

JIS

JAPANESE INDUSTRIAL STANDARD

Test methods for melting point and melting range of chemical products

JIS K 0064—1992

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Test methods for melting point and melting range
of chemical products

K 0064-1992

1. Scope This Japanese Industrial Standard specifies general methods to measure the melting point and melting range, ranging from ordinary temperature to 300°C, of chemical products.

Remarks 1. The melting point shall be applied to the substance which gives a definite melting point.

2. The melting range shall be applied to the substance, such as glass, amorphous, or multi-component mixture as oil-and-fat, wax, or vaseline which gives no definite melting point.

3. When the measuring method other than the methods in this standard are prescribed in the standard of other individual product or group of products, the test should conform to the method in the standard.

4. In some chemical products, the safety for the test cannot always be secured when the tests are carried out because of its volatility, explosiveness, or radioactivity. The methods prescribed in this standard should be applicable to the products of which safety is satisfactorily confirmed since they are only general methods.

5. The standards cited in this standard are as follows.

JIS B 7410 Liquid-in-Glass Thermometers for Testing of Petroleum Products

JIS K 0050 General Rules for Chemical Analysis

JIS K 0211 Technical Terms for Analytical Chemistry (General Part)

JIS R 3503 Glass Apparatus for Chemical Analysis

JIS Z 8401 Rules for Rounding off of Numerical Values

The Japanese Pharmacopoeia (the 12th revised)

2. General matters

2.1 Definition of terms The definition of the terms used in this standard shall conform to JIS K 0050 and JIS K 0211.

2.2 Matters in common The matters in common to the tests in this standard shall conform to JIS K 0050.

Glassware shall conform to JIS R 3503, and the rounding-off of numbers to JIS Z 8401.

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3. Methods to measure melting point The methods to measure melting point shall follow either of the following.

- (1) Method by visual observation
- (2) Method by measuring transmitted light

3.1 Method by visual observation

3.1.1 Summary Heat a sample shut in a glass capillary set in heating liquid, and find the melting point by visual observation.

3.1.2 Equipment and apparatus Equipment and apparatus shall be as follows.

- (1) Melting-point measuring apparatus Fix the capillary keeping the sample on a thermometer so as to let the part of capillary where the sample is shut contact with the mercury bulb of the thermometer, and immerse it in the center of heating liquid kept in a heating vessel and to let its end be about 20 mm above the bottom of the heating vessel. The example of the apparatus shall be as shown in Attached Fig. 1.

Alternatively, a melting-point measuring apparatus run by the heating-liquid circulating method by stirring can be also serviceable. The example of this apparatus shall be shown in Attached Fig. 2. In this case, the lower end of the capillary filled with the sample shall be fixed to be as near as possible to the mercury bulb of the thermometer.

- (2) Heating vessel This vessel is made of the first grade hard glass, and its shape and dimensions are exemplified in Attached Fig. 1.
- (3) Heating liquid Such as water or silicone oil is used, and silicone oil should be heat-resisting at the point higher than measuring temperature and have 50 to 100 mm²/s kinematic viscosity at 25°C.
- (4) Cover Made of ethylene tetrafluoride resin, rubber, or cork.
- (5) Thermometer This is the thermometer with an immersion line (stick type) of No. 1 to No. 6 specified in the Japanese Pharmacopoeia (the 12th revised). Provided that, if an anticipated melting point is 70°C or lower, SOP 57 to 59 specified in JIS B 7410 may be serviceable.
- (6) Capillary tube This tube, measuring 0.8 to 1.2 mm inside diameter, 0.2 to 0.3 mm wall thickness, and about 150 mm length, is made of hard glass with one end closed.
- (7) Heater The heater can heat heating liquid from 15°C lower than the anticipated melting point to 5°C higher than that, and can control temperature rising rate of heating liquid to be about 3°C/min and about 1°C/min.
- (8) Desiccator Containing silica gel as desiccant.