NEATH PORT TALBOT COLLEGE
COLEG CASTELL NEDD PORT TALBOT

School of Maths & Science
Science Practical

Preparation and Purification of 4-MethylBenzeneCarboxylic Acid

◆ Aim

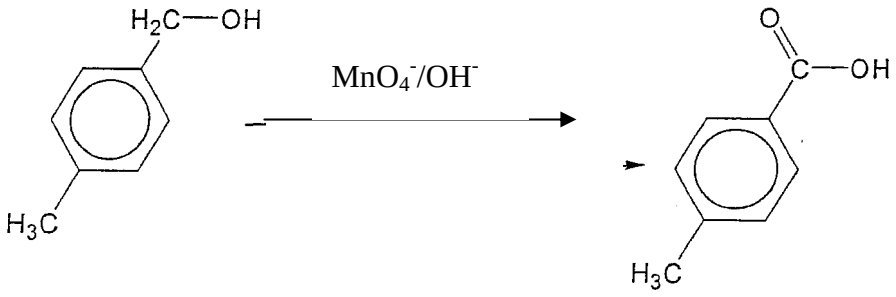
At the end of this experiment you should be able to:

1) Oxidise a primary alcohol to a carboxylic acid and outline the equations and oxidation states of the species involved.
2) Obtain a pure sample of 4-methylbenzenecarboxylic acid and analyse its purity.

◆ Introduction

Both primary and secondary alcohols of the general formula RCH2OH and R1R2CH(OH), respectively, readily undergo oxidation reactions with a variety of reagents. In the case of primary alcohols the oxidation had sometimes be arrested at the intermediate stage of the aldehyde, RCHO, but complete oxidation to the carboxylic acid, RCOOH, occurs more often. On the other hand, for secondary alcohols, oxidation leads only to a single product, the ketone R1R2CO.

In this investigation, the primary alcohol (4-methylphenyl) methanol, CH3C6H4CH2OH, on oxidation produces 4-methylbenzenecarboxylic acid, CH3C6H4COOH, which, as a solid, is somewhat easier to manipulate than a liquid.
The $CH_3C_6H_4CH_2OH$ to $CH_3C_6H_4COOH$ conversion uses MnO_4^- ions under alkaline conditions



◆ Safety

Control Measures

- The wearing of **safety goggles, gloves and a laboratory coat** at all times will be sufficient to take account of most hazards and significant risks.
- Keep stoppers on bottles as much as is possible.
- All waste is to be placed in the labelled container immediately after use.
- You are reminded of the need of good laboratory practise in order to maintain a safe working environment.

Hazards

- **Corrosive** Sat. Potassium permanganate solution, Na_2CO_3, Sodium Sulfite solution (25wt%)
- **Harmful/Irritant** (4-methylphenyl)methanol

♦ Procedure

1) Weigh 2 g of (4-methylphenyl) methanol into a 100 cm^3 round bottom flask (record the weight)

2) Weight out 1g of sodium carbonate into a beaker and add to 45 cm^3 of a sat. potassium permanganate (Vll) solution (stir to dissolve the sodium carbonate).

3) Add the solution (from Part 2) to the round bottom flask and reflux the mixture for 15 mins. (This leads to the formation of the sodium salt of 4-methylbenzenecarboxylic acid and the precipitation of brown manganese(IV) oxide).

4) After cooling, remove the condenser and place your flask in the fumehood.

5) CAREFULLY acidify the mixture by cautiously adding concentrated sulfuric acid in small quantities (drops), accompanied by drops of a sodium sulfite (Na_SO_3) solution, while stirring the solution.
NOTE:the flask will become very hot as this is an exothermic process.

6) Repeat the addition (in Part 5) until all of the brown precipitate of manganese dioxide ($Mn0_2$) has dissolved (you should get a colourless solution).

NOTE: be patient, the reaction of the brown precipitate of $Mn0_2$ with the acidified sodium sulfite may take a few minutes to get underway and for the precipitate to clear. If

the precipitate does not to disappear, test the pH, if the solution is acidic, bring the contents of the flask to the boil and filter hot.
DUE to the heat generated by this reaction, the methylbenzoic acid will be dissolved in solution, if you add too much acid, you will find it difficult to get the methylbenzoic acid to crystallise.

7) Transfer the mixture to a beaker and cool on ice to allow the acid to crystallise (**if you get no crystals forming AND the solution is around 6 °C, you might be allowed to boil off some of the solvent, however, you must ask permission to do this!!!**)

8) Collect the crystals by Buchner filtration and wash with 10 cm^3 of COLD water. Re-crystallise from water to obtain pure crystals (initially dry on the pump, then dry in an oven at 50 °C). Record the yield and melting point.

♦ Results:

Weight of (4-methylphenyl)methanol used: _____

Weight of 4-methylbenzoic acid obtained: _____

Melting point determined for 4-methylbenzoic acid: _____

♦ Calculations

Calculation of yield:

1) Write the overall equation for the reaction:

2) Calculate the number of moles of (4-methylphenyl)methanol used

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3) What is the mole ratio of (4-methylphenyl) methanol to 4-methylbenzoic acid? (consider reaction equation)

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4) Calculate the ***theoretical yield*** for the reaction: i.e., the mass of 4-methylbenzoic acid expected if 100% of the (4-methylphenyl) methanol reacted and no product was lost during the experiment.

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5) Calculate your ***percentage yield*** ([actual yield/theoretical yield] x 100)

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1) The oxidation of (4-methylphenyl) methanol to 4-methylbenzoic acid is accompanied by a colour change of the reaction of the $Mn0_4^-$ ions (purple) to $Mn0_2$ (brown). What is the oxidation state of Mn in both of these species.

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2) The oxidation reaction that occurred here may be represented by the following two half equations:

$CH_3C_6H_4CH_2OH + 4OH^- - 4e^- \longrightarrow CH_3C_6H_4COOH + 3H_20$

and $MnO_4^- + 2H_2O + 3e^- \longrightarrow MnO_2 + 40H^-$

Combine these two equations to deduce an overall stoichiometric equation for the reaction.

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3) In the procedure MnO_2 is removed by reacting with sodium sulfite. This is a redox process. Give the half equations for the reactions that are occurring (Hint, $Mn0_2$ is reduced)

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4) If we oxidised a primary alcohol using potassium dichromate, give the colour change observed and the oxidation states of the chromium ions giving rise to each colour.

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