International Standard 5278

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Benzene — Determination of crystallizing point

Benzène — Détermination du point de cristallisation

First edition — 1980-04-01

UDC 547.532 : 532.785 **Ref. No. ISO 5278-1980 (E)**

Descriptors : aromatic hydrocarbons, benzene, physical tests, solidification point, test equipment, thermometers.

Price based on 3 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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 5278 was developed by Technical Committee ISO/TC 78, *Aromatic hydrocarbons,* and was circulated to the member bodies in September 1977.

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

Australia	Hungary	Philippines
Austria	India	Romania
Bulgaria	Italy	South Africa, Rep. of
Czechoslovakia	Korea, Rep. of	Turkey
Egypt, Arab Rep. of	Mexico	United Kingdom
Germany, F.R.	Netherlands	USSR

The member bodies of the following countries expressed disapproval of the document on technical grounds :

France
Poland

Printed in Switzerland

Benzene — Determination of crystallizing point

WARNING — Benzene is highly flammable and toxic by inhalation, ingestion or skin absorption.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of the crystallizing point of benzene to the nearest 0,01 °C.

NOTE — This method differs so much from that specified in ISO 1392, *Determination of crystallizing point — General method*, that the latter could not be adopted.

2 REFERENCE

ISO 1995, *Aromatic hydrocarbons — Sampling.* [1)]

3 PRINCIPLE

Determination of the crystallizing point on a test portion saturated with water and application of a correction to obtain the crystallizing point of the anhydrous material.

4 APPARATUS

The apparatus required is shown in figure 1 and comprises the following components :

4.1 Test tube, of internal diameter 35 ± 2 mm, length 200 ± 5 mm and of wall thickness about 1,5 mm.

NOTE — Badly scratched test tubes can promote premature crystallization and should therefore be discarded.

The test tube (4.1) shall be fitted with a cork stopper through the central hole of which the thermometer (4.4) can easily pass. A tight rubber ring shall be placed on the stem of the thermometer in such a position that, when this is resting on the stopper, the thermometer is hanging freely in the test tube with the bottom of the bulb 10 to 12 mm from the bottom of the test tube.

These shall be a second hole near to the edge of the stopper to enable the glass rod (4.2) to be supported in a similar manner.

4.2 Glass rod, supported on a rubber ring on the cork stopper in such a position that it may be used to scratch the inside of the tube as required.

4.3 Dewar flask, of internal diameter at least 80 mm, for use as a cooling bath.

4.4 Thermometer, as shown in figure 2, complying with the following specification :

Range	4 to 6 °C
For test at	5,4 °C
Sub-divisions	0,01 °C
Long lines at each	0,10 °C
Short lines at each	0,05 °C
Number at each	0,20 °C
Maximum scale error	0,01 °C
Permit heating to	35 °C

4.5 Separating funnel, stoppered, of capacity 250 ml.

5 SAMPLING

Take a representative sample of not less than 500 ml from the bulk of the material.

Recommended methods of sampling are given in ISO 1995.

6 THERMOMETER CHECK

Changes in the dimensions of the bulbs of thermometers of this accuracy necessitate frequent checks of the mercury position relative to the scale. These checks are made by reference to benzene, of known crystallizing point, as follows.

Carry out the test as described in clause 7 on benzene having a certified crystallizing point (wet). If the determined value is below the certified crystallizing point (wet), add the difference between the certified and the determined crystallizing points to the reading obtained in the test. If it is above, subtract the difference.

1) At present at the stage of draft.

7 PROCEDURE

Fill the Dewar flask (4.3) with a quantity of water such that, when the test tube (4.1) is in position, the level of the water is approximately 5 mm from the top of the flask. Adjust the temperature of the water to between 2 and 3 °C, and maintain it at this temperature by adding, if necessary, small quantities of crushed ice.

Prepare the test sample by vigorously shaking 100 ml of the laboratory sample with 50 ml of distilled water for 1 min in the stoppered separating funnel (4.5). Draw off the aqueous layer after allowing to settle for 3 min.

Measure 80 ml of the test sample into the test tube and place the tube, fitted with its cork stopper, the thermometer (4.4) and the glass rod (4.2), in the Dewar flask. Stir the test portion gently with the thermometer for 30 s by lightly gripping the thermometer just above the cork and moving it with a circular motion, taking care not to touch the wall of the test tube.

Remove the test tube assembly from the Dewar flask and examine the test portion. If no turbidity due to precipitation of water has occured, shake the test portion with water as described above, and recommence the test. If the test portion is turbid, replace the test tube assembly in the Dewar flask and clamp in such a position that the 0 °C point on the thermometer is level with the top of the flask. Stir the test portion gently with the thermometer, as described above, until the temperature falls to below 4 °C, taking care not to touch the wall of the test tube.

Promote crystallization by scratching the wall of the test tube with the glass rod. Stir the test portion gently for about 5 s and leave the thermometer stationary in the middle of the tube. Gently tap the thermometer throughout this stage; an automatic thermometer vibrator or tapper is recommended and is available commercially.

Record the highest temperature reached, to the nearest 0,005 K, and apply the correction as described in clause 6.

8 EXPRESSION OF RESULTS

Record the corrected temperature as the crystallizing point (wet). Convert the value to the anhydrous basis by adding 0,090 K and report this result as the crystallizing point (dry).

9 PRECISION

9.1 Repeatability *(r)*

The value below which the absolute difference between two single test results, on identical material, obtained by one operator in one laboratory using the same equipment within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability, is 0,010 °C.

9.2 Reproducibility *(R)*

The value below which the absolute difference between two single test results, on identical material, obtained by operators in different laboratories, using the standardized test method, may be expected to lie with a 95 % probability, is 0,020 °C.

10 TEST REPORT

The test report shall contain at least the following information:

a) the type and identification of the product under test;

b) a reference to this International Standard;

c) any deviation, by agreement or otherwise, from the test procedure described;

d) any unusual features noted during the determination;

e) the result of the test;

f) the date of the test.

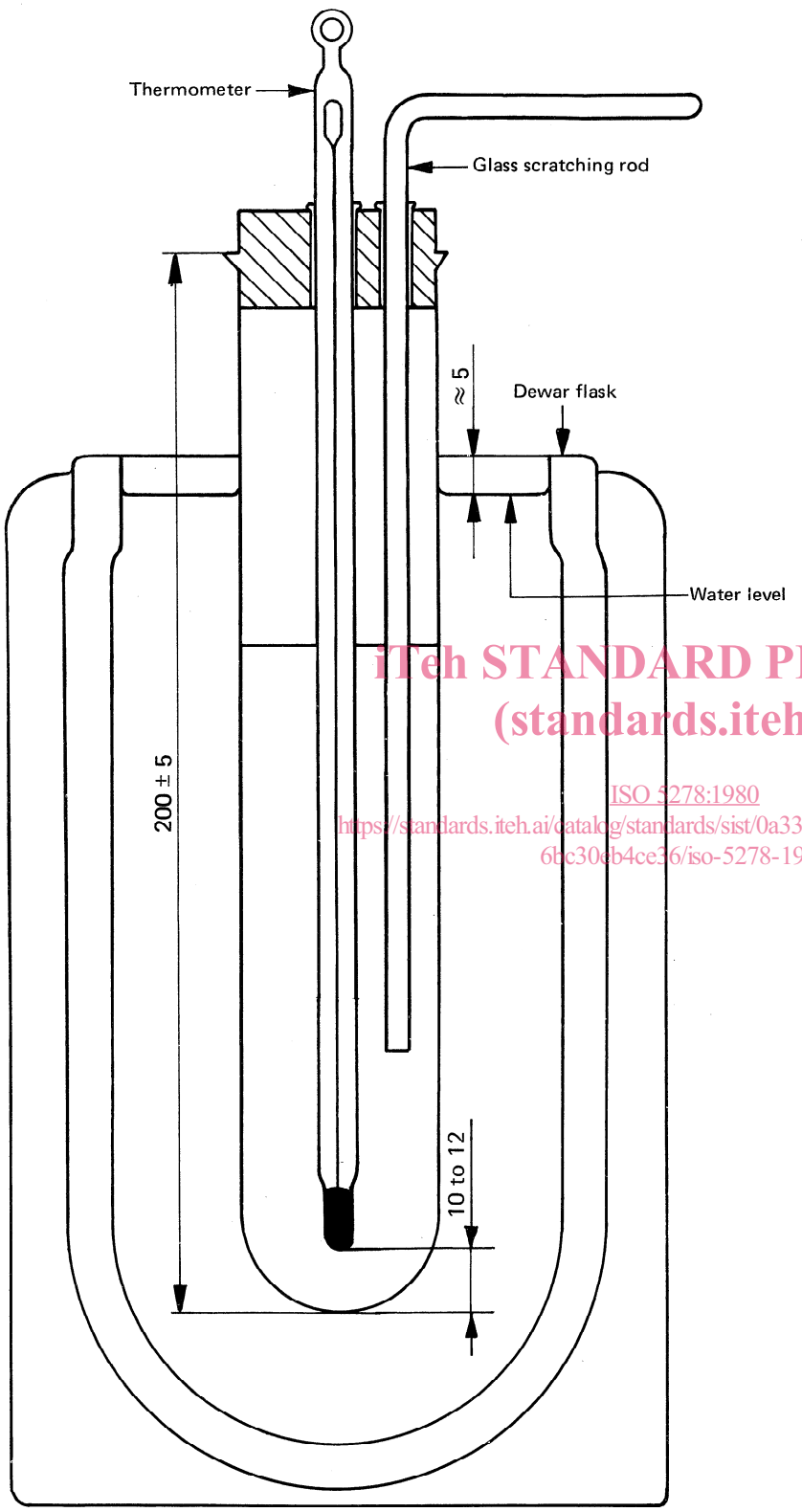
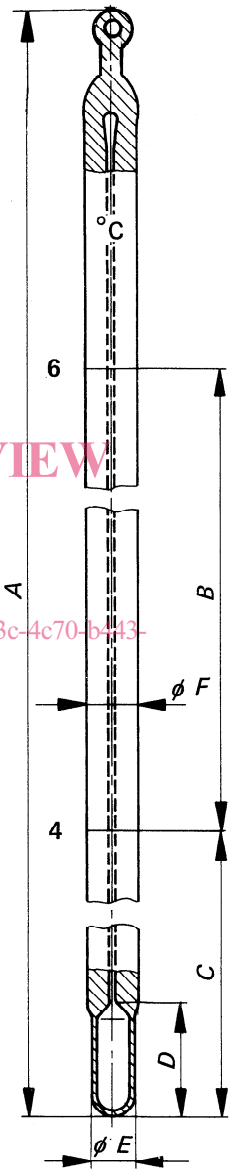


FIGURE 1 — **Crystallizing point assembled apparatus**

Dimensions in millimetres



A	250 ± 5
B	85 min.
C	95 ± 5
D	65 max.
E	< ϕ stem (*F*)
F	6 ± 0,5

FIGURE 2 — **Suitable thermometer (4.4)**

This page intentionally left blank

INTERNATIONAL STANDARD ISO 5278-1980 (E)/ERRATUM

Published 1981-12-01

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Benzene — Determination of crystallizing point

ERRATUM

Page 2

Clause 7

Paragraph 4 :

a) in line 3, correct the spelling of "occurred";

b) in line 6, replace "0 °C" by "5 °C".

This page intentionally left blank

This page intentionally left blank