

Methods of sampling and test for

Liquefied anhydrous ammonia

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Committees responsible for this British Standard

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- Association of Public Analysts
- British Coal Corporation
- Chemical Industries Association
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Fertiliser Manufacturers' Association Ltd.
- Institute of Trading Standards Administration
- Ministry of Agriculture, Fisheries and Food
- Ministry of Defence

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Foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee. It supersedes BS 4431:1969, which is withdrawn. The principal differences between this revision and the previous edition are as follows:

- a) a general procedure for taking the test portion has been introduced, based on international test methods;
- b) a method of sampling for the determination of oxygen is described;
- c) a general test report clause has been introduced.

For many years the UK has participated in the work of Sub-committee 4, of Technical Committee 47, Chemistry, of the International Organization for Standardization (ISO). However, ISO 7103-1982 “*Liquefied anhydrous ammonia for industrial use — Sampling — Taking a laboratory sample*”, which was produced by Technical Committee 47, was disapproved by the UK as the procedure is not used in the UK. ISO test methods applicable to anhydrous ammonia will not be implemented as Parts of BS 4431 as they refer to ISO 7103. This revision retains the guidance on sampling given in the previous edition and therefore the use of the evaporation factor has been retained for the calculation of results. Where appropriate, the methods have been based on international test methods, and the International Standards that are related to the methods described in the clauses are as follows.

Clause	Related International Standard
2	} ISO 4276
3	
4	} ISO 7106
5	
6	ISO 7105

The method for determination of water content, described in clause 6, refers frequently to BS 2511:1970, possession of which is essential.

It was assumed in the drafting of this British Standard that the methods described would be carried out by fully trained and supervised personnel.

WARNING. Liquefied anhydrous ammonia is a highly corrosive, toxic substance, which boils at – 33.3 °C at standard atmospheric pressure. Its action on the skin is strongly corrosive, producing severe and painful burns. Contact with the eyes can cause permanent blindness. Its vapour is strongly irritant to the mucous membrane and eyes, and produces a suffocating effect on the respiratory tract. Obtain advice for the safe storage and handling of liquefied anhydrous ammonia from manufacturers and suppliers. Prohibit smoking and sources of ignition during the sampling of ammonia.

NOTE Mixtures of ammonia gas and air can explode, if ignited, when the proportion of ammonia gas is within the limits of 16 % to 27 % by volume.

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 to 12, 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 British Standard describes methods of test and sampling for liquefied anhydrous ammonia suitable for general industrial purposes.

NOTE 1 In the methods described the ammonia is sampled by volume but the calculations are by mass. The density has been taken as 0.68 g/mL, at about -33°C , at which temperature liquid ammonia has a vapour pressure of $10^5 \text{ Pa}^{1)}$. A corrective evaporation factor is applied in each case to allow for loss through sampling at atmospheric pressure.

Clause 2 describes a general method for collecting the test portion with minor variations given for particular methods.

Clause 3 describes a method for the determination of residue on evaporation, which applies to products giving a residue value greater than or equal to 100 mg/kg.

Clause 4 describes a gravimetric method for the determination of the oil content (non-volatile at about 105°C), which applies to products having an oil content greater than or equal to 10 mg/kg.

Clause 5 describes a method for the determination of the oil content (non-volatile at about 105°C) in which the method used for the isolation of the oil content is as described in clause 4, but the determination is made using infra-red spectrometry. This is more sensitive than the gravimetric method and applies to products with an oil content less than 10 mg/kg.

Clause 6 describes a method for the determination of the water content by the Karl Fischer method, which applies to products with a water content greater than or equal to 25 mg/kg.

Clause 7 describes the determination of carbon dioxide by a titrimetric method, which applies to products with a carbon dioxide content greater than or equal to 15 mg/kg.

Clause 9 describes a method of sampling for the subsequent determination of oxygen by gas chromatography.

In Appendix A guidance is given on the sampling of anhydrous ammonia and in Appendix B a method for the determination of the evaporation factor, F , is described.

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

¹⁾ $10^5 \text{ Pa} = 10^5 \text{ N/m}^2 = 1 \text{ bar}$.

2 Method for collecting the test portion

2.1 Principle

A known volume of anhydrous ammonia is collected in a graduated, chilled (-35°C to -40°C) glass tube fitted with a modified Drechsel head and is allowed to begin to evaporate at ambient temperature and pressure.

2.2 Reagents

2.2.1 General. During the collection of the test portion, use only reagents of recognized analytical grade and water complying with grade 3 of BS 3978.

2.2.2 Ethanediol (ethylene glycol), of water content not greater than 0.1 % (m/m).

NOTE Care should be taken to avoid absorption of atmospheric moisture as ethanediol is very hygroscopic.

2.2.3 Barium hydroxide, standard volumetric solution, $c(\text{Ba}(\text{OH})_2) = 0.05 \text{ mol/L}$. Weigh, to the nearest 0.0005 g, 15.774 g of barium hydroxide octahydrate ($\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$), dissolve in a little water, transfer quantitatively the solution to the one-mark volumetric flask (2.3.2), dilute to the mark and mix.

2.2.4 Freezing mixture, consisting of solid carbon dioxide and methanol (technical grade is suitable), capable of reaching a temperature of between -35°C and -40°C .

2.3 Apparatus

2.3.1 General. Use ordinary laboratory apparatus and the following.

2.3.2 One-mark volumetric flask, 1 000 mL, complying with BS 1792.

2.3.3 Apparatus for collecting the test portion (see Figure 1).

2.3.3.1 Glass test tube of total capacity 250 mL, having a reference mark at 200 mL, fitted via a 24/29 ground glass modified Drechsel head to an arm with a three-way stopcock, with a key made from polytetrafluoroethylene (PTFE), and a side arm (3).

2.3.3.2 Dewar vessel, capable of containing the test tube (2.3.3.1) and the freezing mixture (2.2.4) while leaving the graduation mark visible.

2.4 Procedure

2.4.1 General. Carry out the procedure in accordance with 2.4.2, if appropriate, and 2.4.3. Do not attempt to subdivide the test portion.

2.4.2 Additional details for test portions to be used in the methods described in clauses 3, 6 and 7.

2.4.2.1 Clause 3. Initially weigh the test tube assembly (2.3.3.1) to the nearest 0.0001 g.

2.4.2.2 Clause 6. Introduce 2 mL of the ethanediol (2.2.2) into the test tube immediately before assembling the apparatus (2.3.3).

2.4.2.3 Clause 7. Add 10.0 mL of the standard barium hydroxide solution (2.2.3) to the test tube before assembling the apparatus (2.3.3).

NOTE When sampling direct from a cylinder or other pressurized vessel (see Appendix A), note the gauge pressure at that time in order to determine the evaporation factor, F (see Appendix B).

2.4.3 Common details. Three-quarters fill the Dewar vessel (2.3.3.2) with the freezing mixture (2.2.4), immerse the test tube (2.3.3.1) in the Dewar vessel (2.3.3.2) and insert the modified Drechsel head (see 2.3.3.1). Connect the inlet arm (1) to the ammonia sample point and attach to the outlet arms (2) and (3) pieces of tubing of sufficient length to lead away the escaping ammonia safely.

Ensure that the liquid anhydrous ammonia flow is controlled by means of a by-pass so that the apparatus is not subjected to high pressures.

Quickly turn the stopcock to introduce the liquid anhydrous ammonia into the test tube. As soon as the liquid anhydrous ammonia has reached the 200 mL graduation mark, turn the stopcock to isolate the branch into the test tube and to open the outlet arm (2).

Close the liquid anhydrous ammonia sample point valve to allow the liquid ammonia in the inlet and outlet arms (1 and 2 respectively) to evaporate before disconnecting the apparatus.

Remove the test tube containing the test portion from the Dewar flask and place it in a bath of cold running water. Allow the ammonia to evaporate slowly through arm (3). Turn the stopcock to the open position to ensure that no liquid anhydrous ammonia is trapped within the valve.

NOTE Numbers in parentheses in medium type refer to Figure 1.

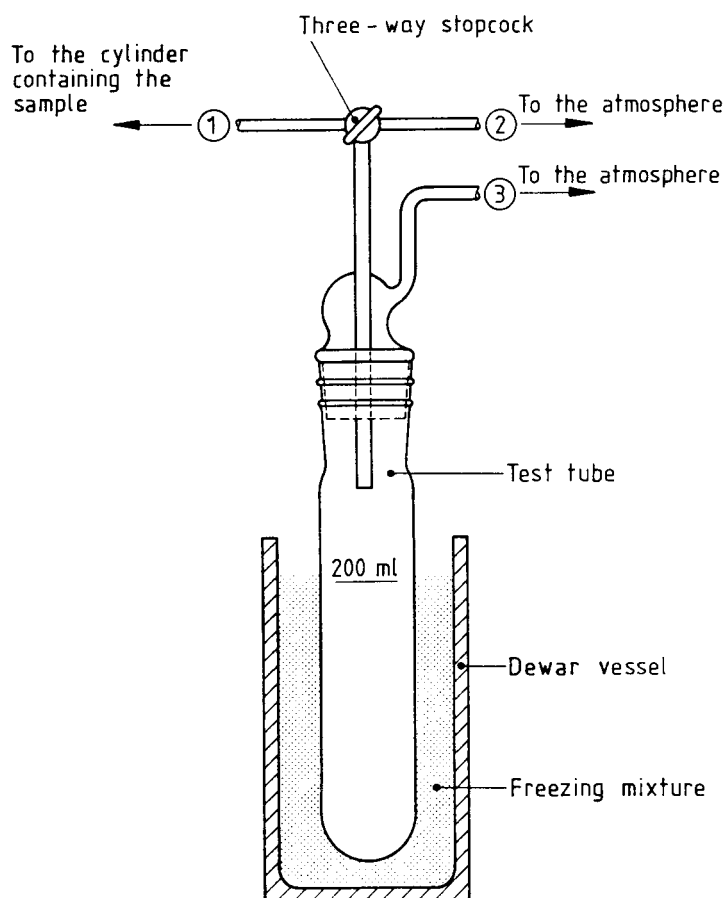


Figure 1 — Apparatus for collecting the test portion