

Acetic acid for industrial use —

Part 2: Methods of test for acetic acid

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- British Pharmacopoeia Commission
- British Society of Perfumers
- Chemical Industries Association
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Royal Society of Chemistry

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Foreword

This Part of BS 576 has been prepared under the direction of the Chemicals Standards Committee and provides a comprehensive series of test methods for acetic acid for industrial use. Although applicable to the material in general, it includes all the test methods required to assess compliance with BS 576-1. This Part, together with BS 576-1, supersedes BS 576:1969, which is withdrawn.

In preparing this standard, the opportunity has been taken to implement various International Standards describing test methods for acetic acid. These have been prepared, with the active participation of the UK, by Technical Committee 47, Chemistry, of the International Organization for Standardization (ISO), as separate Parts of ISO 753 and constitute the revision of ISO Recommendation R 753. Appendix A gives the relationship between International Standards and this British Standard, together with the relationship between general test methods and corresponding International Standards.

Appendix B contains a graph showing the relationship between crystallizing point and acetic acid content, based on the assumption that water is the only impurity present.

This Part of BS 576 specifies methods of test only and should not be used as a specification defining limits of purity. Reference to this Part of BS 576 should indicate that the methods of test used are in accordance with the appropriate clause(s) of BS 576-2.

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 Part of BS 576 describes methods for testing acetic acid for industrial use.

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

2 Sampling and storage of sample

Take a representative sample from the bulk liquid.

NOTE Methods for sampling liquid chemical products are described in BS 5309-1 and BS 5309-3.

Store the laboratory sample in a clean, dry and airtight, ground glass stoppered bottle, or a screw-capped bottle fitted with a polyethylene cone insert, of such capacity that it is almost filled by the sample. Sufficient ullage should be left in the bottle to avoid excessive pressure changes that could arise from temperature variations during storage and handling. About 10 % ullage is recommended. If it is necessary to seal the bottle, take care to avoid any risk of contamination of the contents. Store the sample in a cool place in the dark.

3 Measurement of colour

Determine the colour by the method described in BS 5339.

4 Determination of crystallizing point

Determine the crystallizing point by the method described in BS 4633, using a thermometer no. A20C/100 complying with BS 593 in the inner tube.

NOTE A graph showing the relationship between crystallizing point and acetic acid content is given in Appendix B.

5 Determination of acetic acid content

5.1 Principle

A test portion of acetic acid is titrated with standard volumetric sodium hydroxide solution, using phenolphthalein as indicator. The acid content is then calculated, making allowance for any formic acid present.

5.2 Reagents

5.2.1 General. Use only reagents of recognized analytical grade, only methylated spirits complying with BS 3591, and only water complying with grade 3 of BS 3978.

5.2.2 Sodium hydroxide, standard volumetric solution, $c(\text{NaOH}) = 1 \text{ mol/L}$.

5.2.3 Phenolphthalein, 5 g/L ethanolic solution.

Dissolve 0.5 g of phenolphthalein in 100 mL of 95 % (V/V) ethanol, or 95 % (V/V) industrial methylated spirits, and add 4 g/L sodium hydroxide solution until a pale pink coloration is obtained.

NOTE The use of industrial methylated spirits is governed by the Methylated Spirits Regulations, 1983 (S.I. 1983 No. 252). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquor Duties Act 1979, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

5.3 Apparatus

Ordinary laboratory apparatus together with the following.

5.3.1 Weighing pipette, of capacity 10 mL.

5.3.2 Burette, of capacity 50 mL, complying with class A of BS 846.

5.4 Procedure

5.4.1 Test portion. Using the weighing pipette (5.3.1), weigh, to the nearest 0.0001 g, a mass of the laboratory sample containing 2 g to 3 g of glacial acetic acid. Suitable masses are given in Table 1.

Transfer the test portion to a 250 mL conical flask containing 50 mL of water which has been boiled and cooled in a carbon dioxide-free atmosphere.

Table 1 — Mass of test portion for determination of acetic acid content

Nominal acetic acid content of laboratory sample	Mass of test portion
% (m/m)	g
98 to 100	2.5
80	3.0
60	4.0
40	6.0

5.4.2 Determination. Add 0.5 mL of the phenolphthalein solution (5.2.3) to the conical flask containing the test portion (5.4.1) and titrate with the sodium hydroxide solution (5.2.2) from the burette (5.3.2) until a pink colour, which persists for about 5 s, is obtained.

5.5 Expression of results

The acetic acid (CH_3COOH) content A , expressed as a percentage by mass, is given by equation:

$$A = \frac{0.060 \times V \times 100}{M} - 1.3F$$

$$= \frac{6V}{M} - 1.3F$$

where

V is the volume (in mL) of the standard volumetric sodium hydroxide solution used for the determination;

M is the mass (in g) of the test portion;

F is the formic acid content, expressed as a percentage by mass, determined by the method described in clause 11;

- 1.3 is the factor for the conversion of formic acid to acetic acid;
- 0.060 is the mass (in g) of acetic acid corresponding to 1 mL of sodium hydroxide solution, $c(\text{NaOH}) = 1.00 \text{ mol/L}$.

6 Determination of residue on evaporation on a water bath

Determine the residue on evaporation by the method described in BS 4524.

7 Determination of iron content

7.1 General

Determine the iron content by the method described in BS 6337-3 using a 50 mL aliquot portion of a test solution prepared as described in 7.2. Calculate the result from the equation given in 7.3.

7.2 Test portion and preparation of the test solution

Weigh, to the nearest 0.001 g, 40 g of the laboratory sample and transfer this test portion quantitatively to a 100 mL one-mark volumetric flask containing about 40 mL of water. Dilute to the mark and mix.

7.3 Calculation of result

The iron content B , expressed as a percentage by mass, is given by the equation:

$$B = \frac{2(M_2 - M_3) \times 100}{M_1 \times 10^6}$$

$$= \frac{M_2 - M_3}{5000 \times M_1}$$

where

- M_1 is the mass (in g) of the test portion;
- M_2 is the mass (in μg) of iron found in the test solution;
- M_3 is the mass (in μg) of iron found in the blank test solution.

8 Limit test for inorganic chlorides

8.1 Principle

The turbidity obtained by adding a silver nitrate solution to a test solution, acidified with nitric acid, and the turbidity obtained from a chloride solution, of known concentration, treated in the same manner are compared visually.

8.2 Reagents

8.2.1 General. Use only reagents of recognized analytical grade and only water complying with grade 3 of BS 3978.

8.2.2 Nitric acid, 315 g/L solution.

8.2.3 Silver nitrate, 50 g/L solution.

8.2.4 Standard chloride solution, containing 0.1 g of Cl^- per litre.

Transfer 28.2 mL of a standard volumetric hydrochloric acid solution, $c(\text{HCl}) = 0.1 \text{ mol/L}$, to a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix.

1 mL of this solution contains 0.1 mg of Cl^- .

8.3 Apparatus

Ordinary laboratory apparatus together with the following.

8.3.1 Filter papers, chloride-free.

8.3.2 Two matched Nessler cylinders, graduated at 100 mL.

8.4 Procedure

8.4.1 Test portion. Weigh ($M \pm 0.01 M$) g of the laboratory sample, where the value of M depends on the limit set for the inorganic chlorides content, $x \%$ (m/m), and is given in Table 2.

Table 2 — Mass of test portion and volume of aliquot portion of test solution for limit test for inorganic chlorides

Limit for inorganic chlorides content, x	Mass of test portion, M	Volume of aliquot portion of test solution
% (m/m)	g	mL
Less than 0.0005	$(50 \times 0.0005)/x$	100
0.0005 to 0.05	50	$0.05/x$
Greater than 0.05	$(50 \times 0.05)/x$	1

8.4.2 Preparation of the test solution. Transfer the test portion (8.4.1) quantitatively to a 250 mL one-mark volumetric flask containing approximately 50 mL of water. Dilute to the mark and mix.

If the solution is cloudy, filter it through one of the filter papers (8.3.1). If the solution is still cloudy, carry out an extraction with a suitable solvent, for example light petroleum, to remove any wax which might be present.

8.4.3 Preparation of standard turbidimetric solution. Transfer 1.0 mL of the standard chloride solution (8.2.4) to one of the Nessler cylinders (8.3.2), dilute to the 100 mL mark with water, add 2 mL of the nitric acid solution (8.2.2) and mix.

8.4.4 Test. Transfer an aliquot portion of the test solution (8.4.2), as specified in Table 2, to the second Nessler cylinder (8.3.2), dilute to the 100 mL mark with water, add 2 mL of the nitric acid solution (8.2.2) and mix.