

Methods of test for Nitric acid

Co-operating organizations

The Chemicals Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and industrial organizations:

- Board of Trade
- British Steel Industry
- Chemical Industries Association*
- Fertiliser Manufacturers' Association Ltd.*
- Gas Council*
- Institute of Vitreous Enamellers
- Institution of Gas Engineers
- Ministry of Health
- National Sulphuric Acid Association
- Royal Institute of Public Health and Hygiene

The industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

- British Sulphate of Ammonia Federation Limited
- Ministry of Defence, Army Department
- National Coal Board
- Pharmaceutical Society of Great Britain
- United Kingdom Atomic Energy Authority

This British Standard, having been approved by the Chemicals Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 21 October 1968

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Contents

	Page
Co-operating organizations	Inside front cover
Foreword	ii
1 Scope	1
2 Determination of total acidity	1
3 Determination of sulphuric acid content	1
4 Determination of nitrous acid content	2
5 Calculation of nitric acid content	3
6 Determination of combined ammonia content	3
7 Determination of total sulphate content	4
8 Determination of chloride content	5
9 Determination of iron content	6
10 Determination of lead content	7
11 Determination of sulphated ash	8
Figure 1 — Pressure pipette	10
Figure 2 — Typical device for use in the determination of sulphuric acid content	11
Figure 3 — Ammonia distillation apparatus	12
Figure 4 — Splash head for apparatus for the distillation of ammonia	13
Figure 5 — Tap funnel and double-bulb splash head	14

Foreword

This standard makes reference to the following British Standards:

BS 612, *Nessler cylinders*.

BS 975, *Density-composition tables for aqueous solutions of nitric acid*.

BS 1647, *pH scale*.

BS 1752, *Laboratory sintered or fritted filters*.

BS 1792, *One-mark volumetric flasks*.

BS 1848, *Glass condensers*.

BS 3591, *Industrial methylated spirits*.

BS 3978, *Water for laboratory use*.

BS, *Methods of test for sodium hypochlorite solution*¹⁾.

The preparation of this British Standard was authorized by the Chemicals Industry Standards Committee in order to provide methods for the analysis of nitric acid of commercial quality and intended for general industrial use.

This standard prescribes methods of test only and should not be used or quoted as a specification defining limits of purity. References to the standard should be in a form of words indicating the methods of test used conform to BS 4367.

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, pages 1 to 14 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⁾ In course of preparation.

1 Scope

This British Standard describes methods of test for commercial nitric acid suitable for general industrial purposes.

2 Determination of total acidity

2.1 Outline of method. A weighed quantity of the acid is diluted with water and titrated directly with standard sodium hydroxide solution.

2.2 Apparatus. The following apparatus is required:

- 1) *Pressure pipette*, as shown in Figure 1.
- 2) *Flask*, conical 150 ml capacity, fitted with a glass stopper.

2.3 Reagents. The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978²⁾ shall be used throughout.

- 1) *Sodium hydroxide*, N standard volumetric solution.
- 2) *Bromophenol blue indicator solution*, 0.4 g/l. Warm 0.1 g of bromophenol blue with 3.0 ml of 0.05N sodium hydroxide solution and 5 ml of 95 % (v/v) ethanol³⁾; after solution is effected, add 50 ml of 95 % (v/v) ethanol³⁾ and dilute to 250 ml with water.

2.4 Procedure. Put 30–50 ml of water into the flask, carefully dry the neck of the flask and the stopper and ensure that the outside of the flask is thoroughly dry. Replace the stopper and weigh the flask to the nearest milligramme.

Using the pressure pipette transfer about 2 ml of the sample to the flask, taking care not to allow the pipette to touch the water or sides of the flask. Immediately replace the stopper, rotate the flask to mix the contents and allow to stand for five minutes.

Momentarily withdraw the stopper to adjust the pressure. Carefully wipe the outside of the flask and reweigh to the nearest milligramme.

Add a few drops of the bromophenol blue indicator and titrate without delay with the sodium hydroxide until the colour just changes to a true blue. At the end-point, the addition of a further drop of the sodium hydroxide will produce no further change in colour.

NOTE For the highest accuracy it is essential to take into account corrections arising from calibration of the burettes and to correct the volumes of standard sodium hydroxide solution used for any deviation of temperature from that at which the solution was standardized. Relevant tables for this purpose are given in "Chemical analysis. The working tools": C. R. N. Strouts, H. N. Wilson, R. T. Parry-Jones, eds., Volume 1, page 222, Clarendon Press 1962.

2.5 Calculation. Total acidity, calculated as nitric acid, HNO_3 ,

$$\text{percentage by mass} = \frac{6.302 \times T_1}{M_1}$$

where T_1 = volume, in millilitres, of N sodium hydroxide solution used in the titration

and M_1 = mass, in grammes, of sample taken.

3 Determination of sulphuric acid content

3.1 Outline of method. Nitrogen acids are removed by volatilization in a current of hot air and the residual sulphuric acid is titrated with standard sodium hydroxide solution.

3.2 Apparatus. The following apparatus is required:

- 1) *Conical flask*, 300 ml capacity, with a suitable device to aid evaporation. One such device is shown in Figure 2; the mouth of the flask is closed by a loosely fitting, hollow, tapered closure, designed so that heated filtered air may be blown into the flask and the volatile nitrogen acids allowed to escape. The cup at the top serves as a deflector.
- 2) *Air line*, in which filtered compressed air is heated by passing through a coil of metal tubing immersed in boiling water.

²⁾ BS 3978, "Water for laboratory use".

³⁾ Ethanol may be replaced by industrial methylated spirits, 66 degrees O.P., complying with BS 3591, "Industrial methylated spirits". It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations 1952 (S.I. 1952 No. 2230).

3.3 Reagents. The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978⁴⁾ shall be used throughout.

- 1) *Sodium hydroxide*, 0.1N standard volumetric solution.
- 2) *Hydrochloric acid*, 0.1N non-standardized volumetric solution.
- 3) *Bromophenol blue indicator solution*, 0.4 g/l. Warm 0.1 g of bromophenol blue with 3.0 ml of 0.05N sodium hydroxide solution and 5 ml of 95 % (v/v) ethanol⁵⁾ after solution is effected, add 50 ml of 95 % (v/v) ethanol⁵⁾ and dilute to 250 ml with water.
- 4) *Litmus paper*, neutral.

3.4 Procedure. Transfer a suitable quantity of the sample, according to the expected sulphuric acid content, to the flask and place the closure in position. Place the flask in a boiling water-bath in a fume cupboard and connect to the heated, filtered air supply. Continue passing the air until the escaping air is acid free when tested with the moistened litmus paper. Wash down the closure and the walls of the flask, using about 10 ml of water and repeat the evaporation. Again wash down with about 10 ml of water and repeat the evaporation.

To 50 ml of water add a few drops of the indicator solution and just neutralize it with the hydrochloric acid solution. Use this water to wash down the walls of the flask and the inside of the closure and collect the washings in the flask. Titrate the solution in the flask with the sodium hydroxide solution.

3.5 Calculation. Sulphuric acid content, calculated as H_2SO_4 ,

$$\text{percentage by mass} = \frac{0.4904 \times T_2}{V_1 \times d}$$

where d = relative density^a of the sample at room temperature,

V_1 = volume, in millilitres, of sample taken

and T_2 = volume, in millilitres, of 0.1N sodium hydroxide solution used.

^a Useful information is given in BS 975, "Density-composition tables for aqueous solutions of nitric acid".

4 Determination of nitrous acid content

4.1 Outline of method. The nitrous acid is oxidized with excess ceric sulphate solution, and the excess titrated with standard ammonium ferrous sulphate solution.

4.2 Reagents. The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978⁴⁾ shall be used throughout.

- 1) *Ammonium ferrous sulphate*, 0.1N standard volumetric solution.

NOTE This solution requires restandardization each week.

- 2) *Ceric sulphate*, 0.1N non-standardized volumetric solution. To 55 g of dry ammonium ceric nitrate add 50 ml of concentrated sulphuric acid ($d = 1.84$) and evaporate almost to dryness. Dissolve the residue in approximately N sulphuric acid solution and dilute to 1 000 ml with this acid.

- 3) *N-Phenylanthranilic acid indicator solution*, 2 g/l. Dissolve 0.1 g of N-phenylanthranilic acid in 10 ml of N sodium hydroxide solution and dilute to 50 ml with water. This solution is stable for about 1 week.

4.3 Procedure. Into a stoppered weighing bottle, weigh to the nearest milligramme, about 10 g of the sample. Transfer the weighing bottle and stopper to a wide-necked conical flask containing about 200 ml of water and 25.0 ml of the ceric sulphate solution. Swirl the flask to mix the contents, add 1 drop of the N-phenylanthranilic acid solution and titrate with the ammonium ferrous sulphate solution.

At the same time titrate under the same conditions 25.0 ml of the ceric sulphate solution.

4.4 Calculation. Nitrous acid content, calculated as HNO_2 ,

$$\text{percentage by mass} = \frac{0.235 (T_3 - T_4)}{M_2}$$

where T_3 = volume, in millilitres, of 0.1N ammonium ferrous sulphate solution used for the 25.0 ml of ceric sulphate solution,

T_4 = volume, in millilitres, of 0.1N ammonium ferrous sulphate solution used in the determination

and M_2 = mass, in grammes, of sample taken.

⁴⁾ BS 3978, "Water for laboratory use".

⁵⁾ Ethanol may be replaced by industrial methylated spirits, 66 degrees O.P., complying with BS 3591, "Industrial methylated spirits". It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations 1952 (S.I. 1952 No. 2230).