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INTERNATIONAL STANDARD ISO 1592

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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	Romania
Austria	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Israel	Sweden
Canada	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	Turkey
France	Peru	U.S.S.R.
Germany	Poland	Yugoslavia
Greece	Portugal	

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

United Kingdom

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

Printed in Switzerland

INTERNATIONAL STANDARD ISO 1592-1977 (E)

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 ($CuSO_4.5H_2O$).

3.2 Sulphuric acid, ρ 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.

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

5.3 Determination

5.3.1 *Preparation of test solution*

Add to the Kjeldahl flask (4.1), containing the test portion (5.1), 25 ml of water, 50 ml of the sulphuric acid solution (3.2) and 0,75 g of the copper sulphate (3.1). Close the Kjeldahl flask with its pear-shaped stopper and warm gently until all the carbon dioxide has been driven off. Steadily increase the heating until evolution of white fumes and then continue heating for a further 20 min. Allow to cool and carefully add 300 ml of water, cooling and stirring during the addition.

Quantitatively transfer the solution to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

5.3.2 *Distillation*

Place 50,0 ml of the test solution (5.3.1) in the distillation flask (A). Add about 300 ml of water, a few drops of the mixed indicator solution (3.6) and a few anti-bumping granules.

Coat the joints of the apparatus with a silicone grease. Mount the splash head (B) on the flask (A) and connect it to the condenser (D).

Place 40,0 ml of the standard volumetric sulphuric acid solution (3.4), about 80 ml of water and a few drops of the mixed indicator solution (3.6) into the flask (E). Connect the flask (E) to the condenser (D), ensuring that all the joints of the apparatus are firm, by means of the spring clamps (F) in the case of spherical joints.

Into the flask (A), introduce sufficient of the sodium hydroxide solution (3.3) to neutralize the solution and then 25 ml in excess, through the dropping-funnel (C), taking care to leave at least a few millimetres of liquid above the tap.

Distil until a volume of about 250 to 300 ml has collected in the flask (E). Stop the heating, open the tap of the dropping funnel (C), disconnect the splash head (B) and wash the condenser (D) carefully, collecting the wash water in the flask (E). Finally disconnect the flask (E).

5.3.3 *Titration*

Carefully mix the solution contained in the flask (E) and in the two side bulbs, and back-titrate the excess of the standard volumetric sulphuric acid solution (3.4) with the standard volumetric sodium hydroxide solution (3.5), to the colour change of the indicator.

During the titration, stir carefully to ensure that the solution is completely mixed.

6 EXPRESSION OF RESULTS

The nitrogen content, expressed as a percentage by mass of nitrogen (N), is given by the formula

$$(V_1 - V_2) \times 0{,}007\,004 \times \frac{500}{50} \times \frac{100}{m}$$

$$= \frac{7{,}004\,(V_1 - V_2)}{m}$$

where

V_1 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.5) used for the back-titration of the excess of the standard volumetric sulphuric acid solution (3.4) placed in the flask (E) for the blank test;

V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.5) used for the back-titration of the excess of the standard volumetric sulphuric acid solution (3.4) placed in the flask (E) for the determination;

m is the mass, in grams, of the test portion (5.1);

0,007 004 is the mass, in grams, of nitrogen corresponding to 1 ml of exactly 0,5 N sulphuric acid solution.

NOTE – If the concentrations of the standard volumetric solutions are not exactly as specified in the list of reagents, appropriate corrections should be made.

7 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.

Dimensions in millimetres

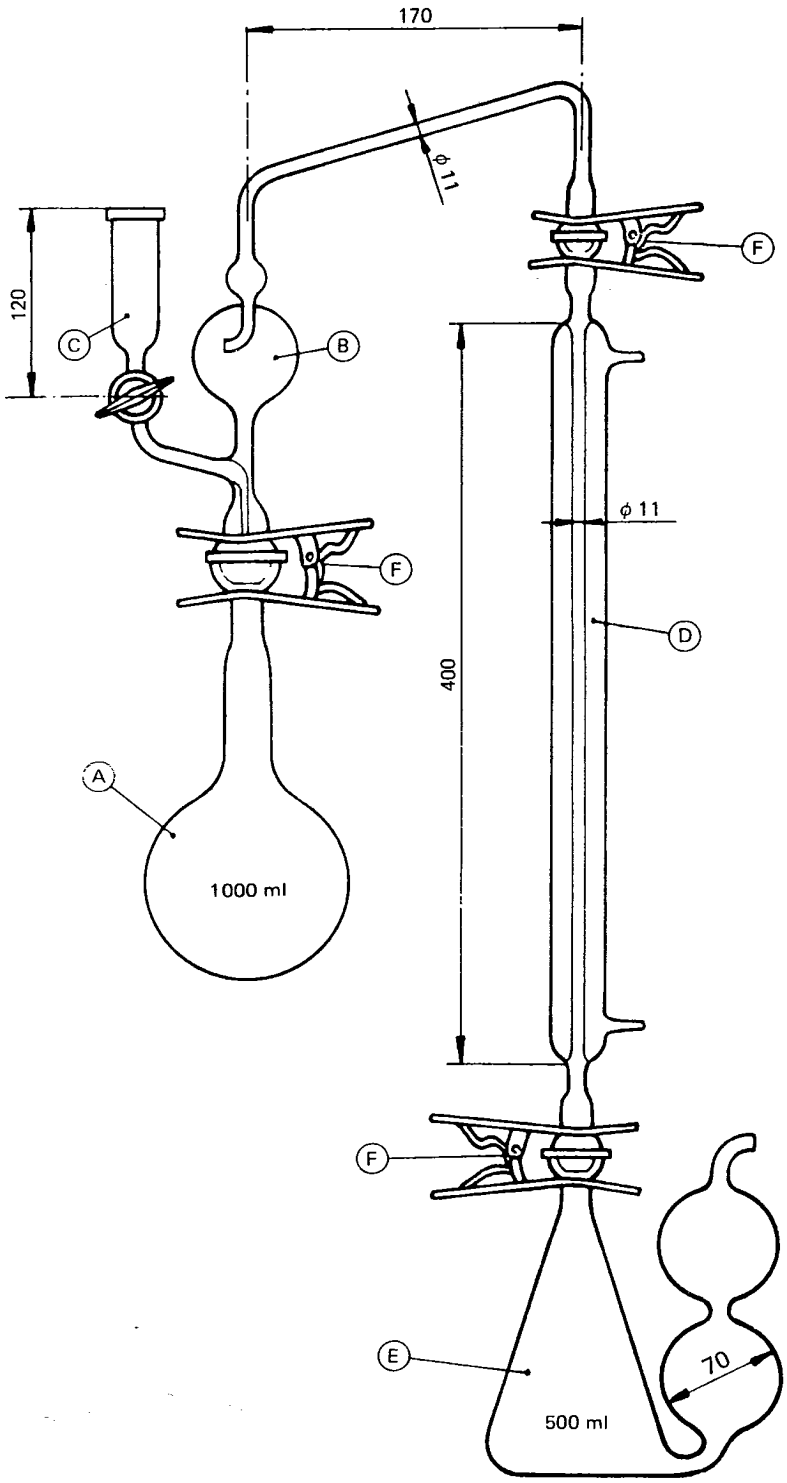

FIGURE — **Typical distillation apparatus (4.2)**

ANNEX

ISO PUBLICATIONS RELATING TO UREA FOR INDUSTRIAL USE

ISO 1592 – Determination of nitrogen content – Titrimetric method after distillation.

ISO 1593 – Determination of alkalinity – Titrimetric method.

ISO 1594 – Determination of ash.

ISO/R 1595 – Determination of iron content – 2,2′-Bipyridyl photometric method.

ISO 2749 – Measurement of the pH of a solution of urea of conventional concentration (100 g/l) – Potentiometric method.

ISO 2750 – Measurement of colour in Hazen units (platinum-cobalt scale) of a urea-formaldehyde solution.

ISO 2751 – Determination of the buffer coefficient – Potentiometric method.

ISO 2752 – Measurement of the variation of pH in the presence of formaldehyde – Potentiometric method.

ISO 2753 – Determination of water content – Karl Fischer method.

ISO 2754 – Determination of biuret content – Photometric method.

ISO 4274 – Determination of biuret content – Flame atomic absorption and photometric absorption methods.