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ISO RECOMMENDATION

R 918

TEST METHOD FOR DISTILLATION
(DISTILLATION YIELD AND DISTILLATION RANGE)

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Vérifié par :

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

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.

ISO Recommendation R 918 January 1969

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 ± 1.0 mm
– wall thickness	1.0 to 1.5 mm
– length of straight portion of longer limb	600 ± 10 mm
– length of shorter limb	55 ± 5 mm
– angle included between longer and shorter limb	97 ± 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 ± 10 mm in length, and its external diameter 35 ± 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 is 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.

5. ADJUSTMENTS TO BE APPLIED TO SPECIFIED TEMPERATURES BEFORE COMMENCING THE DISTILLATION

(For yield)

5.1 Adjustments for barometric pressure

When the barometric pressure (corrected as described in section 8) deviates from 760 mmHg, apply adjustments to the specified distillation temperatures, as indicated in the specification for the material under test. It should be noted that these adjustments are valid only for pressures above 700 mmHg.

5.2 Adjustments for thermometer error

If the thermometer gives incorrect readings at the specified distillation temperatures adjusted in accordance with clause 5.1, further adjust the temperatures by adding the amount of error if the thermometer is reading high or subtracting the amount of error if the thermometer is reading low.

Also make corrections for the emergent stem of the thermometer when using a thermometer calibrated for total immersion.

6. PROCEDURE

6.1 Measure 100 ml of the sample in the receiver (3.3). Transfer the liquid as completely as possible to the distillation flask (3.1), add a few small pieces of clean, dry porous pot. Place the flask, thermometer (3.2) and receiver in position and ensure that the condenser has a steady supply of water*. Light the burner and regulate the flame so that the first drop of distillate falls from the end of the condenser after 5 to 10 minutes for materials boiling below 100 °C, after 10 to 15 minutes for materials boiling above 100 °C, or after any other interval as indicated in the specification for the material.

6.2 Further regulate the flame so that the distillate is collected at the rate of 3 to 4 ml per minute, unless otherwise stated in the specification for the material under test, adjusting the distance between the burner and the bottom of the flask so that the flame of minimum size only is necessary.

6.3 Record

- *either* the temperatures corresponding to volumes of distillate in the standard for the product under test (distillation range); these temperatures should be corrected as described in section 7 unless the standard for the product provides only for a difference of temperature between two volumes of distillate;
- *or* the volume of distillate in the receiver when the thermometer indicates each of the distillation temperatures (previously adjusted as described in section 5) specified in the standard for the product under test (distillation yield).

7. CORRECTIONS TO BE APPLIED TO THE OBSERVED TEMPERATURES AFTER THE DISTILLATION

(For range)

7.1 Corrections for thermometer error

If the thermometer gives incorrect readings at the observed initial boiling point or observed dry point, correct the readings by subtracting the amount of error if the thermometer is reading high, or adding the amount of error if the thermometer is reading low.

Also make corrections for the emergent stem of the thermometer when using a thermometer calibrated for total immersion.

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* For materials expected to boil below 70 °C, the temperature of the water supply should not exceed 20 °C.

7.2 Correction for barometric pressure

When the barometric pressure (corrected as in section 8) deviates from 760 mmHg, apply further corrections to the observed temperatures as indicated in the specification for the product under test. It should be noted that these last corrections are valid only for pressures above 700 mmHg.

8. CORRECTIONS TO BAROMETER READING

Ascertain the atmospheric pressure by taking a reading on a mercury barometer* having a millimetre scale and applying the following corrections :

8.1 Index correction

Correct the observed barometer reading for index error in accordance with the certificate issued with the barometer. The reading thus corrected gives the atmospheric pressure as indicated by a correctly adjusted barometer at the temperature of observation, and under the gravity prevailing at the place of observation, and must be further corrected to give the equivalent value in millimetres of mercury at 0 °C.

8.2 Temperature correction to 0 °C

If the mercury barometer is of the Fortin type, or of some other type in which the mercury in the cistern is set to a fiducial level when the barometric reading is taken, apply the temperature correction given in Table 1 to the value obtained in clause 8.1.

If the barometer used is of the Kew type, i.e. one in which the barometric reading is taken without adjusting the level of the mercury in the cistern, the temperature correction will differ somewhat from that given in Table 1. The temperature coefficient of a Kew pattern barometer depends to a small extent on its dimensions, but sufficient accuracy will be obtained, in general, if the temperature correction to a reading of a Kew pattern barometer is taken to be 5 % in excess of that given in Table 1 for a Fortin barometer.

8.3 Correction to standard gravity

The following information is given for use when distillations are carried out in latitudes where the value of gravity differs widely from the standard value.

The reading as corrected in accordance with clause 8.2 gives the pressure in terms of standard millimetres of mercury at 0 °C at the place of observation. To obtain the equivalent pressure at 0 °C and under standard gravity, multiply the value obtained as above by $\frac{g}{980.665}$, where g is the value of gravity in cm/s^2 at the place of observation. Changes in gravity due to causes other than change in latitude, e.g. height above sea level, may be neglected and the reduction to standard gravity carried out by means of Table 2 which gives the appropriate corrections for different latitudes. When the sign of the correction in Table 2 is positive, add, and when negative, subtract, the quantity noted to or from the barometric reading.

9. EXPRESSION OF RESULTS

State in the test report the characteristics required for the product concerned.

10. TEST REPORT

Give the following information :

(*a*) the reference of the method used;

(*b*) the results and the method of expression used;

(*c*) any unusual features noted during the determination;

(*d*) any operation not laid down in this ISO Recommendation or regarded as optional.

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* Alternatively, an aneroid barometer, having a scale calibrated in standard millimetres of mercury, may be employed.

TABLE 1 – Correction of barometer readings to 0 °C Fortin barometer with brass scale

(Subtract the correction from the barometric reading)

Temperature of barometer	Barometer reading, mmHg						
°C	700	720	740	760	780	800	820
10	1.14	1.17	1.21	1.24	1.27	1.30	1.34
11	1.26	1.29	1.33	1.36	1.40	1.44	1.47
12	1.37	1.41	1.45	1.49	1.53	1.57	1.60
13	1.48	1.53	1.57	1.61	1.65	1.70	1.74
14	1.60	1.64	1.69	1.73	1.78	1.83	1.87
15	1.71	1.76	1.81	1.86	1.91	1.96	2.00
16	1.82	1.88	1.93	1.98	2.03	2.09	2.14
17	1.94	1.99	2.05	2.10	2.16	2.22	2.27
18	2.05	2.11	2.17	2.23	2.29	2.35	2.40
19	2.17	2.23	2.29	2.35	2.41	2.48	2.54
20	2.28	2.34	2.41	2.47	2.54	2.60	2.67
21	2.39	2.46	2.53	2.60	2.67	2.73	2.80
22	2.51	2.58	2.65	2.72	2.79	2.86	2.94
23	2.62	2.69	2.77	2.84	2.92	2.99	3.07
24	2.73	2.81	2.89	2.97	3.05	3.12	3.20
25	2.85	2.93	3.01	3.09	3.17	3.25	3.33
26	2.96	3.04	3.13	3.21	3.30	3.38	3.47
27	3.07	3.16	3.25	3.34	3.42	3.51	3.60
28	3.19	3.28	3.37	3.46	3.55	3.64	3.73
29	3.30	3.39	3.49	3.58	3.68	3.77	3.87
30	3.41	3.51	3.61	3.71	3.80	3.90	4.00

TABLE 2 – Correction of barometer readings to standard gravity
($g = 980.66$ cm/s^2)

Latitude degrees	Barometer reading, mmHg 700	720	740	760	780
0	- 1.88	- 1.93	- 1.99	- 2.04	- 2.10
5	- 1.85	- 1.90	- 1.95	- 2.01	- 2.06
10	- 1.77	- 1.82	- 1.87	- 1.92	- 1.97
15	- 1.63	- 1.68	- 1.72	- 1.77	- 1.82
20	- 1.45	- 1.49	- 1.53	- 1.57	- 1.61
25	- 1.22	- 1.25	- 1.29	- 1.32	- 1.36
30	- 0.96	- 0.98	- 1.01	- 1.04	- 1.07
35	- 0.67	- 0.68	- 0.70	- 0.72	- 0.74
40	- 0.36	- 0.37	- 0.38	- 0.39	- 0.40
45	- 0.04	- 0.04	- 0.04	- 0.04	- 0.04
50	+0.29	+0.29	+0.30	+0.31	+0.32
55	+0.60	+0.61	+0.62	+0.65	+0.67
60	+0.89	+0.91	+0.94	+0.97	+0.99
65	+1.15	+1.19	+1.22	+1.25	+1.29
70	+1.38	+1.42	+1.46	+1.50	+1.54

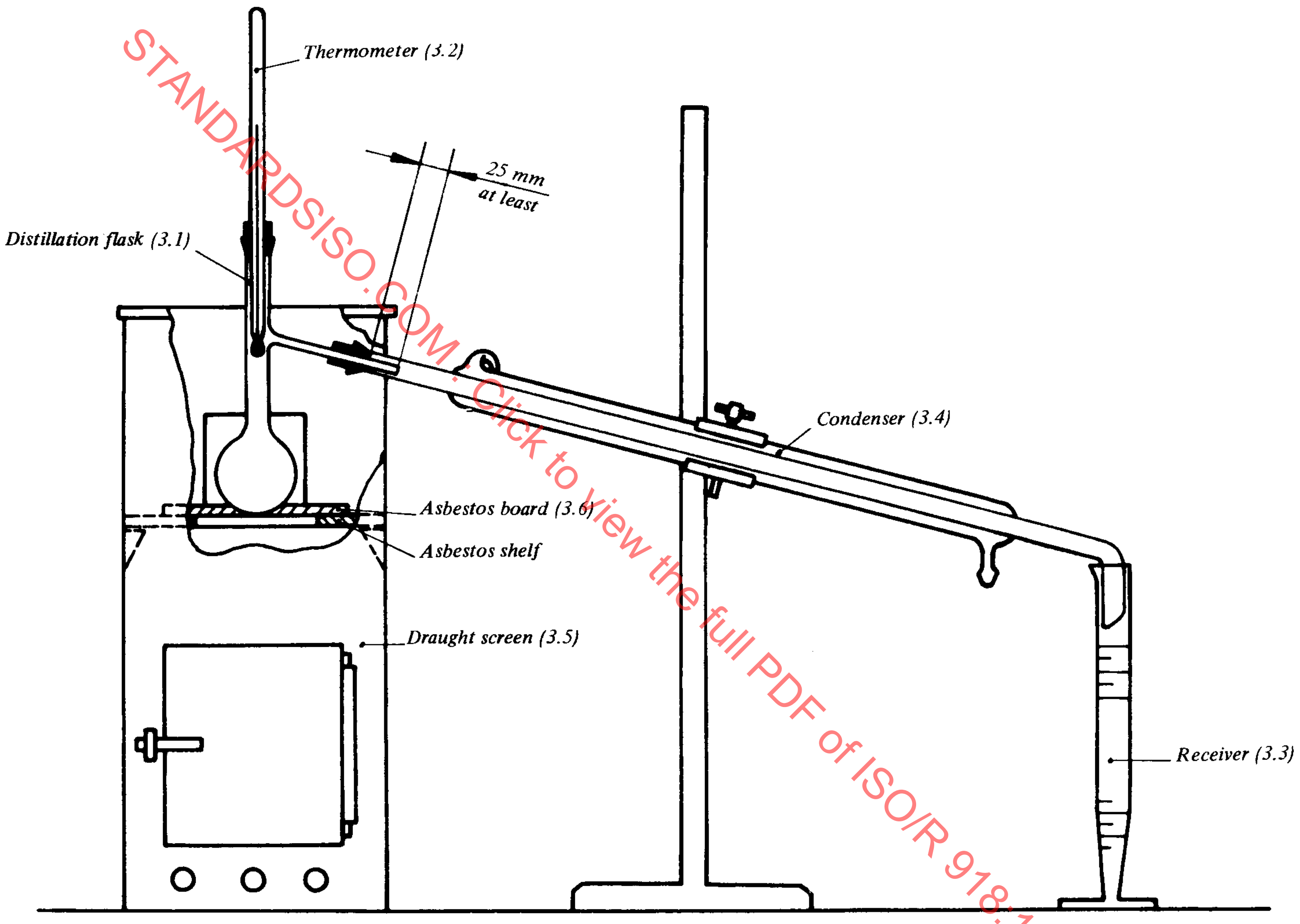

FIG. 1 – **Assembly of distillation apparatus**