

***Calibration of the apparatus.*** The apparatus may be calibrated using melting point reference substances such as those of the World Health Organisation or other appropriate substances.

融点

FCC

## MELTING RANGE OR TEMPERATURE

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For purposes of the FCC, the melting range or temperature of a solid is defined as those points of temperature within which or the point at which the solid coalesces and is completely melted when determined as directed below. Any apparatus or method capable of equal accuracy may be used. The accuracy should be checked frequently by the use of one or more of the six USP Melting Point Reference Standards, preferably the one that melts nearest the melting temperature of the compound to be tested.

Five procedures for the determination of melting range or temperature are given herein, varying in accordance with the nature of the substance. When no class is designated in the monograph, use the procedure for *Class I*.

The procedure known as the mixed melting point determination, whereby the melting range of a solid under test is compared with that of an intimate mixture of equal parts of the solid and an authentic specimen of it, may be used as a confirmatory identification test. Agreement of the observations on the original and the mixture usually constitutes reliable evidence of chemical identity.

**Apparatus** The melting range apparatus consists of a glass container for a bath of colorless fluid, a suitable stirring device, an accurate thermometer (see Appendix I), and a controlled source of heat. The bath fluid is selected consistent with the temperature required, but light paraffin is used generally, and certain liquid silicones are well adapted to the higher temperature ranges. The fluid is deep enough to permit immersion of the thermometer to its specified immersion depth so that the bulb is still about 2 cm above the bottom of the bath. The heat may be supplied electrically or by an open flame. The capillary tube is about 10 cm long, with an internal diameter of 0.8 to 1.2 mm, and with walls 0.2 to 0.3 mm thick.

The thermometer is preferably one that conforms to the specifications provided under *Thermometers*, Appendix I, selected for the desired accuracy and range of temperature.

**Procedure for Class I** Reduce the sample to a very fine powder, and unless otherwise directed, render it anhydrous when it contains water of hydration by drying it at the temperature specified in the monograph, or when the substance contains no water of hydration, dry it over a suitable desiccant for 16 to 24 h.

Charge a capillary glass tube, one end of which is sealed, with a sufficient amount of the dry powder to form a column in the bottom of the tube 2.5 to 3.5 mm high when packed down as closely as possible by moderate tapping on a solid surface.

Heat the bath until a temperature approximately  $30^{\circ}$  below the expected melting point is reached, attach the capillary tube to the thermometer, and adjust its height so that the material in the capillary is level with the thermometer bulb. Return the thermometer to the bath, continue the heating, with constant stirring, at a rate of rise of approximately  $3^{\circ}/\text{min}$  until a temperature  $3^{\circ}$  below the expected melting point is attained, then carefully regulate the rate to about  $1^{\circ}$  to  $2^{\circ}/\text{min}$  until melting is complete.

The temperature at which the column of the sample is observed to collapse definitely against the side of the tube at any point is defined as the beginning of melting, and the temperature at which the sample becomes liquid throughout is defined as the end of melting. The two temperatures fall within the limits of the melting range.

**Procedure for Class Ia** Prepare the sample and charge the capillary glass tube as directed for *Class I*. Heat the bath until a temperature  $10^{\circ} \pm 1^{\circ}$  below the expected melting range is reached, then introduce the charged tube, and heat at a rate of rise of  $3^{\circ} \pm 0.5^{\circ}/\text{min}$  until melting is complete. Record the melting range as for *Class I*.

**Procedure for Class Ib** Place the sample in a closed container, and cool to  $10^{\circ}$  or lower for at least 2 h. Without previous powdering, charge the cooled material into the capillary tube as directed for *Class I*, immediately place the charged tube in a vacuum desiccator, and dry at a pressure not exceeding 20 mm Hg for 3 h. Immediately upon removal from the desiccator, fire-seal the open end of the tube. As soon as is practicable, proceed with the determination of the melting range as follows: Heat the bath until a temperature of  $10^{\circ} \pm 1^{\circ}$  below the expected melting range is reached, then introduce the charged tube, and heat at a rate of rise of  $3^{\circ} \pm 0.5^{\circ}/\text{min}$  until melting is complete. Record the melting range as directed in *Class I*.

If the particle size of the material is too large for the capillary, precool the sample as directed above, then with as little pressure as possible, gently crush the particles to fit the capillary, and immediately charge the tube.

**Procedure for Class II** Carefully melt the material to be tested at as low a temperature as possible, and draw it into a capillary tube that is left open at both ends to a depth of about 10 mm. Cool the charged tube at  $10^{\circ}$ , or lower, for 24 h, or in contact with ice for at least 2 h. Then attach the tube to the thermometer by means of a rubber band, adjust it in a water bath so that the upper edge of the material is 10 mm below the water level, and heat as directed for *Class I*, except within  $5^{\circ}$  of the expected melting temperature, regulate the rate of rise of temperature to  $0.5^{\circ}$  to  $1.0^{\circ}/\text{min}$ . The temperature at which the material is observed to rise in the capillary tube is the melting temperature.

**Procedure for Class III** Melt a quantity of the substance slowly, while stirring, until it reaches a temperature of 90° to 92°. Remove the source of heat, and allow the molten substance to cool to a temperature of 8° to 10° above the expected melting point. Chill the bulb of an ASTM 14C thermometer (see Appendix 1) to 5°, wipe it dry, and while it is still cold, dip it into the molten substance so that approximately the lower half of the bulb is submerged. Withdraw it immediately, and hold it vertically away from the heat until the wax surface dulls, then dip it for 5 min into a water bath having a temperature not higher than 16°.

Fix the thermometer securely in a test tube so that the lower point is 15 mm above the bottom of the test tube. Suspend the test tube in a water bath adjusted to about 16°, and raise the temperature of the bath at the rate of 2°/min to 30°, then change to a rate of 1°/min, and note the temperature at which the first drop of melted substance leaves the thermometer. Repeat the determination twice on a freshly melted portion of the sample. If the variation of three determinations is less than 1°, take the average of the three as the melting point. If the variation of three determinations is greater than 1°, make two additional determinations and take the average of the five.

## MELTING RANGE

Before determining the melting range of a substance, the sample should be dried under the conditions specified for Loss on Drying in the individual monograph. If a temperature is not specified in the monograph, the sample should be dried for 24 h in a desiccator.

Transfer a quantity of the dried powder to a dry capillary-tube about 10 cm long and sealed at one end (thickness of the wall, 0.10-0.15 mm; internal diameter 0.9-1.1 mm) and pack the powder by tapping the tube on a hard surface so as to form a tightly-packed column 2-4 mm in height.

Attach the capillary-tube and its contents to a standard thermometer so that the closed end is at the level of the middle of the bulb, and heat in a suitable apparatus containing an appropriate liquid (liquid paraffin or silicone oil) and fitted with a stirring device and an auxiliary thermometer. Regulate the rise in temperature during the first period to 3° per min. When the temperature has risen to 5° below the lowest figure of the range for the substance being tested, heat more slowly: if no other directions are given, the rate of rise in temperature should be 1°-2° per min.

Unless otherwise directed, read the temperature at which the substance is observed to form droplets against the side of the tube and the temperature at which it is completely melted, as indicated by the formation of a definitive meniscus.

To the temperature readings, apply the emergent-stem correction determined as follows:

Before starting the determination of the melting range, adjust the auxiliary thermometer so that the bulb touches the standard thermometer at a point midway between the graduation for the expected melting range and the surface of the heating material. When the substance has melted, read the temperature on the auxiliary thermometer. Calculate the correction to be added to the temperature reading of the standard thermometer from the following formula:

$$0.00015 N(T - t)$$

where T is the temperature reading of the standard thermometer;

t is the temperature reading of the auxiliary thermometer;

N is the number of degrees of the scale of the standard thermometer between the surface of the heating material and the level of the mercury.

The statement "melting range, a°-b°" means that the corrected temperature at which the material is observed to form droplets must be at least a°, and that the material must be completely melted at the corrected temperature b°.

## 11. 凝固点測定法

凝固点は、次の方法で測定する。

### 装 置

概略は、第1図による。

A：ガラス製円筒（内外の両壁に曇り止めのためシリコン油を塗る。）

B：試料容器（硬質ガラス製試験管で、管の両壁に曇り止めのためシリコン油を塗る。ただし、試料に接する部分には塗らない。A中に差し込み、コルク栓で固定する。）

C：標線

D：ガラス製又はプラスチック製冷却浴

E：ガラス製又はステンレス製かき混ぜ棒（径3 mm、下端を外径18 mmの輪状にしたもの）

F：浸線付温度計（棒状）

G：補助温度計

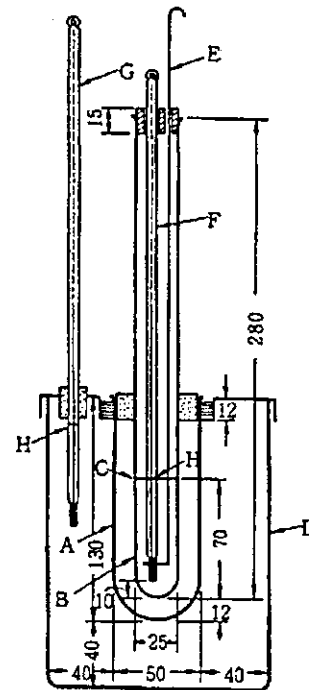
H：浸線

### 操作法

ガラス製又はプラスチック製冷却浴Dに予想される凝固点よりも5℃低い温度の水をほぼ全満する。試料が常温で液体の場合は、Dの水を予想した凝固点より10～15℃低くする。試料を試料容器Bの標線Cまで入れる。試料が固体の場合は、予想される凝固点よりも20℃以上高くないように注意して加温して溶かし、Bに入れる。Bをガラス製円筒A中に差し込み、浸線付温度計Fの浸線Hを試料のメニスカスに合わせた後、試料の温度が予想される凝固点よりも5℃高い温度まで冷却されたとき、かき混ぜ棒Eを毎分60～80回の割合で上下に動かし、30秒間ごとに温度を読む。温度は、徐々に下がるが、結晶が析出し始めて温度が一定になるか、又はやや上がり始めたとき、かき混ぜをやめる。

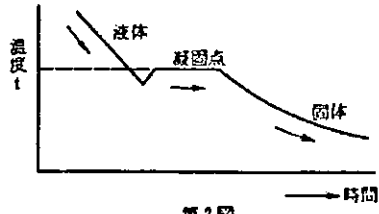
通例、温度は、上昇の後にしばらく一定になる。この維持された最高温度（Fの示度）を読み取る（第2図）。温度上昇の起こらない場合は、しばらく静止した温度を読み取る（第3図）。連続4回以上の読み取り温度の範囲が0.2℃以内のとき、その平均値をとり、凝固点とする。

なお試料中に混在する不純物が多い場合は、凝固点曲線は、第2図のようにはならず、第3図、第4図又は第5図のようになる。第4図と第5図の場合は、固相と液相の延長線の交点をグラフから求めて凝固点とし、第3図の場合は、第2図に準ずる。

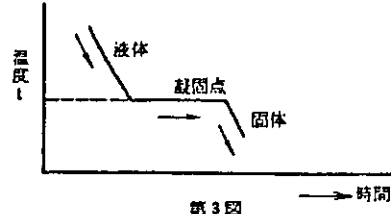


(単位 mm)

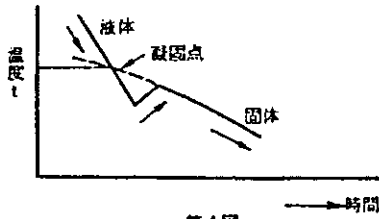
第1図



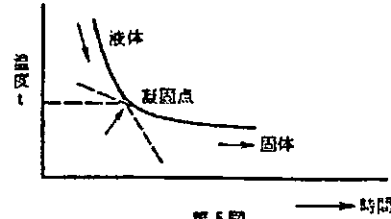
第2図



第3図



第4図



第5図

注意：過冷の状態が予想される場合は、Bの内壁をこするか、温度が予想される凝固点に近づいたとき、固体試料の小片を投入して凝固を促進させる。





### 2.2.18. FREEZING POINT

The freezing point is the maximum temperature occurring during the solidification of a supercooled liquid.

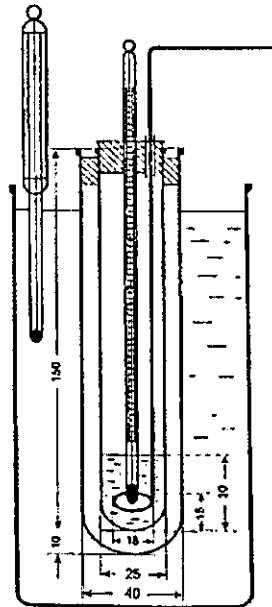


Figure 2.2.18.-1. - Apparatus for the determination of freezing point  
Dimensions in millimetres

*Apparatus.* The apparatus (see Figure 2.2.18.-1) consists of a test-tube about 25 mm in diameter and 150 mm long placed inside a test-tube about 40 mm in diameter and 160 mm long. The inner tube is closed by a stopper which carries a thermometer about 175 mm long and graduated in 0.2 °C fixed so that the bulb is about 15 mm above the bottom of the tube. The stopper has a hole allowing the passage of the stem of a stirrer made from a glass rod or other suitable material formed at one end into a loop of about 18 mm overall diameter at right angles to the rod. The inner tube with its jacket is supported centrally in a 1 litre beaker containing a suitable cooling liquid to within 20 mm of the top. A thermometer is supported in the cooling bath.

*Method.* Place in the inner tube sufficient quantity of the liquid or previously melted substance to be examined, to cover the thermometer bulb and determine the approximate freezing point by cooling rapidly. Place the inner tube in a bath about 5 °C above the approximate freezing point until all but the last traces of crystals are melted. Fill the beaker with water or a saturated solution of sodium chloride, at a temperature about 5 °C lower than the expected freezing point, insert the inner tube into the outer tube, ensuring that some seed crystals are present, and stir thoroughly until solidification takes place. Note the highest temperature observed during solidification.

## B. Determination of freezing point

(Ph. Eur. method 2.2.18)

The freezing point is the maximum temperature occurring during the solidification of a supercooled liquid.

### *Apparatus*

A test tube about 150 mm  $\times$  25 mm placed inside a test tube about 160 mm  $\times$  40 mm; the inner tube is closed by a stopper which carries a stirrer and a thermometer (about 175 mm long and with 0.2° graduations) fixed so that the bulb is about 15 mm above the bottom of the tube (Fig. 5B-1). The stirrer is made from a glass rod or other suitable material formed at one end into a loop of about 18 mm overall diameter at right angles to the rod. The inner tube with its jacket is supported centrally in a litre beaker containing a suitable cooling liquid to within 20 mm of the top. A thermometer is supported in the cooling bath.

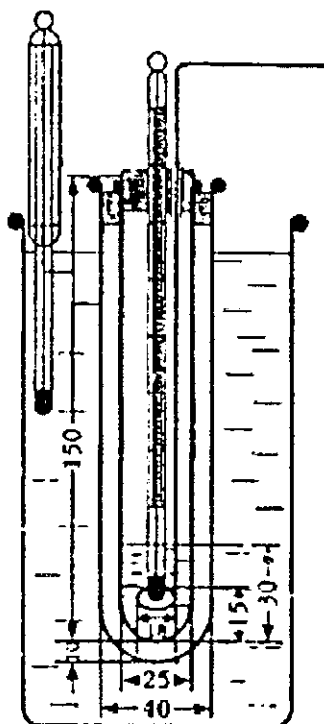


Fig. 5B-1 Apparatus for Determination of Freezing Point  
Dimensions in mm

### *Method*

Place a quantity of the substance, previously melted if necessary, in the inner tube such that the thermometer bulb is well-covered and determine the approximate freezing point

**Procedure** Adjust the temperature of the cooling bath to about 5° below the expected solidification point. Fit the thermometer and stirrer with a cork stopper so that the thermometer is centered and the bulb is about 20 mm from the bottom of the sample container. Transfer a sufficient amount of the sample, previously melted if necessary, into the sample container to fill it to a depth of about 90 mm when in the molten state. Place the thermometer

and stirrer in the sample container, and adjust the thermometer so that the immersion line will be at the surface of the liquid and the end of the bulb is  $20 \pm 4$  mm from the bottom of the sample container. When the temperature of the sample is about 5° above the expected solidification point, place the assembled sample tube in the air jacket.

Allow the sample to cool while stirring, at the rate of 20 to 30 strokes/min, in such a manner that the stirrer does not touch the thermometer. Stir the sample continuously during the remainder of the test.

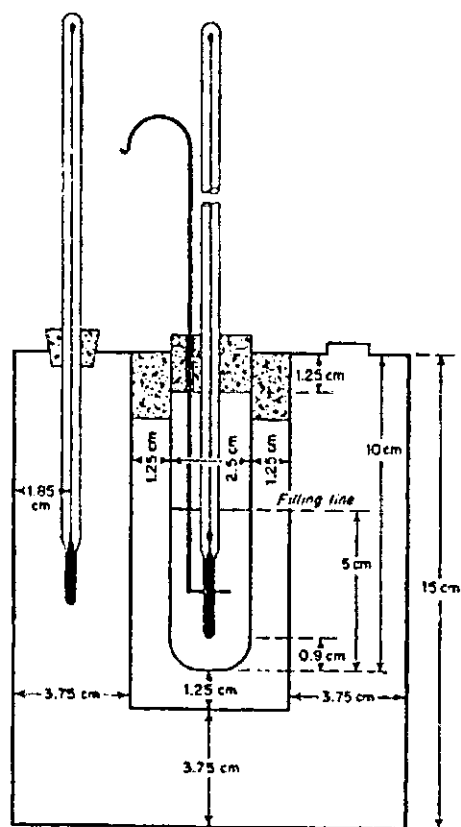
The temperature at first will gradually fall, then will become constant as crystallization starts and continues under equilibrium conditions, and finally will start to drop again. Some chemicals may supercool slightly below (0.5°) the solidification point; as crystallization begins, the temperature will rise and remain constant as equilibrium conditions are established. Other products may cool more than 0.5° and cause deviation from the normal pattern of temperature change. If the temperature rise exceeds 0.5° after the initial crystallization begins, repeat the test, and seed the melted compound with small crystals of the sample at 0.5° intervals as the temperature approaches the expected solidification point. Crystals for seeding may be obtained by freezing a small sample in a test tube directly in the cooling bath. It is preferable that seed of the stable phase be used from a previous determination.

Observe and record the temperature readings at regular intervals until the temperature rises from a minimum, due to supercooling, to a maximum and then finally drops. The maximum temperature reading is the solidification point. Readings 10 s apart should be taken to establish that the temperature is at the maximum level and should continue until the drop in temperature is established.

## (651) CONGEALING TEMPERATURE

The temperature at which a substance passes from the liquid to the solid state upon cooling is a useful index to purity if heat is liberated when the solidification takes place, provided that any impurities present dissolve in the liquid only, and not in the solid. Pure substances have a well-defined freezing point, but mixtures generally freeze over a range of temperatures. For many mixtures, the congealing temperature, as determined by strict adherence to the following empirical methods, is a useful index of purity. The method for determining congealing temperatures set forth here is applicable to substances that melt between  $-20^{\circ}$  and  $150^{\circ}$ , the range of the thermometer used in the bath. The congealing temperature is the maximum point (or lacking a maximum, the point of inflection) in the temperature-time curve.

**Apparatus**—Assemble an apparatus similar to that illustrated, in which the container for the substance is a 25- × 100-mm test tube. This is provided with a suitable, short-range thermometer suspended in the center, and a wire stirrer, about 30 cm long, bent at its lower end into a horizontal loop around the thermometer. Use a thermometer having a range not exceeding  $30^{\circ}$ , graduated in  $0.1^{\circ}$  divisions, and calibrated for, but not used at, 76-mm immersion. A suitable series of thermometers, covering a range from  $-20^{\circ}$  to  $+150^{\circ}$ , is available as the ASTM E1 series 89C through 96C. Other temperature-measuring devices may be used if they are validated for this procedure (see *Thermometers* (21)). Dimensions should be within  $\pm 20\%$  of those given in the illustration.



Congealing Temperature Apparatus

The specimen container is supported, by means of a cork, in a suitable water-tight cylinder about 50 mm in internal diameter and 11 cm in length. The cylinder, in turn, is supported in a suitable bath sufficient to provide not less than a 37-mm layer surrounding the sides and bottom of the cylinder. The outside bath is provided with a suitable thermometer.

**Procedure**—Melt the substance, if a solid, at a temperature not exceeding 20° above its expected congealing point, and pour it into the test tube to a height of 50 to 57 mm. Assemble the apparatus with the bulb of the test tube thermometer immersed halfway between the top and bottom of the specimen in the test tube. Fill the bath to about 12 mm from the top of the tube with suitable fluid at a temperature 4° to 5° below the expected congealing point.

In case the substance is a liquid at room temperature, carry out the determination using a bath temperature about 15° below the expected congealing point.

When the test specimen has cooled to about 5° above its expected congealing point, adjust the bath to a temperature 7° to 8° below the expected congealing point. Stir the specimen continuously during the remainder of the test by moving the loop up and down between the top and bottom of the specimen, at a regular rate of 20 complete cycles per minute.

Congelation frequently may be induced by rubbing the inner walls of the test tube with the thermometer, or by introducing a small fragment of the previously congealed substance. Pronounced supercooling may cause deviation from the normal pattern of temperature changes. If the latter occurs, repeat the test, introducing small particles of the material under test in solid form at 1° intervals as the temperature approaches the expected congealing point.

Record the reading of the test tube thermometer every 30 seconds. Continue stirring only so long as the temperature is gradually falling, stopping when the temperature becomes constant or starts to rise

slightly. Continue recording the temperature in the test tube every 30 seconds for at least 3 minutes after the temperature again begins to fall after remaining constant.

The average of not less than four consecutive readings that lie within a range of 0.2° constitutes the congealing temperature. These readings lie about a point of inflection or a maximum, in the temperature-time curve, that occurs after the temperature becomes constant or starts to rise and before it again begins to fall. The average to the nearest 0.1° is the congealing temperature.

## SOLIDIFICATION POINT

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**Scope** This method is designed to determine the solidification point of food-grade chemicals having appreciable heats of fusion. It is applicable to chemicals having solidification points between  $-20^{\circ}$  and  $+150^{\circ}$ . Necessary modifications will be noted in individual monographs.

**Definition** Solidification Point is an empirical constant defined as the temperature at which the liquid phase of a substance is in approximate equilibrium with a relatively small portion of the solid phase. It is measured by noting the maximum temperature reached during a controlled cooling cycle after the appearance of a solid phase.

The solidification point is distinguished from the freezing point in that the latter term applies to the temperature of equilibrium between the solid and liquid state of pure compounds.

Some chemical compounds have more than one temperature at which there may be an equilibrium between the solid and liquid state depending on the crystal form of the solid that is present.

**Apparatus** The apparatus illustrated in Figs. 3 and 4 consists of the components described in the following paragraphs.

**Thermometer** A thermometer having a range not exceeding  $30^{\circ}$ , graduated in  $0.1^{\circ}$  divisions, and calibrated for 76-mm immersion should be employed. A satisfactory series of thermometers, covering a range from  $-20^{\circ}$  to  $+150^{\circ}$ , is available as ASTM-E1 89C through 96C (see *Thermometers*, Appendix D). A thermometer should be chosen such that the solidification point is not obscured by the cork stopper of the sample container.

**Sample Container** Use a standard glass 25- × 150-mm test tube with a lip, fitted with a two-hole cork stopper to hold the thermometer in place and to allow adequate stirring with a stirrer.

**Air Jacket** For the air jacket, use a standard glass 38- × 200-mm test tube with a lip and fitted with a cork or rubber stopper bored with a hole into which the sample container can easily be inserted up to the lip.

**Cooling Bath** Use a 2000-mL beaker or a similar, suitable container as a cooling bath. Fill it with an appropriate cooling medium such as glycerin, mineral oil, water, water and ice, or alcohol-dry ice.

*Stirrer* The stirrer (Fig. 4) consists of a 1-mm in diameter (B & S gauge 18), corrosion-resistant wire bent into a series of three loops about 25 mm apart. It should be made so that it will move freely in the space between the thermometer and the inner wall of the sample container. The shaft of the stirrer should be of a convenient length designed to pass loosely through a hole in the cork holding the thermometer. Stirring may be hand operated or mechanically activated at 20 to 30 strokes/min.

*Assembly* Assemble the apparatus in such a way that the cooling bath can be heated or cooled to control the desired temperature ranges. Clamp the air jacket so that it is held rigidly just below the lip, and immerse it in the cooling bath to a depth of 160 mm.

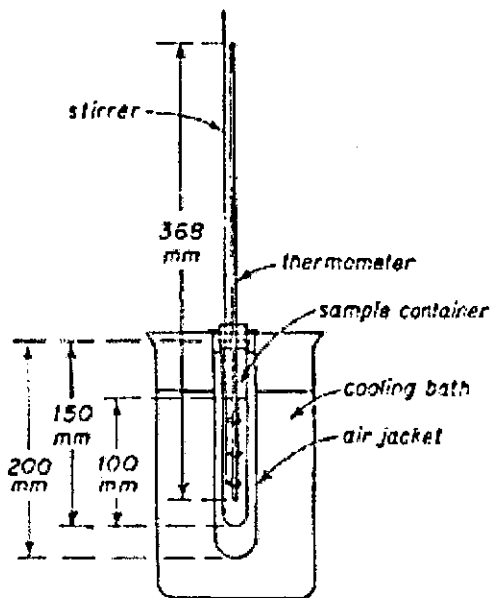


FIGURE 3 Apparatus for Determination of Solidification Point

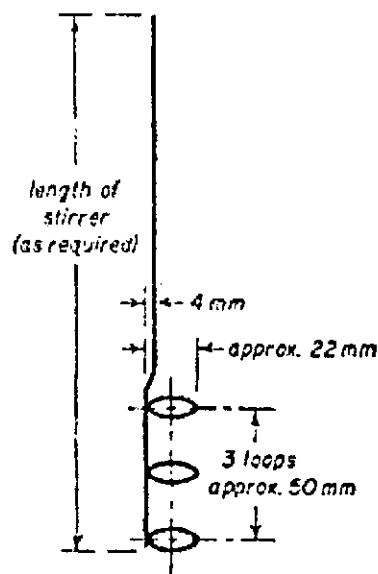


FIGURE 4 Stirrer for Solidification Point Determination.

**Sample Preparation** The solidification point of chemicals is usually determined as they are received. Some may be hygroscopic, however, and will require special drying. If this is necessary, it will be noted in the individual monographs.

Products that are normally solid at room temperature must be carefully melted at a temperature about  $10^{\circ}$  above the expected solidification point. Care should be observed to avoid heating in such a way as to decompose or distill any portion of a sample.

**Procedure** Adjust the temperature of the cooling bath to about  $5^{\circ}$  below the expected solidification point. Fit the thermometer and stirrer with a cork stopper so that the thermometer is centered and the bulb is about 20 mm from the bottom of the sample container. Transfer a sufficient amount of the sample, previously melted if necessary, into the sample container to fill it to a depth of about 90 mm when in the molten state. Place the thermometer and stirrer in the sample container, and adjust the thermometer so that the immersion line will be at the surface of the liquid and so that the end of the bulb is  $20 \pm 4$  mm from the bottom of the sample container. When the temperature of the sample is about  $5^{\circ}$  above the expected solidification point, place the assembled sample tube in the air jacket.

Allow the sample to cool while stirring, at the rate of 20 to 30 strokes/min, in such a manner that the stirrer does not touch the thermometer. Stir the sample continuously during the remainder of the test.

The temperature at first will gradually fall, then will become constant as crystallization starts and continues under equilibrium conditions, and finally will start to drop again. Some chemicals may supercool slightly below ( $0.5^{\circ}$ ) the solidification point; as crystallization begins, the temperature will rise and remain constant as equilibrium conditions are established. Other products may cool more than  $0.5^{\circ}$  and cause deviation from the normal pattern of temperature change. If the temperature rise exceeds  $0.5^{\circ}$  after the initial crystallization begins, repeat the test, and seed the melted compound with small crystals of the sample at  $0.5^{\circ}$  intervals as the temperature approaches the expected solidification point. Crystals for seed-

ing may be obtained by freezing a small sample in a test tube directly in the cooling bath. It is preferable that seed of the stable phase be used from a previous determination.

Observe and record the temperature readings at regular intervals until the temperature rises from a minimum, due to supercooling, to a maximum and then finally drops. The maximum temperature reading is the solidification point. Readings 10 s apart should be taken to establish that the temperature is at the maximum level and should continue until the drop in temperature is established.



## SOLIDIFICATION POINT

### Scope

This method is designed to determine the solidification point of food grade chemicals having appreciable heats of fusion. It is applicable to chemicals having solidification points between  $-20^{\circ}$  and  $+150^{\circ}$ .

Solidification point is an empirical constant defined as the temperature at which the liquid phase of a substance is in approximate equilibrium with a relatively small portion of the solid phase. It is measured by noting the maximum temperature reached during a controlled cooling cycle after the appearance of a solid phase.

Solidification point is distinguished from freezing point in that the latter term applies to the temperature of equilibrium between the solid and liquid state of pure compounds. Some chemical compounds have two temperatures at which there may be a temperature equilibrium between solid and liquid state depending upon the crystal form of the solid that is present.

### Apparatus

The apparatus is illustrated on the following page and consists of the components described in the following paragraphs.

#### Sample container

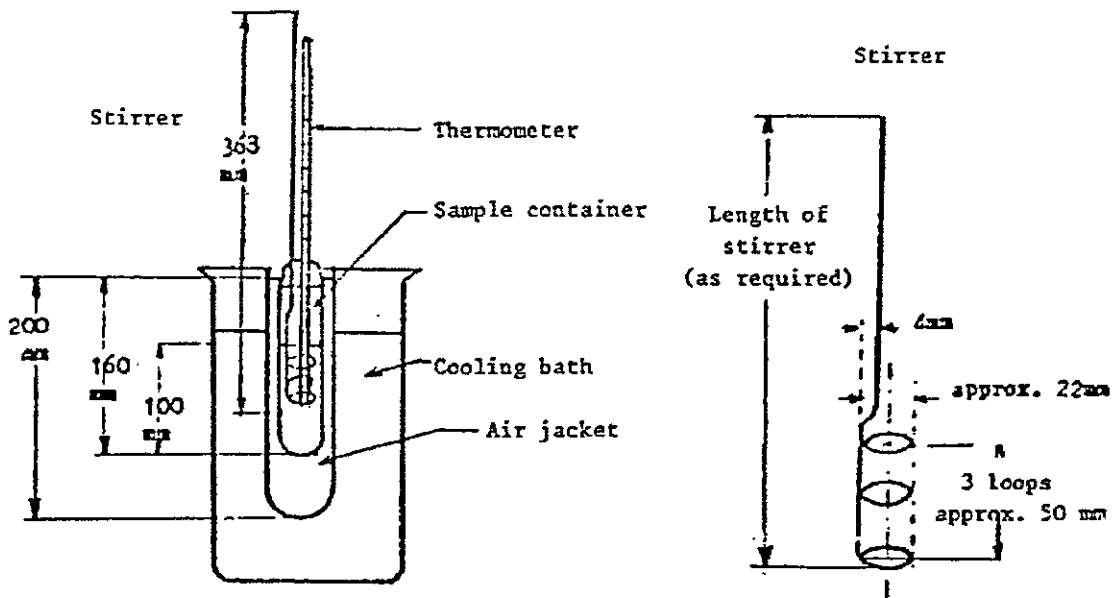
Use a standard glass 25 x 150 mm test-tube with lip, fitted with a cork stopper bored to hold the thermometer in place and to allow stirring with stirrer.

#### Thermometer

A thermometer having a range not exceeding  $30^{\circ}$  graduated in  $0.1^{\circ}$  divisions, and calibrated for 76 mm immersion, should be employed. A satisfactory series of thermometers, covering a range from  $-20^{\circ}$  to  $+150^{\circ}$  is available as ASTM-EI 89C through 96C. A thermometer should be so chosen that the solidification point is not obscured by the cork stopper of the sample container.

#### Stirrer

The stirrer consists of a 1 mm diameter (B & S gauge 18) corrosion-resistant wire bent in a series of 3 loops about 25 mm apart. It should be made so that it will move freely in the space between the thermometer and the inner wall of the sample container. The shaft of the stirrer should be of a convenient length designed to pass loosely through a hole in the cork holding the thermometer. Stirring may be hand-operated or mechanically activated at 20 to 30 strokes per min.



### Apparatus for Determining Solidification Point

#### Air jacket

Use a standard glass 38 x 200 mm test-tube with lip, fitted with a cork stopper bored with a hole into which the sample container can easily be inserted up to the lip.

#### Cooling bath

Use a 2-L beaker or similar suitable container as a cooling bath. Fill it with an appropriate cooling medium such as glycerine, mineral oil, water, water and ice or alcohol-dry ice.

#### Assembly

Assemble the apparatus in such a way that the cooling bath can be heated or cooled to control the desired temperature ranges. Clamp the air jacket so that it is held rigidly just below the lip and immerse it in the cooling bath to a depth of 160 mm.

### Preparation of Sample

The solidification point is usually determined on chemicals as they are received. Some may be hygroscopic, however, and require special drying. Where this is necessary it will be noted in the monograph. Products which are normally solid at room temperature must be carefully melted at a temperature about 10° above the expected solidification point. Care should be observed to avoid heating in such a way as to decompose or distil any portion of the sample.

### Procedure

Adjust the temperature of the cooling bath to about 5° below the expected solidification point. Fit the thermometer and stirrer with a cork stopper so that the thermometer is centred and the bulb is about 20 mm from the bottom of the sample container. Transfer a sufficient amount of the sample, previously melted if necessary, into the sample container to fill it to a depth of about 90 mm when in molten state. Place the thermometer and stirrer in the sample container and adjust the thermometer so that the immersion line will be at the surface of the liquid and the end of the bulb  $20 \pm 4$  mm from the bottom of the sample container. When the temperature of the sample is about 5° above the expected solidification point, place the assembled sample tube in the air jacket.

Allow the sample to cool while stirring at the rate of 20 to 30 strokes per min, in such a manner that the stirrer does not touch the thermometer. Stir the sample continuously during the remainder of the test.

The temperature at first will gradually fall, then become constant as crystallization starts and continues under equilibrium conditions, and finally will start to drop again. Some chemicals may supercool slightly below (0.5°) the solidification point; as crystallization begins the temperature will rise and remain constant as equilibrium conditions are established. Other products may cool more than 0.5° and cause deviation from the normal pattern of temperature changes. If the temperature rise exceeds 0.5° after the initial crystallization begins, repeat the test and seed the melted compound with small crystals of the sample at 0.5° intervals as the temperature approaches the expected solidification point. Crystals for seeding may be obtained by freezing a small sample in a test-tube directly in the cooling bath. It is preferable that seeds of the stable phase be used from a previous determination.

Observe and record the temperature readings at regular intervals until the temperature rises from a minimum, due to supercooling, to a maximum and then finally drops. The maximum temperature reading is the solidification point. Readings 10 sec apart should be taken in order to establish that the temperature is at the maximum level and continues until the drop in temperature is established.

## 第7版 食品添加物公定書

### 比旋光度測定法

旋光度は、光学的活性物質又はその液が偏光面を回転する角度であり、旋光計によって測定する。旋光の性質は、偏光の進行方向に向きあって、偏光面を右に回転するものを右旋性、左に回転するものを左旋性とし、偏光面を回転する角度を示す数字の前に、それぞれ記号+又は-を付け、角度を表す数字の右肩に°を付ける。

旋光度  $\alpha_{t/x}$  とは、特定の単色光  $x$ （波長又は名称で記載する。）を用い、温度  $t$  °Cで測定したときの旋光度を意味し、単に旋光度と記載した場合は、別に規定するもののほか、温度は 20°C、層長は 100mm、光線はナトリウムスペクトル中のD線で測定した旋光度  $\alpha =$  を示す。

比旋光度  $[\alpha]_{t/x}$  は、次の式で表す。

$$[\alpha]_{t/x} = 100 \alpha / l c$$

ただし、 $t$ ：測定時の温度

$x$ ：用いたスペクトルの特定の単光色の波長又は名称（D線を用いた場合は、Dと記載する。）

$\alpha$ ：偏光面を回転した角度

$l$ ：測定した液の層、すなわち、測定に用いた測定管の長さ（mm）

$c$ ：測定した液 1 ml 中に存在する試料の g 数

以下、本試験法を用いる場合において、例えば、「 $[\alpha]_{20/D} = +20.5 \sim +21.5^\circ$ （1 g、新たに煮沸し冷却した水、10ml、乾燥物換算）」とあるのは、本品約 1 g を精密に量り、新たに煮沸し冷却した水を加えて溶かして正確に 10ml とし、この液について測定し、乾燥物換算を行うとき、比旋光度が  $+20.5 \sim +21.5^\circ$  であることを示す。