BRITISH STANDARD

BS 4267-7: 1987

Ammonium nitrate —

Part 7: Methods for determination of nitrite 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.

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Foreword

This Part of BS 4267 has been prepared under the direction of the Chemicals Standards Committee. It supersedes clause **9** of BS 4267:1968, to which it is technically equivalent and which has been deleted by amendment.

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 4267-7:1987.

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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. 86/53124) 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 methods for determination of the nitrite content of ammonium nitrate for industrial use. The method is applicable to products with a nitrite content of not more than 5 mg/kg.

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

2 Principle

Sulphanilic acid is diazotized by the nitrite present in a test portion. The resulting diazo compound obtained is coupled with Cleve's acid to give a magenta red colour which is measured spectrometrically or visually.

3 Reagents

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

3.2 *Glacial acetic acid*, ρ = 1.05 g/mL.

3.3 *Sulphanilic acid solution*. Dissolve 0.5 g of sulphanilic acid in 120 mL of water and add 30 mL of the glacial acetic acid (**3.2**).

3.4 *Cleve's acid solution.* Dissolve 0.2 g of Cleve's acid in 120 mL of water warming to 40 °C to aid solution. Filter the solution, cool, add 12 mL of the glacial acetic acid (**3.2**) and dilute to 150 mL with water.

NOTE The usual reagent available commercially is essentially 1-naphthylamine-7-sulphonic acid mixed with a smaller quantity of 1-naphthylamine-6-sulphonic acid. The latter has been found to be only half as sensitive as the former and it may be necessary to exercise some selection to obtain satisfactory sensitivity.

3.5 *Sodium nitrite, standard solution,* freshly prepared, using freshly boiled water.
Dissolve 0.986 g of sodium nitrite, previously dried at 105 °C, in water and dilute to 1 000 mL. Further dilute 5.0 mL of this solution to 1 000 mL. 1 mL of the second solution contains 1 µg of nitrite nitrogen.

4 Apparatus

4.1 *Ordinary laboratory apparatus*

4.2 *Spectrometer,* capable of measuring absorbance at a wavelength of approximately 525 nm and provided with cells of 10 mm optical path length.

4.3 *Seven Nessler cylinders*, complying with BS 612.

4.4 *Nine* 50 mL *stoppered measuring cylinders*, complying with BS 604.

5 Procedure

5.1 Test portion

According to the expected nitrite content weigh, to the nearest 0.01 g, a test portion containing up to 15 µg of nitrite nitrogen and, in any case, of mass not more than 25 g.

5.2 Calibration

Prepare fresh calibration solutions as follows. Using one of the measuring cylinders (**4.4**) for each calibration solution, add successively 0.0, 3.0, 6.0, 9.0, 12.0 and 15.0 mL of the standard nitrite solution (**3.5**). Make up each measuring cylinder to about 40 mL with water, add 2 mL of the sulphanilic acid solution (**3.3**), mix thoroughly and allow to stand at 22 ± 1 °C for 20 min to 30 min. Add 5 mL of the Cleve's acid solution (**3.4**), dilute to 50 mL with water, mix and allow to stand for a further 20 min to 30 min at 22 ± 1 °C.

NOTE The 0.0 mL calibration solution is the reagent blank.

Using the spectrometer (**4.2**), measure the absorbance of each calibration solution at approximately 525 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 nitrite nitrogen (in µg) against absorbance.

5.3 Determination

5.3.1 *General*. Carry out the determination at the same time as the procedure described in **5.2**, following one of the methods described in **5.3.2** or **5.3.3**.

5.3.2 *Determination by spectrometer*. Dissolve the test portion (**5.1**) in a small amount of water and transfer into one of the measuring cylinders (**4.4**). Dilute the solution to 40 mL, add 2 mL of the sulphanilic acid solution (**3.3**), mix thoroughly and allow to stand at 22 ± 1 °C for 20 min to 30 min. Add 5 mL of the Cleve's acid solution (**3.4**), dilute to 50 mL, mix and allow to stand for a further 20 min to 30 min at 22 ± °C.

Using the spectrometer (**4.2**), measure the absorbance of this test solution at the wavelength used in the calibration and with water as the reference. Note the corresponding mass of nitrite nitrogen (in μg) from the calibration chart.

5.3.3 *Visual determination*. Compare the colour of the test solution prepared (see **5.3.1**) with the series of prepared colour standards (see **5.2**) in matched Nessler cylinders. Note the nitrite content of the standard that most closely matches the test solution.

6 Calculation and expression of results

6.1 Determination by spectrometer

Calculate the corrected absorbance from the following expression:

$A_1 - A_2$

where

A_1 is the absorbance of the test solution (**5.3.2**)

A_2 is the absorbance of the reagent blank (**5.2**).

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

The nitrite content, expressed as nitrite nitrogen, N, in mg/kg, is given by the following expression:

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

where

m_1 is the mass of nitrite nitrogen found (in μg);

m_0 is the mass of the test portion (**5.1**) (in g).

NOTE To convert the nitrite nitrogen content to ammonium nitrite content, multiply the value by a factor of 4.57.

6.2 Visual determination

The nitrite content, expressed as nitrite nitrogen, N, in mg/kg, is given by the following expression:

$$\frac{m_2}{m_0}$$

where

m_0 is the mass of the test portion (**5.1**) (in g);

m_2 is the mass of nitrite nitrogen in the closest matching calibration standard (**5.3.3**) (in μg).

7 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 4267-7:1987;

c) the results expressed in accordance with **6.1** or **6.2**;

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 612, *Specification for Nessler cylinders.*
BS 3978, *Specification for water for laboratory use.*

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