BRITISH STANDARD

BS 4267-9: 1987

Ammonium nitrate —

Part 9: Method for determination of sulphate 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

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. 86/53126) announced in *BSI News,* September 1986

ISBN 0 580 16122 6

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

Foreword

This Part of BS 4267 has been prepared under the direction of the Chemicals Standards Committee. It supersedes clause **12** of BS 4267:1968, to which it is technically equivalent and which has been deleted by amendment.

This Part of BS 4267 is related to ISO 3329 *"Ammonium nitrate for industrial use — Determination of sulphur compounds — Method by reduction and titrimetry"* published by the International Organization for Standardization (ISO).

This standard 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 is in accordance with BS 4267-9:1987.

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.

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

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.

1 Scope

This Part of BS 4267 describes a method for determination of sulphate content of ammonium nitrate for industrial use. The method is applicable to products with a sulphate content of between 10 mg/kg and 200 mg/kg.

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

2 Principle

A test portion is volatilized after the addition of sodium carbonate. The soluble residue is dissolved in hot water, the solution filtered and the sulphate precipitated by the addition of barium chloride solution. The sulphate content is determined by a visual turbidimetric method, using standard solutions of sulphuric acid for reference.

3 Reagents

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

3.2 *Sodium carbonate,* free from sulphate (see **5.3**).

3.3 *Hydrochloric acid solution, c*(HCl) = 2 mol/L approximately.

3.4 *Sulphuric acid solution,* $c(\frac{1}{2}H_2SO_4)$ = 0.002 mol/L.

3.5 *Sulphate seeding reagent*, freshly prepared.

Dissolve 2.0 g of barium chloride dihydrate, $BaCl_2.2H_2O$, in 75 mL of water, add 20 mL of 95 % (*V*/*V*) ethanol and 5 mL of the sulphuric acid solution (**3.4**).

4 Apparatus

4.1 *Six matched Nessler cylinders*, complying with BS 612.

4.2 *Burette*, class A, 25 mL, graduated in 0.05 mL, complying with BS 846.

4.3 *One-mark volumetric flask*, 50 mL, complying with BS 1792.

4.4 *Porcelain dish*, about 100 mm in diameter.

4.5 *Furnace*, capable of being maintained at 600 ± 10 °C.

5 Procedure

5.1 Test portion

Weigh, to the nearest 0.1 g, approximately 50 g of the test sample.

5.2 Standard solutions

Prepare the range of standard solutions at the same time as the determination. Using one Nessler cylinder (**4.1**) for each standard solution, add successively 2.0, 4.0, 6.0, 8.0 and 10.0 mL of the sulphuric acid solution (**3.4**). Make up each cylinder to 20 mL with water, add 1 mL of the sulphate seeding reagent (**3.5**), mix well and stand for 5 min. These standard solutions contain 196 µg to 980 µg sulphate, as H_2SO_4, i.e. approximately 200 µg to 1 000 µg.

5.3 Blank test

Weigh 0.02 g of the sodium carbonate (**3.2**) and dissolve it in 20 mL of water in a Nessler cylinder (**4.1**). Add 0.4 mL of the hydrochloric acid solution (**3.3**) and mix. Add 1 mL of the sulphate seeding reagent (**3.5**) and stand for 5 min.

If turbidity is observed discard the sodium carbonate. Repeat this procedure with sodium carbonate from different samples, until no turbidity is observed.

5.4 Determination

5.4.1 *Preparation of the test solution*. Transfer the test portion (**5.1**) into the porcelain dish (**4.4**) and dissolve in about 40 mL of water. Add 0.05 g of the sodium carbonate (**3.2**) and place the dish on a sand bath at a temperature of approximately 120 °C in a fume cupboard. When the water has evaporated, heat over a low flame until white fumes have ceased to be evolved. Heat the dish at 600 ± 10 °C in the furnace (**4.5**) for 15 min. Allow to cool.

WARNING. If the ammonium nitrate is heated too rapidly, a violent decomposition may result.

Dissolve the residue in 10 mL of water, add 1 mL of the hydrochloric acid solution (**3.3**) and filter into the volumetric flask (**4.3**). Wash the filter paper with several portions of water and dilute to the mark.

5.4.2 *Turbidity test*. If the sulphate content is expected to be between 10 mg/kg and 50 mg/kg transfer 20.0 mL of the test solution (**5.4.1**) to one of the Nessler cylinders (**4.1**).

If the sulphate content is expected to be between 40 mg/kg and 200 mg/kg transfer 5.0 mL of the test solution (**5.4.1**) to one of the Nessler cylinders (**4.1**) and add 15.0 mL of water.

Add 1 mL of the sulphate seeding reagent, mix well and stand for 5 min.

Compare the turbidity produced with that of each of the standard solutions (**5.2**) and note the sulphate content (in µg) of the sulphate standard which it most nearly matches.

6 Expression of results

The sulphate content, expressed as sulphuric acid (H_2SO_4), in mg/kg, is given by the following expression:

$$\frac{M}{V}$$

where

M is the mass of H_2SO_4(in μg), in the sulphate standard, the turbidity of which most nearly matches that produced from the test solution (**5.4.2**);

V is the volume of the test solution (**5.4.1**) taken (in mL).

7 Test report

The test report shall include the following information:

a) an identification of the sample;

b) a reference to this British Standard method, i.e. BS 4267-9:1987;

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

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 612, *Specification for Nessler cylinders.*
BS 846, *Specification for burettes.*
BS 1792, *Specification for one-mark volumetric flasks.*
BS 3978, *Specification for water for laboratory use.*

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: 020 8996 9000. Fax: 020 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 020 8996 9001. Fax: 020 8996 7001.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: 020 8996 7111. Fax: 020 8996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: 020 8996 7002. Fax: 020 8996 7001.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL