
INTERNATIONAL STANDARD



3422

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

Ammonium hydrogen carbonate for industrial use (including foodstuffs) – Determination of total carbon dioxide content – Titrimetric method

Bicarbonate d'ammonium à usage industriel (y compris les industries alimentaires) – Dosage du dioxyde de carbone total – Méthode titrimétrique

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Descriptors : ammonium compounds, chemical analysis, determination of content, carbon dioxide, volumetric analysis.

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.

International Standard ISO 3422 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in March 1974.

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

Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Bulgaria	Israel	Switzerland
Chile	Italy	Thailand
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia
Hungary	Romania	

No Member Body expressed disapproval of the document.

Ammonium hydrogen carbonate for industrial use (including foodstuffs) – Determination of total carbon dioxide content – Titrimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a titrimetric method for the determination of the total carbon dioxide content (bicarbonate, carbonate and carbamate) of ammonium hydrogen carbonate for industrial use (including foodstuffs).

2 PRINCIPLE

Treatment of a test portion with an excess of carbonate-free sodium hydroxide solution. Elimination of ammonia by boiling. Precipitation of carbon dioxide by an excess of barium chloride. Neutralization with hydrochloric acid solution, using thymolphthalein as indicator. Addition of an excess of standard volumetric hydrochloric acid solution and back-titration with standard volumetric sodium hydroxide solution, using methyl red as indicator.

3 REAGENTS

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity, freshly boiled, cooled and neutral to methyl red.

3.1 Barium chloride dihydrate, 100 g/l solution.

3.2 Sodium hydroxide, carbonate-free solution.

Dissolve 5 g of barium chloride dihydrate in a little water, add 200 ml of sodium hydroxide, approximately 400 g/l solution, dilute to 1 000 ml and mix.

Allow to stand for 48 h. Use for the analysis only that part of the solution which is perfectly clear.

3.3 Hydrochloric acid, 0,5 N standard volumetric solution.

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

3.5 Methyl red, 1 g/l ethanolic solution.

Dissolve 0,10 g of methyl red in 95 % (V/V) ethanol and dilute to 100 ml with the same ethanol.

3.6 Thymolphthalein, 1 g/l ethanolic solution.

Dissolve 0,10 g of thymolphthalein in 95 % (V/V) ethanol and dilute to 100 ml with the same ethanol.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Conical flasks, capacity 250 ml, with ground necks and fitted with male joints with bent adapters.

4.2 Guard device (column or U-tube), filled with soda asbestos or soda lime.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,001 g, about 5 g of the test sample.

5.2 Determination

5.2.1 Preparation of the test solution

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

5.2.2 Titration

Transfer 50,0 ml of the test solution (5.2.1) to one of the open conical flasks (4.1).

Add 10 ml of the carbonate-free sodium hydroxide solution (3.2) and some glass beads, and boil for 15 min. Fit the adapter (4.1), connect with the guard device (4.2) and cool to room temperature. Add 25 ml of the barium chloride solution (3.1) and 0,2 ml of the thymolphthalein solution (3.6) to the flask and neutralize with the standard volumetric hydrochloric acid solution (3.3) until the solution is decolorized. Add 0,2 ml of the methyl red solution (3.5) and 50,0 ml of the standard volumetric hydrochloric acid solution (3.3), running the acid down the inner wall of the flask in order to remove any precipitate adhering to it. Boil for 5 min and back-titrate the cooled solution, using the standard volumetric sodium hydroxide solution (3.4), until the yellow end-point of the indicator is reached.

6 EXPRESSION OF RESULTS

The total carbon dioxide content, expressed as a percentage by mass of CO₂, is given by the formula

$$\frac{(50 - V) \times 0,011 \times 10 \times 100}{m} = \frac{11 (50 - V)}{m}$$

where

V is the volume, in millilitres, of the standard volumetric sodium hydroxide solution (3.4) used for the back-titration of the excess of the standard volumetric hydrochloric acid solution (3.3);

0,011 is the mass, in grams, of carbon dioxide corresponding to 1 ml of an exactly 0,5 N standard volumetric hydrochloric acid solution;

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

NOTE — If the concentrations of the standard volumetric solutions used are not exactly as specified in the list of reagents, an appropriate correction 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.

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ANNEX

**ISO PUBLICATIONS RELATING TO AMMONIUM HYDROGEN CARBONATE FOR INDUSTRIAL USE
(INCLUDING FOODSTUFFS)**

ISO 2515 – Determination of ammoniacal nitrogen content – Volumetric method after distillation.

ISO 2516 – Determination of total alkalinity – Volumetric method.

ISO 3420 – Determination of ash content – Gravimetric method.

ISO 3421 – Determination of lead content – Dithizone photometric method.

ISO 3422 – Determination of total carbon dioxide content – Titrimetric method.

ISO 4275 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.

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