Shawdon Molavi
CHEM 475 – Barron
2/21/12
Melting Point Analysis

Intro

- Melting point is a physical property
- Quick/Easy analysis to qualitatively identify relatively pure samples (<10% impurity)
- Also possible to use to quantitatively determine purity

Equipment

- Many types but all have
    - Heating block/oil bath
    - Thermometer
    - Temperature control
    - Magnifying glass viewer
    - Backlight to view sample
- Modern ones are digital and programmable (start temp, end temp, temp change rate)

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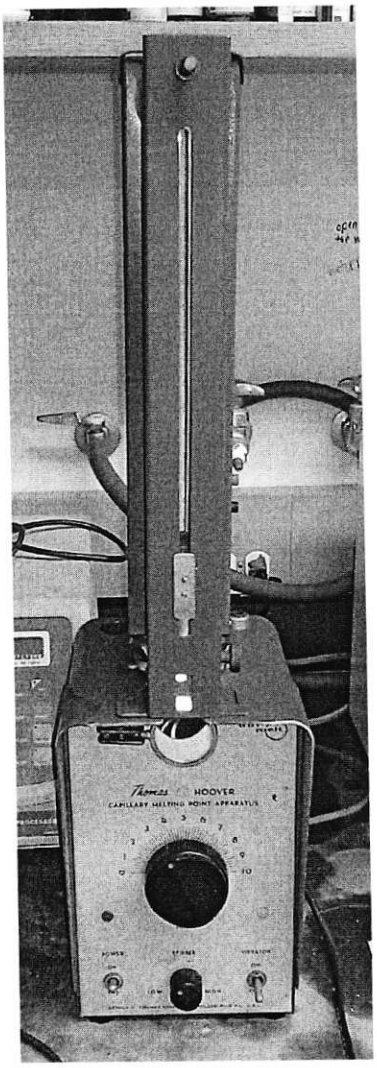
- 

A Thomas Hoover Melting Point Apparatus

Sample preparation

- Pack dry sample into melting point analysis capillary tube (has one closed end). Only need 3-5 mm of sample
- Pack into closed end by tapping/controlled dropping or vibration. Some machine have a vibrator
- Place capillary tube into machine. Some machines can run multiple samples

Recording/Interpreting Data

- Watch sample for melting
- When melting begins, note temperature
- When sample is fully melted, note temperature
- That is the melting point range

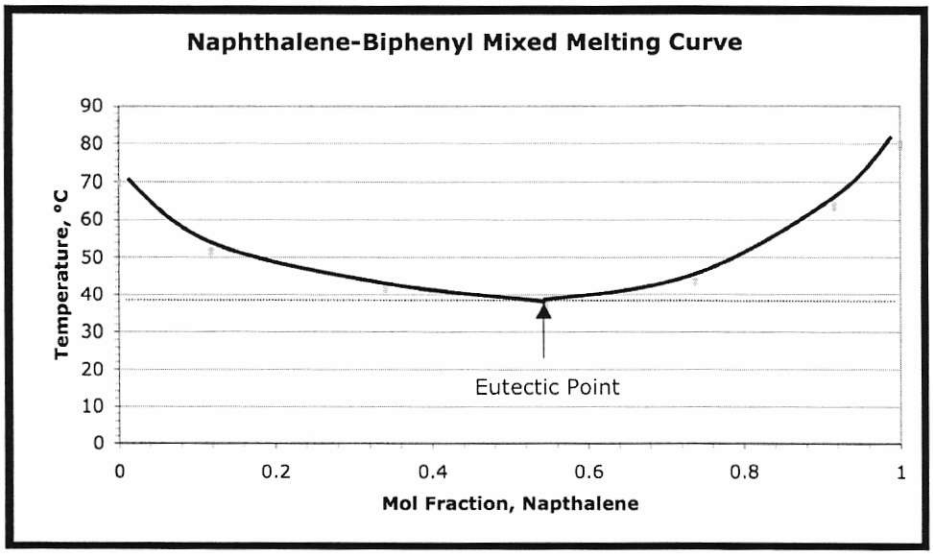
  - Usually 1 to 2 °C for pure samples
  - Can be broadened due to colligative properties

A Good video showing sample prep and discussing melting point analysis in general

- http://www.youtube.com/watch?v=9RNRYLvlbXM

Interpreting Data

- For Identification (Qualitative)
  - Compare experimental melting point range to literature values
    - There are vast databases
  - Obtain pure sample of suspected substance and mix a small amount of unknown with it
  - Conduct melting point analysis again
  - If sharp melting point range is observed at similar temperatures, then unknown likely successfully identified
  - If melting point range is depressed or broadened (due to colligative properties), then unknown was not successfully identified
- For Purity (Quantitative)
  - First, the identity of the solvent (main constituent of sample) needs to be known as well as the main solute
    - Many GC-MS set-ups can do this
  - Because melting point (aka freezing point) depression is unique between chemicals, a graph needs to either be obtained or prepared comparing melting point to molar fraction
    - Can be prepared by simply performing melting point analysis on standards with known molar fraction ratios
  - Compare experimental melting point to graph to determine molar fractions in sample



Mixed melting curve for Naphthalene and Biphenyl (not sure about the "adapted from" details. It's from an organic lab pre-lab and protocol for Clark College)

Specificity/Accuracy

- Melting point is a unique physical characteristic of a substance
- Thermometer readings are accurate
- Melting point is dependent on pressure as well, so this can affect experimental values. Should be considered when comparing to literature values
- Detection of melting is done visually. Introduction of error is high due to this

Pros

- Quick, easy, and cheap preliminary analysis if sample is mostly pure and sample has suspected identity
- Uses very small samples

Cons/Limitations

- Sample must be solid
- Destructive
- Samples with more than one solute cannot be analyzed quantitatively

Questions??

There is "Melting Temperature Analysis" done on DNA to determine ratios of point mutations, but it operates using a dye to determine when double stranded DNA is denatured to single stranded DNA. When half of the DNA sample has been denatured, that temperature is considered the "melting temperature". **This IS NOT the same as melting point analysis, but should I try to include it anyway?** I feel like the fact that one is a chemical reaction and one is a physical reaction makes it a bit of a stretch to call it a "Modern Adaptation".