BRITISH STANDARD

BS 4267-8: 1987

Ammonium nitrate —

Part 8: Methods for determination of chloride content

NOTE It is recommended that this Part of BS 4267 be read in conjunction with the information in the "General introduction", published separately as BS 4267-0.

WARNING. Ammonium nitrate is a strong oxidizing agent. If necessary, break the test sample up by crushing rather than grinding.

UDC 661.525:546.39'175:543

Foreword

This Part of BS 4267 has been prepared under the direction of the Chemicals Standards Committee. It supersedes clause **10** of BS 4267:1968, which has been deleted by amendment.

A colorimetric method is given which is technically equivalent to the method that was described in clause **10** of BS 4267:1968, except that the field of application has been limited to chloride contents of not more than 150 mg/kg. A potentiometric method has been included which refers to BS 6337-4:1984 (identical with ISO 6227:1982).

This standard describes methods 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 methods of test used are in accordance with BS 4267-8:1987.

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.

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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
30 September 1987

The Committees responsible for this British Standard are shown in Part 0

The following BSI references relate to the work on this standard:
Committee reference CIC/21
Draft (ref. 86/53125) announced in *BSI News*, September 1986

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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 4267 describes two methods for determination of the chloride content of ammonium nitrate for industrial use.

Clause **2** describes a colorimetric method, which is applicable to products having chloride contents of not more than 150 mg/kg.

Clause **3** describes a potentiometric method, which is applicable to products having chloride contents greater than 150 mg/kg.

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

2 Colorimetric method

2.1 Principle

The chloride ions present in a test portion are reacted with mercury (II) thiocyanate and ammonium iron (III) sulphate. The absorbance of the resulting solution is measured at a wavelength within the range 460 nm to 470 nm and the chloride content is determined 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 BS 3978.

2.2.2 *Mercury (II) thiocyanate solution.* Dissolve 0.1 g of mercury (II) thiocyanate and 2 g of ammonium nitrate in 100 mL of nitric acid solution, $c(HNO_3)$ = 63 g/L, warming gently to aid dissolution. Filter the solution if necessary.

2.2.3 *Ammonium iron (III) sulphate solution.* Dissolve 10.7 g of $(NH_4)_2SO_4,Fe_2(SO_4)_3.24H_2O$ in 100 mL of nitric acid solution, $c(HNO_3)$ = 378 g/L.

2.2.4 *Sodium chloride, standard solution.* Dissolve 0.1649 g of sodium chloride in water in the volumetric flask (**2.3.4**) and dilute to the mark. Further dilute 5 mL of this solution to 100 mL immediately before use. In the second solution 1 mL contains 10 µg of chloride ions.

2.2.5 *Nitric acid solution*, $c(HNO_3)$ = 378 g/L approximately.

2.3 Apparatus

2.3.1 *Ordinary laboratory apparatus*

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 *Nine* 25 mL *stoppered measuring cylinders,* complying with BS 604.

2.3.4 *One-mark volumetric flask*, 500 mL, complying with BS 1792.

2.4 Procedure

2.4.1 *Test portion.* Weigh, to the nearest 1 mg, approximately 0.3 g of the test sample.

2.4.2 *Calibration*. Using one measuring cylinder (**2.3.3**) for each calibration solution, add successively 0.0, 1.0, 2.0, 3.0, 4.0 and 5.0 mL of the dilute standard chloride solution (**2.2.4**). Make up each measuring cylinder to about 18 mL with water, add 4 mL of the ammonium iron (III) sulphate solution (**2.2.3**), insert the stopper and mix the solutions. Remove each stopper, add 2 mL of the mercury (II) thiocyanate solution (**2.2.2**), dilute to 25 mL with water, replace the stopper, mix the contents of the measuring cylinder and allow it to stand for 10 min to 20 min.

NOTE The 0.0 mL calibration solution is the reagent blank.

Using the spectrometer (**2.3.2**), measure the absorbance of each calibration 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 from that of each calibration solution. Prepare a calibration chart by plotting chloride (in µg) against the corrected absorbance value.

2.4.3 *Preparation of the sample blank.* Add 0.3 g of the test sample to one of the measuring cylinders (**2.3.3**), dissolve in about 18 mL of water, add 4.5 mL of the nitric acid solution (**2.2.5**), insert the stopper and mix the solutions. Remove the stopper, dilute to 25 mL with water, replace the stopper, mix the contents of the measuring cylinder and allow it to stand for 10 min to 20 min. Using the spectrometer (**2.3.2**), measure the absorbance of the solution at the wavelength used in the calibration and with water as the reference.

NOTE This blank compensates for the inherent colour or turbidity occasionally found in samples of ammonium nitrate solution.

2.4.4 *Determination.* Transfer the test portion (**2.4.1**) to one of the measuring cylinders (**2.3.3**), dissolve in about 18 mL of water and allow the solution to reach room temperature. Add 4 mL of the ammonium iron (III) sulphate solution (**2.2.3**), insert the stopper and mix the solutions. Remove the stopper, add 2 mL of the mercury (II) thiocyanate solution (**2.2.2**), dilute to 25 mL with water, replace the stopper, mix the contents of the measuring cylinder and allow it to stand for 10 min to 20 min.

Using the spectrometer (**2.3.2**), measure the absorbance of the test solution at the wavelength used in the calibration and with water as the reference.

2.5 Calculation and expression of results

Calculate the corrected absorbance from the following expression:

$A_1 - (A_2 + A_3)$

where

A_1 is the absorbance of the test solution;

A_2 is the absorbance of the reagent blank (**2.4.2**);

A_3 is the absorbance of the sample blank (**2.4.3**).

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

The chloride content, expressed as chlorine, Cl, in mg/kg, is given by the following expression:

$$\frac{m_1}{m_0}$$

where

m_1 is the mass of chloride found (in μg);

m_0 is the mass of the test portion (**2.4.1**) (in g).

3 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.

3.3 Apparatus

3.3.1 *General*. The apparatus described in clause **5** of BS 6337-4:1984 and the item described in **3.3.2** are required.

3.3.2 100 mL *one-mark volumetric flask*, complying with class B of BS 1792.

3.4 Procedure

3.4.1 *Test portion and preparation of the test solution*. Weigh, to the nearest 0.01 g, approximately 20 g of the test sample and transfer this test portion to the one-mark volumetric flask (**3.3.2**). Dissolve the test portion in water, dilute to the mark and mix.

3.4.2 *Determination*. Determine the chloride content of the test solution by following the procedure described in clause **6** of BS 6337-4:1984, using an appropriate amount of the test solution depending on the expected chloride ion content.

3.5 Calculation and expression of results

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

The chloride content, expressed as a percentage by mass of chlorine (Cl), is given by the following expression:

$$\frac{m_1}{m_0} \times 100$$

where

m_1 is the mass of chloride in the test solution (in g);

m_0 is the mass of the test portion (in g).

4 Test report

The test report shall include the following information:

a) an identification of the sample;

b) a reference to this British Standard, i.e. BS 4267-8:1987;

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

d) any unusual features noted during the determination;

e) any operation not included in this Part of BS 4267 or regarded as optional.

Publications referred to

BS 604 , *Specification for graduated glass measuring cylinders.*
BS 1792, *Specification for one-mark volumetric flasks.*
BS 3978, *Specification for water for laboratory use.*
BS 6337, *General methods of chemical analysis.*
BS 6337-4, *Method of determination of chloride ions by potentiometry .*

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