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# International Standard



# 4626

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials

*Liquides organiques volatils — Détermination de l'intervalle de distillation des solvants organiques utilisés comme matières premières*

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**Descriptors** : paints, varnishes, organic materials, volatile matter, liquids, tests, boiling temperature tests, measurement, boiling point, distillation range.

## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4626 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in September 1976.

It has been approved by the member bodies of the following countries :

Australia	Iran	Poland
Austria	Israel	Romania
Brazil	Italy	South Africa, Rep. of
Bulgaria	Korea, Rep. of	Sweden
Canada	Mexico	Switzerland
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	United Kingdom
France	Norway	Yugoslavia
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The member bodies of the following countries expressed disapproval of the document on technical grounds : <https://standards.iteh.ai/catalog/standards/sist/0bbf6478-ef1f-4d4f-abb6-ae212df9e7dd/iso-4626-1980>

Germany  
U.S.A.

This International Standard is one of three ISO publications dealing with the determination of distillation characteristics. The two others are :

- ISO/918, *Test method for distillation (distillation yield and distillation range)*.
- ISO 3405, *Petroleum products — Determination of distillation characteristics*.

It is recognized that there may be some overlapping between these three documents which were developed in separate technical committees. In the absence of agreement on a general International Standard on the subject, it has however been thought necessary to publish them.

The completion in the future of such a general standard may therefore lead to the amendment or cancellation of this International Standard.

# Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials

## SAFETY PRECAUTIONS

### A Peroxide formation

Certain solvents and chemical intermediates, particularly but not only ethers and unsaturated compounds, may form peroxides during storage. These peroxides may present an explosion hazard when the product is distilled, especially as the dry point is approached.

When peroxide formation is likely, either because of the chemical nature of the product, the type of the product or its length of storage, the material should be analysed for peroxides and, if they are present, appropriate precautions should be taken, such as destruction of the peroxides before distillation, or protection of the operator.

#### Test for peroxides

Add 0,5 to 1,0 ml of the material to be tested to an equal volume of glacial acetic acid to which has been added about 100 mg of sodium or potassium iodide crystals.

Carry out a blank determination. A comparatively yellow colour indicates a low and a brown colour a high concentration of peroxide in the sample.

### B Flammability

Most organic solvents and chemical intermediates are flammable. A fire hazard exists when a distillation is carried out and safety precautions should be taken.

Before starting the test, the flask should be examined for flaws and care should be taken that good seals are obtained where the side arm connects with the condenser and where the thermometer fits into the neck. Some solvents are liable to auto-ignition if distilled. Distillation of these products should be avoided. During the distillation, a suitable catch-pan and shield should be used to contain spilled liquid in the event of accidental breakage of the distillation flask.

Adequate ventilation should be provided to maintain the solvent vapour concentration below the explosive limit in the immediate vicinity of the distillation apparatus, and below the threshold limit value in the general work area.

## 1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies a method for determining the boiling range of liquids that boil between 30 and 300 °C at normal pressure, and that are chemically stable and do not corrode the apparatus during the distillation.

1.2 The method is applicable to organic liquids such as

hydrocarbons, esters, alcohols, ketones, ethers and similar products.

NOTE — The method differs from that described in ISO/R 918 with respect to the volume of the distillation flask, the type of cooler and the distillation receiver.

The method differs from that specified in ISO 3405 with respect to the volume of the distillation flask and the diameter of the hole in the flask support.

## 2 REFERENCES

ISO 842, *Raw materials for paints and varnishes — Sampling*.

ISO/R 918, *Test method for distillation (distillation yield and distillation range)*.

ISO 3165, *Sampling of chemical products for industrial use — Safety in sampling*.

ISO 3405, *Petroleum products — Determination of distillation characteristics*.

## 3 DEFINITIONS

**3.1 initial boiling point:** The temperature noted (corrected if required) at the moment when the first drop of condensate falls from the tip of the condenser during a distillation carried out under standardized conditions.

**3.2 dry point:** The temperature noted (corrected if required) at the moment of vaporization of the last drop of liquid at the bottom of the flask during a distillation carried out under standardized conditions, disregarding any liquid on the side of the flask and on the thermometer.

**3.3 boiling range:** The temperature interval between the initial boiling point and the dry point.

**3.4 end point; final boiling point:** The maximum temperature noted (corrected, if required) during the final phase of a distillation carried out under standardized conditions.

## 4 PRINCIPLE

Distillation of a 100 ml test portion under prescribed conditions which are equivalent to a simple batch distillation. Systematic observation of thermometer readings and volumes of condensate and calculation of the results from these data with correction to standard atmospheric pressure.

## 5 APPARATUS

The apparatus, a suitable form of which is shown in figures 1 to 4, shall comprise the following items:

**5.1 Distillation flask,** of heat-resistant glass, of capacity 200 ml, conforming to the dimensions shown in figure 1.

NOTE — Superheating of liquid in a new flask may be prevented by depositing a small amount of carbon in the bottom of the flask. This may be accomplished by heating and decomposing a pinch of tartaric acid in the bottom of the flask. The flask is then prepared for use by washing with water, rinsing with acetone, and drying.

An exception is made for diacetone alcohol: in order to avoid an erratic value for the initial boiling point, the distillation flask should be clean and free of any residual carbon deposit.

**5.2 Thermometers,** mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, and conforming to the requirements in table 1.

NOTE — The thermometer should have been artificially aged by means of a suitable treatment before graduation, in order to ensure stability of the lowest point on the scale. This treatment should have been such that, after the procedure described below, the rise at a fiducial point is not greater than the maximum error specified, and the accuracy of the thermometer is within the limits specified.

Heat the thermometer to a temperature equal to its highest reading and keep it at this temperature for 5 min. Allow the thermometer to cool, either naturally in still air or slowly in the test bath (at a specified rate), to 20 °C above ambient temperature or to 50 °C, whichever is the lower, and then determine the lowest point on the scale. If rapid cooling is used, the lowest point on the scale shall be determined within 1 h. Heat the thermometer again to a temperature equal to its highest reading, keep it at this temperature for 24 h, allow the thermometer to cool to one of the two temperatures referred to above, at the same rate as at the start of the test, and re-determine the lowest point on the scale under the same conditions as before.

## 5.3 Draught screen

### 5.3.1 For use with a gas burner

The draught screen shall be rectangular in cross-section and open at the top and bottom. It shall have the dimensions shown in figure 2 and be made of sheet of metal of thickness approximately 0,8 mm.

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Dimensions in millimetres

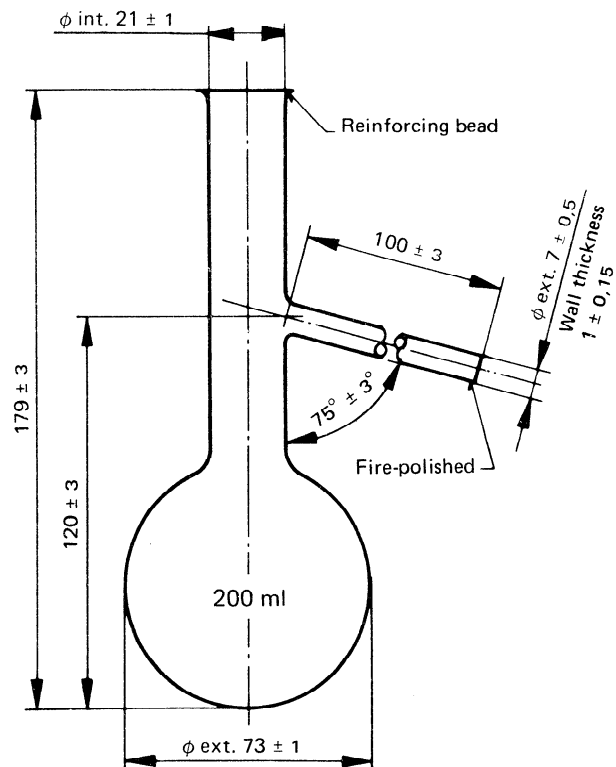


FIGURE 1 — Distillation flask (5.1)

TABLE 1 — Solvent distillation thermometers

Designation <sup>1)</sup>	38 °C-75	39 °C-75	40 °C-75	41 °C-62	42 °C-62	102 °C-65	103 °C-65	104 °C-75	105 °C-65	106 °C-75	107 °C-75
Immersion, mm	100	100	100	100	100	100	100	100	100	100	100
Range, °C	24 to 78	48 to 102	72 to 126	98 to 152	95 to 255	123 to 177	148 to 202	173 to 227	198 to 252	223 to 277	248 to 302
Graduation, °C	0,2	0,2	0,2	0,2	0,5	0,2	0,2	0,2	0,2	0,2	0,2
Longer lines at each, °C	1	1	1	1	1	1	1	1	1	1	1
Figured at each, °C	2	2	2	2	5	2	2	2	2	2	2
Scale error not to exceed, °C	0,2	0,2	0,2	0,2	0,5	0,2 up to 150 °C 0,3 over 150 °C	0,4	0,4	0,4 up to 225 °C 0,6 over 225 °C	0,8	1,0
Overall length, mm	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5
Stem diameter, mm	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 8,0	6,0 to 8,0
Bulb length, mm	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20
Distance from bottom of bulb, mm	24 °C 125 to 145	48 °C 125 to 145	72 °C 125 to 145	98 °C 125 to 145	95 °C 125 to 145	123 °C 125 to 145	148 °C 125 to 145	173 °C 125 to 145	198 °C 125 to 145	223 °C 125 to 145	248 °C 125 to 145
— to	78 °C 335 to 360	102 °C 335 to 360	126 °C 335 to 360	152 °C 335 to 360	255 °C 335 to 360	177 °C 335 to 360	202 °C 335 to 360	227 °C 335 to 360	252 °C 335 to 360	277 °C 335 to 360	302 °C 335 to 360
Expansion chamber to allow heating to, °C	105	130	150	180	280	200	225	250	275	300	325

1) These designations correspond to those in ASTM E1, *Standard specification for ASTM thermometers*.

In each of the two narrower sides of the draught screen, there shall be two circular holes of diameter 25 mm, the centres of which are situated 215 mm below the top of the shield, as shown in figure 2.

In each of the four sides of the draught screen, there shall be three circular holes of diameter 12,5 mm, the centres of which are situated 25 mm above the base of the draught screen. These holes shall occupy the positions shown in figure 2.

At the middle of each of the wider sides, a vertical slot for the condenser tube, dimensioned approximately as shown in figure 2, shall be cut downwards from the top of the screen. A removable shutter of suitable dimensions shall be provided for closing whichever vertical slot is not in use. This arrangement enables the condenser (5.6) to be placed on either side of the draught screen.

A shelf of ceramic material, of thickness 3 to 6 mm and possessing a centrally cut circular hole of diameter 75 to 100 mm, shall be supported horizontally in the screen and shall fit closely to the sides of the screen, to ensure that hot gases from the source of heat (5.5) do not come in contact with the sides or neck of the flask (5.1). The supports for this shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

A board as described in 5.4 shall rest on this shelf.

In one of the narrower sides of the screen, a door shall be provided having the approximate dimensions shown in figure 2 and overlapping the opening in the screen by approximately 5 mm all round.

### 5.3.2 For use with an electric heater

When an electric heater is employed, the portion of the draught screen above the shelf shall be as described in 5.3.1, but the lower portion (including the shelf) may be modified or omitted, provided that the change does not expose the distillation flask to draughts (see figure 3). Provision shall be made for adjustment of the shelf to facilitate fitting of the flask.

**5.4 Ceramic boards**, of thickness 3 to 6 mm, with central holes of diameter 32 or 38 mm respectively and overall dimensions not less than 150 mm square. When a gas heater is employed, this board shall rest on the shelf described in 5.3.1. When an electric heater is employed, the same arrangement shall be adopted if the shelf is present; alternatively, the board may be placed directly on the heater or it may form the top of the heater. Provision shall be made for adjusting the height of the heater.

Whichever type of heater is employed, direct heat shall only be applied to the flask through the central hole in the ceramic board.

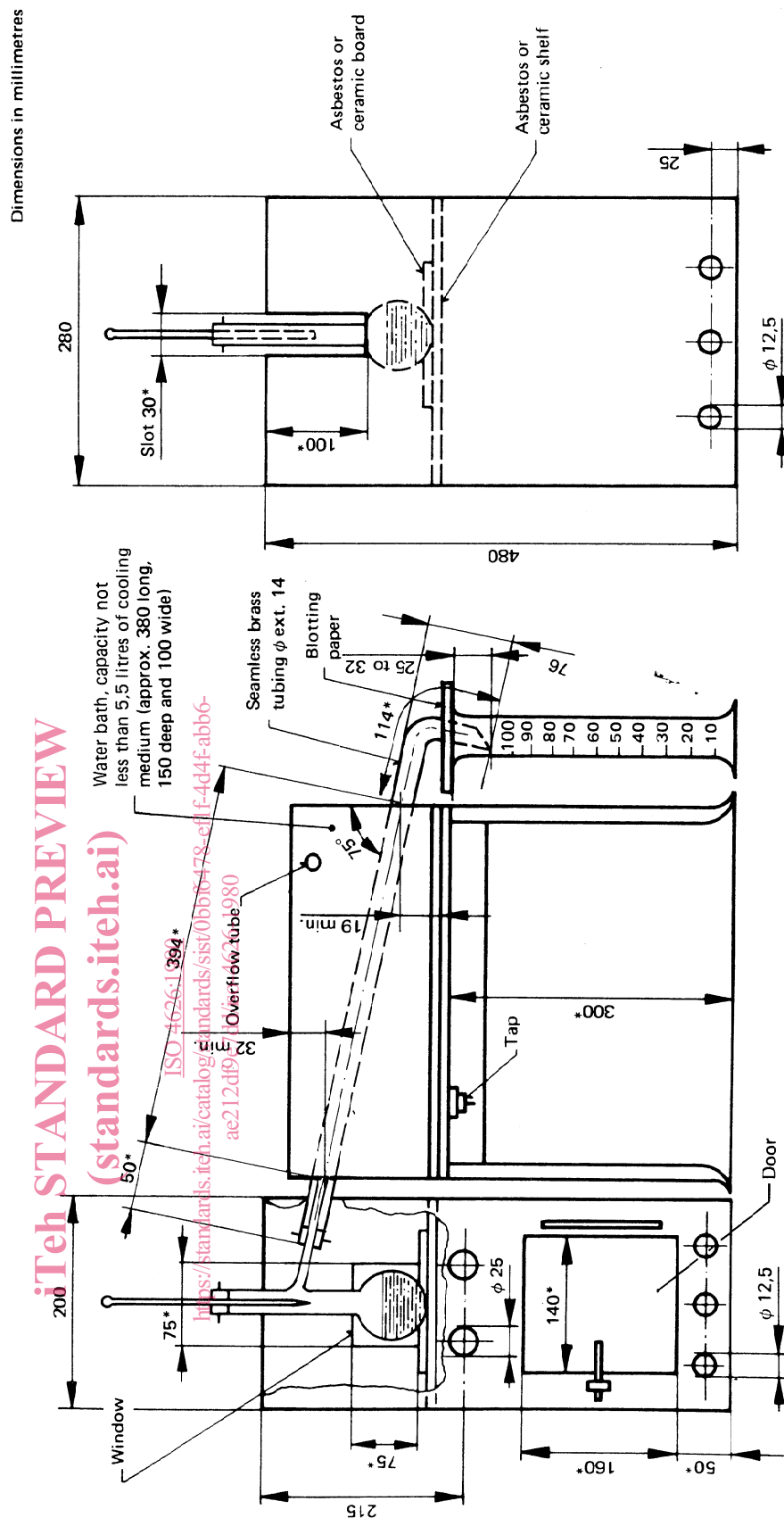
**5.5 Source of heat**, comprising either a gas burner so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in 8.3.1 (a sensitive regulating valve or governor are desirable adjuncts) or an electric heater capable of complying with the same requirements. (A heater of low heat retention, adjustable from 0 to 1 kW, has been found satisfactory.) See also 9.6.

**5.6 Condenser**, of seamless brass tubing, of length 560 mm, outside diameter 14 mm and wall thickness 0,8 to 0,9 mm, surrounded by a metal cooling bath, preferably of copper or brass. The tube shall be set so that a length of approximately 390 mm is in contact with the cooling medium in the cooling bath, with about 50 mm outside the cooling bath at the upper end, and about 115 mm outside at the lower end. The length of the tube projecting at the upper end shall be straight and set at an angle of  $75^\circ$  to the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous smooth curve; the average gradient of this section shall be 0,26 mm per linear millimetre of the condenser tube (sine of angle of  $15^\circ$ ), and no part of it shall have a gradient less than 0,24 mm nor more than 0,28 mm per linear millimetre of the tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and slightly backward so as to ensure contact with the wall of the receiver (5.7) at a point 25 to 32 mm below the top of the receiver when it is in a position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle so that the tip may be brought into contact with the wall of the receiver.

The capacity of the cooling bath shall be not less than 5,5 litres of cooling medium. The arrangement of the tube in the cooling bath shall be such that its centre line is not less than 32 mm below the plane of the top of the bath at its point of entrance, and not less than 19 mm above the floor of the bath at its exit. Clearances between the condenser tube and the walls of the bath shall be at least 13 mm, except for the section adjacent to the points of entrance and exit.

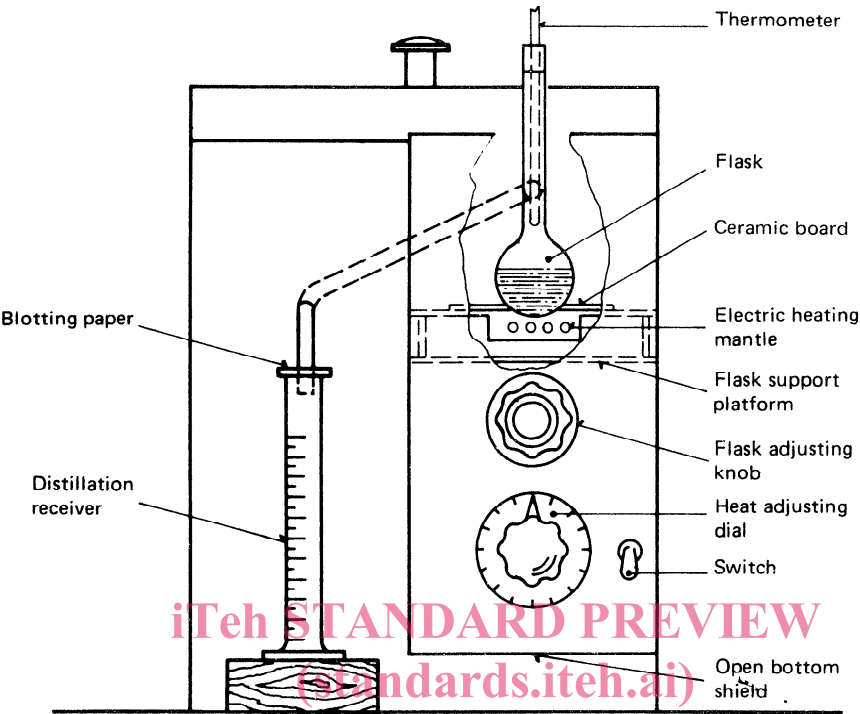
The cooling bath may be provided with a tap at the bottom for drainage or inlet, and with an overflow tube near the top.

The main dimensions of the tube and cooling bath are shown in figure 2.



**FIGURE 2 – Distillation apparatus using gas burner**





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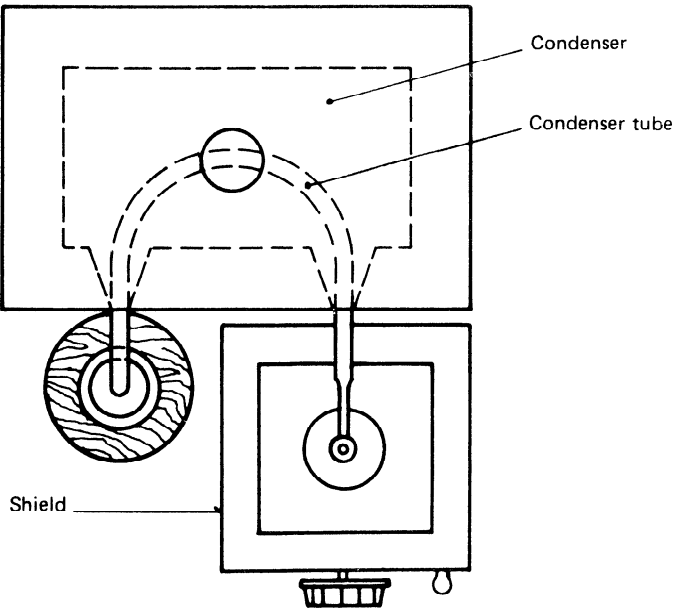


FIGURE 3 — Distillation apparatus using electric heater



**5.7 Receiver**, of capacity 100 ml, complying with the details shown in figure 4. None of the graduation lines shall be in error by more than 1 ml. The shape of the base is optional but it shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of  $15^\circ$  to the horizontal.

**5.8 Barometer**, accurate to the nearest 1 mbar, 0,1 kPa or 1 mmHg.

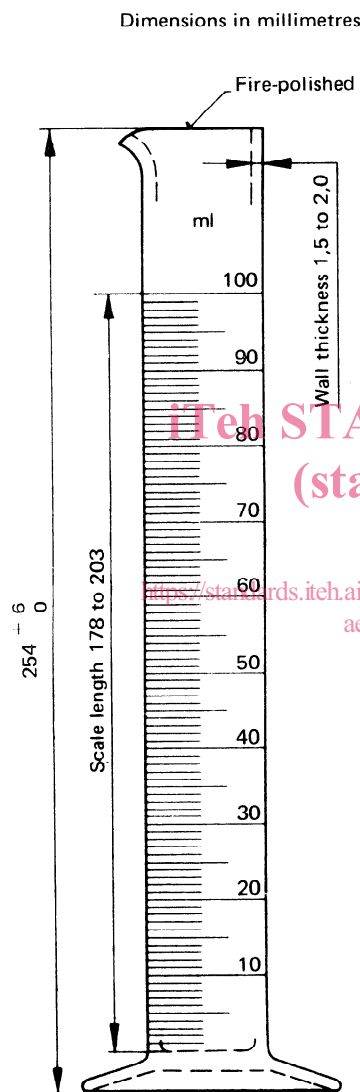


FIGURE 4 — Distillation receiver

## 6 SAMPLING

Representative samples, each having a volume of not less than 500 ml, should, wherever possible, be taken in triplicate from one or more original and previously unopened containers or from the bulk during packing, as may be agreed between the interested parties, and shall be packaged in clean, dry, airtight containers of dark glass or metal. The containers shall be of such a size that they are

nearly filled by the sample. Each sample container so filled shall be sealed with a material unaffected by the contents and marked with the full details and date of sampling.

Guidance on sampling is given in ISO 842.

## 7 ASSEMBLY AND PREPARATION OF APPARATUS

### 7.1 Assembly

Assemble the apparatus as shown in figure 2 or figure 3, swabbing out the condenser with a piece of lint-free cloth attached to a wire cord or by any other suitable means, and paying attention to the following details.

#### 7.1.1 Position and choice of thermometer

Use the thermometer listed in the specification for the product under test, or that indicated in table 3. If no thermometer is specified, select one from table 1, with the smallest graduations that will cover the entire boiling range of the product.

Centre the thermometer into the neck of the flask through a tight-fitting silicone-rubber or cork stopper so that the upper end of the contraction chamber is level with the lower side of the vapour tube at its junction with the neck of the flask.

**NOTE** — It is far more important that the greatest possible volume of mercury be immersed in the refluxing zone than that the immersion mark on the thermometer be placed at any specific point.

#### 7.1.2 Support for flask

If a draught screen with ceramic shelf is used, place the appropriate ceramic board (see 5.4 and 8.3) on top of the shelf so that the two holes are concentric.

#### 7.1.3 Connection of flask to condenser

Make a leak-proof connection of the flask (5.1) to the tube of the condenser (5.6) by means of a tight-fitting silicone-rubber or cork stopper through which the vapour tube of the flask passes. Connect the flask to the condenser so that the flask is in a vertical position; the end of the vapour tube shall extend at least 25 mm and not more than 50 mm beyond the cork into the condenser tube and shall be co-axial with it.

Place the flask in such a position on the board that the base completely closes the hole in the board.

### 7.2 Filling of cooling bath

Fill the bath with water or with water and cracked ice in sufficient quantity to cover the condenser tube, so as to ensure that the temperature of the bath at the start of and during distillation is as shown in table 2.