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Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials

Liquides organiques volatils — Détermination de l'intervalle de distillation des solvants organiques utilisés comme matières premières

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Foreword

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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 document 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).

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This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

This second edition results from the reinstatement of ISO 4626:1980, which was withdrawn in 2017.

The main changes are as follows:

— the normative references have been updated.

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.

Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials

1 Scope

This document specifies a method for determining the boiling range of liquids that boil between 30 °C and 300 °C at normal pressure, and that are chemically stable and do not corrode the apparatus during the distillation.

The method is applicable to organic liquids such as hydrocarbons, esters, alcohols, ketones, ethers and similar products.

NOTE 1 The method differs from that described in ISO 918 with respect to the volume of the distillation flask, the type of cooler and the distillation receiver.

NOTE 2 The method differs from that specified in ISO 3405 with respect to the volume of the distillation flask and the diameter of the hole in the flask support.

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 3165, *Sampling of chemical products for industrial use — Safety in sampling*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

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

ISO and IEC maintain terminology 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
initial boiling point
temperature noted (corrected if required) at the moment when the first drop of condensate falls from the tip of the condenser during a distillation carried out under standardized conditions

3.2
dry point
temperature noted (corrected if required) at the moment of vaporization of the last drop of liquid at the bottom of the flask during a distillation carried out under standardized conditions, disregarding any liquid on the side of the flask and on the thermometer

3.3
boiling range
temperature interval between the *initial boiling point* (3.1) and the *dry point* (3.2)

3.4
end point
final boiling point
maximum temperature noted (corrected, if required) during the final phase of a distillation carried out under standardized conditions

4 Principle

100 ml of a test portion are distilled under prescribed conditions which are equivalent to a simple batch distillation. Thermometer readings and volumes of condensate are observed systematically and the results from these data with correction to standard atmospheric pressure are calculated.

5 Apparatus

The apparatus, a suitable form of which is shown in Figure 1 to Figure 4, shall comprise the following items.

5.1 Distillation flask, of heat-resistant glass, of capacity 200 ml, conforming to the dimensions shown in Figure 1.

Superheating of liquid in a new flask may be prevented by depositing a small amount of carbon in the bottom of the flask. This may be accomplished by heating and decomposing a pinch of tartaric acid in the bottom of the flask. The flask is then prepared for use by washing with water, rinsing with acetone, and drying.

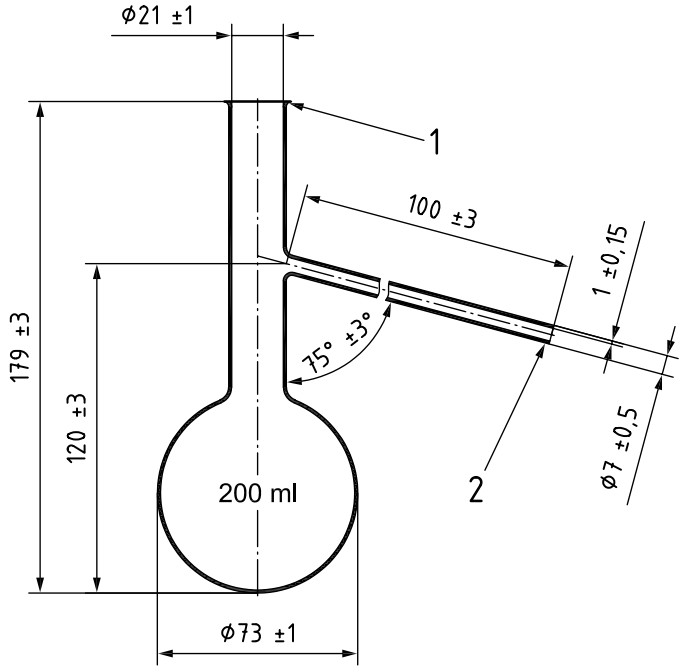
An exception is made for diacetone alcohol: in order to avoid an erratic value for the initial boiling point, the distillation flask should be clean and free of any residual carbon deposit.

5.2 Thermometers, mercury-in-glass type, nitrogen-filled, graduated on the stem, enamel-backed, and conforming to the requirements in Table 1.

The thermometer should have been artificially aged by means of a suitable treatment before graduation, in order to ensure stability of the lowest point on the scale. This treatment should have been such that, after the procedure described below, the rise at a fiducial point is not greater than the maximum error specified, and the accuracy of the thermometer is within the limits specified.

Heat the thermometer to a temperature equal to its highest reading and keep it at this temperature for 5 min. Allow the thermometer to cool, either naturally in still air or slowly in the test bath (at a specified rate), to 20 °C above ambient temperature or to 50 °C, whichever is the lower, and then determine the lowest point on the scale. If rapid cooling is used, the lowest point on the scale shall be determined within 1 h. Heat the thermometer again to a temperature equal to its highest reading, keep it at this temperature for 24 h. Allow the thermometer to cool to 20 °C above ambient temperature or to 50 °C, at the same rate as at the start of the test, and re-determine the lowest point on the scale under the same conditions as before.

Dimensions in millimetres



Key

1 reinforcing bead
2 fire-polished

Figure 1 — Distillation flask

Table 1 — Solvent distillation thermometers

Designation[a]	38 C-75	39 C-75	40 C-75	41 C-62	42 C-62	102 C-65	103 C-65	104 C-75	105 C-65	106 C-75	107 C-75
Immersion mm	100	100	100	100	100	100	100	100	100	100	100
Range °C	24 to 78	48 to 102	72 to 126	98 to 152	95 to 255	123 to 177	148 to 202	173 to 227	198 to 252	223 to 277	248 to 302
Graduation °C	0,2	0,2	0,2	0,2	0,5	0,2	0,2	0,2	0,2	0,2	0,2
Longer lines at each °C	1	1	1	1	1	1	1	1	1	1	1
Figured at each °C	2	2	2	2	5	2	2	2	2	2	2
Scale error not to exceed °C	0,2	0,2	0,2	0,2	0,5	0,2 up to 150 0,3 over 150	0,4	0,4	0,4 up to 225 0,6 over 225	0,8	1,0
Overall length mm	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5	395 ± 5
Stem diameter mm	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0	6,0 to 8,0	6,0 to 8,0
Bulb length mm	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20	15 to 20
Distance from bottom of bulb — to	24 °C 125 mm to 145 mm	48 °C 125 mm to 145 mm	72 °C 125 mm to 145 mm	98 °C 125 mm to 145 mm	95 °C 125 mm to 145 mm	123 °C 125 mm to 145 mm	148 °C 125 mm to 145 mm	173 °C 125 mm to 145 mm	198 °C 125 mm to 145 mm	23 °C 125 mm to 145 mm	248 °C 125 mm to 145 mm
— to	78 °C 335 mm to 360 mm	102 °C 335 mm to 360 mm	126 °C 335 mm to 360 mm	152 °C 335 mm to 360 mm	255 °C 335 mm to 360 mm	177 °C 335 mm to 360 mm	202 °C 335 mm to 360 mm	227 °C 335 mm to 360 mm	252 °C 335 mm to 360 mm	277 °C 335 mm to 360 mm	302 °C 335 mm to 360 mm
Expansion chamber to allow heating to °C	105	130	150	180	280	200	225	250	275	300	325

[a] These designations correspond to those in ASTM E1.

5.3 Draught screen

5.3.1 For use with a gas burner

The draught screen shall be rectangular in cross-section and open at the top and bottom. It shall have the dimensions shown in Figure 2 and be made of a sheet of metal, approximately 0,8 mm thick.

In each of the two narrower sides of the draught screen, there shall be two circular holes of diameter of 25 mm, the centres of which are situated 215 mm below the top of the shield, as shown in Figure 2.

In each of the four sides of the draught screen, there shall be three circular holes of diameter 12,5 mm, the centres of which are situated 25 mm above the base of the draught screen. These holes shall occupy the positions shown in Figure 2.

At the middle of each of the wider sides, a vertical slot for the condenser tube, dimensioned approximately as shown in Figure 2, shall be cut downwards from the top of the screen. A removable shutter of suitable dimensions shall be provided for closing whichever vertical slot is not in use. This arrangement enables the condenser (5.6) to be placed on either side of the draught screen.

A shelf of ceramic material, of thickness 3 mm to 6 mm and possessing a centrally cut circular hole of diameter 75 mm to 100 mm, shall be supported horizontally in the screen and shall fit closely to the sides of the screen, to ensure that hot gases from the source of heat (5.5) do not come in contact with the sides or neck of the flask (5.1). The supports for this shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

A board as described in 5.4 shall rest on this shelf.

In one of the narrower sides of the screen, a door shall be provided, having the approximate dimensions shown in Figure 2 and overlapping the opening in the screen by approximately 5 mm all round.