BRITISH STANDARD

BS 894:1956

*Incorporating Amendment No. 1*

## Specification for

# Ubbelohde apparatus for flow and drop points

Confirmed November 2011

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# Co-operating organizations

The Scientific Glassware and Related Laboratory Apparatus Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

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This British Standard, having been approved by the Scientific Glassware and Related Laboratory Apparatus Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on
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# Contents

# Foreword

This standard makes reference to the following British Standard:

BS 593, *Laboratory thermometers*.

This British Standard was first published in 1940 under the title "*The determination of the flow and drop points of fats and allied substances*," and at that time only a glass cup was specified. In 1947 an amendment was issued specifying a metal cup as an alternative, but it was pointed out that "it must not be assumed that the glass cups and metal cups may in all circumstances be used indiscriminately", and experience has shown that the results obtained for a given material may differ significantly according to which type of cup is used.

Similar apparatus and methods of use have been specified both by the Institute of Petroleum and by the British Pharmacopœia Commission. The Institute of Petroleum, however, have decided only to specify the metal cup, and in the 1953 edition of the "British Pharmacopœia" this has been introduced as an alternative to the glass cup. The chief objection to the latter is the difficulty of complying in manufacture with the precise tolerances on dimensions which are essential to secure interchangeability of the parts and reproducible results.

Glass cups strictly complying with the British Standard have, in fact, been very difficult to obtain, and this consideration outweighs the advantage of the glass cup that air bubbles included during filling can be more easily detected. It has therefore been decided to specify only the metal cup in this revision.

In order that the cup should be held firmly in the case and to secure interchangeability, both parts had previously to be made to very close tolerances, with consequent increase in cost. An important change in this edition is the provision on the case of spring clips which grip the cup. The tongues previously specified are not split, but are retained as guides for inserting the cup.

Sleeves complying with the requirements of the 1940 edition of this standard may still be used provided they are fitted with spring clips as specified.

Other changes made in the apparatus specification are of minor importance. The previous edition contained a short section describing the method of use, but enquiries have shown that the precise method of filling and using the cup needs to be specified in detail, and varies according to the type of material which is being tested. It is not possible in this British Standard to specify suitable methods for all materials, but for information and guidance the methods in use for a number of important materials are given in Appendix B.

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.

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**Summary of pages**

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8 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 specifies a metal cup and ancillary apparatus of the Ubbelohde type for the determination of the flow and drop points of fats and allied substances.

## 2 Assembly of apparatus

The apparatus shall consist of the following components:

A. *Metal cup.*
B. *Thermometer for cup.*
C. *Sleeve and case.*
D. *Boiling tube.*
E. *Cork.*
F. *Beaker.*
G. *Stirrer.*
H. *Heating liquid.*
I. *Auxiliary thermometer.*
J. *Stand with clamps.*
K. *Source of heat.*

The general assembly is shown in Figure 1.

## 3 Metal cup

The cup shall be made from chromium plated brass, or from other suitable metal which is not affected by the material being tested. The top of the cup and the bottom of the tube forming the orifice shall be smooth, parallel to each other and at right angles to the axis of the cup. The cup shall conform to the dimensions given in Table 1 and Figure 2. The wide part of the cup shall have an approximately hemispherical lower portion, and an internal depth such that when a steel ball 7.00 mm in diameter is placed in the cup, the top of the ball shall be $12.20 \pm 0.15$ mm above the bottom of the tube forming the orifice. The bottom edge of the orifice shall not be chamfered or radiused.

**Table 1 — Dimensions of metal cup**

| | mm |
|---|---|
| Internal diameter of wide part | $7.50 \pm 0.15$ |
| External diameter of wide part | $9.99 \begin{smallmatrix} +0.00 \\ -0.04 \end{smallmatrix}$ |
| Internal diameter of orifice | $3.15 \pm 0.05$ |
| External diameter of orifice | $5.55 \pm 0.05$ |
| Overall length | $15.2 \pm 0.2$ |

## 4 Thermometer for cup

The thermometer used shall be a 100 mm immersion type selected as required from the ranges specified in Series C of BS 593,[1] and shall comply with the relevant provisions in Table 2 or Table 4 of that British Standard, except in so far as they conflict with the following special requirements:

a) The diameter of the bulb shall be $3.5 \pm 0.15$ mm.

b) The length of the bulb shall be not more than 6 mm.

c) The stem immediately above the bulb shall have a diameter approximately equal to, but not less than that of the bulb, for a length of at least 26 mm, measured from the bottom of the bulb.

d) The inscriptions on the thermometer shall be as given in Clause **14**.

e) The thermometer shall be finished with a plain top.

Alternatively, where a particular thermometer is included in the specification to which the product is being tested, then this thermometer shall be used.

## 5 Sleeve and case

To the lower end of the thermometer the cylindrical metal sleeve [see Figure 3(a)] shall be securely and coaxially cemented.[2] To this shall be screwed the metal case (see Figure 4), which shall have two small apertures, one in front and one behind, to act as air vents. The case shall have a rim stop, guide lugs to aid the coaxial insertion of the cup into the case, and spring clips which enable the cup to be retained firmly with its top edge against the stop. The design, dimensions, and method of fixing of the spring clips shown in Figure 4 are not mandatory but are recommended as suitable.

The sleeve shall be fixed to the thermometer in such a position that, when the case is screwed on to the sleeve, the bottom of the thermometer bulb shall be $8.0 \pm 0.1$ mm below the rim stop, and the thermometer stem shall be coaxial with the sleeve and case.

## 6 Boiling tube

The boiling tube shall be of glass. Suitable dimensions for it are:

Overall length 110 mm
Internal diameter 25 mm

[1] BS 593, "*Laboratory thermometers*".
[2] While it does not form a mandatory part of this standard, it is suggested that the accurate location of the thermometer in the sleeve may be assisted by the use of a metal jig such as is shown in Figure 3(b). The inside of the sleeve may with advantage be roughened to provide a key for the cement. The cement used shall be suitable for the temperatures covered by the thermometer.

## 7 Cork

The cork shall be approximately 25 mm long, shall be bored with a central hole to fit the thermometer specified in Clause **4**, and shall have a notch cut along the side.

## 8 Beaker

The beaker shall be of glass, and of such a size that when the apparatus is assembled the boiling tube can be immersed vertically to two-thirds of its length in the liquid heating medium with its lower end about 25 mm above the bottom of the beaker. (A 600 ml squat form beaker is suitable.)

## 9 Stirrer

The stirrer shall consist of any convenient device which will ensure uniformity of temperature throughout the liquid in the beaker.

## 10 Heating liquid

Any suitable liquid may be used as the heating medium in the beaker. Water is recommended for flow and drop points below 80 °C and glycerine or liquid paraffin for flow and drop points above 80 °C.

## 11 Auxiliary thermometer

Any suitable thermometer may be used for measuring the temperature of the heating liquid. A 50 mm immersion thermometer of suitable range, constructed in accordance with the provisions for Series C of BS 593,[3)] is convenient for this purpose.

## 12 Stand with clamps

Any suitable stand may be used to hold the boiling tube and auxiliary thermometer in position in the beaker, and to hold the beaker above a source of heat.

## 13 Source of heat

Any suitable means may be used to heat the liquid in the beaker.

## 14 Inscriptions

The metal cup specified in Clause **3** and the thermometer specified in Clause **4** shall each have permanently and legibly marked on it:

a) An identification number.

b) The maker's or vendor's name or mark.

c) The number of this British Standard, i.e. "BS 894".[4)]

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[3)] BS 593, "*Laboratory thermometers*".

[4)] The mark "BS 894" on the product is an indication by the manufacturer that it purports to comply with the requirements of this British Standard.

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Further particulars of the terms of licence may be obtained from the Director, British Standards Institution, 2 Park Street, London, W.1.

## Appendix A Testing of British Standard Ubbelohde apparatus

The National Physical Laboratory is prepared to accept metal cups and the thermometers for them for examination for compliance with the requirements of this British Standard. Particulars of the fees charged can be obtained on application to the Director, National Physical Laboratory, Teddington, Middlesex.

## Appendix B Examples of methods adopted for using the Ubbelohde apparatus

### 1. British Pharmacopœia 1953.

### Melting point of hydnocarpus oil, soft paraffin, and wool fat.

*Method.* The glass cup is filled with the substance and smoothed so that no air bubbles are included. The cup is then pushed, without lateral movement, into the metal case as far as the stop and the excess fat squeezed out of the bottom of the tube is wiped away. Care is taken that the pressure equalizing aperture is not stopped. The thermometer, with the cup attached, is next fitted through the bored cork to the boiling tube, and the immersion line on the thermometer is adjusted to the top of the cork. The boiling tube is fixed vertically within the beaker so that at least two-thirds of its length is immersed in water contained in the beaker and kept stirred. The temperature of the outer bath is so adjusted that the temperature of the substance rises at the rate of 1 °C per minute. The temperature at which the first drop of melted liquid falls from the glass cup is regarded as the melting point of the substance.

NOTE The glass cup is that specified in earlier editions of BS 894. The British Pharmacopœia 1953 also states:

"Alternatively, a metal cup of suitable dimensions may be used, provided that the result obtained is the same as that given when a glass cup complying with the above specification is used."

### 2. Institute of Petroleum Standard Methods for Testing of Petroleum and its Products.

### Method I.P. 31/55 Drop Point of Greases.

*Scope.* In general, the drop point is an indication of the temperature of transition from the solid or semi-solid to the liquid state, and does not necessarily have any bearing upon service performance.

*Definition.* The drop point is the temperature at which a drop of the sample first detaches itself from the main bulk, when it is steadily heated under closely specified conditions. In some cases a drop does not become detached and the sample flows in a continuous stream. The drop point is then the temperature recorded when the stream has travelled a specified distance.

*Procedure.*

a) By means of a spatula, fill the cup with the sample and cut away any excess material. Take precautions to exclude air bubbles, but do not melt the grease.

b) Push the cup, without lateral movement, into the metal case as far as the stop and cut away any excess material squeezed out of the bottom. Take care that the aperture in the side of the metal case is not blocked. Fit the thermometer, with the cup attached, centrally in the boiling tube, through a bored cork having a side notch, so that the bottom of the cup is 25 mm ± 1.0 mm above the bottom of the boiling tube. Then fix the boiling tube vertically in the beaker containing the liquid heating medium, so that two-thirds of its length is immersed and the bottom is about 25 mm above the bottom of the beaker.

c) Heat the liquid bath, keeping it stirred, at such a rate that the drop point thermometer shows a rise in temperature of 1 °C per minute for 20 °C below the drop point of the sample. Record the temperature at which the first drop, whatever its composition, falls from the cup or at which the continuous stream of material, if formed, reaches the bottom of the boiling tube.

*Reporting.* Report the temperature recorded to the nearest 1.0 °C.

*Precision.* Results of duplicate tests should not differ by more than the following amounts:

| Grease type | Repeatability (One operator and apparatus) | Reproducibility (Different operators and apparatus) |
|---|---|---|
| Lime-base grease | 2 °C | 4 °C |
| Soda-base grease | 5 °C | 10 °C |
| Other greases | Not established | Not established |

**3. Methods adopted for inclusion in revision (in course of preparation) of BS 684, "*Methods of analysis of oils and fats*".**

*Definitions:*

*Flow point.* The flow point of a fat is the temperature at which it forms an approximately hemispherical protuberance at the orifice of the cup under the specified conditions of test.

*Drop point.* The drop point of a fat is the temperature at which the first drop falls from the cup under the specified conditions of test.

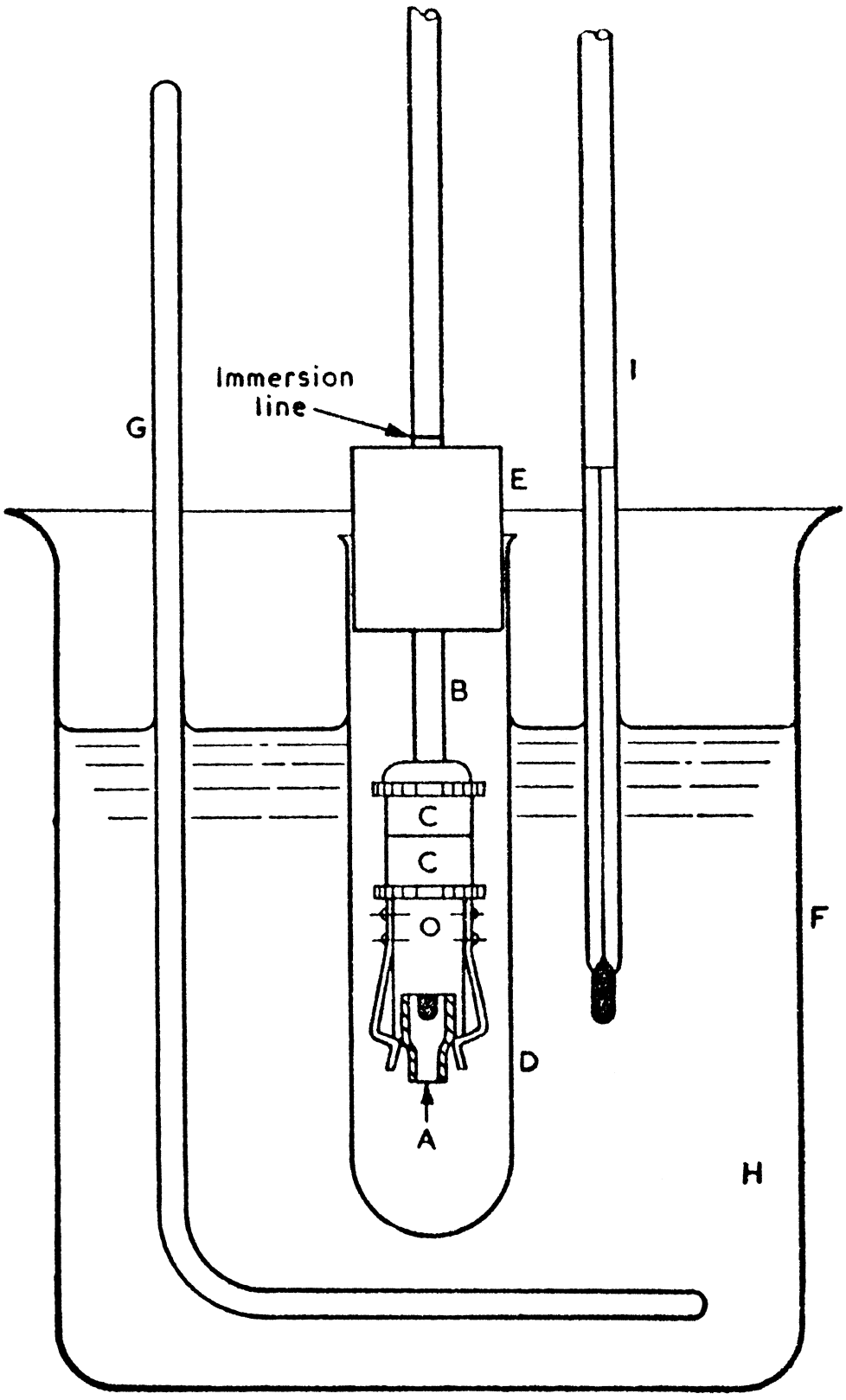
*Method for use with soft fats.* Fill[5)] the cup by means of a spatula with the fat to be tested and smooth it so that no air bubbles are included. Push the cup, without lateral movement, into the metal case as far as the stop and wipe away the excess fat squeezed out of the bottom of the tube. Care should be taken not to seal the pressure equalizing aperture in the side of the metal case. Fit the thermometer, with the cup attached, through the bored cork to the boiling tube and adjust the immersion line on the thermometer to the top of the cork. Fix the boiling tube vertically within the beaker so that at least two-thirds of its length is immersed in a suitable liquid heating medium contained in the beaker and kept stirred.

Adjust the temperature of the outer bath so that the temperature of the fat rises at the rate of 1 °C per minute over a range of about 10 °C immediately below the flow point.

*Method for use with hard fats.* Warm the fat with gentle stirring to about 10 °C above its melting point, and introduce sufficient into a small beaker (e.g. 100-ml squat type) to give approximately ¾ in. to 1 in. depth. Drop the cup into the molten fat and allow it to reach the same temperature, then remove it by means of tweezers and place it on a watch glass standing on crushed ice. Fill the cup immediately with the molten fat and fit the thermometer whilst the fat is still fluid. After standing the apparatus for 5 minutes at 0 °C (preferably in a refrigerator), remove the surplus fat and allow the apparatus to stand at room temperature (17 °C) overnight (16 hours). Fix the boiling tube vertically within the beaker so that at least two-thirds of its length is immersed in a suitable liquid heating medium contained in the beaker and kept stirred.

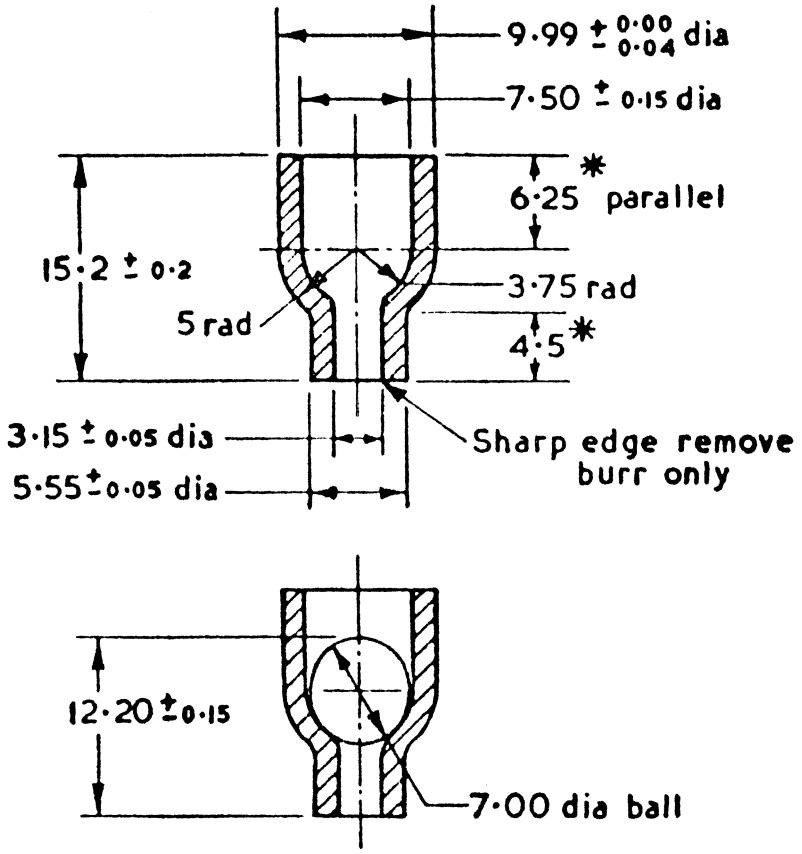
Adjust the temperature of the outer bath so that the temperature of the fat rises at a rate of 1 °C per minute over a range of about 10 °C immediately below the flow point.

[5)] The precise method for filling the cup varies with different substances. When the use of this test is prescribed for any substance, the method of filling with that substance must be defined.



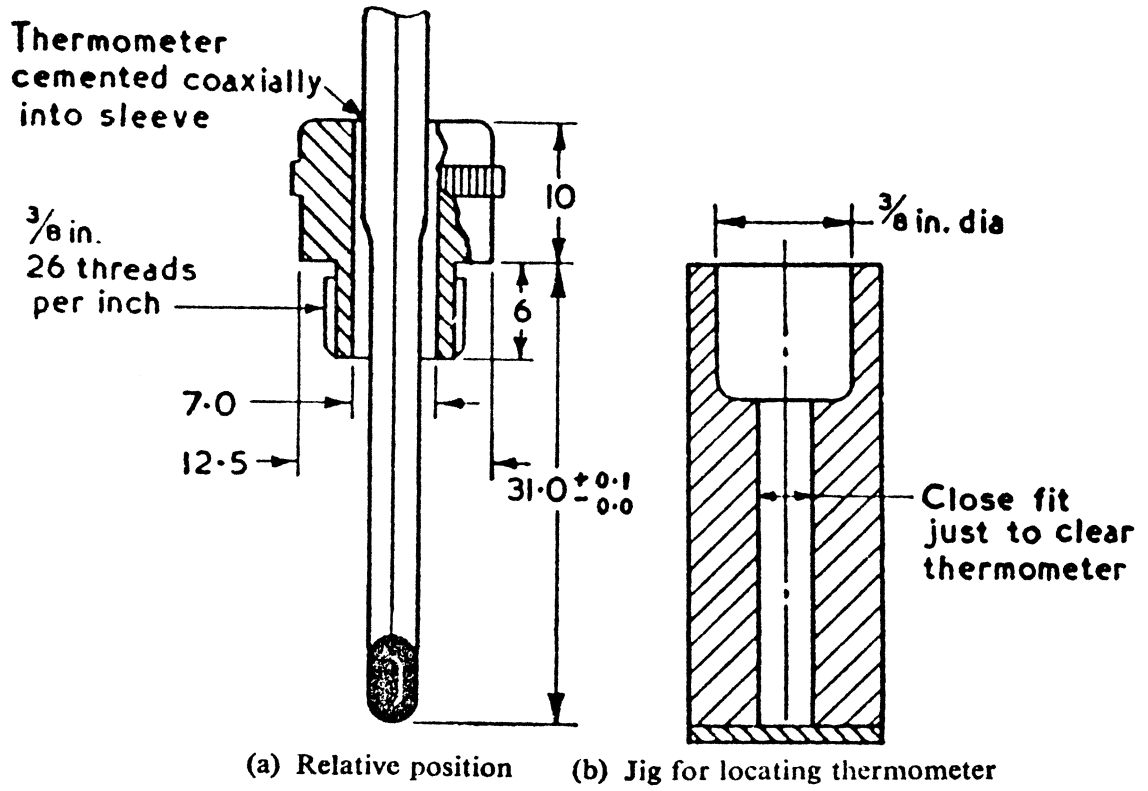

(For key to components see Clause **2**)

**Figure 1 — Ubbelohde apparatus — assembly**



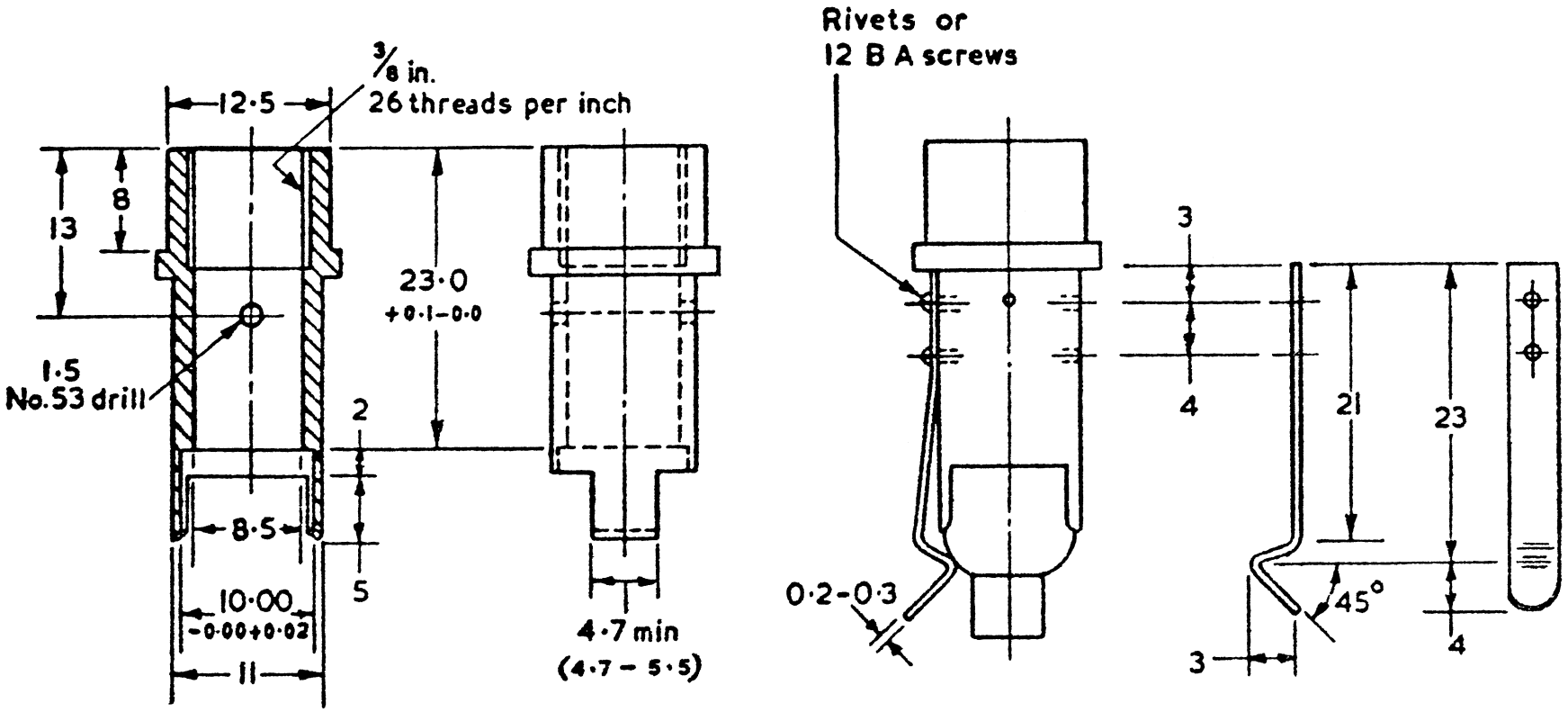

All dimensions are in millimetres
Dimensions marked with an asterisk are for guidance only
Where no tolerances are shown, normal working tolerances are allowed

**Figure 2 — Ubbelohde cup**




All dimensions are in millimetres except where otherwise shown
Where no tolerances are shown, normal working tolerances are allowed

**Figure 3 — Ubbelohde apparatus — thermometer and sleeve**




All dimensions are in millimetres except where otherwise shown. Dimensions shown without tolerances are included for guidance

**Figure 4 — Ubbelohde apparatus — case**

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