

Fertilizers — Determination of different forms of nitrogen in the same sample, containing nitrogen as nitric, ammoniacal, urea and cyanamide nitrogen

ICS 65.080

National foreword

This British Standard is the UK implementation of EN 15604:2009. It supersedes DD CEN/TS 15604:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

Fertilizers - Determination of different forms of nitrogen in the same sample, containing nitrogen as nitric, ammoniacal, urea and cyanamide nitrogen

Engrais - Détermination des différentes formes d'azote dans un même échantillon contenant l'azote sous forme nitrique, ammoniacale, uréique et cyanamidique

Düngemittel - Bestimmung verschiedener, nebeneinander anwesender Stickstoff-Formen in derselben Probe mit Stickstoff in Form von Ammonium, Nitrat, Harnstoff und Cyanamid

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Foreword

This document (EN 15604:2009) 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of an identical text or by endorsement, at the latest by July 2009, and conflicting national standards shall be withdrawn at the latest by July 2009.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 any one form of nitrogen in the presence of any other form.

The method is applicable to any fertilizer provided for in Annex I of the Regulation (EC) No 2003/2003 [1] containing nitrogen in various forms.

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 15475, *Fertilizers — Determination of ammoniacal nitrogen*

EN 15562, *Fertilizers — Determination of cyanamide nitrogen*

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

4.1 Total soluble and insoluble nitrogen

According to the list of standard fertilizers given in Annex I of [1], this determination is applicable to products containing calcium cyanamide.

In the absence of nitrates, the test sample is mineralized by direct Kjeldahl digestion.

In the presence of nitrates, the test sample is mineralized by Kjeldahl digestion after reduction with the aid of metallic iron and stannous chloride.

In both cases, the ammonia is determined according to EN 15475.

NOTE If analysis shows an insoluble nitrogen content of more than 0,5 %, one concludes that the fertilizer contains other forms of insoluble nitrogen not included in the list in [1], Annex I.

4.2 Forms of soluble nitrogen

4.2.1 General

The forms of soluble nitrogen referred to in 4.2.2 to 4.2.7 are determined from different aliquots taken from the same solution of the test sample:

4.2.2 Total soluble nitrogen

4.2.2.1 In the absence of nitrates, by direct Kjeldahl digestion. The ammonia is then determined (by the same method as that described in EN 15475).

4.2.2.2 In the presence of nitrates, by Kjeldahl digestion on an aliquot part taken from the solution after reduction according to Ulsch. The ammonia is then determined (by the same method as that described in EN 15475).

4.2.3 Total soluble nitrogen with the exception of nitrate nitrogen

By Kjeldahl digestion after elimination in an acid medium of nitrate nitrogen with ferrous sulfate. The ammonia is then determined (by the same method as that described in EN 15475).

4.2.4 Nitrate nitrogen by difference

4.2.4.1 In the absence of calcium cyanamide, by determining the difference between the nitrogen determined as summarized in 4.2.2.2 and that determined as summarized in 4.2.3 or between total soluble nitrogen (4.2.2) and the sum of ammoniacal nitrogen and ureic organic nitrogen (4.2.5 + 4.2.6).

4.2.4.2 In the presence of calcium cyanamide, by determining the difference between the nitrogen determined as summarized in 4.2.2.2 and that determined as summarized in 4.2.3 or between the nitrogen determined as summarized in 4.2.2.2 and the sum of that determined as summarized in 4.2.5 + 4.2.6 + 4.2.7.

4.2.5 Ammoniacal nitrogen

4.2.5.1 Solely in the presence of ammoniacal nitrogen and ammoniacal plus nitrate nitrogen, according to EN 15475.

4.2.5.2 In the presence of urea nitrogen and/or cyanamide nitrogen by cold distillation after making slightly alkaline, the ammonia is absorbed in a standard solution of sulfuric acid and determined according to EN 15475.

4.2.6 Urea nitrogen

4.2.6.1 By conversion using urease, into ammonia which is titrated with a standard solution of hydrochloric acid.

or

4.2.6.2 By gravimetry with xanthidrol: the co-precipitated biuret can be counted with urea nitrogen without great error, its content remaining generally low in absolute value in compound fertilizers.

or

4.2.6.3 By difference according to Table 1.

Table 1 — Determination of urea nitrogen by difference

Case	Nitrate nitrogen	Ammoniacal nitrogen	Cyanamidic nitrogen	Difference
1	Absent	Present	Present	$(4.2.2.1) - (4.2.5.2 + 4.2.7)$
2	Present	Present	Present	$(4.2.3) - (4.2.5.2 + 4.2.7)$
3	Absent	Present	Absent	$(4.2.2.1) - (4.2.5.2)$
4	Present	Present	Absent	$(4.2.3) - (4.2.5.2)$

4.2.7 Cyanamide nitrogen

By precipitation as a silver compound, the nitrogen being determined in the precipitate by the Kjeldahl method.

5 Reagents

5.1 General

Use only reagents of recognized analytical grade and distilled or de-mineralized water of grade 3 according to EN ISO 3696:1995.

5.2 Potassium sulfate

p.a.

5.3 Iron powder, reduced with hydrogen.

The prescribed quantity of iron shall be able to reduce at least 50 mg of nitrate nitrogen.

5.4 Potassium thiocyanate, p.a.

5.5 Potassium nitrate, p.a.

5.6 Ammonium sulfate, p.a.

5.7 Urea, p.a.

5.8 Diluted sulfuric acid

Dilute one volume of sulfuric acid ($\rho_{20} = 1,84$ g/ml) in one volume of water.

5.9 Standard solution of sulfuric acid, $c = 0,1$ mol/l.

5.10 Sodium hydroxide solution, aqueous solution of about 30 % (mass concentration), free from ammonia.

5.11 Standard solution of sodium or potassium hydroxide, $c = 0,2$ mol/l, free from carbonates.