
International Standard



918

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ISO 918:1983

Volatile organic liquids for industrial use — Determination of distillation characteristics

Liquides organiques volatils à usage industriel — Détermination des caractéristiques de distillation

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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 developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 918 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1982.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Portugal
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
China	Italy	Switzerland
Czechoslovakia	New Zealand	USSR
Egypt, Arab Rep. of	Nigeria	
France	Poland	

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

This International Standard cancels and replaces ISO Recommendation R 918-1969, of which it constitutes a technical revision.

This International Standard is one of three ISO publications dealing with the determination of distillation characteristics. The two others are :

- ISO 3405, *Petroleum products — Determination of distillation characteristics.*
- ISO 4626, *Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials.*

It is recognized that there may be some overlapping between these three documents which were developed in separate technical committees. In the absence of agreement on a general International Standard on the subject, it has however been thought necessary to publish them.

The completion in the future of such a general standard may therefore lead to the amendment or cancellation of this International Standard.

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Volatile organic liquids for industrial use — Determination of distillation characteristics

1 Scope and field of application

This International Standard specifies an apparatus and method for the determination of distillation characteristics of volatile organic liquids which (excluding petroleum products) having a boiling point in the range 30 to 300 °C approximately and are stable during distillation in atmospheric conditions.

2 References

ISO 3405, *Petroleum products — Determination of distillation characteristics*.

ISO 4626, *Volatile organic liquids — Determination of boiling range of organic solvents used as raw materials*.

3 Definitions (see ISO 3405 and ISO 4626)

3.1 initial boiling point : The 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 final boiling point : The maximum temperature noted (corrected, if required) during the final phase of the distillation of a sample carried out under standardized conditions.

3.3 dry point : The temperature noted (and corrected, if required) at the moment of vaporization of the last drop of liquid at the bottom of a flask, during a distillation carried out under standardized conditions.

3.4 boiling range : The temperature interval between the initial boiling point and the dry point.

4 Principle

Distillation of a test portion, under carefully defined conditions in order to determine either

4.1 the temperatures corresponding to the condensate volumes defined in the International Standard for the product under test; these temperatures shall be corrected as indicated in clause 9, unless the nature of the product provides only for a difference of temperature between two volumes of condensate, (plotting temperature as a function of volume); or

4.2 the volume of condensate when the thermometer indicates each of the distillation temperatures (previously adjusted as indicated in clause 9) specified in the International Standard for the product under test (plotting volume as a function of temperature).

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Distillation apparatus, as shown in figure 1 comprising

5.1.1 Distillation flask, available capacity 100 ml, of borosilicate glass, as shown in figure 2.

5.1.2 Thermometer, of the mercury-in-glass type, graduated in 0,2 °C intervals, having a maximum error of $\pm 0,2$ °C and covering a range suitable for the products being tested.

The distance between the top of the bulb and the first graduation mark is at least 100 mm. The thermometer has no contraction chamber unless this is very close to the bulb or contained therein.

The scale is calibrated for use at 100 mm immersion (or alternatively for total immersion).

Additional requirements, in particular concerning the range of the thermometer, may be given in the International Standards for the products under test.

5.1.3 Receiver, 100 ml capacity, graduated as shown in figure 3.

5.1.4 Condenser of the Liebig/West type, of borosilicate glass, as shown in figure 4. The inner tube shall have the following dimensions :

Internal diameter	14,0 $\pm$ 1,0 mm
Wall thickness	1,0 to 1,5 mm
Length of straight portion of longer limb	600 $\pm$ 10 mm
Length of shorter limb	55 $\pm$ 5 mm
Angle included between longer and shorter limb	97 $\pm$ 3°

The inlet of the inner tube of the condenser shall be finished square with its axis and its outlet, smoothly ground at an angle

of approximately 45° to the axis of the tube at that point, as shown in figure 4.

The length of the central straight portion of the water jacket shall be 450 ± 10 mm, and its external diameter 35 ± 3 mm.

5.1.5 Draught screen, rectangular in cross-section and open at the top and bottom. It shall have the dimensions shown in figure 5 and shall be made of sheet metal 0,7 mm thick.

In each of the two narrower sides of the draught screen are two air-vent holes 25 mm in diameter situated below the heat-resistant shelf, as shown in figure 5.

In each of the four sides of the draught screen are three air-vent holes with their centre 25 mm above the base of the draught screen. These holes occupy the positions shown in figure 5, the diameter of the holes situated centrally in the wider side being 25 mm, and the diameter of the remaining 10 holes 12,5 mm.

At the middle of each of the wider sides, a vertical slot for the side tube of the distillation flask, dimensioned as shown in figure 5, is cut downwards from the top of the screen. A removable shutter, conforming to the dimensions in figure 5, is provided for closing whichever vertical slot is not in use.

A shelf of hard heat-resistant board 6 mm in thickness and possessing a centrally cut circular hole 110 mm in diameter, is supported horizontally in the screen and fits closely to the sides of the screen, to ensure that hot gases from the source of heat do not come into contact with the sides or neck of the flask. The supports for shelf may consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

In one of the narrower sides of the screen, a door is provided having the dimensions shown in figure 5 and overlapping an opening in the screen by approximately 5 mm all round.

In each of the narrower sides of the screen, a mica window is placed centrally with the bottom of the window level with the top of the heat-resistant shelf. The dimensions and the positions of the windows are shown in figure 5.

5.1.6 Heat-resistant board.

In addition to the heat-resistant shelf referred to in 5.1.5, a heat-resistant board 150 mm square and 6 mm in thickness is required. It has a central hole of 50 mm diameter, unless otherwise stated in the specification for the material under test.

5.2 Assembly of the apparatus

Assemble the distillation apparatus as shown in figure 1.

5.2.1 Position of thermometer

The thermometer shall be held in the neck of the flask by means of a well-fitting stopper of a material which is not attacked by

the liquid under test. The junction of the capillary tube and the main bulb of the thermometer shall be maintained level with the lower edge of the joint between the side tube and the neck of the flask. The stopper shall project about 10 mm above the top of the neck of the flask.

When the thermometer is fixed in position as indicated above, the immersion line on the thermometer will be in the neighbourhood of the top of the stopper holding the thermometer in position in the neck of the flask.

5.2.2 Support for flask

The heat-resistant board (5.1.6) shall be placed on the top of the heat-resistant shelf of the draught screen so that the two holes are approximately concentric. The flask is then placed in position so as to close the hole in the heat-resistant board completely.

5.2.3 Connection of flask to condenser

The flask shall be connected to the condenser so that the end of the side tube projects at least 25 mm into the condenser and is coaxial with it.

6 Sampling¹⁾

Store the laboratory sample in a clean, dry and air-tight dark-glass flask, fitted either with a ground glass stopper or with a screw cap containing an air-tight inert plastics seal. The bottle should be of such capacity that it is almost entirely filled by the sample. If it is necessary to seal the container, take care to avoid any contamination of the contents.

If special precautions are necessary, they shall be given in the appropriate International Standard test methods.

To carry out the test specified in this International Standard, a laboratory sample of 500 ml is recommended.

7 Procedure

7.1 Test portion

Using the receiver (5.1.3) take 100 ± 1 ml of the laboratory sample.

If the expected distillation temperature lies below 70 °C, adjust the temperature of the sample, before taking it, to the appropriate value shown in table 1.

Table 1 — Sampling temperatures

Values in degrees Celsius

Initial boiling point	Condenser	Sample
Below 50	0 to 3	0 to 3
50 to 70	0 to 10	10 to 20
70 to 150	25 to 30	20 to 30
Above 150	35 to 50	20 to 30

1) The sampling of liquid chemical products for industrial use will form the object of a future International Standard.