BRITISH STANDARD

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Ammonia solution —

Part 6: Methods for determination of chloride 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 – 145.2:543.73:546.131

Foreword

This Part of BS 4651 has been prepared under the direction of the Chemicals Standards Committee and supersedes clause **6** of BS 4651:1971 which is withdrawn. A colorimetric method is given which is technically equivalent to that described in clause **6** of BS 4651:1971, except that the field of application is limited to products with chloride contents of not more than 150 mg/kg. For products with higher chloride contents, a potentiometric method has been included which refers largely to that described in BS 6337-4:1984, possession of which is essential.

NOTE The term "ammonia solution" is used to describe grades of product containing 25.0 % to 35.0 % (*m*/*m*) 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 4651-6:1988.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

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 to 4, 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 two methods for the determination of the chloride content of ammonia solution for industrial use.

The methods are applicable to solutions containing 25.0 % to 35.0 % (*m*/*m*) of ammonia.

Method 1 is a colorimetric method applicable to products having a chloride content of not more than 150 mg/kg.

Method 2 is a potentiometric method applicable to products having a chloride content of greater than 150 mg/kg.

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

2 Method 1. Colorimetric method

2.1 Principle

Reaction of the chloride ions present in a test portion with mercury (II) thiocyanate and ammonium iron (III) sulphate, measurement of the absorbance of the resulting solution at a wavelength within the range 460 nm to 470 nm and determination of the chloride content by means of a calibration graph.

2.2 Reagents

2.2.1 *General.* During the analysis, use only reagents of recognized analytical grade and water complying with grade 3 of BS 3978.

2.2.2 *Mercury (II) thiocyanate solution.* Dissolve approximately 0.1 g of mercury (II) thiocyanate and approximately 2 g of ammonium nitrate in 100 mL of nitric acid solution, *c* (HNO_2) = 6 mol/L approximately, warming gently to aid dissolution. Filter the solution if necessary.

Prepare a fresh solution after one week.

2.2.3 *Ammonium iron (III) sulphate solution.* Dissolve 10.7 g of $(NH_4)_2\,SO_4.Fe_2\,(SO_4)_3.24H_2O$ in 100 mL of nitric acid solution, *c* (HNO_3) = 6 mol/L approximately.

2.2.4 *Standard chloride solution.* Dissolve 0.1649 g of sodium chloride in water in a beaker. Transfer quantitatively to a 500 mL one-mark volumetric flask and dilute to the mark. Using a pipette, transfer 5.0 mL of this solution to a 100 mL one-mark volumetric flask and dilute to the mark. One mL of the second solution contains 10 µg of chloride ions.

2.2.5 *Sodium carbonate solution, freshly prepared.* Dissolve 0.1 g of anhydrous sodium carbonate in 500 mL of water.

2.3 Apparatus

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

2.3.2 *Spectrometer.* Capable of measuring absorbances at wavelengths in the range 460 nm to 470 nm and provided with cells of 40 mm optical path length.

2.3.3 *One-mark volumetric flasks,* (eight are required), 25 mL, complying with BS 1792.

2.3.4 *Platinum dish*

2.4 Procedure

NOTE Take every precaution to avoid chloride contamination while following this procedure, as only low quantities of chloride are to be determined.

2.4.1 *Test portion.* According to the expected chloride content, transfer an appropriate volume of the test sample containing 10 µg to 50 µg of chlorine, from a burette to the small platinum dish (**2.3.4**), noting the volume to the nearest 0.1 mL.

2.4.2 *Calibration.* Into a series of six one-mark volumetric flasks (**2.3.3**) add respectively 0.0 mL, 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL, and 5.0 mL of the dilute standard chloride solution (**2.2.4**). Dilute each to about 18 mL with water, add 4.0 mL of the ammonium iron (III) sulphate solution (**2.2.3**), insert the stopper and mix. Remove each stopper, add 2.0 mL of the mercury (II) thiocyanate (**2.2.2**) solution, dilute to 25 mL with water, replace the stopper, mix the contents of the flask and allow it to stand for 20 min.

NOTE The 0.0 mL calibration solution is the first reagent blank, R_1.

Using the spectrometer (**2.3.2**), measure the absorbance of each solution at a wavelength within the range 460 nm to 470 nm with water as the reference. Subtract the absorbance value of the reagent blank, R_1, from that measured in each case.

Prepare a calibration chart by plotting chloride (in µg) against the corrected absorbance value.

NOTE The graph is not strictly linear over this full calibration range.

2.4.3 *Determination.* Add to the test portion (**2.4.1**), a portion of the sodium carbonate solution (**2.2.5**) in the ratio of 1 : 5 (for example, add 5 mL for 25 mL of test portion) noting the volume. Evaporate to dryness on a boiling water bath, taking care to avoid any loss by spray.

Redissolve the residue in 5 mL of water, then allow to cool. Transfer quantitatively the solution to a one-mark volumetric flask (**2.3.3**) and dilute to about 18 mL with water. Continue by following the procedure described in **2.4.2**, from ". . ., add 4.0 mL of the ammonium . . .", but ignore the reference to the calibration chart.

2.4.4 *Preparation of the second reagent blank (R_2).* Prepare the second reagent blank, R_2, as follows. To a one-mark volumetric flask (**2.3.3**) transfer about 18 mL of water. Add a portion of the sodium carbonate solution equivalent to that used for the determination (see **2.4.3**). Continue by following the procedure described in **2.4.2**, from "…, add 4.0 mL of the ammonium …", but ignore the reference to the calibration chart.

2.5 Calculation and expression of results

Calculate the corrected absorbance from the following expression.

$$A_1 - A_2$$

where

A_1 is the absorbance of the test portion (see **2.4.3**);

A_2 is the absorbance of the second reagent blank, R_2 (**2.4.4**).

Using the corrected value of the absorbance, read the corresponding mass of chloride from the calibration chart.

The chloride content, expressed as milligrams of chlorine, CI, per kilogram, is given by the following expression.

$$\frac{m_1}{V \times \rho}$$

where

m_1 is the mass of chlorine determined (**2.4.3**) (in µg);

V is the volume of the test portion (**2.4.1**) (in mL);

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

3 Method 2. Potentiometric method

3.1 Principle

The principle is described in clause **3** of BS 6337-4:1984.

3.2 Reagents

The reagents described in clause **4** of BS 6337-4:1984 are required. Use water complying with grade 3 of BS 3978.

3.3 Apparatus

3.3.1 *General.* The apparatus described in clause **5** of BS 6337-4:1984 and the following are required.

3.3.2 *Platinum dish,* approximately 75 mL capacity.

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

3.4 Procedure

3.4.1 *Test portion.* Fill the dry one-mark volumetric flask (**3.3.3**) to the mark with the test sample. If the expected chloride content of the sample is greater than 750 mg/kg, take a smaller test portion (see **6.1** of BS 6337-4:1984).

3.4.2 *Preparation of the test solution.* Pour some of the test portion (**3.4.1**) into the platinum dish (**3.3.2**) and place it on a boiling water bath in a well-ventilated fume cupboard. Carefully evaporate the solution adding more of the test portion until the whole of the 100 mL has been used. Rinse the one-mark volumetric flask with water and add the washings to the sample in the platinum dish. Continue the evaporation until the majority of the ammonia has been released and about 10 mL of the solution is estimated to remain. Transfer quantitatively this residue to a clean dry beaker of suitable capacity (for example, 400 mL), keeping the volume of the solution to less than 50 mL.

3.4.3 *Determination.* Determine the chloride content of the test solution by following the procedure given in clause **6** of BS 6337-4:1984 and using the 0.1, mol/L silver nitrate solution.

3.5 Calculation and expression of results

Calculate the mass of chloride, expressed as chlorine, Cl, in g, in the test solution from the expression given in clause **7** of BS 6337-4:1984.

The chloride content, expressed as milligrams of chlorine, Cl, per kilogram, is given by the following expression.

$$\frac{m_2 \times 10^6}{V \times \rho}$$

where

m_2 is the mass of chloride found (in g);

V is the volume of the test portion (**3.4.1**) (in mL);

ρ is the density of the test portion at 20°, determined in accordance with BS 4651-1 (in g/m L).

4 Test report

The test report shall include the following information:

a) a reference to this British Standard, i.e. BS 4651-6:1988;

b) a reference to the method used, i.e. method 1 or method 2;

c) a complete identification of the sample;

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

e) the results expressed in accordance with **2.5** or **3.5**;

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

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Publications referred to

BS 1792, *Specification for one-mark volumetric flasks.*
BS 3978, *Specification for water for laboratory use.*
BS 4651, *Ammonia solution.*
BS 4651-0, *Methods for sampling.*
BS 4651-1, *Method for determination of density at 20 °C.*
BS 6337, *General methods of chemical analysis.*
BS 6337-4, *Method for determination of chloride ions by potentiometry.*

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BSI
389 Chiswick High Road
London
W4 4AL