BRITISH STANDARD

BS 509-2:1984

Incorporating Amendment No. 1

Acetone for industrial use —

Part 2: Methods of test

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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/4 upon which the following bodies were represented:

Chemical Industries Association
Ministry of Defence
Oil and Colour Chemists' Association
Royal Society of Chemistry
Society of Chemical Industry

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

British Pharmacopoeia Commission
British Society of Perfumers
Cosmetic, Toiletry and Perfumery Association Limited
Department of Trade and Industry (Laboratory of the Government Chemist)

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on
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5446	January 1987	Indicated by a sideline in the margin

Contents

Foreword

This Part of this British Standard has been prepared under the direction of the Chemicals Standards Committee and provides a comprehensive series of test methods for acetone for industrial use. Although applicable to the material in general, it includes all the test methods required to assess compliance with BS 509-1.

In preparing this standard, the opportunity has been taken to implement, without technical alteration, the International Standards describing test methods for acetone. 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 757 and constitute the revision of ISO Recommendation R 757. Table 1 gives the relationship between International Standards and this British Standard, together with the relationship between general test methods and corresponding International Standards.

Subsequent International Standards in the ISO 757 series, if approved by the UK, will be published as additions to this British Standard, without technical alteration.

This Part of this standard specifies methods of test only and should not be used as a specification defining limits of purity. Reference to this Part of this standard should indicate that the methods of test used are in accordance with the appropriate clause(s) of BS 509-2.

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 4, 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 509 describes methods for testing acetone for industrial use.

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

2 Sampling[1)]

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 Determination of distillation characteristics

Determine the distillation characteristics by the method described in BS 4591:1971, except that the following thermometer and temperature corrections should be used.

a) *Thermometer* (**3.2** of BS 4591:1971). Use a thermometer No. F 75C/1000 complying with the requirements of BS 593.

b) *Corrections to be applied to observed temperatures* (**7.2** of BS 4591:1971). If the corrected barometric pressure deviates from 1 013 mbar[2)] apply a correction to the observed temperature by subtracting 0.029 °C for every millibar above, or adding 0.029 °C for every millibar below, 1 013 mbar.

4 Determination of water content

Determine the water content using the method described in clause **4** of BS 2511:1970, using as test portions (see **4.4.3** of BS 2511:1970) 20 mL of the material mixed with 100 mL of dry pyridine.

5 Determination of density at 20 °C

Determine the density at 20 °C by the method described in BS 4522.

6 Determination of dry residue after evaporation on a water bath

Determine the dry residue after evaporation by the method described in BS 4524.

7 Determination of acidity to phenolphthalein

7.1 Field of application. This method is applicable to products having acidities, expressed as acetic acid (CH_3 COOH), equal to or greater than 0.0006 % (*m*/*m*).

7.2 Principle. Dilution of a test portion with carbon dioxide-free water.

Titration of the test solution, if acid, with standard volumetric sodium hydroxide solution, using phenolphthalein as indicator.

7.3 Reagents

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

7.3.2 *Water, carbon dioxide-free.* Boil distilled water and allow it to cool in a flask fitted with a stopper carrying a soda-lime guard tube.

7.3.3 *Sodium hydroxide,* standard volumetric solution, *c* (NaOH) = 0.1 mol/L.

7.3.4 *Phenolphthalein,* 5 g/L ethanolic solution.

Dissolve 0.5 g of phenolphthalein in 100 mL of 95 % (*V*/*V*) ethanol and add the sodium hydroxide solution (**7.3.3**) until a pale pink coloration is obtained.

NOTE The ethanol may be replaced for this purpose by industrial methylated spirits, 95 % (*V*/*V*) complying with BS 3591. It should be noted that 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.

7.4 Apparatus

7.4.1 *Ordinary laboratory apparatus*

7.4.2 *Conical flask,* of capacity 250 mL, of borosilicate glass, fitted with a ground glass stopper carrying a soda-lime guard tube.

7.4.3 *Burette,* of capacity 10 mL, graduated in 0.02 mL divisions.

7.5 Procedure

7.5.1 *Test portion.* Take 100 ± 0.1 mL of the laboratory sample.

7.5.2 *Determination.* Place 80 mL of the water (**7.3.2**) in the conical flask (**7.4.2**), add 0.5 mL of the phenolphthalein solution (**7.3.4**) and make faintly pink by the addition of a few drops of the sodium hydroxide solution (**7.3.3**).

[1)] Additional guidance is given in BS 5309-1 and BS 5309-3.

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

Add the test portion (**7.5.1**) and a further 0.5 mL of the phenolphthalein solution (**7.3.4**). If the solution is acid (colourless), titrate it with the sodium hydroxide solution (**7.3.3**), stoppering the flask and swirling its contents after each addition, until a pink coloration, persisting for about 15 s, is obtained.

7.6 Expression of results. The acidity, expressed as acetic acid (CH_3 COOH), in % (*m*/*m*), is given by the formula

$$\frac{0.006\ V_1}{\rho}$$

where

V_1 is the volume, in mL, of the sodium hydroxide solution (**7.3.3**) used for the determination;

ρ is the density, in g/mL, of the sample at 20 °C (determined by method described in BS 4522);

0.006 is the mass, in g, of acetic acid corresponding to 1.00 mL of sodium hydroxide solution, *c* (NaOH) = 0.100 mol/L.

NOTE If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

8 Test for miscibility with water

8.1 Principle. Addition of water to a test portion, under specified conditions, and examination for opalescence or turbidity.

8.2 Reagents. During the test, use only water complying with BS 3978.

8.3 Apparatus

8.3.1 *Ordinary laboratory apparatus*

8.3.2 *Two matched Nessler cylinders,* of capacity 100 mL, similar in every respect.

8.4 Procedure

8.4.1 *Test portion.* Take, by means of a safety pipette, 5 mL of the laboratory sample at a temperature of about 20 °C and transfer to one of the Nessler cylinders (**8.3.2**).

8.4.2 *Test.* Add slowly, with thorough mixing, 95 mL of water to the test portion (**8.4.1**) in the Nessler cylinder. Examine for opalescence during the addition. Adjust the temperature of the mixture to 20 °C.

Examine vertically for opalescence or turbidity against a black background with side illumination, using as a standard the second cylinder containing 100 mL of water.

8.5 Expression of results. Report the formation of opalescence or turbidity or whether the solution remained clear.

9 Determination of permanganate time

9.1 Definition

For the purposes of this clause of BS 509-2, the following definition applies.

9.1.1
permanganate time

the number of minutes required, after adding 2 mL of 0.2 g/L potassium permanganate solution to 50 mL of the sample, for the colour to match that of a colour standard

9.2 Principle. Addition to a test portion, under specified conditions, of potassium permanganate solution. Determination of the time taken for the colour of this test solution to match that of a cobalt(II) chloride and uranyl nitrate colour standard.

9.3 Reagents

9.3.1 *General.* During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only water complying with BS 3978.

9.3.2 *Potassium permanganate,* 0.2 g/L *solution.* Use water previously boiled for 30 min with sufficient dilute potassium permanganate solution to give a stable faint pink coloration. Cool the water to ambient temperature before preparation of the solution.

Prepare this solution immediately before use and protect it from light.

9.3.3 *Cobalt(II) chloride and uranyl nitrate,* colour standard. To 5 mL of a 50 g/L solution of cobalt(II) chloride hexahydrate ($CoCl_2.6H_2O$), add 7 mL of a 40 g/L solution of uranyl nitrate hexahydrate [$UO_2\ (NO_3)_2.6H_2O$], and dilute with water to 50 mL.

Prepare this solution on the day of use.

9.4 Apparatus

NOTE Clean the glassware used so as to avoid any risk of contamination.

9.4.1 *Ordinary laboratory apparatus*

9.4.2 *Water bath,* capable of being controlled at 25 ± 0.2 °C.

9.4.3 *Two matched cylinders,* of capacity 100 mL, of colourless transparent glass, graduated at 50 mL and fitted with ground glass stoppers.

9.4.3 *Pipette,* of capacity 2 mL.

0.1 Procedure

0.1.1 *Test portion.* Carry out the test as soon as possible after receipt of the sample.

Rinse one of the cylinders (**9.4.3**), first with 15 mL to 20 mL of hydrochloric acid, of density approximately 1.19 g/mL, about 38 % (*m*/*m*) solution, then six times with tap water, twice with distilled water and finally with some of the laboratory sample.

Immediately fill the cylinder to the mark with more of the laboratory sample at a temperature of about 25 °C.

0.1.2 *Determination.* Rinse the second cylinder (**9.4.2**) as specified in **0.1.1** but omitting the last rinse with the laboratory sample.

Fill the cylinder to the mark with the colour standard solution (**9.3.3**). Place the cylinder containing the test portion (**0.1.1**) in the water bath (**9.4.2**), controlled at 25 ± 0.2 °C, so that the water level in the bath is approximately 25 mm below the neck of the cylinder. After 15 min, remove the cylinder from the water bath and, using the pipette (**9.4.4**), add 2.0 mL of the potassium permanganate solution (**9.3.2**). Note the time. Immediately stopper the cylinder, shake, and replace in the water bath.

Remove the cylinder from the water bath, from time to time, and compare the colour, viewing vertically downwards against a white background, with the colour standard. Towards the end of the determination, compare the colour at intervals of 1 min. Avoid exposing the test solution to strong daylight.

Note the time when the colour of the test solution matches that of the colour standard.

0.2 Expression of results. Report the time, in minutes, from the addition of the potassium permanganate solution, for the colour of the test solution to match that of the colour standard.

1 Control test with Agulhon's reagent

1.1 Principle. Treatment of a test portion with Agulhon's reagent under specified conditions. The presence of certain impurities, in particular alcoholic ones, is indicated by a blue or violet coloration.

1.2 Reagents

1.2.1 *General.* During the test, use only reagents of recognized analytical grade and water complying with BS 3978.

1.2.2 *Agulhon's reagent.* Dissolve 0.50 g of potassium dichromate in 30 mL of water and add about 65 mL of nitric acid solution, of density approximately 1.40 g/mL. Cool the solution to about 20 °C and dilute to 100 mL with more of the nitric acid solution.

1.3 Apparatus

1.3.1 *Ordinary laboratory apparatus*

1.4 Procedure

1.4.1 *Test portion.* Take by means of a safety pipette, 1 mL of the laboratory sample.

1.4.2 *Test.* Place the test portion (**1.4.1**) in a test tube and add 3 mL of the Agulhon's reagent (**1.2.2**). Mix and allow the solution to stand at approximately 15 °C for 5 min.

After this period, examine the colour of the solution.

1.5 Expression of results. If no blue or violet colour appears, report the absence of alcoholic impurities.

2 Measurement of colour

Determine the colour by the method described in BS 5339.

3 Test reports

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

a) an identification of the sample;

b) the reference to the method used;

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.

4 Detection of alkalinity

4.1 Principle. Neutralization of water using bromothymol blue as indicator and the transfer of equal volumes to each of two glass cylinders. Mixing of a test portion with the water in one cylinder and comparison of its colour with that of the blank.

4.2 Reagents

4.2.1 *General.* During the test, use only reagents of recognized analytical grade and water complying with BS 3978.

Table 1 — Methods of test for acetone: relationship between British Standard and international series

a) Relationship between BS 509-2 and International Standards

Clause no.	Corresponding International Standard no.	Subject	Relationship of BS 509-2 to international test method
2	757/1	Sampling	Related
3, **4**, **5**, **6**, **2**	757/1	General methods by cross reference	Related (extends technical content of ISO 757/1)
7	757/2	Acidity to phenolphthalein	Technically equivalent
8	757/3	Miscibility with water	Technically equivalent
9	757/4	Permanganate test	Technically equivalent
1	757/5	Control test with Agulhon's reagent	Technically equivalent

b) Relationship between British Standards describing general test methods and corresponding International Standards[a]

BS no.	Corresponding International Standard no.[a]	Subject	Relationship of British Standard to international test method
2511	760	Water content	Related
4522	758	Density at 20 °C	Technically equivalent
4524	759	Residue on evaporation on a water bath	Identical
4591	918	Distillation characteristics	Related
5339	2211	Colour measurement	Identical

[a] These International Standards are the general test methods specified in ISO 757/1 as being applicable to acetone.

4.2.2 *Hydrochloric acid,* 0.73 g hydrogen chloride per litre of solution.

4.2.3 *Sodium hydroxide,* 0.80 g/L *solution.*

4.2.4 *Bromothymol blue indicator,* 0.4 g/L *solution.* Warm 0.1 g of bromothymol blue with 3.2 mL of 2 g/L sodium hydroxide solution and 5 mL of 95 % (*V*/*V*) ethanol. When dissolved, add 50 mL of 95 % (*V*/*V*) ethanol and dilute to 250 mL with water.

NOTE The ethanol may be replaced for this purpose by industrial methylated spirits, 95 % (*V*/*V*) complying with BS 3591. It should be noted that 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.

4.3 Procedure. To 200 mL of freshly boiled and cooled water add two drops (approximately 0.1 mL) of the bromothymol blue indicator solution (**4.2.4**) and adjust to the neutral colour of the indicator by addition of the hydrochloric acid solution (**4.2.2**) or the sodium hydroxide solution (**4.2.3**) as necessary. Transfer 100 mL of the neutralized water to each of two 250 mL glass cylinders. To one of these add 100 mL of the sample and mix. Compare the colour of the mixture containing the sample with that of the blank, viewing both downwards to compensate for dilution.

Note if any change of colour towards blue, indicating the presence of alkalinity, takes place.

Publications referred to

BS 509, *Acetone for industrial use.*

BS 509-1, *Specification for acetone.*

BS 593, *Laboratory thermometers.*

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

BS 3591, *Industrial methylated spirits.*

BS 3978, *Water for laboratory use.*

BS 4522, *Method for the determination of density of liquids at 20 °C.*

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

BS 4591, *Method for the determination of distillation characteristics.*

BS 5309, *Method 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.*

ISO 757, *Acetone for industrial use — Methods of test.*

ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 degrees C.*

ISO 759, *Volatile organic liquids for industrial use — Determination of dry residue after evaporation on water bath — General method.*

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

ISO 918, *Volatile organic liquids for industrial use — Determination of distillation characteristics.*

ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum-cobalt scale).*

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