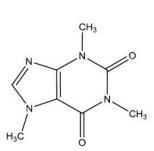
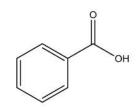
Problem 8: Compound confusion

Pre-Lab answers

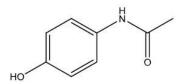
1.



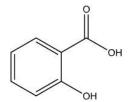


benzoic acid, m.p. 122 °C

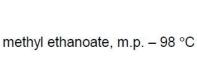
caffeine, m.p. 238 °C

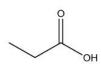


paracetamol, m.p. 170 °C



2-hydroxybenzoic acid, m.p. 159 °C





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;

$$\begin{array}{rrrr} \mathsf{C_6H_5COOH} + & \mathsf{NaHCO_3} \rightarrow \mathsf{C_6H_5COO^-Na^+} + & \mathsf{H_2O} + & \mathsf{CO_2} \\ 2 & \mathsf{C_6H_5COOH} + & \mathsf{Na_2CO_3} \rightarrow 2 & \mathsf{C_6H_5COO^-Na^+} + & \mathsf{H_2O} + & \mathsf{CO_2} \end{array}$$

For 2-hydroxybenzoic acid;

 $C_6H_5(OH)COOH$ + NaHCO₃ → $C_6H_5(OH)COO^-Na^+$ + H₂O + CO₂ 2 $C_6H_5(OH)COOH$ + Na₂CO₃ → 2 $C_6H_5(OH)COO^-Na^+$ + H₂O + CO₂

For propanoic acid;

 $\begin{array}{l} C_2H_5COOH + NaHCO_3 \rightarrow C_2H_5COO^-Na^+ + H_2O + CO_2 \\ 2 C_2H_5COOH + Na_2CO_3 \rightarrow 2 C_2H_5COO^-Na^+ + H_2O + CO_2 \end{array}$



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 $CHCI_3$ or $(CH_3)_2SO_2$ present in the bottles of the deuterated solvents.
- c) CDCl₃ Residual solvent peak in ¹³C NMR δ C 77.2 ppm, triplet DMSO Residual solvent peak in ¹³C NMR δ C 39.5 ppm, septuplet

5.

m/z 43 = acylium ion н₃с

 $\left[\mathsf{HOC}_{6}\mathsf{H}_{4}\mathsf{NHC}(\mathsf{O})\mathsf{CH}_{3}\right]^{\textbf{+}}\rightarrow\ \left[\mathsf{CH}_{3}\mathsf{C}(\mathsf{O})\right]^{\textbf{+}}\ \textbf{+}\ \left[\mathsf{HOC}_{6}\mathsf{H}_{4}\mathsf{NH}\right]^{\textbf{+}}$



Teacher and Technician Pack



Proposed method

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 (v 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 KLearn Chemistry



This resource was downloaded from <u>https://rsc.li/3PsEwvB</u>

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

