Methods of test for

# Desiccants —

Part 8: Estimation of ammonia and ammonium compounds content

NOTE It is recommended that this Part be read in conjunction with BS 3482-1 "Sampling, and preparation and storage of test samples" which is issued separately.



# Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Policy Committee (CIC/-) to Technical Committee CIC/2, upon which the following bodies were represented:

Chemical Industries Association
Ministry of Defence
Royal Society of Chemistry
Society of British Aerospace Companies Limited
Society of Environmental Engineers

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### Contents

		Page
Committees responsible		Inside front cover
For	reword	ii
1	Scope	1
2	Method A. Nessler method	1
3	Method B. Nessler method with Rochelle salt	1
Publication(s) referred to		Inside back cover

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### **Foreword**

This Part of BS 3482 has been prepared under the direction of the Chemicals Standards Policy Committee. This revision of BS 3482 comprises Part 1, which describes procedures for sampling and preparation and storage of test samples and a number of Parts describing methods of test for desiccants. This revision supersedes BS 3482:1962 which is withdrawn. It differs from BS 3482:1962 in that individual methods have been published in separate Parts and have been generally updated. Some additional methods not included in BS 3482:1962 have also been incorporated.

This Part of BS 3482 also supersedes the methods given in Appendix C of BS 2540:1960 and Appendix C of BS 2541:1960 which are withdrawn.

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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.

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#### 1 Scope

This Part of BS 3482 describes two pass/fail methods for the estimation of the level of ammonia and ammonium compounds in desiccants. Method A is for general use and method B is for use where precipitation of interfering hydroxides is likely.

NOTE 1 The desiccant product standards specify which of the methods is to be used

NOTE 2 The titles of the publications referred to in this standard are listed on the inside back cover.

#### 2 Method A. Nessler method

#### 2.1 Principle

Filtered aqueous extract and an ammonia standard solution are treated with Nessler's reagent. The intensities of the colours produced are compared to give a pass/fail result.

#### 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** *Nessler's reagent*, commercially available or prepared as follows.

Dissolve 3.5 g of potassium iodide and 1.25 g of mercury(II) chloride in 80 mL of water contained in a 500 mL glass flask. Add a cold saturated aqueous solution of mercury(II) chloride, stirring constantly, until a slight red precipitate remains, then add 12 g of sodium hydroxide. Allow this to dissolve, add a little more of the saturated mercury(II) chloride solution until a slight turbidity is obtained and dilute with 100 mL of water. Allow to settle, decant and store in the dark.

**2.2.3** Standard ammonia and ammonium compounds solution. Freshly prepare a 0.031 g/L solution of ammonium chloride.

#### 2.3 Apparatus

- **2.3.1** Ordinary laboratory apparatus
- 2.3.2 A matched pair of 100 mL Nessler tubes
- **2.3.3** *Graduated pipettes*, complying with BS 700.

#### 2.4 Procedure

#### 2.4.1 Sample test portion

Measure 25 mL of the filtered aqueous extract (see clause 3 of BS 3482-1:1991) into one of the Nessler tubes (3.3.2) and dilute to 50 mL with water.

#### 2.4.2 Standard solution test portion

Transfer 2.5 mL of the standard ammonia and ammonium compounds solution (2.2.3) using the graduated pipette (2.3.3) into the other Nessler tube (2.3.2) and dilute to 50 mL with water.

#### 2.4.3 Determination

Add 1 mL of the Nessler's reagent (2.2.2) to each test portion (2.4.1 and 2.4.2), mix well and allow to stand for 10 min. Stand both tubes on a white background and, looking down through the solution, compare the intensities of the colours.

#### 2.5 Expression of results

The test is recorded as pass or fail depending upon the relative strengths of the colours.

Record PASS if the colour of the standard solution (2.4.2) is more intense then the sample solution (2.4.1).

Record FAIL if the colour of the standard solution (2.4.2) is less intense than the sample solution (2.4.1).

#### 2.6 Test report

The test report shall include the following information.

- a) A complete identification of the sample.
- b) A reference to this British Standard method, i.e. method A of BS 3482-8:1991.
- c) The results, expressed in accordance with 2.5.
- d) Any unusual features noted during the estimation.

# 3 Method B. Nessler method with Rochelle salt

#### 3.1 Principle

Filtered aqueous extract and an ammonia standard solution are treated with Nessler's reagent and potassium sodium tartrate. The intensities of the colours produced are compared to give a pass/fail result.

#### 3.2 Reagents

- **3.2.1** *General.* During the analysis, use only reagents of recognized analytical grade and water complying with Grade 3 of BS 3978.
- **3.2.2** *Nessler's reagent*, commercially available or prepared as in **2.2.2**.
- **3.2.3** Potassium sodium tartrate, 50 % (m/m) solution.
- **3.2.4** Standard ammonia and ammonium compounds solution. Prepare as in **2.2.3**.

#### 3.3 Apparatus

- 3.3.1 Ordinary laboratory apparatus
- **3.3.2** A matched pair of 100 mL Nessler tubes
- **3.3.3** *Graduated pipette*, complying with BS 700.

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#### 3.4 Procedure

#### 3.4.1 Sample test portion

Measure 25 mL of the filtered aqueous extract (see clause 3 of BS 3482-1:1991) into one of the Nessler tubes (3.3.2) and dilute to 50 mL with water.

#### 3.4.2 Standard solution test portion

Transfer 2.5 mL of the standard ammonia and ammonium compounds solution (3.2.4) using the graduated pipette (3.3.3) into the other Nessler tubes (3.3.2) and dilute to 50 mL with water.

#### 3.4.3 Determination

Add 1 mL of the Nessler's reagent (3.2.2) and 1 mL of the potassium sodium tartrate solution (3.2.3) to each test portion (3.4.1 and 3.4.2) mix well and allow to stand for 10 min. Stand both tubes on a white background and, looking down through the solutions, compare the intensities of the colours.

#### 3.5 Expression of results

The test is recorded as pass or fail depending upon the relative strengths of the colours.

Record PASS if the colour of the standard solution (3.4.2) is more intense than the sample solution (3.4.1).

Record FAIL is the colour of the standard solution (3.4.2) is less intense than the sample solution (3.4.1).

#### 3.6 Test report

The test report shall include the following information.

- a) A complete identification of the sample.
- b) A reference to this British Standard method, i.e. method B of BS 3482-8:1991.
- c) The results, expressed in accordance with 3.5.
- d) Any unusual features noted during the estimation.

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# Publication(s) referred to

 ${\rm BS}\ 700, {\it Graduated\ pipettes}.$ 

BS 3482, Methods of test for desiccants.

BS 3482-1, Sampling, and preparation and storage of samples.

BS 3978, Specification for water for laboratory use.

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