BRITISH STANDARD

BS 576-2:1988

Acetic acid for industrial use —

Part 2: Methods of test for acetic acid

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Committee (CIC/-) to Technical Committee CIC/51, upon which the following bodies were represented:

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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Contents

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 This Part, together with BS 576-1, supersedes BS 576:1969, which is withdrawn.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.

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, $c(\mathrm{NaOH}) = 1$ 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:

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

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

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, $c(\text{NaOH}) = 1.00$ 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, $c(\text{HCl}) = 0.1$ 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.

Add to each Nessler cylinder 1 mL of the silver nitrate solution (**8.2.3**) and mix. Allow the cylinders to stand in the dark for 5 min and then compare the turbidity produced by the aliquot portion of the test solution with that produced by the standard turbidimetric solution (**8.4.3**).

8.5 Interpretation of results

The inorganic chlorides content does not exceed the limit of x % (m/m) of Cl^- if the turbidity produced by the test solution does not exceed that produced by the standard turbidimetric solution.

9 Limit test for inorganic sulphates

9.1 Principle

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

9.2 Reagents

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

9.2.2 *Sodium carbonate,* 53 g/L solution of the anhydrous salt.

9.2.3 *Hydrochloric acid,* 36.5 g/L solution of hydrogen chloride.

9.2.4 *Barium chloride dihydrate,* 100 g/L solution.

9.2.5 *Standard sulphate solution,* containing 0.1 g of SO_4^{2-} per litre.

Transfer 20.8 mL of a standard volumetric sulphuric acid solution, $c(1/2\ H_2SO_4) = 0.1$ 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 SO_4^{2-}.

9.3 Apparatus

Ordinary laboratory apparatus together with the following.

9.3.1 *Filter papers,* sulphate-free.

9.3.2 *Two matched Nessler cylinders,* graduated at 100 mL.

9.4 Procedure

9.4.1 *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 sulphates content, y % (m/m), and is given in Table 3.

Table 3 — Mass of test portion and volume of aliquot portion of test solution for limit test for inorganic sulphates

Limit for inorganic sulphates content, y	Mass of test portion, M	Volume of aliquot portion of test solution
% (m/m)	g	mL
Less than 0.001	$(100 \times 0.001)/y$	100
0.001 to 0.1	100	$0.1/y$
Greater than 0.1	$(100 \times 0.1)/y$	1

9.4.2 *Preparation of the test solution.* Transfer the test portion (**9.4.1**) quantitatively to a porcelain evaporating dish of suitable capacity. Add 0.2 mL of the sodium carbonate solution (**9.2.2**) and evaporate to dryness on a boiling water bath in a fume cupboard. Dissolve the residue in water containing 1 mL of the hydrochloric acid solution (**9.2.3**), transfer the solution quantitatively to a 250 mL one-mark volumetric flask, dilute to the mark with water and mix.

If the solution is cloudy, filter it through one of the filter papers (**9.3.1**). 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.

9.4.3 *Preparation of standard turbidimetric solution.* Transfer 4.0 mL of the standard sulphate solution (**9.2.5**) to one of the Nessler cylinders (**9.3.2**), dilute to the 100 mL mark with water, add 2 mL of the hydrochloric acid solution (**9.2.3**) and mix.

9.4.4 *Test.* Transfer an aliquot portion of the test solution (**9.4.2**), as given in Table 3, to the second Nessler cylinder (**9.3.2**), dilute to the 100 mL mark with water, add 2 mL of the hydrochloric acid solution (**9.2.3**) and mix.

Add to each Nessler cylinder 2 mL of the barium chloride solution (**9.2.4**) and mix. Allow the cylinders to stand for 5 min, agitate each cylinder to mix its contents again and then compare the turbidity produced by the aliquot portion of the test solution with that produced by the standard turbidimetric solution (**9.4.3**).

9.5 Interpretation of results

The inorganic sulphates content does not exceed the limit of y % (m/m) of SO_4^{2-} if the turbidity produced by the test solution does not exceed that produced by the standard turbidimetric solution.

10 Limit test for heavy metals (including iron)

NOTE The method detects only the heavy metals present in non-complex form and is not specific for any one heavy metal.

10.1 Principle

Heavy metals, such as lead, copper and iron, present in a test solution of acetic acid, are converted to their sulphides by treatment with sodium sulphide in the presence of ammonia. The colour produced is compared with that of a lead solution, of known concentration, treated in the same manner.

10.2 Reagents

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

10.2.2 *Ammonia solution,* ρ 0.88 g/mL, approximately 34.3 % (m/m) solution.

10.2.3 *Sodium sulphide nonahydrate* ($Na_2S \cdot 9H_2O$), 100 g/L solution.

10.2.4 *Standard lead solution,* containing 0.01 g of Pb per litre.

Weigh, to the nearest 0.0001 g, 0.0160 g of lead nitrate [$Pb(NO_3)_2$], dissolve in water and transfer the solution to a 1 000 mL one-mark volumetric flask. Dilute to the mark and mix.

1 mL of this solution contains 0.01 mg of Pb.

Prepare this solution immediately before use.

10.2.5 *Litmus paper* (pH range 5 to 8).

10.3 Apparatus

Ordinary laboratory apparatus together with the following.

10.3.1 *Two matched Nessler cylinders,* graduated at 50 mL.

10.4 Procedure

10.4.1 *Test portion.* Weigh ($M \pm 0.004\ M$) g of the laboratory sample, where the value of M depends on the limit set for the heavy metals content, z % (m/m), and is given in Table 4.

Table 4 — Mass of test portion and volume of aliquot portion of test solution for limit test for heavy metals

Limit for heavy metals content, z	Mass of test portion, M	Volume of aliquot portion of test solution
% (m/m)	g	mL
Less than 0.0005	$(25 \times 0.0005)/z$	40
0.0005 to 0.04	25	$0.02/z$
Greater than 0.04	$(25 \times 0.04)/z$	0.5

10.4.2 *Preparation of the test solution.* Transfer the test portion (**10.4.1**) quantitatively to a 250 mL one-mark volumetric flask containing about 50 mL of water. Dilute to the mark and mix.

10.4.3 *Preparation of standard matching solution.* Place 20 mL of water in one of the Nessler cylinders (**10.3.1**) and add 2.0 mL of the standard lead solution (**10.2.4**) and 1 mL of the ammonia solution (**10.2.2**). Dilute to the 50 mL mark with water and mix. Add 0.1 mL (two drops) of the sodium sulphide solution (**10.2.3**) and mix again.

10.4.4 *Test.* Transfer an aliquot portion of the test solution (**10.4.2**), as given in Table 4, to the second Nessler cylinder (**10.3.1**). Add the ammonia solution (**10.2.2**) until the solution is alkaline to the litmus paper (**10.2.5**) (blue colour), dilute to the 50 mL mark with water and mix. Add 0.1 mL (two drops) of the sodium sulphide solution (**10.2.3**) and mix again.

Compare the depth of colour of this solution with that of the standard matching solution (**10.4.3**).

10.5 Interpretation of results

The heavy metals content does not exceed the limit of z % (m/m), expressed as Pb, if the depth of colour produced by the test solution does not exceed that of the standard matching solution.

11 Determination of formic acid content

NOTE This method is applicable to products having formic acid contents between 0.02 % (m/m) and 0.35 % (m/m).

11.1 Principle

The total reducing matter in a test portion is determined by oxidation with an excess of sodium hypobromite solution and the excess sodium hypobromite is determined by iodometry.

The reducing compounds other than formic acid are determined in a further test portion by oxidation with an excess of potassium bromide-bromate solution, in an acid medium, and the excess potassium bromide-bromate is determined by iodometry.

The formic acid content is calculated from the difference between the two determinations.

11.2 Reactions

The following reactions occur during the test.

$$HCOOH + NaOBr \rightarrow NaBr + CO_2 + H_2O$$

$$NaOBr + 2KI + 2HCl \rightarrow 2KCl + NaBr + H_2O + I_2$$

$$KBrO_3 + 5KBr + 6HCl \rightarrow 6KCl + 3Br_2 + 3H_2O$$

$$3Br_2 + 6KI \rightarrow 6KBr + 3I_2$$

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

11.3 Reagents

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

11.3.2 *Hydrochloric acid solution,* containing approximately 220 g of concentrated hydrochloric acid per litre.

11.3.3 *Potassium iodide,* approximately 250 g/L solution.

11.3.4 *Sodium hypobromite solution,* c(1/2 NaOBr) = 0.1 mol/L, approximately.

Place 100 mL of 80 g/L sodium hydroxide solution in a 1 000 mL one-mark volumetric flask, add 500 mL of water and then carefully add 2.8 mL of bromine. Stir until dissolution is complete, dilute to the mark with water and mix.

WARNING. Bromine is very toxic and causes severe burns. Prevent inhalation of vapour and contact with skin and eyes.

11.3.5 *Potassium bromide-bromate solution,* c(1/6 $KBrO_3$) = 0.1 mol/L, approximately.

In a 400 mL beaker, dissolve 10 g of potassium bromide and 2.78 g of potassium bromate in 200 mL of water. Quantitatively transfer the solution into a 1 000 mL one-mark volumetric flask, dilute to the mark with water and mix.

11.3.6 *Sodium thiosulphate,* standard volumetric solution, $c(Na_2S_2O_3)$ = 0.1 mol/L, approximately.

Standardize this solution immediately before use.

11.3.7 *Starch solution.* Triturate 1.0 g of soluble starch with 5 mL of water and add the mixture, while stirring, to 100 mL of boiling water. Boil for a few minutes and allow to cool.

Discard this solution after 2 weeks.

11.4 Apparatus

Ordinary laboratory apparatus together with the following.

11.4.1 *Conical flasks,* of minimum capacity 500 mL, resistant under vacuum, with ground glass necks (see Figure 1).

11.4.2 *Dropping funnel,* of capacity 50 mL to 100 mL, resistant under vacuum, with a bottom ground glass joint matching the necks of the flasks (**11.5.2**) (see Figure 1).

11.4.3 *Vacuum pump,* capable of maintaining a residual pressure down to 100 mbar[1].

11.4.4 *Weighing pipette,* of capacity 10 mL.

11.5 Procedure

WARNING. All operations involving a vacuum should be carried out behind a screen.

11.5.1 *Determination of total reducing matter*

11.5.1.1 *Test portion.* Using the weighing pipette (**11.4.4**), weigh, to the nearest 0.01 g, approximately 10 g of the laboratory sample.

11.5.1.2 *Blank test.* Carry out a blank test following the same procedure as for the determination (**11.5.1.3**), using the same amounts of all reagents [except the sodium thiosulphate solution (**11.3.6**)] as used for the determination, but replacing the test portion with 10 mL of water.

11.5.1.3 *Determination.* Introduce 80 mL of water into a flask (**11.4.1**) and fit the dropping funnel (**11.4.2**) to it. Open the stopcock of the funnel and connect the funnel to the vacuum pump (**11.4.3**). Evacuate the flask to produce a residual pressure sufficient for the intake of at least 200 mL of liquid. Close the stopcock of the funnel and disconnect the vacuum pump.

Introduce 25.0 mL of the sodium hypobromite solution (**11.3.4**) into the funnel. Open the stopcock sufficiently to draw most of the solution into the flask and close it again.

NOTE It is essential that, on each addition to the flask, sufficient liquid is left in the funnel to form a seal and prevent the release of the vacuum.

Rinse down the inside of the funnel with two 5 mL portions of water, each time drawing most of the water into the flask by opening and closing the stopcock.

Introduce the test portion (**11.5.1.1**) into the funnel and draw most of it into the flask. Repeat the rinsing with two 5 mL portions of water, drawing most of the water into the flask. Mix and allow to stand for about 10 min at room temperature to complete the reaction.

Then introduce, via the funnel, 5 mL of the potassium iodide solution (**11.3.3**), followed by 20 mL of the hydrochloric acid solution (**11.3.2**) and shake for 30 s. Open the stopcock to release the vacuum, disconnect the funnel and add 50 mL of water to the flask. Titrate the liberated iodine with the sodium thiosulphate solution (**11.3.6**) to a pale yellow colour. Add about 2 mL of the starch solution (**11.3.7**) and continue the titration until the blue colour is discharged.

11.5.2 *Determination of reducing compounds other than formic acid*

11.5.2.1 *Test portion.* Using the weighing pipette (**11.4.4**), weigh, to the nearest 0.01 g, approximately 10 g of the laboratory sample.

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[1] 1 mbar = 100 N/m^2 = 100 Pa.

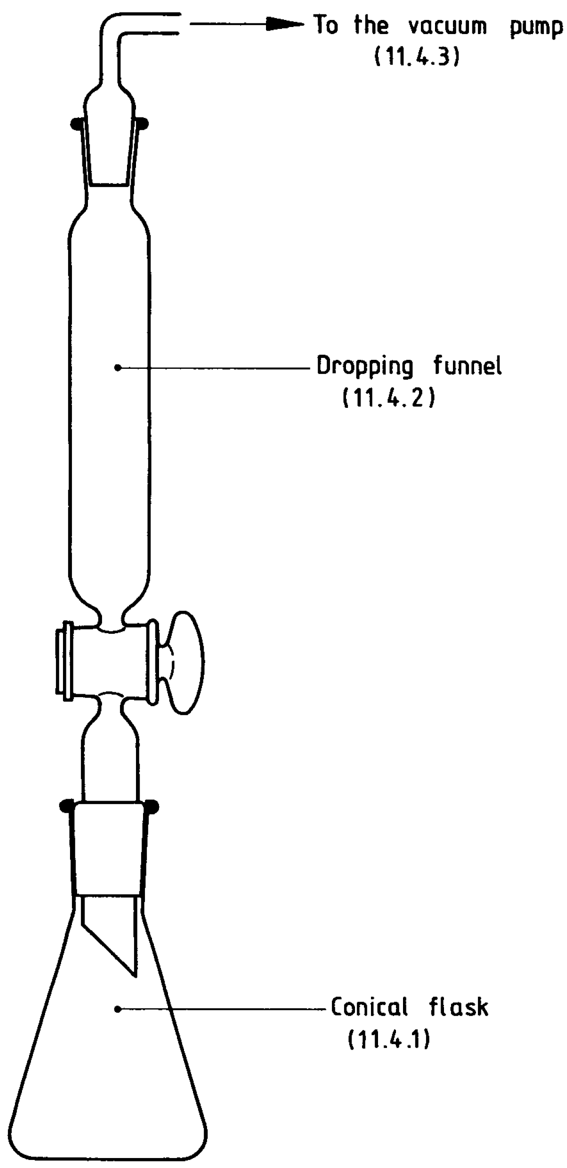


Figure 1 — Apparatus for determination of formic acid content

11.5.2.2 *Blank test.* Carry out a blank test following the same procedure as for the determination (**11.5.2.3**), using the same amounts of all reagents [except the sodium thiosulphate solution (**11.3.6**)] as used for the determination, but replacing the test portion with 10 mL of water.

11.5.2.3 *Determination.* Introduce 90 mL of water and 25.0 mL of the potassium bromide-bromate solution (**11.3.5**) into a flask (**11.4.1**) and fit the dropping funnel (**11.4.2**) to it. Open the stopcock of the funnel and connect the funnel to the vacuum pump (**11.4.3**). Evacuate the flask to produce a residual pressure sufficient for the intake of at least 200 mL of liquid. Close the stopcock of the funnel and disconnect the vacuum pump.

Introduce the test portion (**11.5.2.1**) into the funnel. Open the stopcock sufficiently to draw most of the test portion into the flask and close it again.

NOTE It is essential that, on each addition to the flask, sufficient liquid is left in the funnel to form a seal and prevent the release of the vacuum.

Rinse down the inside of the funnel with two 5 mL portions of water, each time drawing most of the water into the flask by opening and closing the stopcock.

Introduce 10 mL of the hydrochloric acid solution (**11.3.2**) into the funnel and draw most of the solution into the flask. Repeat the rinsing with two 5 mL portions of water, drawing most of the water into the flask. Mix and allow to stand at room temperature for about 10 min.

Then introduce, via the funnel, 5 mL of the potassium iodide solution (**11.3.3**), followed by 50 mL of water and mix. Open the stopcock to release the vacuum and disconnect the funnel. Titrate the liberated iodine with the sodium thiosulphate solution (**11.3.6**) to a pale yellow colour. Add about 2 mL of the starch solution (**11.3.7**) and continue the titration until the blue colour is discharged.

11.6 Expression of results

The formic acid content F, expressed as a percentage by mass, is given by the equation:

$$F = \frac{V_1 - V_2}{M_1} - \frac{V_3 - V_4}{M_2} \times C \times 0.023 \times 100$$

where

V_1 is the volume (in mL) of the sodium thiosulphate solution used for the titration of the first blank;

V_2 is the volume (in mL) of the sodium thiosulphate solution used for the determination of total reducing matter;

V_3 is the volume (in mL) of the sodium thiosulphate solution used for the titration of the second blank;

V_4 is the volume (in mL) of the sodium thiosulphate solution used for the determination of reducing compounds other than formic acid;

C is the actual concentration (in mol/L) of the sodium thiosulphate solution;

M_1 is the mass (in g) of the test portion taken for the determination of total reducing matter;

M_2 is the mass (in g) of the test portion taken for the determination of reducing compounds other than formic acid;

0.023 is the mass (in g) of formic acid corresponding to 1.00 mL of sodium thiosulphate solution, $c(Na_2S_2O_3) = 1.000$ mol/L.

12 Determination of acetaldehyde monomer content

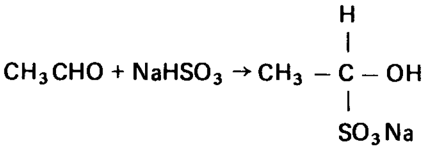
NOTE This method is applicable to products having acetaldehyde monomer contents equal to or greater than 0.01 % (*m*/*m*).

12.1 Principle

The acetaldehyde present in a test portion is reacted with an excess of sodium hydrogen sulphite solution. The residual sodium hydrogen sulphite is determined by iodometric titration.

12.2 Reactions

The following reactions occur during the test.



$$CH_3CHO + NaHSO_3 \rightarrow CH_3-\underset{SO_3Na}{\overset{H}{C}}-OH$$

$$NaHSO_3 + I_2 + H_2O \rightarrow NaHSO_4 + 2HI$$

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

12.3 Reagents

12.3.1 *General.* During the analysis, use only reagents of recognized analytical grade and only water complying with grade 3 of BS 3978.

12.3.2 *Sodium hydrogen sulphite,* 18.2 g/L solution.

Dissolve 1.66 g of disodium disulphite ($Na_2S_2O_5$) in water and dilute to 100 mL.

Prepare this solution immediately before use.

12.3.3 *Iodine,* standard volumetric solution, $c(1/2\ I_2) = 0.02$ mol/L.

12.3.4 *Sodium thiosulphate,* standard volumetric solution, $c(Na_2S_2O_3) = 0.02$ mol/L, approximately.

Standardize this solution immediately before use.

12.3.5 *Starch solution.* Triturate 1.0 g of soluble starch with 5 mL of water and add the mixture, while stirring, to 100 mL of boiling water. Boil for a few minutes and allow to cool.

Discard this solution after 2 weeks.

12.4 Apparatus

Ordinary laboratory apparatus together with the following.

12.4.1 *Weighing pipette,* of capacity 20 mL.

12.4.2 *Conical flasks,* of capacity 250 mL, fitted with ground glass stoppers.

12.4.3 *Burettes,* of capacity 10 mL, complying with class A of BS 846.

12.5 Procedure

12.5.1 *Test portion.* Using the weighing pipette (**12.4.1**), weigh, to the nearest 0.01 g, approximately 10 mL of the laboratory sample.

12.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure (**12.5.3**), using the same amounts of all reagents [except the sodium thiosulphate solution (**12.3.4**)] as used for the determination, but omitting the test portion.

12.5.3 *Determination.* Transfer the test portion (**12.5.1**) to a 50 mL one-mark volumetric flask containing 10 mL of water. Add 5.0 mL of the sodium hydrogen sulphite solution (**12.3.2**) from a burette (**12.4.3**). Dilute to the mark with water, mix and allow to stand for about 30 min. At the same time as preparing this solution, transfer 50.0 mL of the iodine solution (**12.3.3**) into a conical flask (**12.4.2**) and allow it to stand in a bath containing a mixture of ice and water.

At the end of the 30 min period, transfer 20.0 mL of the solution in the volumetric flask to the conical flask containing the iodine solution. Titrate with the sodium thiosulphate solution (**12.3.4**), from a second burette (**12.4.3**), to a pale yellow colour. Add 0.5 mL of the starch solution (**12.3.5**) and continue the titration until the blue colour is discharged.

12.6 Expression of results

The acetaldehyde (CH_3CHO) monomer content D, expressed as a percentage by mass, is given by the equation:

$$D = 0.022(V_2 - V_1) \times C \times \frac{50}{20} \times \frac{100}{M}$$

$$= \frac{5.5(V_2 - V_1)C}{M}$$

where

V_1 is the volume (in mL) of the sodium thiosulphate solution used for the blank test;

V_2 is the volume (in mL) of the sodium thiosulphate solution used for the determination;

C is the actual concentration (in mol/L) of the sodium thiosulphate solution;

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

0.022 is the mass (in g) of acetaldehyde corresponding to 1.00 mL of sodium thiosulphate solution, $c(Na_2S_2O_3) = 1.000$ mol/L.

13 Determination of total acetaldehyde content

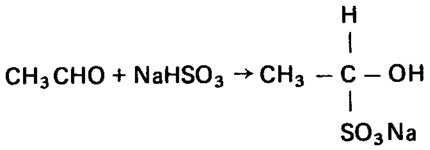
NOTE This method is applicable to products having total acetaldehyde (polymerized and monomeric acetaldehyde) contents equal to or greater than 0.004 % (*m*/*m*).

13.1 Principle

A test portion is heated in an acid medium to depolymerize any paraldehyde present and to entrain, by distillation, both regenerated acetaldehyde and the monomeric acetaldehyde originally present. The acetaldehyde in the distillate reacts with an excess of sodium hydrogen sulphite solution. The residual sodium hydrogen sulphite is determined by iodometric titration.

13.2 Reactions

The following reactions occur during the test.



$$CH_3CHO + NaHSO_3 \rightarrow CH_3 - \underset{SO_3Na}{\overset{H}{C}} - OH$$

$$NaHSO_3 + I_2 + H_2O \rightarrow NaHSO_4 + 2HI$$

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

13.3 Reagents

13.3.1 *General.* During the analysis, use only reagents of recognized analytical grade and only water complying with grade 3 of BS 3978.

13.3.2 *Sodium hydrogen sulphite,* 12.6 g/L solution.

Dissolve 1.15 g of disodium disulphite ($Na_2S_2O_5$) in water and dilute to 100 mL.

Prepare this solution immediately before use.

13.3.3 *Phosphoric acid,* ρ approximately 1.70 g/mL, about 85 % (*m*/*m*) solution.

13.3.4 *Iodine,* standard volumetric solution, $c(1/2\ I_2) = 0.1$ mol/L.

13.3.5 *Sodium thiosulphate,* standard volumetric solution, $c(Na_2S_2O_3) = 0.1$ mol/L, approximately.

Standardize this solution immediately before use.

13.3.6 *Starch solution.* Triturate 1.0 g of soluble starch with 5 mL of water and add the mixture, while stirring, to 100 mL of boiling water. Boil for a few minutes and allow to cool.

Discard this solution after 2 weeks.

13.4 Apparatus

Ordinary laboratory apparatus together with the following.

13.4.1 *Weighing pipette,* of capacity 20 mL.

13.4.2 *Distillation apparatus,* as shown in Figure 2, with ground glass joints, consisting of the following items:

a) distillation flask, of capacity 250 mL, of borosilicate glass;

b) splash-head adapter with a recovery bend;

c) water condenser;

d) receiver adapter.

13.4.3 *Conical flask,* of capacity 200 mL, graduated at 50 mL and 100 mL.

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

13.5 Procedure

13.5.1 *Test portion.* Using the weighing pipette (**13.4.1**), weigh, to the nearest 0.0001 g, approximately 10 mL of the laboratory sample.

13.5.2 *Blank test.* Carry out a blank test at the same time as the determination, following the same procedure (**13.5.3**), using the same amounts of all reagents [except the sodium thiosulphate solution (**13.3.5**)] as used for the determination, but omitting the test portion.

13.5.3 *Determination.* Transfer the test portion (**13.5.1**) to the distillation flask [**13.4.2** a)] containing 100 mL of water. Add 2 g of the phosphoric acid solution (**13.3.3**) to the distillation flask and place 40 mL of water and 10.0 mL of the sodium hydrogen sulphite solution (**13.3.2**) in the conical flask (**13.4.3**). Assemble the apparatus as shown in Figure 2, partially immersing the conical flask in a bath containing a mixture of ice and water.

Heat the distillation flask so that its contents are gently boiling after a few minutes and then slowly distil about 50 mL of liquid into the conical flask. Remove the source of heat, disconnect the distillation flask and remove the bath of ice and water. Rinse the inner walls of the condenser and of the receiver adapter with water, collecting the washings in the conical flask and allow to stand at ambient temperature for 30 min.

Add 30.0 mL of the iodine solution (**13.3.4**) to the conical flask and titrate the excess of iodine with the sodium thiosulphate solution (**13.3.5**), from the burette (**13.4.4**), to a pale yellow colour. Add 0.5 mL of the starch solution (**13.3.6**) and continue the titration until the blue colour is discharged.

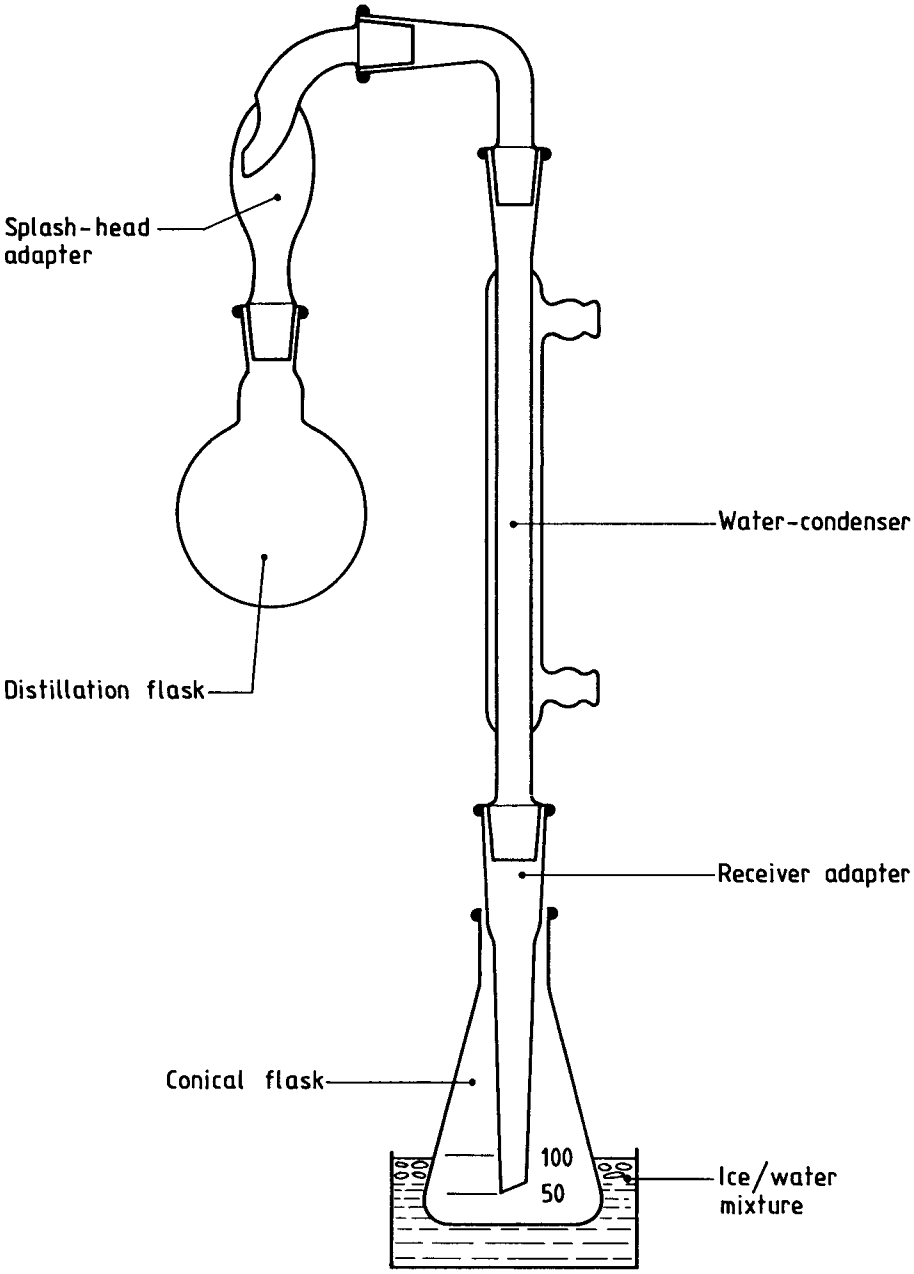


Figure 2 — Apparatus for determination of total acetaldehyde content

13.6 Expression of results

The total acetaldehyde (CH_3CHO) content E, expressed as a percentage by mass, is given by the equation:

$$E = 0.022\ (V_2 - V_1) \times C \times \frac{100}{M}$$

$$= \frac{2.2\ (V_2 - V_1) \times C}{M}$$

where

V_1 is the volume (in mL) of the sodium thiosulphate solution used for the blank test;

V_2 is the volume (in mL) of the sodium thiosulphate solution used for the determination;

C is the actual concentration (in mol/L) of the sodium thiosulphate solution;

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

0.022 is the mass (in g) of acetaldehyde corresponding to 1.00 mL of sodium thiosulphate solution, $c(Na_2S_2O_3) = 1.000$ mol/L.

14 Determination of water content

Determine the water content by one of the methods described in BS 2511.

15 Determination of arsenic content

15.1 General

Determine the arsenic content by the method described in BS 4404 with the additional reagents specified in **15.2** and following the instructions in **15.3** for the test portion and preparation of test solution.

15.2 Reagents

Use the reagents specified in clause **5** of BS 4404:1968 together with the following.

15.2.1 *Hydrogen peroxide,* approximately 100 g/L solution.

15.2.2 *Sulphuric acid,* approximately 200 g/L solution.

15.3 Test portion and preparation of test solution (see **6.2.1** and **6.2.2** of BS 4404:1968).

Weigh, to the nearest 0.001 g, a quantity of the laboratory sample containing 1 µg to 10 µg of arsenic (generally 25 g). Place the test portion in a beaker containing 50 mL of water. Add 5 mL of the hydrogen peroxide solution (**15.2.1**) and evaporate almost to dryness on a sand bath. Carefully add 5 mL of the concentrated sulphuric acid [**5.2** 2) of BS 4404:1968] and evaporate, in a well-ventilated fume cupboard, until white fumes are evolved. Dissolve the residue in approximately 5 mL of water and transfer the solution quantitatively to the conical flask [**4.1** 1) of BS 4404:1968] using the sulphuric acid solution (**15.2.2**) for the washings. Make up the volume to about 40 mL with the same sulphuric acid solution.

16 Test reports

The test report, for each determination, shall contain the following information:

a) a complete identification of the sample;

b) a reference to the British Standard method used, e.g. clause **12** of BS 576-2:1987;

c) the results, and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in the appropriate clause of this standard or in the British Standards to which reference is made, or regarded as optional.

Appendix A Relationship between BS 576-2 and International Standards

The relationship between the methods described in BS 576-2 and International Standards is given in Table 5.

Table 5 — Relationship between the methods described in BS 576-2 and International Standards describing methods of test for acetic acid

BS 576-2 clause reference	Corresponding International Standard	Subject	Relationship of BS 576-2 to international test method
2	ISO 753-1	Sampling	Related
3, **4**, **6**, **14**, **15**	ISO 753-1	General methods by cross reference	Related (extends technical content of ISO 753-1)
5	ISO 753-2	Acetic acid content	Technically equivalent
7	None	Iron content by general method	—
8	ISO 753-8	Limit test for inorganic chlorides	Technically equivalent
9	ISO 753-9	Limit test for inorganic sulphates	Technically equivalent
10	ISO 753-10	Limit test for heavy metals (including iron)	Technically equivalent
11	ISO 753-3	Formic acid content	Technically equivalent
12	ISO 753-4	Acetaldehyde monomer content	Technically equivalent
13	ISO 735-5	Total acetaldehyde content	Technically equivalent

The relationship between those British Standards describing general test methods which are referred to in BS 576-2 and corresponding International Standards is given in Table 6.

Table 6 — Relationship between those British Standards describing general test methods which are referred to in BS 576-2 and corresponding International Standards

British Standard	Corresponding International Standard	Subject	Relationship of British Standard to international test method
BS 2511	ISO 760	Water content	Related
BS 4404	ISO 2590	Arsenic content	Technically equivalent
BS 4524	ISO 759	Residue on evaporation on a water bath	Identical
BS 4633	ISO 1392	Crystallizing point	Related
BS 5339	ISO 2211	Colour measurement	Identical
NOTE These International Standards are the general test methods described in ISO 763-1 as being applicable to acetic acid.			

Appendix B Relationship between crystallizing point and acetic acid content

A graph of the relationship between crystallizing point and acetic acid content is shown in Figure 3. This graph is based on the assumption that water is the only impurity present.

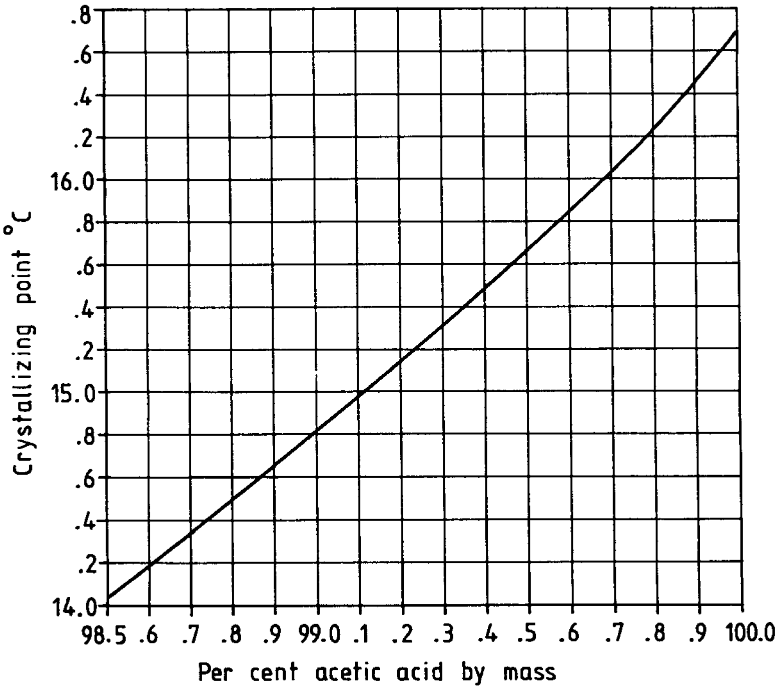


Figure 3 — Relationship between crystallizing point and acetic acid content

Publications referred to

BS 576, *Acetic acid for industrial use*[2].

BS 576-1, *Specification for acetic acid.*

BS 593, *Specification for laboratory thermometers.*

BS 846, *Specification for burettes.*

BS 2511, *Methods for the determination of water (Karl Fischer method).*

BS 3591, *Specification for industrial methylated spirits.*

BS 3978, *Specification for water for laboratory use.*

BS 4404, *Method for the determination of arsenic (silver diethyldithiocarbamate procedure).*

BS 4524, *Method for determination of residue on evaporation on a water bath*[3].

BS 4633, *Method for the determination of crystallizing point.*

BS 5309, *Methods for sampling chemical products.*

BS 5309-1, *Introduction and general principles.*

BS 5309-3, *Sampling of liquids.*

BS 5339, *Method of measurement of colour in Hazen units (platinum-cobalt scale) of liquid chemical products*[3].

BS 6337, *General methods of chemical analysis.*

BS 6337-3, *Method for determination of iron content (1,10-phenanthroline spectrophotometric method).*

ISO 753, *Acetic acid for industrial use — Methods of test.*

ISO 760, *Determination of water — Karl Fischer method (General method).*

ISO 1392, *Determination of crystallizing point — General method.*

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method.*

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[2] Referred to in the foreword only.

[3] ISO 759 and ISO 2211, referred to in Table 6 are identical with BS 4524 and BS 5339 respectively.

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