PJJ's Chemistry

Mandatory Experiments Summary

2008

Leaving Certificate 2007 *Mandatory Experiments*

Syllabus section	**Title** - **click on the experiment title to go straight to the summary of the experiment**
1.1	**Flame tests**
1.2	**Redox reactions of Group VII elements.** **Halogens as oxidising agents with bromides, iodides, Iron(II) and sulphites**
	Displacement reactions of metals
2.1	**Tests for anions in aqueous solutions:**
3.1	**Determination of the relative molecular mass of a volatile liquid**
4.1	**Preparation of a standard solution of sodium carbonate**
4.2	**Standardisation of a hydrochloric acid solution**
4.2a	Hydrochloric acid / Sodium hydroxide titration and use of this reaction to make salt NaCl
4.3	**Determination of the concentration of ethanoic acid in vinegar**
4.4	**Determination percentage water of crystallisation in a sample of hydrated sodium carbonate**
4.5	**A potassium manganate (VII) / ammonium iron(II) sulphate titration**
4.6	**Determination of the amount of iron in an iron tablet**
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4.8	**Determination of the percentage of hypochlorite in bleach**
5.1	**Determination of the heat of reaction of hydrochloric acid with sodium hydroxide**
5.2	**Preparation and properties of ethyne**
6.1	**Monitoring the rate of production of oxygen from hydrogen peroxide using MnO_2**
6.2	**Effect of concentration on reaction rate using sodium thiosulphate and hydrochloric acid.**
6.2a	**Effect of temperature on reaction rate using sodium thiosulphate and hydrochloric acid.**
7.1	**Recrystallisation of benzoic acid and determination of its melting point**
7.2	**Preparation of soap**
7.3	**Preparation_and_Properties_of_ethene**
7.4	**Preparation and properties of ethanal**
7.5	**Preparation and properties of ethanoic acid**
7.6	**Extraction of clove oil from cloves [or similar] by stem distillation**
7.7	**Separation of a mixture of indicators using paper, thin layer or column chromatography**
8.1	**Simple experiments to Illustrate Le Chatelier's Principle**
8.1a	**To show effects of concentration changes on the equilibrium mixture**
9.1	**Colorimetric experiment to estimate free chlorine in swimming pool water or bleach**
9.2	**Determination of total suspended and dissolved solids in p.p.m.**
9.2a	**Determination of pH**
9.3	**Estimation of total hardness using ethylenediaminetetraacetic acid. [edta]**
9.4	**Estimation of dissolved oxygen by redox titration**
	Calculating Percentage Yields
	Titration Precautions
	Organic Synthesis

Exp 1.1 Flame tests

- Place sample of salt on clock glass
- Moisten with conc. HCl
- Pick up sample with clean nichrome or platinum wire
- Hold in non-luminous Bunsen flame
- Note colour of flame
  - **Sodium Yellow**
  - **Potassium Lilac [crimson through blue glass]**
  - **Copper Blue-green**
  - **Lithium Crimson**
  - **Strontium Red**
  - **Barium Green**
- Line spectra can be seen using a spectroscope

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Exp 1.2 Redox Reactions of Group VII and Displacement Reactions of Metals

Redox reactions

- Make solutions of
  - sodium sulphite
  - Iron(II) *sulphate*
  - *Sodium* Bromide
  - *Potassium* Iodide
- Make chlorine gas by reacting conc. HCl with $KMnO_4$ crystals
- Pour it into each solution, shake and note what happens
  - Sulphite is oxidised to sulphate *[use test from Exp. 2.1]*
  - Green iron(II) is oxidised to brown iron(III)
  - Colourless bromide solution is oxidised as Red bromine forms
  - Colourless iodide solution is oxidised as brown iodine forms

Displacement reactions

- Place copper sulphate solution in 2 test tubes
- Add Zn to one and Mg to another
- Blue colour of Cu^{2+} disappears as Cu is displaced by Zn
- Blue colour of Cu^{2+} disappears as Cu is displaced by Mg
- More active metal displaces less active metal from solution
- $Zn_{(s)} + Cu^{2+}{}_{(aq)} = CU_{(s)} + Zn^{2+}{}_{(aq)}$

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Exp 2.1 Identifying Anions

Anions: Negatively charged ions.
Attracted to opposite charge of the anode in electrolysis.

Chloride Cl^-

- Place in test tube and dissolve
- Add acidified silver nitrate solution
- White precipitate forms
- Add Ammonia solution
- White Precipitate re-dissolves

Nitrate NO_3^{1-}: *[Brown Ring Test]*:

- Place in a test tube and dissolve
- Add Iron (II) sulphate solution and mix

- Pour in Concentrated sulphuric acid (Hold test tube at 45 degrees)
- Acid sinks to bottom and brown ring forms between the layers

Sulphite: SO_3^{2-}

- Place in a test tube and dissolve
- Add Barium Chloride solution
- White precipitate of Barium Sulphite should form
- Add some dilute HCl
- White precipitate should re-dissolve *[compare to sulphate]*

Sulphate SO_4^{2-}

- Place in a test tube and dissolve
- Add some Barium Chloride solution
- White precipitate of Barium Sulphate forms
- Does not re-dissolve in dilute HCl *[compare to sulphite]*

Carbonate: CO_3^{2-}

- Place sample in test tube and add dilute HCl
- If a gas is produced test with lime water - if CO_2 then lime water goes milky
- Substance is either a carbonate or hydrogencarbonate.
- Add magnesium sulphate to solution
- White precipitate then carbonate if not it is a hydrogencarbonate

Hydrogencarbonate HCO_3^{2-}

- Place sample in test tube and add dilute HCl
- If a gas is produced test with lime water - if CO_2 then lime water goes milky
- Substance is either a carbonate or hydrogencarbonate.
- Add magnesium sulphate solution
- If no white precipitate then it is a hydrogencarbonate

Phosphate PO_4^{3-}

- Place in a test tube and dissolve
- Add a few drops of Ammonium molybdate solution
- Add a few drops of concentrated HNO_3
- A yellow precipitate forms [may need to be heated in water bath]

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Exp. 3.1 To Measure the Relative Molecular Mass of a Volatile Liquid

- Find mass of dry conical flask, rubber band and aluminium foil
- Pour 10cm³ of volatile liquid [chloroform] into conical flask
- Seal top with foil and rubber band – put small hole in foil
- Place flask in a large beaker of boiling water
- Leave flask till all liquid has evaporated at 100^0C
- Remove flask and allow to cool - the vapour condenses back to liquid
- Dry outside of flask and foil
- Reweigh the flask, foil and band
- Calculate mass of the liquid whose vapour filled flask [change in mass]
- Find volume of flask by filling with water and pouring into graduated cylinder
- Record atmospheric pressure using barometer.

- Calculate the volume of the vapour at S.T.P using: $V_1 = P_2V_2T_1/ P_1T_2$
- V_2 = volume of vapour at S.T.P.
- Know the mass of volume V_2
- Find the mass of 1cm³ [mass / V_2]
- Multiply by 22,400 to get the mass of 22,400cm³ of vapour = RMM

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Exp 4.1 Preparing a Standard solution of Sodium Carbonate

- Weigh out 2.65 g of anhydrous Na_2CO_3 on a clock glass [[23*2] + 12 +[16*3]/10 *250/1000]
- Place in beaker with 100 ml deionised water with washings
- Stir to dissolve
- Place into 250 ml volumetric flask with washings
- Make up to mark with deionised water
- On level surface with bottom of meniscus and eye level with mark
- Invert 20 times to make homogeneous
- Label

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Exp 4.2 To standardise a solution of HCl using a standard solution of Na_2CO_3

- Pipette 25 cm^3 dil. HCl into a 250cm^3 volumetric flask
- Make up to the mark with deionised water (dilutes acid to a factor of 10)
- Mix well to make homogeneous
- Pipette 20ml HCl into a conical flask
- Add methyl orange indicator [SAWBMO] and note the colour
- Add Na_2CO_3 from burette
- Mix constantly
- Wash any drops from side with deionised water [doesn't affect amount acid in flask]
- Note volume as indicator changes colour (red → orange)
- Do one rough and two accurate titres and take the average of two accurate
- Use $V_aM_a/n_a = V_bM_b/n_b$ to solve
- Multiply answer by 10 to allow for dilution

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Exp 4.3 To determine the percentage of Ethanoic acid in vinegar

- Pipette 20 ml NaOH into conical flask,
- Add a few drops of phenolphthalein indicator [WASBPH], turns pink.
- Put Ethanoic acid in burette (remove air bubble).
- Record initial volume
- Add to NaOH in flask
- When pink turns colourless note volume (adding dropwise near end).
- Do one rough and two accurate and take the average of two accurate

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Exp 4.4 To determine the percentage of water of crystallisation in hydrated sodium carbonate (washing soda)

- weigh out 5g washing soda crystals [Na_2CO_3]
- dissolve in 100cm^3 deionised water, stir

- transfer to 250 cm^3 flask with washings, make solution up to mark, invert 20 times
- pipette 25cm^3 of Sodium carbonate solution into conical flask
- add 3 drops of methyl orange indicator goes yellow [SAWBMO]
- fill burette with HCl solution
- titrate: colour change from yellow to pink
- do one rough and 3 accurate – average of 2 accurate

- Calculation - use: $\frac{Va*Ma}{na} = \frac{Vb*Mb}{nb}$
- Mass of anhydrous Na_2CO_3 = M * 106
- mass of water = mass of crystals - mass of anhydrous Na_2CO_3 =
- **% anhydrous Na_2CO_3** = $\frac{\text{mass of anhydrous } Na_2CO_3}{\text{mass of crystals}}$ **% water** = $\frac{\text{mass of water}}{\text{mass of crystals}}$
- $\frac{\%\text{ anhydrous } Na_2CO_3}{106} : \frac{\%\text{ water}}{18}$

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Exp 4.5 Potassium manganate(VII) / Ammonium iron(II) sulphate titration

- Ammonium iron(II) sulphate primary standard solution
- $KMnO_4$ in burette – read from top of meniscus
- Pipette 25 ml ammonium iron(II) sulphate into conical flask
- Add 10 ml dil. H_2SO_4 to make sure doesn't stick at brown pptte. of MnO_2
- Mn^{2+} autocatalyst - product of reaction catalyses reaction
- Acts as own indicator
- End point when pink colour remains permanently
- Calculation - use: $\frac{Va*Ma}{na} = \frac{Vb*Mb}{nb}$
- Equation $MnO_4^{1-} + 5\ Fe^{2+} + 8\ H^+ = Mn^{2+} + 5\ Fe^{3+} + 4\ H_2O$

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Exp 4.6 To determine the amount iron in iron tablet.

- Find the mass of the five iron tablets.
- Crush with pestle and mortar in dilute H_2SO_4 [to stop Fe^{2+} - Fe^{3+}]
- Transfer the paste with washings to beaker
- Stir to dissolve the paste.
- Transfer solution into 250cm^3 volumetric flask with washings
- Make up to mark with deionised water. Eye/meniscus level with mark. Invert 20 times.
- Pipette 25cm^3 of tablet solution into conical flask.
- Add 20cm^3 of dilute sulphuric acid to ensure full reaction
- Titrate with $KMnO_4$ from burette until a permanent pink colour remains
- Equation $MnO_4^{1-} + 5\ Fe^{2+} + 8\ H^+ = Mn^{2+} + 5\ Fe^{3+} + 4\ H_2O$
- Mn^{2+} autocatalyst - product of reaction catalyses reaction
- One rough and two accurate. Average of 2 accurate
- Calculation - use: $\frac{Va*Ma}{na} = \frac{Vb*Mb}{nb}$
- Mass = molarity * RMM = M * 152 =
- Multiply by 1000 to give mg
- Divide by 5 to give mass of each tablet

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Exp 4.7 An Iodine/Thiosulphate Titration

- Make up 0.05M solution of iodine
- By reacting 0.017 M potassium iodate with excess potassium iodide
- The iodide then reacts with the sodium thiosulphate, which is added from a burette.
- $I_2 + 2\ S_2O_3^{2-} = S_4O_6^{2-} + 2I^-$
- Weigh 6.25g of sodium thiosulphate crystals onto clock glass
- Transfer crystals to beaker containing 100cm^3 of de-ionised water
- Stir and when dissolved transfer to 250cm^3 volumetric flask with washings
- Make up to mark with de-ionised water. Invert 20 times.
- Pipette 25cm^3 of potassium iodate
- Use graduated cylinder to add 20cm^3 of dilute sulphuric acid, followed by 10cm^3 of 0.5M potassium iodide solution to the conical flask. Note a reddish/brown colour of liberated iodine.
- Titrate. Until pale yellow. Add a few drops of starch indicator - blue colour is observed.
- Continue adding thiosulphate solution drop by drop until blue colour disappears, to give colourless solution. Note the titration figure.
- One rough and two accurate titres. Average two accurate
- $V_o * M_o / n_o = V_r * M_r / n_r$

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Exp 4.8 To determine the percentage (w/v) of sodium hypochlorite NaOCl in bleach

- Pipette 25 ml concentrated bleach into 250 ml volumetric flask
- Fill up to the mark with deionised water, invert it 20 times.
- Diluted by factor 10
- Pipette 25 ml bleach into conical flask, add 20 ml sulphuric acid and 10 ml of 0.5M potassium iodide (KI) solution. Note reddish/brown colour
- Titrate until a pale yellow colour
- Add a few drops of starch indicator, note blue colour
- Add thiosulfate drop by drop until colour change from blue to colourless.
- Note titration figure.
- Do one rough and two accurate readings. Take average of two accurate
- Calculate molarity of sodium hypochlorite $NaClO_3$
- $\mathbf{V_o * M_o / n_o = V_r * M_r / n_r}$
- Multiply by 10 for dilution
- Calculate mass of hypochlorite in 1 litre [mass = molarity * RMM of hypochlorite]
- Divide by 10 to get mass in 100g

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Exp 5.1 Determine the heat of reaction on of hydrochloric acid with sodium hydroxide

- 100 ml 0.1 M HCl
- 100 ml 0.1 M NaOH
- take temperature of both and average = starting temp
- mix in polystyrene cup [insulator so no heat escapes]
- Record highest temp
- Calculate change in temp
- $H = mc\Theta$
- Mass = 0.2 kg
- C = 2.4 kJ kg^{-1}
- Θ = temp change
- Heat change for 0.1 mole therefore multiply by 10 for 1 mole

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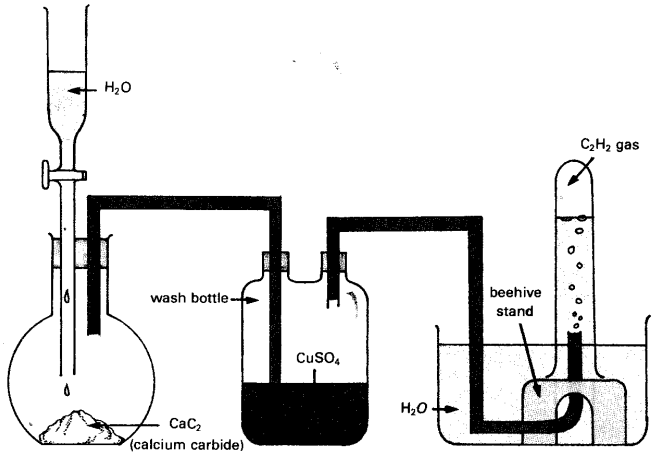
Exp 5.2 Preparation and Properties of Ethyne

$$CaC_{2(s)} + 2\,H_2O_{(l)} \;\Longrightarrow\; C_2H_{2(g)} + Ca(OH)_{2(s)}$$

Calcium Dicarbide — Ethyne — Slaked Lime

- Calcium dicarbide is a grey lumpy solid
- As the reaction proceeds lots of heat is produced [very exothermic] causes fizzing and spattering.
- The solid product, $Ca(OH)_2$, is a white powder which occupies more space than the CaC_2
- Ethyne can be purified by passing it through acidified $CuSO_4$ solution
- To remove impurities such as Hydrogen Sulphide and Phosphine
- Produced by the reaction of the water with traces of Calcium Sulphide [CaS] and Calcium Phosphide [Ca_3P_2] in the CaC_2.
- Ethyne has a sickly sweet smell



- Burns in air with a very smoky yellow flame. Very hot in excess air - oxyacetylene burner
- Unsaturated $C \equiv C$
- Decolourises bromine water from Red to colourless – quickly
- Decolourises acidified manganate(VII) from purple to colourless MnO_4^- to Mn^{2+} – quickly

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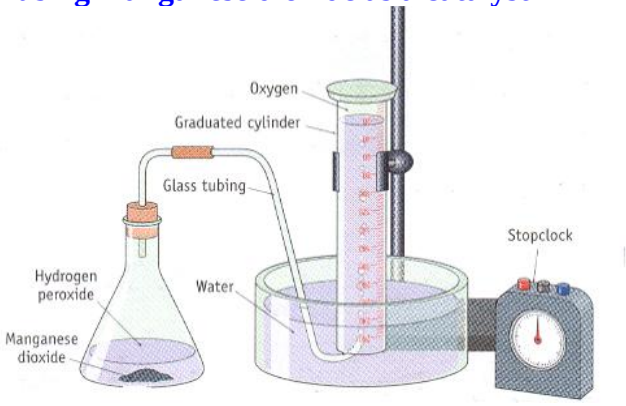
Exp 6.1 To monitor the rate of production of oxygen from hydrogen peroxide using manganese dioxide as a catalyst



- Place $50cm^3$ of hydrogen peroxide solution in conical flask
- Hold flask horizontal
- Add small amount of manganese dioxide to neck of conical flask, insert rubber stopper
- Turn flask to normal position and MnO_2 will fall in and the reaction will start.
- At 30 second intervals record volume of gas in cylinder till reaction over
- Plot a graph of volume of oxygen against time
- $H_2O_2 = H_2O + ½\ O_2$
- Average rate of evolution of oxygen $= \dfrac{\text{Total volume given off}}{\text{Time}}$
- Instantaneous rate = rise/ run of tangent drawn at any point on graph

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Exp 6.2 To determine the effect of concentration and temperature on the rate of reaction.

1: Temperature.

- Using sodium thiosulphate and dilute HCl
- Make up different % solutions (100%, 80%, etc.) in conical flask
- Pipette 50 ml thiosulphate into conical flask
- Place flask on paper marked with cross
- Add 10 ml dilute HCl
- Record time in seconds it takes for cross to disappear
- Repeat for each concentration
- Draw graph: rate of reaction [1000/s] against concentration
- Rate of reaction directly proportional to concentrations of reactants
- **Doubling concentration of one reactant doubles the rate of reaction**

2: Concentration

- Pipette 50 ml thiosulphate into conical flask \Using sodium thiosulphate and dilute HCl
- Place conical flask in water bath at different temperatures and Heat to required temperature
- Place the flask on paper marked with cross
- Add 10 ml dilute HCl
- Record time in seconds it takes for cross to disappear
- Repeat for each temperature
- Draw graph: rate of reaction [1000/s] against temperature
- **10°C rise doubles rate of reaction**

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Exp 7.1 Recrystallisation of Benzoic Acid and determination of its melting point

[a] Recrystallising

- Dissolve impure benzoic acid in minimum hot solvent [water]
- Filter using a hot apparatus to remove any insoluble impurities
- Cool to recrystallise
- Filter to retrieve crystals
- Wash crystals with cold solvent [wash away last traces of impurity with minimum solution of crystals]
- Dry [in desiccator]

[b] Melting point determination

- Take a very small amount
- Heat on aluminium block with thermometer in it
- Melts over several degrees = impure
- Melts sharply = pure

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Exp 7.2 Preparation of Soap

- Reaction is called **Saponification** or **alkaline hydrolysis**
- Mix olive oil [or any vegetable oil] with NaOH [The NaOH is 20% W/V] in ethanol
- **Alkaline hydrolysis** leads to formation of the sodium salt of the fatty acid
- Boil under **reflux** to speed up reaction and drive to completion
- Reflux stops volatile components escaping
- Ethanol removed by **distillation**
- **Residue** is excess NaOH, glycerol and soap dissolved in minimum hot water

- Cool and pour into concentrated NaCl solution [called **salting out**]
- Stir and allow to stand and cool
- Glycerol and NaOH dissolve in brine [concentrated salt solution]
- Soap does not dissolve in brine and forms a cake on top of the liquid
- Waterand glycerol and brine removed by Buchner Filtration [vacuum filtration].
- Soap rinsed with ice-cold water to reduced amount lost by dissolving

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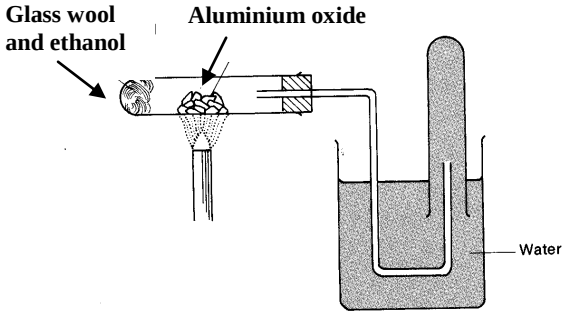
Exp 7.3 Preparation and Properties of Ethene

- Pass ethanol vapour over heated Al_2O_3 and collect ethene over water
- Al_2O_3 acts as a catalyst
- Glass wool holds the ethanol in place to stop it reaching the Al_2O_3 powder as a liquid.

$$\mathbf{C_2H_5OH_{(l)} = C_2H_{4(g)} + H_2O_{(l)}}$$

or

$$\mathbf{C_2H_5OH_{(l)} - H_2O_{(l)} = C_2H_{4(g)}}$$



- Colourless gas same density as air
- Insoluble in water and polar solvents
- Soluble in non-polar solvents e.g. cyclohexane
- Burns in air to give CO_2 and H_2O and heat [sometimes explosively]
- Unsaturated C = C shown by
- Ethene decolourises bromine water from Red to colourless quickly
- Ethene decolourises acidified manganate (VII) from purple to colourless quickly

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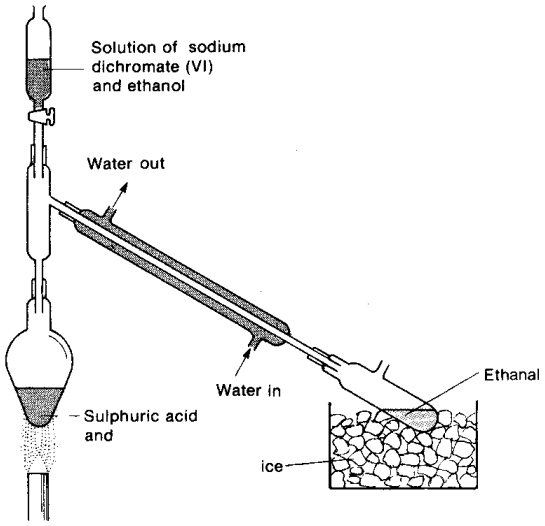
Exp 7.4 Preparation and Properties of Ethanal

- Use a **Primary Alcohol**
- Make sure **alcohol is in excess [or $Cr_2O_7^{2-}$ limiting reactant**
- Put conc. H_2SO_4 into the pear shaped flask.
- If diluting the acid, add acid to water, mix constantly and cool, because the acid reacts very exothermically with water.
- Add **anti-bumping granules**.[Stops **bumping** (large bubbles) which may damage apparatus by forming lots of small bubbles instead of a few large ones]
- Put a mixture of dichromate dissolved in alcohol [ethanol] into a dropping funnel.
- Heat acid to boiling and stop heating
- Then add alcohol/dichromate mixture at a rate such that
  (i) the acid keeps boiling {**exothermic reaction**} and
  (ii) the rate of addition of the mixture equals the rate of production of ethanal.



- Solution of ethanol/dichromate is **amber** due to dichromate $\mathbf{Cr_2O_7^{2-}}$
- As reaction proceeds it goes **green** as $\mathbf{Cr^{3+}}$ is formed
- Remove the ethanal as soon as it is formed so no chance of it reacting further into a carboxylic acid.
- Pass through Leibig Condenser and collect ethanal in an ice bath - it is **volatile** [BP 20.8°C] and the ice bath stops it evaporating.
- **Water in at base and out at top** of condenser
- Distillate contains small amounts of impurities - water and ethanol boiled over with the ethanal.
- Dry by adding **anhydrous sodium sulphate** and shaking for 10 mins - then filter off Na_2SO_4.

- **Re-distil** distillate and collect the **fraction** boiling 20 to 23^{o}C. - leaves the last of the alcohol behind.
- Soluble in water due to polar carbonyl group – insoluble in cyclohexane
- Ethanal reduces **Fehling's solution** from blue to red precipitate when heated with it.
- Forms silver mirror on clean test tube when heated with Tollen's Reagent **ammoniacal silver nitrate**

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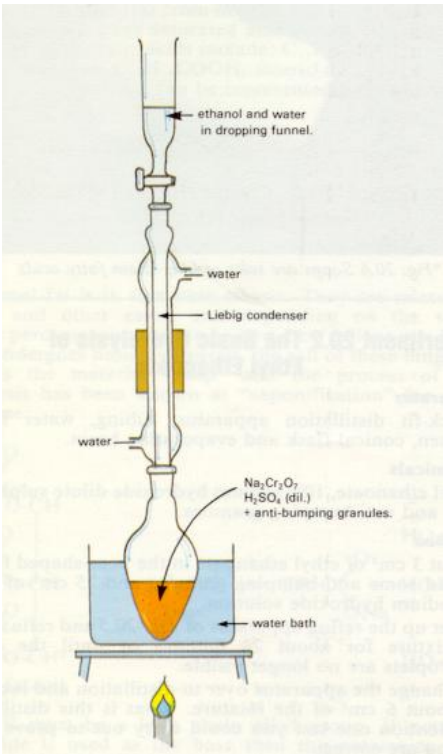
Exp 7.5 Preparation and Properties of Ethanoic Aid



- Heat Ethanol with **acidified dichromate** in **reflux** apparatus for **30 mins.**
- Make sure that the **oxidising agent** ($Cr_2O_7^{2-}$) is in **excess**.

$3\ C_2H_5OH + 2\ Cr_2O_7^{2-} + 16\ H^+ = 3\ CH_3COOH + 4\ Cr^{3+} + 11\ H_2O$

- Reflux stops volatile components escaping
- Alcohol is converted first to aldehyde and then onto a carboxylic acid.
- As reactions happen the orange dichromate ($Cr_2O_7^{2-}$) is turned into the green chromium(III) ion **(Cr^{3+})**.
- pH 3-4 because **weak acid** only partly dissociates in aqueous solution
- Turns **UI orange/yellow**
- Reacts with Na_2CO_3 to give sodium ethanoate, CO_2 and water.

$2\ CH_3COOH + Na_2CO_3 = 2\ CH_3COONa + CO_2 + H_2O$

- Reacts with Mg to give magnesium ethanoate and hydrogen

$Mg + 2\ CH_3COOH = (CH_3COO)_2Mg + H_2$

- Reacts with ethanol to give ethyl ethanoate and water [ester + water]. Add a few drops of conc. sulphuric acid as a catalyst
- Fruity smell of ester
- **$CH_3COOH + C_2H_5OH = CH_3COOC_2H_5 + H_2O$**
- Ethanoic acid removed fitting the condenser sideways and collecting the distillate.
- Impurities are water and ethanol.
- Remove water by shaking with anhydrous Na_2SO_4. for 5 mins.
- To remove alcohol distil and discard fraction boiling at 80 ^{o}C
- Uses of carboxylic acids / condiment / preservative / making esters/ cellulose acetate film

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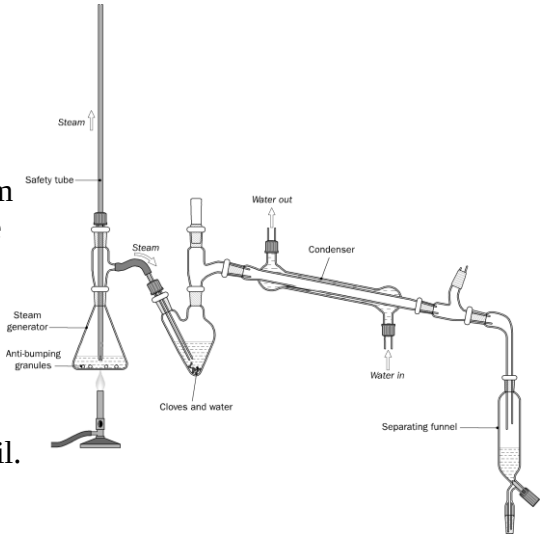
Exp 7.6 Extraction of Clove Oil from Cloves



- Make sure you have a safety opening to the atmosphere
- Steam distillation used because some components of clove oil have high BP and this temp would damage molecules in the oil
- Some organic compounds are immiscible with water. Usually these compounds have a low vapour pressure. After mixing them with water, however, the mixture will distil when the sum of the two vapour pressures reaches atmospheric pressure. It follows, then, that this must happen below the boiling point of water. This process is known as steam distillation.
- Note the mass of the cloves, and place them in the pear-shaped flask. Cover with a little warm water (about 5 cm^3).
- Place plenty of water in the **steam generator**, connect it and boil. Use anti-bumping granules in the steam generator.

- If the level of the boiling water in the steam generator falls too low, the system will not work smoothly. Remove the heat, carefully loosen the safety valve, and top up the steam generator with **hot** water. Reconnect everything and heat again.
- After 20 to 30 minutes disconnect steam generator to avoid **suck-back** then turn off the heat.
- Collect 40 - 50 cm^3 of the pale milky distillate [emulsion]. Note the smell
- Oil separated by dissolving in solvent, placing in separating funnel
- Collecting organic solvent fraction and then evaporating solvent.

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Exp 7.7 Separation of a mixture of indicators using Paper Chromatography

- Set up chromatography tank
- Place 1 cm depth of Solvent in tank
- Draw line 3 cm from bottom of paper and one near top
- Spot line with individual indictors and the mixture of indicators several times allowing to dry between applications
- Place end of chromatogram in solvent making sure spots are above solvent
- Cover tank and allow to run till solvent front reaches line near top
- Remove and dry
- Calculate and record Rf values [distance moved by substance / distance moved by solvent front] of each substance
- Ammonia vapour used to locate [develop] phenolphthalein spot

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Exp 8.1 Illustrations of Le Chatelier's Principle

[a] equilibrium between $CoCl_4^{2-}$ and $Co(H_2O)_6^{+}$ $CoCl_4^{2-} + 6\ H_2O = Co(H_2O)_6^{+} + 4\ Cl^-$
Blue Pink

- **Effect of Concentration**
- dissolve 4 g of cobalt chloride – 6 water in 40 cm^3 deionised water
- pink solution formed
- add Conc. HCl in fume cupboard till blue colour forms [gets Cl^- down by going left]
- add water pink colour returns [gets water down by going right]
- repeat several times

- **Effect of temperature**
- place test tube in water at 90°C – goes blue – system tries to lower temp by going to left endothermic
- place test tube in crushed ice – goes pink – system tries to raise temp by going to right exothermic

[b] Equilibrium between $Cr_2O_7^{2-}$ and CrO_4^{2-} $Cr_2O_7^{2-} + H_2O = CrO_4^{2-} + 2\ H^+$
orange yellow

- **Effect of Concentration**
- place $Na_2Cr_2O_7$ soln. in test tube – orange
- add some dil. NaOH - goes yellow - uses up H^+ system replaces by making H^+ and chromate [CrO_4^{2-}]
- Add dil. H_2SO_4 goes orange add H^+ system uses it up by making dichromate [$Cr_2O_7^{2-}$]

[c] Equilibrium between Fe^{3+} and $Fe(CNS)^2$ $Fe^{3+} + CNS^- = Fe(CNS)^{2+}$
yellow red

- Mix 5 ml of iron(III) chloride and Potassium thiocyanate in beaker – red because the equilibrium is to right

- Add conc. HCl – red colour disappears – Cl^- reacts with Fe^{3+} and removes it
- $Fe(CNS)^{2+}$ used up as Fe^{3+} is replaced – red colour goes

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Exp 9.1 To Estimate the Concentration of Free Chlorine in Swimming Pool Water or Bleach. (Using a colorimeter)

- HOCl called free chlorine put in as calcium hypochlorite [$Ca(OCl)_2$]
- pH low to keep conc. of free chlorine at maximum
- chlorine kills bacteria – if conc. Too high skin problems
- Colorimeter works on principle that absorbance[colour] is proportional to concentration
- Calibrate colorimeter. 0% = light switched off and 100% distilled water
- Wavelength for maximum absorbance
- Make up stock [standard] solutions 1, 2, 4, 8, 16 p.p.m. and run absorbance for each
- Draw graph absorbance vs. concentration
- Take unknown solution add 2% KI and ethanoic acid
- Turns brown due to release of iodine [I_2]
- Take unknown solution add Run unknown solution
- Read value from graph

Exp 9.2 To determine the total suspended solids (in p.p.m.) of a sample of water by filtration, (a) the total dissolved solids (in p.p.m.) of a sample of water, (b) the pH of a sample of water.

A. To measure the total suspended solids by filtration

- Fill volumetric flask to the mark with the sample of water.
- Find the mass of a dry filter paper.
- Filter the known quantity of water through the filter paper.
- Allow filter paper to dry.
- Find new mass of filter paper
- Calculate the mass of suspended solids in sample [the change in mass of filter paper]
- Calculate the mass of suspended solids in 1 L
  [mass of suspended solids in sample / volume of sample * 1000]

B. To measure the total suspended solids by Evaporation

- Find the mass of a clean dry beaker.
- Add a known quantity of filtered water from a graduated cylinder.
- Evaporate the water to dryness.
- Note the dissolved solids remain in the beaker.
- Allow the beaker to cool
- Dissolved solids = mass of beaker at end – mass of clean dry beaker [in mg]
- Mass in 1 Litre = mass of dissolved solids / volume of sample * 1000
- Results are in mg/L i.e. p.p.m.

C. To measure the pH of a sample of tap water

- Most accurate method is to use a pH meter.
- Calibrate meter in buffer solution
- Wash then Place electrode of the meter in a sample of water
- Read off the pH.

- If a pH meter is not available, pH paper may be used.

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Exp 9.3 Estimation of Total Hardness of water using Ethylenediaminetetraacetic acid

- Place 50 ml of hard water in conical flask
- Place 0.01 M EDTA in burette
- Add 1 cm^3 of buffer solution [pH 10] to keep alkaline so indicator works properly
- Add 5 drops of Erichrome black – gives wine red colour
- Add EDTA from burette until solution turns blue
- Take 1 rough and 2 accurate titres - average 2 accurate
- 1 cm^3 of 0.1 M EDTA ≡ I mg $CaCO_3$
- Multiply average titre value by 20 to find mg L^{-1} [p.p.m.]
- Unboiled = permanent + temporary
- Boiled water = permanent only
- Temporary = Unboiled value – boiled value

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Exp 9.4 To measure the amount of Dissolved Oxygen by Winkler Method

- Rinse bottle to stop bubbles forming
- Fill and stopper under water
- add 1cm^3 of conc. manganese(II) sulphate [so not to upset volume]
- add 1cm^3 of conc. alkaline KI [so not to upset volume]
- Brown precipitate forms – if white precipitate forms no oxygen present.
- Add 1cm^3 of conc. H_2SO_4.
- Solution goes brown colour due to the liberated iodine.
- Put 100 cm^3 of iodine solution into conical flask.
- 0.005 M Sodium thiosulphate in burette is standard solution
- Titrate until a pale straw coloured
- Add a few drops of starch indicator
- Continue titrating until the blue/black colour disappears.
- Do one rough and 2 accurate – average 2 accurate
- Calculate the concentration of O_2 in water expressing your results in p.p.m.
- Let dissolved oxygen = a and Let thiosulphate = b
- Ratio of dissolved oxygen to thiosulphate = 1:4 therefore $n_a = 4$ and $n_b = 1$
- Calculate molarity of the oxygen
- Multiply by 32 to convert to grams per L
- Multiply by 1000 to convert grams to mg/L [p.p.m.]

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Calculating Percentage Yield

The organic practical question usually contains a calculation

Percentage yield = actual yield / theoretical yield * 100

Actual yield

This is the amount of substance actually produced by the experiment
This is often given as a volume along with a density
e.g. 10 cm^3 of ethanol density 0.8 g cm^{-3}

Mass = density * volume = 10 * 0.8 = 8 g

Theoretical yield

This has to be worked out from the equation
It assumes 100% conversion of reactants to products

Question

10cm^3 of ethanol [density = 0.8 g cm^{-3}] is reacted with excess ethanoic acid to form ethyl ethanoate. If 2.0g of ethyl ethanoate is formed what is the percentage yield?

Write balanced equation	**CH_3COOH + C_2H_5OH = $CH_3COOC_2H_5$ + H_2O**	
Underline substances given and asked about	**CH_3COOH + $\underline{C_2H_5OH}$ = $\underline{CH_3COOC_2H_5}$ + H_2O**	
Write down the number of moles of each of these substances in the equation.	1 mole	1 mole
Write down the units given and asked for	g	g
Convert the number of moles of each of these substances to the units given and asked for	46g	88g
Bring the substance you know the actual amount of down to one and do the same to the other side of the equation. [divide both sides by the given number]	$\frac{46}{46} = 1$	$\frac{88}{46}$
Bring it up to the given amount [by multiplying both sides by the given amount]	8 * 1 = 8 g	$\frac{88}{46} * 8 =$ **15.3g**

% yield = $\frac{\text{actual yield}}{\text{theoretical yield}} * 100$ $\frac{2}{15.3} * 100 =$ **13.07 %**

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Titration Precautions

Pipette

- Rinse with deionised water *to wash out any impurities*
- Then with the solution it is going to contain *to wash out the deionised water.*
- Fill using a pipette filler - *the solution may be poisonous or caustic*.
- Read from the bottom of meniscus
- meniscus level with the ring on the stem
- eye level with this.
- Empty into the conical flask and touch tip against the surface.
- DON'T BLOW it is calibrated to allow for the drop at the tip.

Burette

- Rinse with deionised water and then with the solution it is going to contain.
- Fill using a funnel and remove it as drops may fall from it or it may dip into the liquid giving a false level.
- Remove the air bubble from the tip by opening tap quickly
- Read from the bottom of meniscus - with eye level with this point.
- $KMnO_4$ - read from the top of the meniscus *(or from the bottom with a light behind)*
- Don't put NaOH in burette it may react with glass of burette or block tap *[not really valid now]*.

Conical Flask

- Rinse out with deionised water only.
- Place on white tile - *to see colour change more easily*.
- Mix continuously.
- Add only a few drops of indicator
  (They are weak acids or bases and may upset the results)
- Wash down drops on the side of the flask with deionised water.
  (This wont affect amount of reactant in flask or change the result.)

Volumetric Flask

- Long thin neck to make it accurate.
- Read from bottom of meniscus at eye level.
- Make sure it is at room temperature - *it is calibrated at 20^oC.*
- Mix by inverting 20 times to make sure the solution is homogeneous - *long thin neck makes this necessary.*

Titration

- Use the correct indicator *[SAWBMO]*
- Only 3 - 4 drops of indicator
- One rough and 2 accurate titres
- two accurate should be within 0.1 cm^3
- Average the 2 accurate titres
- Mix well by swirling
- Add from burette drop by drop near the **end point.**
- **Point at which reaction is complete - *shown by colour change***
- Identify the standard solution *[one you are given the concentration of] - for calculations that are to follow.*

Other Precautions

MnO_4^-

- must be acidified (with dil. sulphuric acid)
- Otherwise reaction stops at MnO_2 [brown precipitate.] instead of going on to the colourless Mn^{2+}.
- It acts as its own indicator going from purple to colourless.
- The **Mn^{2+}** acts as an **autocatalyst**. (One of the products of the reaction catalyses the reaction)
- Read from the top of the meniscus as it is too deeply coloured to see through it clearly

Iodine / thiosulphate [I_2 / $S_2O_3^{2-}$]

- Use **starch indicator**
- Blue/black while iodine (I_2) is present - colourless when iodine has gone i.e. I_2 all been turned to iodide (I^-).
- Don't add the starch indicator too early - *it may complex with iodine and ruin the result.*
- Add it when the iodine solution is straw coloured. *(e.g. Winkler Method)*

Indicator Choice

- SASBANY - Strong Acid Strong Base - Any indicator
- SAWBMO - Strong Acid Weak Base - Methyl Orange
- WASBPH - Weak Acid Strong Base - Phenolphthalein
- WAWBNONE - Weak Acid Weak Base - None

Standards

- **Standard Solution** is one whose concentration is known **accurately**
- **Primary Standard** is one that can be made up **directly** using a measured amount of **pure** solid.
- **Secondary Standard** Make up a solution and then standardise this solution using a primary standard.
- **Standardise** means to find the concentration of using titration
- Can't use MnO_4^- as **Primary Standard** - *it cant be got pure.*
- Can't use iodine [I_2] because *it sublimes*.
- Can't use KOH or NaOH they absorb CO_2 and moisture from air
- Can't use conc. H_2SO_4 absorbs water from air

Making a salt e.g. NaCl

- Use HCl and NaOH
- Do titration with indicator
- Get volumes
- Do without indicator
- Evaporate water leaving pure salt

Indicator Colours

- Methyl Orange Acid Red Alkali Orange/yellow
- Phenolphthalein Acid clear Alkaline Purple/pink

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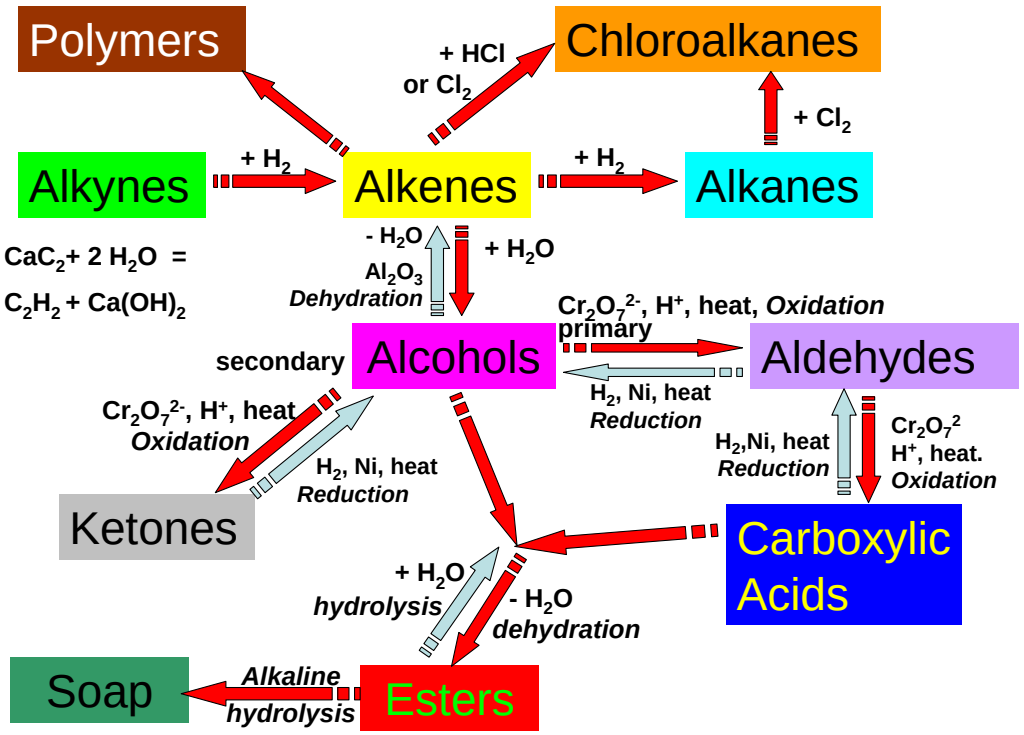
Organic Synthesis

Making complex molecules from simple ones by a series of reactions e.g. aspirin, DDT, penicillin, ibuprofen [know 2]

You need to be able to do three steps on this flow chart giving conditions



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