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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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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.

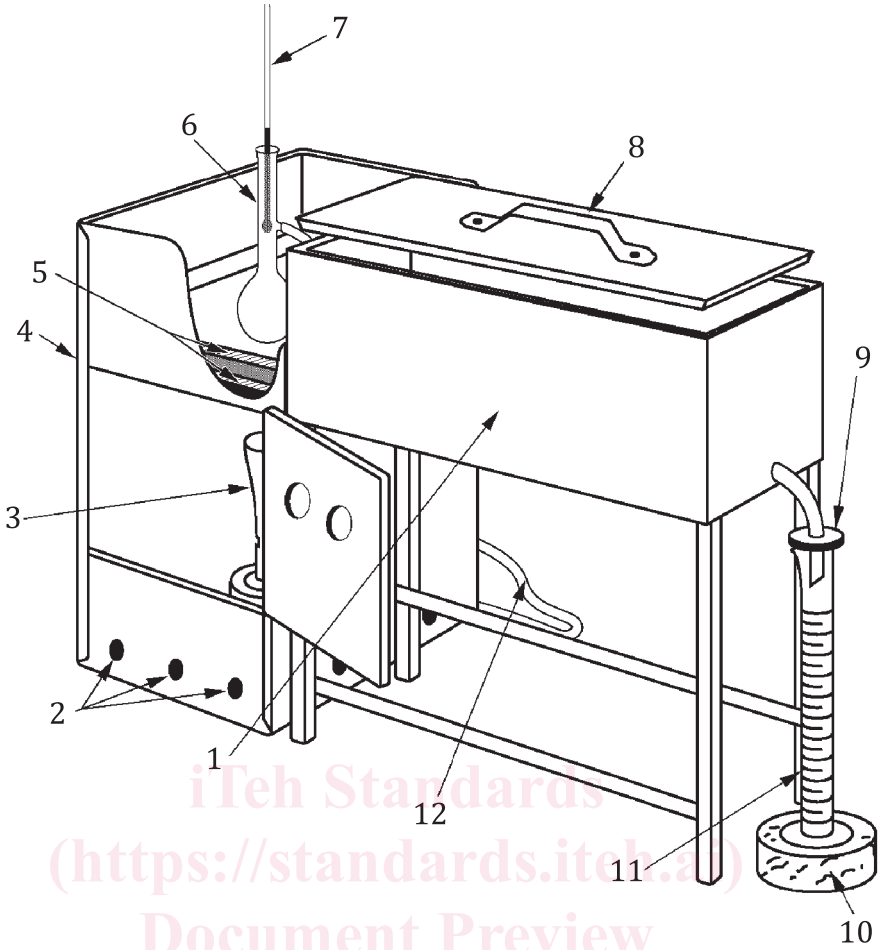
5.3.4 The volume and design of the cooling bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single cooling bath may be used for several condenser tubes.

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).

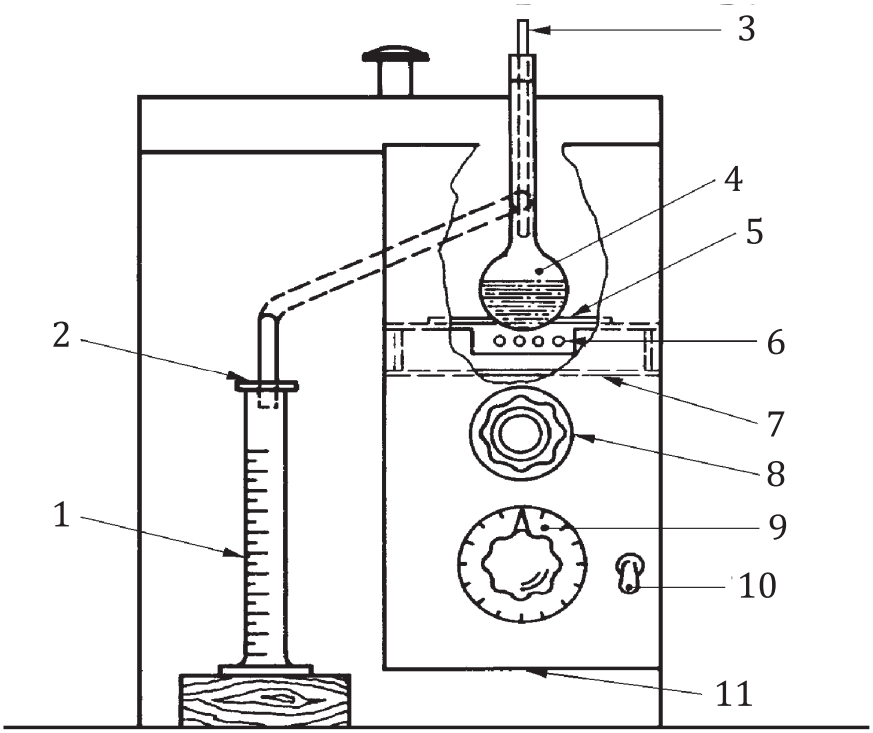
NOTE 2 A typical shield for a unit fitted with an electric heater would be 440 mm high, 200 mm long and 200 mm wide, made of sheet metal approximately 0,8 mm in thickness (see Figure 2).

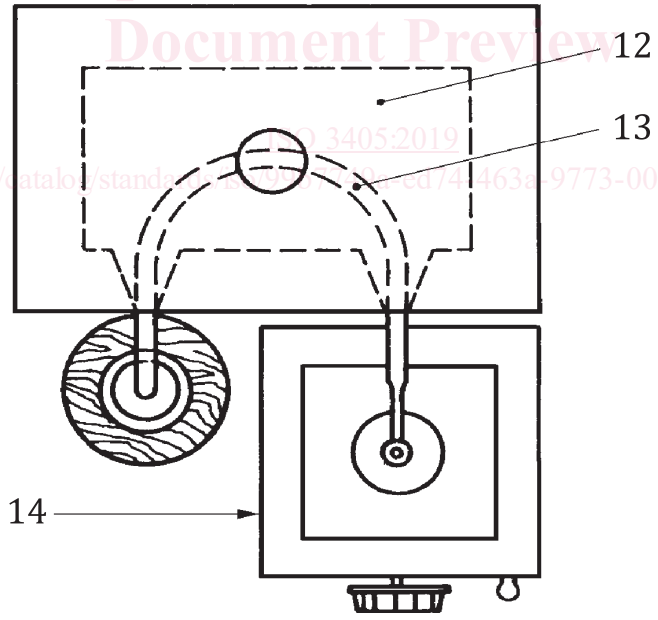


Key

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