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SIST EN 15558:2009

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EUROPEAN STANDARD

EN 15558

NORME EUROPÉENNE

EUROPÄISCHE NORM

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English Version

## Fertilizers - Determination of nitric and ammoniacal nitrogen according to Ulsch

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

Düngemittel - Bestimmung von Nitrat- und Ammoniumstickstoff nach Ulsch

This European Standard was approved by CEN on 30 November 2008.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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## Foreword

This document (EN 15558:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.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 15558:2007.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

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**EN 15558:2009 (E)****1 Scope**

This European Standard specifies a method for the determination of nitrate and ammoniacal nitrogen with reduction according to Ulsch.

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)*

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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.

**4 Principle**

Reduction of nitrates and nitrites to ammonia by means of metallic iron in an acid medium and displacement of the ammonia thus formed by the addition of an excess of sodium hydroxide: distillation of the ammonia and determination of the yield of ammonia in a known volume of standard sulfuric acid solution and 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 concentrated hydrochloric acid solution,  $\rho(\text{HCl}) = 1,18 \text{ g/ml}$ , with one volume of water.

**5.3 Sulfuric acid**,  $c = 0,05$  mol/l.

**5.4 Sodium or potassium hydroxide solution**, carbonate free,  $c = 0,1$  mol/l.

**5.5 Sulfuric acid solution**, approximately 30 %  $H_2SO_4$  (mass concentration), ammonia free.

**5.6 Powdered iron**, reduced in hydrogen.

The prescribed quantity of iron shall be able to reduce at least 0,05 g of nitrate nitrogen.

**5.7 Sodium hydroxide solution**, 30 % mass concentration, of approximately  $\rho(NaOH) = 1,33$  g/ml, ammonia free.

## 5.8 Indicator solutions

### 5.8.1 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.

### 5.8.2 Methyl red indicator solution [SIST EN 15558:2009](#)

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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 5.8.1.

**5.9 Anti-bump granules**, for example pumice stone, washed in hydrochloric acid and calcined.

**5.10 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.

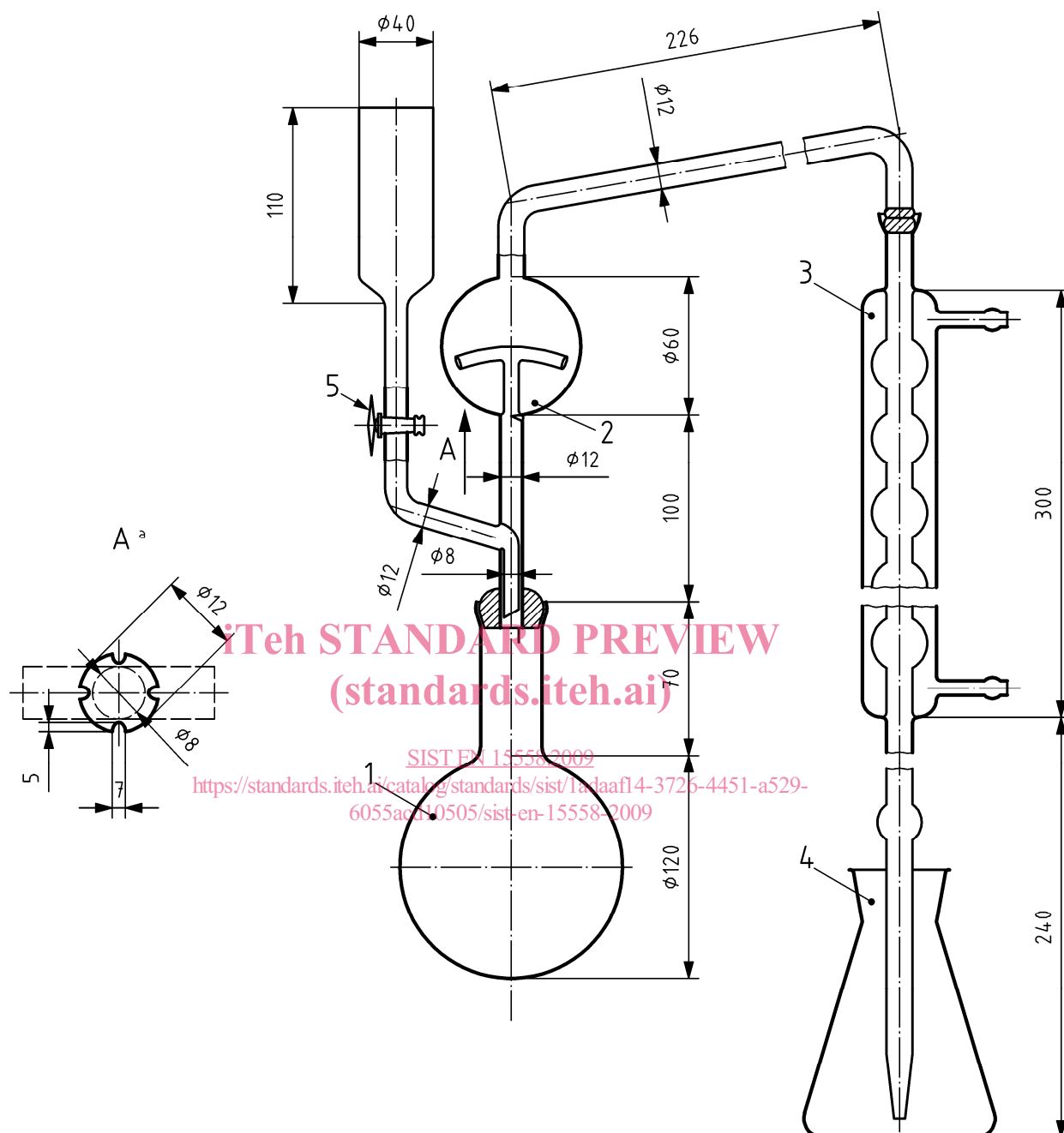
NOTE The different types of equipment recommended for this determination are reproduced, showing all the features of construction, in Figures 1, 2, 3 and 4.

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





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 (5) 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
- <sup>a</sup> enlarged description

Figure 2 — Distillation apparatus 2