

Problem 5: Coursework conundrum

Curriculum links;

oxidation of alcohols, carboxylic acids

Practical skills;

recrystallisation, thin layer chromatography

A lazy student has contacted the students for help with purification of his sample of benzoic acid (contaminated with benzyl alcohol and Cr^{3+} residues). Recrystallisation of the sample is followed by TLC analysis to prove its purity.

Extension discussion points:

- What is the chemistry behind the potassium permanganate TLC stain?
- Why is it important to dissolve the sample in the minimum quantity of hot solvent?

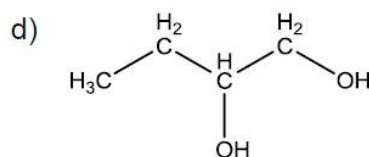
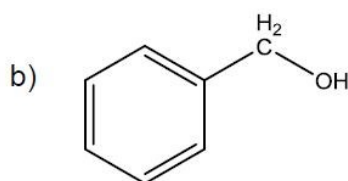
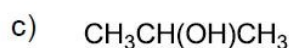
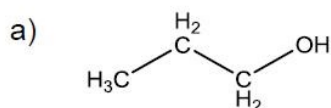
Pre-Lab questions

(Remember to give full references for any information beyond A-level that you find out)

1. Acidified potassium dichromate is a common oxidising agent for alcohols. In the process of the reaction $\text{Cr}_2\text{O}_7^{2-}$ is reduced to Cr^{3+} . Write two half equations for the oxidation of ethanol to ethanoic acid using acidified potassium dichromate [HINT: In the oxidation process, the carbon bonded to the oxygen is the atom undergoing oxidation (oxidation state $-1 \rightarrow +3$)].

Combine the two half equations to give the full redox equation for the reaction.

2. For each of the alcohols below, name the alcohol and draw and name the product obtained from the oxidation of the alcohol after refluxing in excess acidified potassium dichromate.



3. Impure solids can be purified by recrystallisation. Outline the steps involved in the purification of a solid by recrystallisation

4. Generally, organic compounds are insoluble in polar solvents such as water and ethanol. However carboxylic acids are an exception, with short chain carboxylic acids being totally miscible with both water and ethanol. Use your understanding of the interactions involved to explain the data below (taken from http://www.auburn.edu/~deruija/pda1_acids1.pdf)

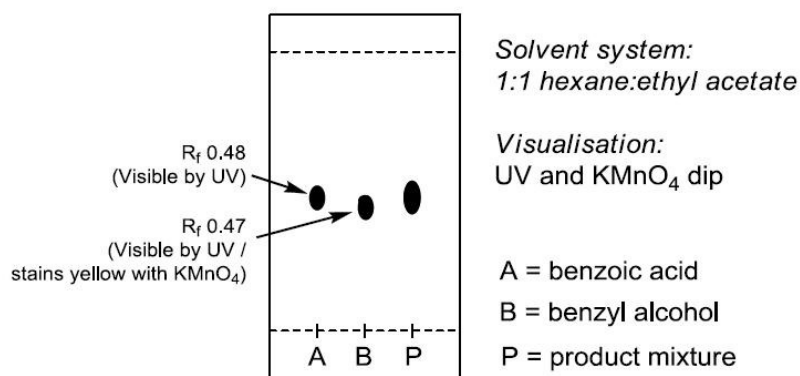
RCOOH; R=	Solubility in water / g per 100 cm³	Solubility in ethanol / g per 100 cm³
H-	soluble	soluble
CH ₃ -	soluble	soluble
CH ₃ CH ₂ -	soluble	soluble
CH ₃ CH ₂ CH ₂ -	soluble	soluble
CH ₃ CH ₂ CH ₂ CH ₂ -	3.7	soluble
CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ -	1.0	soluble
C ₆ H ₅ -	0.34	soluble
CH ₃ (CH ₂) ₈ -	0.015	soluble
CH ₃ (CH ₂) ₁₀ -	Insoluble	100
CH ₃ (CH ₂) ₁₂ -	Insoluble	soluble
CH ₃ (CH ₂) ₁₆ -	Insoluble	5.0

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Dear scientist,

Help! My A-level chemistry coursework is due in at the end of the week and I have a problem. My product is not pure ;-(

The coursework practical involved the oxidation of benzyl alcohol to form benzoic acid by refluxing the alcohol in acidified potassium dichromate overnight. However, when isolated my reaction has not gone to completion and my product is not clean. When I run a TLC of my product, despite appearing to be only one spot by TLC analysis, staining with KMnO_4 reveals that it is in fact a mixture. My product is also slightly green indicating the presence of left over Cr^{3+} salts plus I think I left some of the anti-bumping granules in the mixture as well!



How can I purify my product? I have included a sample of my product mixture with this letter. I know this is cheating but can you check that my product is indeed impure by repeating the TLC and then purify it for me please. As I will need to submit details of my practical please also provide a full procedure and rationale for the method used together with a sample of pure benzoic acid and a TLC plate showing its purity.

You're a lifesaver ;-)

L. A. Zyudent

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