INTERNATIONAL STANDARD

ISO 15604

First edition
2016-05-01

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

Reference number
ISO 15604:2016(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

ISO 15604 was prepared by CEN/TC 260 as EN 15604:2009 and was adopted (without modification other than those stipulated below) by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*.

Modifications were made as follows:

a) 5.2: p.a. = pro analysis = analytical grade;

b) 6.2: add "Refer to Figure 1".

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

1 Scope

This International 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 the Regulation (EC) No 2003/2003, Annex I[2] containing nitrogen in various forms.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 14820-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

ISO 25475, *Fertilizers — Determination of ammoniacal nitrogen*

EN 12944-1, *Fertilizers and liming materials — Vocabulary — Part 1: General terms*

EN 12944-2, *Fertilizers and liming materials — Vocabulary — Part 2: Terms relating to fertilizers*

EN 15562, *Fertilizers — Determination of cyanamide nitrogen*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1 and EN 12944-2 apply.

4 Principle

4.1 Total soluble and insoluble nitrogen

According to the list of standard fertilizers given in Regulation (EC) No 2003/2003, Annex I,[2] 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 ISO 25475.

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 Regulation (EC) No 2003/2003, Annex I.[2]

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 ISO 25475).

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 ISO 25475).

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 ISO 25475).

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 (see 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 and 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 ISO 25475.

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 ISO 25475.

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 xanthydrol: 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 ISO 3696.

5.2 Potassium sulfate, p.a. (p.a. = pro analysis = analytical grade).

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 Sulfuric acid diluted.

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

5.9 Sulfuric acid, standard solution, c = 0,1 mol/l.

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

5.11 Sodium or potassium hydroxide, standard solution, c = 0,2 mol/l, free from carbonates.

5.12 Stannous chloride solution.

Dissolve 120 g of $SnCl_2 \cdot 2H_2O$ in 400 ml of concentrated hydrochloric acid (ρ_{20} = 1,18 g/ml) and make up to 1 l with water. The solution shall be perfectly clear and prepared immediately before use.

It is essential to check the reducing power of stannous chloride: dissolve 0,5 g of $SnCl_2 \cdot 2H_2O$ in 2 ml of concentrated hydrochloric acid (ρ_{20} = 1,18 g/ml) and make up to 50 ml with water. Then, add 5 g