Problem 8: Compound confusion

Pre-Lab answers

1. 

![Caffeine](image1.png)  
**caffeine, m.p. 238 °C**

![Benzoic acid](image2.png)  
**benzoic acid, m.p. 122 °C**

![Paracetamol](image3.png)  
**paracetamol, m.p. 170 °C**

![Methyl ethanoate](image4.png)  
**methyl ethanoate, m.p. –98 °C**

![2-hydroxybenzoic acid](image5.png)  
**2-hydroxybenzoic acid, m.p. 159 °C**

![Propanoic acid](image6.png)  
**propanoic acid, m.p. –21 °C**

2. 

A pure compound will melt over a relatively narrow temperature range. Impurities both lower the temperature at which the compound melts and widens the range over which it melts.

3. 

For benzoic acid:

\[
\begin{align*}
C_6H_5COOH + NaHCO_3 &\rightarrow C_6H_5COO^-Na^+ + H_2O + CO_2 \\
2 C_6H_5COOH + Na_2CO_3 &\rightarrow 2 C_6H_5COO^-Na^+ + H_2O + CO_2
\end{align*}
\]

For 2-hydroxybenzoic acid:

\[
\begin{align*}
C_6H_4(OH)COOH + NaHCO_3 &\rightarrow C_6H_4(OH)COO^-Na^+ + H_2O + CO_2 \\
2 C_6H_4(OH)COOH + Na_2CO_3 &\rightarrow 2 C_6H_4(OH)COO^-Na^+ + H_2O + CO_2
\end{align*}
\]

For propanoic acid:

\[
\begin{align*}
C_3H_7COOH + NaHCO_3 &\rightarrow C_3H_7COO^-Na^+ + H_2O + CO_2 \\
2 C_3H_7COOH + Na_2CO_3 &\rightarrow 2 C_3H_7COO^-Na^+ + H_2O + CO_2
\end{align*}
\]
4. 
   a) A deuterium atom is an isotope of hydrogen with atomic mass 2 (1 proton, 1 neutron, 1 electron)
   b) The residual solvent peak is a result of the small quantities of CHCl₃ or (CH₃)₂SO₂ present in the bottles of the deuterated solvents.
   c) CDCl₃ Residual solvent peak in ¹³C NMR – δC 77.2 ppm, triplet 
       DMSO Residual solvent peak in ¹³C NMR – δC 39.5 ppm, septuplet

5.

\[
\text{m/z 43} = \text{acylium ion} \\
\text{[HOOC₆H₄NHC(O)CH₃]⁺} \rightarrow \text{[CH₃C(O)]⁺} + \text{[HOOC₆H₄NH]⁺}
\]
Students can identify compounds C and D as methyl ethanoate [Highly flammable, Irritant] and propanoic acid [Corrosive] because they are liquid at RTP. A simple carbonate test will identify the carboxylic acid as C.

Sample E can be identified as paracetamol [Harmful] and sample F as caffeine [Harmful] from the elemental analyses.

This leaves samples A and B as benzoic acid [Harmful] or 2-hydroxybenzoic acid [Harmful]. A weak O-H stretch (ν 3200 cm⁻¹) is visible in the IR of sample B, but a melting point analysis of each will allow for final identification of A as benzoic acid and B as 2-hydroxybenzoic acid.

A = benzoic acid; B = 2-hydroxybenzoic acid
C = propanoic acid; D = methyl ethanoate
E = paracetamol; F = caffeine

Spectral set 1 = methyl ethanoate
Spectral set 2 = 2-hydroxybenzoic acid
Spectral set 3 = benzoic acid
Spectral set 4 = paracetamol
Spectral set 5 = propanoic acid
Spectral set 6 = caffeine

Full spectral analysis can be found on the SpectraSchool link on Learn Chemistry.
Equipment list

Each group will need;

- Small samples of each of the compounds labelled A – F
- benzoic acid [Harmful] labelled Sample A
- 2-hydroxybenzoic acid [Harmful] labelled Sample B
- propanoic acid [Corrosive] labelled Sample C
- methyl ethanoate [Highly flammable; Irritant] labelled Sample D
- paracetamol [Harmful] labelled Sample E
- caffeine [Harmful] labelled Sample F
- Access to a melting point apparatus
- Melting point tubes
- Test tubes
- Sodium carbonate [Irritant] or sodium hydrogen carbonate [Low hazard]
- Spatula
- Disposable pipettes
- Universal Indicator solution