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SIST EN 15559:2009

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EUROPEAN STANDARD

EN 15559

NORME EUROPÉENNE

EUROPÄISCHE NORM

January 2009

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Supersedes CEN/TS 15559:2007

English Version

Fertilizers - Determination of nitric and ammoniacal nitrogen according to Arnd

Engrais - Dosage de l'azote nitrique et ammoniacal selon
Arnd

Düngemittel - Bestimmung von Nitrat- und
Ammoniumstickstoff nach Arnd

This European Standard was approved by CEN on 6 December 2008.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN Management Centre or to any CEN member.

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Foreword

This document (EN 15559:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 15559:2007.

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 implement this European Standard: 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 the United Kingdom.

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EN 15559:2009 (E)**1 Scope**

This European Standard specifies a method for the determination of nitric and ammoniacal nitrogen with reduction according to Arnd (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 improvers — 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 neutral aqueous solution by means of a metallic alloy composed of 60 % Cu and 40 % Mg (Arnd's alloy) in the presence of magnesium chloride.

Distillation of the ammonia and determination of the yield in a known volume of standard sulfuric acid solution. 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 concentrated hydrochloric acid solution, $\rho(\text{HCl}) = 1,18 \text{ g/ml}$, 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 NOTES in 8.2), $c = 0,1$ mol/l.
- 5.6 Sodium or potassium hydroxide solution** (for variant b, see NOTES in 8.2), carbonate free, $c = 0,2$ mol/l.
- 5.7 Sulfuric acid** (for variant c, see NOTES in 8.2), $c = 0,25$ mol/l.
- 5.8 Sodium or potassium hydroxide solution** (for variant c, see NOTES in 8.2), carbonate free, $c = 0,5$ mol/l.
- 5.9 Sodium hydroxide solution**, approximately $c = 2$ mol/l.
- 5.10 Arnd's alloy**, powdered so as to pass through a sieve with apertures less than 1 mm square.
- 5.11 Magnesium chloride solution**, $\rho = 20$ %.

Dissolve 200 g of magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in approximately 600 ml to 700 ml of water in a 1 l flat-bottomed flask. To prevent frothing, add 15 g of magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$).

After dissolution add 2 g of magnesium oxide and a few anti-bump granules of pumice stone and concentrate the suspension to 200 ml by boiling, thus expelling any trace of ammonia from the reagents. Cool, make up the volume to 1 l and filter.

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5.12 Indicator solutions

5.12.1 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 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.

5.12.2 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 5.12.1.

5.12.3 Congo red indicator solution

Dissolve 3 g of Congo red in 1 l of warm water and filter if necessary after cooling. This indicator may be used, instead of that specified in 5.12.1 or 5.12.2, in the neutralization of acid extracts before distillation, using 0,5 ml per 100 ml of liquid to be neutralized.

5.13 Anti-bump granules, for example pumice stone, washed in hydrochloric acid and reclaimed.

EN 15559:2009 (E)**5.14 Sodium nitrate, 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.

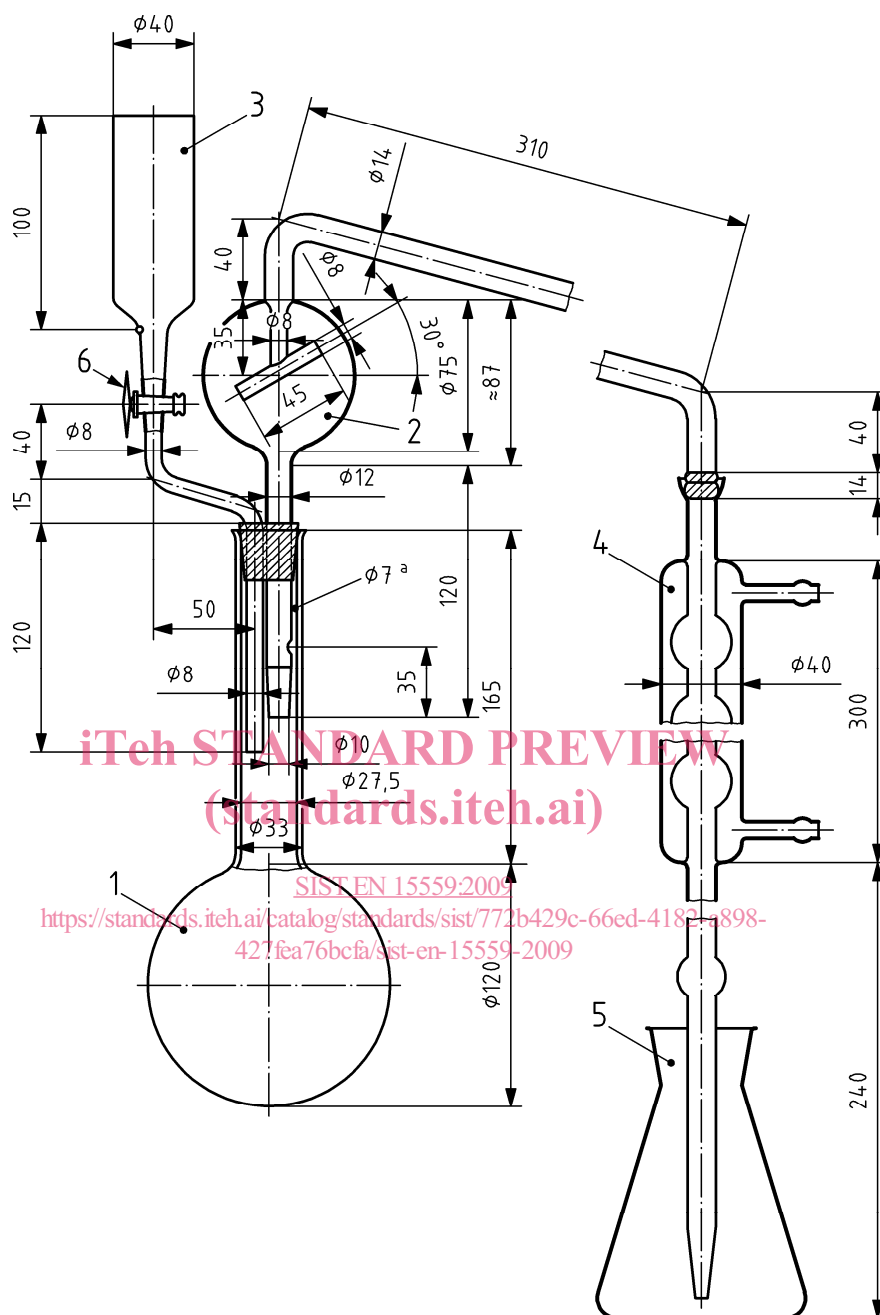
An automatic distillation apparatus may also be used, provided that the results are statistically equivalent.

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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 (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
- ^a hole

Figure 1 — Distillation apparatus 1