# INTERNATIONAL STANDARD



First edition 2016-04-15

# Fertilizers — Determination of ammoniacal nitrogen

Engrais — Détermination de l'azote ammoniacal

# iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO 25475:2016 https://standards.iteh.ai/catalog/standards/sist/c721b165-3a87-4c36-8e3b-1151300a0ed7/iso-25475-2016



Reference number ISO 25475:2016(E)

### iTeh STANDARD PREVIEW (standards.iteh.ai)

<u>ISO 25475:2016</u> https://standards.iteh.ai/catalog/standards/sist/c721b165-3a87-4c36-8e3b-1151300a0ed7/iso-25475-2016



© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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

Page

### Contents

Fore	eword	iv
1	Scope	
2	Normative references	
3	Terms and definitions	
4	Principle	
5	Reagents	
6	Apparatus	
7	Sampling and sample preparation	
8	Procedure8.1Preparation of the solution8.2Analysis of the solution8.3Blank8.4Control test	
9	-	
10	Precision 10.1 Inter-laboratory test	
	10.2 Repeatability	
11	Test report (standards.iteh.ai)	
Ann	nex A (informative) Results of the inter-laboratory tests	
Bibl	ISO 25475:2016 https://standards.iteh.ai/catalog/standards/sist/c721b165-3a87-4c36-8e3b 1151300a0ed7/iso-25475-2016	

### 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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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 <a href="https://www.iso.org/patents">www.iso.org/patents</a>).

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: <a href="http://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

ISO 25475 was prepared by CEN/TC 260 as EN 15475:2009 and was adopted (without modification other than those stipulated below) by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*.

Modifications were made in the following subclauses dards/sist/c721b165-3a87-4c36-8e3b-

- 1151300a0ed7/iso-25475-2016
- a) General: The references to EN 1482-1 and -2 were changed to ISO 14820-1 and -2;
- b) <u>5.9</u>: added "mass fraction";
- c) <u>5.12</u>: p.a. = pro analysis = analytical grade;
- d) <u>6.1</u>: First two sentences joined together.
- e) <u>6.4</u>: "35 to 40 revolutions per minute" was changed to "35 r/min to 40 r/min";
- f) 8.1: "Filter through a filter into a 500 ml graduated flask and make up to the volume with water" was changed to "Pass the solution through a filter into a 500 ml graduated flask and make up to the volume with water";
- g) 8.2, Table 3: "F = factor comprising the amount weighed, the dilution, the aliquot part of solution of the sample to be distilled and the volumetric equivalent. expressed as percentage of ammoniacal nitrogen in the fertilizer" was changed to "F = factor comprising the amount weighed, the dilution, the aliquot part of the solution of the sample to be distilled and the volumetric equivalent".
- h) <u>10.1</u> Line 1: "Test was carried out in 2004 with 24, respectively 22 participating labs" was changed to "A test was carried out in 2004 with 24 participating labs";
- i) Bibliography: Reference [3] was removed.

### Fertilizers — Determination of ammoniacal nitrogen

#### 1 Scope

This International Standard specifies a method for the determination of the ammoniacal nitrogen content in fertilizers. The method is applicable to all nitrogenous fertilizers including compound fertilizers, in which nitrogen is found exclusively either in the form of ammonium salts or ammonium salts together with nitrates.

This International Standard is not applicable to fertilizers containing urea, cyanamide or other organic nitrogenous compounds.

#### 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

(standards.iteh.ai) EN 12944-1, Fertilizers and liming materials — Vocabulary — Part 1: General terms

EN 12944-2, Fertilizers and liming materials 250752014ary — Part 2: Terms relating to fertilizers https://standards.iteh.ai/catalog/standards/sist/c721b165-3a87-4c36-8e3b-1151300a0ed7/iso-25475-2016

#### 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

Displacement of ammonia by means of an excess of sodium hydroxide, distillation and determining the yield of ammonia in a given volume of a standard sulfuric acid and titration of the excess 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 ISO 3696).

- **5.2** Hydrochloric acid, diluted. Mix one volume of  $\rho$ (HCl) = 1,18 g/ml with one volume of water.
- **5.3 Sulfuric acid** (for variant a), *c* = 0,05 mol/l.
- **5.4 Sodium or potassium hydroxide solution** (for variant a), carbonate free, *c* = 0,1 mol/l.
- **5.5** Sulfuric acid (for variant b, see NOTE in 8.2), c = 0.1 mol/l.

**5.6** Sodium or potassium hydroxide solution (for variant b, see NOTE in <u>8.2</u>), carbonate free, c = 0.2 mol/l.

**5.7** Sulfuric acid (for variant c, see NOTE in 8.2), c = 0.25 mol/l.

**5.8** Sodium or potassium hydroxide solution (for variant c, see NOTE in 8.2), carbonate free, c = 0.5 mol/l.

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

#### 5.10 Indicator solutions.

#### 5.10.1 Mixed indicator.

Solution A: Dissolve 1 g of methyl red in 37 ml of sodium hydroxide solution (5.4) 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 A with two volumes of 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.

# 5.10.2 Methyl red indicator solution.

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 (four to five drops) instead of the preceding one. This indicator is red in acid solution and yellow in alkaline solution. 25475:2016 https://standards.iteh.av/catalog/standards/sist/c721b165-3a87-4c36-8e3b-

1151200202d7/220202d7/202010

**5.11** Anti-bump granules (i. e. pumice stone, glass pearls), washed in hydrochloric acid and calcined.

**5.12 Ammonium sulfate**, p. a. (p.a. = pro analysis = analytical grade).

#### 6 Apparatus

#### 6.1 Distillation apparatus

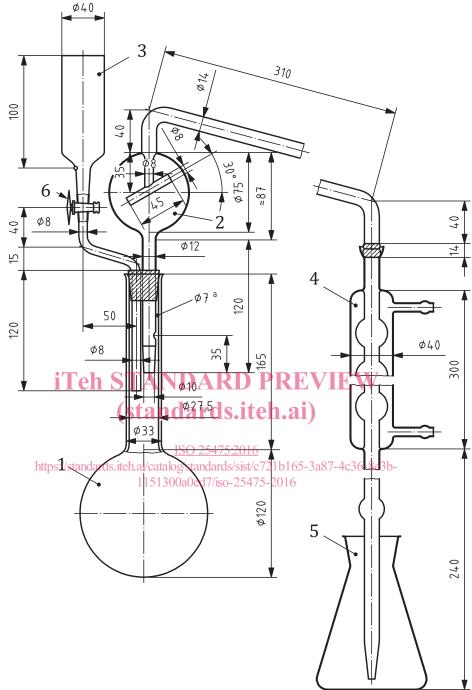
The equipment is made of borosilicate glass and consists of a round-bottomed flask of suitable capacity connected to a condenser by means of a splash head.

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.

#### ISO 25475:2016(E)

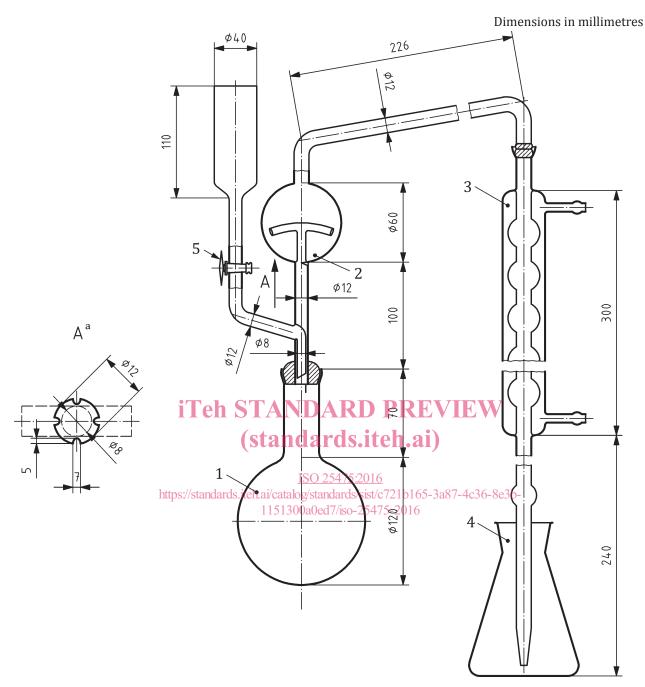
Dimensions in millimetres



#### Key

- 1 round-bottomed, long-necked flask of 1 000 ml capacity
- 2 distillation tube with a 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) for the addition of sodium hydroxide
- 4 six-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 (the tap may likewise be replaced by a rubber connection with a clip)

#### Figure 1 — Distillation apparatus 1



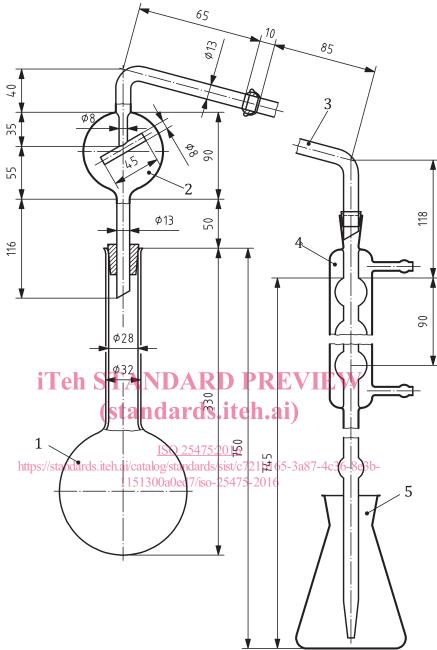
#### Key

- 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

#### ISO 25475:2016(E)

Dimensions in millimetres



#### Key

- 1 round-bottomed, long-necked flask of 750 ml or 1 000 ml capacity with a bell mouth
- 2 distillation tube with a splash head and a spherical joint (No 18) at the issue
- 3 elbow tube with a spherical joint (No 18) at the entrance and a drip cone (the connection to the distillation tube may be effected by means of a rubber tube instead of a spherical joint)
- 4 six-bulb condenser joined at the issue to a glass extension tube by means of a small rubber connection
- 5 500 ml flask in which the distillate is collected

#### Figure 3 — Distillation apparatus 3