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Mineral solvents for paints — White spirits and related hydrocarbon solvents

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FOREWORD

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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.*

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

France

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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	these limits apply to mineral solvents for paints with or without denaturant	
Method a)*	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

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* 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 requirements[1] :

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.

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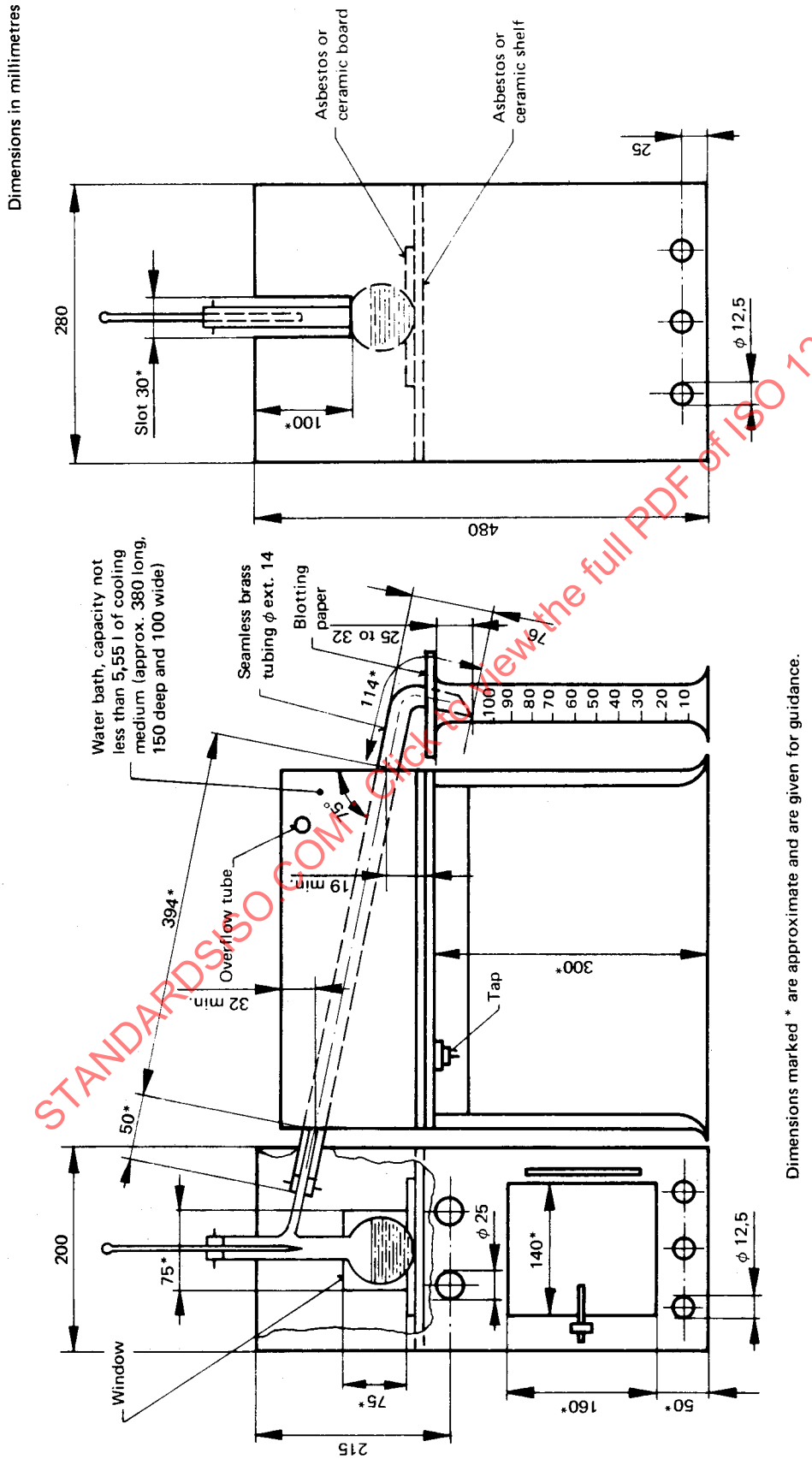

Dimensions marked * are approximate and are given for guidance.

FIGURE 1 – **Distillation apparatus using gas burner**

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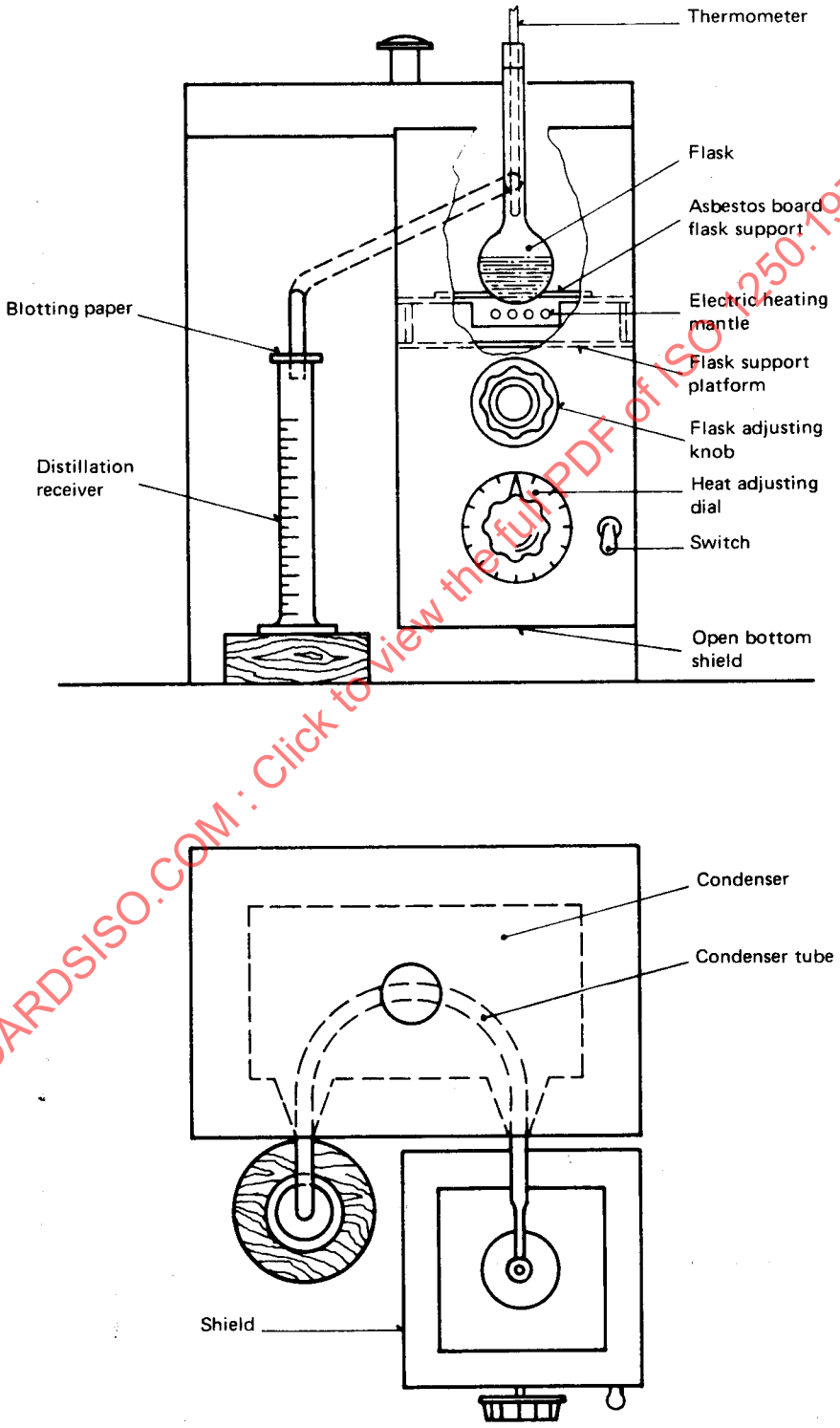

FIGURE 2 – **Distillation apparatus using electric heater**

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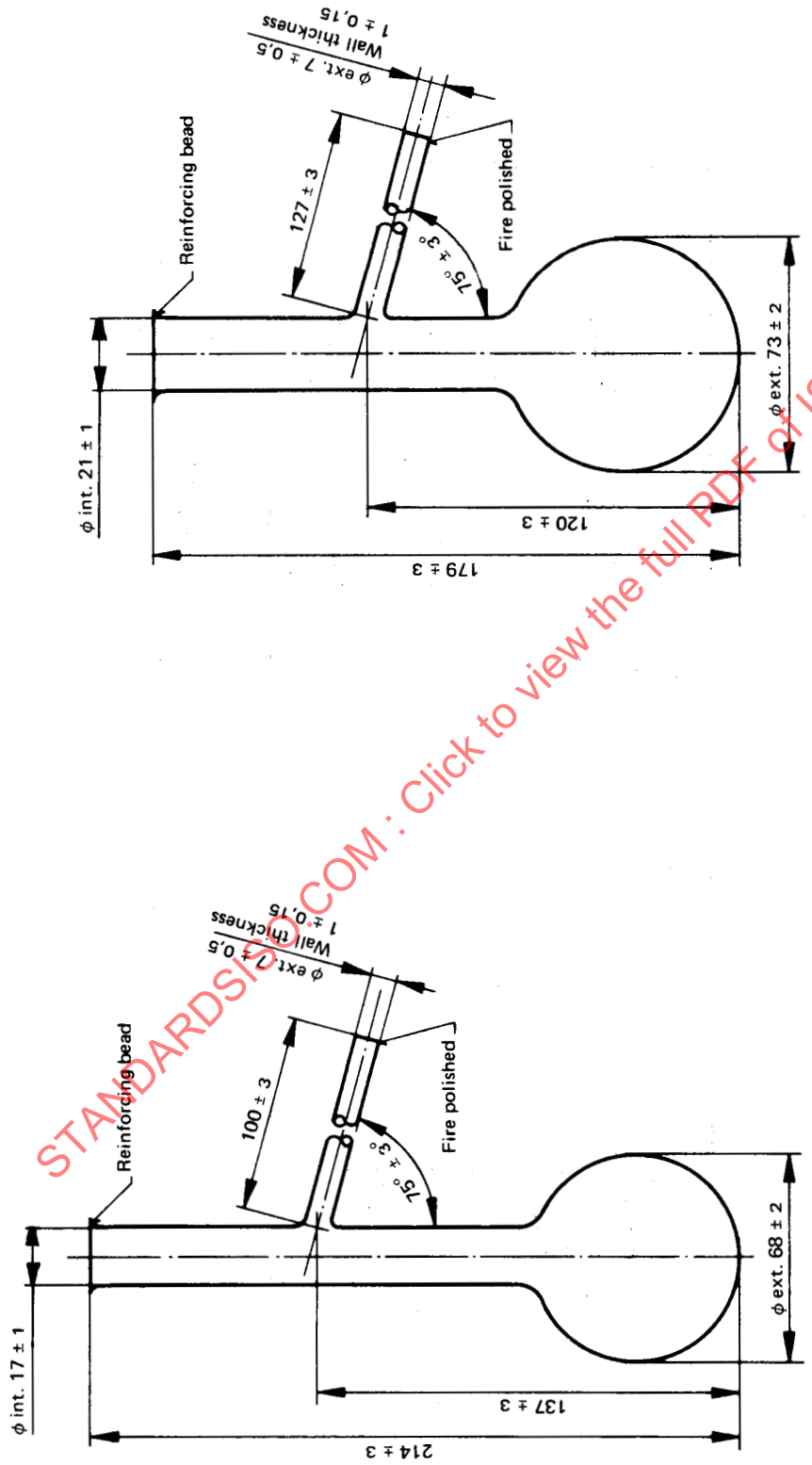
Dimensions in millimetres



b) 200 ml flask for method b)

a) 125 ml flask for method a)

FIGURE 3 – Distillation flasks

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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.

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Dimensions in millimetres

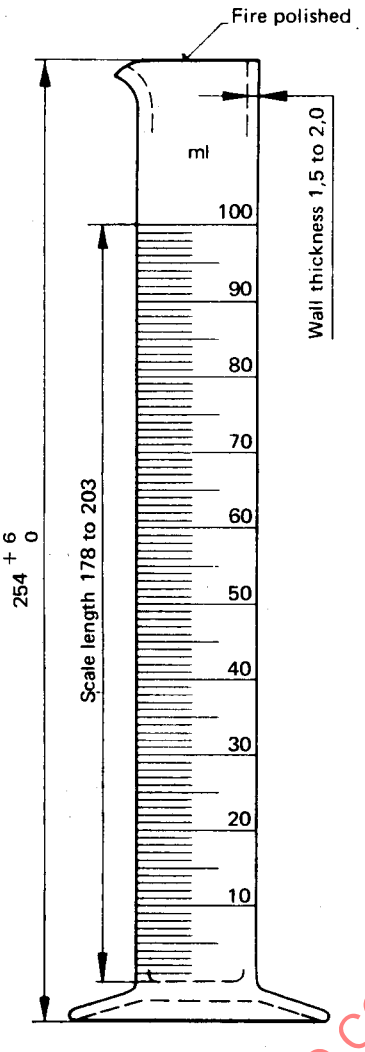

FIGURE 4 — **Distillation receiver**

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).

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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*

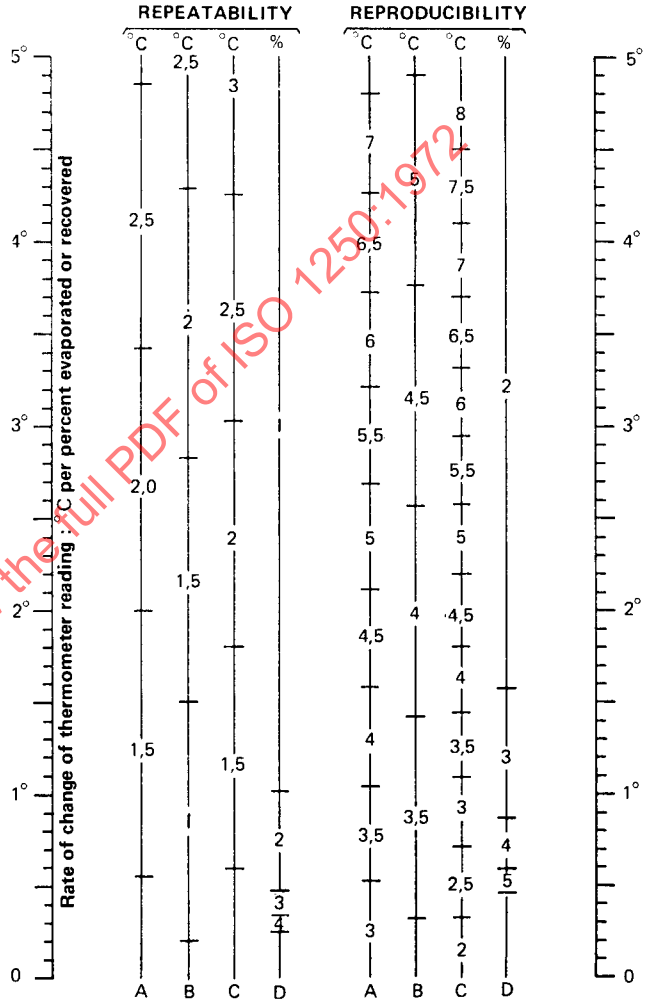
The following criteria shall be used for judging the acceptability of results (95 % probability) :

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.

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5.3.2 *Apparatus*

The apparatus shall comprise :

5.3.2.1 Distillation flask, in heat-resistant glass, of 200 ml distillation capacity, conforming to the dimensions shown in Figure 3 b).

5.3.2.2 Thermometer, mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, and conforming to the following requirements[1] :

Range	95 to 255 °C
Graduation	0,5 °C
Immersion	100 mm
Overall length	390 to 400 mm
Stem diameter	6 to 7 mm
Bulb shape	Cylindrical
Bulb length	15 to 20 mm
Bulb diameter	Not greater than stem
Distance from bottom of bulb	
– to 95 °C	125 to 145 mm
– to 255 °C line	355 to 360 mm
– to top of contraction chamber	35 mm max.
Longer lines at each	1 °C
Figured at each	5 °C
Expansion chamber	To allow heating to 280 °C
Top finish	Ring or plain
Scale error not to exceed	± 0,5 °C

NOTES

1 The length of the unchanged capillary between the contraction chamber and the graduation line nearest to it shall be not less than 10 mm.

2 During manufacture and testing of this thermometer the mean temperature of the emergent mercury column shall be as follows :

Temperature	Mean temperature of emergent column
100 °C	30 °C
150 °C	35 °C
200 °C	40 °C
250 °C	45 °C

5.3.2.3 Draught screen, as described in 5.2.2.3.

5.3.2.4 Hard asbestos or ceramic board, as described in 5.2.3.4 but with a central hole 32 mm in diameter.

5.3.2.5 Source of heat, as described in 5.2.2.5.

5.3.2.6 Condenser, as described in 5.2.2.6.

5.3.2.7 Receiver, as described in 5.2.2.7.

5.3.3 *Assembly of apparatus*

Assemble the cleaned apparatus as described in 5.2.3.1, with the following exceptions :

5.3.3.1 Fill the cooling bath with water at a temperature of 25 to 30 °C and maintain it at this temperature during the test.

5.3.3.2 In relation to 5.2.3.2, the "bottom end of the uniform capillary tube of the thermometer" refers to the main capillary tube above the contraction chamber.

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

5.3.3.3 Use the flask support board according to 5.3.2.4.

5.3.4 *Correction to be applied to the specified distillation temperatures before commencing the distillation*

Apply corrections as described in 5.2.4.

5.3.5 *Procedure*

5.3.5.1 Measure 100 ml of the sample in the receiver, both being at a temperature of 20 to 30 °C, and pour the sample directly into the distillation flask, taking care not to allow any of it to enter the vapour tube, and allowing the receiver to drain for 15 to 20 s. Re-insert the thermometer 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. Close 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.

5.3.5.2 Regulate the application of heat so that the ring of condensing vapour on the wall of the flask takes 10 to 15 min to rise from the lower end of the neck of the flask to the side arm (this is to ensure that the mercury in the thermometer is at the same temperature as that of the refluxing liquid), and the first drop of condensate falls from the condenser within 15 min of the start of heating.

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 reaches each of the corrected temperature points corresponding to the specified distillation temperatures of 132 °C, 148 °C and 204 °C.

Do not make any further adjustements of the heat until the dry point is observed and record this temperature. If a dry point is not obtainable (i.e. if active decomposition occurs before the dry point is reached, as evidenced by a rapid

1) The requirements given are taken from the specification for thermometer ASTM 42C; thermometer IP 82C, although differing slightly in its specification, is also suitable for this test.

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evolution of vapour or heavy fumes; or if there is liquid remaining on the bottom of the flask when the maximum temperature is observed on the distillation thermometer) note this fact.

5.3.6 *Expression of results*

Report the volumes of distillate recorded at the indicated temperatures corresponding (see 5.2.4) to the specified temperatures of 132 °C, 148 °C and 204 °C. Report the dry point suitably corrected by applying the barometric correction and the thermometer correction. Report whether these figures comply with or do not comply with the requirements of 5.2.4. Record the barometric pressure during the test and state that the necessary corrections have been applied.

If the dry point is not obtainable, record this fact and report the maximum temperature observed on the distillation thermometer, after applying the appropriate corrections.

5.3.7 *Precision*

No statistically established data are at present available for Method b) for materials of wide boiling range, but it may be assumed that for white spirits the precision limits applicable to Method b) are of the same order as those for Method a).

5.4 Method for the determination of aromatic content

5.4.1 *Principle*

A small quantity of the sample is introduced into a special glass adsorption column packed with fine activated silica gel, a small layer of which contains a mixture of fluorescent dyes. When all the sample has been adsorbed on the gel, alcohol is added to desorb the sample and force it down the column. The hydrocarbons are separated according to their adsorption affinities into aromatics, olefins and saturated hydrocarbons. The fluorescent dyes are also separated selectively, with the hydrocarbon types, and make the boundaries of the aromatics, olefin and saturate zones visible under ultraviolet light. The volume percentage of each hydrocarbon type is calculated from the length of each zone in the column. Some diolefins and aromatics with olefinic side chains, plus any sulphur, nitrogen, and oxygen compounds are determined as aromatics.

5.4.2 *Reagents and materials*

5.4.2.1 Silica gel, of 150 to 75 μm particle size, manufactured to ensure minimum olefin polymerization activity and to conform to the sieve test requirements shown in Table 3. It is necessary to screen the gel to make certain that the gel size specification is met. Before use, dry the gel in a shallow vessel at 175 ± 5 °C for 3 h. Transfer the dried gel to an airtight container while still hot, and protect it thereafter from atmospheric moisture.

TABLE 3 – **Sieve acceptance tests of silica gel**

Mesh aperture of sieve μm	**Percentage (*m/m*) of particles passing through sieve**
180	100
150	95 min.
75	15 max.

NOTE – Suitable silica gel is obtainable from W.R. Grace and Co., Davison Chemical Division, Baltimore, Md. 21203 or from their appointed agents, by specifying Code 923.

5.4.2.2 Fluorescent indicator dyed gel : standard dyed gel, consisting of a mixture of recrystallized Petrol Red AB4 and purified portions of the olefin and aromatic dyes obtained by chromatographic adsorption following a definite, uniform procedure, and deposited on silica gel.

It is essential that the dyed gel be stored in a dark place under an atmosphere of nitrogen. Even under these conditions the fluorescent dyes can deteriorate resulting in indistinct zone boundaries. Batches stored for longer than six months should be considered suspect.

NOTE – Suitable dyed gel is available from Patent Chemicals, Inc., 335 McLean Blvd , Patterson, N.J., or their appointed agents by requesting "Standard Dyed Gel for the FIA Method of Hydrocarbon Type Analysis".

5.4.2.3 ***Iso*propyl alcohol** (propan-2-ol), refined, 99 % grade.

5.4.2.4 Pressuring gas, air (or nitrogen) delivered to the top of the column at pressures controllable over the range 0 to 103,4 kN/m² (0 to 1,05 kgf/cm² gauge).

5.4.3 *Apparatus*

5.4.3.1 Adsorption columns, made of glass with standard wall tubing as shown in Figure 6 a), or with precision bore tubing as shown in Figure 6 b), and consisting of a charger section with a capillary neck, a separator section, and an analyser section.

5.4.3.1.1 For normal laboratory procedure use standard wall tubing for the analyser section. It is necessary to select tubing of uniform bore and to provide a leakproof connection between the separator and the analyser sections. Draw out one end of the tubing selected for the analyser section to a fine capillary to retain the gel. Connect the other end of the analyser section to the separator section with a 30 mm length of PVC tubing, making certain that the two glass sections touch. To ensure a leakproof glass-to-PVC seal with the analyser section, it is necessary to heat the upper end of the analyser section until it is just hot

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enough to melt the PVC, then insert the upper end of the analyser section into the PVC sleeve. Alternatively, this seal can be made by securing the PVC sleeve to the analyser section by wrapping it tightly with soft wire.

NOTE – Calibrations of standard wall tubing would be impractical; however, any variations of 0,5 mm or greater, as measured by ordinary calipers, in the outside diameter along the tube may be taken as an indication of irregularities in the inner diameter and such tubing shall not be used.

5.4.3.1.2 For referee purposes use precision bore tubing. The inner diameter of the analyser section shall be 1,60 to 1,65 mm and an approximately 100 mm thread of mercury shall not vary in length by more than 0,3 mm when located in any part of the analyser section. In glass-sealing the various sections to each other, long-taper connections shall be made instead of shouldered connections. Support the silica gel by means of a 6 mm diameter portion of phosphor-bronze or equivalent 75 μm aperture wire gauze located between the ball and socket of the S 13 spherical joint and covering the analyser outlet. The column tip attached to the S 13 socket shall have a 2 mm internal diameter. Clamp the ball and socket together and ensure that the tip does not tend to slide from a position in a direct line with the analyser section during the packing and subsequent use of the column.

5.4.3.2 Zone-measuring device. The zones may be marked with a glass-writing pencil and the distances measured with a metre rule, with the analyser section lying horizontally. Alternatively, the metre rule may be fastened adjacent to the column. In this case, it is convenient to have each rule fitted with four movable metal index clips (see Figure 6) for marking zone boundaries and measuring the length of each zone.

5.4.3.3 Ultraviolet light source, with radiation predominantly at 365 nm. A convenient arrangement consists of one or two units about 900 to 1 200 mm long mounted vertically along the apparatus, and adjusted to give the best fluorescence.

5.4.3.4 Vibrator, for packing the silica gel by vibrating individual columns or the frame supporting multiple columns. A small electric vibrator is recommended, but any other means may be used to obtain a uniform packing.

5.4.3.5 Hypodermic syringe, capacity 1 ml, graduated in 0,01 or 0,02 ml, with a needle 100 mm in length. Needles of external diameter 0,7 to 1,2 mm are convenient to use.

5.4.4 *Preparation of apparatus*

Mount the apparatus assembly in a darkened room or area to facilitate observation of zone boundaries. For multiple determinations, assemble an apparatus which includes the ultraviolet light source, a rack to hold the columns, and a gas manifold system with spherical joints to connect to the desired number of columns.

5.4.5 *Procedure*

5.4.5.1 Prepare two standard wall columns for simultaneous, duplicate analysis of each sample, or a single column if precision bore tubing is to be used. Freely suspend each column from a loose-fitting clamp placed immediately below the spherical joint of the charger section. While vibrating the column along its entire length, add small increments of silica gel through a glass funnel into the charger section until the separator section is half full. Stop the vibrator and add a 3 to 5 mm layer of dyed gel. Start the vibrator and vibrate the column while adding additional silica gel. Continue to add silica gel until the tightly packed gel extends 75 mm into the charger section. Wipe the length of the column with a damp cloth while vibrating the column. This aids in packing the column by removing static electricity. Vibrate the column after filling is completed. Vibration for about 4 min is usually satisfactory when the vibrator is used.

NOTE – More than one pair of columns can be prepared simultaneously by mounting them on a frame or rack to which an electric vibrator is attached.

5.4.5.2 Attach the filled columns to the apparatus assembly in the darkened room or area, and when a permanently mounted metre rule is used, fasten the lower end of the column to the fixed rule with a rubber band.

5.4.5.3 Chill the sample and the hypodermic syringe to between 2 and 4 °C. Draw 0,75 ± 0,03 ml of sample into the syringe and inject the sample 30 mm below the surface of the gel in the charger section.

5.4.5.4 Fill the charger section to the spherical joint with *iso*propyl alcohol. Connect the column to the gas manifold and apply 13,8 kN/m² (0,14 kgf/cm²) gas pressure for 2,5 min, to move the liquid front down the column. Increase the pressure to 34,5 kN/m² (0,35 kgf/cm²) for another 2,5 min and then adjust the pressure required to give a transit time of about 1 h. Usually 68,9 to 103,4 kN/m² (0,7 to 1,05 kgf/cm²) gas pressure is needed. The pressure required will depend on the tightness of packing of the gel and the molecular mass of the sample. A transit time of 1 h is generally satisfactory; however, high-molecular mass samples may require longer transit times.

5.4.5.5 Before commencing to read the zone boundaries, carefully wipe the outside of the column with a cloth dampened with ethanol and avoid handling the column with bare hands thereafter.

5.4.5.6 After the red, alcohol-aromatic boundary has advanced 350 mm into the analyser section, make a set of readings by quickly marking the boundary of each hydrocarbon-type zone observed in ultraviolet light (see Note 1) in the following sequence (**CAUTION** – Avoid touching the column with the hands during this operation.)

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Dimensions in millimetres

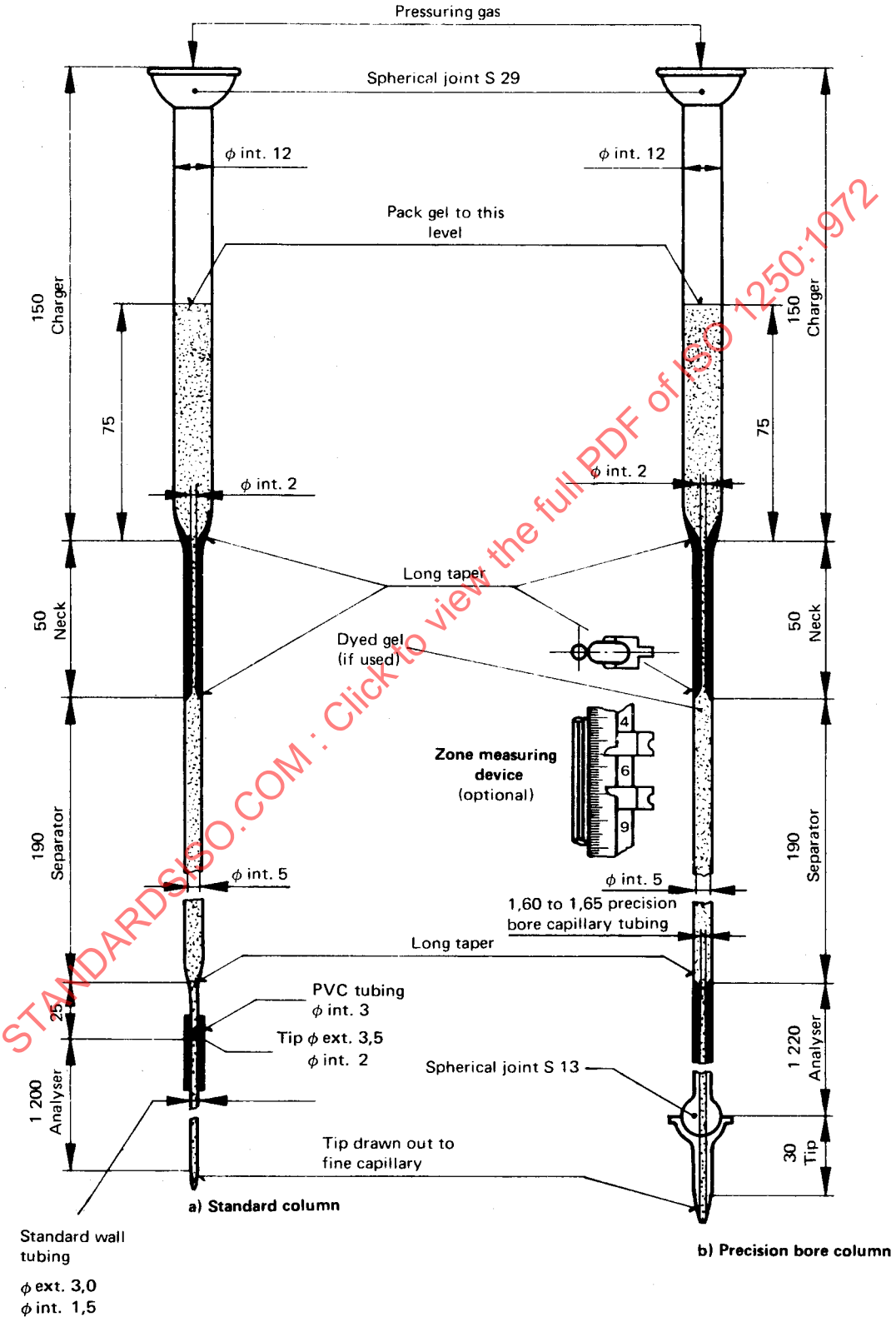

FIGURE 6 – **Alternative adsorption columns with standard wall and precision bore tubing in analyser**

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– for the non-fluorescent saturate zone, mark the front of the charge and the point where the yellow fluorescence first reaches its maximum intensity;

– for the upper end of the second, or olefin zone, mark the point where the first intense blue fluorescence occurs;

– finally, for the upper end of the third or aromatic zone, mark the upper end of a reddish or brown zone (see Note 2).

With colourless distillates, the alcohol-aromatic boundary is clearly defined by a red ring of dye. However, certain impurities may obscure this red ring and give a brown coloration which varies in length, but which shall be counted as a part of the aromatic zone, except that when no blue fluorescence is present, the brown or reddish ring shall be considered as part of the next distinguishable zone below it in the column. If the boundaries have been marked off with index clips, record the measurements.

NOTES

1 **CAUTION** – Direct exposure to ultraviolet light can be harmful, and operators should avoid this as far as possible, particularly with regard to their eyes.

2 When zone boundaries are indistinct, the use of a Wratten No. 47 or No. 50 light filter between the column and the eyes may aid in recognizing the aromatic zone front.

5.4.5.7 When the sample has advanced another 50 mm down the column, make a second set of readings by marking the zones in the reverse order as described in 5.4.5.6 so as to minimize errors due to the advancement of boundary positions during readings. If the marking has been made with a glass-writing pencil, two colours can be used to mark off each set of measurements, and the distances measured at the end of the test with the analyser sections lying horizontally on the bench top. If the boundaries have been marked off with index clips, record the measurements.

NOTE – Erroneous results can be caused by improper packing of the gel or incomplete elution of hydrocarbons by the alcohol. With precision bore columns, incomplete elution can be detected from the total length of the several zones, which must be at least 500 mm for a satisfactory analysis. With standard wall tubing, this criterion of total sample length is not strictly applicable because the inside diameter of the analyser is not the same in all columns. For samples containing substantial amounts of material boiling above 204 °C, the use of *iso*amyl alcohol instead of *iso*propyl alcohol may improve elution.

5.4.5.8 Release the gas pressure and disconnect the column. Discard the analyser section after all measurements have been completed if the standard wall column is used. To remove used gel from the precision bore column, invert it above a sink and insert through the wide end a long piece of about 1 mm diameter hypodermic tubing with a 45° angle tip. By means of 6 mm tubing at the opposite end for attaching a rubber tube, connect to a water tap and flush with a rapid stream of water. Rinse with residue-free acetone (reagent grade preferred) and dry by evacuation.

5.4.6 *Calculation*

5.4.6.1 For each set of observations (four for the two standard wall columns and two for the precision bore column), calculate to the nearest 0,1 % the volume per cent of the hydrocarbon types as follows :

$$\text{Aromatics, \% } (V/V) = \frac{L_a}{L} \times 100$$

$$\text{Olefins, \% } (V/V) = \frac{L_o}{L} \times 100$$

$$\text{Saturates, \% } (V/V) = \frac{L_s}{L} \times 100$$

where

L_a is the length, in millimetres, of the aromatic zone;

L_o is the length, in millimetres, of the olefin zone;

L_s is the length, in millimetres, of the saturate zone;

L is the sum of $L_a + L_o + L_s$.

5.4.6.2 If the standard wall column is used, calculate the average of the four sets of observations obtained from two successive sets of readings on each pair of columns.

5.4.6.3 If the precision bore column is used, calculate the average from two successive readings obtained on a single column.

5.4.6.4 Round off to the nearest 0,1 % the values obtained by using the precision bore column, and to the nearest 1 % those obtained by using the standard wall column, in both cases adjusting if necessary the amount of the largest component in order that the sum of the components is 100 %.

5.4.7 *Expression of results*

Report, as volume per cent of aromatic content in the sample as analysed, the rounded value obtained as detailed in 5.4.6.4, and state whether the standard wall column or the precision bore column was used.

5.4.8 *Precision*

5.4.8.1 Repeatability

Duplicate results by the same operator shall not be considered suspect, unless they differ by more than the following amounts :

Level of result %	Aromatics	Olefins	Saturates
50	1,2	1,9	1,4
40 or 60	1,2	1,8	1,4
30 or 70	1,1	1,7	1,3
20 or 80	1,0	1,5	1,1
10 or 90	0,7	1,1	0,8
5 or 95	0,5	0,8	0,6
1 or 99	0,2	0,3	0,2

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NOTE – These repeatability figures refer to results obtained simultaneously, and not successively as in the strict definition of the term.

5.4.8.2 Reproducibility

The results submitted by each of two laboratories shall not be considered suspect unless the two results differ by more than the following amounts :

Level of result %	Aromatics	Olefins	Saturates
50	4,0	7,7	4,2
40 or 60	3,9	7,6	4,4
30 or 70	3,7	7,1	3,8
20 or 80	3,2	6,2	3,3
10 or 90	2,4	4,6	2,5
5 or 95	1,7	3,3	1,8
1 or 99	0,8	1,5	0,8

5.5 Method for the determination of residue on evaporation

5.5.1 *Definition*

residue on evaporation : The amount of non-volatile residue obtained under the conditions of test.

5.5.2 *Apparatus*

5.5.2.1 Evaporation bath, either a solid metal-block bath using a metal of high thermal conductivity, or a liquid bath fitted with a reflux condenser, electrically heated and constructed in accordance with the general principles shown in Figure 7. The electric supply to the liquid bath shall be sufficient to keep the liquid boiling when air is passing through the preheating coil at the specified rate. The evaporation bath shall be provided with wells and air jets for three or more beakers and be insulated. The preheaters, manifolds, and air outlets shall be so constructed as to allow the required rate of air flow (see 5.5.2.2). The beaker wells shall be made sufficiently deep to allow insertion of the 100 ml beakers to a depth of 70 mm. If a liquid bath is used, it shall be filled to within 25 mm of the top with a stable liquid having a boiling point between 160 and 165 °C; ethanediol (ethylene glycol) containing approximately 3 % of water is suitable for this purpose.

5.5.2.2 Air supply apparatus, capable of supplying filtered air at a pressure not greater than 34,5 kN/m² (0,35 kgf/cm²) to the inlet of the preheating coil of the bath, and at a rate sufficient to provide a flow from each outlet of 1 000 ± 150 ml/s at a temperature of 155 ± 5 °C.

5.5.2.3 Flowmeter, capable of metering a flow of air equivalent to 1 000 ml/s for each outlet of the evaporation bath at the operating temperature. The use of a calibrated flowmeter allowing a flow of 600 ± 90 ml/s, measured at room temperature, will ensure delivery of 1 000 ± 150 ml/s at the operating temperature, provided that the pressure at the outlet of the flowmeter is not greater than 34,5 kN/m² (0,35 kgf/cm²).

5.5.2.4 Beakers, of 100 ml capacity, flat bottomed as shown in Figure 7. The beakers shall be arranged in sets, the number in each set depending on the number of beaker wells in the evaporating bath, each beaker in the set being marked permanently with an identifying number or letter, the lowest mass beaker being reserved for use as a tare. The same beaker may be used as a tare for up to six test beakers, provided that all are heated at the same time.

5.5.2.5 Cooling vessel, suitable covered vessel for cooling the beakers before weighing, such as a glass desiccator or a tightly covered metal vessel for each set of beakers. A drying agent is not to be used in the cooling vessel.

5.5.2.6 Balance, having a sensitivity of at least 0,1 mg, preferably with no drying agent in the case.

5.5.2.7 Thermometer, of the mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, adjusted for 76 mm immersion and allowing temperatures in the range 150 to 165 °C to be measured with an accuracy of ± 1 °C at an average temperature of the emergent mercury column of 65 °C.

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.

5.5.3 *Cleaning liquids*

5.5.3.1 A mixture of equal volumes of **toluene** and **acetone**, of analytical reagent quality.

5.5.3.2 Chromic/sulphuric acid solution.

5.5.4 *Preparation of apparatus*

5.5.4.1 Assemble the apparatus as shown in Figure 7. With the apparatus at room temperature, adjust the air flow to a rate of 600 ml/s at one of the outlets. Measure the flow at the remaining outlets and make any necessary adjustments so that the rate at each outlet is 600 ± 90 ml/s (see 5.5.2.3). Heat the bath and when its temperature reaches 160 to 165 °C, place a beaker in each well (5.5.2.1). Submit air at the rate required with the conical jets in position. Check

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the temperature in each well by placing the bulb of the thermometer on the bottom of the beaker in the well. Do not use any well where the temperature does not lie between 150 and 160 °C.

5.5.4.2 Clean new beakers by immersion in the chromic/sulphuric acid solution (5.5.3.2) for at least 6 h. Remove the beakers from the solution by means of stainless steel forceps and handle only with forceps thereafter. Wash the beakers thoroughly, first with tap water, then with distilled water.

NOTE — Protective clothing such as gloves and goggles must be worn by operators using chromic/sulphuric acid solution.

Clean beakers which have been used in previous determinations by removing the residue with the mixture (5.5.3.1), then immerse them for at least 6 h in chromic/sulphuric acid cleaning solution followed by washing as described above. Clean the tare beakers in a similar manner.

Dry the beakers for 1 h in an oven at 150 °C and allow them to cool for at least 2 h in the cooling vessel placed in the vicinity of the balance.

5.5.5 *Procedure*

For each sample, weigh to the nearest 0,1 mg two test beakers for a duplicate determination, using the tare beaker as a tare on the right-hand pan of the balance. Repeat the weighings without changing the order in which the beakers are weighed, until consecutive masses for the beakers agree within 0,1 mg, and record the masses.

If suspended or solid matter is present, mix the contents of the sample container thoroughly. Immediately filter, at atmospheric pressure, the necessary quantity of the sample through a sintered-glass funnel of porosity grade P.100 (pore size index 40 – 100 μm).

Using a graduated measuring cylinder, pour 50 ml of the sample into each test beaker. Place the full beakers and also the empty tare beaker in the evaporation bath, previously heated to the specified temperature (160 to 165 °C). Replace the conical jet as each beaker is filled, centring the jet vertically above the surface of the liquid. The time elapsing between filling the first and last beakers shall be as short as possible. Supply air at the required rate.

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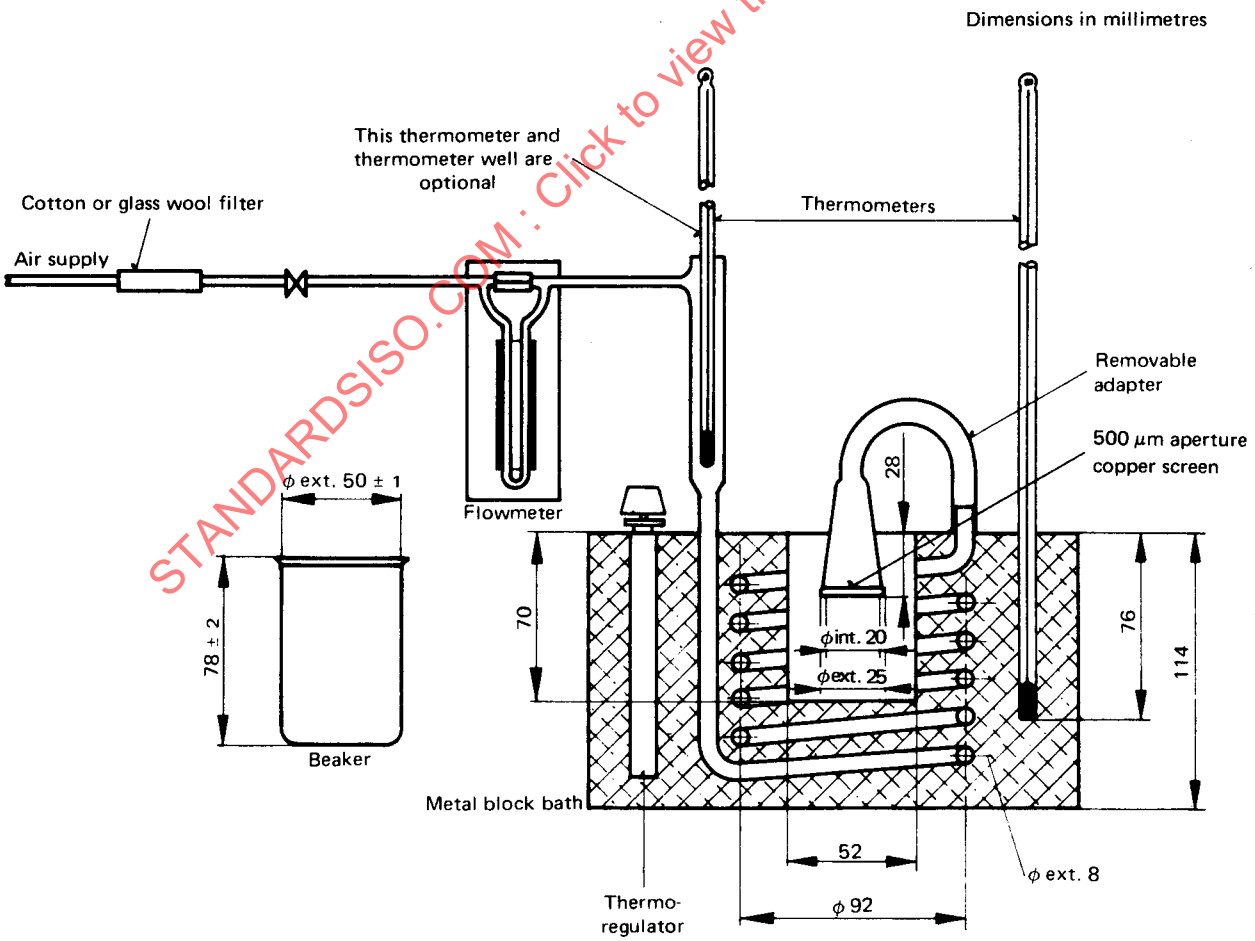

FIGURE 7 — **Apparatus for determining evaporation residue**

After 30 min evaporation, remove the beakers from the bath, place them in the cooling vessel and allow them to cool in the vicinity of the balance for at least 2 h. Weigh the beakers in the same manner and in the same sequence as was followed previously, repeating the weighings until consecutive masses agree within 0,1 mg. If the results of two evaporations differ by more than 1,0 mg, repeat the determinations.

5.5.6 *Expression of results*

5.5.6.1 Calculate the residue on evaporation A, in milligrams per 100 ml, by the formula :

$$A = 2\,000\,(m_1 - m_0)$$

where

m_0 is the mass, in grams, of the empty test beaker;

m_1 is the mass, in grams, of the test beaker plus residue.

5.5.6.2 Report the mean of the sum of the results of the duplicate determinations to the nearest milligram per 100 ml as the residue on evaporation. After the numerical value designate by the word "filtered" if the sample has been so treated.

5.5.7 *Precision*

For results of the order of 10 mg/100 ml (i.e. the maximum permitted residue), duplicate results shall not be considered suspect unless they differ by more than 3 mg/100 ml (repeatability) or 6 mg/100 ml (reproducibility).

5.6 Method for the determination of aniline point

5.6.1 *Definition*

aniline point : The lowest temperature at which equal volumes of aniline and of the product under test are completely miscible under standard conditions.

5.6.2 *Reagents*

5.6.2.1 **Sodium sulphate** or **calcium sulphate**, anhydrous.

5.6.2.2 **Aniline**, redistilled.

Dry a convenient quantity of aniline over solid potassium hydroxide. Filter and distil it, discarding the first and last 10 % fractions. Dry the intermediate fraction for not longer than 24 h over solid potassium hydroxide. The aniline shall be prepared on the day of use.

NOTE – As an alternative to preparing the aniline on the day of use, it may be distilled as described above and the distillate stored under vacuum or in an atmosphere of dry nitrogen in sealed ampoules and kept in a cool dry place for subsequent use. In either case, all precautions shall be taken to prevent contamination by atmospheric moisture. In such conditions, the aniline may be kept for about 6 months.

It is recommended that the aniline should be checked before use by testing with pure *n*-heptane, when it should give an aniline point of 69,3 ± 0,2 °C.

NOTE – **Safety precautions.** Aniline is extremely toxic and must not be sucked into a pipette with the mouth. Aniline is also toxic by absorption through the skin, even in very small quantities, and must be handled with great caution.

5.6.2.3 ***n*-heptane**, pure, as used as a reference fuel in the knock testing of motor fuels and complying with the following requirements :

Density at 20 °C	0,683 8 ± 0,000 15 g/ml
Refractive index n_D at 20 °C	1,387 7 ± 0,000 15
Freezing point, maximum	−90,71 °C
Distillation :	
50 % recovered, °C at 1 013 mbar (760 mmHg)	98,427 ± 0,025
Differential, 80 % recovered minus 20 % recovered, °C	0,02 max.

NOTE – Full references to appropriate test methods for the above requirements are given in ASTM Method D 611, *Aniline point and mixed aniline point of petroleum products and hydrocarbon solvents.*

5.6.3 *Apparatus*

5.6.3.1 **Aniline point apparatus**, as shown in Figure 8, comprising :

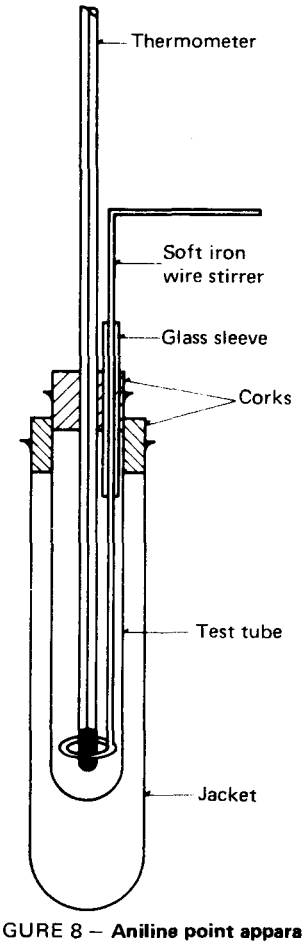

FIGURE 8 – **Aniline point apparatus**

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