INTERNATIONAL STANDARD ISO 2515

G-97-21

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

Ammonium hydrogen carbonate for industrial use (including foodstuffs) — Determination of ammoniacal nitrogen content — Volumetric method after distillation

First edition — 1973-04-15

UDC 661.523 : 546.171 : 543.24 Ref. No. ISO 2515-1973 (E)

Descriptors : ammonium compounds, carbonates, chemical analysis, determination of content, nitrogen, volumetric analysis.

Price based on 3 pages

FOREWORD

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International Standard ISO 2515 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in August 1971.

It has been approved by the Member Bodies of the following countries :

Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Chile	Israel	Switzerland
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Germany	Portugal	
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No Member Body expressed disapproval of the document.

Printed in Switzerland

INTERNATIONAL STANDARD ISO 2515-1973 (E)

Ammonium hydrogen carbonate for industrial use (including foodstuffs) — Determination of ammoniacal nitrogen content — Volumetric method after distillation

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a volumetric method for the determination of the ammoniacal nitrogen content of ammonium hydrogen carbonate for industrial use (including foodstuffs).

2 PRINCIPLE

Distillation of the ammonia after displacement by an alkaline solution; absorption in an excess of standard volumetric sulphuric acid solution and back titration with standard volumetric sodium hydroxide solution in the presence of an indicator.

NOTE — The absorption of the ammonia may also be carried out in an excess of boric acid solution, and the titration of the ammonium ions, in this case, carried out directly with a standard volumetric sulphuric acid solution (see section 7).

3 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

3.1 Sodium hydroxide, 450 g/l solution.

3.2 Sulphuric acid, 0,5 N standard volumetric solution.

3.3 Sodium hydroxide, 0,5 N standard volumetric solution.

3.4 Mixed indicator, ethanolic solution.

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

4 APPARATUS

Ordinary laboratory apparatus and

4.1 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) :

4.1.1 *Distillation flask* (A), capacity 1 000 ml, with female joint;

4.1.2 *Splash head* (B), with parallel inlet and outlet into which 4.1.3 is fused;

4.1.3 *Cylindrical tap funnel* (C), capacity 50 ml, with male joint;

4.1.4 *Liebieg condenser* (D), effective length about 400 mm;

4.1.5 *Conical flask* (E), capacity 500 ml, with female joint, fitted with two side bulbs;

4.1.6 *Spring clips* (F).

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,001 g, about 10 g of the test sample.

5.2 Blank test

Carry out a blank test in parallel with, and following the same procedure as, the determination using the same quantities of all the reagents.

5.3 Determination

5.3.1 *Preparation of the test solution*

Place the test portion (5.1) in a 500 ml one-mark volumetric flask, dissolve in water, dilute to the mark and mix.

5.3.2 *Distillation*

Place in the distillation flask (A) 50,0 ml of the test solution (5.3.1). Add about 300 ml of water, a few drops of the mixed indicator solution (3.4) and a few small pieces of pumice.

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).

Introduce into the flask (E) 40,0 ml of the standard volumetric sulphuric acid solution (3.2), about 80 ml of water and a few drops of the mixed indicator solution (3.4). Connect the flask (E) to the condenser (D), ensuring that all the joints of the apparatus are firm (by means of the spring clips (F) in the case of spherical joints).

Through the funnel (C), introduce into the flask (A) the quantity of the sodium hydroxide solution (3.1) necessary for neutralization, and then add an excess of 25 ml of this solution.

If the apparatus described is used, distil until a volume of about 250 to 300 ml is left in the flask (E). Stop the heating, 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 by means of the standard volumetric sodium hydroxide solution (3.3).

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

6 EXPRESSION OF RESULTS

The ammoniacal nitrogen content, expressed as ammonia (NH_3) is given, as a percentage by mass, by the formula :

$$\frac{[(V_1 - V_2) - (V_3 - V_4)] \times 0{,}008\,5 \times 10 \times 100}{m} =$$

$$\frac{8{,}5\,[(V_1 - V_2) - (V_3 - V_4)]}{m}$$

where

V_1 is the volume, in millilitres, of the standard volumetric sulphuric acid solution (3.2) placed in the flask (E) for the determination;

V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.3) used for the back titration of the excess of the standard volumetric sulphuric acid solution (3.2) placed in the flask (E) for the determination;

V_3 is the volume, in millilitres, of the standard volumetric sulphuric acid solution (3.2) placed in the flask (E) for the blank test;

V_4 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.3) used for the back titration of the excess of the standard volumetric sulphuric acid solution (3.2) placed in the flask (E) for the blank test;

m is the mass, in grams, of the test portion;

0,008 5 is the mass, in grams, of ammonia corresponding to 1 ml of 0,5 N sulphuric acid solution.

7 NOTE ON PROCEDURE

It is possible to carry out a direct determination by replacing the 40 ml of standard volumetric sulphuric acid solution (3.2) in the flask (E), by 40 ml of a 50 g/l boric acid solution neutralized by a few drops of approximately 0,1 N sodium hydroxide solution in the presence of the mixed indicator (3.4). The titration of the ammonium ions is then carried out directly using the standard volumetric sulphuric acid solution (3.2).

8 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.

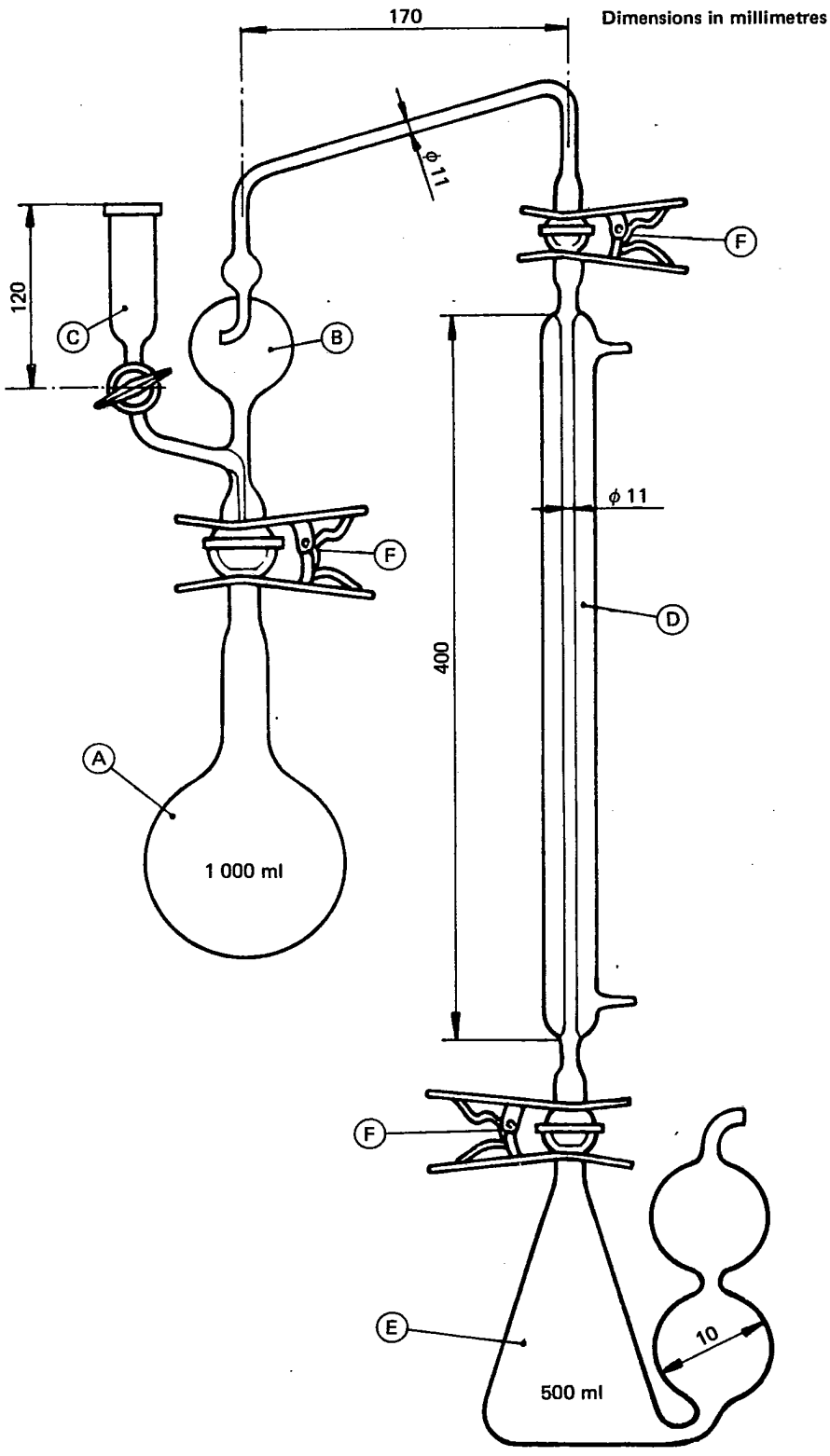

FIGURE — **Distillation apparatus (4.1)**