SLOVENSKI STANDARD
SIST-TS CEN/TS 15750:2008

01-oktober-2008

Gnojila - Določevanje različnih oblik dušika v gnojilih, ki vsebuje dušik v nitratni, amonijevi in setveni obliki z dvema različnima metodama

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

Düngemittel - Bestimmung verschiedener nebeneinander anwesender Stickstoff-Formen in Düngemitteln mit Stickstoff in Form von Ammonium, Nitrat und Harnstoff unter Anwendung von zwei verschiedenen Verfahren

Engrais - Détermination des teneurs des différentes formes d'azote en présence les unes des autres dans les engrais ne contenant l'azote que sous forme nitrique, ammoniacale et uréique en utilisant deux méthodes différentes

Ta slovenski standard je istoveten z: **CEN/TS 15750:2008**

ICS:

65.080	Gnojila	Fertilizers

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TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 15750

August 2008

ICS 65.080

English Version

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

Engrais - Détermination des teneurs des différentes formes d'azote en présence les unes des autres dans les engrais ne contenant l'azote que sous forme nitrique, ammoniacale et uréique en utilisant deux méthodes différentes

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

This Technical Specification (CEN/TS) was approved by CEN on 11 May 2008 for provisional application.

The period of validity of this CEN/TS is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the CEN/TS can be converted into a European Standard.

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

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Foreword

This document (CEN/TS 15750:2008) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

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1 Scope

This document 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 [2]. 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 385:2005, *Laboratory glassware – Burettes (ISO 385:2005)*

EN ISO 3696, *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

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 Technical Specification. A recommended sampling method is given in EN 1482-1 [1].

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

6 Method A

6.1 Reagents

6.1.1 General

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).

6.1.2 Chromium metal, powder, of particle size less than or equal to 250 µm;

6.1.3 Aluminium oxide, fused, pumice is suitable;

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

6.1.5 Ammonium nitrate, dried at 100 °C to constant mass;

6.1.6 Digestion catalyst mixture, finely ground, comprising

— potassium sulfate (K_2SO_4): 1 000 g

— copper(II) sulfate pentahydrate ($CuSO_4.5H_2O$): 50 g

6.1.7 Sulfuric acid, concentrated, ρ approximately 1,84 g/ml;

6.1.8 Hydrochloric acid, concentrated, ρ approximately 1,18 g/ml;

6.1.9 Sodium hydroxide, approximately 400 g/l solution;

6.1.10 Sodium hydroxide, standard volumetric solution, c(NaOH)=0,10 mol/l;

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

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

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

6.1.14 Indicator solution,

Use either the screened methyl red solution (6.1.14.1) or the methyl red solution (6.1.14.2).

6.1.14.1 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.

6.1.14.2 Methyl red, ethanolic indicator solution;

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

6.1.15 pH indicator paper, wide range.

6.2 Apparatus

6.2.1 General

Common laboratory equipment and glassware, in particular equipment according to 6.2.2 to 6.2.6.

6.2.2 Digestion apparatus, comprising an 800 ml Kjeldahl flask and a pear-shaped hollow glass stopper;

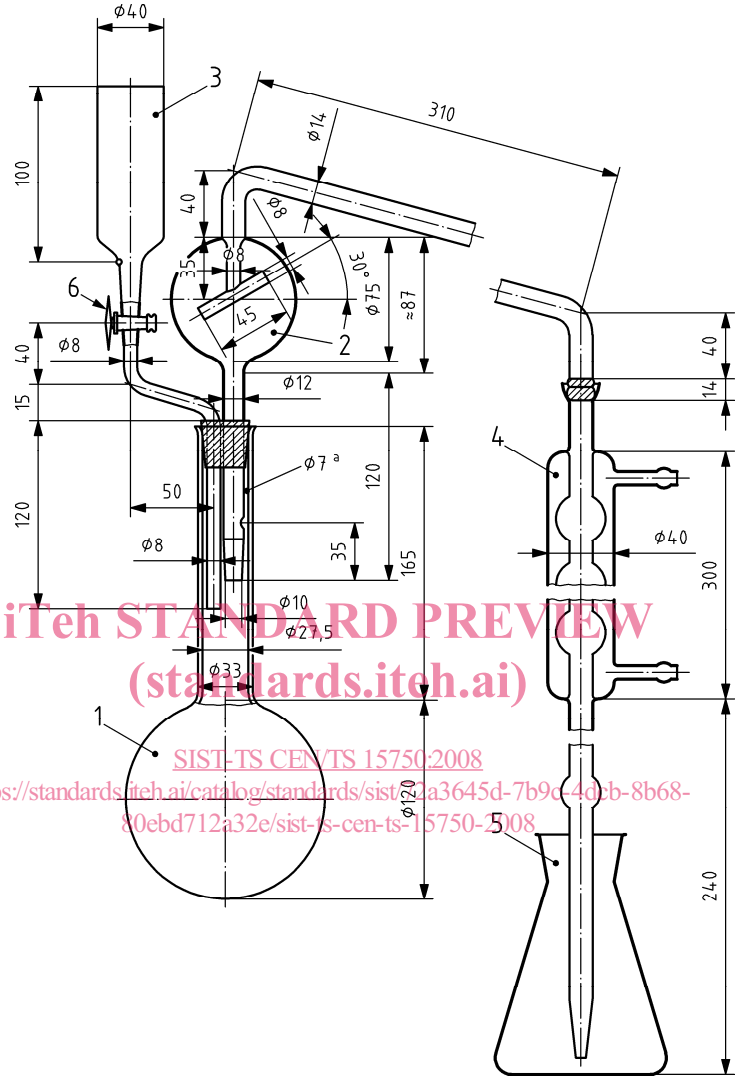
6.2.3 Distillation apparatus

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.

Dimensions in millimetres



Key

1 Kjeldahl flask (6.2.2) or round-bottomed, long-necked flask of 1 000 ml capacity

2 distillation tube with a single-bulb 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) (the tap may likewise be replaced by a rubber connection with a clip)

4 seven-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 – Typical distillation apparatus (using a round bottom flask)