Ammonium nitrate —

Part 4: Method for determination of sulphated ash

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 **5** of BS 4267:1968, to which it is technically equivalent and which has been deleted by amendment.

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 4267-4:1987.

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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Summary of pages

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

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

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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: Committee reference CIC/21 Draft for comment 84/54870 DC

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Amendments issued since publication

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1 Scope

This Part of BS 4267 describes a gravimetric method for determination of the sulphated ash of ammonium nitrate for industrial use. The method is applicable to products with a sulphated ash of not less than 0.1 % (m/m).

2 Principle

A test portion is volatilized at about 225 °C and then heated with sulphuric acid. The residue is then heated at about 850 °C to constant mass.

3 Reagent

3.1 Sulphuric acid, concentrated, $\rho = 1.84$ g/mL, analytical grade.

4 Apparatus

- 4.1 Ordinary laboratory apparatus
- 4.2 Platinum dish, about 75 mm in diameter.
- **4.3** *Muffle furnace*, capable of being maintained at 850 ± 50 °C.

5 Procedure

5.1 Test portion

Weigh, to the nearest $0.1~\mathrm{g}$, about $100~\mathrm{g}$ of the test sample.

5.2 Determination

Heat the empty platinum dish (4.2) in a muffle furnace at 850 ± 50 °C. Cool the dish in a desiccator and weigh to the nearest 0.1 mg. Repeat this process until successive weighings do not differ by more than 1 mg.

Gradually introduce the test portion (5.1), not more than 5 g at a time, into the weighed platinum dish while heating the dish on a hotplate at approximately 200 °C to 250 °C in a fume cupboard. After each addition, wait until volatilization is completed.

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

When all the ammonium nitrate has been volatilized, cool the dish and its contents and then add a few drops of the sulphuric acid (3.1). Heat the dish on a hotplate until no further fumes are evolved, transfer the dish to the muffle furnace (4.3) and ignite the residue at $850\pm50\,^{\circ}\mathrm{C}$. Cool the dish and its contents in a desiccator and weigh to the nearest 0.1 mg. Repeat the process of ignition, cooling and weighing until successive weighings do not differ by more than 1 mg.

6 Expression of results

The sulphated ash, expressed as a percentage by mass, is given by the following expression:

$$\frac{100\times M_1}{M_0}$$

where

 M_1 is the mass of the final residue (in g);

 M_0 is the mass of the test portion (5.1) (in g).

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 4267-4:1987;
- 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.

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