Incorporating Amendment No. 1

Specification for

# isoPropyl acetate

Confirmed January 2011



## 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 scientific and industrial organizations:

Board of Trade

British Iron & Steel Federation

Chemical Industries Association\*

Fertilizer Manufacturers' Association

Gas Council

Institute of Vitreous Enamellers

Institution of Gas Engineers

Ministry of Health

National Sulphuric Acid Association

Royal Institute of Public Health & Hygiene

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

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**British Plastics Federation** 

Ministry of Defence, Army Department

Ministry of Defence, Navy Department

Ministry of Technology — Laboratory of the Government Chemist

Oil and Colour Chemists' Association

Paintmakers Association of Great Britain

Pharmaceutical Society of Great Britain

Research Association of British Paint, Colour & Varnish Manufacturers

Royal Institute of Chemistry

Society of Chemical Industry

Society of Motor Manufacturers and Traders Ltd.

Toilet Preparations Federation

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 22 February 1968

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Committee references CIC/4, CIC/4/5 and CIC/51
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## **Foreword**

This standard makes reference to the following British Standards:

BS 593, Laboratory thermometers.

BS 612, Nessler cylinders.

BS 658, Apparatus for the determination of distillation range (including flasks and receivers).

BS 1792, One-mark volumetric flasks.

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

BS 3591, Industrial methylated spirits.

BS 3978, Water for laboratory use.

BS 4591, Method for determination of distillation characteristics of organic liquids (other than petroleum products).

This standard forms one of a series of British Standards for solvents and allied products, the preparation of which was authorized originally by the Fine Chemicals Industry Standards Committee (now merged in the Chemicals Industry Standards Committee).

This British Standard was first issued in 1952. In the present revision the limits for relative density, water content, residue on evaporation, acidity, and ester content have been made more stringent. The requirements in respect of distillation range have been made more stringent, and now specify initial boiling point and dry point. A specific colour requirement has been included.

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#### 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.

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#### 1 Scope

This British Standard specifies requirements for *iso*propyl acetate suitable for industrial purposes.

#### 2 Description

British Standard *iso*propyl acetate shall be clear, and free from matter in suspension, and shall consist essentially of the acetic ester of propan-2-ol, CH<sub>3</sub>COOCH(CH<sub>3</sub>)<sub>2</sub>.

#### 3 Colour

The colour of the material shall not exceed 10 Hazen units when measured by the method described in Appendix A or by a suitable instrumental method.

#### 4 Relative density

The relative density<sup>1)</sup> of the material at any one of the following temperatures shall be within the appropriate values as shown:

Temperature	Relative density	
°C	min.	max.
20/4 20/20 25/25	0.870 0.872 0.867	0.872 0.874 0.869

#### 5 Distillation range

When the material is distilled by the method described in Appendix B, the initial boiling point at 760 mmHg pressure shall not be below 87.0  $^{\circ}$ C and the dry point at 760 mmHg pressure shall not be above 90.5  $^{\circ}$ C.

#### 6 Water

The material shall not contain more than 0.1 % by mass of water, determined by the method described in clause **2** of BS 2511:1970<sup>2)</sup> and using 20 ml of the material.

#### 7 Residue on evaporation

The material shall not leave more than 50 parts per million by mass of residue when tested by the method described in Appendix C.

#### 8 Acidity

The material shall not contain more than 50 parts per million by mass of acid, calculated as acetic acid,  $CH_3COOH$ , and determined by the method described in Appendix D.

#### 9 Ester content

The material shall show an ester content of not less than 98.5 % by mass, calculated as *iso* propyl acetate, CH<sub>3</sub>COOCH(CH<sub>3</sub>)<sub>2</sub>, and determined by the method described in Appendix E.

#### 10 Sample

A representative sample of the material measuring not less than one litre shall be taken from the bulk for the purpose of examination in accordance with this specification. The sample shall be placed in a clean, dry and airtight glass stoppered glass bottle of such a size that it is nearly filled by the sample.

When it is necessary to seal the container, care shall be taken to avoid the risk of contaminating the contents in any way.

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2) BS 2511:1970, Methods for the determination of water (Karl Fischer method).

<sup>1)</sup> Ratio of density of liquid at a specified temperature to density of water at a specified temperature.

#### Appendix A Limit test for colour

#### A.1 Apparatus

The following apparatus shall be used:

- 1) Two matched Nessler cylinders<sup>3)</sup>, 100 ml capacity.
- 2) One-mark volumetric flask<sup>4)</sup>, 500 ml capacity.
- 3) One-mark volumetric flask<sup>4)</sup>, 250 ml capacity.

#### A.2 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978<sup>5)</sup> shall be used throughout.

- 1) Cobaltous chloride, hexahydrate.
- 2)  $Hydrochloric\ acid$ , concentrated, d = 1.18.
- 3) Chloroplatinic acid reagent. Dissolve 250 mg of platinum in a small quantity of aqua regia contained in a glass or porcelain basin by heating on a water bath. When the metal has dissolved evaporate the solution to dryness. Add 1 ml of the hydrochloric acid and again evaporate to dryness. Repeat this operation twice more.

#### A.3 Preparation of colour standard

Dissolve 0.50 g of the cobaltous chloride hexahydrate and the whole of the chloroplatinic acid (prepared as described above) in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500 ml volumetric flask. Dilute with water to the mark.

Pipette 5.0 ml of this solution into the 250 ml volumetric flask. Dilute with water to the mark. This diluted solution has a colour of 10 Hazen units and should always be freshly prepared.

#### A.4 Procedure

Fill one of the Nessler cylinders to the mark with the sample, and the other with the colour standard. Using a white background compare the colours.

#### Appendix B Method for the determination of distillation range

#### **B.1** Apparatus

- 1) The apparatus required is described in BS 658<sup>6)</sup> and comprises:
- 1) Distillation flask of 100 ml distillation capacity complying with the requirements of BS 658<sup>6</sup>).
- 2) Thermometer, No. F100C/100, complying with the requirements of BS 593<sup>7</sup>).
- 1) Condenser, Type 1, complying with the requirements of BS 658<sup>6</sup>).
- 1) Draught screen, Type B, complying with the requirements of BS 658<sup>6</sup>).

#### **B.2 Procedure**

1) Assemble the apparatus as described in BS  $658^6$ ). Measure 100 ml of the sample into the distillation flask from a graduated measuring cylinder and add a few small anti-bumping granules. Place the flask, thermometer and a suitable receiver in position and ensure that the condenser has a steady supply of water. Adjust the rate of heating so that the first drop of distillate falls from the end of the condenser in 10 minutes to 15 minutes. Read the temperature at the instant the first drop falls from the end of the condenser and record as the observed initial boiling point.

Further adjust the rate of heating so that the distillate is collected at the rate of 3 ml to 4 ml per minute. Read the temperature indicated at the instant the last drop of liquid evaporates from the lowest point in the distillation flask and record as the observed dry point. Disregard any liquid on the side of the flask.

 <sup>&</sup>lt;sup>3)</sup> BS 612, "Nessler cylinders".
 <sup>4)</sup> BS 1792, "One-mark volumetric flasks".
 <sup>5)</sup> BS 3978, "Water for laboratory use".

<sup>6)</sup> BS 658, "Apparatus for the determination of distillation range (including flasks and receivers)". 7) BS 593, "Laboratory thermometers".

#### B.3 Corrections to be applied to the observed temperatures

**B.3.1** If the thermometer gives incorrect readings at the observed initial boiling point or observed dry point, correct the readings by subtracting the amount of error if the thermometer is reading high, or adding the amount of error if the thermometer is reading low.

**B.3.2** Read the barometer and correct-the reading as described in clause 8 of BS 4591:1990<sup>8)</sup>.

**B.3.3** If the corrected barometric pressure deviates from 101.3 kPa (760 mmHg) apply corrections to the observed temperatures, as described in **9.1.2** of BS 4591:1990<sup>8)</sup>, by subtracting 0.031  $^{\circ}$ C for every 0.1 kPa above 101.3 kPa, or adding 0.031  $^{\circ}$ C for every 0.1 kPa below 101.3 kPa.

NOTE These corrections are valid only for pressures above 93.3 kPa.

## Appendix C Method for the determination of residue on evaporation

#### C.1 Procedure

Evaporate 100 ml of the sample to dryness in a weighed basin of platinum, silica or borosilicate glass on a boiling water bath. Dry the residue for 30 minutes in an oven at a temperature of  $100 \pm 2$  °C. Cool in a desiccator and weigh.

#### C.2 Calculation

Residue on evaporation,

parts per million by mass = 
$$\frac{M_1 \times 10\ 000}{d}$$

where  $M_1$  = mass, in grammes, of residue and d = relative density of the sample.

## Appendix D Method for the determination of acidity

#### **D.1 Reagents**

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978<sup>9)</sup> shall be used throughout.

- 1) Sodium hydroxide, 0.1N solution.
- 2)  $Neutralized\ ethanol^{10)}$ . Add a few drops of the phenolphthalein indicator to 95 % (v/v) ethanol; and then add 0.01N sodium hydroxide solution until faintly pink; neutralize the ethanol by adding 0.01N hydrochloric acid until the pink colour is just discharged.
- 3) *Phenolphthalein indicator*. Dissolve 0.5 g of phenolphthalein in 100 ml of 95 % (v/v) ethanol<sup>10)</sup> and make faintly pink by the addition of dilute sodium hydroxide solution.

#### **D.2 Procedure**

To 50 ml of the sample add 50 ml of the neutralized ethanol containing a few drops of the phenolphthalein indicator. Titrate with the 0.1N sodium hydroxide solution using a microburette.

#### D.3 Calculation

Acidity, calculated as acetic acid, CH<sub>3</sub>COOH,

parts per million by mass = 
$$\frac{120 \ T_1}{d}$$

where  $T_1$  = volume, in millilitres, of 0.1N sodium hydroxide solution used and d = relative density of the sample.

<sup>8)</sup> BS 4591:1990, Method for determination of distillation characteristics of organic liquids (other than petroleum products).

<sup>&</sup>lt;sup>9)</sup> BS 3978, "Water for laboratory use".

<sup>&</sup>lt;sup>10)</sup> Ethanol may be replaced by industrial methylated spirits, 95 % (v/v), complying with the requirements of BS 3591. It should be noted that the use of industrial 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.

## Appendix E Method for the determination of ester content

#### E.1 Reagents

The reagents used shall be of a recognized analytical reagent quality. Water complying with BS 3978<sup>11)</sup> shall be used throughout and shall be freshly boiled and cooled.

- 1) Potassium hydroxide, approximately N solution in ethanol. 12)
- 2) Hydrochloric acid, N solution.
- 3) *Phenolphthalein indicator*. Dissolve 0.5 g of phenolphthalein in 100 ml of 95 % (v/v) ethanol<sup>12)</sup> and make faintly pink by the addition of 0.1N sodium hydroxide solution.

#### E.2 Procedure

Using the same pipette, introduce 50.0 ml of the potassium hydroxide solution into each of two dry 250 ml conical flasks with ground-glass joints. Add 5 ml of water to each flask. Close the flasks with their glass stoppers. By means of a weighing pipette, transfer 2.4 g to 2.6 g of the sample, weighed to the nearest 0.001 g, into one of the flasks.

Attach the flasks to water-cooled reflux condensers with ground-glass joints and heat for one hour in a boiling water bath. Withdraw the flasks, still carrying their condensers, and immerse them in cold running water. When cool, wash down the inside of each condenser with two 20 ml portions of water. Disconnect the flasks and wash each joint with a further 20 ml of water. Add 0.5 ml of the phenolphthalein indicator and titrate the mixture immediately with the hydrochloric acid until the pink colour is just discharged.

#### E.3 Calculation

Ester content, calculated as *iso* propyl acetate,

$$\mathrm{CH_{3}COOCH(CH_{3})_{2},\,per\,cent\,by\,mass} = \frac{10.21\,(T_{3}\,-\,T_{2})}{M_{2}}$$

where  $T_3$  = volume, in millilitres, of N hydrochloric acid solution required by the blank,

 $T_2$  = volume, in millilitres, of N hydrochloric acid solution required by the test sample

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

 $<sup>^{11)}</sup>$  BS 3978, "Water for laboratory use".

<sup>12)</sup> Ethanol may be replaced by industrial methylated spirits, 95 % (v/v), complying with the requirements of BS 3591. It should be noted that the use of industrial 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.

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