Freezing Point of Glacial Acetic Acid

INSTRUCTOR RESOURCES

The CCLI Initiative

Learning Objectives

- demonstrate the general features of a cooling curve.
- measure the freezing point of a compound.

Procedure Overview

- temperature probe calibration at a minimum of three points between 0°C and 55°C.
- testing the experiment program and temperature probe.
- apparatus assembly and warming of glacial acetic acid sample.
- data collection for cooling curve with supercooling and spreadsheet treatment of data.

Name ______________________________ Section _________ Date ___________________

Freezing Point of Glacial Acetic Acid

Report Sheet

1. In the space below, record the freezing point temperature for glacial acetic acid obtained in this experiment. For comparison, the *Handbook of Chemistry and Physics, 65th Edition* lists the melting point as 16.6°C.

   Observed freezing point: _____________________

2. Include the following item as part of your report for this experiment:

   (a) printout of your cooling curve graph.

Name ______________________________ Section _________ Date ___________________

Freezing Point of Glacial Acetic Acid

Questions

1. Your instructor will provide the structure of glacial acetic acid. Explain the meaning of the term "supercooling," and on the basis of the structure, suggest a reason why glacial acetic acid supercools so dramatically.

2. Why is it important to have a broad flat portion of the freezing point curve after the “supercooling” portion in order to get a good freezing point value?

3. What could be the cause of a cooling curve in which the cooling curve rises from the “supercooling” to a peak, and then begins a slow, steady drop from there?

4. How can you insure that a proper cooling curve with a broad flat plateau is obtained.

Freezing Point of Glacial Acetic Acid

Suggested Answers to Questions

1. Your instructor will provide the structure of glacial acetic acid. Explain the meaning of the term "supercooling," and on the basis of the structure, suggest a reason why glacial acetic acid supercools so dramatically.

   Supercooling is the process of cooling a liquid below its freezing point without its changing to a solid.

   Supercooling occurs because, as it is cooled, the liquid may not achieve the degree of organization necessary to form solid at the freezing point temperature, and thus it continues to exist as the liquid. At some point the correct ordering occurs and solid rapidly forms, releasing energy in the exothermic process and bringing the temperature back up to the freezing point, where the remainder of the liquid freezes.

2. Why is it important to have a broad flat plateau in the freezing point curve after the "supercooling" portion in order to get a good freezing point value?

   A broad flat plateau is desirable to allow the glacial acetic acid to freeze at a rate that insures all of the amount of the substance present has had time to solidify at the freezing point temperature.

3. What could be the cause of a cooling curve in which the cooling curve rises from the "supercooling" to a peak, and then immediately begins a slow, steady drop from there?

   This phenomenon is caused by cooling the glacial acetic acid too fast, removing heat so rapidly that no plateau can form before the temperature drops below the freezing point.

4. How can you insure that a proper cooling curve with a broad flat plateau is obtained.

   The cooling must be slow enough that all of the glacial acetic acid liquid has time to solidify at the freezing point temperature. The use of the two nested test tubes is an attempt to insure this.

Freezing Point of Glacial Acetic Acid

Tips and Traps

1. Make sure the interface is turned on by pushing the power switch on the right back corner of the interface.

2. In the calibration of the temperature probe, students should be cautioned to allow the red line to merge into the green equilibration area, and for the active graph to flatten out.

3. The best supercooling occurs if the test tube containing the acetic acid is placed directly into the ice slush with no stirring.

4. The best melting point is obtained using the method described in the experiment, but is slow due to the added insulation of the outer test tube. The instructor needs to decide whether the additional time is warranted.

5. After the calibration, the temperature probe should be thoroughly dried before being placed in the melted glacial acetic acid.

6. Never push the temperature probe through the #2 two hole stopper. Slit one side of the stopper. Open the slitted side and insert the temperature probe into the stopper hole to the proper depth.

7. Be sure to keep and reuse the glacial acetic acid. The original 20 x 50 mm test tube serves as a good storage site. Cap the tube with a rubber stopper.

8. The structure of glacial acetic acid, CH_3COOH, is

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  H    O
  |   //
H-C-C
  |   \
  H    OH
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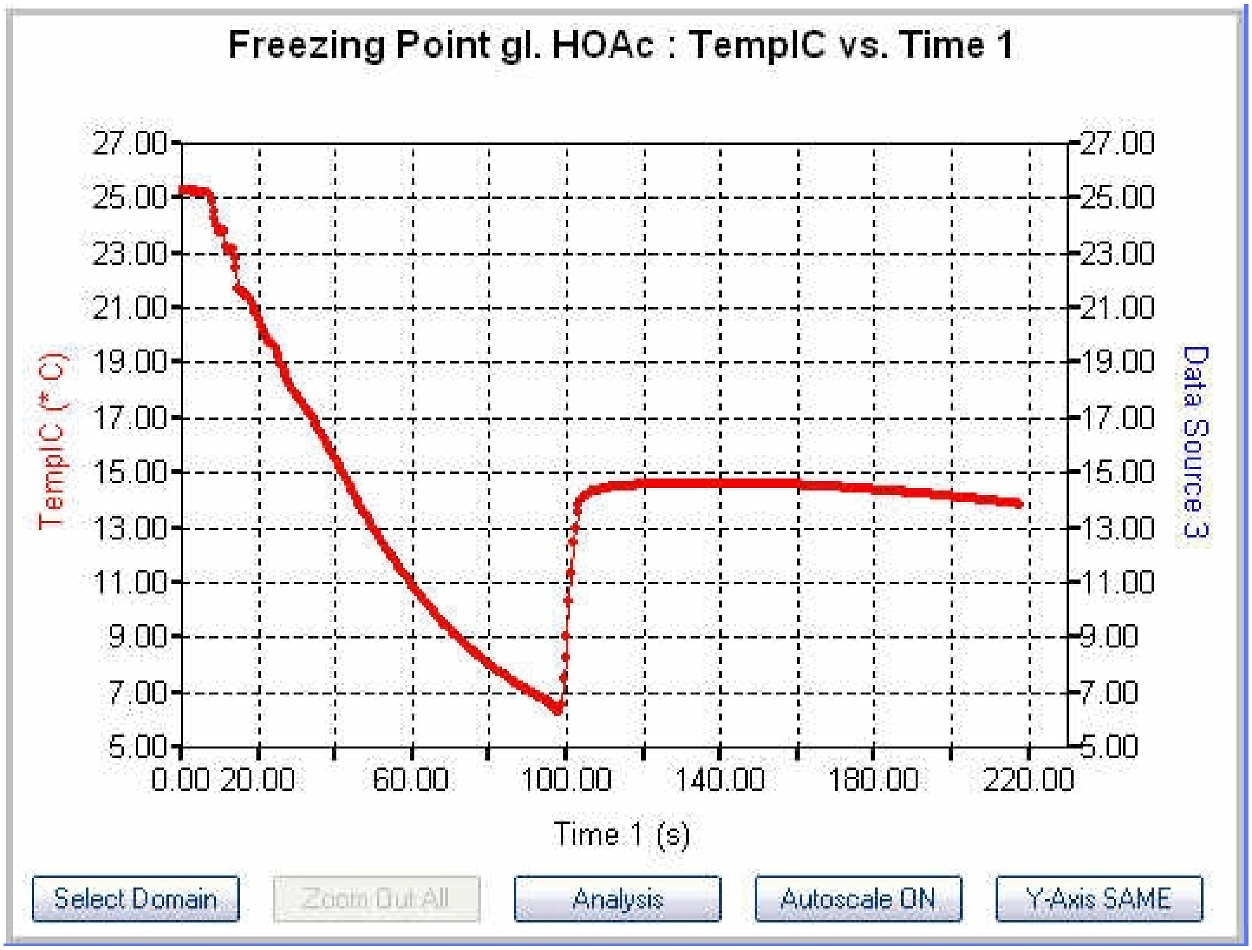
10. Butyl alcohol can be used in place of glacial acetic acid, but supercooling is far less and traces of moisture spoil cooling curves.

Freezing Point of Glacial Acetic Acid

Sample Data

Sample ***MicroLAB*** freezing point curve for acetic acid.



Freezing Point of Glacial Acetic Acid

Laboratory Preparation (per student station)

Equipment

- each freezing point determination apparatus (see figure on page 35 of the experiment) consists of an inner test tube (20 x 50 mm) fitted with a rubber stopper (number 2, two hole) to accommodate the temperature probe and stirrer. The outside of the test tube should be fitted with a rubber ring to allow it to fit snugly into a 40 ml conical centrifuge tube (29 x 118 mm, Corning No. 8320-40, or equivalent).
- temperature probe
- stirring wire, approximately 7.5 inches long (see figure on page 5)
- 250 ml beaker (2)
- 400 ml beaker
- 600 ml beaker
- universal clamp
- ring stand

Supplies

- 150 - 200 g ice for calibration of the temperature probe and cooling

Chemicals

- 5 ml glacial acetic acid

Safety and Disposal

- there are no known safety problems associated with the experiment, and there should be no waste for disposal.