
INTERNATIONAL STANDARD



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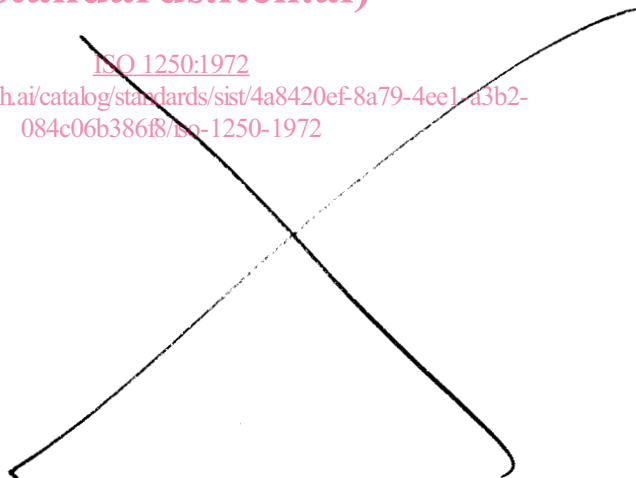
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**Mineral solvents for paints —
White spirits and related hydrocarbon solvents**

First edition — 1972-12-15

iTeh STANDARD PREVIEW
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<https://standards.iteh.ai/catalog/standards/sist/4a8420ef-8a79-4ee1-a3b2-084c06b386f8/iso-1250-1972>



UDC 667.629.2

Ref. No. ISO 1250-1972 (E)

Descriptors : paints, solvents, hydrocarbons, materials specifications, characteristics, tests.

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 1250 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*.

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It was approved in March 1969 by the Member Bodies of the following countries :

Austria	Iran	Spain
Belgium	Israel	Sweden
Brazil	Netherlands	Switzerland
Denmark	New Zealand	Turkey
Egypt, Arab Rep. of	Peru	United Kingdom
Germany	Poland	U.S.S.R.
Greece	Portugal	
India	South Africa, Rep. of	

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The Member Body of the following country expressed disapproval of the document on technical grounds :

France

Mineral solvents for paints – White spirits and related hydrocarbon solvents

0 INTRODUCTION

This International Standard is intended to cover the essential requirements of mineral solvents used in the paint industry, which may be considered as falling into two categories, according to aromatics content, namely: Type A, aromatics content less than 25 %, and Type B, aromatics content of 25 to 50 %. It was at first intended to use the simple title, "White spirit", but discussion showed that this term would not be generally acceptable for such a wide range of solvents, because in some countries it is used with a much more restricted meaning. Accordingly a longer, but more explanatory, title was adopted for the International Standard.

Some of the methods of test given in this document are technically identical with the widely known methods standardized by the American Society for Testing and Materials which are themselves under study by ISO/TC 28, *Petroleum products*. It is expected in due course, therefore, to replace these test methods by cross-references to appropriate International Standards.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements for two categories of mineral solvents for use in paints and varnishes, as follows:

- Type A: having an aromatic content below 25 % (V/V);
- Type B: having an aromatic content of 25 to 50 % (V/V).

2 REFERENCES

ISO/R 842, *Sampling raw materials for paints and varnishes*.

ISO 2049, *Petroleum products – Determination of colour*.

ISO 2160, *Petroleum products – Corrosiveness to copper – Copper strip test*.

3 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Mineral solvents consist essentially of a mixture of hydrocarbons, but the presence of a denaturant is permitted when agreed between the interested parties; they shall have the characteristics shown in Table 1, page 2.

4 SAMPLES

4.1 Representative samples, each having a volume of not less than 500 ml, shall, 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 packed 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/R 842.

4.2 If an agreed sample is required for the purpose of section 3 in relation to odour, it shall comply in all other respects with the requirements of this specification. It shall have a volume of not less than 500 ml and shall be packed in the manner described in 4.1.

TABLE 1 – Required characteristics and their tolerances

Characteristic	Requirement	Test method
Clarity	clear, no solid matter present	visual inspection
Undissolved water at 20 °C	absent	visual inspection
Odour	if required by purchaser, conform to agreed sample	—
Colour	not darker than standard	see 5.1
Distillation at 1 013 mbar; volume of condensate recovered Method a)*	these limits apply to mineral solvents for paints with or without denaturant not more than 1 ml below 130 °C not more than 10 ml below 145 °C not less than 90 ml below 200 °C end point, not above 220 °C	see 5.2
Method b)*	not more than 1 ml below 132 °C not more than 10 ml below 148 °C not less than 90 ml below 204 °C dry point, not above 220 °C.	see 5.3
Aromatic content	type A : less than 25 % (V/V) type B : 25 to 50 % (V/V) if required, more precise limits may be agreed between the interested parties	see 5.4
Residue on evaporation	not more than 10 mg per 100 ml	see 5.5
Neutrality	when 50 ml of sample are shaken with 10 ml of distilled water, the water layer shall be neutral to methyl orange; by agreement between the interested parties, this requirement may be waived or modified in relation to denatured materials.	
Freedom from objectionable sulphur compounds	no more than slight tarnish of copper strip	see ISO 2160
Aniline point	if required, to be agreed between the interested parties	see 5.6
Flash point	if required, to be agreed between the interested parties	to be agreed between the interested parties
Viscosity reduction power	if required, to be agreed between the interested parties	see 5.7

* By Methods a) and b) are intended the methods specified in 5.2 and 5.3 and corresponding respectively to ASTM D 86 and D 1078, and to equivalent national standards.

For comparison of distillation data obtained by the two methods, use the conversion equations :

$$t_b = (t_a \times 1,025) - 0,993$$

$$t_a = (t_b \times 0,975) + 0,969$$

where

t_a is the temperature, in degrees Celsius, corresponding to a given volume of distillate, recorded according to Method a);

t_b is the temperature, in degrees Celsius, corresponding to the same volume of distillate, recorded according to Method b).

These relations were established by T.R. Donlan (See Materials, Research and Standards, October 1962.)

5 METHODS OF TEST

5.1 Method for the comparison of colour

5.1.1 Standard colour solution

Dissolve 4,8 mg of pure anhydrous potassium dichromate in 1 l of distilled water or deionized water of at least equal purity.

5.1.2 Apparatus

Two 50 ml Nessler cylinders with the height of the 50 ml mark above the inside of the base matched to within 1 mm in the range 110 to 116 mm.

5.1.3 Procedure

Pass the sample through a filter paper about 150 mm in diameter and reject the first 10 ml of filtrate. Fill one of the Nessler cylinders to the mark with the filtered sample, and the other with the standard colour solution. Place the cylinders vertically 75 mm above the surface of an opaque opal glass sheet reflecting diffuse daylight, and compare the colour of the sample with that of the standard colour solution. Report the colour of the sample as being equal to, or lighter or darker than the colour of the standard colour solution.

5.1.4 Remark

Alternative methods, such as in ISO 2049, employing permanent colour standards and giving results equivalent to the specified colour, may be used by agreement between the interested parties.

5.2 Distillation test – Method a)

5.2.1 Definitions

5.2.1.1 volume recovered: The volume, expressed in millilitres, of condensate collected in the receiver at the specified temperature readings on the thermometer.

5.2.1.2 end point (final boiling point): The maximum temperature indicated during the distillation. This temperature is usually reached after the evaporation of all liquid from the bottom of the flask.

5.2.2 Apparatus

The apparatus, a suitable form of which is shown in Figures 1 and 2, shall comprise :

5.2.2.1 Distillation flask, in heat-resistant glass, of 125 ml distillation capacity, conforming to the dimensions shown in Figure 3 a).

5.2.2.2 Thermometer, mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, and conforming to the following requirements¹⁾ :

Range	– 2 to + 300 °C
Graduation	1 °C
Immersion	Total
Overall length	385 ± 5 mm
Stem diameter	6 to 7 mm
Bulb shape	Cylindrical
Bulb length	10 to 15 mm
Bulb diameter	5 to 6 mm and not greater than stem
Distance from bottom of bulb	
– to 0 °C line	100 to 110 mm
– to 300 °C	333 to 354 mm
Longer lines at each	5 °C
Figured at each	10 °C
Expansion chamber	Required
Top finish	Ring or plain
Scale error not to exceed	± 0,5 °C up to 300 °C
Stability of zero	See Note

NOTE – The thermometer shall be artificially aged by means of a suitable treatment before graduation, in order to secure stability of zero. This treatment shall be 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 zero. If rapid cooling is used, the zero 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 zero under the same conditions as before.

5.2.2.3 Draught screen

5.2.2.3.1 For use with a gas burner

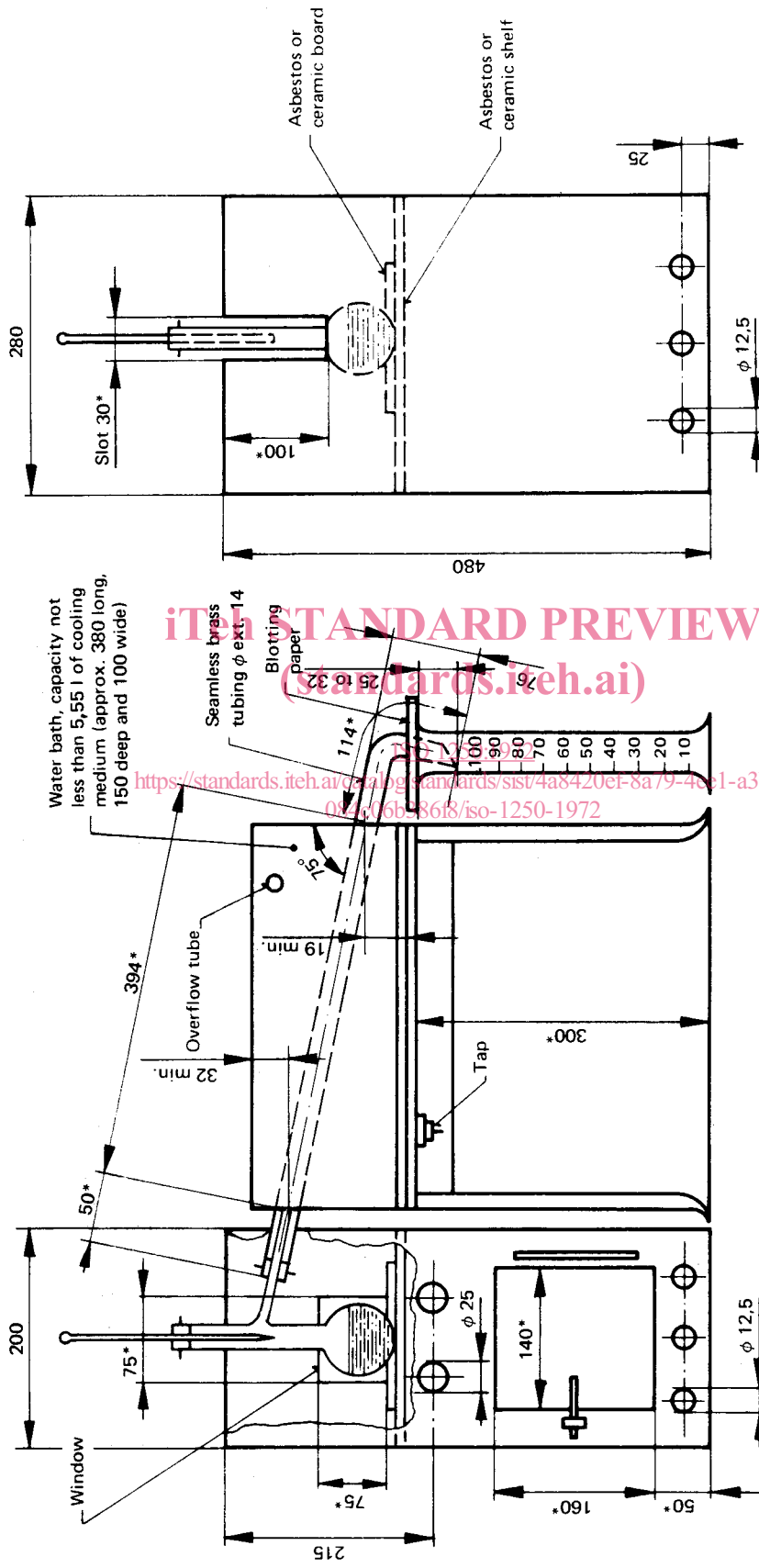
The draught screen shall be rectangular in cross-section and open at the top and bottom. It shall have the dimensions shown in Figure 1 and be made of sheet metal approximately 0,8 mm thick.

In each of the two narrower sides of the draught screen there shall be two circular holes 25 mm in diameter situated 215 mm below the top of the shield, as shown in Figure 1.

In each of the four sides of the draught screen there shall be three holes with their centres 25 mm above the base of the draught screen. These holes shall occupy the positions shown in Figure 1, the diameter of each of the holes being 12,5 mm.

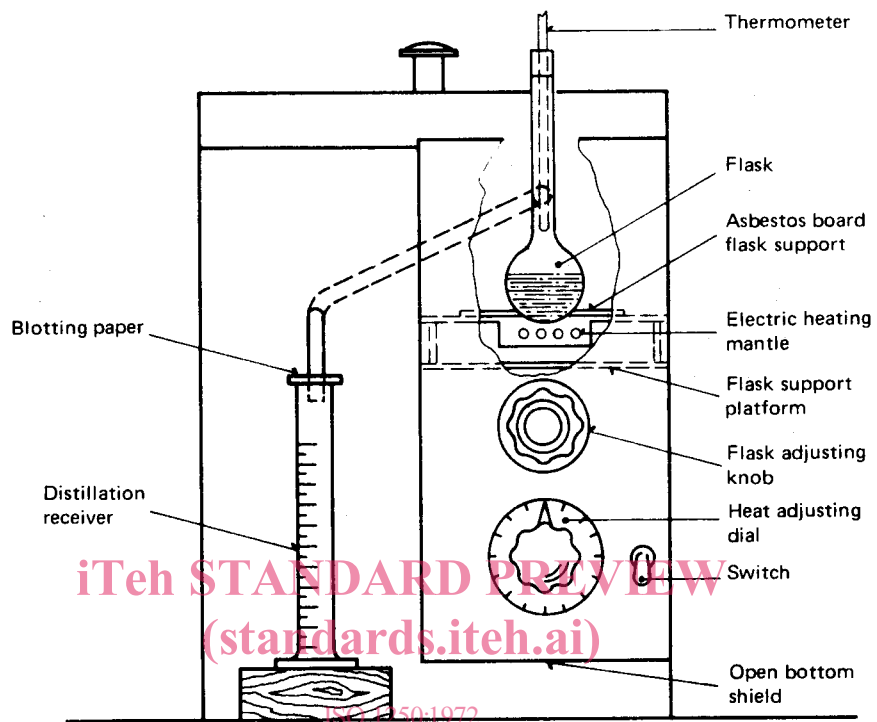
1) The requirements given are taken from the specification for thermometer ASTM 7C; thermometer IP 5C, although differing slightly in its specification, is also suitable for this test.

Dimensions in millimetres



Dimensions marked * are approximate and are given for guidance.

FIGURE 1 — Distillation apparatus using gas burner



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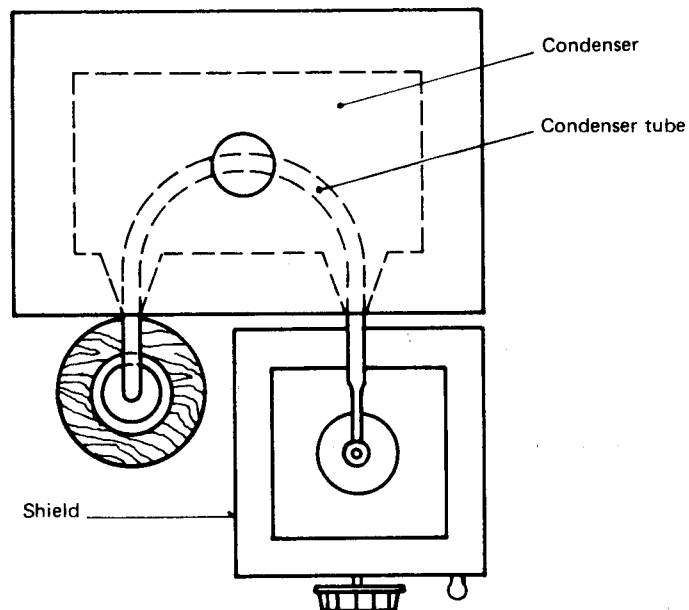
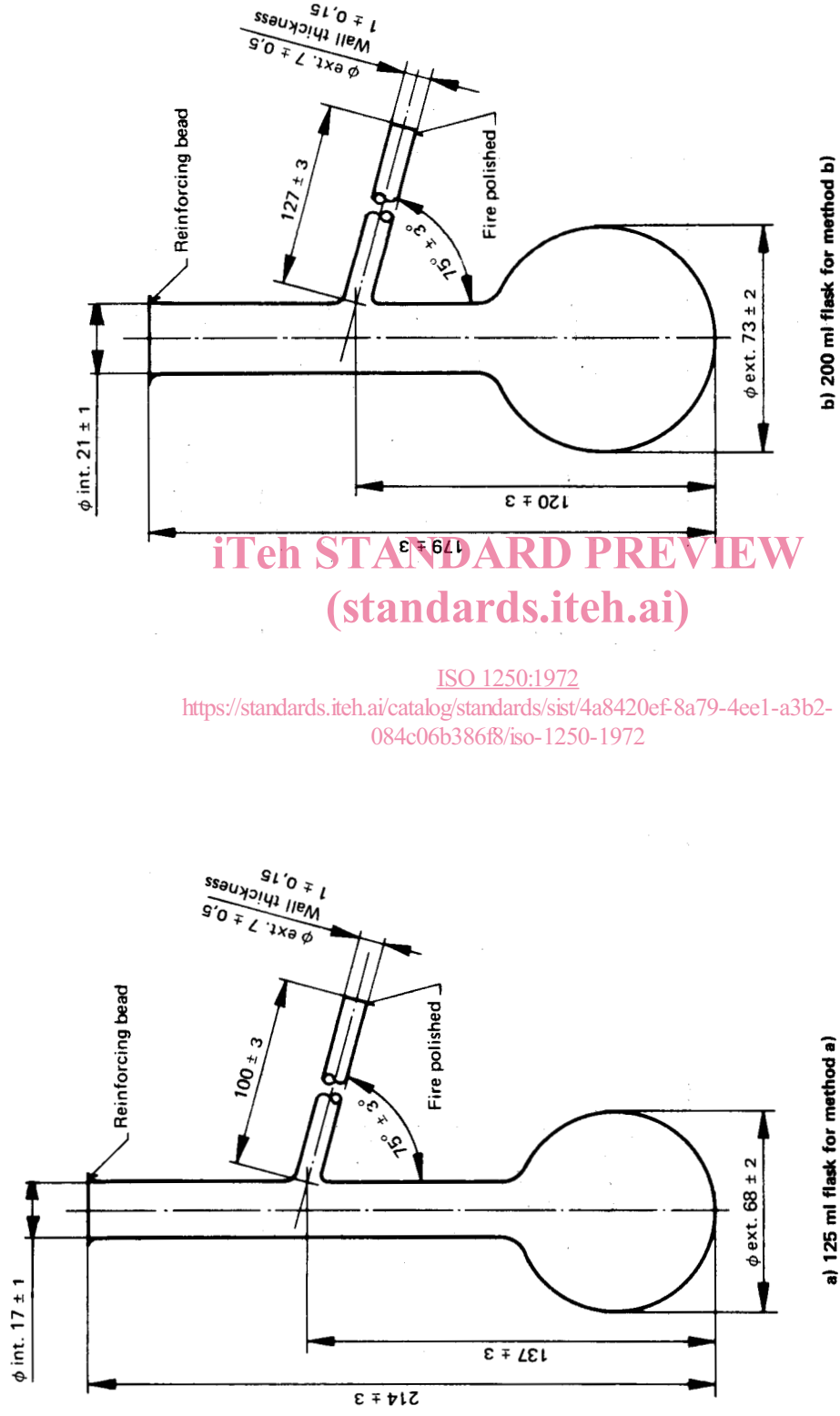


FIGURE 2 – Distillation apparatus using electric heater

Dimensions in millimetres



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FIGURE 3 — Distillation flasks

At the middle of each of the wider sides a vertical slot for the condenser tube, dimensioned approximately as shown in Figure 1 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 to be placed on either side of the draught screen.

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

In one of the narrower sides of the screen a door shall be provided having the approximate dimensions shown in Figure 1 and overlapping the opening in the screen by approximately 5 mm all round.

In each or both of the narrower sides of the screen a mica or heat-resisting glass window may be placed centrally, with the bottom of the window level with the top of the shelf. The approximate dimensions and positions of the windows are shown in Figure 1.

5.2.2.3.2 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 5.2.2.3.1, but the lower portion (including the shelf) may be modified or omitted provided the change does not expose the distillation flask to draughts (see Figure 2).

5.2.2.4 Hard asbestos or ceramic board, 3 to 6 mm thick, with a central hole of 50 mm diameter and overall dimensions not less than 150 mm square. When a gas heater is employed, this board shall rest on the shelf described in 5.2.2.3.1. 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.

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

5.2.2.5 Source of heat: either a gas burner so constructed that sufficient heat can be obtained to distill the product at the uniform rate specified in 5.2.5 (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.)

5.2.2.6 Condenser, of seamless brass tube, 560 mm long, of 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 approximately 390 mm of it are in contact with the cooling medium in the cooling bath with about 50 mm outside the cooling bath at

the upper end, and about 155 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° 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°), 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 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 5,55 l 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 1.

5.2.2.7 Receiver, of 100 ml capacity, 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° to the horizontal.

5.2.3 Assembly of apparatus

5.2.3.1 Assemble the apparatus as shown in Figure 1, swabbing out the condenser with a piece of lint-free cloth attached to a wire cord or by any suitable means, and paying attention to the following details.

5.2.3.2 Position of thermometer

Hold the thermometer concentrically in the neck of the flask by means of a well-fitting stopper of a material which is not attacked by the liquid during the test, the bottom end of the uniform capillary tube of the thermometer being maintained level with the lowest point of the bore of the vapour tube at the joint between the vapour tube and the neck of the flask. The thermometer shall be at a temperature of 13 to 18 °C when it is inserted in the flask at the start of the distillation test.

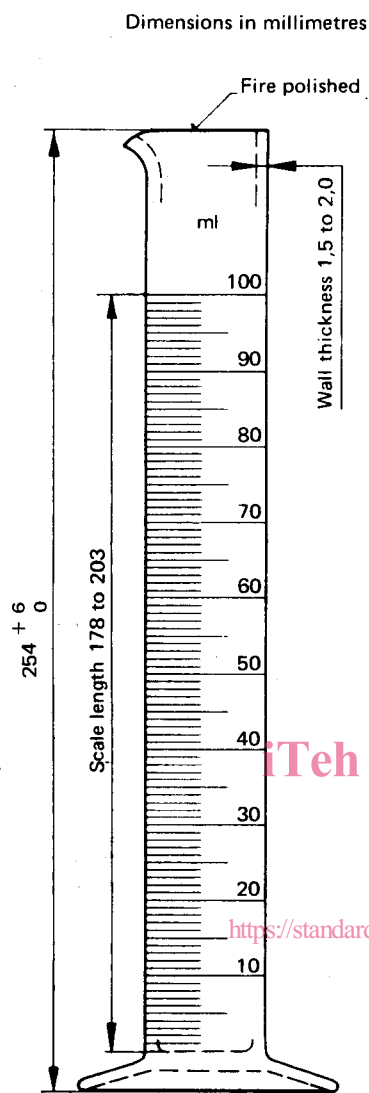


FIGURE 4 – Distillation receiver

5.2.3.3 Support for flask

If a draught screen with asbestos or ceramic shelf is used, place the asbestos or ceramic board (see 5.2.2.4) on top of the shelf so that the two holes are approximately concentric. Place the flask in such a position on the board that the base closes completely the hole in the board.

5.2.3.4 Connection of flask to condenser

Make a leak proof connection to the condenser tube by means of a cork through which the vapour tube passes, and connect the flask to the condenser so that it is in a vertical position and the end of the vapour tube projects at least 25 mm, and not more than 50 mm, beyond the cork into the condenser and is co-axial with it.

5.2.3.5 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 and during distillation is between 0 and 4 °C.

5.2.4 Corrections to be applied to the specified distillation temperatures before commencing the distillation

5.2.4.1 Correction for barometric pressure

When the barometric pressure is other than 1 013 mbar (760 mmHg), apply the corrections given in Table 2 to the specified distillation temperatures. It should be noted that these corrections are only valid for pressures above 933 mbar (700 mmHg).

TABLE 2 – Temperature corrections

Temperature range °C	Correction ¹⁾ per 13 mbar (10 mmHg) difference in pressure °C
110 to 130	0,47
130 to 150	0,50
150 to 170	0,52
170 to 190	0,54
190 to 210	0,57
210 to 230	0,59

1) To be added to the specified distillation temperature when the barometric pressure is above 1 013 mbar (760 mmHg), and to be subtracted when the barometric pressure is below 1 013 mbar (760 mmHg).

5.2.4.2 Correction for thermometer error

If the thermometer gives incorrect readings at the specified distillation temperatures, adjusted in accordance with 5.2.4.1, make a further correction to these temperatures, corresponding to the actual error of the thermometer.

5.2.5 Procedure

5.2.5.1 Measure 100 ml of the sample in the clean and dry receiver, both being at a temperature of 13 to 18 °C, and transfer the sample as completely as possible to the distillation flask, taking care that none of the liquid flows into the vapour tube. Re-insert the thermometer located as described in 5.2.3.1 and place the receiver, without drying it, at the outlet of the condenser tube in such a position that the condenser tube extends centrally into the receiver for at least 25 mm, but not below the 100 ml graduation. If the room temperature is not between 13 and 18 °C, immerse the receiver up to the 100 ml graduation in a transparent bath maintained between these temperatures. Cover the top of the receiver with a piece of blotting paper or its equivalent, cut to fit the condenser tube tightly, to prevent condensed moisture from entering the receiver. Maintain the level of the bath around the receiver so that it is up to the 100 ml mark.

5.2.5.2 Regulate the application of heat so that the first drop of condensate falls from the condenser in not less than 5 and not more than 10 min. After the first drop falls, move the receiver so that the tip of the condenser tube touches its side. Further regulate the heat so that the distillation proceeds at a uniform rate of 4 to 5 ml per minute (approximately 2 drops per second).

Record the volume of distillate collected to the nearest 0,5 ml when the thermometer reading reaches each of the corrected temperature points corresponding to the specified distillation temperatures of 130 °C, 145 °C and 200 °C.

When the volume of residual liquid in the flask is approximately 5 ml, make a final adjustment of the heat if necessary, so that the time from this moment to the end point (final boiling point) does not exceed 5 min. If this condition is not satisfied, repeat the test with appropriate modification of the final heat adjustment. Record the end point and whether the bottom of the flask is dry.

5.2.6 Expression of results

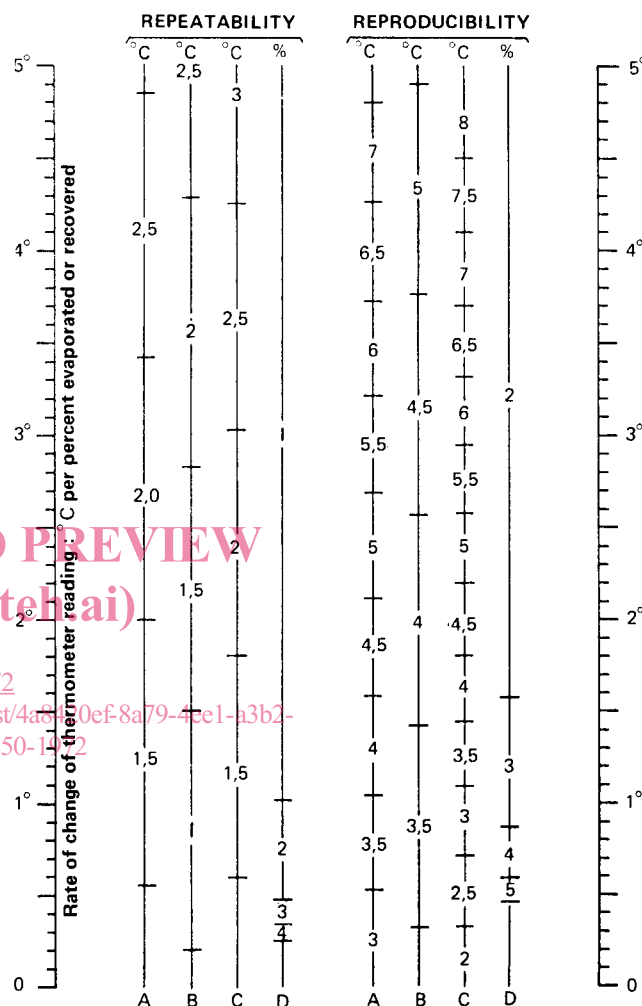
Report the volumes of distillate recorded at the indicated temperatures corresponding (see 5.2.4) to the specified temperatures of 130 °C, 145 °C and 200 °C. Report the final boiling point suitably corrected by applying the barometric correction and the thermometer correction. Report whether these figures comply with or do not comply with the requirement of 5.2.4. Record the barometric pressure during the test and state that the necessary corrections have been applied.

5.2.7 Precision

The following criteria shall be used for judging the acceptability of results (95 % probability):

- a) Duplicate results obtained by the same operator and apparatus shall not be considered suspect unless they differ by more than the repeatability which, according to Figure 5, is appropriate to the test result obtained and to the rate of change in thermometer reading which prevailed at the stage at which the result was obtained.
- b) The results obtained by each of two laboratories shall not be considered suspect unless the two results differ by more than the reproducibility which, according to Figure 5, is appropriate to the test result obtained and to the rate of change in thermometer reading which prevailed at the stage at which the result was obtained.
- c) To facilitate the use of Figure 5, the rate of change in thermometer reading in degrees Celsius per the percentage evaporated or recovered, at any point between the initial boiling point and the end point (final boiling point) or dry point, shall be assumed to be the same as the average rate between two data points which are equidistant above and below the point in question. The span from the point in question to either of the other data points shall not represent more than 10 % evaporated or recovered in any case, nor more than 5 % if the point in question is not included in the 10 to 90 % range. For the initial boiling point, end point (final boiling point), or dry point, the rate of change shall be assumed to be the same as the average rate over an interval, not to exceed 5 % evaporated or recovered, between the extreme point and the next data point above or below it.
- d) In Figure 5, it will be noted that the left and right marginal scales, representing the rate of change in thermometer reading, are identical. This is to facilitate

the establishing of a horizontal line across the chart and at the required level, which may be done in any convenient manner. Wherever this line intersects the appropriate precision scale, the zone in which such intersection falls will indicate the expected repeatability or reproducibility.



A – Initial boiling point, °C
 B – End point (final boiling point) or dry point, °C.
 C – Thermometer reading at prescribed per cent evaporated or recovered, °C.
 D – Per cent evaporated or recovered at prescribed thermometer readings.

FIGURE 5 – Precision of distillation

5.3 Distillation test – Method b)

5.3.1 Definitions

5.3.1.1 volume recovered: The volume, expressed in millilitres, of condensate collected in the receiver at the specified temperature readings on the thermometer.

5.3.1.2 dry point: The temperature indicated at the instant the last drop of liquid evaporates from the lowest point in the flask, any drops or film of liquid on the side of the flask or on the thermometer being disregarded.