

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 918

TEST METHOD FOR DISTILLATION
(DISTILLATION YIELD AND DISTILLATION RANGE)

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BRIEF HISTORY

The ISO Recommendation R 918, *Test method for distillation (Distillation yield and distillation range)*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led in 1962 to the adoption of a Draft ISO Recommendation.

In November 1963, this Draft ISO Recommendation (No. 658) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Portugal
Austria	India	Romania
Belgium	Israel	Spain
Chile	Italy	U.A.R.
Colombia	Japan	United Kingdom
Czechoslovakia	Korea, Rep. of	U.S.S.R.
France	Netherlands	Yugoslavia
Germany	Poland	

One Member Body opposed the approval of the Draft :

New Zealand

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in January 1969, to accept it as an ISO RECOMMENDATION.

TEST METHOD FOR DISTILLATION (DISTILLATION YIELD AND DISTILLATION RANGE)

1. SCOPE

This document describes an apparatus and method for the determination of distillation characteristics (distillation yield and distillation range) of liquids.

The method is applicable whenever specified by an ISO Recommendation relating to a given product.

2. PRINCIPLE

Distillation of a given volume of the sample, under carefully defined conditions, and determination of either the volume of distillate collected as a function of temperature, or vice versa.

3. APPARATUS

The apparatus required is shown in Figure 1, and comprises the following items :

- 3.1 *Distillation flask*, nominal capacity 100 ml, of borosilicate glass, complying with Figure 2.
- 3.2 *Thermometer* of the mercury-in-glass type, graduated in 0.2 degC intervals, having a maximum error of ± 0.2 degC and covering a range suitable for the sample 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 standard for the material under test.
- 3.3 *Receiver*, 100 ml capacity, complying with Figure 3.
- 3.4 *Condenser* of good quality borosilicate glass, as shown in Figure 4, the inner tube having 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 is finished square with its axis, and its outlet is smoothly ground at an angle of approximately 45° to the axis of the tube at that point, as shown in Figure 4.
The central straight portion of the water jacket (see Fig. 4) is 450 $\pm$ 10 mm in length, and its external diameter 35 $\pm$ 3 mm.

- 3.5 *Draught screen*, rectangular in cross-section and open at the top and bottom. It has the dimensions shown in Figure 5 and is made of sheet metal 0.7 mm thick.

In each of the two narrower sides of the draught screen are two circular air vent holes 25 mm in diameter, situated below the asbestos shelf, as shown in Figure 5.

In each of the four sides of the draught screen are three air vent holes with their centres 25 mm above the base of the draught screen. These holes occupy the positions shown in Figure 5, the diameter of each of the holes centrally situated in the wider sides being 25 mm, and the diameter of the remaining ten 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 asbestos 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 this asbestos shelf may conveniently 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 asbestos shelf. The dimensions and positions of the windows are shown in Figure 5.

- 3.6 *Asbestos board*. In addition to the asbestos shelf referred to in clause 3.5, an asbestos 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.

4. ASSEMBLY OF THE APPARATUS

The apparatus is assembled as shown in Figure 1, and attention is drawn to the following details :

4.1 Position of thermometer

The thermometer is held concentrically in the neck of the flask by means of a well-fitting stopper of a material which is not attacked by the liquid, and the junction of the capillary tube and the main bulb of the thermometer is maintained level with the lower edge of the joint between the side tube and the neck of the flask. The stopper should 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.

4.2 Support for flask

The asbestos board (3.6) is placed on the top of the asbestos shelf of the draught screen so that the two holes are approximately concentric. The flask is then placed in position and pressed down so as to close completely the hole in the asbestos board.

4.3 Connection of flask to condenser

The flask is 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.