

SLOVENSKI STANDARD

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SIST-TS CEN/TS 15750:2008

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Fertilizers - Determination of total nitrogen in fertilizers containing nitrogen only as nitric, ammoniacal and urea nitrogen by two different methods

Düngemittel - Bestimmung von Gesamtstickstoff in Düngemitteln mit Stickstoff in Form von Ammonium, Nitrat und Harnstoff unter Anwendung von zwei verschiedenen Verfahren

[SIST EN 15750:2010](#)

Engrais - Détermination de la teneur en azote totale des engrais ne contenant l'azote que sous forme nitrique, ammoniacale et uréique selon deux méthodes différentes

Ta slovenski standard je istoveten z: **EN 15750:2009**

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65.080

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Fertilizers

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EUROPEAN STANDARD

EN 15750

NORME EUROPÉENNE

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October 2009

ICS 65.080

Supersedes CEN/TS 15750:2008

English Version

**Fertilizers - Determination of total nitrogen in fertilizers
containing nitrogen only as nitric, ammoniacal and urea nitrogen
by two different methods**

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This European Standard was approved by CEN on 20 September 2009.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

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Foreword

This European Standard (EN 15750:2009) 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of an identical text or by endorsement, at the latest by April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.

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

This document supersedes CEN/TS 15750:2008.

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Introduction

This document specifies two different methods for the determination of the total nitrogen content in fertilizers. Based on the statistical results of the inter-laboratory tests, obtained with the same samples, the two methods produce equivalent results, and hence can be used both on decision of the user.

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1 Scope

This European Standard specifies two different methods (Methods A and B) for the determination of the total nitrogen content in fertilizers. Method A specifies the titrimetric method after distillation according to ISO 5315:1984. Method B specifies a method by reduction of nitrate with iron and tin(II)-chloride.

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 385, *Laboratory glassware — Burettes (ISO 385:2005)*

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

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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

4.1 Method A – Titrimetric method after distillation according to ISO 5315:1984

Reduction of nitrate to ammonia by chromium powder in acid medium. Conversion of organic and urea nitrogen into ammonium sulfate by digestion with concentrated sulfuric acid in the presence of a catalyst. Distillation of the ammonia from an alkaline solution, absorption in an excess of standard volumetric sulfuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of methyl red or screened methyl red as indicator.

4.2 Method B – Reduction of nitrate with iron and tin(II)-chloride

Reduction of nitrate to ammonia by iron powder and tin chloride in acid medium. Conversion of organic and urea nitrogen into ammonium sulfate by digestion with concentrated sulfuric acid in the presence of a catalyst. Distillation of the ammonia from an alkaline solution, absorption in an excess of standard volumetric sulfuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of an indicator solution.

5 Sampling and sample preparation

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

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

6 Method A

6.1 Reagents

Use only reagents of recognized analytical grade having, in particular, low nitrogen contents and distilled or demineralized water (grade 3 according to EN ISO 3696).

6.1.1 Chromium metal, powder of particle size less than or equal to 250 µm.

6.1.2 Aluminium oxide, fused, pumice is suitable.

6.1.3 Anti-foaming agent, for example paraffin wax of melting point not lower than 100 °C, or a silicone.

6.1.4 Ammonium nitrate, dried at 100 °C to constant mass.

6.1.5 Digestion catalyst mixture, finely ground, comprising

— potassium sulfate (K_2SO_4): 1 000 g;

— copper(II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$): 50 g;

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6.1.6 Sulfuric acid, concentrated, ρ approximately 1,84 g/ml.

6.1.7 Hydrochloric acid, concentrated, ρ approximately 1,18 g/ml.

6.1.8 Sodium hydroxide, approximately 400 g/l solution.

6.1.9 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0,10$ mol/l.

6.1.10 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0,25$ mol/l.

6.1.11 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0,10$ mol/l.

6.1.12 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0,05$ mol/l.

6.1.13 Indicator solution,

use either the screened methyl red solution (6.1.13.1) or the methyl red solution (6.1.13.2).

6.1.13.1 Screened methyl red, ethanolic indicator solution;

mix 50 ml of a 2 g/l ethanolic solution of methyl red with 50 ml of a 1 g/l ethanolic solution of methylene blue.

6.1.13.2 Methyl red, ethanolic indicator solution;

dissolve 0,1 g of methyl red in 50 ml of 95 % (volume fraction) ethanol.

6.1.14 pH indicator paper, wide range.**6.2 Apparatus**

Common laboratory equipment and glassware, in particular equipment according to 6.2.1 to 6.2.5.

6.2.1 Digestion apparatus, comprising an 800 ml Kjeldahl flask and a pear-shaped hollow glass stopper.**6.2.2 Distillation apparatus**

The components of the distillation apparatus may be connected by means of rubber bungs and tubing or by the use of spherical ground glass joints.

Spherical ground glass joints should be held spring clamps to ensure that they are leak tight. Rubber bungs and tubing shall be replaced when they begin to perish or show signs of wear.

Suitable apparatus is illustrated in Figure 1. An automatic distillation apparatus may also be used, provided that the results are statistically equivalent.

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