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Petroleum and related products from natural or synthetic sources — Determination of distillation characteristics at atmospheric pressure

Produits pétroliers et connexes d'origine naturelle ou synthétique — Détermination des caractéristiques de distillation à pression atmosphérique

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

This fifth edition cancels and replaces the fourth edition (ISO 3405:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

- extension of the scope to include synthetic and biological products in general and automotive petrol-ethanol blends and to diesel with up to 30 % (V/V) FAME specifically;
- the procedure has been aligned with ASTM D86[1] and ASTM International has granted usage of its precision data on 5 July 2017;
- update of the precision (for automated apparatus) for groups 1, 2, and 3, with the slope-based precision obtained from a 2010 Interlaboratory Study[2];
- for T95, group 4 now has a valid range of 260 °C to 360 °C and an updated precision, as a review of a 2006 Interlaboratory Study revealed the absence of some group 4 samples having a final boiling point near 360 °C, as well final boiling points above;
- the test report example in Annex F has been updated as group 0 is not addressed since the fourth edition of this document;
- introduction of a solution for the replacement of mercury-in-glass thermometers.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Petroleum and related products from natural or synthetic sources — Determination of distillation characteristics at atmospheric pressure

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document specifies a laboratory method for the determination of the distillation characteristics of light and middle distillates derived from petroleum and related products of synthetic or biological origin with initial boiling points above 0 °C and end-points below approximately 400 °C, utilizing either manual or automated equipment. Light distillates are typically automotive engine petrol, automotive engine ethanol fuel blends with up to 85 % (*V*/*V*) ethanol, and aviation petrol. Middle distillates are typically aviation turbine fuel, kerosene, diesel, diesel with up to 30 % (*V*/*V*) FAME, burner fuel, and marine fuels that have no appreciable quantities of residua.

NOTE For the purposes of this document, the term "% (*V*/*V*)" is used to represent the volume fraction of a material.

The distillation (volatility) characteristics of hydrocarbons and related products of synthetic or biological origin have an important effect on their safety and performance, especially in the case of fuels and solvents. The boiling range gives important information on composition and behaviour during storage and use, and the rate of evaporation is an important factor in the application of many solvents. Limiting values to specified distillation characteristics are applied to most distillate petroleum product and liquid fuel specifications in order to control end-use performance and to regulate the formation of vapours which may form explosive mixtures with air, or otherwise escape into the atmosphere as emissions (VOC).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 918, *Volatile organic liquids for industrial use — Determination of distillation characteristics*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
decomposition point
thermometer reading (corrected) which coincides with the first indications of thermal decomposition of the liquid in the flask

Note 1 to entry: Characteristic indications of thermal decomposition are an evolution of fumes and erratic thermometer readings which usually show a decided decrease after any attempt has been made to adjust the heat.

3.2
dry point
thermometer reading (corrected) that is observed 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

Note 1 to entry: The end-point (final boiling point), rather than the dry point is intended for general use. The dry point can be reported in connection with special purpose naphthas, such as those used in the paint industry. Also, it is substituted for the end-point (final boiling point) whenever the sample is of such a nature that the precision of the end-point cannot consistently meet the precision requirements given in Clauses 13 or 14.

3.3
final boiling point
end-point maximum thermometer reading (corrected) obtained during the test

Note 1 to entry: This usually occurs after evaporation of all liquid from the bottom of the flask.

3.4
initial boiling point
thermometer reading (corrected) that is observed at the instant that the first drop of condensate falls from the lower end of the condenser tube

3.5
percent evaporated
sum of the percent recovered and the percent loss

3.6
percent loss
calculated amount of uncondensed material

Note 1 to entry: Sometimes called "front-end loss"; this is the amount of uncondensed material lost in the initial stages of the distillation.

3.7
corrected loss
percent loss corrected for barometric pressure

3.8
percent recovered
volume of condensate observed in the receiving cylinder at any point in the distillation, expressed as a percentage of the charge volume, in connection with a simultaneous temperature reading

3.9
percent recovery
maximum percent recovered, as observed in accordance with 9.10 or 10.10

3.10
percent residue
volume of residue measured in accordance with 9.11 or 10.11, and expressed as a percentage of the charge volume

3.11

percent total recovery

combined percent recovery and residue in the flask, as determined in accordance with 11.1

3.12

thermometer reading

temperature recorded by the sensor of the saturated vapour measured in the neck of the flask below the vapour tube, under the specified conditions of this test

3.13

temperature reading

thermometer or temperature-measurement device reading (3.12) which is corrected to 101,3 kPa standard pressure

3.14

emergent stem effect

offset in temperature reading caused by the use of a total immersion mercury-in-glass thermometer in the partial immersion mode

Note 1 to entry: The emergent part of the mercury column is at a lower temperature than the immersed portion, resulting in a lower temperature reading than that obtained when the thermometer was completely immersed for calibration.

3.15

temperature lag

offset in temperature reading between a mercury-in-glass thermometer and an electronic temperature-measurement device, caused by the different response time of the systems involved

4 Principle

The sample is assigned into one of four groups based on its composition and expected volatility characteristics, each group defining the apparatus arrangement, condenser temperature and operational variables. A 100 ml test portion is distilled under the specified conditions appropriate to the group into which the sample falls, and systematic observations of thermometer readings and volumes of condensate recovered are made. The volume of the residue in the flask is measured, and the loss on distillation recorded. The thermometer readings are corrected for barometric pressure, and the data are then used for calculations appropriate to the nature of the sample and the specification requirements.

5 Apparatus

5.1 General

Typical assemblies of the manual apparatus are shown in Figures 1 and 2. In addition to the basic components described in Clause 5, automated apparatus are equipped with a system to measure and automatically record the vapour temperature and the associated recovered volume in the receiving cylinder.

Automated equipment manufactured from 1999 onwards shall be equipped with a device to automatically shut down power to the unit and to spray an inert gas or vapour in the chamber where the distillation flask is mounted in the event of fire.

NOTE Some causes of fires are breakage of the distillation flask, electrical shorts, and foaming and spilling of liquid sample through the top opening of the flask.

5.2 Distillation flasks

The distillation flasks shall have a capacity of 125 ml and be constructed of heat-resistant glass, according to the dimensions and tolerances shown in Figure 3.

For tests specifying the dry point, especially selected flasks with bottoms and walls of uniform thickness are recommended.

5.3 Condenser tube and cooling bath

5.3.1 Typical types of condenser and cooling bath are illustrated in Figures 1 and 2.

Other types of apparatus may be used, provided that the test results obtained by their use are such as to correlate with the results obtained with those illustrated, and to satisfy the precision criteria given in Clauses 13 or 14.

5.3.2 The condenser shall be made of seamless non-corrosive metal tubing, 560 mm ± 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0,8 mm to 0,9 mm.

NOTE Brass or stainless steel are suitable materials.

5.3.3 The condenser shall be set so that 393 mm ± 3 mm of the tube is in contact with the cooling medium, with 50 mm ± 3 mm outside the cooling bath at the upper end, and 114 mm ± 3 mm outside at the lower end. The portion of tube projecting at the upper end shall be set at an angle of 75° ± 3° to the vertical. The portion of the tube inside the cooling bath shall be either straight or bent in any suitable continuous smooth curve. The average gradient shall be 15° ± 1° with respect to the horizontal, and no 100 mm section shall have a gradient outside a 15° ± 3° range. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and the lower end cut off at an acute angle. Provisions shall be made to enable the flow of distillate to run down the side of the receiving cylinder. Figure 4 gives an illustration of the lower end of the condenser tube.

The flow of distillate down the side of the graduated cylinder can be accomplished either by using a drip-deflector which is inserted in the receiver, or by having the downward length of the condenser tube curve slightly backwards so as to ensure contact with the wall of the receiving cylinder at a point 25 mm to 32 mm below the top of the receiving cylinder when it is in position to receive distillate.

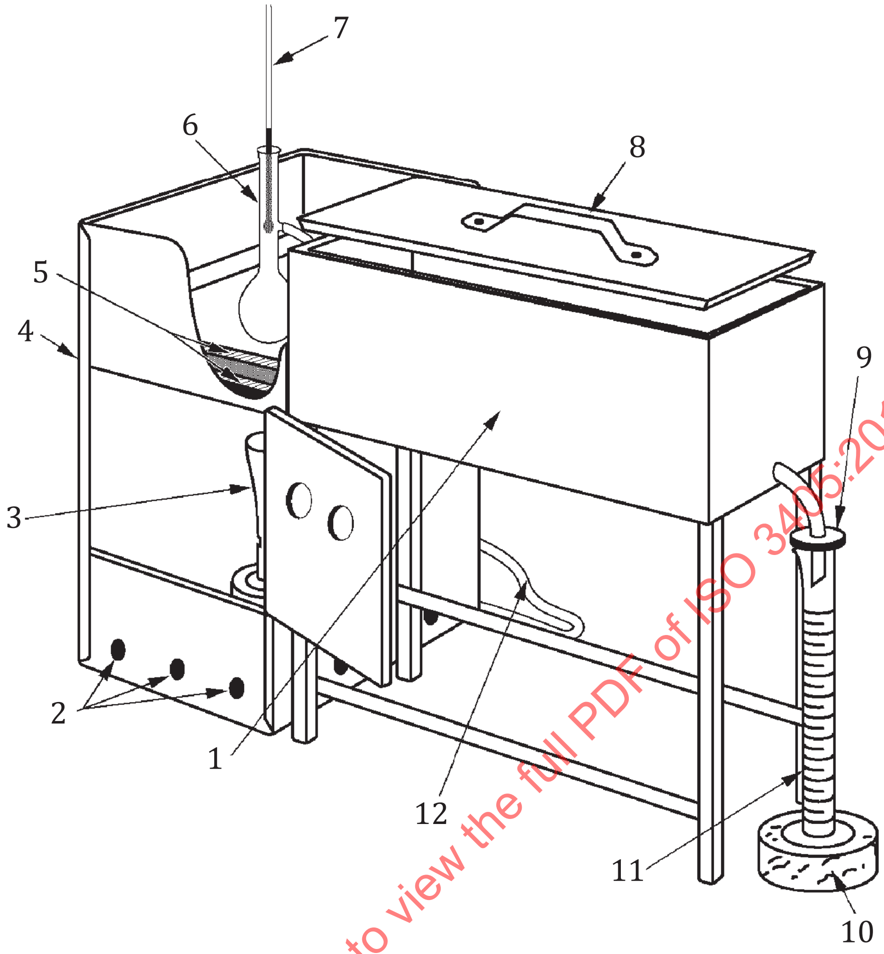
5.3.4 The volume and design of the cooling bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single cooling bath may be used for several condenser tubes.

5.4 Metal shield or enclosure for flask (manual apparatus only)

Shields shall be provided to protect the operator from damage from the unit during operation, and to protect the distillation flask from draughts. They shall allow easy access to the distillation during operation, and be provided with at least one window to observe the dry point at the end of the distillation.

NOTE 1 A typical shield for a unit fitted with a gas burner would be 480 mm high, 280 mm long and 200 mm wide, made of sheet metal approximately 0,8 mm in thickness (see Figure 1).

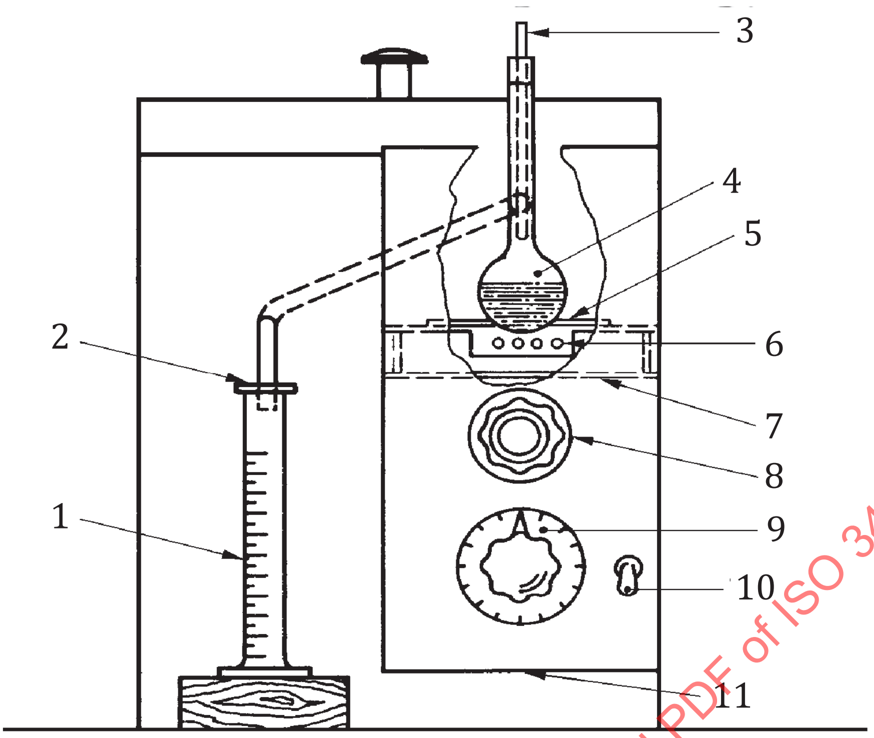
NOTE 2 A typical shield for a unit fitted with an electric heater would be 440 mm high, 200 mm long and 200 mm wide, made of sheet metal approximately 0,8 mm in thickness (see Figure 2).

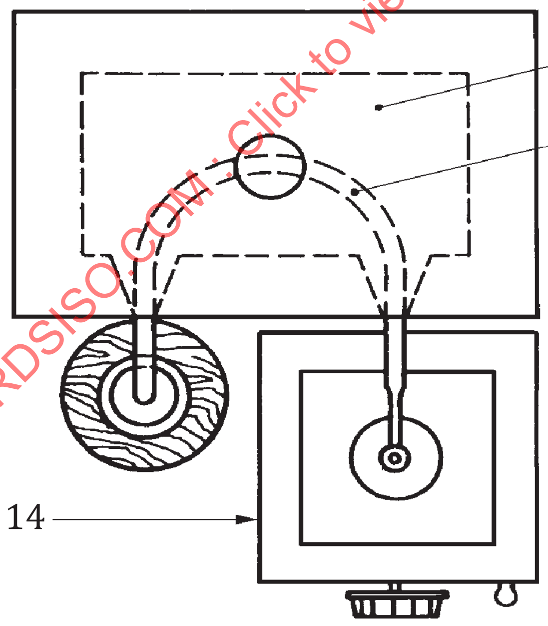


Key

1	cooling bath	7	thermometer
2	air vents	8	bath cover
3	burner	9	blotting paper
4	shield	10	support
5	heat-resistant boards	11	graduated cylinder
6	distillation flask	12	gas line

Figure 1 — Apparatus assembly using a gas burner





Key

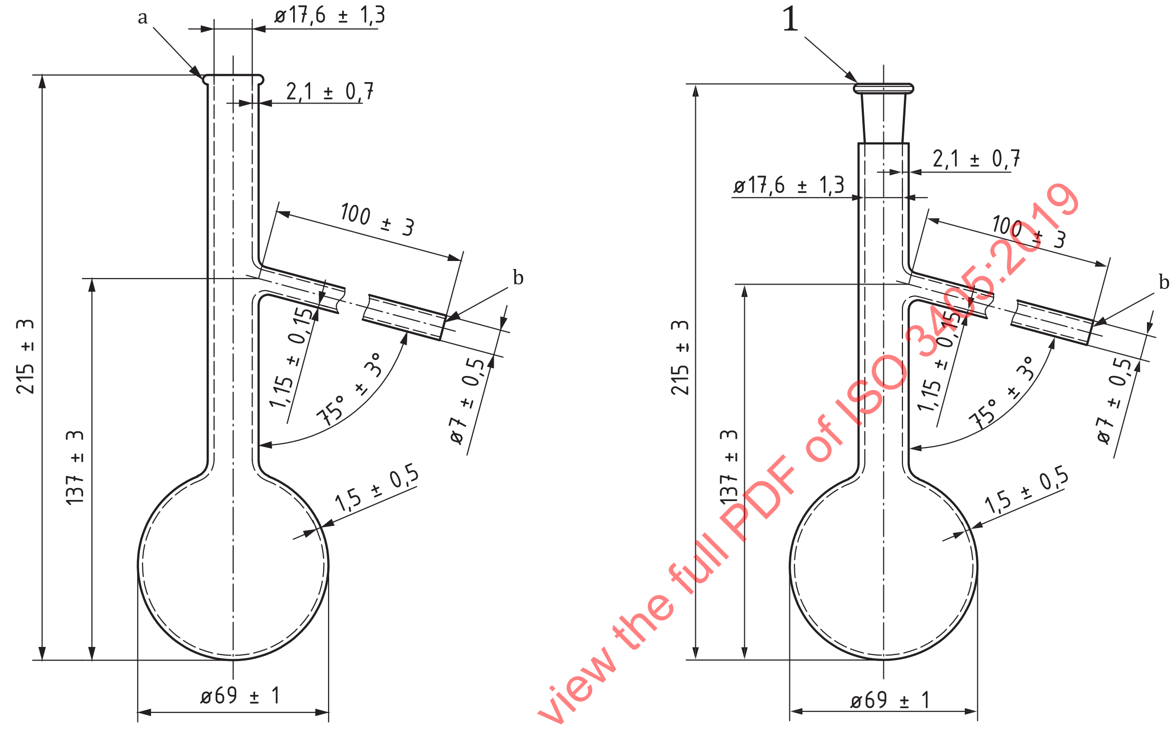
1	receiving cylinder
2	blotting paper
3	thermometer
4	distillation flask
5	flask-support board
6	electric heating element
8	flask-adjusting knob
9	indicating dial
10	switch
11	open bottom shield
12	cooling bath
13	condenser tube

7 flask-support platform

14 shield

Figure 2 — Apparatus assembly using an electric heater

Dimensions in millimetres



Key

1 19/22 neck or 19/26 neck

a Reinforcing bead.

b Fire polished.

Figure 3 — 125 ml flasks — Alternative neck designs

Dimensions in millimetres

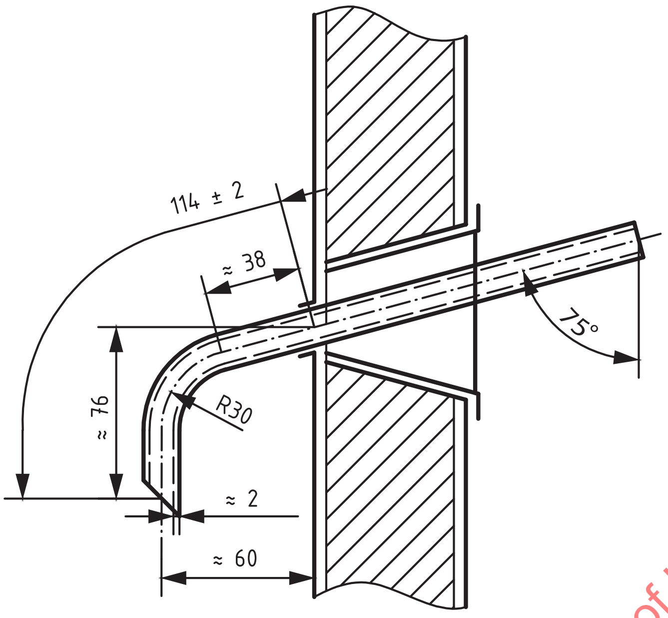


Figure 4 — Lower end of condenser tube

5.5 Heat source(s)

5.5.1 Gas burner (see Figure 1), capable of bringing over the first drop from a cold start within the time specified, and continuing the distillation at the specified rate. A sensitive regulating valve and gas pressure governor to give complete control of heating shall be provided.

5.5.2 Electric heater (see Figure 2), of low heat retention and adjustable from 0 W to 1 000 W.

5.6 Flask-support

5.6.1 Type 1 for use with gas burner (see Figure 1). Either a ring support of the ordinary laboratory type, 100 mm or larger in diameter, supported on a stand inside the shield, or a platform adjustable from the outside of the shield shall be used.

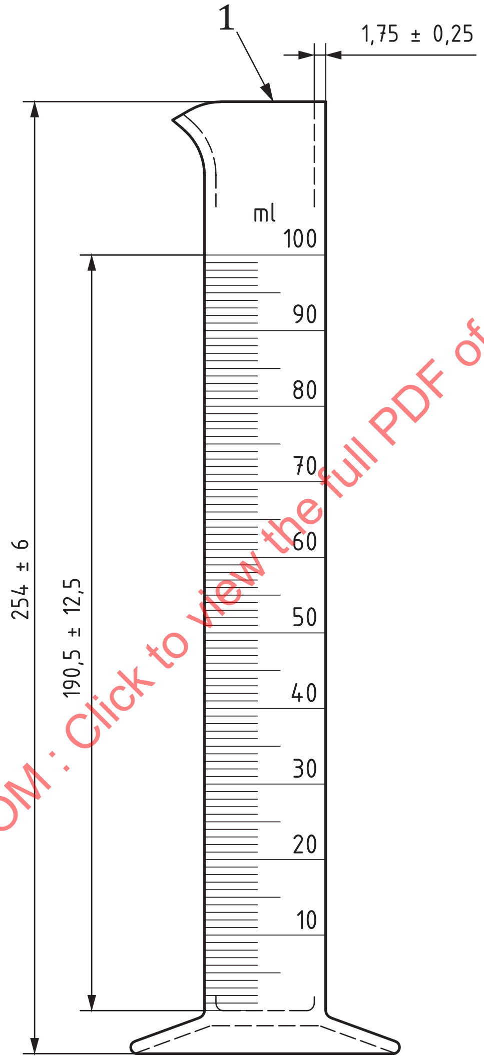
The flask support board shall be constructed of ceramic or other heat-resistant material, 3 mm to 6 mm in thickness and shall have a central opening conforming to the dimensions given in Table 2. The flask support board shall be of sufficient dimension to ensure that thermal heat to the flask only comes from the central opening and that extraneous heat to the flask other than through the central opening is minimized. The flask-support board may be moved slightly in accordance with the directions for positioning the distillation flask so that direct heat is applied to the flask only through the opening in this board. The position of the flask is set by adjusting the length of the side-arm inserted into the condenser.

5.6.2 Type 2 for use with an electric heater (see Figure 2). The flask-support is a platform on top of the electric heater and adjustable from the outside of the shield. The flask support board described in 5.6.1 is mounted on this support. Provision shall be made for moving the upper (flask-support) board slightly in the horizontal plane to ensure that direct heat is applied only through the specified opening in this board. The flask-support assembly shall be able to move vertically to ensure contact of the flask-support board with the bottom of the distillation flask during the distillation, and to allow for easy mounting and removal of the distillation flask from the unit.

5.7 Graduated cylinders

5.7.1 Receiving cylinder, of 100 ml ± 1,0 ml capacity, nominally in accordance with ISO 4788. It shall be graduated at intervals of 1 ml beginning at least at the 5 ml mark and have a graduation at the 100 ml mark. The shape of the base shall be such that the receiver does not topple when placed empty on a surface inclined at an angle of 13° to the horizontal. Construction details and tolerances for the graduated cylinder are shown in Figure 5.

Dimensions in millimetres



Key

1 fire polished top end

Figure 5 — 100 ml receiving cylinder (tolerance ± 1,0 ml)

For automated apparatus, the cylinder shall conform to the physical specifications described in this subclause, with the exception of all graduations but that at 100 ml. Receiving cylinders for use in automated units may also have a metal base.

If required, the receiving cylinder shall be either immersed in a cooling bath containing cooling liquid, such as a tall-form beaker of clear glass or transparent plastic, up to above the 100 ml graduation line, or placed in a thermostatically controlled air-circulation chamber.

5.7.2 Residue cylinder, of 5 ml capacity, generally in accordance with ISO 4788.

5.8 Temperature measurement system

5.8.1 Thermometers, if used, shall be of the mercury-in-glass type, nitrogen filled, graduated on the stem and enamel backed, and shall conform to the specifications given in Annex A.

CAUTION — Under certain test conditions, the bulb of the thermometer can be 28 °C above the temperature indicated, and at an indicated temperature of 370 °C, the temperature of the bulb is approaching a critical range in the glass. It is thus strongly recommended that distillation temperature readings above 370 °C are avoided, but in those cases where thermometers have been exposed to observed temperature readings above 370 °C, they shall not be re-used without checking their ice point to verify calibration.

5.8.2 Electronic temperature-measurement devices, if used, shall exhibit the same temperature lag, emergent stem effect and accuracy as the equivalent mercury-in-glass thermometer.

To simulate the temperature lag of a mercury-in-glass thermometer, the circuitry and/or the algorithms used for the electronic system shall take this fact into account.

Alternatively, place the sensor in a casing with the tip covered, so that the assembly, because of its adjusted thermal mass and conductivity, has a temperature lag time similar to that of mercury-in-glass thermometers.

In case of dispute, unless otherwise agreed, the referee test shall be carried out using the specified mercury-in-glass thermometers.

When it is required to determine the difference in lag time between an electronic measurement system and mercury-in-glass thermometers Annex B shall be used.

NOTE The lag time difference determination is more applicable for equipment manufacturers.

5.9 Centring device

The temperature sensor shall be fitted through a snug-fitting device designed for mechanically centring the sensor in the neck of the distillation flask without vapour leakage. The use of a cork or silicone rubber stopper with a hole drilled through the centre is not acceptable for this purpose. Examples of acceptable centring devices are shown in Figures 6 and 7.

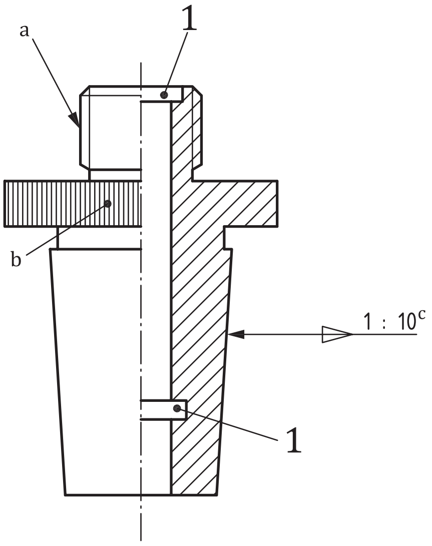
When running tests by the manual method, products with a low initial boiling point can have one or more temperature readings obscured by the centring device.

Centring devices not shown in Figures 6 and 7 are also acceptable provided they position and hold the temperature sensor in the middle of the neck of the distillation flask.

5.10 Barometer

The barometer shall be capable of measuring atmospheric pressure with an accuracy of 0,1 kPa or better, at the same elevation relative to sea level as the apparatus in the laboratory. Do not take readings from aneroid barometers that are pre-corrected to give sea level pressures.

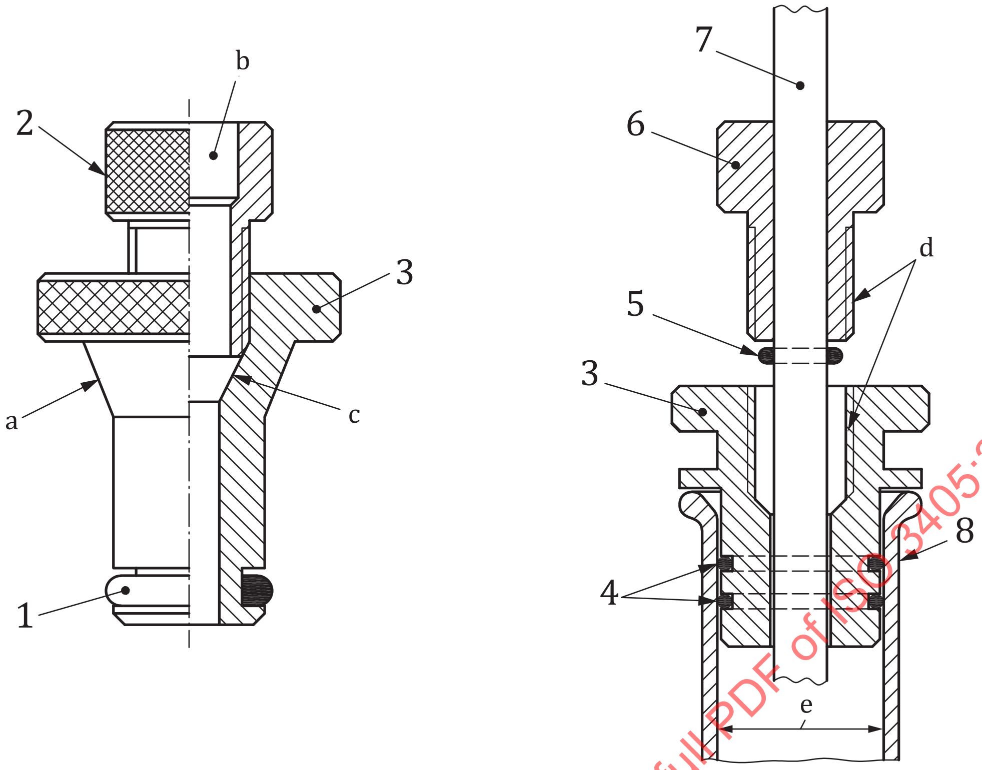
The barometer should ideally be located in the room in which the distillation is carried out.



Key

1	O-ring
a	Screwcap.
b	Knurled knob.
c	Cone male NS 19/26.

Figure 6 — PTFE centring device for ground-glass joint



Key

1	single O-ring Viton or perfluoro elastomer
2	compression nut (PTFE)
3	PTFE body
4	double O-rings Viton or perfluoro elastomer
5	compression O-ring
6	compression nut
7	thermometer or Pt 100 probe
8	neck of distilling flask

a	Cone-shaped for perfect centring in neck of distillation flask.
b	Drilled out to fit Pt 100 probe.
c	Compression without O-ring.
d	Threads.
e	Internal diameter of flask to be precision bore.

Figure 7 — Two illustrative centring device designs for straight-bore neck

6 Samples and sampling

6.1 Sample grouping

Determine the nature of the product to be sampled and place it in the appropriate group according to Table 1, which also gives general guidance on sampling conditions.

6.2 Sample maintenance prior to testing

6.2.1 General

Unless otherwise specified, sampling shall be carried out by the procedures described in ISO 3170 or ISO 3171, bearing in mind the special conditions described in Table 1. Maintain samples prior to testing at the specified temperatures given in Table 1, away from sources of direct heat or sunlight.

6.2.2 Groups 1 and 2

Collect the sample in a container previously cooled to below 10 °C, when necessary. Condition the container preferably by immersing it in the liquid, where possible, and discarding the first sample. Where immersion is not possible, the sample shall be drawn off into the previously cooled container in such a manner that agitation is kept at a minimum. Close the container immediately with a tight-fitting stopper, and place the sample in an ice bath or refrigerator to maintain the sample below the specified temperature. Maintain the sample below 10 °C prior to testing, and preferably store at or below this temperature. Where maintenance and/or storage at below 10 °C is not possible or practicable, a temperature up to 20 °C is acceptable provided that the sample is always conditioned to a temperature below 10 °C before the container is opened.

6.2.3 Groups 3 and 4

Maintain the sample at ambient temperature. If the sample is not fluid at ambient temperature, maintain it at a temperature of 9 °C to 21 °C above its pour point. Shake the sample vigorously prior to subsampling to ensure homogeneity, and disregard the temperature range shown in Table 2 for the receiving cylinder. Prior to analysis, heat the receiving cylinder to approximately the same temperature as the sample, and pour the heated test portion precisely to the 100 ml mark. Transfer the test portion as rapidly and completely as possible to the distillation flask.

CAUTION — A tightly-sealed, full, cold container of sample is likely to break if heated.

Table 1 — Sample groups and sampling conditions

Group number	1	2	3	4
Typical sample type	Gasoline	Gasoline	Wide-cut aviation fuel	Kerosene/ gas oil
Vapour pressure (DVPE), kPa	≥65,5	<65,5	<65,5	<65,5
Initial boiling point (IBP), °C	—	—	≤100	>100
Final boiling point (FBP), °C	≤250	≤250	>250	>250
Temperature of sample bottle, °C	<10	—	—	—
Temperature of sample at sampling, °C	≤10	≤10	Ambient[a]	Ambient[a]
Temperature of stored sample, °C	<10[b]	<10[b]	Ambient	Ambient
If sample is wet	Resample or dry	Resample or dry	Dry	Dry
(Reference)	(6.3.2)	(6.3.2)	(6.3.3)	(6.3.3)

[a] Samples shall always be at 9 °C to 21 °C above their pour point if not fluid at ambient temperature.

[b] When no facilities available for storage below 10 °C, the sample may be stored below 20 °C provided the container is tightly sealed. See 6.2.2.

6.3 Removing water from sample

6.3.1 General

Samples of materials that are visibly hazy (suspended water) or are suspected of containing water are not suitable for testing.

6.3.2 Groups 1 and 2

If the sample is not dry, obtain another sample for testing that is free from suspended water. If such a sample cannot be obtained, add a sufficient amount of anhydrous sodium sulfate or other suitable drying agent to the sample maintained at 0 °C to 10 °C, and physically remove the water by shaking. Once the sample shows no visible signs of water, use a decanted portion of the sample, maintained at 0 °C to 10 °C, for the analysis. Record that the sample has been dried by desiccant.

Data from a round-robin exercise show that suspended water in hazy samples in group 1 and group 2 may be removed by the above procedure without statistically affecting the results of the test.

6.3.3 Groups 3 and 4

In cases where a water-free sample is not practical, remove the suspended water by shaking the sample with anhydrous sodium sulphate or another suitable drying agent, and separate it from the drying agent by decantation.

7 Preparation of apparatus

7.1 Refer to Table 2 and prepare the apparatus by choosing the appropriate distillation flask, temperature-measurement system and flask-support board as directed for the indicated group. If gas heating is used, use a Type 1 flask-support (5.6.1); if electric heating is used, use a Type 2 flask-support (5.6.2). Bring the temperature of the receiving cylinder, the flask, the temperature sensor and the cooling bath to the indicated temperature.

7.2 Make any necessary provisions so that the temperature of the cooling bath and receiving cylinder will be maintained at their specified temperatures. The receiving cylinder shall be positioned in a bath such that either the liquid level is at least as high as the 100 ml mark, or the entire receiving cylinder is surrounded by an air-circulation chamber.

For groups 1, 2 and 3, use suitable media for low temperature baths such as chopped ice and water, refrigerated brine and refrigerated ethylene glycol.

For group 4, use suitable media for ambient and higher bath temperatures such as cold water, hot water and heated ethylene glycol.

7.3 Remove any residual liquid in the condenser tube by swabbing with a piece of soft, lint-free cloth attached to a cord or wire.

7.4 For samples in groups 1, 2 and 3, fit a low-range temperature sensor, provided with a snug-fitting cork or silicone rubber stopper, tightly into the neck of the sample container, and bring the temperature of the sample to that specified in Table 2.

Table 2 — Preparation of apparatus

Group number	**1**	**2**	**3**	**4**
Temperature-measurement system (see 5.8)	Low-range	Low-range	Low-range	High-range
Diameter of hole in flask-support board, mm	38	38	50	50
Temperature at start of test, °C				
— flask and thermometer	13 to 18	13 to 18	13 to 18	≤ambient
— flask-support board and shield	≤ambient	≤ambient	≤ambient	—
— receiving cylinder and sample	13 to 18	13 to 18	13 to 18	13 to ambient

7.5 When the temperature of the sample is confirmed, measure the test portion precisely to the 100 ml mark of the receiving cylinder, and then transfer it as completely as practical to the distillation flask, taking care that none of the liquid flows into the vapour tube.

NOTE 1 Any material which evaporates during the transfer will contribute to the loss; any material which remains in the receiving cylinder will contribute to the observed recovery volume at the time of the initial boiling point.

If irregular boiling (bumping) is expected, add a small volume of clean and dry boiling chips to the test portion.

NOTE 2 Although theoretically the volume of deposit on the chips is part of the residue, this amount is negligible and can be disregarded.

7.6 Fit the appropriate temperature sensor through a centring device (5.9). In the case of a mercury-in-glass thermometer, the bulb is centred in the neck and the lower end of the capillary is level with the highest point on the bottom of the inner wall of the vapour tube (see Figure 8). In the case of a thermocouple/resistance thermometer, follow the manufacturer's instructions as to placement so that the sensing position will be just below the level of the side arm of the distillation flask.

Use a small quantity of vacuum grease on the mating surface of the polytetrafluoroethylene (PTFE) centring device to facilitate dismantling after use.

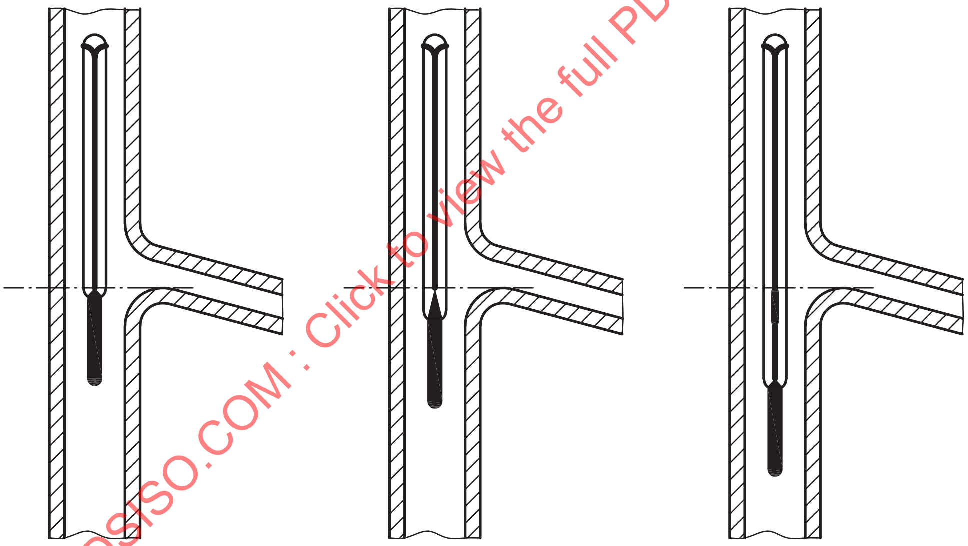


Figure 8 — Position of thermometer in distillation flask neck

7.7 Fit the flask vapour tube, provided with a snug-fitting, well-rolled cork or silicone rubber stopper, tightly into the condenser tube. Adjust the distillation flask in a vertical position so that the vapour tube extends into the condenser tube for a distance of 25 mm to 50 mm. Raise and adjust the flask-support board to fit snugly against the bottom of the flask.

7.8 Place the receiving cylinder that was used to measure the test portion, without drying, fitted with a drip deflector if necessary, into the bath under the lower end of the condenser tube so that the end of the condenser tube is centred in the receiving cylinder and extends therein for a distance of at least 25 mm, but not below the 100 ml mark. For manual distillations, cover the receiving cylinder closely with a piece of blotting paper or similar material that has been cut to fit the condenser tube snugly. For automated distillations, cover the receiving cylinder, if necessary, with the device provided with the apparatus.

7.9 Record the room temperature and prevailing barometric pressure. Proceed at once with the distillation, as described in Clauses 9 or 10.

8 Apparatus verification

8.1 Level follower

For automated apparatus, the level follower/recording mechanism of the apparatus shall have a resolution of 0,1 ml with a maximum error of 0,3 ml between the 5 ml and 100 ml points. The calibration of the assembly shall be verified according to the manufacturer's instructions at intervals of not more than three months.

NOTE The typical verification procedure involves verifying the output with the receiving cylinder containing 5 ml and 100 ml of material respectively.

8.2 Electronic temperature-measurement devices

8.2.1 Verification of the calibration of these temperature sensors shall be carried out at intervals of not more than 6 months. The electronic circuit for resistance thermometers shall be verified against a standard precision resistance. When performing this verification, ensure that any algorithms used to correct the temperature for lag and emergent stem effect are not applied (see the manufacturer's instructions). The response of the temperature-measurement system itself shall be verified by one of the procedures described in 8.2.2 or 8.2.3 or 8.2.4.

8.2.2 Distil reagent grade toluene in accordance with ISO 918 and determine whether the 50 % (*V*/*V*) recovered temperature is within the following temperature range. Use reagent grade toluene for this test unless it can be shown that the grade proposed is of sufficient purity to permit its use without lessening the accuracy of the determination.

Toluene is used as a verification fluid for bias; it will yield almost no information on how well an electronic measurement system simulates the temperature lag of a mercury-in-glass thermometer. Annex B describes a method to determine this difference in lag time.

To verify the response at elevated temperatures, use hexadecane.

NOTE 1 Toluene is shown in reference manuals as boiling at 110,6 °C, and hexadecane at 287,0 °C, under the conditions of ISO 918, which uses a partial immersion thermometer. Because this document uses thermometers calibrated for total immersion, the results will be lower, and different for each. The approximate values obtained from an interlaboratory study are shown below:

	Manual apparatus	**Automated apparatus**
Toluene (group 1,2,3)	105,9 °C to 111,8 °C	108,5 °C to 109,7 °C
Hexadecane (group 4)	272,2 °C to 283,1 °C	277,0 °C to 280,0 °C

8.2.3 Distil a reference fluid of a specific product group, under the conditions of this document, using in sequence, the appropriate mercury-in-glass thermometer followed by the alternative temperature-measurement device or system. Record systematic deviations over the range of temperatures measured to ensure that no deviation exceeds the repeatability of this test method, and that the algebraic sum of the deviations is close to zero.

8.2.4 Distil a certified reference fluid of a specific product group, under the conditions of this document. Record systematic deviations over the range of temperatures measured to ensure that no deviation exceeds the repeatability of this test method, and that the algebraic sum of the deviations is close to zero.

NOTE 2 Certified reference fluids, consisting of specified mixtures of single compounds, are commercially available for group 1 and group 4 product types.

8.3 Electronic pressure measuring device

At intervals of not more than six months, and after the system has been repaired or replaced, the barometric reading of the device shall be verified against a barometer as described in 5.10.

9 Procedure — Manual apparatus

9.1 Apply heat to the distillation flask (5.2) and contents, with the drip tip of the condenser (see 5.3) away from the wall of the receiving cylinder (5.7.1) allowing observation for the first condensation. Regulate the heating (5.5) at this stage so that the time interval between the first application of heat and the initial boiling point is as specified in Table 3.

9.2 Observe and record the first drop of condensate as the initial boiling point to the nearest 0,5 °C. If a receiver deflector is not being used, immediately move the receiving cylinder so that the tip of the condenser touches the inner wall.

Table 3 — Conditions during test procedure

Group number	1	2	3	4
Temperature of condenser bath, ° C	0 to 1	0 to 5	0 to 5	0 to 60
Temperature of medium around receiving cylinder, ° C	13 to 18	13 to 18	13 to 18	±3 of charge
Time from first heat to IBP, min	5 to 10	5 to 10	5 to 10	5 to 15
Time from IBP to 5 % (*V*/*V*) recovered, s	60 to 100	60 to 100	—	—
Uniform average rate of condensation from 5 % (*V*/*V*) recovered to 5 ml in flask, ml/min	4 to 5	4 to 5	4 to 5	4 to 5
Time from 5 ml residue in flask to FBP, min	≤5	≤5	≤5	≤5

9.3 Regulate the heating so that the time from initial boiling point to 5 % (*V*/*V*) recovered is as indicated in Table 3.

9.4 Continue to regulate the heating so that the uniform average rate of condensation from 5 % (*V*/*V*) recovered to 5 ml residue in the flask is 4 ml/min to 5 ml/min.

9.4.1 Due to the configuration of the distillation flask and the conditions of the test, the vapour and liquid around the temperature sensor (see 5.8) are not in thermodynamic equilibrium. The distillation rate will consequently have an effect on measured vapour temperature, and thus this should be kept as constant as possible throughout the test.

9.4.2 In the context of this test method, “uniform average rate of condensation” has the following intention. Heating of the boiling flask shall be regulated to maintain as best as possible a uniform flow of condensation, which will then provide the most desired precision for the test. However, some distillation tests can have one or more short-term rates of condensation which deviate from the 4 ml/min to 5 ml/min indicated in 9.4 and Table 3, this is a common occurrence for some sample types.

The periods of these short-term deviations may last for several percent of material condensed until the temperature slope regains constancy, and may occur at several periods along the entire condensation range. These deviations will typically correct after the temperature slope again becomes constant.

These short-term deviations shall not occur over the entire range of condensation. Typically, these short-term deviations should not occur for more than ten contiguous percent volume. The precision of the temperature readings will be significantly affected during these periods. When the overall calculated average rate of condensation between 5 % recovered and 5 ml residue is within the prescribed rate, the requirement of 9.4 and Table 3 is satisfied.

As example, those samples containing a 10 % ethanol-fuel blend or those that exhibit a significant change of temperature slope at points during the distillation can have a short-term rate of condensation which deviates from the 4 ml/min to 5 ml/min indicated in 10.4 and Table 3.

NOTE When testing group 1 or group 2 materials, the condensate can form non-miscible phases and bead upon the thermometer and in the neck of the distillation flask at a vapour temperature of approximately 160 °C. This is accompanied by a sharp (approximately 3 °C) dip in vapour temperature and a drop in recovery rate. The phenomenon can last for 10 s to 40 s, before the temperature recovers and the condensate starts flowing again. This point is referred to as the “hesitation point”.

9.5 Repeat any distillation that does not meet the conditions described in 9.1 to 9.4.

9.6 If a decomposition point is observed, discontinue the heating and proceed as directed in 9.10.

9.7 In the interval between the initial boiling point and the end of the distillation, observe and record data necessary for the calculation and reporting of the results of the test as required by the specification involved, or as previously established for the material under test.

These observed data may include thermometer readings at specified percentages recovered, or percentages recovered at specified thermometer readings.

Record all volumes in the receiving cylinder to the nearest 0,5 ml and all thermometer readings to the nearest 0,5 °C.

For groups 1, 2, 3 and 4, in cases in which no specific data requirements have been indicated, record the initial boiling point, the final boiling point or dry point, or both, and thermometer readings at 5 %, 15 %, 85 % and 95 % volume recovered, and at each 10 % multiple of volume recovered from 10 % to 90 % inclusive.

For group 4, when the high-range thermometer is used for testing kerosene, pertinent thermometer readings can be obscured by the centring device. If these readings are required, perform a second distillation in accordance with the requirements of group 3. In such cases, readings from the low-range thermometer are reported in place of the obscured high-range thermometer readings, and the test report shall so indicate. If, by agreement, the obscured readings are waived, the test report shall indicate this (see Clause 15).

When required to report the thermometer reading at a specified percent evaporated or recovered for a sample which has a rapidly changing slope of the distillation curve in the region of the specified percent evaporated or recovered reading, record thermometer readings at every 1 % (V/V) recovered. The slope is considered rapidly changing if the change in slope, C, of the data points in that particular area is greater than 0,6 as calculated from Formula (1).

$$C=\frac{(T_2-T_1)}{(V_2-V_1)}-\frac{(T_3-T_2)}{(V_3-V_2)} \qquad (1)$$

where

T_1 is the thermometer reading at the volume percent recorded one reading prior to that in question, expressed in degrees Celsius;

T_2 is the thermometer reading at the volume percent recorded in question, expressed in degrees Celsius;

T_3 is the thermometer reading at the volume percent recorded following that in question, expressed in degrees Celsius;

V_1 is the volume percent recorded one reading prior to that in question;

V_2 is the volume percent recorded in question;

V_3 is the volume percent recorded following that in question.

9.8 When the residual liquid in the distillation flask is approximately 5 ml, make a final adjustment to the heat. To determine when the residual liquid in the distillation flask is approximately 5 ml, subtract the estimated loss from 93,5 ml and estimate the amount recovered in the receiving cylinder at that time.

The time from the 5 ml of liquid residue in the distillation flask to the final boiling point shall be within the limits specified in Table 3. If this condition is not satisfied, repeat the test, with appropriate modification of the final heat adjustment.

If the actual loss differs by more than 2 ml from the estimated value, repeat the test.

Since it is extremely difficult to determine the amount of boiling residual liquid in the distillation flask, this is estimated from the amount of liquid recovered in the receiving cylinder at this time. The dynamic hold-up has been determined to be approximately 1,5 ml at this point, and thus with no losses, the amount of 5 ml in the distillation flask may be assumed to correspond to an amount of 93,5 ml in the receiving cylinder.

9.9 Observe and record the final boiling point, or dry point, or both, as required, and discontinue heating.

9.10 Allow the distillate to continue to drain into the receiving cylinder, observe and note the volume of condensate, to the nearest 0,5 ml, at 2 min intervals until two successive observations agree. Measure this volume accurately, and record it, to the nearest 0,5 ml, as percent recovery. If the distillation was previously discontinued under the conditions of a decomposition point, deduct the percent recovery from 100, report this difference as percent residue and loss, and omit the procedure described in 9.11.

9.11 After the distillation flask has cooled, and no more vapours are observed, disconnect the distillation flask from the condenser, pour its contents into the 5 ml residue cylinder (5.7.2), and with the distillation flask suspended over the 5 ml residue cylinder, allow the flask to drain until no appreciable increase in volume of the liquid in the 5 ml residue cylinder is observed. Record the volume in the residue cylinder, to the nearest 0,1 ml, as percent residue.

For viscous residue, pour the contents into the 5 ml residue receiver while the flask is warm and allow to cool.

CAUTION — If boiling chips were added, care should be taken to avoid their inclusion in this volume.

If the 5 ml residue cylinder does not have graduations below 1 ml, and the volume of liquid is less than 1 ml, prefill the residue cylinder with 1 ml of heavy oil to obtain a better reading of the volume of residue.

For group 4 products, examine the condenser arm and the side arm of the distillation flask for waxy or solid deposits. If found, repeat the test, adjusting the temperature of the condenser bath upwards within the range specified in Table 3.

9.12 If only the percent evaporated or percent recovered at a predetermined corrected thermometer reading is required, the procedure shall be modified as described in Annex C.

10 Procedure — Automated apparatus

10.1 Apply heat to the distillation flask (5.2) and contents. Ensure that the drip deflector tip is touching the wall of the receiving cylinder. Regulate the heating (5.5) at this stage so that the time interval between the first application of heat and the initial boiling point is as specified in Table 3.

10.2 Record the first drop of condensate as the initial boiling point to the nearest 0,1 °C.

10.3 Regulate the heating so that the time from initial boiling point to 5 % (V/V) recovered is as indicated in Table 3.

10.4 Continue to regulate the heating so that the uniform average rate of condensation from 5 % (V/V) recovered to 5 ml residue in the flask is 4 ml/min to 5 ml/min.

10.4.1 Due to the configuration of the distillation flask and the conditions of the test, the vapour and liquid around the temperature sensor (see 5.8) are not in thermodynamic equilibrium. The distillation rate will consequently have an effect on measured vapour temperature, and thus this should be kept as constant as possible throughout the test.

10.4.2 In the context of this test method, "uniform average rate of condensation" has the following intention. Heating of the boiling flask shall be regulated to maintain as best as possible a uniform flow of condensation, which will then provide the most desired precision for the test. However, some distillation tests can have one or more short-term rates of condensation which deviate from the 4 ml/min to 5 ml/min indicated in 10.4 and Table 3, this is a common occurrence for some sample types.

The periods of these short-term deviations may last for several percent of material condensed until the temperature slope becomes constant again, and may occur at several periods along the entire condensation range. These deviations will typically correct after the temperature slope again becomes constant. These short-term deviations shall not occur over the entire range of condensation.

Typically, these short-term deviations should not occur for more than ten contiguous percent volume. The precision of the temperature readings will be significantly affected during these periods. When the overall calculated average rate of condensation between 5 % recovered and 5 ml residue is within the prescribed rate, the requirement of 10.4 and Table 3 is satisfied.

As example, those samples containing a 10 % ethanol-fuel blend or those that exhibit a significant change of temperature slope at points during the distillation can have a short-term rate of condensation which deviates from the 4 ml/min to 5 ml/min indicated in 10.4 and Table 3.

NOTE When testing group 1 or group 2 materials, the condensate can form non-miscible phases and bead upon the thermometer and in the neck of the distillation flask at a vapour temperature of approximately 160 °C. This is accompanied by a sharp (approximately 3 °C) dip in vapour temperature and a drop in recovery rate. The phenomenon can last for 10 s to 40 s, before the temperature recovers and the condensate starts flowing again. This point is referred to as the "hesitation point".

10.5 Repeat any distillation that does not meet the conditions described in 10.1 to 10.4.

10.6 If a decomposition point is observed, discontinue the heating and proceed as directed in 10.10.

10.7 In the interval between the initial boiling point and the end of the distillation, record data necessary for the calculation and reporting of the results of the test as required by the specification involved, or as previously established for the material under test.

These data may include vapour temperature readings at specified percentages recovered, or percentages recovered at specified vapour temperature readings.

Record all volumes in the receiving cylinder to the nearest 0,1 ml and all temperature readings to the nearest 0,1 °C.

In cases in which no specific data requirements have been indicated, record the initial boiling point, the final boiling point or dry point, or both, and thermometer readings at 5 %, 15 %, 85 % and 95 % volume recovered, and at each 10 % multiple of volume recovered from 10 % to 90 % inclusive.

10.8 When the residual liquid in the distillation flask is approximately 5 ml, make the final adjustment to the heat. To determine when the residual liquid in the distillation flask is approximately 5 ml, subtract the estimated loss from 93,5 ml and estimate the amount recovered in the receiving cylinder at that time.

The time from the 5 ml of liquid residue in the distillation flask to the final boiling point shall be within the limits specified in Table 3. If this condition is not satisfied, repeat the test, with appropriate modification of the final heat adjustment.

If the actual loss differs by more than 2 ml from the estimated value, repeat the test.

Since it is extremely difficult to determine the amount of boiling residual liquid in the distillation flask, this is estimated from the amount of liquid recovered in the receiving cylinder at this time. The dynamic hold-up has been determined to be approximately 1,5 ml at this point, and thus with no losses, the amount of 5 ml in the distillation flask may be assumed to correspond to an amount of 93,5 ml in the receiving cylinder.

10.9 Record the final boiling point, or dry point, or both, as required, and discontinue heating.

10.10 Allow the distillate to continue to drain into the receiving cylinder, monitor the volume of condensate, to the nearest 0,1 ml, at 2 min intervals until two successive observations agree. Record this volume to the nearest 0,1 ml, as percent recovery.

10.11 After the distillation flask has cooled, disconnect the distillation flask from the condenser, pour its contents into the 5 ml residue cylinder (5.7.2), and with the distillation flask suspended over the 5 ml residue cylinder, allow the flask to drain until no appreciable increase in volume of the liquid in the 5 ml residue cylinder is observed. Measure and record the volume in the residue cylinder, to the nearest 0,1 ml, as percent residue.

For viscous residue, pour the contents into the 5 ml residue receiver while the flask is warm and allow to cool.

CAUTION — If boiling chips were added, care should be taken to avoid their inclusion in this volume.

If the 5 ml residue cylinder does not have graduations below 1 ml, and the volume of liquid is less than 1 ml, prefill the residue cylinder with 1 ml of heavy oil to obtain a better reading of the volume of residue.

For group 4 products, examine the condenser arm and the side arm of the distillation flask for waxy or solid deposits. If found, repeat the test, adjusting the temperature of the condenser bath upwards within the range specified in Table 3.

11 Calculations

11.1 The percent total recovery is the sum of the percent recovery (9.10 or 10.10) and the percent residue (9.11 or 10.11). Deduct the percent total recovery from 100 to obtain the percent loss.

11.2 Correct temperature readings to 101,3 kPa pressure. Obtain the correction, T_C, to be applied to each temperature reading by means of the Sydney Young formula, see Formula (2) below, or by reference to Table 4.

$$T_C = 0{,}000\ 9(101{,}3 - p_k)(273 + T) \tag{2}$$

where

p_k is the barometric pressure prevailing at the time and location of the test, expressed in kilopascals;

T is the observed temperature reading, expressed in degrees Celsius.

The absolute value of the temperature correction is to be added to the recorded temperature reading if the barometric pressure is less than 101,3 kPa; it is to be subtracted if the barometric pressure is greater than 101,3 kPa.

For absolute accuracy, observed barometric pressures should be corrected to 0 °C and standard gravity, to compensate for changes in the gravity of the earth with respect to latitude. These compensations are small, and would result in a further correction of less than 0,2 °C in boiling point in the most extreme case. Unless specifically requested, these compensations are not required.

Temperature readings are not corrected to 101,3 kPa when product definitions, specifications, or agreements between the parties involved indicate, specifically, that such correction is not required, or that correction shall be made to some other base pressure.

After applying the corrections and rounding each result to the nearest 0,5 °C for manual apparatus or 0,1 °C for automated apparatus, use the corrected temperature readings in all further calculations and reporting.

Table 4 — Approximate temperature sensor reading corrections

Temperature range °C	**Correction** °C/kPa	**Temperature range** °C	**Correction** °C/kPa
10 to 29,5	0,27	210 to 229,5	0,45
30 to 49,5	0,29	230 to 249,5	0,48
50 to 69,5	0,31	250 to 269,5	0,49
70 to 89,5	0,32	270 to 289,5	0,51
90 to 109,5	0,35	290 to 309,5	0,53
110 to 129,5	0,36	310 to 329,5	0,55
130 to 149,5	0,38	330 to 349,5	0,57
150 to 169,5	0,40	350 to 369,5	0,58
170 to 189,5	0,42	370 to 389,5	0,60
190 to 209,5	0,44	390 to 410	0,62

11.3 Correct the actual loss to 101,3 kPa pressure when the temperature readings are corrected to 101,3 kPa pressure. Calculate the corrected loss, L_c, in percent volume, from Formula (3).

$$L_c = 0{,}5 + \frac{L - 0{,}5}{1 + (101{,}3 - p) \times 0{,}125} \tag{3}$$

where

L is the percent loss calculated from test data, expressed in percent volume;

P is the observed barometric pressure, expressed in kilopascals.

NOTE Formula (3) has been derived from experimental data, which is referenced in ASTM D86-96 and later editions[1].

11.4 Calculate the corresponding corrected percent recovery, R_c, in percent volume, from Formula (4).

$$R_c = R + (L - L_c) \tag{4}$$

where

R is the observed recovery, expressed in percent volume;

L is the observed percent loss, expressed in percent volume;

L_c is the corrected percent loss, expressed in percent volume.

11.5 To report percentages evaporated at specified temperature readings, add the observed percent loss to each of the observed percentages recovered at the specified temperature readings, and report these results as the respective percentages evaporated, as seen in Formula (5).

$$P_e = P_r + L \tag{5}$$

where

P_e is the percentage volume evaporated;

P_r is the percentage volume recovered;

L is the observed percent loss, expressed in percent volume.

11.6 Manual percent evaporated procedure is as follows.

To report temperature readings at specified percentages evaporated, use either of the procedures given below, and indicate on the test report whether the graphical procedure or the arithmetical procedure has been used.

11.6.1 For the arithmetical procedure, deduct the observed distillation loss from each specified percentage evaporated in order to obtain the corresponding percentages recovered. Calculate each required temperature reading, T, in degrees Celsius, using Formula (6).

$$T = T_l + \frac{(T_h - T_l)(R - R_l)}{R_h - R_l} \tag{6}$$

where

R is the percent volume recovered corresponding to the specified percent volume evaporated;

R_h is the percent volume recovered adjacent to, and higher than R;

R_l is the percent volume recovered adjacent to, and lower than R;

T_h is the temperature reading recorded at R_h, expressed in degrees Celsius;

T_l is the temperature reading recorded at R_l, expressed in degrees Celsius.

Values obtained by the arithmetical procedure are affected by the extent to which the distillation graphs are non-linear. Intervals between successive data points shall, at any stage of the test, be no wider than the intervals indicated in 9.7. In no case shall a calculation be made that involves extrapolation.

11.6.2 For the graphical procedure, use graph paper with uniform subdivisions, plotting each thermometer reading, corrected for barometric pressure, if required (see 11.2), against its corresponding percent volume recovered. Plot the initial boiling point at 0 % volume recovered. Draw a smooth curve connecting the points. For each specified percent volume evaporated, deduct the distillation loss, in order to obtain the corresponding percent volume recovered, and take from the graph the thermometer reading which this percent volume recovered indicates. Values obtained by graphical interpolation procedures are affected by the care with which the plot is made.

NOTE See Annex D for numerical examples illustrating these arithmetical procedures.

11.7 The automated percent evaporated procedure is as follows.

Distillations carried out with automated instruments which collect temperature-volume data and store them in memory, require neither of the procedures described in 11.6. Obtain the temperature directly from the database as that closest to and within 0,1 % (V/V) of the specified percent evaporated.

12 Expression of results

12.1 Report the sampling procedure used.

12.2 Report all volume percentages to the nearest 0,5 % (V/V) or 0,1 % (V/V), and all temperature readings to the nearest 0,5 °C or 0,1 °C, appropriate to the apparatus being used. The 0,5 % (V/V) and 0,5 °C apply to the manual procedure, and the 0,1 % (V/V) and 0,1 °C apply to the automated procedure.

12.3 Report a reference to manual or automated procedure, whichever was used.

12.4 Unless specifically requested (see paragraph 5 in 11.2), temperature readings shall be corrected to 101,3 kPa barometric pressure before being reported. When such special arrangements have been made, report the observed values in accordance with the rounding rules described in 12.2, together with the laboratory barometric pressure. In these cases, also report the residue and loss as “observed”.

12.5 For group 1, or any other material with a loss exceeding 2,0 % (V/V), report the relationship between the temperature reading and the percent volume evaporated. For groups 2, 3 and 4 generally, report the relationship between the temperature reading and the percent volume recovered. Clearly identify which relationship is being used.

12.6 Report samples in groups 1 and 2 as “dried” if the default procedure described in 6.3.2 has been carried out.

12.7 Report the substitution of a low-range thermometer for the high-range thermometer, if the conditions of 9.7 (paragraph 5) have been applied.

NOTE See Annex F for examples of reports.

13 Precision (Manual Apparatus)

13.1 General

The precision as determined by statistical examination according to ISO 4259-1[3] of interlaboratory test results on matrices of petrols, kerosenes and gas oils by manual procedures is given in 13.2 and 13.3, and factors and/or precision are indicated in Tables 6 and 7. Statements of bias are given in 14.4.

To determine the precision of results other than the initial boiling point (IBP) and final boiling point (FBP), it is generally necessary to determine the rate of change of temperature at that particular point. This variable, $\Delta C/\Delta V$, is equal to the change in temperature per percent recovered or evaporated, and is calculated from Formula (7).

$$\frac{\Delta C}{\Delta V}=\frac{T_{\mathrm{U}}-T_{\mathrm{L}}}{V_{\mathrm{U}}-V_{\mathrm{L}}} \quad (7)$$

where

T_{U} is the upper temperature, expressed in degrees Celsius;

T_{L} is the lower temperature, expressed in degrees Celsius;

V_{U} is the percent volume recovered or evaporated corresponding to T_{U};

V_{L} is the percent volume recovered or evaporated corresponding to T_{L}.

Table 5 gives the data points to be used to determine the slope at specific percentages recovered or evaporated. For samples in group 1, the precision data reported are based on slope values calculated from percent evaporated data, and for samples in groups 2, 3 and 4, the precision data reported are based on slope values calculated from percent recovered data.

In the event that the final boiling point occurs prior to the 95 % (V/V) evaporated or recovered point, the slope at the final boiling point, if required, is calculated from Formula (8).

$$\frac{\Delta C}{\Delta V}=\frac{T_{\mathrm{FBP}}-T_{\mathrm{H}}}{V_{\mathrm{FBP}}-V_{\mathrm{H}}} \quad (8)$$

where

T is the temperature, expressed in degrees Celsius;

V is the volume, expressed in percent volume;

where the subscripts are

FBP is the final boiling point;

H is the highest volume reading, to the nearest 5 % (*V*/*V*), prior to the final boiling point.

Table 5 — Data points for determination of slope

Slope at, %	**IBP**	**5**	**10**	**20**	**30**	**40**	**50**	**60**	**70**	**80**	**90**	**95**	**FBP**
T_L at, %	0	0	5	10	20	30	40	50	60	70	80	90	95
T_U at, %	5	10	20	30	40	50	60	70	80	90	90	95	V_{FBP}
$V_U - V_L$	5	10	15	20	20	20	20	20	20	20	10	5	V_{FBP}−95

13.2 Repeatability

13.2.1 Group 1. The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 6 in only one case in 20.

13.2.2 Groups 2, 3 and 4. The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 7 in only one case in 20.

13.3 Reproducibility

13.3.1 Group 1. The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 6 in only one case in 20.

13.3.2 Groups 2, 3 and 4. The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 7 in only one case in 20.

Table 6 — Precision for group 1 (manual)

Percentage evaporated % (*V*/*V*)	**Repeatability** °C	**Reproducibility** °C
IBP	3,3	5,6
5	$r_1 + 0,66$	$R_1 + 1,11$
10	r_1	R_1
20	r_1	R_1
30 to 70	r_1	R_1
80	r_1	R_1
90	r_1	$R_1 - 1,22$
95	r_1	$R_1 - 0,94$
FBP	3,9	7,2

NOTE r_1, and R_1 are variables, and each is a constant function of the slope, $\Delta C/\Delta V$, at each distillation point in question. The values of these variables are calculated from the following formulae:

$r_1 = 0,864\ (\Delta C/\Delta V) + 1,214$ $R_1 = 1,736\ (\Delta C/\Delta V) + 1,994$

Table 7 — Precision for groups 2, 3 and 4 (manual)

Percentage recovered % (V/V)	Repeatability °C	Reproducibility °C
IBP	0,35 ($\Delta C/\Delta V$) + 1,0	0,93 ($\Delta C/\Delta V$) + 2,8
5 to 95	0,41 ($\Delta C/\Delta V$) + 1,0	1,33 ($\Delta C/\Delta V$) + 1,8
FBP	0,36 ($\Delta C/\Delta V$) + 0,7	0,42 ($\Delta C/\Delta V$) + 3,1
% (V/V) at T °C	0,7 + 0,92/($\Delta C/\Delta V$)	1,5 + 1,78/($\Delta C/\Delta V$)
NOTE This table has been derived from the nomograph that represented this set of precision data in all previous versions of this document, and parallel standards published by other bodies.		

14 Precision (automated apparatus)

14.1 General

The precision as determined by statistical examination according to ISO 4259-1[3] of interlaboratory test results on matrices of petrols, automotive engine ethanol fuels up to 85 % (V/V) of ethanol, aviation petrols, kerosenes, diesel fuels, diesel blends with FAME up to 30 % (V/V), heating oils, aviation turbine fuels, petroleum spirits and marine fuels by automated procedures is given in 14.2 and 14.3. The reproducibility for certain specified distillation data has been calculated from subsets of specification fuels, this information is stated in C.1.5.

To determine the precision of results (groups 1, 2, and 3), other than the initial boiling point (IBP) and final boiling point (FBP), it is generally necessary to determine the rate of change of temperature at that particular point. This variable, $\Delta C/\Delta V$, is equal to the change in temperature per percent recovered or evaporated and is calculated from Formula (7) given in 13.1.

NOTE In the 2006 and 2010 interlaboratory study the precision of materials meeting group 1, 2 and 3 conditions using automated apparatus were determined to be statistically equivalent and were combined in a single statement.

14.2 Repeatability

14.2.1 Group 1, 2 and 3. The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 8 in only one case in 20.

14.2.2 Group 4. The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 9 in only one case in 20.

14.3 Reproducibility

14.3.1 Group 1, 2 and 3. The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 8 in only one case in 20.

14.3.2 Group 4. The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 9 in only one case in 20.

Table 8 — Precision for groups 1, 2, and 3 (automated)

Percentage evaporated % (V/V)	Repeatability °C	Reproducibility °C	Valid range °C
IBP	2,7	4,7	20 to 50
5	$1,4 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$2,5 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	30 to 60
10	$0,9 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$1,9 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	35 to 65
20	$0,9 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$2,0 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	45 to 70
30	$0,8 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$1,8 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	50 to 85
40	$0,9 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$2,0 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	60 to 100
50	$1,0 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$1,9 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	65 to 115
60	$1,1 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$2,0 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	70 to 125
70	$1,5 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$2,1 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	105 to 140
80	$1,1 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$2,0 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	120 to 155
90	$1,8 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$2,8 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	145 to 175
95	$2,0 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	$3,6 + 2,8(0,43(\Delta C/\Delta V) + 0,24)$	155 to 195
FBP	3,3	7,1	175 to 260
Where ($\Delta C/\Delta V$) is the slope or rate of change of temperature in degrees Celsius calculated from 13.1 Formula (7).			

Table 9 — Precision for group 4 (automated)

Percentage recovered % (V/V)	Repeatability °C	Reproducibility °C	Valid range °C
IBP	0,018 T	0,055 T	145 to 220
5	0,010 9 T	0,03 T	160 to 255
10	0,009 4 T	0,022 T	160 to 265
20	0,007 28 T	0,020 8 T	175 to 275
30	0,005 82 T	0,016 5 T	185 to 285
40	0,005 T	0,014 T	195 to 290
50	1,0	3,0	170 to 295
60	0,003 57 T	0,011 7 T	220 to 305
70	0,003 55 T	0,012 5 T	230 to 315
80	0,003 77 T	0,013 6 T	240 to 325
90	0,004 1 T	0,015 T	180 to 340
95	0,013 18 (T – 140)	0,041 05 (T – 140)	260 to 360
FBP	2,2	7,1	195 to 365
Where T is the temperature at the percentage recovered within valid range prescribed.			

14.4 Bias

14.4.1 Bias

Since there is no accepted reference material suitable for determining the bias for the procedure in these test methods, bias has not been determined.

14.4.2 Relative bias

An interlaboratory study conducted in 2003 using manual and automated apparatus has concluded that there is no statistical evidence to suggest that there is a bias between manual and automated results. See ASTM Research Report RR:D02-1580[2].

15 Test report

The test report shall contain at least the following information:

a) a reference to this document;

b) the type and complete identification of the product tested;

c) the result of the test (see Clause 12);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

NOTE See Annex F for examples of reports.

Annex A
(normative)

Thermometer specifications

A.1 General

Table A.1 gives the specifications for the mercury-in-glass thermometers specified in 5.8.1.

Table A.1 — Thermometer specifications

	Low-range	**High-range**
Range, °C	−2 to 300	−2 to 400
Subdivisions, °C	1	1
Immersion, mm	Total	Total
Overall length, mm	381 to 391	381 to 391
Stem diameter, mm	6 to 7	6 to 7
Bulb shape	Cylindrical	Cylindrical
Bulb length, mm	10 to 15	10 to 15
Bulb diameter, mm	5 to 6	5 to 6
Distance of bottom of bulb to		
— 0 °C line, mm	100 to 110	25 to 45
— 300 °C line, mm	333 to 354	—
— 400 °C line, mm	—	333 to 354
Longer lines at each, °C	5	5
Numbering at each, °C	10	10
Scale error, maximum °C	0,5 up to 300	1,0 up to 370
Maximum line width, mm	0,23	0,23
Expansion chamber	Required[a]	—
Heat stability	See cautionary note in 5.8.1	See cautionary note in 5.8.1

NOTE Thermometers ASTM 7C/IP 5C (low-range) and ASTM 8C/IP 6C (high-range) conform to the above requirements.

[a] An expansion chamber is provided for relief of gas pressure to avoid distortion of the bulb at higher temperatures. It is not for the purpose of joining mercury separations. Under no circumstances shall the thermometer be heated above its highest temperature reading.

Annex B
(normative)

Determination of temperature-sensor lag times

B.1 Temperature-sensor lag times

B.1.1 General

The response time of an electronic temperature-measurement device is inherently more rapid than that of a mercury-in-glass thermometer. The assembly in general use, consisting of the sensor and its casing, and/or an electronic system and its associated software, is designed such that the device will simulate the temperature lag of the mercury-in-glass thermometer.

NOTE 1 Annex E contains information on typical corrections.

NOTE 2 This Annex is under revision. Verification of the response time of the electronic thermometer by testing synthetic blends as reference fuels instead of direct comparison of the electronic thermometer with the mercury in glass thermometer, is being executed by ISO.

NOTE 3 The use and reliability of automated equipment with electronic temperature measuring devices has been accepted for application by industry for over ten years. Manufacturers have developed reliable response time algorithms (refer to manufacturer's instructions for calibration of temperature measurement systems).

B.1.2 Determination

B.1.2.1 Select a sample of material with known temperature properties representative of the products normally analysed, not a pure compound, nor any narrow range product, nor a synthetic blend of less than five compounds. Ensure that the difference between 5 % (*V*/*V*) recovered and 95 % (*V*/*V*) recovered is at least 100 °C.

NOTE The certified reference fluids (8.2.4) are suitable.

B.1.2.2 Carry out an automated distillation by the procedure described in this document, with the electronic temperature-measurement device in place.

B.1.2.3 Replace the electronic measurement device by the appropriate mercury-in-glass thermometer and repeat the distillation, manually recording the thermometer readings at each percent recovery appropriate to the product as described in 9.7. See NOTES.

B.1.2.4 Calculate the difference in value at each observed slope ($\Delta C/\Delta V$) by both procedures. This difference at any point shall be equal or less than the repeatability of the test method at that point.

B.1.2.5 If the difference is larger than the test method repeatability, adjust the electronics of the electronic measurement device, and repeat until a satisfactory agreement is obtained.

Annex C
(normative)

Determination of specified distillation data

C.1 Specified distillation data

C.1.1 General

Many specifications for petrol, kerosene, and gas oil require specific percentages evaporated or recovered, either as maxima, minima or ranges, at specified temperature readings. These are frequently designated the terms "Exxx" or "Rxxx".

NOTE Typical specified temperatures are E70, E100 and E180 for petrols, R200 for kerosenes, and R250 and R350 for gas oils.

C.1.2 Procedure

C.1.2.1 From the observed barometric pressure, calculate the temperature readings equivalent to those desired, using the procedure given in 11.2.

C.1.2.2 Carry out a distillation as described in Clause 9 or Clause 10, but in the range approximately 10 °C below and 10 °C above the desired temperature reading calculated in C.1.2.1. Record the temperature readings at intervals of 1 % (*V*/*V*) or smaller. Observe and record the amount of distillate recovered closest to the exact temperature reading required.

If the intent of the distillation is only to determine Rxxx, the distillation may be discontinued after a minimum of 2 ml of distillate has been recovered above the highest specified temperature.

C.1.3 Calculation

To determine the value of Exxx, add the observed loss to the observed volume recovered at or around the required corrected temperature reading. Obtain the value at the exact temperature, using either the arithmetical or graphical procedures described in 11.6.

C.1.4 Precision

The statistical determination of precision of specified distillation data for manual apparatus and specified distillation points for automated apparatus, except for those in C.1.5, has not been directly carried out on the basis of Exxx or Rxxx. However, this can be calculated on the basis that it is equivalent to the precision of temperature measurement divided by the rate of change of temperature measurement versus the volume recovered or evaporated. This calculation becomes less valid at high slope values. Formulae (C.1) and (C.2) describe this relationship:

$$r_V = \frac{r}{\Delta C / \Delta V} \tag{C.1}$$

$$R_\text{V} = \frac{R}{\Delta C / \Delta V} \tag{C.2}$$