
**Petroleum products — Determination of
distillation characteristics at atmospheric
pressure**

*Produits pétroliers — Détermination des caractéristiques de distillation à
pression atmosphérique*



Reference number
ISO 3405:2011(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 3405 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This fourth edition cancels and replaces the third edition (ISO 3405:2000), which has been technically revised. It has been aligned with ASTM D86¹⁾.

1) ASTM D86, *Standard Method for Distillation of Petroleum Products at Atmospheric Pressure*.

Introduction

The distillation (volatility) characteristics of hydrocarbons 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 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 (volatile organic compounds or VOCs).

Petroleum products — Determination of distillation characteristics at atmospheric pressure

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a laboratory test method, utilizing either manual or automated equipment, for determining the distillation characteristics of light and middle distillates derived from petroleum and having initial boiling points above 0 °C and end points below approximately 400 °C.

Light distillates are typically automotive engine petrols, automotive engine petrols with up to 10 % (V/V) ethanol and aviation petrols. Middle distillates are aviation turbine fuels, kerosenes, diesel, diesel with up to 20 % (V/V) FAME (fatty acid methylesters), burner fuels and marine fuels that have no appreciable quantities of residua.

NOTE For the purposes of this International Standard, “% (V/V)” is used to represent the volume fraction of a material.

2 Normative references

The following referenced documents are indispensable for the application 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 4259, *Petroleum products — Determination and application of precision data in relation to methods of test*

ISO 4788:2005, *Laboratory glassware — Graduated measuring cylinders*

3 Terms and definitions

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

3.1 decomposition point

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

NOTE 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 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. 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 Clause 13 or 14.

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

NOTE 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 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
front-end loss
amount of uncondensed material lost in the initial stages of the distillation, equal to 100 % minus the total recovery

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 in connection with a simultaneous temperature reading

NOTE It is expressed as a percentage of the charge volume.

3.9
percent recovery
maximum percent recovered, as observed in accordance with this International Standard

NOTE See 9.10.

3.10
percent residue
volume of residue measured in accordance with this International Standard

NOTE 1 See 9.11.

NOTE 2 It is 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 this International Standard

NOTE See 10.1.

3.12**thermometer reading**

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

3.13**temperature reading**

thermometer reading or other temperature measurement device reading which is corrected to 101,3 kPa barometric 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 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 with the thermometer 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 times of the systems involved

4 Principle

The sample is assigned to one of four groups, based on its composition and expected volatility characteristics, with 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 this clause, automated apparatus also are equipped with a system for measuring and automatically recording the vapour temperature and the associated recovered volume in the receiving cylinder.

Automated equipment manufactured in or after the year 1999 shall be equipped with a device for automatically shutting down power to the unit and for spraying an inert gas or vapour in the chamber where the distillation flask is mounted in the event of fire.

NOTE Some causes of fire 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.

NOTE For tests specifying the dry point, specially selected flasks with bottoms and walls of uniform thickness are desirable.

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 in Figures 1 and 2 and to satisfy the precision criteria given in Clause 13 or 14.

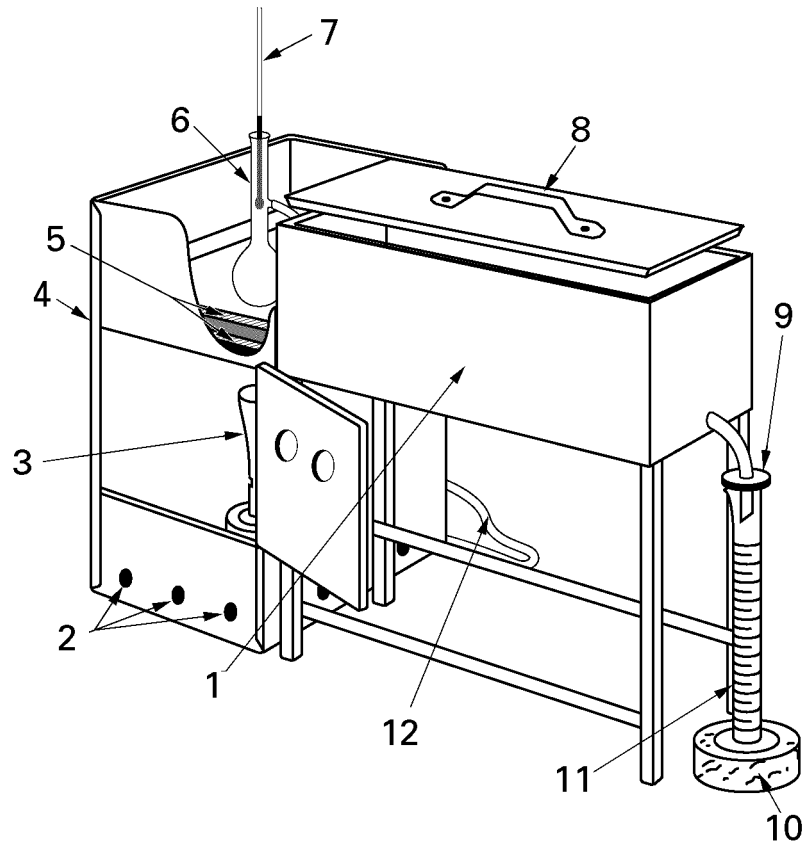
5.3.2 The condenser shall be made of seamless non-corrosive metal tubing, 560 mm \pm 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0,8 mm to 0,9 mm.

NOTE Brass or stainless steel are suitable materials.

5.3.3 The condenser shall be set so that a length of 393 mm \pm 3 mm of the tube is in contact with the cooling medium, with 50 mm \pm 3 mm outside the cooling bath at the upper end, and 114 mm \pm 3 mm outside at the lower end. The portion of tube projecting at the upper end shall be set at an angle of 75° 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° \pm 1° with respect to the horizontal, and no 100 mm section shall have a gradient outside the range of 15° \pm 3°. 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 may be accomplished either by using a drip deflector 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.

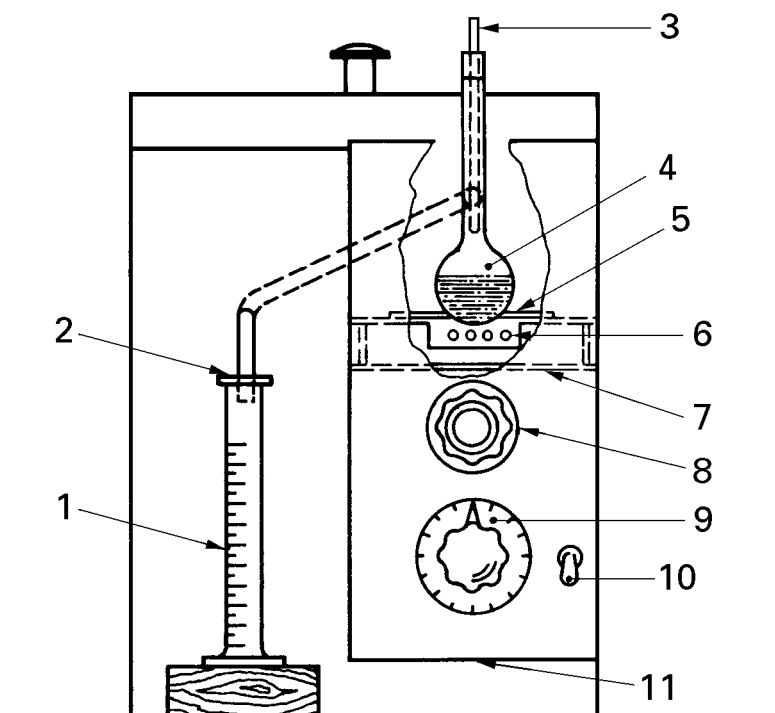
5.3.4 The volume and design of the cooling bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single cooling bath may be used for several condenser tubes.



Key

- 1 cooling bath
- 2 air vents
- 3 burner
- 4 shield
- 5 heat-resistant boards
- 6 distillation flask
- 7 thermometer
- 8 bath cover
- 9 blotting paper
- 10 support
- 11 graduated cylinder
- 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
- 7 flask-support platform
- 8 flask-adjusting knob
- 9 indicating dial
- 10 switch
- 11 open-bottom shield
- 12 cooling bath
- 13 condenser tube
- 14 shield

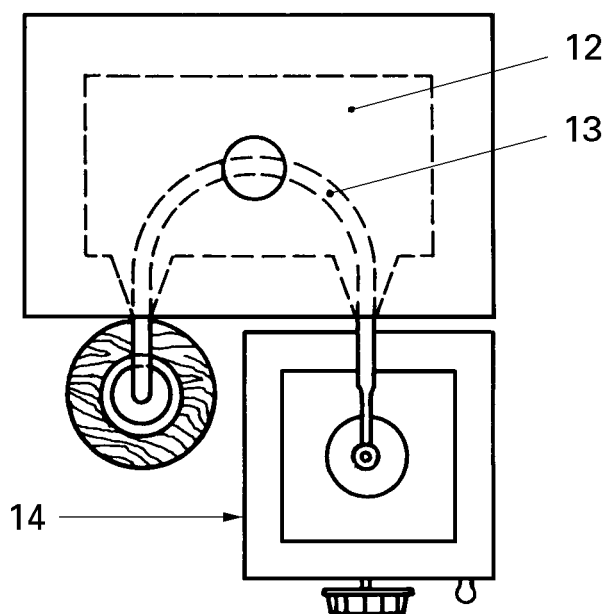
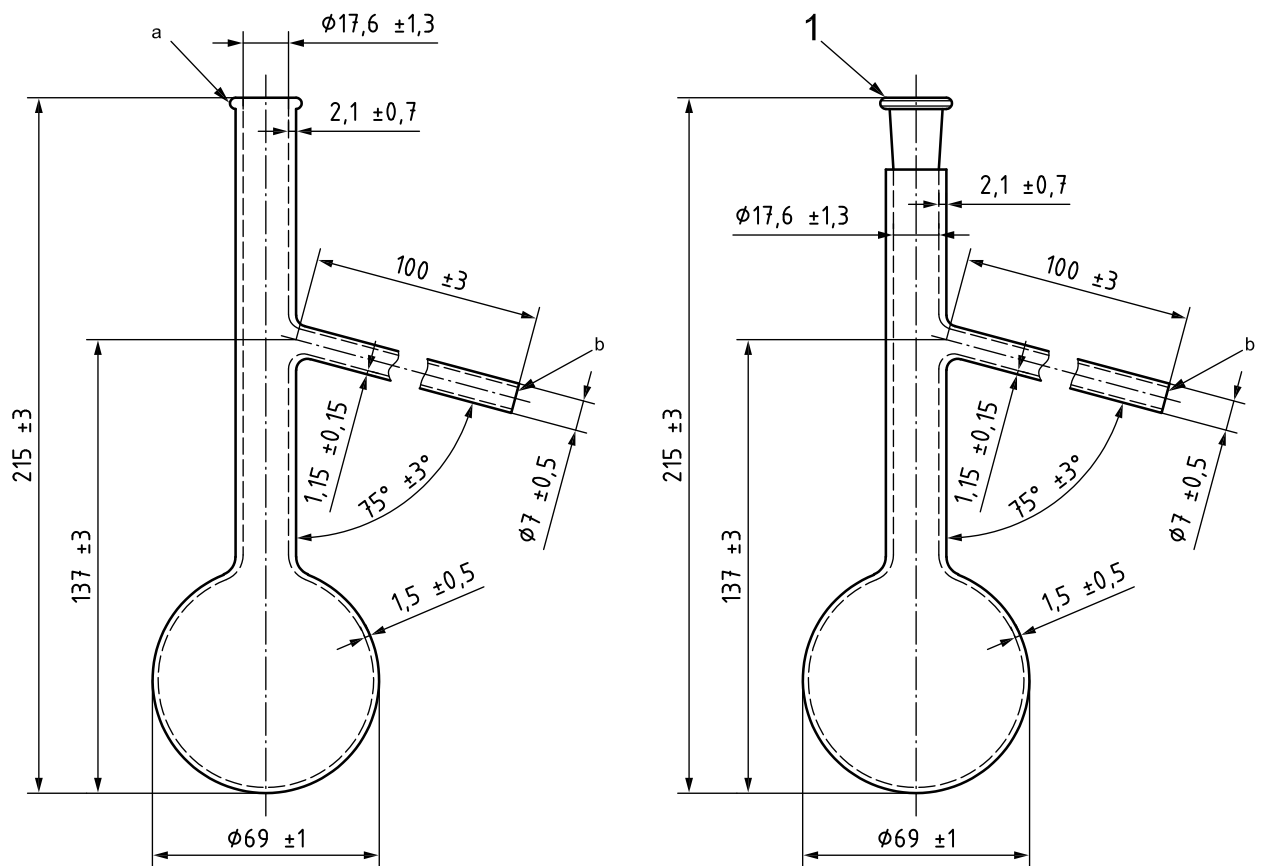


Figure 2 — Apparatus assembly using an electric heater

Dimensions in millimetres



Key

- 1 19/22 or 19/26
- a Reinforcing bead.
- b Fire-polished.

Figure 3 — 125 ml flasks — Alternative neck designs

Dimensions in millimetres

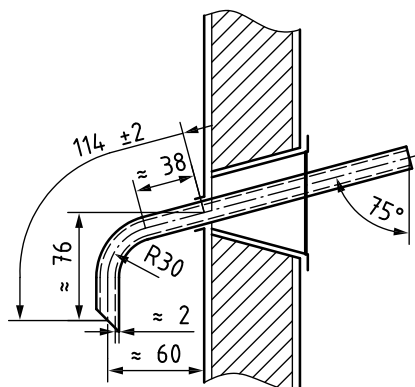


Figure 4 — Lower end of condenser tube

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

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

NOTE 1 A typical shield for a unit fitted with a gas burner would be 400 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).

5.5 Heat source

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, 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

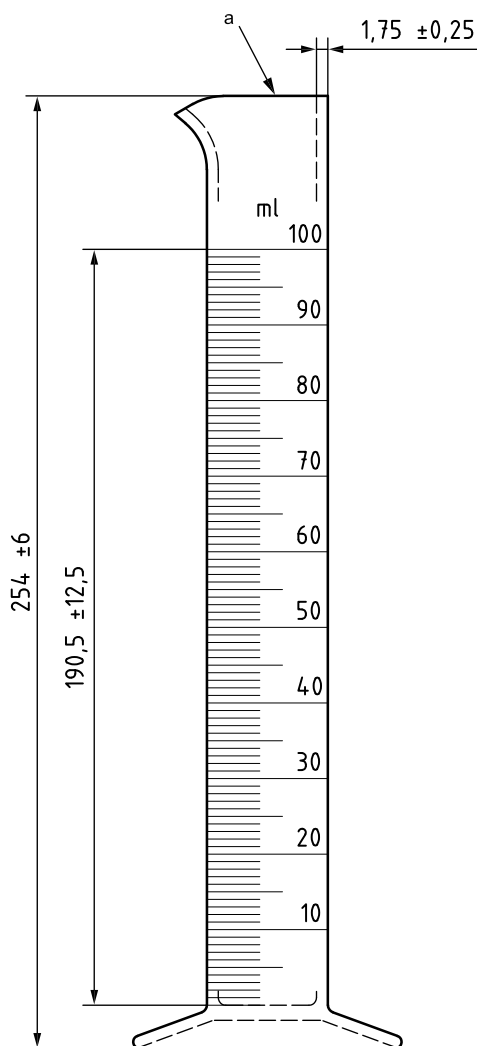
5.7.1 Receiving cylinder, of 100 ml capacity, generally in accordance with ISO 4788. It shall be graduated at intervals of 1 ml beginning at least at the 5 ml mark and have a graduation at the 100 ml mark. The shape of the base shall be such that the receiver does not topple when placed empty on a surface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

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 immersed to above the 100 ml graduation line in a cooling liquid contained in a cooling bath, such as a tall-form beaker of clear glass or transparent plastic, or placed in a thermostatically controlled air-circulation chamber.

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

Dimensions in millimetres



a Fire-polished.

Figure 5 — 100 ml receiving cylinder (tolerance $\pm 1,0$ ml)

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 $28\text{ }^{\circ}\text{C}$ above the temperature indicated, and at an indicated temperature of $371\text{ }^{\circ}\text{C}$, the temperature of the bulb is approaching a critical range in the glass. It is thus strongly recommended that distillation temperature readings above $371\text{ }^{\circ}\text{C}$ be avoided, but in those cases where thermometers have been exposed to observed temperature readings above $371\text{ }^{\circ}\text{C}$, 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 provision 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 a mercury-in-glass thermometer.

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

A means of determining the difference in lag time between an electronic temperature-measurement system and a mercury-in-glass thermometer is given in Annex B.

5.9 Centring device

The temperature sensor shall be fitted through a snug-fitting device designed to mechanically centre 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.

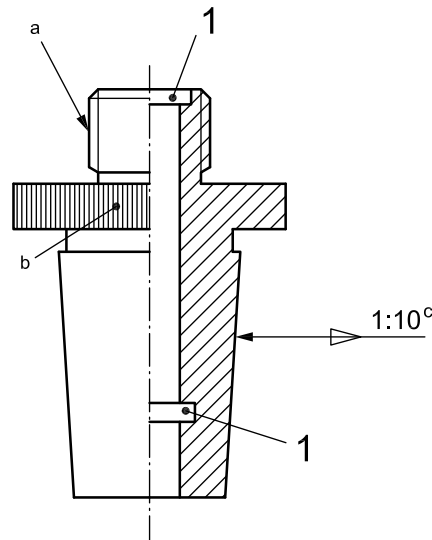
Centring devices different from those shown in Figures 6 and 7 are also acceptable provided that they position and hold the temperature sensor in the middle of the neck of the distillation flask.

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

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 precorrected to give sea level pressures.

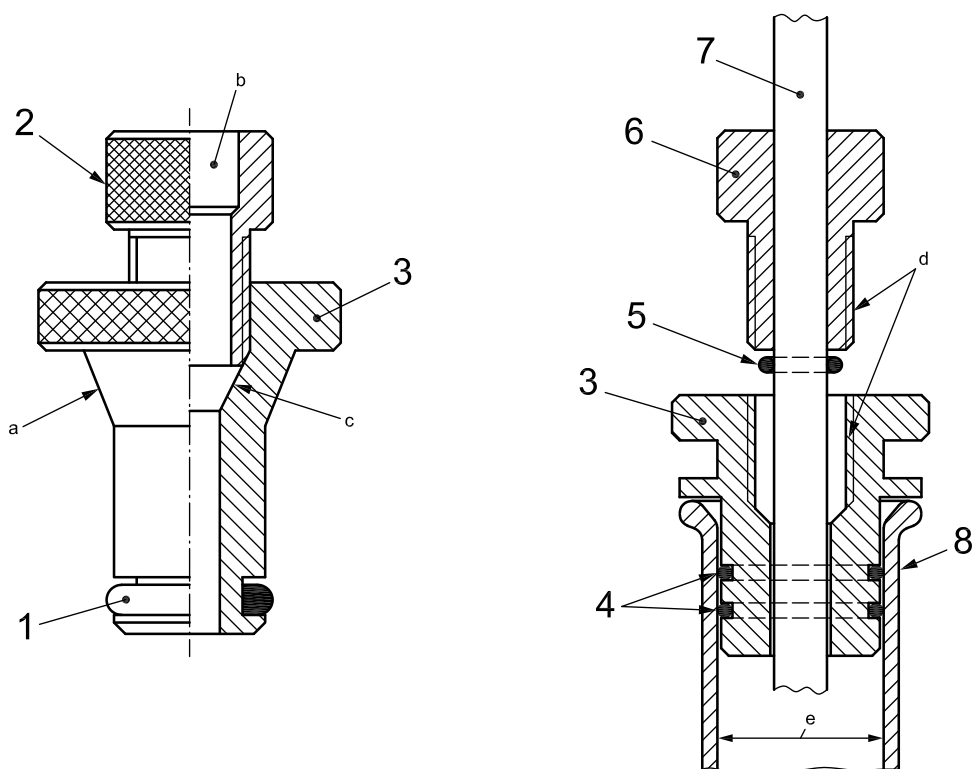
NOTE The barometer should ideally be located in the room in which the distillation is carried out.



Key

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

Figure 6 — PTFE (polytetrafluoroethylene) centring device for ground-glass joint



Key

- | | | | |
|---|--|---|-----------------------------|
| 1 | single O-ring: Viton ²⁾ or perfluoro elastomer | 5 | compression O-ring |
| 2 | compression nut (PTFE) | 6 | compression nut |
| 3 | PTFE body | 7 | thermometer or Pt 100 probe |
| 4 | double O-rings: Viton ²⁾ or perfluoro elastomer | 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-bored.

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.

2) Viton® is the trade name of a product supplied by DuPont. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

6.2 Sample maintenance prior to testing

6.2.1 General

Unless otherwise specified, sampling shall be carried out in accordance with 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. 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 so 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 of 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
Reid vapour pressure, kPa	≥ 65,5	< 65,5	< 65,5	< 65,5
Distillation				
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
See subclause:	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 are available for storage below 10 °C, the sample may be stored at 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 the fact that the sample has been dried by desiccant.

NOTE Data from a round-robin exercise show that suspended water in hazy samples in groups 1 and 2 can 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 sulfate 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 required 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 temperatures.

7.2 Make any necessary provisions so that the temperature of the cooling bath and receiving cylinder are 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 volume 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 is 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 PTFE centring device to facilitate dismantling after use.

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 in accordance with Clause 9 or 10, depending on whether the procedure is manual or automatic.

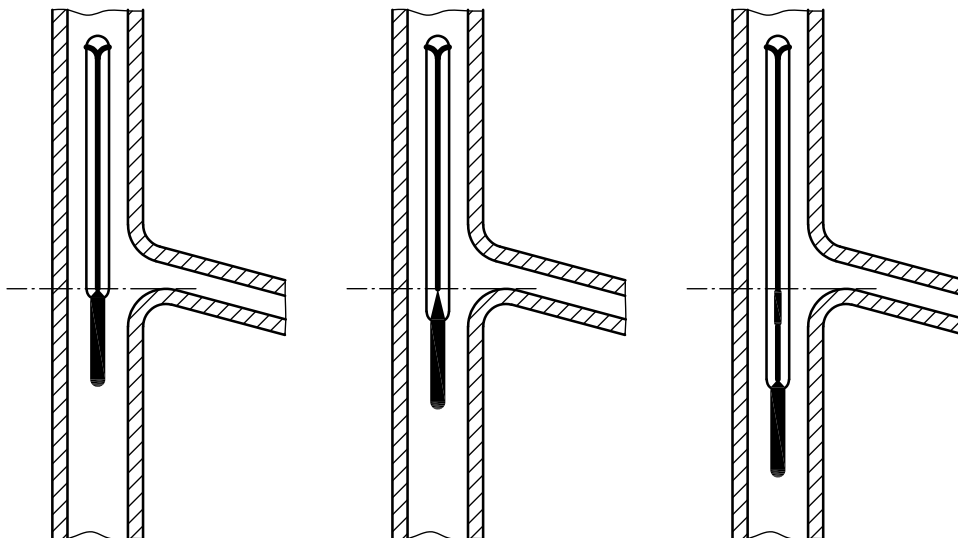


Figure 8 — Position of thermometer in distillation flask neck

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 Temperature-measurement devices using other than the specified mercury-in-glass thermometers shall exhibit the same temperature lag, emergent-stem effect and accuracy as the equivalent mercury-in-glass thermometer. Verification of the calibration of these temperature sensors shall be carried out at intervals of not more than six 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 also be verified by one of the procedures described in 8.2.2 or 8.2.3.

8.2.2 Distil reagent grade toluene in accordance with ISO 918 and compare the 50 % (V/V) recovered temperature with that shown by the above-mentioned mercury-in-glass thermometer when carrying out a manual test under the same conditions. 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 for determining this difference in lag time.

To verify the response at elevated temperatures, use hexadecane.

NOTE Toluene is shown in reference manuals as boiling at 110,6 °C, and hexadecane at 287,0 °C, under the conditions specified in ISO 918, which uses a partial immersion thermometer. Because this International Standard 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 here:

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

8.2.3 Distil a certified reference fluid of a specific product group, under the manual conditions given in this International Standard, 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.

NOTE 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 specified 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 (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 used, immediately move the receiving cylinder so that the tip of the condenser touches the inner wall.

Table 3 — Test conditions

Group number	1	2	3	4
Temperature of condenser bath, °C	0 to 1	0 to 4	0 to 4	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 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.

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

NOTE When testing group 1 or group 2 materials, the condensate may 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 may last for 10 s to 20 s before the temperature recovers and the condensate starts flowing again. This point may be 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 according to 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 test results as required by the particular specification or as previously established for the material under test.

NOTE These observed data may include thermometer readings at specified percents recovered or percents 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 kerosenes, 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 indicate this. If, by agreement, the obscured readings are waived, the test report shall also indicate this (see Clause 15).

When required to report the thermometer reading at a specified percent evaporated or percent recovered for a sample which has a rapidly changing distillation curve slope in the region of the specified percent evaporated or percent recovered reading, record thermometer readings at every 1 % (*V/V*) recovered. The slope is considered to be rapidly changing if the change in the slope, *C*, of the data points described above for groups 1, 2, 3 and 4 in that particular area is greater than 0,6, as calculated from Equation (1):

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

where

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

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

V_2 is the volume percent recorded in question;

V_3 is the volume percent recorded following the one in question.

9.8 When the residual liquid in the distillation flask is approximately 5 ml, make a final heat adjustment. To determine when the residual liquid in the distillation flask is approximately 5 ml, subtract the estimated loss from 93,5 ml to 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.

NOTE 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 can 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 the 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 the percent residue and percent loss, and omit the procedure according to 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 the percent residue.

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, refill 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 in accordance with Annex C.

10 Procedure — Automated apparatus

10.1 Apply heat to the distillation flask (5.2) and its 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.

Due to the configuration of the distillation flask and the conditions of the test, the vapour and liquid around the temperature sensor (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.

NOTE When testing group 1 or group 2 materials, the condensate may 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 may last for 10 s to 20 s, before the temperature recovers and the condensate starts flowing again. This point may be referred to as the “hesitation point”.

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

10.6 If a decomposition point is observed, discontinue the heating and proceed according to 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.

NOTE These data may include vapour temperature readings at specified percents recovered, or percents 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 a final heat adjustment. To determine when the residual liquid in the distillation flask is approximately 5 ml, subtract the estimated loss from 93,5 ml to 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.

NOTE 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 can 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 the 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 the percent residue.

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 (see 9.10 or 10.10) and the percent residue (see 9.11 or 10.11). Deduct the percent total recovery from 100 to obtain the percent loss.

11.2 Correct temperature readings to a pressure of 101,3 kPa. Obtain the correction, T_C , to be applied to each temperature reading by means of the Sydney Young equation [Equation (2)] or by reference to Table 4:

$$T_C = 0,0009(101,3 - p_k)(273 + T) \tag{2}$$

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.

NOTE 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 is required to 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 a pressure of 101,3 kPa when the temperature readings are corrected to 101,3 kPa. Calculate the corrected loss, L_c , in percent volume, using Equation (3):

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

where

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

p is the observed barometric pressure, expressed in kilopascals.

NOTE Equation (3) has been derived from experimental data referenced in ASTM D86-96 and later editions.

11.4 Calculate the corresponding corrected percent recovery, R_c , in percent volume, from Equation (4):

$$R_c = R + (L - L_c) \quad (4)$$

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 percents evaporated at specified temperature readings, add the observed percent loss to each of the observed percents recovered at the specified temperature readings, and report these results as the respective percents evaporated, as seen in Equation (5):

$$P_e = P_r + L \quad (5)$$

where

P_e is the percent volume evaporated;

P_r is the percent volume recovered;

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

11.6 Manual percent evaporated procedure: 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 Equation (6):

$$T = T_1 + \frac{(T_h - T_1)(R - R_l)}{R_h - R_l} \quad (6)$$

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 specified 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 Automated percent evaporated procedure: distillations carried out with automated instruments which collect temperature-volume data and store them in memory require neither of the procedures specified 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) for the manual procedure or to the nearest 0,1 % (V/V) for the automated procedure, and record all temperature readings to the nearest 0,5 °C for the manual procedure or to the nearest 0,1 °C for the automated procedure, as appropriate for the apparatus being used.

12.3 Report whether the manual or automated procedure has been used.

12.4 Unless specifically requested (see Note in 11.2), temperature readings shall be corrected to a barometric pressure of 101,3 kPa before being reported. When such special arrangements have been made, report the observed values in accordance with the rounding rules given 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 evaporated. For groups 2, 3 and 4 generally, report the relationship between the temperature reading and the percent recovered. Clearly identify which relationship is being used.

12.6 Report samples in groups 1 and 2 as “dried” if the default procedure according to 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 4) have been applied.

NOTE See Annex F for examples of test reports.

13 Precision — Manual apparatus

13.1 General

The precision as determined by statistical examination according to ISO 4259 of interlaboratory test results on matrices of petrols, kerosines and gas oils is obtained by means of the manual procedures given in 13.2 and 13.3, with factors and/or precision are indicated in Tables 6 and 7, while 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 Equation (7).

$$\frac{\Delta C}{\Delta V} = \frac{T_U - T_L}{V_U - V_L} \quad (7)$$

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 Equation (8):

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

where

T is the temperature, expressed in degrees Celsius;

V is the volume, expressed in percent volume;

FBP denotes the final boiling point temperature or volume;

H denotes the highest volume or temperature reading, to the nearest 5 % (V/V) or 0,5 °C, prior to the FBP.

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	$V_{\text{FBP}} - 95$

13.2 Repeatability

13.2.1 Group 1: the difference between two test results obtained by the same operator, using the same apparatus under constant operating conditions on identical test material, would, in the long run, and with normal and correct application 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, using the same apparatus under constant operating conditions on identical test material, would, in the long run and with normal and correct application 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, and with normal and correct application 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, and with 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

Each of the variables r_1 and R_1 is a constant function of the slope, $\Delta C/\Delta V$, at each distillation point in question, with values calculated from: $r_1 = 0,864 (\Delta C/\Delta V) + 1,214$; $R_1 = 1,736 (\Delta C/\Delta V) + 1,994$.

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	$1,00 (\Delta C/\Delta V) + 0,5$	$1,89 (\Delta C/\Delta V) + 1,3$

NOTE This table is derived from the nomograph representing this set of precision data published in all previous editions of this International Standard as well as in other, parallel, standards.

14 Precision — Automated apparatus

14.1 General

The precision as determined by statistical examination according to ISO 4259 of interlaboratory test results on matrices of petrols, petrols with oxygenates up to 10 %, ethanol or MTBE, aviation petrols, kerosines, diesels, diesels with FAME up to 20 %, heating oils, aviation turbine fuels, petroleum spirits and marine fuel 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.

NOTE 1 In the 2006 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.

NOTE 2 An interlaboratory study is planned to address concerns that many laboratories testing gasoline are not able to meet the precision for 50 % (V/V) evaporated temperature. The results may lead to a future revision of this International Standard.

14.2 Repeatability

14.2.1 Group 1, 2 and 3: the difference between two test results obtained by the same operator using the same apparatus under constant operating conditions on identical test material would, in the long run, and with normal and correct application 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, and with normal and correct application 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, and with 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, and with normal and correct application 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	0,029 5($E + 51,19$)	0,059 5($E + 51,19$)	20–70
10	1,33	3,20	35–95
50	0,74	1,88	65–220
90	0,007 55($E + 59,77$)	0,019($E + 59,77$)	110–245
FBP	3,33	6,78	135–260

E is the temperature at the percentage evaporated within the prescribed valid range.

Table 9 — Precision for group 4 — Automated

Percentage evaporated % (V/V)	Repeatability °C	Reproducibility °C	Valid range °C
IBP	0,018 <i>T</i>	0,055 <i>T</i>	145–220
10	0,009 4 <i>T</i>	0,022 <i>T</i>	160–265
50	0,94	2,97	170–295
90	0,004 1 <i>T</i>	0,015 <i>T</i>	180–340
95	0,015 15(<i>T</i> – 140)	0,042 27(<i>T</i> – 140)	260–340
FBP	2,2	7,1	195–365

T is the temperature at the percentage recovered within the prescribed valid range.

14.4 Bias

14.4.1 General

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.

15 Test report

The test report shall contain at least the following information:

- a) reference to this International Standard;
- 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 test 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 to 300	1,0 to 370
Maximum line width, mm	0,23	0,23
Expansion chamber	Required ^a	—
Heat stability	See caution, 5.8.1	See caution, 5.8.1
NOTE Thermometers ASTM 7C/IP 5C (low-range) and ASTM 8C/IP 6C (high-range) conform to these 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 Annex E gives information on typical corrections.

B.1.2 Determination

Select a sample of material representative of the products normally analysed: not a pure compound, narrow range product or a synthetic blend of less than six 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 (see 8.2.3) are suitable.

B.1.2.1 Carry out an automated distillation by the procedure described in this International Standard, with the electronic temperature-measurement device in place.

B.1.2.2 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 in accordance with 9.7.

B.1.2.3 Calculate the difference in value at each observed slope ($\Delta C/\Delta V$) by both procedures. This difference at any point shall be less than or equal to the repeatability of the test method at that point.

B.1.2.4 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 percents evaporated or recovered, either as maxima, minima or ranges, at specified temperature readings. These are frequently designated by the terms "Exxx" or "Rxxx".

EXAMPLE Typical specified temperatures are E70, E100 and E180 for petrols, R200 for kerosene, 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 in accordance with Clause 9, but in the range approximately 10 °C below and 10 °C above the desired temperature reading calculated according to 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. Equations (C.1) and (C.2) describe this relationship:

$$r_V = \frac{r}{\Delta C / \Delta V} \quad (\text{C.1})$$

$$R_V = \frac{R}{\Delta C / \Delta V} \quad (\text{C.2})$$

where

r_V is the repeatability of the volume percent evaporated or recovered;

r is the repeatability of the temperature at the specified temperature, obtained from 13.2;

R_V is the reproducibility of the volume percent evaporated or recovered;

R is the reproducibility of the temperature at the specified temperature, obtained from 13.3.

C.1.5 Reproducibility for certain specified fuels — Automated apparatus

An interlaboratory study was conducted in 2006, containing specification petrol and diesel samples analysed by automated apparatus. From this data, the following reproducibility statements were estimated for the specification temperatures and percentages.

Reproducibility of diesel percent volume recovered at specified temperatures

For R200, R250 and R350:

$$R = 2,7 \% (V/V)$$

where Rxxx is the percent recovered at specified temperature “xxx”, in degrees Celsius.

Reproducibility of petrol percent volume evaporated at specified temperatures

For E70: $R = 2,7 \% (V/V)$

For E100: $R = 2,2 \% (V/V)$

For E150: $R = 1,3 \% (V/V)$

For E180: $R = 1,1 \% (V/V)$

where Exxx is the percent evaporated at specified temperature “xxx”, in degrees Celsius.

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Annex D (informative)

Examples of data calculations

D.1 Example distillation

D.1.1 General

The example distillation described in this annex concerns a group 1 product, yielding a recovery of 96,7 % (V/V), a residue of 0,8 % (V/V), and a loss of 2,5 % (V/V) under an observed barometric pressure of 98,6 kPa. Table D.1 gives the thermometer readings before and after corrections.

D.1.2 Thermometer reading correction

Each thermometer reading is corrected using Equation (2). The correction becomes:

$$T_C = 0,000\ 9(101,3 - 98,6)(273 + t) \quad (D.1)$$

where t is the observed thermometer reading, expressed in degrees Celsius.

D.1.3 Loss correction

The loss is corrected by Equation (3). The corrected loss becomes:

$$\begin{aligned} L_C &= 0,5 + \frac{2,5 - 0,5}{1 + (101,3 - 98,6)/8,00} \\ &= 0,5 + \frac{2,0}{1,34} \\ &= 2,0 \text{ \% (V/V)} \end{aligned} \quad (D.2)$$

D.1.4 Recovery correction

The corrected recovery according to Equation (4) becomes:

$$\begin{aligned} R_C &= 96,7 + (2,5 - 2,0) \\ &= 97,2 \text{ \% (V/V)} \end{aligned} \quad (D.3)$$

Table D.1 — Observed and corrected illustrative data

	Observed data	Corrected data
Initial boiling point (IBP), °C	27,1	27,8
2 % (V/V) recovered at, °C	32,8	33,5
5 % (V/V) recovered at, °C	38,7	39,5
10 % (V/V) recovered at, °C	48,5	49,3
20 % (V/V) recovered at, °C	61,9	62,7
30 % (V/V) recovered at, °C	75,6	76,4
40 % (V/V) recovered at, °C	91,4	92,3
50 % (V/V) recovered at, °C	104,5	105,4
60 % (V/V) recovered at, °C	118,7	119,7
70 % (V/V) recovered at, °C	131,3	132,3
80 % (V/V) recovered at, °C	148,8	149,8
90 % (V/V) recovered at, °C	164,2	165,3
95 % (V/V) recovered at, °C	183,5	184,6
Final boiling point (FBP), °C	206,0	207,2
Recovery, % (V/V)	96,7	97,2
Residue, % (V/V)	0,8	0,8
Loss, % (V/V)	2,5	2,0

D.2 Conversion to percentages evaporated

To convert thermometer readings (corrected) at specified percentages recovered to corresponding percentages evaporated, use Equation (6).

An example derivation for T_{50E} is shown in Equation (D.4), using values from Table D.1:

$$T_{50E} = 92,3 + (119,7 - 92,3)(47,5 - 40)/(60 - 40) \quad (D.4)$$

$$T_{50E} = 102,6 \text{ °C}$$

Other examples are

$$5 \text{ % (V/V) recovered at [7,5 % (V/V) evaporated at]} = 39,5 \text{ °C}$$

$$5 \text{ % (V/V) evaporated at} = 34,4 \text{ °C}$$

$$20 \text{ % (V/V) recovered at [22,5 % (V/V) evaporated at]} = 62,7 \text{ °C}$$

$$20 \text{ % (V/V) evaporated at} = 59,5 \text{ °C}$$

$$90 \text{ % (V/V) recovered at [92,5 % (V/V) evaporated at]} = 165,3 \text{ °C}$$

$$90 \text{ % (V/V) evaporated at} = 161,4 \text{ °C}$$

NOTE 1 The initial boiling point is not corrected, although in theory it should be. Because of the variability of non-recoverable light materials, the slope of the distillation curve at the initial boiling point is not a reliable instrument for extrapolation to 0 % (V/V) evaporated.

NOTE 2 If the slope of the distillation curve changes significantly between the two points considered, then the above estimates may be in error. In those cases, the graphical procedure described in 11.6 is to be preferred. This is particularly true at the higher percentages recovered.

D.3 Determination of percent evaporated at specified temperatures

D.3.1 Determination

The percent evaporated can be easily calculated using the arithmetical or graphical procedures given in 11.6. To calculate directly from the observed thermometer readings, correct the values for the specified temperatures for barometric pressure and follow a simple interpolation.

Examples for the illustrated distillation are

70,8 °C for 70 °C

100,9 °C for 100 °C

181,1 °C for 180 °C

D.3.2 Precision

To calculate the precision for the above three temperatures, first obtain the $\Delta C/\Delta V$ for each, using Equation (7) and Table D.1. This gives the following:

For E70: $0,1(E_{32,5} - E_{22,5}) = 0,1(76,4 - 62,7) = 1,37$

For E100: $0,1(E_{52,5} - E_{42,5}) = 0,1(105,4 - 92,3) = 1,31$

For E180: $0,2(E_{97,5} - E_{92,5}) = 0,2(184,6 - 165,3) = 3,86$

Obtain the precision from Table 6. Use approximately 30 %, 50 % and 95 % values. These calculate as:

$r_{170} = 2,40$ $R_{170} = 4,37$ $r_{1100} = 2,37$ $R_{1100} = 4,32$ $r_{1180} = 4,55$ $R_{1180} = 8,69$

Using Equations (C.1) and (C.2), the precision becomes:

$rE_{70} = 2,40/1,37 = 1,75 \% (V/V)$ $RE_{70} = 4,37/1,37 = 3,19 \% (V/V)$

$rE_{100} = 2,37/1,31 = 1,81 \% (V/V)$ $RE_{100} = 4,32/1,31 = 3,30 \% (V/V)$

$rE_{180} = 4,55/3,86 = 1,18 \% (V/V)$ $RE_{180} = 8,69/3,86 = 2,25 \% (V/V)$

Annex E (informative)

Emulation of emergent-stem errors

E.1 When an electronic or other sensor without an emergent-stem error is used to emulate a mercury-in-glass thermometer, the output of this sensor or the associated data system should be corrected for this error. The averaged Equations (E.1) and (E.2) have been derived from information supplied by four manufacturers of automated distillation equipment.

Equations (E.1) and (E.2) have limited applicability and are shown for information purposes only. In addition, the correction for the emergent stem, the electronic sensor and the associated data system will also have to emulate the lag in response time observed for mercury-in-glass thermometers.

E.2 When the alternative sensor replaces a low-range thermometer, no stem correction is to be applied below 20 °C. Above this temperature, the emulated temperature, T_{elr} , in degrees Celsius, is calculated from:

$$T_{\text{elr}} = T_{\text{t}} - 0,000\ 162(T_{\text{t}} - 20)^2 \quad (\text{E.1})$$

where T_{t} is the true temperature, expressed in degrees Celsius.

E.3 When the alternative sensor replaces a high-range thermometer, no stem correction is to be applied below 35 °C. Above this temperature, the emulated temperature, T_{ehr} , in degrees Celsius, is calculated from:

$$T_{\text{ehr}} = T_{\text{t}} - 0,000\ 131(T_{\text{t}} - 35)^2 \quad (\text{E.2})$$

where T_{t} is the true temperature, expressed in degrees Celsius.

⋮

Annex F (informative)

Examples of test reports

"Percent Recovered" Report Form

Date:
 Time:
 Operator:

Ambient temperature (°C)
 Atmospheric pressure (kPa)
 Condenser temperature (°C)
 Temperature of the bath around receiving cylinder (°C)

	Percent Recovered	Corrected Temperature Reading (°C)	Time or mL / min
	IBP		
	5		
	10		
	15		
	20		
	25		
	30		
	35		
	40		
	45		
	50		
	55		
	60		
	65		
	70		
	75		
	80		
	85		
	90		
5 ml residue			
	95		
FBP			

Percent Recovery
 Percent Residue
 Percent Total Recovery
 Percent Loss
 Corrected Percent Recovery Corrected Loss
 Corrected Total Recovery

Comments:

Ambient temperature at the start of the test

Ambient barometric pressure at the start of the test

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 simultaneous temperature reading

Temperature measuring device readings which are corrected to 101,3 kPa barometric pressure

Group 1, 2 & 3: 5 to 10 minutes
Group 4: 5 to 15 minutes

Group 1 & 2: 60 to 100 seconds

4 to 5 ml / min uniform average rate from 5% recovered to 5 ml in flask

Volume of condensate observed in the receiving cylinder when the 5ml conditions are reached

Volume of condensate observed in the receiving cylinder when the final boiling point is observed

Maximum percent recovered

Volume of residue in the flask expressed as a percentage of the charge volume

Combined Percent Recovery and Percent Residue in the flask

Time from 5 ml in flask to FBP = < 5 minutes

100 minus the Total Recovery

Percent Recovery corrected for barometric pressure

Percent Loss corrected for barometric pressure

Combined Percent Recovery and Percent Residue in the flask corrected for barometric pressure

"Percent Evaporated" Report Form

Laboratory: _____

Date: _____
 Time: _____
 Operator: _____

Ambient temperature (°C) _____
 Atmospheric pressure (kPa) _____
 Condenser temperature (°C) _____
 Temperature of the bath around receiving cylinder (°C) _____

Percent Recovered	Corrected Temperature Reading (°C)	Time or mL / min	Percent Evaporated	Temperature Readings at prescribed percent evaporated (°C)
IBP			IBP	
5			5	
10			10	
15			15	
20			20	
25			25	
30			30	
35			35	
40			40	
45			45	
50			50	
55			55	
60			60	
65			65	
70			70	
75			75	
80			80	
85			85	
90			90	
5 ml residue				
95			95	
FBP			FBP	

Percent Recovery _____
 Percent Residue _____
 Percent Total Recovery _____
 Percent Loss _____
 Corrected Percent Recovery _____
 Corrected Total Recovery _____

Comments: _____

Ambient temperature at the start of the test

Ambient barometric pressure at the start of the test

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 simultaneous temperature reading

Temperature measuring device readings which are corrected to 101,3 kPa barometric pressure

Sum of the percent recovered and the percent loss

Temperature measuring device readings at specified percentages evaporated calculated with arithmetical or graphical procedures

Group 0: 2 to 5 minutes
 Group 1, 2 & 3: 5 to 10 minutes
 Group 4: 5 to 15 minutes

Group 1 & 2: 60 to 100 seconds

Group 0: time from first application of heat to 10% recovered = 3 to 4 minutes
 Group 0, 1, 2, 3 & 4: 4 to 5 ml / min uniform average rate from 5% recovered to 5 ml in flask

Volume of condensate observed in the receiving cylinder when the 5ml conditions are reached

Volume of condensate observed in the receiving cylinder when the final boiling point is observed

Maximum percent recovered

Volume of residue in the flask expressed as a percentage of the charge volume

Combined Percent Recovery and Percent Residue in the flask

Time from 5 ml in flask to FBP = < 5 minutes

100 minus the Total Recovery

Percent Recovery corrected for barometric pressure

Percent Loss corrected for barometric pressure

Combined Percent Recovery and Percent Residue in the flask corrected for barometric pressure

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