UDC 661.717.5 : 543.24 : 546.17

Ref. No. : ISO/R 1592-1970 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1592

UREA FOR INDUSTRIAL USE

DETERMINATION OF NITROGEN

VOLUMETRIC METHOD

1st EDITION

July 1970

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 1592, *Urea for industrial use – Determination of nitrogen – Volumetric method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1592 which was circulated to all the ISO Member Bodies for enquiry in February 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Body opposed the approval of the Draft :

United Kingdom

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 1592 July 1970

UREA FOR INDUSTRIAL USE

DETERMINATION OF NITROGEN

VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of nitrogen in urea for industrial use.

2. PRINCIPLE

Catalytic conversion of the nitrogen to ammoniacal nitrogen by heating in concentrated sulphuric acid solution. Distillation of the ammonia in the presence of alkali; its absorption in an excess of standard sulphuric acid and back titration with standard sodium hydroxide solution using an indicator.

3. REAGENTS

Distilled water or water of equal purity should be used in the test.

3.1 *Copper (II) sulphate,* crystals ($CuSO_4.5H_2O$).

3.2 *Sulphuric acid,* approximately ρ 1.84 (g/ml), 96 % (m/m) or 36 N solution.

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. When this has dissolved, dilute to 100 ml with the same ethanol.

4. APPARATUS

Ordinary laboratory apparatus and

4.1 *Kjeldahl flask,* 500 ml, fitted with a pear-shaped stopper.

4.2 *Distillation apparatus with ground glass joints,* preferably spherical, or any other form of apparatus, that ensures quantitative distillation and absorption. For example the apparatus consists of (see Figure) :

Distillation flask (A),1000 ml, with a ground glass socket.

Bent distillation tube (B),fitted with a splash head, and connected to the dropping funnel (C).

Dropping funnel (C), cylindrical, 50 ml capacity, with a tap and ground glass cone.

Condenser, Liebig (D), useful length about 40 cm.

Flask (E), conical, 500 ml, with a ground glass socket, and two side bulbs.

Spring clips (F).

5. PROCEDURE

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 at the same time, and using the same procedure, a blank test with the same quantity of all the reagents used for the determination.

5.3 Determination

5.3.1 *Preparation of the sample 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 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 minutes. 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.* Transfer 50.0 ml of the sample solution (5.3.1) to the distillation flask (A). Add about 300 ml of water, several drops of the mixed indicator solution (3.6) and several small pieces of pumice. Smear the joints of the apparatus with silicone grease. Fit the distillation tube (B) to the flask (A) and connect it to condenser (D).

Transfer to flask (E), 40.0 ml of the sulphuric acid solution (3.4), about 80 ml of water and several drops of the mixed indicator solution (3.6). Attach the flask (E) to condenser (D), ensuring that all the joints are gas-tight, using spring clips (F) for apparatus containing spherical joints.

Through the dropping funnel (C), add to the flask (A) sufficient sodium hydroxide solution (3.3) to neutralize the solution and then add 25 ml in excess.

When using the apparatus described, distil over into flask (E) a volume of about 250 to 300 ml. Cease heating, disconnect the delivery tube (B) and carefully wash condenser (D), collecting the washings in flask (E). Finally disconnect flask (E).

5.3.3 *Titration.* Thoroughly mix the solution in the flask and its two side bulbs and back titrate the excess sulphuric acid solution with the sodium hydroxide solution (3.5) to the colour change of the indicator.

During titration keep the solution thoroughly mixed by stirring.