SLOVENSKI STANDARD
SIST ISO 1392:1995

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Dolo anje kristalizacijske to ke - Splo na metoda

Determination of crystallizing point -- General method

Détermination du point de cristallisation -- Méthode générale

Ta slovenski standard je istoveten z: **ISO 1392:1977**

ICS:

71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

SIST ISO 1392:1995 **en**

INTERNATIONAL STANDARD 1392

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Determination of crystallizing point — General method

Détermination du point de cristallisation — Méthode générale

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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.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 1392-1970 and found it technically suitable for transformation. International Standard ISO 1392 therefore replaces ISO Recommendation R 1392-1970, to which it is technically identical.

ISO Recommendation R 1392 had been approved by the member bodies of the following countries :

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

No member body had expressed disapproval of the Recommendation.

No member body disapproved the transformation of the Recommendation into an International Standard.

Printed in Switzerland

Determination of crystallizing point — General method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a general method for the determination of crystallizing points in the range from about − 50 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 Acetone.

3.2 Solid carbon dioxide.

3.3 Ice.

3.4 Calcium sulphate, dried at about 170 °C.

Dry calcium sulphate dihydrate ($CaSO_4.2H_2O$) for 24 h at about 170 °C, allow to cool in a desiccator 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 the following items :

4.1 Crystallizing tube, external diameter approximately 25 mm and length approximately 150 mm.

4.2 Outer protection tube, internal diameter approximately 28 mm, length approximately 120 mm and wall thickness approximately 2 mm.

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 scale error of 0,1 °C, and with the range stated in the specific test method for the particular material.

4.5 Dewar vessel of approximately 500 ml 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 suitable 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 (4.1) to a depth of approximately 60 mm with the untreated sample and proceed as specified in 5.3.

5.1.2 *Solid products*

Before the determination of the crystallizing point of these products, they shall be melted in a water bath, drying oven or oil bath [this can be carried out in the crystallizing tube (4.1), using the heating bath (4.7)], 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 specified in 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. less than or equal to 2 % (*m/m*)] shall be dried in the crystallizing tube (4.1) by addition of calcium sulphate. Fill the crystallizing

tube 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 specified in 5.3.

In some cases, other methods of drying may be required; these will be specified in the specific test method 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. less than or equal to 2 % (*m/m*)]

Solid products with melting points below approximately 150 °C shall be dried with calcium sulphate. 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 specified in 5.3.

Solid products with melting points above approximately 150 °C shall be dried 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 specified in 5.3.

For these higher melting point samples, the method and time of drying will be indicated in the specific test method for the particular material; in some cases, alternative drying methods may be given 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) shall 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 on the molten sample. Fill the crystallizing tube (4.1) to a depth of approximately 60 mm with the molten sample and proceed as specified in 5.3.

In addition, with products melting below approximately 150 °C, add some calcium sulphate (3.4) (normally 2 to 5 g), in the crystallizing tube before commencing the determination.

The method of drying, the time of drying or alternative drying methods will be indicated in the specific test method for the particular material.

NOTE — Before the determination of the crystallizing point, solid samples shall be melted in a water bath, drying oven or oil bath [this can be carried out in the crystallizing tube using the heating bath (4.7)], 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 (4.3) into the crystallizing tube (4.1) prepared as specified in 5.1 and 5.2. Secure the specified thermometer (4.4) 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 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 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 ocurred. 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. Record this temperature to the nearest 0,1 °C as the crystallizing point of the product under test.

NOTES

1 The stirrer may be omitted and the stirring carried out by hand using the thermometer but care should be taken that the thermometer does not touch the walls of the crystallizing tube.

2 For the correct determination of the crystallizing point of a solid sample, it is necessary that the product 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 shall include 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 International Standard or regarded as optional.

Approximate dimensions in millimetres

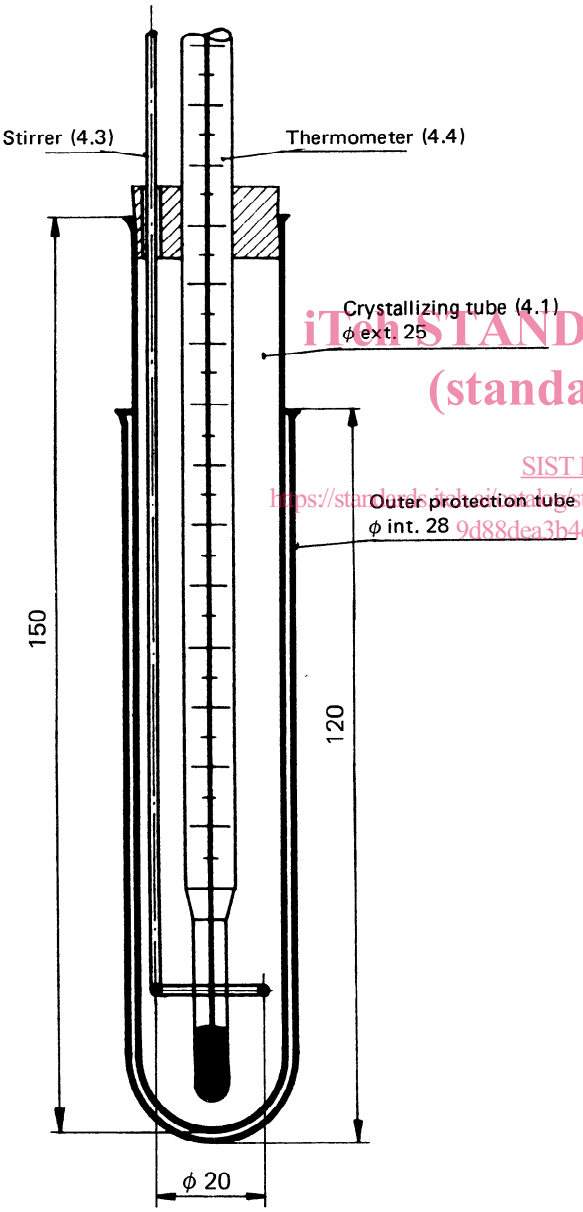

FIGURE 1 — **Apparatus for determination of crystallizing point**

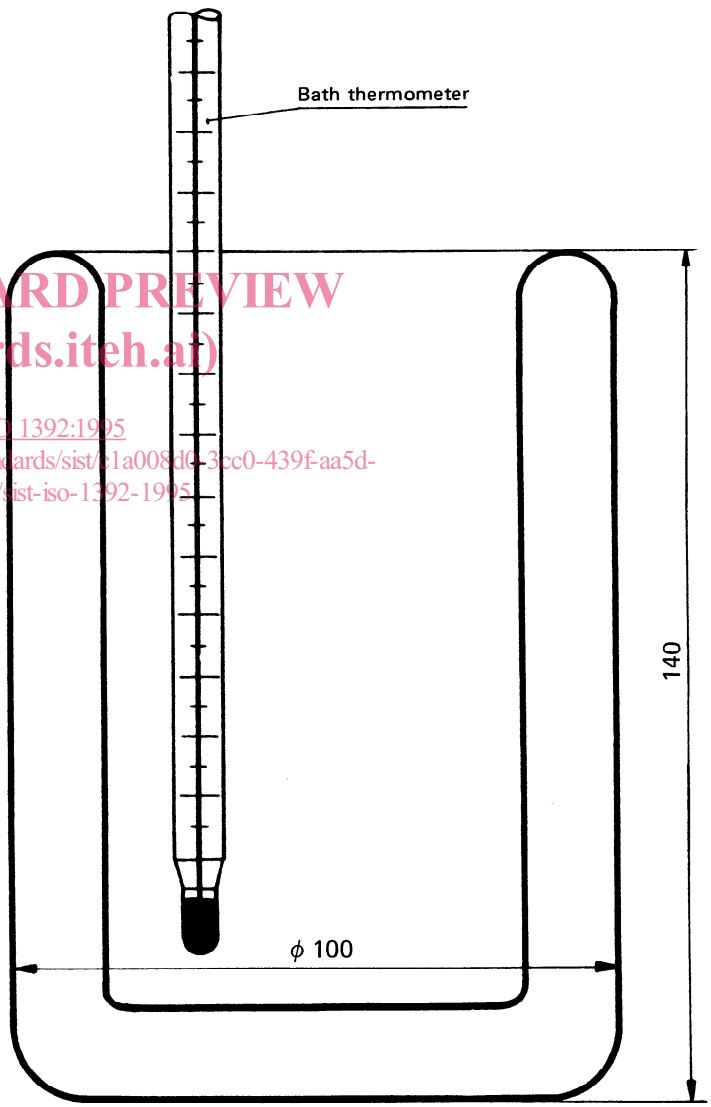

FIGURE 2 — **Dewar vessel (4.5)**

Approximate dimensions in millimetres

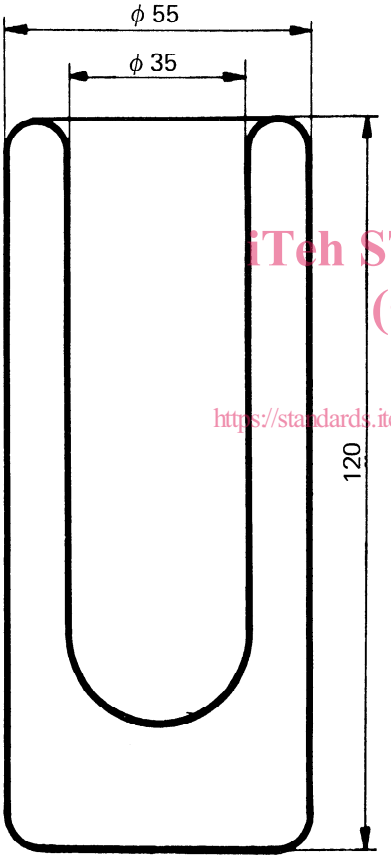

FIGURE 3 — **Dewar vessel (4.6)**

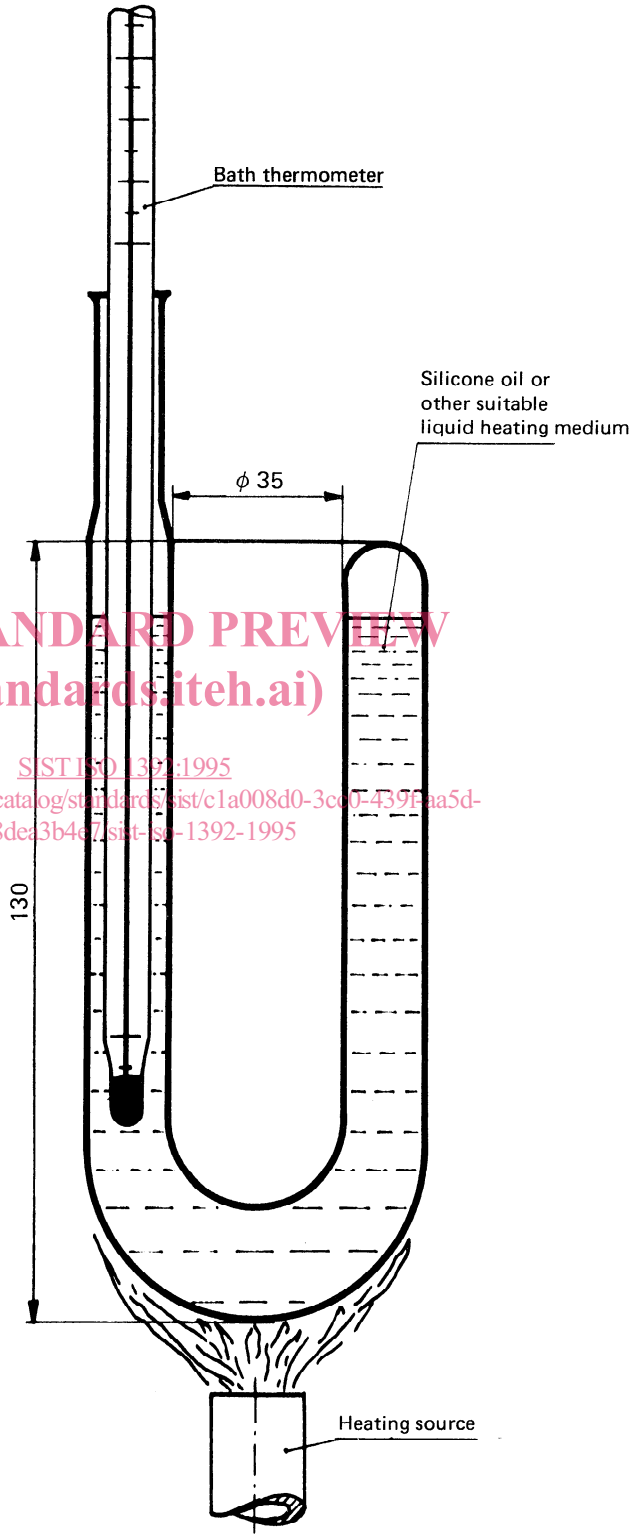

FIGURE 4 — **Heating bath**