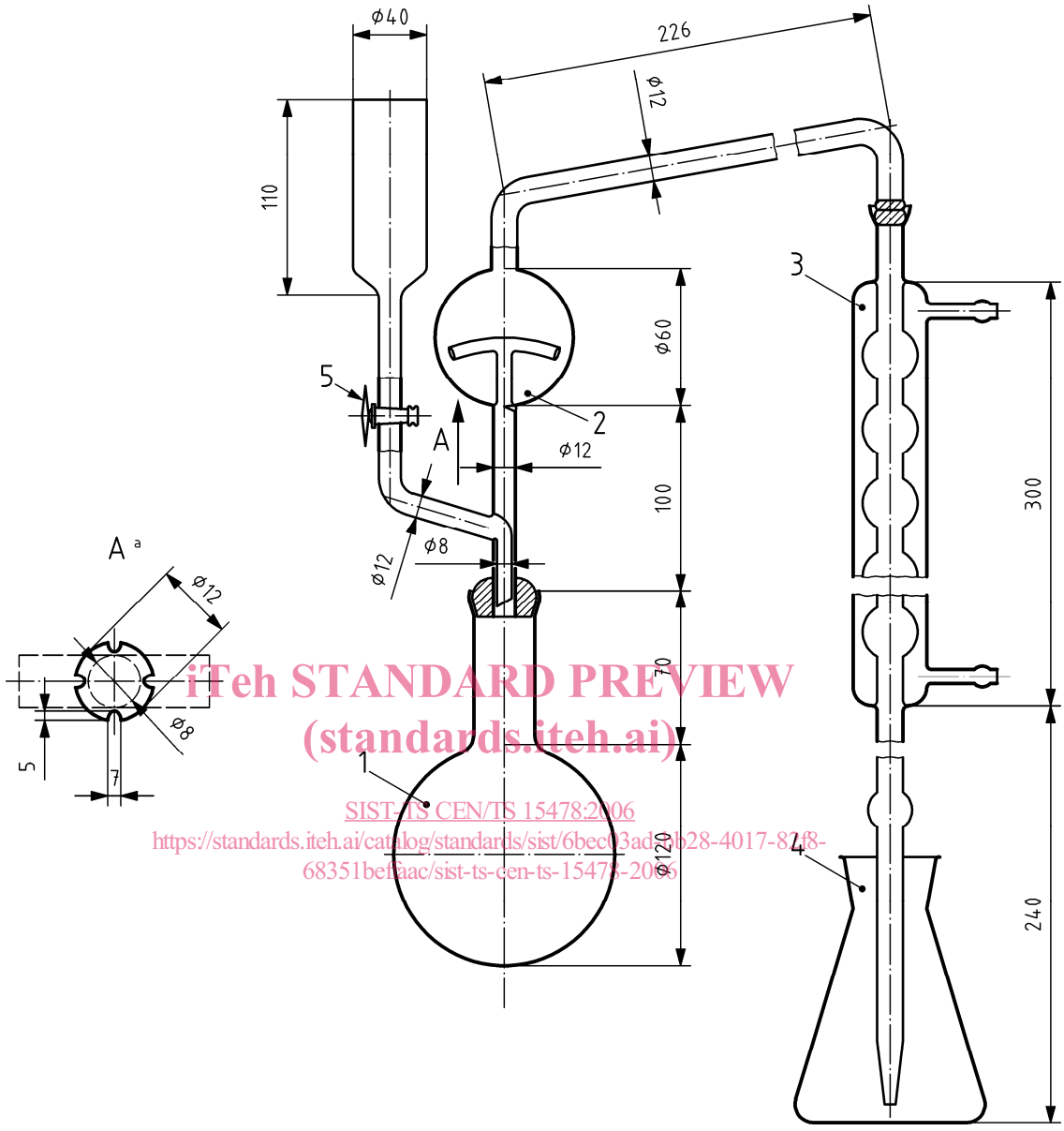
4 six-bulb condenser with spherical joint (No 18) at the entrance, and joined at the issue to a glass extension tube by means of a small rubber connection (when the connection to the distillation tube is effected by means of a rubber tube, the spherical joint may be replaced by a suitable rubber bung)

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

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 (6) 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