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Petroleum products — Determination of distillation characteristics at atmospheric pressure

Produits pétroliers — Détermination des caractéristiques de distillation à pression atmosphérique

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Contents

Page

Foreword v

Introduction vi

1 Scope 1

2 Normative references 1

3 Terms and definitions 1

4 Principle 3

5 Apparatus 3
5.1 General 3
5.2 Distillation flasks 4
5.3 Condenser tube and cooling bath 4
5.4 Metal shield or enclosure for flask (manual apparatus only) 8
5.5 Heat source 8
5.6 Flask support 8
5.7 Graduated cylinders 8
5.8 Temperature measurement system 9
5.9 Centring device 10
5.10 Barometer 10

6 Samples and sampling 11
6.1 Sample grouping 11
6.2 Sample maintenance prior to testing 12
6.3 Removing water from sample 13

7 Preparation of apparatus 13

8 Apparatus verification 15
8.1 Level follower 15
8.2 Electronic temperature-measurement devices 15
8.3 Electronic pressure-measuring device 16

9 Procedure — Manual apparatus 16

10 Procedure — Automated apparatus 18

11 Calculations 20

12 Expression of results 22

13 Precision — Manual apparatus 23
13.1 General 23
13.2 Repeatability 24
13.3 Reproducibility 24

14 Precision — Automated apparatus 25
14.1 General 25
14.2 Repeatability 25
14.3 Reproducibility 25
14.4 Bias 26

15 Test report 26

Annex A (normative) Thermometer specifications 27

Annex B (normative) Determination of temperature-sensor lag times 28

Annex C (normative) Determination of specified distillation data 29

Annex D (informative) **Examples of data calculations** **31**
Annex E (informative) **Emulation of emergent-stem errors** **34**
Annex F (informative) **Examples of test reports** **35**

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 D86[1)].

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.

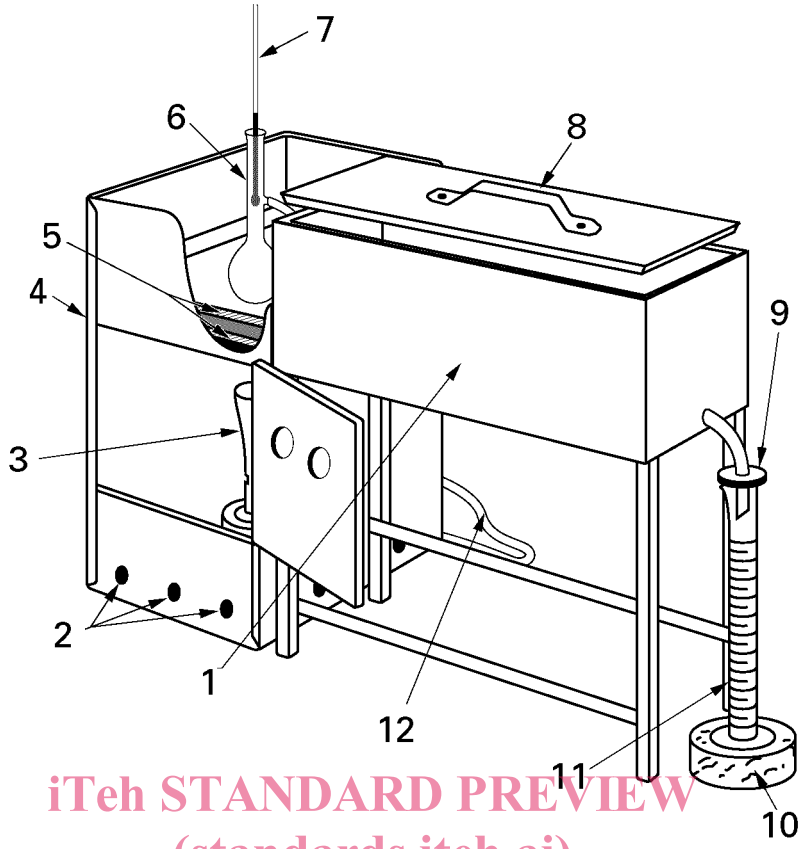
5.3.2 The condenser shall be made of seamless non-corrosive metal tubing, 560 mm ± 5 mm in length, with an outside diameter of 14 mm and a wall thickness of 0,8 mm to 0,9 mm.

NOTE Brass or stainless steel are suitable materials.

5.3.3 The condenser shall be set so that a length of 393 mm ± 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° 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 the range of 15° ± 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.

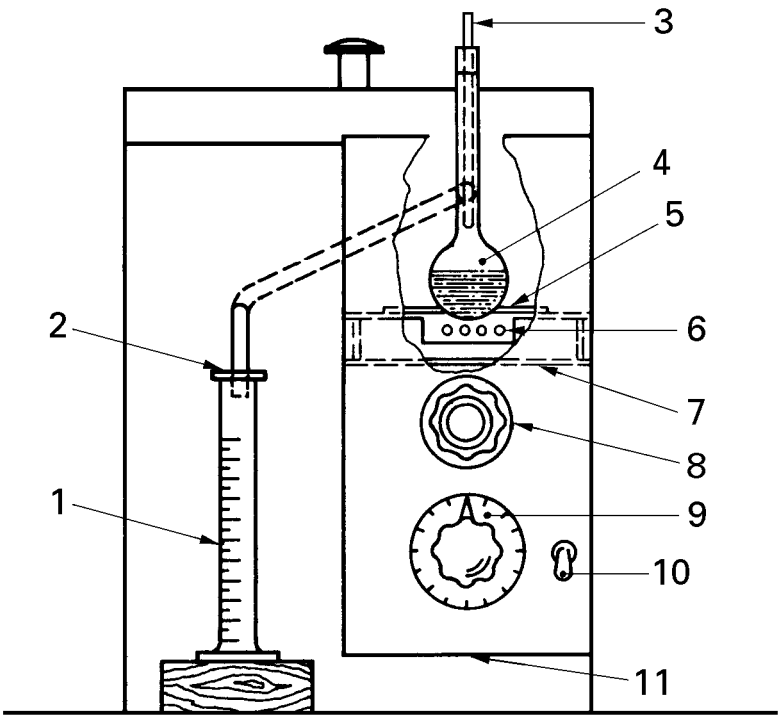
5.3.4 The volume and design of the cooling bath will depend on the cooling medium employed. The cooling capacity of the bath shall be adequate to maintain the required temperature for the desired condenser performance. A single cooling bath may be used for several condenser tubes.

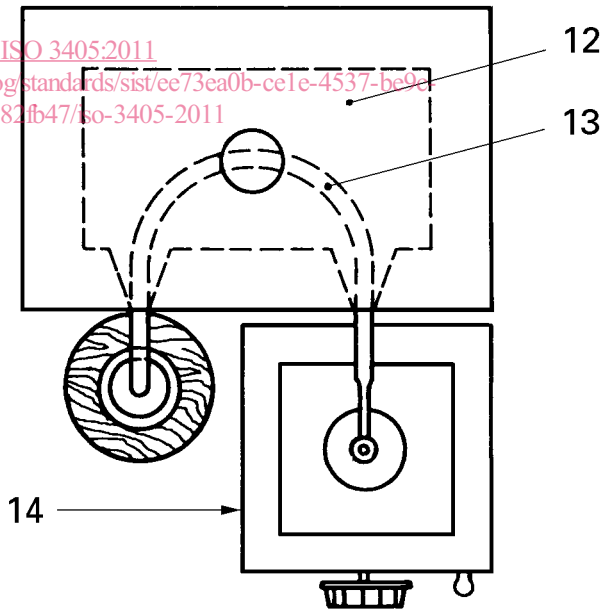


Key

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



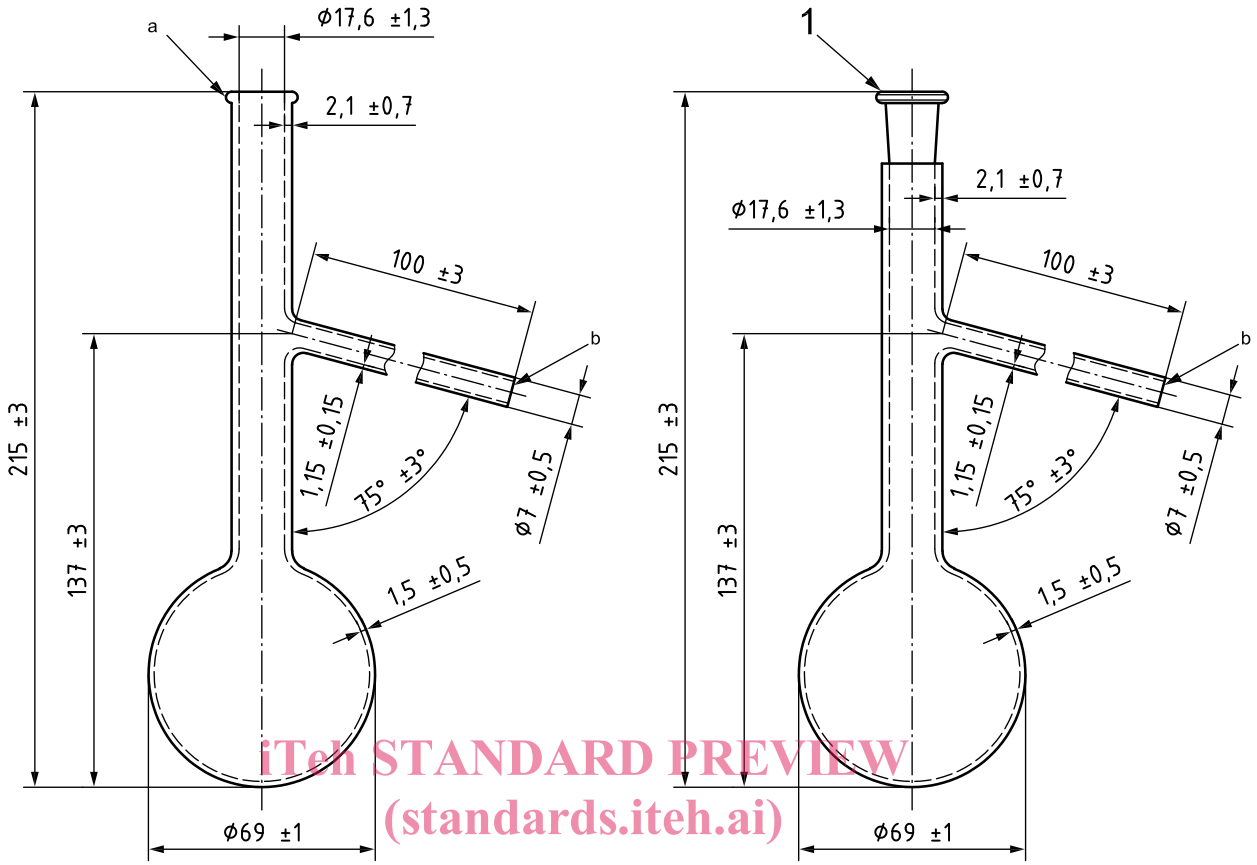


Key

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

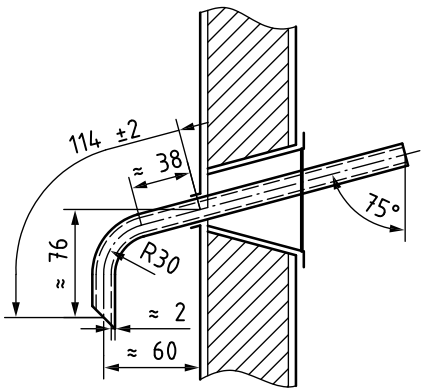


Figure 4 — Lower end of condenser tube