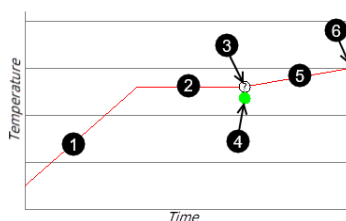


Melting Point of Aspirin

Background

A pure substance has a well-defined melting point while an impure substance may have a lower, complex melting point that occurs over a broader range. The broader the melting range, the greater the impurity. Impurities disrupt crystal lattice structures and make it easier for a substance to transition from a solid to a liquid at a lower temperature. The chemical name for aspirin is acetylsalicylic acid (ASA) and its melting point is 135.0 °C. Commercial aspirin tablets contain ASA plus inactive ingredients. In this investigation, you will use a Melting Point Apparatus to heat up an aspirin sample in a slow and controlled manner to assure the temperature inside the sample tube is the same as the metal block heating it. The Heating Profile feature in Capstone is designed for accurate melting point range determination. The profile steps are visually represented in an image like the one below. The steps in the table are arranged in a typical sequence, however, steps can be reordered, added, or deleted as needed. The image is a model and the shape and completion time of the actual temperature plot will differ.



#	Step	Purpose
①	Quick Ramp	Rapidly approaches a specified temperature (in °C) in an asymptotic manner. Goal: Bring the sample's temperature a few degrees below the expected melting point.
②	Hold	Maintains the block at a steady temperature for a specified time in minutes. Goal: Allow the system and sample to reach thermal equilibrium. Choose a Quick Ramp temperature a few degrees below the melting point so the sample remains solid during Hold.
③	Prompt	Pauses the Heating Profile until the user clicks OK in the pop-up dialog that appears. Goal: Notifies the user when the sequence is about to go through the expected melting point so they can pay closer attention. If no action is taken within 30 minutes of the prompt's initial appearance, the sequence will automatically proceed to the next step.
④	Start Image Capture*	Begins taking a sequence of images from the USB Camera* at a specified interval until stopped, indicated by a green circle. The minimum interval is 5 seconds between images. Goal: Pinpoint temperatures where melting first begins and ends. To avoid large file sizes, snapshots are only taken during the Ramp phase where melting is expected. <i>*Optional; for use with the USB Camera (SE-6215) or other compatible camera</i>
⑤	Ramp	Slowly heat a sample to approach, meet, and exceed its anticipated melting point at a specified ending temperature and heating rate. Goal: Linearly approach and move through the expected melting point temperature at a rate between 1-2 °C per minute. Divide the increase in temperature over the time period to get the heating rate. Example: If Quick Ramp is set to (140 °C), and Ramp is set to (10 minutes, 150 °C), temperature will increase by 10 °C over 10 minutes therefore the heating rate equals 1 °C per minute.
⑥	Stop Image Capture	Indicates when to stop taking video snapshots if using a USB Camera (red rectangle).

Driving Questions

How does the melting point range of an aspirin tablet compare to the melting point of pure aspirin? How does melting point range indicate sample purity? What are the signs that indicate when a phase change from solid to liquid first begins, and comes to completion?

Materials and Equipment

- Computer with Capstone software
- Wireless Melting Point Apparatus
- USB Camera and camera adapter*
- Precision balance (readability: 0.001 g)
- Mortar and pestle (2)
- Spatulas (2)
- Glass capillary tubes, one end closed, 1.5-mm OD
- Watch glasses (2)
- Aspirin tablets (in original packaging)
- Acetylsalicylic acid (ASA, reagent grade)

*Optional; the camera adapter is included with the Wireless Melting Point Apparatus while the USB camera (SE-6215) is sold separately; check pasco.com to see which cameras are compatible with the apparatus, your system, and your software.

Safety

Follow these important safety precautions in addition to your regular classroom procedures:

- Wear gloves if available.
- Handle glass capillary tubes with care and dispose of used tubes as directed by your instructor.

Procedure

Part 1 - Setup

1. Begin your lab report with a 1-paragraph background description of the phenomena you will study in this investigation. Use your own words, and end with a clearly-stated question that can either be supported or denied with data collected during this investigation.
2. Plug the Wireless Melting Point Apparatus (MPA) into a wall outlet and switch the power on.
3. If available, connect the USB Camera to your computer (before you open software).
4. Open Capstone and connect the MPA to your computer through the Hardware Setup panel. Close the panel, then open the Quick Start file called *Determining a Melting Point*. When the pop-up window appears, select *Discard*.

NOTE: If the *Quick Start* file is not available, update your software to the latest version and start over.

5. For USB Camera users: Verify the camera is displaying a live preview.
 - If the preview is not displayed, find the Camera settings above the Heating Profile and select the camera called MD100. If this does not work, close Capstone, disconnect the camera from your device, and start over from step 3.

Part 2 - Capillary Tube Preparation

1. Obtain an individual aspirin tablet from a bottle. Record its mass, brand name, manufacturer's determined ASA content (in mg), inactive ingredients*, and any other relevant information from the container.

NOTE: *List only the first 6 inactive ingredients. If not listed, locate the small-print NDC number on the container. Perform an online search of the number (including the NDC prefix) and go to the National Institutes of Health (.gov) link, which should rank at or near the top of your search results.

2. Grind the tablet with a mortar and pestle until the entire sample is a fine powder.
3. Transfer a small pile of the sample to the center of a watch glass, then tap the open end of the capillary tube into the sample as shown in Figure 1Ⓐ. Load it 2-3 mm deep.

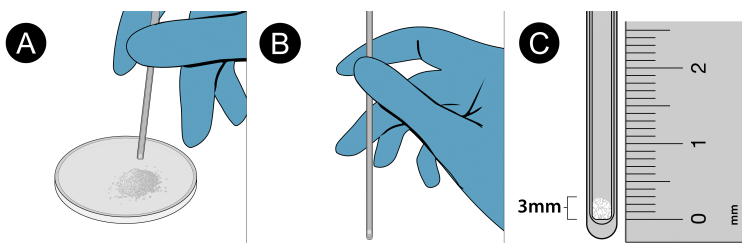


Figure 1. Collect a sample in a capillary tube

4. Invert the tube. Tap the bulbous tube end on the lab bench several times to compact the sample into the closed end (Figure 1Ⓑ).
5. Collect and compact additional sample if necessary to load the tube with a sample height of 2-3 mm (Figure 1Ⓒ). Do not exceed 3 mm.
6. Insert the closed end of the capillary tube into an open chamber in the MPA as shown in Figure 2. A magnified image of the sample will be visible on your screen if you are using a USB Camera (①) or in the eyepiece (②).

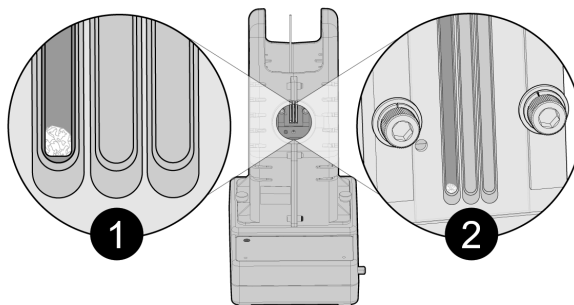




Figure 2. Melting Point Apparatus with sample in capillary tube

7. Choose either (a) or (b) depending on your viewing setup:
 - a. Eyepiece: Rotate the capillary tube and adjust the eyepiece position for the best focus and sample view.
 - b. USB Camera: Rotate the capillary tube and adjust the camera position to focus it for the best sample view. Use the Light Level slider below the profile to adjust sample lighting. To adjust image sharpness and light or color balance, hover over the live camera preview and select the gear icon that appears to open the Properties menu. Expand the Movie Recording menu, then select Advanced Camera Properties.

NOTE: If Capstone appears to be grayed-out, minimize the Capstone window to reveal the Advanced Camera Properties dialog. OK the dialog, then restore the Capstone window.

Part 3 - Melting Profile and Melting Point Range (Tablet)





1. Since you are not working with pure ASA, set up the Melting Profile to quickly estimate the melting point range:
 - a. Select the Ramp step near the bottom of the profile. Leave Time set as-is. Set Temperature to 350 °C.
 - b. Select Quick Ramp in the Melting Profile and delete it . Delete all remaining steps until only Ramp remains.
 - c. Locate the live temperature reading above the Melting Profile, and find the Digits display **1.23** that will show temperature data once recording begins. Assign lab partner roles: (1) sample monitor, and (2) temperature monitor. Lab partners should not switch roles during this investigation.
 - d. Start recording data. The sample monitor must let the temperature monitor know when melting begins and when it is complete. The temperature monitor will note the approximate temperatures at the start and end of melting.
 - e. Stop data recording a few degrees after melting is complete.
2. Select the Cool button above the Melting Profile. Allow the block to cool to at least 20 °C below the estimated first melting temperature, then turn off Cool.
3. Remove the used capillary tube from the MPA. Load a second capillary tube with the sample and set it in the same chamber. Select the Preview Camera icon  above the camera image to display a live image. Adjust position and focus if needed.

4. Revise the profile to apply a slower heating sequence based on the melting point range data determined in step 1:
 - a. Select the Select Profile Template button above the Melting Profile, then select Default from the drop-down list.
 - b. Select Quick Ramp in the Melting Profile. Change the temperature to a few degrees C below the observed temperature at which melting first began.
 - c. Set Hold to 3 minutes; allow Prompt to follow Hold.
 - d. Select Start Image Capture. If you are NOT using a USB Camera, delete this profile step. If you are using a USB Camera, set the image capture interval to 5 s.

NOTE: During analysis it is beneficial to have more images captured, however, consider your device's performance with large files. The faster the image capture rate, the larger the file size.

- e. Select Ramp. Set the ending temperature to a few degrees C above the observed temperature where melting ended, then determine the time required to set the rate of temperature increase to 1-2 °C per minute (see the Ramp step in the table in the Background section).
 - f. Leave Stop Image Capture as the last profile step, or delete it if not using a USB camera.
5. Start recording data; the active step in the Melting Profile will be highlighted. Monitor the sample and respond to the Prompt when it appears. If the sample has already begun to melt before the Prompt appears, stop recording data. Cool the block as before and revise the Melting Profile as needed. Repeat steps 3 through 5 until your Heating Profile ensures the sample remains completely solid during Hold.

NOTE: Know the difference between glistening and melting. Melting begins when you can distinctly see some amount of liquid in the sample, especially on the sides of the capillary tube. Glistening involves light changing or sample settling, without the appearance of liquid. Melting is complete when the entire sample is in liquid form and is transparent.

6. You should now be in the Ramp step. Record the temperatures at which melting begins and ends, then allow the melting point profile sequence to complete (or, stop recording data when it is clear that melting is complete). If you used a USB camera: review snapshots after data recording ends to pinpoint melting temperatures:
 - a. Switch from Recording Mode  to Playback Mode .
 - b. Use the playback controls to identify the beginning of the snapshot that first shows a distinct liquid forming. Record the temperature shown in the Digits display **1.23**.
 - c. Find the beginning of the snapshot that first shows melting is complete and record the temperature.
 - d. Before you begin the next run, switch back to Recording Mode . Next, select the Preview Camera icon  above the camera image and make any image adjustments as necessary. Return to step 3 to begin the next run.
7. Repeat steps 2-6 until you have at least 2 runs with melting point range temperatures that agree within 0.50 °C.

Part 4 - Melting Profile and Melting Point Range: Pure ASA

1. Allow the block to cool to at least 115 °C. Remove the used capillary tube from the MPA.
2. Use a clean watch glass, spatula, and mortar and pestle to prepare a sample of pure ASA. Load a capillary tube and make adjustments for the best sample view. If using a camera, return to Recording Mode and turn on the camera preview.

Melting Point of Aspirin

3. Revise the profile to apply a more narrow heating sequence based on the expected melting point near 135.0 °C..
4. Start recording data. Record the exact temperatures at which melting begins and ends. Repeat until you have at least 2 runs with melting point range temperatures that agree within 0.50 °C.

Analysis

Answer all questions in your lab report.

1. For your aspirin tablet: Of the two runs that agree within 0.5 °C, select the lowest value for start of melting and the highest value for end of melting, then round to the nearest tenth degree C to determine the melting point range. Next, determine the melting point range for the pure ASA sample in the same manner. What is the melting point range of your aspirin tablet, and what is the range for pure ASA?
2. Find the mean melting point temperature for the aspirin tablet and for the pure ASA sample. Do you recommend reporting a mean melting temperature as a measure of ASA content? Why or why not? Support your answer with data.
3. Convert the tablet mass to mg, then use the stated ASA content to determine the aspirin tablet's percent-by-mass ASA content. Show work.

Questions

Answer all questions in your lab report.

1. Report all relevant information about your aspirin tablet, and comment on how well your experimentally determined melting point range reflects the purity of the tablet in terms of ASA content. Support your answer with data.
2. Predict how the melting point range of an enteric-coated, low-dose (81 mg) aspirin tablet would compare to the melting point range you determined for the tablet in this investigation. Explain your answer.
3. Where would you expect the most agreement among class results: melting point range of an identical aspirin tablet, or melting point range of pure ASA? Explain your reasoning.

Conclusion

Summarize your results to complete your lab report. Address the driving questions with generalizations from your data and observations. Use your understanding of intermolecular forces, colligative properties, phase changes, and data from this investigation to support your concluding statements. Comment on your accuracy based on your percent error calculations. Describe potential sources of error and their impacts on your results.