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R 1392

DETERMINATION OF CRYSTALLIZING POINT

GENERAL METHOD

1st EDITION

April 1970

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 1392, *Determination of crystallizing point – General method*, 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 to the adoption of Draft ISO Recommendation No. 1392 which was circulated to all the ISO Member Bodies for enquiry in June 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Iran	South Africa, Rep. of
Belgium	Israel	Spain
Chile	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
France	New Zealand	Turkey
Germany	Peru	U.A.R.
Hungary	Portugal	United Kingdom
India	Romania	U.S.S.R.

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1970, to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1392 | April 1970

DETERMINATION OF CRYSTALLIZING POINT

GENERAL METHOD

1. SCOPE

This ISO Recommendation describes a general method for the determination of crystallizing points in the range from about −50 °C to about + 250 °C.

The crystallizing point can be determined directly on the sample as received, or on the dried sample, or on both. In which of these conditions the sample is to be tested and, if the determination is to be made with the dried sample, what method of drying is to be used, will be stated in the specific test method for each material.

2. PRINCIPLE

Cooling the liquid or liquefied sample, and determination of the crystallizing point by observation of the temperature during crystallization under defined conditions.

3. REAGENTS

3.1 *Solid carbon dioxide.*

3.2 *Acetone.*

3.3 *Ice.*

3.4 *Calcium sulphate,* dried at 170 °C.

Dry the calcium sulphate ($CaSO_4 .2H_2 O$) for 24 hours at 170 °C and then store it in an airtight container.

4. APPARATUS

Ordinary laboratory apparatus and the apparatus shown in Figures 1, 2, 3 and 4 comprising :

4.1 *Crystallizing tube*

External diameter :	25 mm (approximately)
Length :	150 mm (approximately)

4.2 *Outer protection tube*

Internal diameter :	28 mm (approximately)
Length :	120 mm (approximately)
Wall thickness :	2 mm (approximately)

4.3 *Stirrer,* of glass or stainless steel, with a loop approximately 20 mm in diameter; it may be operated by hand or mechanically, to provide approximately one 30 mm stroke per second.

4.4 *Precision thermometer* graduated at intervals of 0.1 °C, with a known maximum error of 0.1 °C, and with the range specified for the test in the relevant specification for the product in question.

4.5 *Dewar vessel of 500 ml* (approximate) capacity, containing the appropriate cooling mixture (carbon dioxide/acetone or ice/water or water) and provided with a suitable laboratory thermometer. An example of such a Dewar vessel is given in Figure 2, but other vessels of the same capacity may also be used.

4.6 *Dewar vessel,* as shown in Figure 3. (It is not necessary for the inner surfaces of the vessel to be silvered.)

4.7 *Heating bath,* as shown in Figure 4, containing silicone oil or other appropriate liquid heating medium, and provided with a suitable laboratory thermometer.

5. PROCEDURE

5.1 Preparation of the sample for the direct determination of the crystallizing point on the sample as received

5.1.1 *Liquid products.* Fill the crystallizing tube to a depth of approximately 60 mm with the untreated sample and proceed as described in clause 5.3.

5.1.2 *Solid products.* Before the determination of the crystallizing point of these products, they should be melted in a waterbath, drying oven or oilbath (this can be carried out in the crystallizing tube, using the heating bath shown in Figure 4), care being taken to ensure that the temperature of the molten product does not exceed its melting point by more than a few degrees. Fill the crystallizing tube to a depth of approximately 60 mm with the molten sample and proceed as described in clause 5.3.

5.2 Preparation of the sample for the determination of the crystallizing point on the dried sample

5.2.1 *Liquid products.* Liquid products of normal water content (i.e. 2 % maximum) should be dried in the crystallizing tube (4.1) by the direct addition of the calcium sulphate (3.4). Fill the crystallizing tube (4.1) to a depth of approximately 60 mm with the liquid sample, add the calcium sulphate (3.4) – 2 to 5 g are usually required – and proceed as described in clause 5.3.

In some cases other methods of drying may be required, and these will be described in the specification for the particular material.

5.2.2 *Solid products.* The drying method for solid products depends on the water content of the sample and on the value of the crystallizing point.

5.2.2.1 PRODUCTS WITH A LOWER WATER CONTENT (i.e. 2 % maximum). Solid products with melting points below approximately 150 °C should be dried with the calcium sulphate (3.4). Fill the crystallizing tube (4.1) to a depth of approximately 60 mm with the molten sample, add the calcium sulphate (3.4) – 2 to 5 g are usually required – and proceed as described in clause 5.3.

Solid products with melting points above approximately 150 °C should be dried either in an oven at 60 °C, or under vacuum, or by air drying and the determination of the crystallizing point then carried out on the molten sample. Fill the crystallizing tube (4.1) to a depth of approximately 60 mm with the molten sample and proceed as described in clause 5.3.

For these higher melting point samples the method and time of drying will be given in the specification for the particular material; alternative drying methods may be given in some cases for a material, whatever its melting point.

5.2.2.2 PRODUCTS WITH A HIGHER WATER CONTENT. Samples with a higher water content (for example pastes) should in every case be dried before determination of the crystallizing point, for example in an oven at 60 °C or under vacuum, etc. The determination is then carried out after the sample has been melted. Fill the crystallizing tube (4.1) to a depth of approximately 60 mm with the molten sample and proceed as described in clause 5.3.

In addition, with products melting below approximately 150 °C, some calcium sulphate (3.4) (normally 2 to 5 g), should be added in the crystallizing tube (4.1) before the determination is commenced.

The method of drying, the time of drying or alternative drying methods should be given in the specification for the particular material.

NOTE. – Before the determination of the crystallizing point, solid samples should be melted in a waterbath, drying oven or oilbath (this can be carried out in the crystallizing tube using the heating bath shown in Figure 4), care being taken to ensure that the temperature of the molten sample does not exceed its melting point by more than a few degrees.

5.3 Preparation of the apparatus

Insert the stirrer into the crystallizing tube (4.1) prepared in accordance with clauses 5.1 and 5.2. Secure the specified thermometer vertically in the liquid or molten product with its bulb approximately 15 mm above the bottom of the crystallizing tube. Fit the outer tube (4.2) to this assembly (if necessary by means of a cork shive or a rubber sleeve), and place the whole in position as follows :

(*a*) *for crystallizing points in the range from room temperature down to approximately −50 °C* : in the Dewar vessel (4.5) filled with the appropriate cooling mixture (carbon dioxide/acetone or ice/water or water) at a temperature approximately 3 to 5 °C below the crystallizing point to be determined;

(*b*) *for crystallizing points in the range from room temperature up to approximately 100 °C* : in the Dewar vessel (4.6);

(*c*) *for crystallizing points in the range from approximately 100 to approximately 250 °C* : in the heating bath (4.7) at a temperature approximately 5 to 7 °C below the crystallizing point to be determined.

5.4 Determination

Check that the sample is still liquid at this stage; stir the sample and take temperature readings (these should decrease uniformly at first, then rise suddenly as the substance crystallizes; sometimes the temperature remains constant for a short time). If the temperature rise exceeds 1 to 2 °C, this indicates that excessive supercooling has occurred. In this case the determination should be repeated, seeding the liquid or the melt to prevent excessive supercooling. Read the highest temperature attained after crystallization and adjust the reading for scale error and emergent stem correction, if any. Record this temperature to the nearest 0.1 °C as the crystallizing point of the substance under test.

NOTES

1. Instead of using a stirrer, the stirring can be effected by hand with the thermometer, but in this case care must be taken that the latter does not come into contact with the walls of the crystallizing tube.

2. For the correct determination of the crystallizing point of a solid sample, it is necessary that the substance should melt during the test without any decomposition. That this condition is fulfilled may be checked by repeating the test and comparing the two results. If the two crystallizing temperatures are the same, this indicates that the above condition has been met.

6. EXPRESSION OF RESULTS

Record the crystallizing point thus determined to the nearest 0.1 °C, indicating the condition of the sample, i.e. whether tested in the dried or the undried condition, or in both.

7. TEST REPORT

The test report should give the following particulars :

(*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 included in this ISO Recommendation or regarded as optional.

Approximate dimensions in millimetres

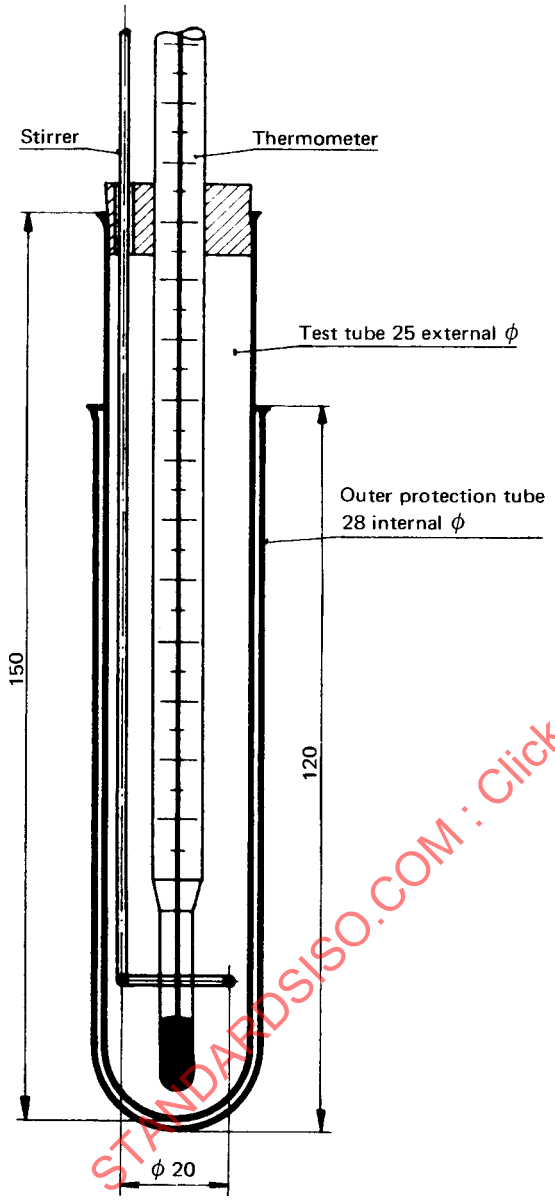

FIGURE 1

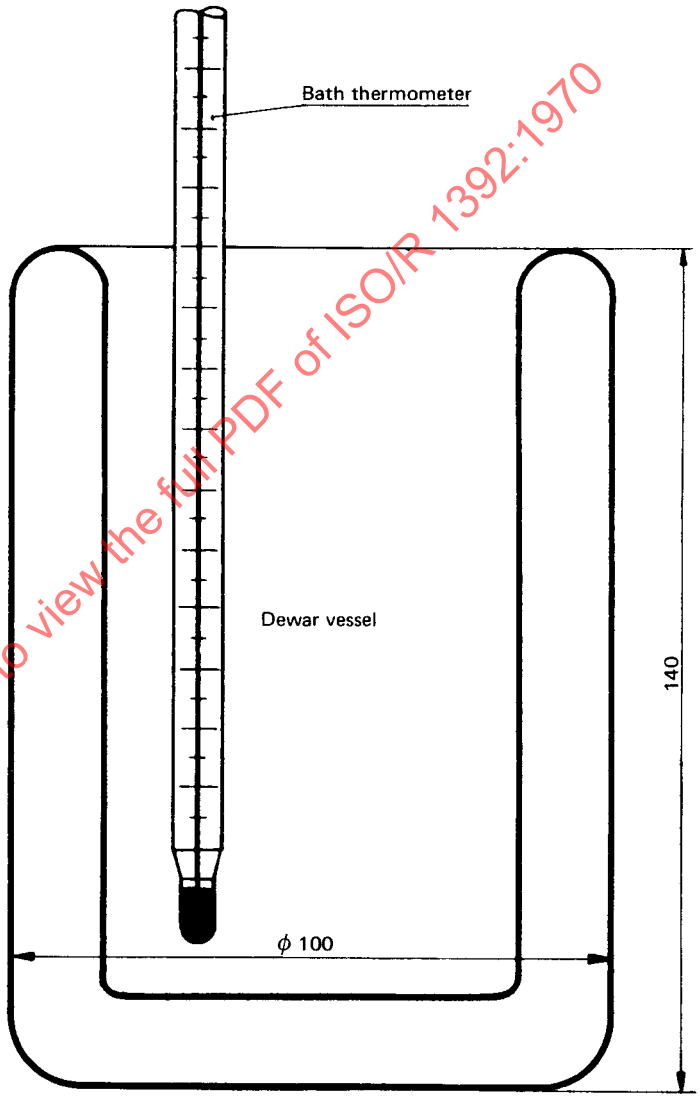

FIGURE 2