

CONFIRMED
DECEMBER 2007

Dean and stark apparatus

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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 Metropolitan Water Board
 Office of the High Commissioner for India
 Oil and Colour Chemists Association
 Research Association of British Paint, Colour and Varnish Manufacturers
 University of London (Faculty of Science)
 Individual manufacturers

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Foreword

This standard makes reference to the following British Standards:—

BS 572, *Interchangeable conical ground glass joints*.

BS 1751, *General purpose glass stopcocks*.

Apparatus of the Dean and Stark type was first specified in 1936, when BS 614 “*Graduated receivers for Dean and Stark apparatus*” was published. At that time ground glass joints were not specified, but in 1937 was added BS 756, “*Apparatus for the determination of small quantities of water by distillation with an immiscible liquid*”, which included requirements for condenser, receiver and flask with ground joint connections.

In 1939, when the two specifications were combined as a revision of BS 756, standard ground joints were becoming popular, but were still considered somewhat of a luxury in many laboratories; although it was recognized that their use afforded greater accuracy in the apparatus in question, it was still considered advisable on the score of cheapness to provide for the use of corks, despite their shortcomings. In the present revision, however, standard joints only are specified for the connection to the condenser, although there is, for most sizes, a choice of standard joint or cork for the connection to the distillation vessel.

A further change is the introduction of the B19 joint for the connection between condenser and receiver. Whereas the joints previously specified (A16 for the condenser and B16 for the receiver) were practically confined to this apparatus, the B19 joint is very widely used; a condenser from the laboratory stock with a B19 joint can therefore be employed in an emergency if the Dean and Stark condenser is not available. This change will not, however, render obsolete any older apparatus that may still be in use.

Investigations have shown that two sizes of condenser are not normally required for the apparatus, and a jacket length of 25 cm. has therefore been standardized in place of the 20 cm. and 30 cm. sizes previously in use. A single size of spray tube is likewise specified, and it is emphasized that this is an optional part of the apparatus.

Additional receivers have been included as developed by the Road Research Laboratory for tests on soils. These are the 25 and 100 ml. receivers with stopcock (each with a plain vapour tube for cork connection).

In the 1939 edition the distance which separated the vapour tube from the body of the receiver was fixed at 70 mm. but experience has shown that this may result in the base of the receiver being unduly close to the source of heat under the distillation vessel. It has therefore been decided, for all Type 1 receivers, except the 2 ml. size, to revert to the 150 mm. separation originally specified in 1936 in BS 614.

The Type 2 apparatus of the 1939 edition has been replaced by the modified Dean and Stark type specified by the Institute of Petroleum for the determination of motor fuel diluent in crankcase oil, which may also be used for the determination of water, using a heavier liquid. As such liquids, e.g. trichlorethylene or perchlorethylene, are generally non-inflammable this apparatus may commend itself in many laboratories owing to the absence of the fire hazard. The 12.5 ml. size is that specified by the Institute of Petroleum and a 3 ml. size has been added for the convenience of other users.

Little information on assembly of apparatus or on methods of use has been included in this standard as these points are generally dealt with in the test methods which require the use of the apparatus.

The following British Standards make reference to an apparatus of the Dean and Stark type. Consultations have taken place with committees of the Institution which are responsible for the production of these standards, and it is understood that the appropriate size and type of apparatus herein described can be satisfactorily employed in each case, and will be proposed for inclusion in future revisions. Agreement has also been reached with the Institute of Petroleum and the Standardization of Tar Products Tests Committee to adopt British Standard apparatus for inclusion in future editions of their handbooks on methods of test.

BS 76:1943, *Tars for road purposes.*

BS 209:1947, *Fuels for oil engines.*

BS 241:1935, *White oil pastes for paints.*

BS 261:1936, *Ready mixed paints (oil gloss).*

BS 331:1938, *Driers for paints.*

BS 390:1938, *Oil pastes (excluding white lead, zinc oxide and lithopone) for paints.*

BS 517:1948, *Cresylic acid of specified orthocresol content.*

BS 521:1948, *Cresylic acid of specified metacresol content.*

BS 523:1948, *Phenol.*

BS 524:1948, *Refined cresylic acid.*

BS 544:1934, *Linseed oil putty.*

BS 598:1950, *Sampling and examination of bituminous road mixtures.*

BS 618:1935, *Emulsions of road tar and of road tar-asphaltic bitumen mixtures for penetration (grouting and semi-grouting) and surface dressing.*

BS 628-32 and BS 650-56:1950, *Vegetable oils.*

BS 684:1950, *Methods of analysis of oils and fats.*

BS 735:1944, *Sampling and analysis of coal and coke for performance and efficiency tests on industrial plant.*

BS 742:1947, *Fuel oils for burners.*

BS 929:1947, *Ready mixed oil paints.*

BS 1011:1942, *Red lead ready mixed paints.*

BS 1016:1942, *Methods for the analysis and testing of coal and coke.*

BS 1017:1942, *Methods for the sampling of coal and coke.*

BS 1033:1942, *Priming paint (lead base) for the protection of steel sheet.*

BS 1067:1942, *Coal tar pitch felt damp-proof courses for temporary war-time building.*

BS 1070:1942, *Black paint (tar base) for use on iron and steel.*

BS 1177:1944, *Pitch mastic flooring incorporating lake asphalt.*

BS 1375:1947, *Coloured pitch mastic flooring.*

BS 1450:1948, *Black pitch mastic flooring.*

BS 1469:1948, *Coal tar liquid fuels.*

BS 1673-2, *Methods of testing raw rubber and unvulcanized compounded rubber: Methods of chemical analysis.*

BS 1715:1951, *Methods for the analysis of soaps.*

BS 1737:1951, *Jointing materials and compounds for water, town gas and low-pressure steam installations.*

BS 434:1952, "*Bitumen road emulsion for penetration (grouting and semi-grouting) and surface dressing*", also specifies an apparatus of the Dean and Stark type, but this has a 25 ml. receiver of special design with a longer scale graduated in 0.1 ml. and has therefore been omitted from the present standard.

SUBSIDIARY STANDARD TEMPERATURE OF 27 °C.

At the Second Meeting, in 1951, of Technical Committee ISO/TC 48 — Laboratory Glassware and Related Apparatus, of the International Organization for Standardization, it was agreed to qualify as follows the acceptance of 20 °C. as the standard temperature for volumetric glassware :—

"When it is necessary in tropical countries to work at an ambient temperature considerably above 20 °C., and these countries do not wish to use exclusively the standard temperature of 20 °C., it recommended that they should adopt a temperature of 27 °C."

In order to meet the requirements of such tropical countries it has been decided to amend the British Standards for volumetric glassware to permit 27 °C., as an alternative to 20 °C.

In the present British Standard this amendment applies to Clause 7 and to the Figure 5 to Figure 12.

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 to iv, pages 1 to 18, 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 specifies suitable apparatus for the determination of water by the method of distillation with an immiscible liquid. The method is applicable to a wide range of materials in industrial processes and laboratory practice.

2 Assembly of apparatus

The apparatus shall consist of the following components, assembled as shown in Figure 1 :—

- a) Distillation vessel.
- b) Condenser, including spray tube if required.
- c) Receiver.

3 Material

The receiver and condenser shall be made of borosilicate glass as free as possible from visible defects, and shall be well annealed.

4 Design and dimensions

The general design and dimensions of the apparatus shall be as shown in Figure 1 to Figure 12. The only mandatory dimensions are those given in Clause 6 and Table 2. The remaining dimensions are given for the guidance of manufacturers and are not a mandatory part of the specification, but an apparatus which, on visual inspection, shows any gross departure from them shall be deemed not to comply with the specification.

5 Distillation vessel

For different purposes a flask made of borosilicate glass or a metal flask or pot is suitable. If a glass flask is used, the connection to the receiver shall be made by means of a cork or by a ground glass joint complying with BS 572¹⁾, as specified in Table 1. If a metal vessel is used, the connection shall be made by a cork or by other suitable means such as that shown in Figure 2.

6 Condenser

- a) *Design and dimensions.* The condenser shall be a glass water-cooled reflux type, of the design and dimensions shown in Figure 3. The only mandatory dimensions (see Clause 4) for the condenser are the external diameters of the inner tube and of the jacket, which shall be 16–17 mm. and 23–25 mm. respectively.

b) *Construction.* In the construction of the condenser, the following points should be noted:—

- i) The joints A and B should be neatly finished as shown in Figure 3; in particular, the bore at B should have the minimum disturbance.
 - ii) The shoulder above the cone of the B19 joint at C should be elongated as shown in Figure 3, thus avoiding a sharp re-entrant shape which may restrict the free flow of liquid down the inner wall.
 - iii) The cone should be extended beyond the length appropriate to the B19 joint, and the lower end ground at an angle of approximately 60° to the axis. The drainage tip should be at the front of the condenser when the lower water-connection is to the left, and the finish should be either smooth or fire-polished.
- c) *Spray tube.* The spray tube, if provided, shall be sealed at one end and shall have four small holes spaced regularly round its circumference near the closed end; it shall be of the design illustrated in Figure 4.

7 Receiver

a) *Type.* Two types of receiver are included, as follows:—

Type 1 is based on the Dean and Stark apparatus proper, and is used with light liquids, such as petroleum spirit or toluole.

Type 2 is based on the apparatus specified by the Institute of Petroleum for determining the diluent content of crankcase oil, and is used with heavy liquids such as trichlorethylene or perchlorethylene.

b) *Sizes.* Five sizes of Type 1 receiver and two sizes of Type 2 receiver are specified, and are illustrated in, Figure 5 to Figure 12. The connection to the condenser is provided by a ground glass joint of size B19 complying with BS 572¹⁾. Some sizes are provided with stopcocks and with alternative connections to the distillation vessel, as shown in Table 1.

¹⁾ BS 572, "Interchangeable conical ground glass joints".

Table 1 — Sizes and connections of receivers for dean and stark apparatus

Type	Size	Illustrated in	Stopcock	Connection to distillation vessel	Separation of graduated tube from vapour tube
1	2 ml.	Figure 5	Without	Straight tube	70
				B24 joint	70
				B34 joint	70
1	7.5	Figure 6	Without	Straight tube	150
				B24 joint	150
1	10	Figure 7	With	Straight tube	150
				B24 joint	150
1	25	Figure 8	Without	Straight tube	150
				B24 joint	150
		Figure 9	With	Straight tube	150
1	100	Figure 10	With	Straight tube	150
2	3	Figure 11	Without	B24 joint	70
2	12.5	Figure 12	Without	Straight tube	70
				B24 joint	70

Table 2 — Mandatory dimensions and tolerances for dean and stark receivers

Dimension, etc.	Type 1					Type 2	
	2 ml.	7.5 ml.	10 ml.	25 ml.	100 ml.	3 ml.	12.5 ml.
Volume equivalent to smallest sub-division ml.	0.05	0.1	0.1	0.2	1	0.05	0.1
Scale length mm.	95 ± 10	110 ± 10	110 ± 10	110 ± 10	130 ± 15	85 – 100	125 – 140
Length of cylindrical tube above upper graduation mark mm.	10 – 15	10 – 25	10 – 30	10 – 30	min.20	min.10	10 – 20
Tolerance on capacity ± ml.	0.02	0.04	0.06	0.1	1.0	0.03	0.06
Maximum permissible leakage rate of stopcock ml/min.	—	—	0.004	0.006	0.01	—	—

c) *Dimensions.* The only mandatory dimensions (see Clause 4) for the receivers are the scale length and the length of cylindrical tube above the upper graduation mark, which shall be as given in Table 2.

Table 3A and Table 3b provide for the guidance of manufacturers additional recommended dimensions for the receivers. These dimensions are not a mandatory part of the specification, but a receiver which, on visual inspection, shows any gross departure from them shall be deemed not to comply with the specification.

d) *Construction.* The shoulder of the upper chamber of the receiver immediately below the conical joint shall be finished square, as shown in Figure 5 to Figure 12.

The graduated portion of the receiver shall be cylindrical throughout its length. The bottom of the graduated tube of the Type 1 receiver, in the cases where a stopcock is not provided, shall be sealed, the end of the tube being approximately hemispherical in shape.

e) *Graduation.* The graduation lines shall be clear, permanent, uniform lines of thickness not greater than 0.3 mm., numbered and subdivided as shown in Figure 5 to Figure 12²⁾. All lines shall be in a plane at right angles to the longitudinal axis of the tube with no evident irregularity in the spacing.

In all sizes of receiver except the 100 ml. size the numbered graduation marks (and, in the 25 ml. receiver, also those mid-way between them) shall be carried completely round the tube, except that a gap not exceeding 10 % of the circumference is permitted, the shortest graduation marks shall be carried halfway round the tube, and the graduation marks of intermediate length shall be carried approximately two-thirds of the way round the tube and shall project equally at each end beyond the shortest graduation marks.

Table 3A — Recommended dimensions for dean and stark receivers (Types 1 and 2)

Dimension	Type 1					Type 2	
	2 ml.	7.5 ml.	10 ml.	25 ml.	100 ml.	3 ml.	12.5 ml.
External diameter of body mm.	28	28	28	28	28	28	28
Length of body mm.	50	50	50	50	50	50	50
External diameter of side tube mm.	14	14	14	14	14	14	14
Angle of side tube	60°	60°	60°	60°	60°	70°	70°
Length of jet mm.	—	—	30	30	30	—	—
Internal diameter of jet mm.	—	—	1.5–2	1.5–2	1.5–2	—	—
Separation of graduated tube from vapour tube mm.	70	150	150	150	150	70	70
Vertical separation between bottom of straight side tube and:							
<i>a.</i> lowest point of receiver without stopcock mm.	90	90	—	90	—	—	90
<i>b.</i> underside of stopcock mm.	—	—	120	120	140	—	—
Vertical separation between bottom of joint or flanged end (see Figure 2) on side tube and:							
<i>a.</i> lowest point of receiver without stopcock mm.	40	40	—	40	—	40	40
<i>b.</i> underside of stopcock mm.	—	—	70	—	—	—	—
Distance from top of receiver to stopcock mm.	—	—	—	—	260	—	—

²⁾ For convenience, in these drawings the middle portions of the graduated scales have been left blank.

Table 3b — Recommended dimensions for dean and stark receivers (Type 2 only)

Dimension	3 ml.	12.5 ml.
Length of cylindrical portion of receiver (including bulb on 3 ml. size) mm.	180	180
Vertical separation between top graduation mark and junction of return tube and side tube mm.	25–30	25–30
Angle of upper portion of return tube	7°	7°
External diameter of return tube mm.	6	6

In the 100 ml. receivers the graduation marks shall be confined to the front of the tube and shall be varied in length as shown in Figure 10.

f) *Definition of capacity.* The capacity corresponding to any graduation mark is defined as the volume of water at 20 °C., expressed in millilitres, required to fill the graduated portion to that mark at 20 °C., the axis of the graduated portion being vertical and the lowest point of the water meniscus being set on the graduation mark. Where a stopcock is provided, the volume of the bore of the stopcock key and the volume of the jet below the stopcock shall not be included as part of the measured volume.

g) *Tolerance on capacity.* The error at any point on the receiver scale, and also the difference between the errors at any two points on the scale, shall not exceed the figures given for the receiver concerned in Table 2.

h) *Stopcock.* If provided on the receiver, the stopcock shall be either of the 2 mm oblique-bore type stopcock having the general design shown in Figure 7, Figure 9 and Figure 10 or a glass stopcock of unconventional design. In both cases the stopcock key may be made from a suitably inert material other than glass. In all cases, stopcocks shall permit smooth and precise control of outflow. The rate of leakage tested with the stopcock free from grease, the barrel and key wetted with water (where applicable), the receiver filled initially with water to the top of the scale, and the key in any fully shut-off position shall not exceed the figures given in Table 2.

i) *Inscriptions.* Each receiver shall have permanently and legibly marked on it:

- The abbreviation “ml.”
- The inscription “C.20 °C.” to indicate that the receiver is graduated for content at 20 °C.
- An identification number. Where a stopcock is provided, this number shall also appear on the key.
- The maker’s or vendor’s name or mark.
- The appropriate joint size numbers in accordance with BS 572³⁾.
- The number of this British Standard, i.e. “BS 756”.

³⁾ BS 572, “Interchangeable conical ground glass joints”.

Appendix Testing of British Standard dean and stark receivers

The National Physical Laboratory is prepared to accept receivers for test for compliance with this British Standard. If required, certificates of values can be supplied for all satisfactory receivers, except the 100 ml. size. Particulars of the fees charged can be obtained on application to the Director, the National Physical Laboratory, Teddington, Middlesex.

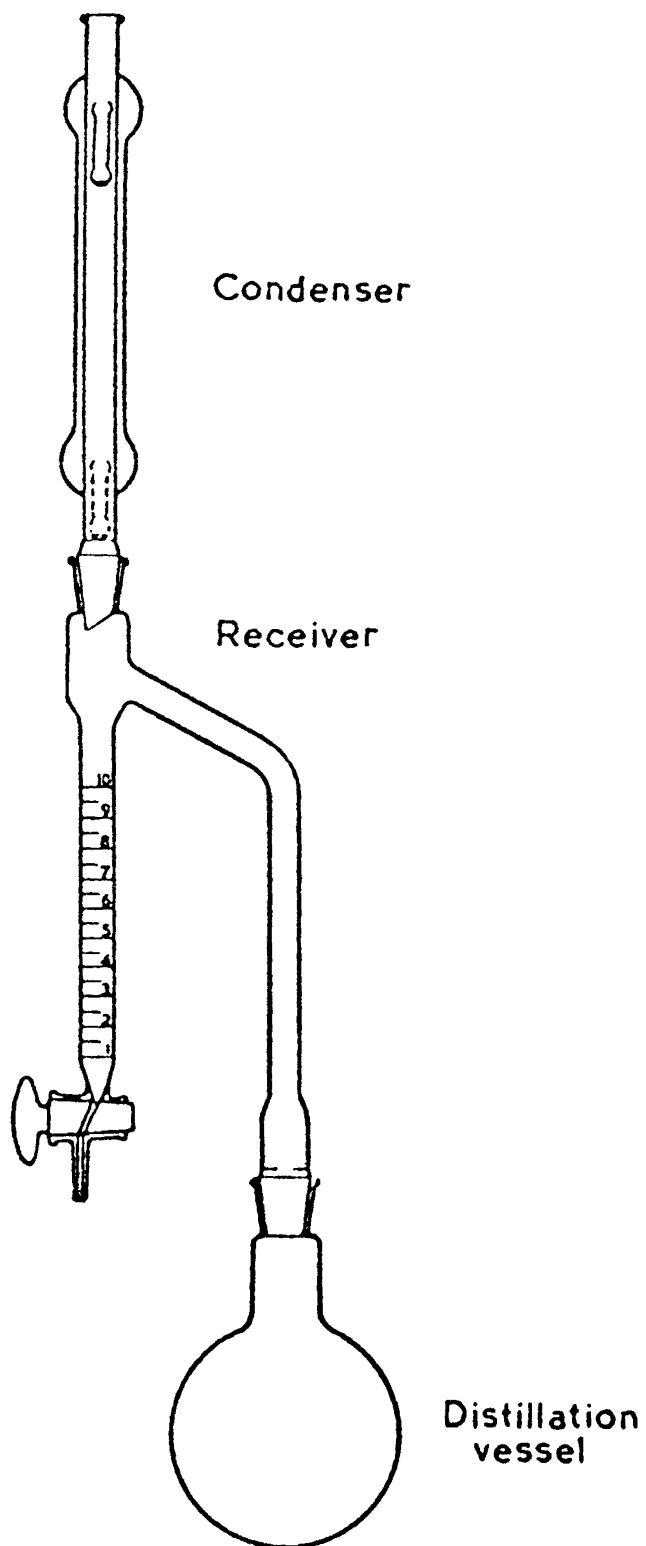


Figure 1 — Typical assembly of Dean and Stark apparatus

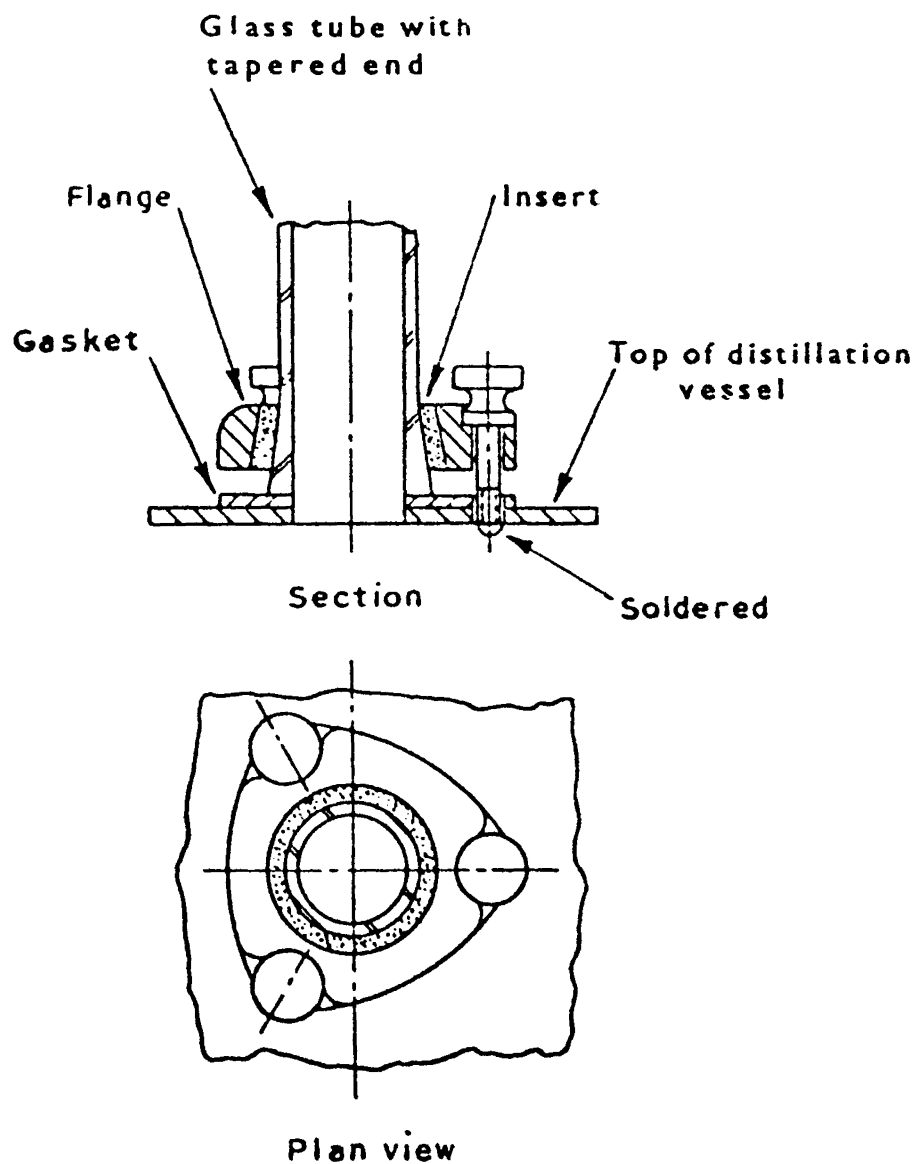


Figure 2 — Flanged joint suitable for use with metal distillation vessel

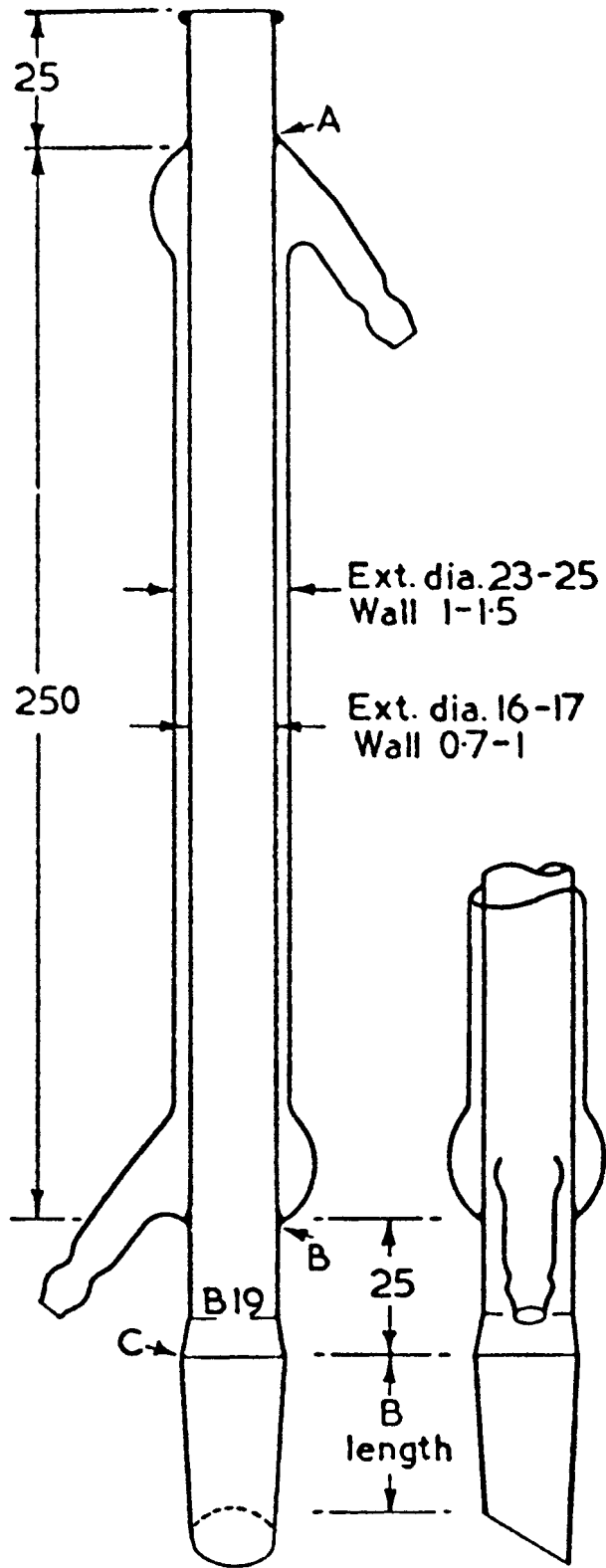


Figure 3 — Condenser
 (All dimensions are in millimetres)

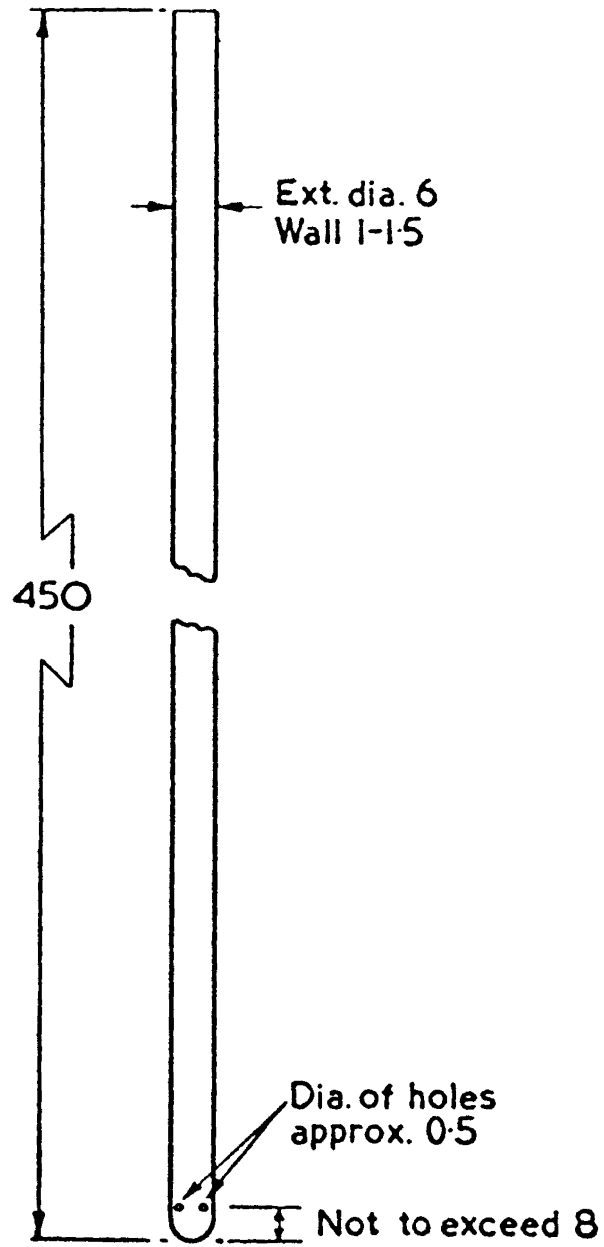


Figure 4 — Spray tube
(All dimensions are in millimetres)

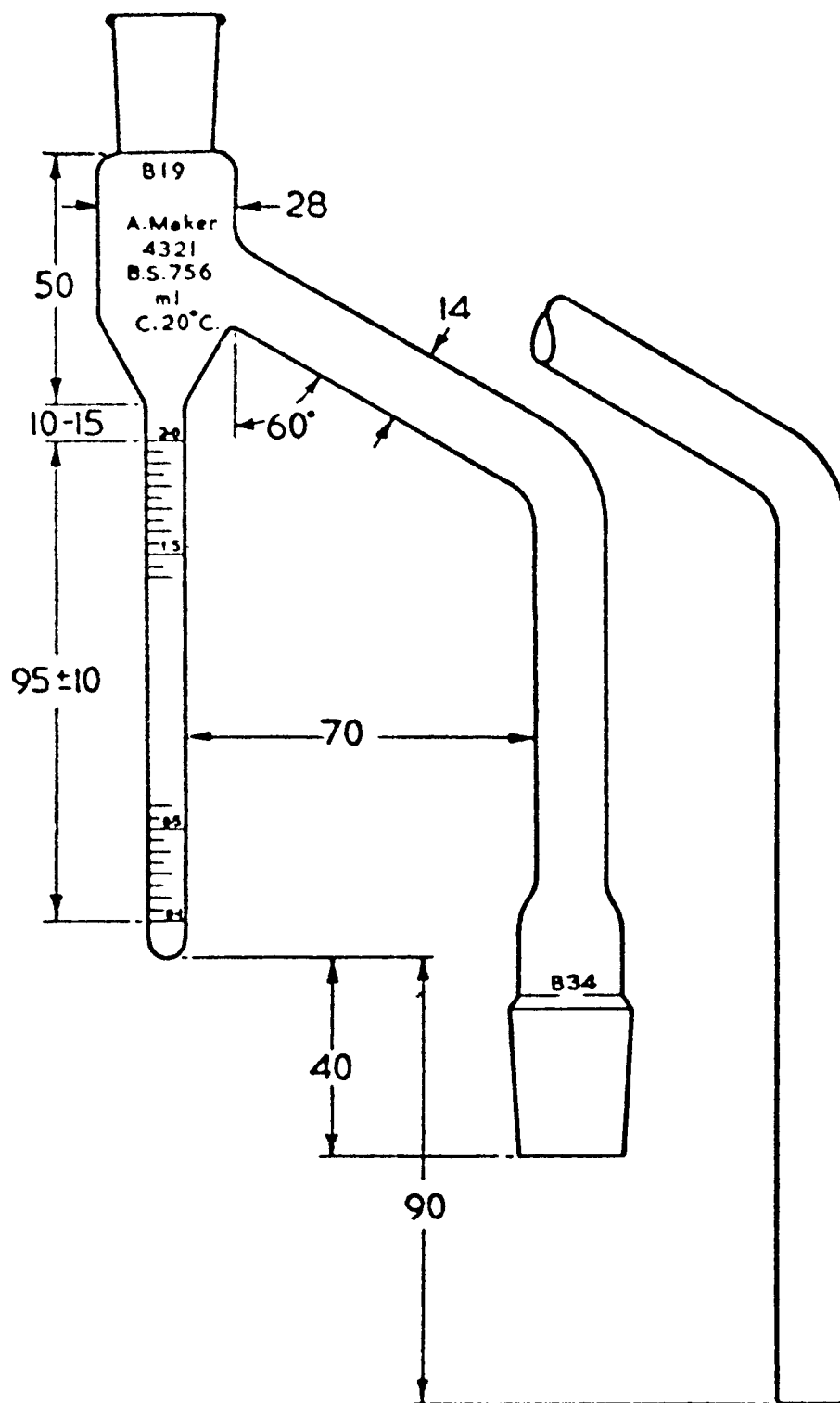


Figure 5 — 2 ml. receiver showing alternative connections to distillation vessel

(Also available with B24 joint)

(All dimensions are in millimetres)

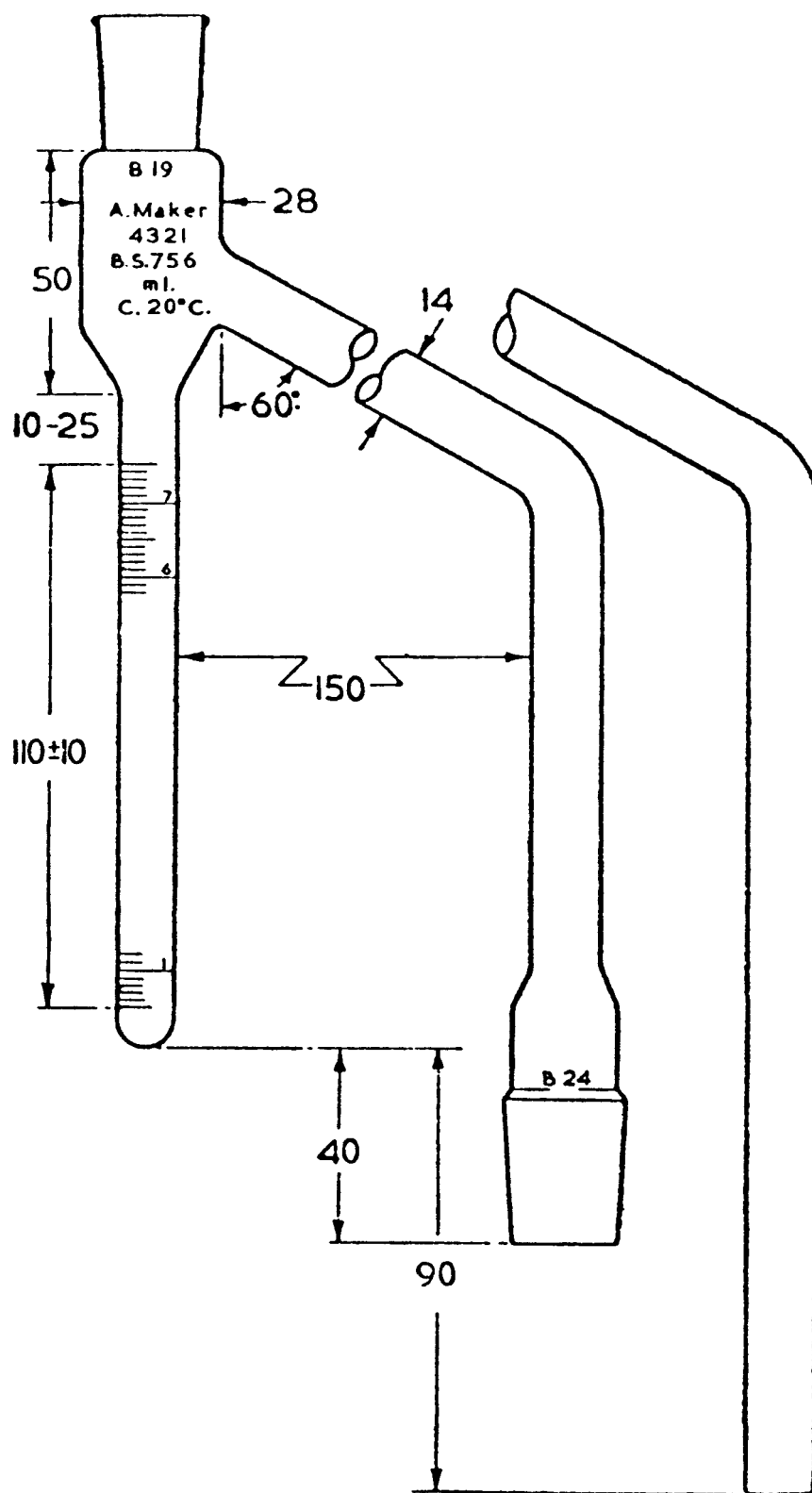


Figure 6 — 7.5 ml. receiver showing alternative connections to distillation vessel
(All dimensions are in millimetres)

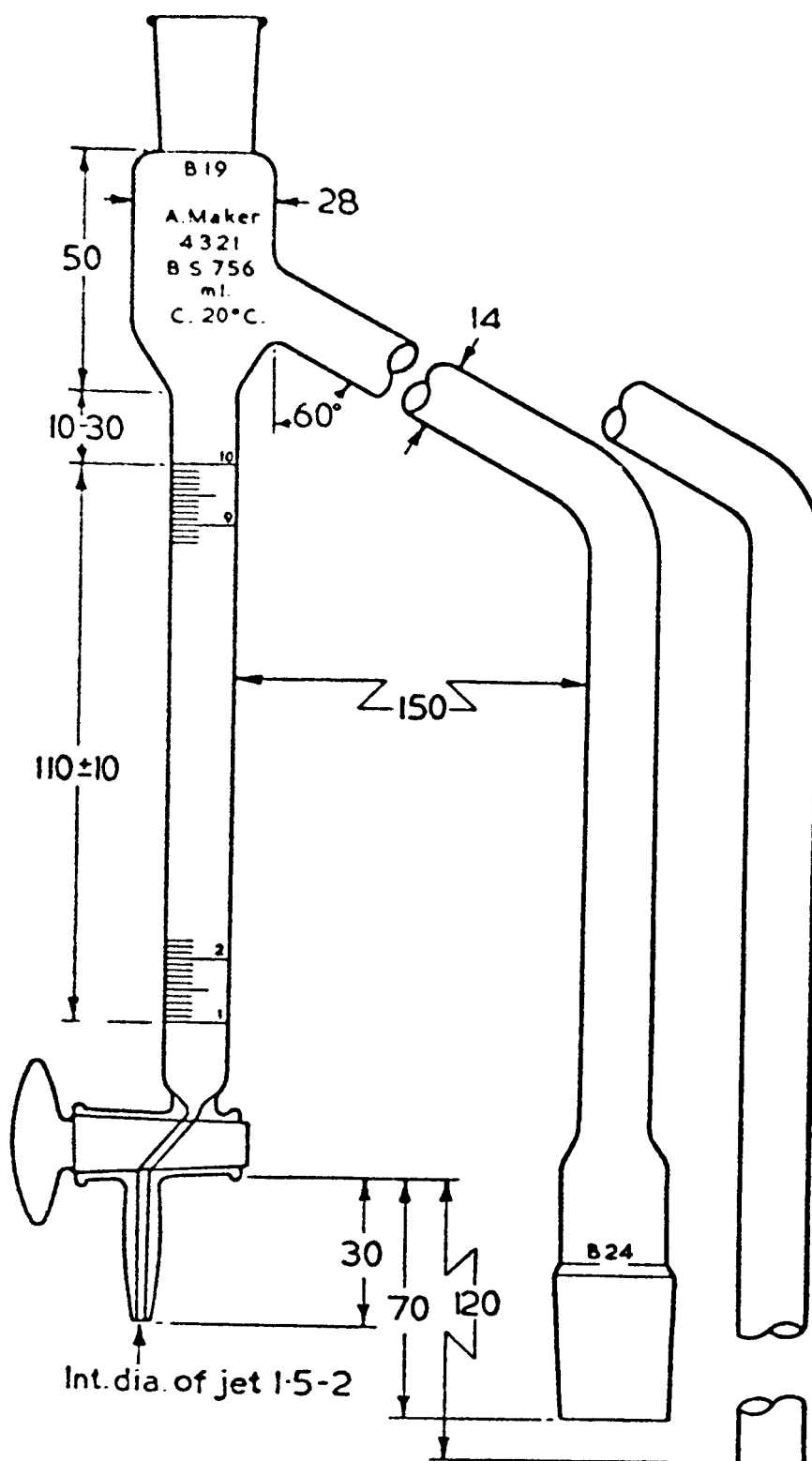


Figure 7 — 10 ml. receiver, showing alternative connection to distillation vessel
(All dimensions are in millimetres)

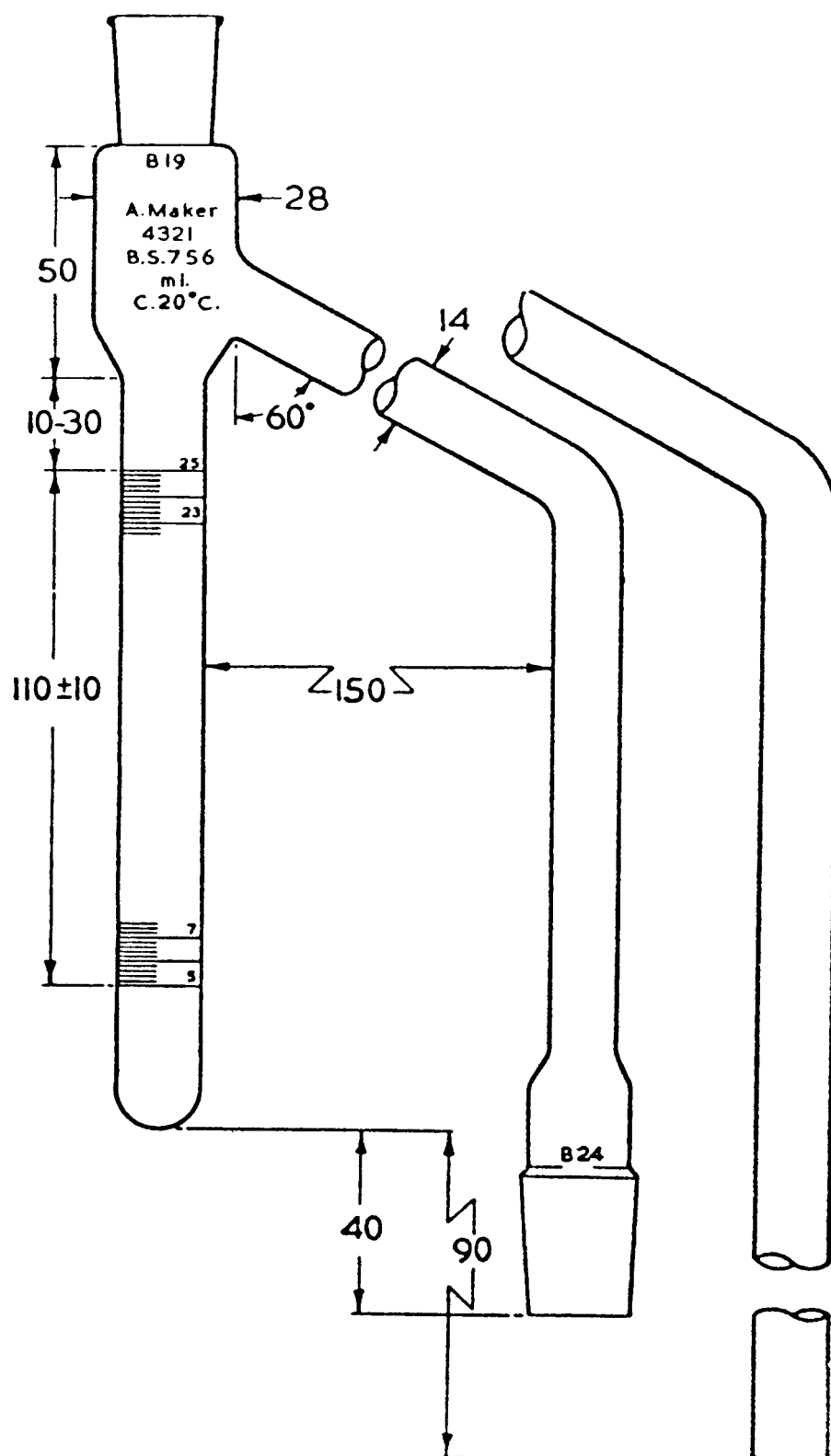


Figure 8 — 25 ml. receiver without stopcock, showing alternative connections to distillation vessel

(All dimensions are in millimetres)

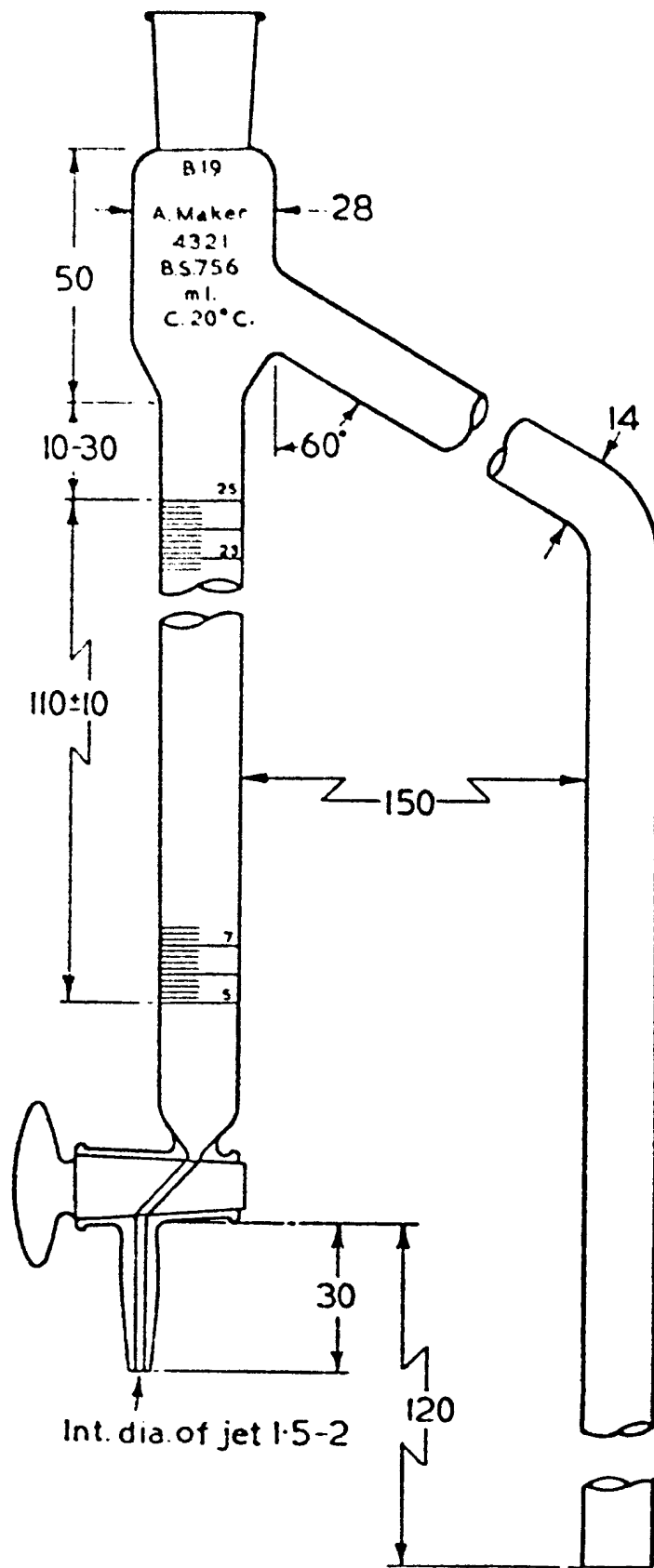


Figure 9 — 25 ml. receiver with stopcock
 (All dimensions are in millimetres)

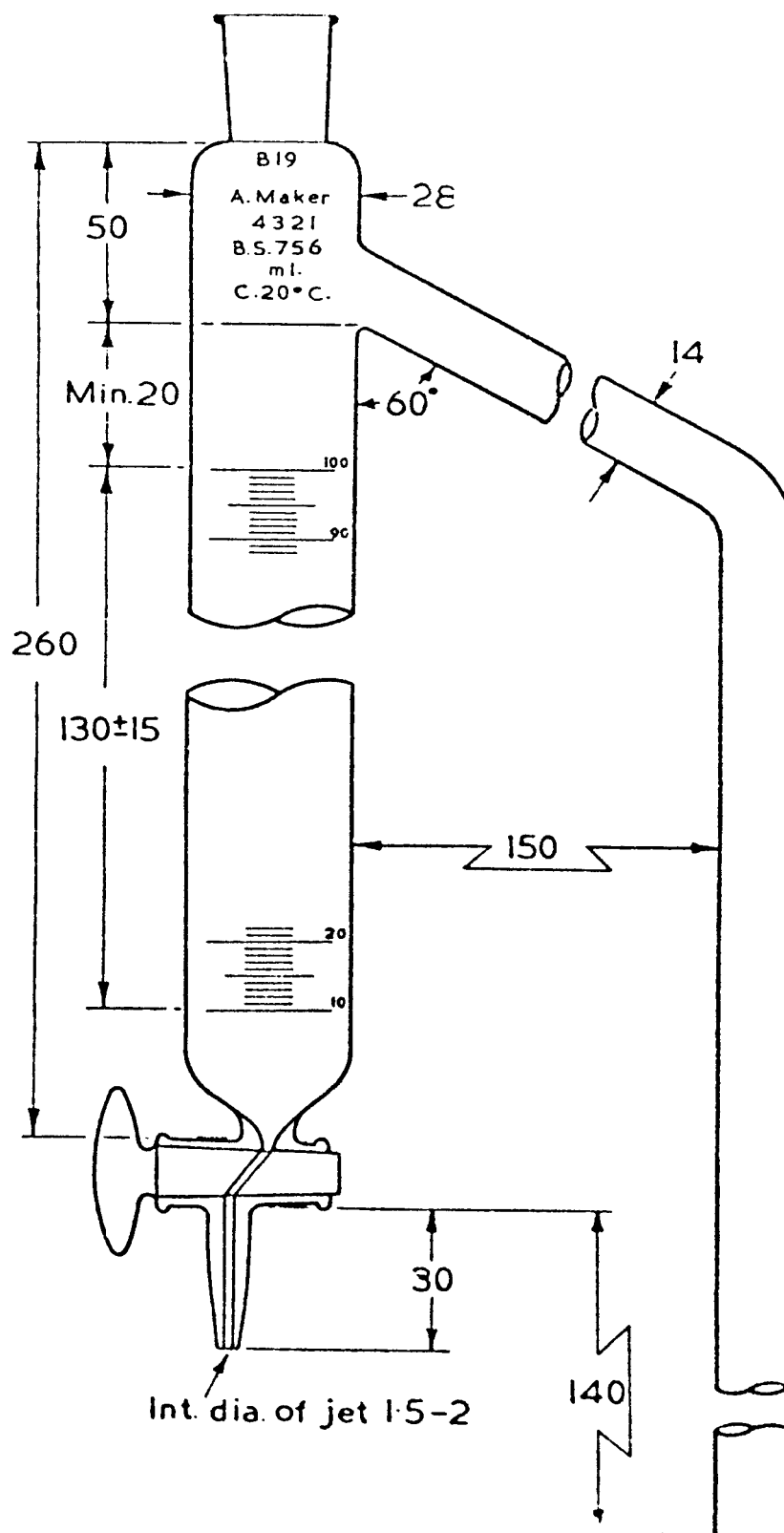


Figure 10 — 100 ml. receiver
(All dimensions are in millimetres)

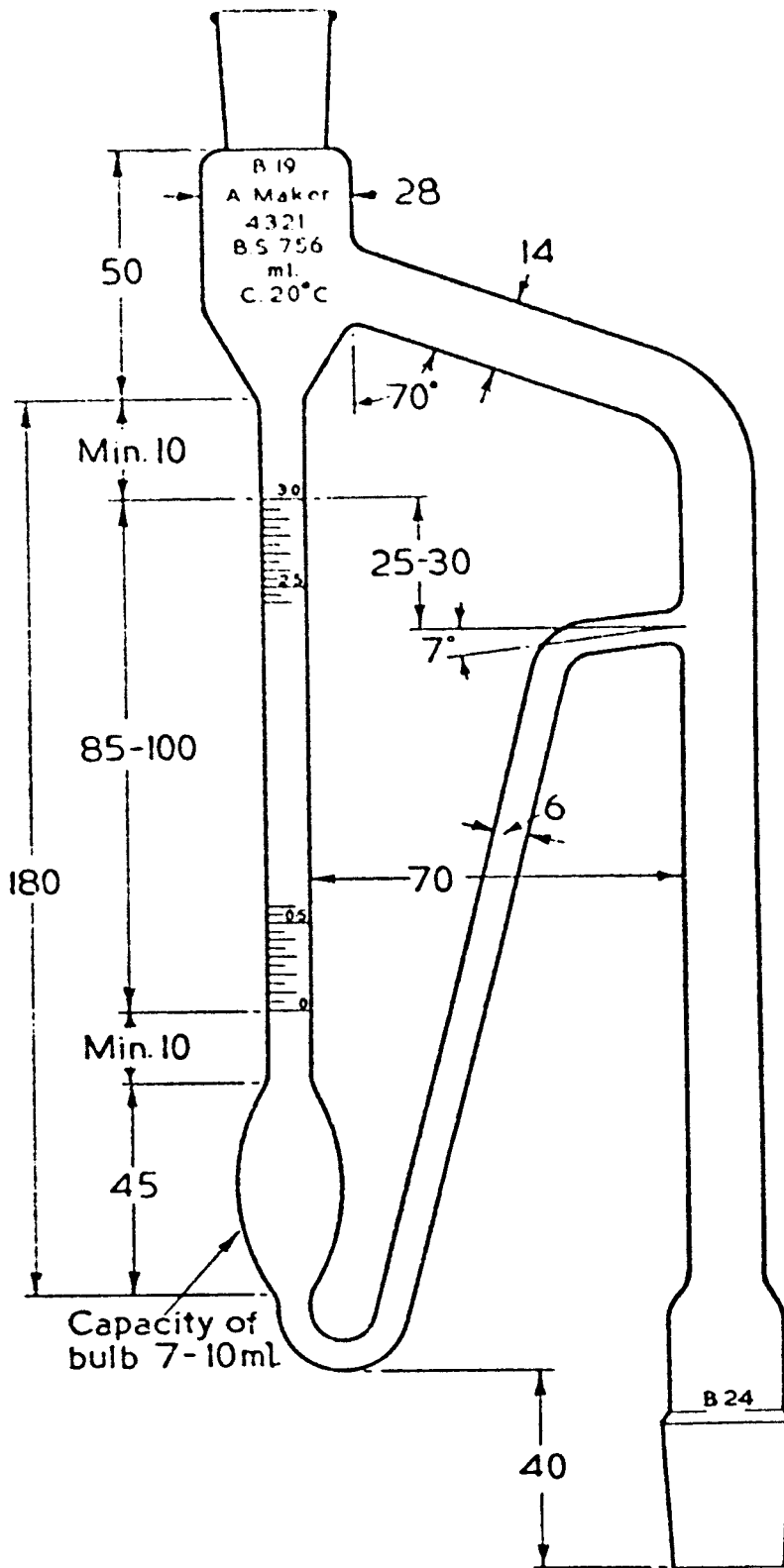


Figure 11 — 3 ml. Type 2 receiver
(All dimensions are in millimetres)

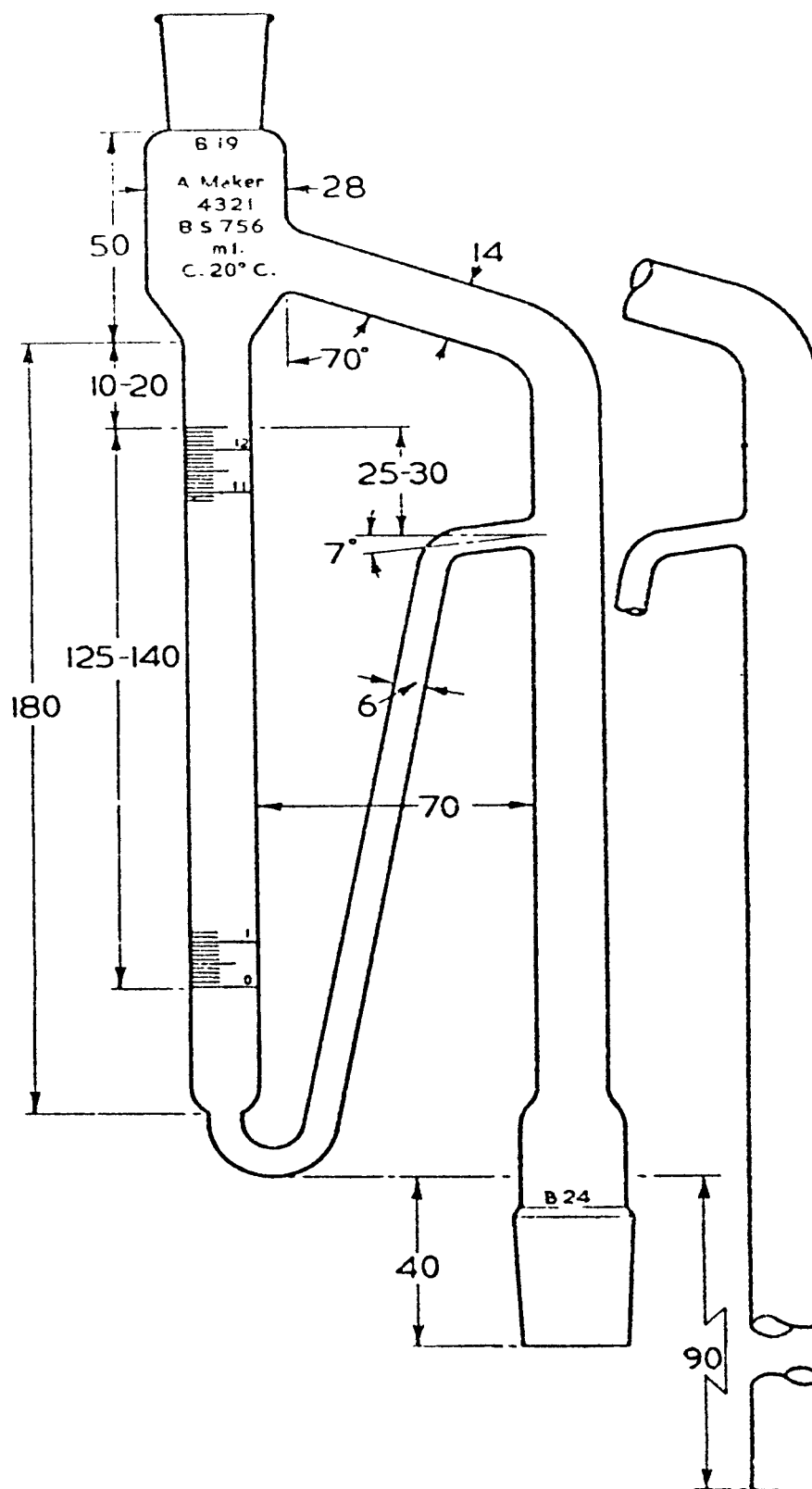


Figure 12 — 12.5 ml. Type 2 receiver, showing alternative connections to distillation vessel
(All dimensions are in millimetres)

British Standards

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