INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ •ORGANISATION INTERNATIONALE DE NORMALISATION

# Urea for industrial use — Determination of nitrogen content — Titrimetric method after distillation

Urée à usage industriel – Dosage de l'azote – Méthode titrimétrique après distillation

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#### **FOREWORD**

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1592-1970 and found it technically suitable for transformation. International Standard ISO 1592 therefore replaces ISO Recommendation R 1592-1970, to which it is technically identical.

ISO Recommendation R 1592 had been approved by the member bodies of the following countries:

Australia Hungary Austria India Belgium Iran Brazil Israel Canada Italy Netherlands Czechoslovakia Egypt, Arab Rep. of New Zealand France Peru Germany Poland

Romania South Africa, Rep. of

Spain Sweden Switzerland Thailand Turkey U.S.S.R. Yugoslavia

The member body of the following country had expressed disapproval of the Recommendation on technical grounds:

Portugal

## United Kingdom

The member body of the United Kingdom also disapproved the transformation of the Recommendation into an International Standard.

Greece

## Urea for industrial use — Determination of nitrogen content — Titrimetric method after distillation

#### 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method, after distillation, for the determination of the nitrogen content of urea for industrial use.

#### 2 PRINCIPLE

Catalytic conversion of the nitrogen present in a test portion to ammonia by heating in concentrated sulphuric acid solution. Distillation and absorption of the ammonia in an excess of standard volumetric sulphuric acid solution and back-titration with standard volumetric sodium hydroxide solution in the presence of an indicator.

#### 3 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

- 3.1 Copper(II) sulphate pentahydrate (CuSO<sub>4</sub>.5H<sub>2</sub>O).
- **3.2 Sulphuric acid**,  $\rho$  approximately 1,84 g/ml, about 96 % (m/m) solution or approximately 36 N.
- **3.3 Sodium hydroxide**, 450 g/l solution.
- **3.4** Sulphuric acid, 0,5 N standard volumetric solution.
- **3.5** Sodium hydroxide, 0,5 N standard volumetric solution.
- 3.6 Mixed indicator, ethanolic solution.

Dissolve 0,1 g of methyl red in about 50 ml of 95 % (V/V) ethanol and add 0,05 g of methylene blue. After dissolution, dilute to 100 ml with the same ethanol and mix.

#### 4 APPARATUS

Ordinary laboratory apparatus and

- **4.1 Kjeldahl flask**, capacity 500 ml, fitted with a pear-shaped stopper.
- **4.2 Distillation apparatus,** with, preferably, spherical ground glass joints, or any apparatus that will ensure quantitative distillation and absorption.

The apparatus may, for example, be made up from the following items (see figure):

- **4.2.1** *Distillation flask* (A), capacity 1 000 ml, with female joint.
- **4.2.2** Splash head (B), with male joints and parallel inlet and outlet into which is fused a cylindrical dropping-funnel (C), capacity 50 ml.
- 4.2.3 Liebig condenser (D), effective length about 400 mm, fitted with a female joint at the inlet and a male joint at the outlet.
- **4.2.4** *Conical flask* (E), capacity 500 ml, with female joint, fitted with two side bulbs.
- 4.2.5 Spring clamps (F).

### 5 PROCEDURE 1

#### 5.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the test sample and transfer to the Kjeldahl flask (4.1).

#### 5.2 Blank test

Carry out a blank test at the same time as the determination, following the same procedure and using the same reagents as used during the determination, but omitting the test portion.

<sup>1)</sup> The procedure is described in terms of the apparatus specified in 4.2 and will require modification if other apparatus is used.