BRITISH STANDARD

BS 4651-7: 1988

Ammonia solution —

Part 7: Method for determination of carbon dioxide content

NOTE It is recommended that this Part be read in conjunction with the information on methods for sampling in BS 4651-0, published separately.

UDC 546.171.1 – 145.2:543.73:546.264 – 31

Foreword

This Part of BS 4651 has been prepared under the direction of the Chemicals Standards Committee and supersedes clause **7** of BS 4651:1971 which is withdrawn. This method is technically equivalent to that described in clause **7** of BS 4651:1971.

NOTE The term "ammonia solution" is used to describe grades of product containing 25.0 % to 35.0 % (*m*/*m*) of ammonia.

WARNING. Ammonia solution is a moderately strong alkali which exerts a local irritant action on the skin. Strong solutions which come into contact with the eyes, even for a short period, can cause serious and permanent damage.

Ingestion of ammonia solution will result in the destruction of the mucous lining of the mouth, throat and stomach.

Ammonia vapour is readily released from ammonia solution and is combustible in air between the concentrations of 16 % and 27 % (*V*/*V*) and may explode in confined spaces.

When sampling ammonia solution, take the precautions described in BS 4651-0.

This Part of BS 4651 describes a method of test only and should not be used or quoted as a specification defining limits of purity. Reference to this Part should indicate that the method of test used complies with BS 4651-7:1988.

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This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 August 1988

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The committees responsible for this British Standard are shown in Part 0.
The following BSI references relate to the work on this standard:
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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

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1 Scope

This Part of BS 4651 describes a titrimetric method for the determination of the carbon dioxide content of ammonia solution for industrial use.

The method is applicable to solutions containing 25.0 % to 35.0 % (*m*/*m*) of ammonia and with a carbon dioxide content of not lower than 10 mg/kg.

NOTE The publications referred to in this standard are listed on the inside back cover.

2 Principle

The carbon dioxide present is precipitated as barium carbonate and the filtered precipitate is washed free from alkali and dissolved in a known excess of standard acid, which is back-titrated with standard sodium hydroxide solution.

3 Reagents

3.1 *General.* During the analysis, use only reagents of recognized analytical reagent grade and water complying with grade 3 of BS 3978, freshly boiled to be free from carbon dioxide.

3.2 *Barium chloride solution,* 100 g/L.

3.3 *Sodium hydroxide solution, c*(NaOH) = 40 g/L, approximately.

3.4 *Sodium hydroxide,* standard volumetric solution, *c*(NaOH) = 0.100 mol/L.

3.5 *Hydrochloric acid,* standard volumetric solution, *c*(HCl) = 0.100 mol/L.

3.6 *Screened methyl orange indicator solution,* 1 g/L. Dissolve 0.25 g of methyl orange and 0.15 g of xylene cyanol FF in 50 mL of 95 % (*V*/*V*) ethanol and dilute to 250 mL with water.

3.7 *Phenolphthalein indicator solution,* 10 g/L in 95 % (*V*/*V*) ethanol.

NOTE For the purposes of **3.6** and **3.7**, ethanol may be replaced by industrial methylated spirits 95 % (*V*/*V*) complying with BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquors Duties Act 1972, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative.

4 Apparatus

4.1 *General.* Ordinary laboratory apparatus and the following.

4.2 *One-mark volumetric flask,* 100 mL, complying with BS 1792.

4.3 *Carbon-dioxide absorber guard tube*

5 Procedure

5.1 Test portion

Fill the one-mark volumetric flask (**4.2**) to the mark with the test sample.

5.2 Determination

Transfer the test portion (**5.1**) to a clean, dry, 250 mL conical flask. Add 10 mL of the barium chloride solution (**3.2**) and 5 mL of the sodium hydroxide solution (**3.3**).

Boil gently until all trace of ammonia odour is absent and close the flask with a stopper fitted with the guard tube (**4.3**).

Allow the flask to cool. Remove the guard tube (**4.3**) and add a few drops of the phenolphthalein indicator solution (**3.7**). Partially neutralize the excess sodium hydroxide by carefully adding 45.0 mL of the hydrochloric acid solution (**3.5**). Filter the contents of the flask through a small, fine, acid-washed filter paper. Rinse the flask well with carbon dioxide-free water, passing the washings through the filter paper. Wash the filter thoroughly until 10 mL to 20 mL of the filtrate are shown to be free from sodium hydroxide when tested with the phenolphthalein indicator solution (**3.7**).

Place the filter paper and contents in the original flask together with 50 mL of water previously neutralized to the screened methyl orange indicator solution (**3.6**). Add, from a burette, a portion of the hydrochloric acid solution (**3.5**), sufficient just to dissolve the barium carbonate and to give a slight excess. Note the volume added to the nearest 0.1 mL. Warm gently for 10 min, ensuring that any precipitate adhering to the sides of the flask is dissolved. Cool, add 0.2 mL of the screened methyl orange indicator solution (**3.6**) and back-titrate the excess acid with the sodium hydroxide standard solution (**3.4**).

5.3 Blank test

Carry out a blank test by following the procedure described in **5.2**, but with 100 mL of the carbon dioxide-free water replacing the sample.

6 Expression of results

The carbonate content, expressed as milligrams of carbon dioxide (CO_2) per kilogram, is given by the following expression.

$$\frac{0.0022 \times [(V_1 - V_2) - (V_3 - V_4)] \times 10^6}{500 \times \rho}$$

where

V_1 is the volume of hydrochloric acid solution (**3.5**) added in the final titration (see **5.2**) (in mL);

V_2 is the volume, of sodium hydroxide solution (**3.4**) used in the back titration (see **5.2**) (in mL);

V_3 is the volume of hydrochloric acid solution (**3.5**) added to the reagent blank in the final titration (see **5.3**) (in mL);

V_4 is the volume of sodium hydroxide solution (**3.4**) added to the reagent blank in the back titration (see **5.3**) (in mL);

500 is the volume of the test portion (**5.1**) (in mL);

ρ is the density of the test sample at 20 °C, determined in accordance with the method described in BS 4651-1 (in g/mL);

0.0022 is the mass of carbon dioxide equivalent to 1 mL of the hydrochloric acid solution (**3.5**) (in g).

7 Test report

The test report shall include the following information:

a) a reference to this British Standard, i.e. BS 4651-7:1988;

b) a complete identification of the sample;

c) details of any unusual features noted during the determination;

d) the results expressed in accordance with clause **6**;

e) any operation not included in this British Standard or regarded as optional.

Publications referred to

BS 1792, *Specification for one-mark volumetric flasks.*
BS 3591, *Specification for industrial methylated spirits.*
BS 3978, *Specification for water for laboratory use.*
BS 4651, *Ammonia solution.*
BS 4651-0, *Methods of sampling.*
BS 4651-1, *Method for determination of density at 20 °C.*

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