SLOVENSKI STANDARD
SIST-TS CEN/TS 15476:2006

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Gnojila ï Doloļ evanje nitratnega in amonijevega duġika po Devardi

Fertilizers - Determination of nitric and ammoniacal nitrogen according to Devarda

Düngemittel - Bestimmung von Nitrat- und Ammoniumstickstoff nach Devarda

Engrais - Détermination de l'azote nitrique et ammoniacal selon Devarda

Ta slovenski standard je istoveten z: **CEN/TS 15476:2006**

ICS:

65.080 Gnojila Fertilizers

SIST-TS CEN/TS 15476:2006 **en,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

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CEN/TS 15476

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ICS 65.080

English Version

Fertilizers - Determination of nitric and ammoniacal nitrogen according to Devarda

Engrais - Détermination de l'azote nitrique et ammoniacal selon Devarda

Düngemittel - Bestimmung von Nitrat- und Ammoniumstickstoff nach Devarda

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (CEN/TS 15476:2006) 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 nitrate and ammoniacal nitrogen with reduction using Devarda alloy (modified for each of the variants a, b and c).

The method is applicable to all nitrogenous fertilizers, including compound fertilizers, in which nitrogen is found exclusively in nitrate form or in ammoniacal and nitrate form.

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*

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.

4 Principle

Reduction of nitrates and nitrites to ammonia in a strongly alkaline solution by means of a metallic alloy composed of 45 % Al, 5 % Zn and 50 % Cu (Devarda alloy). Distillation of the ammonia and determination of the yield in a known volume of standard sulfuric acid; titration of the excess sulfuric 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 ρ(HCl) = 1,18 g/ml with one volume of water.

5.3 Sulfuric acid (for variant a)

c = 0,05 mol/l

5.4 Sodium or potassium hydroxide solution (for variant a)

carbonate free, c = 0,1 mol/l

5.5 Sulfuric acid (for variant b, see NOTE 2 in 8.4)

c = 0,1 mol/l

5.6 Sodium or potassium hydroxide solution (for variant b, see NOTE 2 in 8.4)

carbonate free, c = 0,2 mol/l

5.7 Sulfuric acid (for variant c, see NOTE 2 in 8.4)

c = 0,25 mol/l

5.8 Sodium or potassium hydroxide solution (for variant c, see NOTE 2 in 8.4)

carbonate free, c = 0,5 mol/l

5.9 Devarda alloy for analysis

Powdered in such away that a mass fraction of 90 % to 100 % will pass through a sieve with apertures less than 0,25 mm square, a mass fraction of 50 % to 75 % will pass through a sieve with apertures of less than 0,075 mm square.

Pre-packed bottles containing a maximum of 100 g are recommended.

5.10 Sodium hydroxide solution

30 % of approximately ρ(NaOH) = 1,33 g/ml, ammonia free

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 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.

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

5.12 Ethanol

with a mass fraction of 95 % to 96 % ethanol

5.13 Sodium nitrate

p. a.

6 Apparatus

6.1 Distillation apparatus

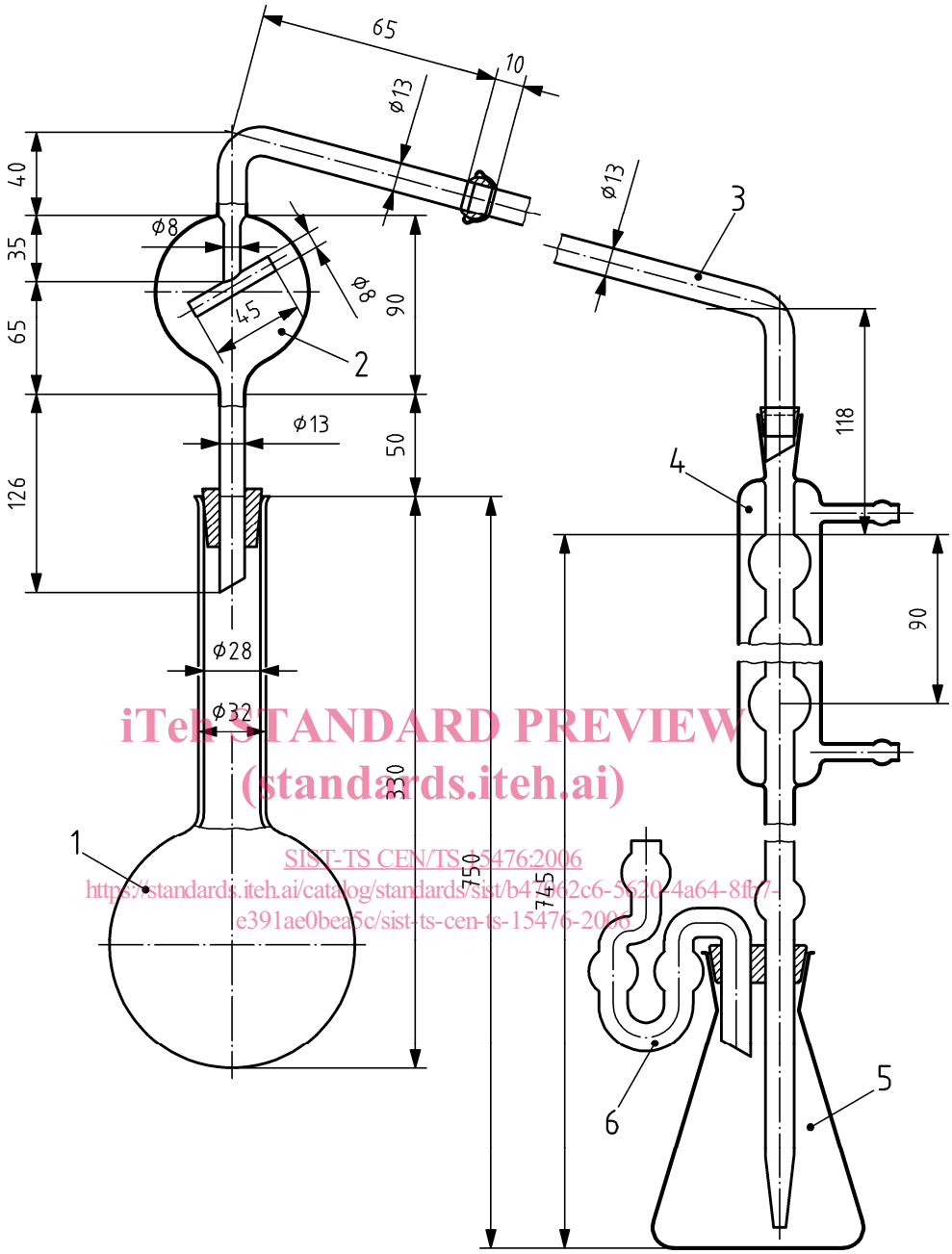
Consisting of a round-bottomed flask of suitable capacity, connected to a condenser by a distillation tube with a splash head, additionally equipped with a bubble trap on the receiving flask to prevent any loss of ammonia.

The type of apparatus recommended for this determination is reproduced, showing all the features of construction, in Figure 1.

The equipment is made of borosilicate glass.

An automatic distillation apparatus may be used as well provided that the results are statistically equivalent.

Dimensions in millimetres



Key

1 750 ml or 1 000 ml round-bottomed, long-necked flask with a bell mouth.

2 distillation tube with a splash head and a No 18 spherical joint at the issue

3 elbow tube with a No 18 spherical joint at the entrance, and a drip cone at the issue (a suitable rubber connection may be used instead of the spherical joint)

4 six-bulb condenser with an extension tube mounted on a rubber bung holding a bubble trap

5 750 ml receiving flask

6 bubble trap to prevent loss of ammonia

Figure 1 — Distillation apparatus