Determination of the melting point of salicylic acid

Aims of the experiment

- Determination of the melting temperature of a solid
- Characterisation of solids by their melting point

Principles

The melting point is defined as the temperature at which a substance changes from a solid to a liquid state at 1013 hPa. To melt a substance, it must be supplied with thermal energy, which increases the vibrational energy of the particles. At the melting point, the energy of the particles is sufficiently high for them to leave the solid lattice sites and change to a more disordered state – the substance liquefies. Not all pure substances have a melting point at a defined temperature: Amorphous substances (such as glass) liquefy over a melting interval while other substances (eg, many plastics) change (decompose) chemically even before reaching the liquid state so that that there is no specified melting point.

As a characteristic quantity of pure substances, the melting point is an important material property. With its help, substances can be identified and tested for purity: If it is suspected that an initially unknown substance is substance A, its determined melting point can be compared with values recorded in tables. Furthermore, the substance can be mixed with a sample that is demonstrably A, and the melting point determined again. If the materials are identical, the melting point of pure A will be found; if the melting point is different, it is not A. Similarly, it can be determined whether a substance is contaminated: If its melting point deviates from that of the pure substance, the substance is contaminated.

The melting point for many solids can be determined using the Thiele apparatus. For this purpose, it is filled with a heating fluid, which is externally heated either by means of a burner or a heating bath. The design of the apparatus ensures distribution of heat by means of convection. A thermometer is inserted into the central opening of the apparatus. The substance to be tested is placed in a melting point detection tube and inserted far enough through one of the two side arms of the apparatus so that the substance is level with the liquid reservoir of the thermometer. Next, the apparatus is heated and the temperatures are determined at which the substance begins to melt and at which it has completely melted (melting interval). Pure substances have a melting interval of max 1 °C. In order to obtain precise values, the temperature rise in the heating fluid in the region of the melting point should be no more than about 1 to 2 °C per minute. In this experiment, the melting point of salicylic acid is determined in this manner.

Fig. 1: Experiment set-up.

Risk assessment

When using the burner, long hair must be tied back to protect it from catching fire.

Protective goggles should be worn during the experiment as salicylic acid must not come in contact with the eyes.

When heating the apparatus, the paraffin used as the heating fluid expands significantly. Therefore, fill the device no more than described in the instructions. As a precaution, place a collecting vessel below when beginning the heating process.

The viscous paraffin used as the heating fluid can be easily heated to 200 °C, but if a heating bath with a higher temperature is required, another heating fluid must be chosen.

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Salicylic acid

Signal word:
Danger

Hazard statements

H302 Harmful if swallowed.
H318 Causes serious eye damage.

Safety Advice

P305+P351+P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do – continue rinsing.
P313 Get medical advice/attention.

Equipment and chemicals

No.	Material	Cat. number
1	Melting point determination apparatus	667 500
1	Melting point detection tubes, set of 100	661 085
1	Silicone gaskets, GL 18/6, set of 10	667 306
1	Chemical thermometer, -10...+250 °C/1 K	666 161
1	Magnetic stirrer with hotplate	666 8471
1	Stirring magnet, 50 mm x 8 mm diam.	666 854
1	Stand rod, 450 x 12 mm diam., M10 thread	666 523
1	Universal clamp 0...80 mm	666 555
1	Bosshead S	301 09
1	Laboratory dish, 140 mm diam., 900 ml	602 725
1	Watch glass dish 80 mm Ø	664 154
1	Powder spatula, steel, 185 mm	604 5682
1	Funnel PP, 75 mm Ø	665 009
1	Teclu burner, universal	656 017
1	Safety gas hose with end clamp, 1 m	667 187
1	Paraffin, thick, 1 l	674 0820
1	Salicylic acid, 100 g	674 6210

Set-up and preparation of the experiment

1. Screw the stand rod into the threaded hole on the magnetic stirrer.
2. Use the bosshead and universal clamp to affix the melting point determination apparatus to the stand rod.
3. Fill the melting point determination apparatus with paraffin oil to just below the two lateral branches, using the funnel as an aid.
4. Insert the thermometer through the GL screw cap, provided with a silicone ring, and secure it in the vertical tube of the melting point determination apparatus so that the centre of the thermometer reservoir is at the level of the two lateral branches.
5. Place a spatula tip of salicylic acid on the watch glass.
6. Take some of the substance from the watch glass using the open end of a melting point detection tube and compact the substance at the closed end of the tube: Drop the tube, with the closed end downwards, several times onto a table from a height of a few centimetres. The filling height should be 2–3 mm after compaction. If the substance is too coarse to be taken up with the melting point detection tube, it must first be ground in a mortar.
7. Cut a piece of silicone hose about 1 cm long in half lengthwise. Using a hot needle, pierce a hole in the centre and pass the melting point detection tube through the hole. Now insert the tube into one of the two lateral glass tubes of the melting point determination apparatus until the substance is at the level of the thermometer reservoir. The piece of hose prevents the tube from slipping.
8. Get the burner ready.
9. Get the heating bath ready: Fill the lab dish a little more than half full with paraffin and add the magnetic stirrer.

Performing the experiment

Rough determination of the melting point of a substance with unknown melting point

1. Carefully heat the full length of the lower leg of the apparatus with a roaring flame from the Bunsen burner. The temperature in the heating fluid should not rise faster than 10 °C per minute.
2. Constantly observe the substance in the melting point detection tube and immediately read the temperature when the first signs of liquefaction of the substance are seen. Read off the temperature again as soon as the entire amount of the substance has liquefied.

Precise determination of the melting point of a substance with known melting point

1. Place the heating bath on the magnetic stirrer, turn it on, and adjust the stirrer so that the heating bath is stirred thoroughly but not too vigorously.
2. Lower the Thiele apparatus into the heating bath until the lower leg is completely immersed in the heating bath.
3. Fill a new melting point detection tube with fresh substance and place in the apparatus (see preparation).
4. Adjust the heating plate of the magnetic stirrer so that the paraffin in the apparatus heats up at a rate of about 10 °C per minute. If the paraffin has reached a temperature of about 20 °C below the expected melting point, adjust the heating plate so that the paraffin heats at a rate of 1–2 °C per minute.
5. Constantly observe the substance in the melting point detection tube and immediately read the temperature when the first signs of liquefaction of the substance are seen. Read off the temperature again as soon as the entire amount of the substance has liquefied.

Observation

At 157 °C it can be seen that part of the salicylic acid in the melting point determination tube liquefies. At 159 °C, the entire substance is present in liquid form.

Result

According to literature salicylic acid has a melting point of 159 °C. When investigating the melting point of pure substances, they usually liquefy within a very small temperature interval of less than one degree Celsius. Here the liquefaction

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was observed over a temperature interval of two degrees Celsius, which indicates a slight contamination of the salicylic acid. An attempt can be made to further purify the salicylic acid by recrystallizing it from water in order to be able to observe the melting in a smaller temperature interval. Overall, this method can be used to determine the melting point of salicylic acid with very good accuracy: The melting point determined experimentally is 159 °C and thus does not deviate from the literature value.

Cleaning and disposal

The paraffin oil from the Thiele apparatus and the heating bath can be collected and stored for further experiments.

The melting point detection tubes are collected in the contaminated glass waste collection bin.

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