SLOVENSKI STANDARD
SIST EN 15476:2009

01-junij-2009

Nadomeġļa:
SIST-TS CEN/TS 15476:2006

Gnojila - Doloļevanje 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: EN 15476:2009

ICS:

65.080 Gnojila Fertilizers

SIST EN 15476:2009 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15476

January 2009

ICS 65.080

Supersedes CEN/TS 15476:2006

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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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 15476:2009: E

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Foreword

This document (EN 15476:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.

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1 Scope

This European Standard 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.

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 inprovers — 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

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

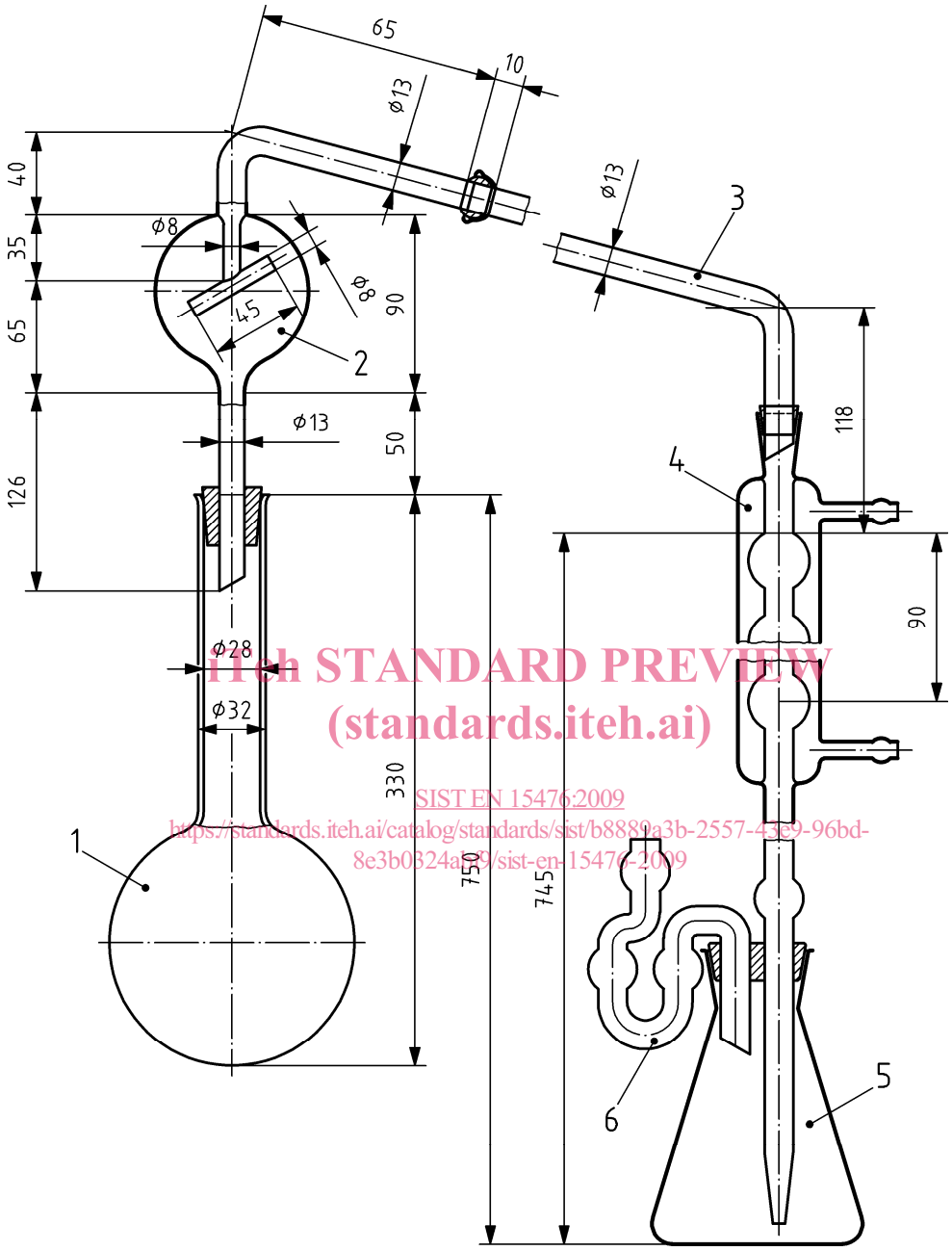
Consisting of a round-bottomed flask of suitable capacity, connected to a condenser by a distillation tube with a splash head, additionally equipped with a bubble trap on the receiving flask to prevent any loss of ammonia.

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

6.2 Pipettes, capacity of 10 ml, 20 ml, 25 ml, 50 ml, 100 ml and 200 ml.

6.3 Graduated flask, capacity 500 ml.

6.4 Rotary shaker, 35 to 40 revolutions per minute.

7 Sampling and sample preparation

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2.

8 Procedure

8.1 Preparation of the solution

According to Table 1, Table 2 or Table 3, depending on the variant chosen weigh to the nearest 0,001 g, a quantity of 5 g, 7 g or 10 g of the prepared sample and transfer into a beaker glass of 250 ml.

Add about 50 ml of water and 20 ml hydrochloric acid (5.2).

Cover the beaker, heat until boiling and cool down.

Filter the content of the beaker and collect the filtrate and the rinse water in a volumetric flask of 500 ml, dilute to the mark with water and mix homogeneously.

8.2 Analysis of the solution

The quantity of nitric nitrogen present in the aliquot part of the solution shall not exceed the maximum quantity expressed in Table 1, Table 2 or Table 3.

According to the variant chosen, place in the receiving flask an exactly measured quantity of standard sulfuric acid as indicated in Table 1. Add the appropriate quantity of the chosen indicator solution (5.11.1 or 5.11.2) and finally, sufficient water to give a volume of 50 ml. The end of the extension tube of the condenser shall be underneath the surface of the solution. Fill the bubble trap with water.

Using a precision pipette, take an aliquot part as indicated in Table 1. Place it in the distillation flask.

Add sufficient water to the distillation flask to obtain a volume of 250 ml to 300 ml, 5 ml ethanol (5.12) and 4 g Devarda's alloy (5.9), (see NOTE).

Taking the necessary precautions to avoid loss of ammonia, add to the flask about 30 ml of 30 % sodium hydroxide solution (5.10) and finally, in the case of acid soluble samples an additional quantity sufficient to neutralize the quantity of hydrochloric acid (5.2) present in the aliquot part taken for the analysis. Connect the distillation flask to the apparatus, ensuring the tightness of connections. Carefully shake the flask to mix the contents.

Warm gently, so that the release of hydrogen decreases appreciably over about half an hour and the liquid will boil. Continue the distillation, increasing the heat so that at least 200 ml liquid distils in about 30 min (do not prolong the distillation beyond 45 min).

When the distillation is complete, disconnect the receiving flask from the apparatus; carefully wash the extension tube and bubble trap, collecting the rinsing in the titration flask and titrate the surplus acid with the standard solution of sodium or potassium hydroxide prescribed for the variant adopted (see NOTE 2 in 8.4).