BRITISH STANDARD

BS 4774:1972

Methods of test for

Chloroform for industrial use

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:

British Steel Industry
Chemical Industries Association*
Department of Health and Social Security
Department of Trade and Industry
Department of Trade and Industry — Laboratory of the Government Chemist*
Fertilizer Manufacturers' Association Ltd.
Institute of Vitreous Enamellers
Institution of Gas Engineers
Ministry of Agriculture, Fisheries and Food
National Sulphuric Acid Association
Royal Institute of Public Health and Hygiene
Soap and Detergent Industry Association

The Government department and industrial organization marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the formulation of the United Kingdom point of view in the international work leading to the publication of this British Standard:

British Plastics Federation
Dyers' and Cleaners' Research Organization
Fire Extinguishers Trades Association
Institute of Metal Finishing
Institute of Refrigeration
Man-made Fibre Producers' Committee
Ministry of Defence, Army Department
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Paintmakers' Association of Great Britain
Pharmaceutical Society of Great Britain
Research Association of British Paint, Colour and Varnish Manufacturers
Royal Institute of Chemistry
Society of Chemical Industry
Society of Motor Manufacturers and Traders Ltd.

This British Standard, having been approved by the Chemicals Industry Standards Committee, was published under the authority of the Executive Board on 26 May 1972

The following BSI references relate to the work on this standard:
Committee references CIC/4 and CIC/4/6
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Amendments issued since publication

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Contents

Foreword

This standard makes reference to the following British Standards:

BS 593, *Laboratory thermometers.*

BS 1792, *One-mark volumetric flasks.*

BS 2511, *Determination of water (Karl Fischer method).*

BS 3978, *Water for laboratory use.*

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

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

For some years the United Kingdom has participated in the work of preparing methods of test for halogenated hydrocarbons for industrial use, carried out within Working Group 6 of Technical Committee 47 — Chemistry, of the International Organization for Standardization (ISO). As international agreement is reached on the methods of test for each product, it is proposed to publish them as British Standards.

This British Standard is technically identical with ISO Recommendation R 1870, *"Chloroform for industrial use — Methods of test"*, and is published at the same time as three other British Standards similarly produced:

BS 4775, *"Methods of test for 1, 4-dichlorobenzene for industrial use".*

BS 4776, "*Methods of test for 4-chlorotoluene for industrial use*" and

BS 4777, *"Methods of test for 2-chlorotoluene for industrial use".*

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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.

1 Scope

This British Standard describes methods of test for chloroform for industrial use.

NOTE The titles of the British Standards referred to in this standard are listed on page ii.

2 Determination of distillation characteristics

Use the method described in BS 4591:1971, subject to the following additional details and modifications appropriate for chloroform.

2.1 Principle (see **1** of BS 4591:1971). This determination indicates the difference between the temperatures corresponding to the collection of two volumes of distillate, *A* and *B*. These two volumes will be indicated in the specifications for the product agreed between the interested parties.

2.2 Thermometer (see **3.2** of BS 4591:1971). Use a thermometer including the interval 59 °C to 71 °C (see BS 593).

2.3 Distillation rate (see **6.2** of BS 4591:1971). Carry out the distillation at a rate of 4 ml to 5 ml per minute.

2.4 Temperature correction. The temperature correction, which is necessary only when the scope of the determination is to ascertain the two temperatures corresponding to the volumes of distillate *A* and *B*, is equal to 0.040 (760 – *p*) °C where *p* is the barometric pressure in millimetres of mercury. This correction shall be added to the observed temperatures.

3 Determination of density at 20 °C

Use the method described in BS 4522.

4 Determination of residue on evaporation

4.1 Principle. The principle of this method is weighing of the residue obtained after evaporation of the test portion at its boiling point and drying at 110 °C to constant mass.

CAUTION. This test should be carried out in a fume cupboard with a good draught.

4.2 Apparatus. The following apparatus is used.

1) *Platinum dish* or, in the absence of this, a borosilicate glass dish, of approximately 140 ml capacity and 85 mm diameter.

2) *Apparatus* for heating the dish by transmission of heat obtained by boiling chloroform under reflux.

Figure 1 shows an example of such an arrangement.

3) *Oven*, controlled at 110 ± 2 °C.

4.3 Procedure. Weigh the dish to the nearest 0.2 mg, after heating for 30 min in the oven at 110 °C and cooling in a desiccator, for 30 min in the case of a platinum dish or for 45 min in the case of a borosilicate glass dish.

Introduce 100.0 ml of the test sample.

Place the dish on the heating apparatus, taking care to place an inverted funnel over it for protection against the deposition of dust.

Wait until all the liquid has evaporated, then place the dish, containing the residue, in the oven with the temperature previously set at 110 ± 2 °C.

Dry until the mass is constant to within ± 0.2 mg, leaving the dish and contents to cool in a desiccator before each weighing for the same time as when weighing out.

4.4 Expression of results. Residue on evaporation is expressed as a percentage (m/m) by the formula:

$$\frac{m \times 100}{\rho_{20} \times V} = \frac{m}{\rho_{20}}$$

where

m is the mass of the weighed residue (g);

ρ_{20} is the density of the test sample at 20 °C (g/ml);

V is the volume of the test portion, i.e. 100.0 ml.

5 Determination of water content

Use any of the methods described in BS 2511.

6 Determination of acidity

6.1 Principle. The principle of this method is titration of the acidity of the aqueous extract separated from a test portion with standard sodium hydroxide solution, using bromocresol green as indicator.

6.2 Apparatus. Ordinary laboratory apparatus is to be used.

6.3 Reagents. The following reagents are to be used.

1) *Sodium hydroxide*.
Approximately 0.01N solution, standardized against 0.01N hydrochloric acid solution under the same conditions as in the determination.

2) *Bromocresol green*. 1 g/l solution in 95 % (v/v) ethanol.

3) *Distilled water neutral to bromocresol green*. Add distilled water contained in a conical flask fitted with a ground glass stopper, 1 % (v/v) of the bromocresol green solution [**6.3** 2)] and neutralize with the standard sodium hydroxide solution [**6.3** 1)] until the colour becomes a clear blue.

NOTE Attention is drawn to BS 3978, *"Water for laboratory use"*.

6.4 Procedure

6.4.1 *Test portion*. Take 50.0 ml of the laboratory sample.

6.4.2 *Determination*. Place in a 250 ml separating funnel, 100.0 ml of neutral distilled water [**6.3** 3)] at approximately 20 °C.

Add the test portion (**6.4.1**) and agitate for exactly 3 min, measured with the aid of a chronometer. Allow to settle.

If the sample is acid, the aqueous phase will be yellow in colour. Carefully take 50.0 ml of this phase and transfer to a 250 ml conical flask.

Titrate with the sodium hydroxide solution [**6.3** 1)] until a clear blue colour is obtained.

6.5 Calculation. The acidity, expressed in milli-equivalents per litre, is given by the formula:

$$V \times 0.01 \times \frac{100}{50} \times \frac{1000}{50} = 0.4V$$

where V is the volume, in millilitres, of the standard sodium hydroxide solution used for the titration.

7 Measurement of colour

7.1 Principle. The principle of this method is comparison of the colour of the sample with coloured standards, expressing the result in Hazen (platinum-cobalt) units.

NOTE The Hazen unit is by definition, the colour of a solution containing 1 part per million of platinum in the form of chloroplatinic acid, in the presence of 2 parts per million of cobaltous chloride hexahydrate ($CoCl_2.6H_2O$).

7.2 Apparatus. Use the following apparatus.

1) *Ordinary laboratory apparatus*, and

2) *Matched flat bottom colorimetric tubes*, (two) with a graduation mark at least 100 mm from the base.

7.3 Reagents. Use only distilled water or water of equivalent purity throughout the analysis.

The following reagents are to be used.

1) *Cobaltous chloride*, hexahydrate.

2) *Hydrochloric acid*, concentrated 36 % (m/m) (11N).

3) Either

a) *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 [**7.3** 2)] and again evaporate to dryness. Repeat this operation twice more.

or

b) *Potassium chloroplatinate*.

7.4 Preparation of colour standard. Dissolve 0.50 g of the cobaltous chloride hexahydrate [**7.3** 1)] and either the whole of the chloroplatinic acid (prepared as described in **7.3** 3) a) or 0.623 g of the potassium chloroplatinate [**7.3** 3) b), in 50 ml of the hydrochloric acid [**7.3** 2)]. Warm if necessary to obtain a clear solution and after cooling, pour into a 500 ml one-mark volumetric flask. Dilute with water to the mark.

NOTE Attention is drawn to BS 3978, *"Water for laboratory use"*, and to BS 1792, *"One-mark volumetric flasks"*.

From this solution prepare a series of colour standards ranging from 0 Hazen colour units upwards at intervals of 10 units. For each 10 units pipette 5.0 ml of the solution into a 250 ml one-mark volumetric flask and dilute with water to the mark. These diluted solutions shall always be freshly prepared.

7.5 Procedure. Fill one of the colorimetric tubes [**7.2** 2)] to the mark with the sample, and the other with the selected colour standard. Using a white background compare the colours, viewing vertically. Repeat with other colour standards, if necessary, until the closest match is obtained.

If the colour of the sample does not match that of any of the colour standards, report if possible an approximate value or give a description of the colour with an appropriate note.

8 Test report

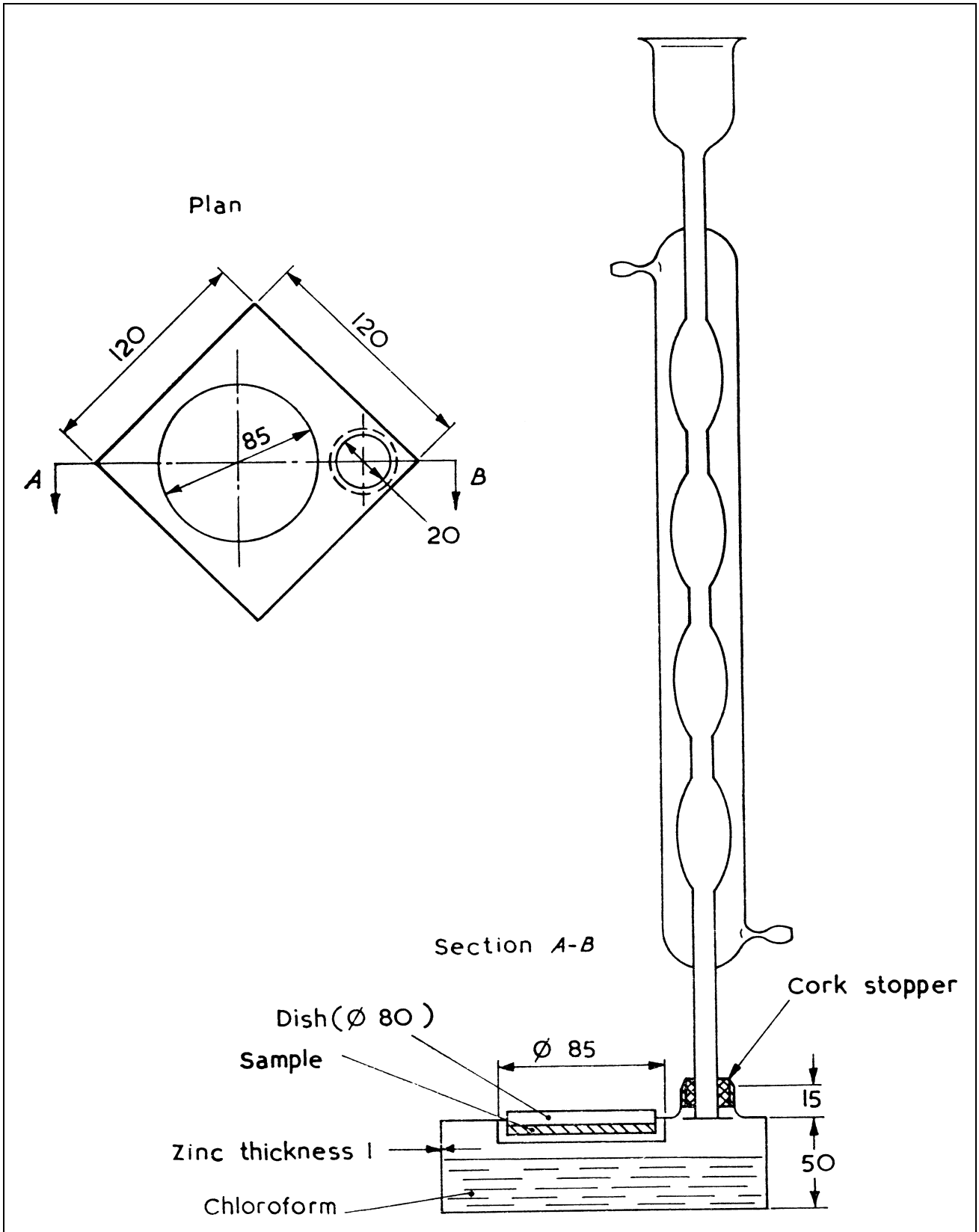
For each test state the following in the report:

1) the reference of the method used, i.e. "in accordance with BS 4774";

2) the results and the method of expression used;

3) any unusual features noted during the determination;

4) any operation not included in this standard or those to which reference is made or regarded as optional.



Approximate dimensions in millimetres

Figure 1 — Apparatus for determination of residue on evaporation

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