Edexcel International Chemistry A-level

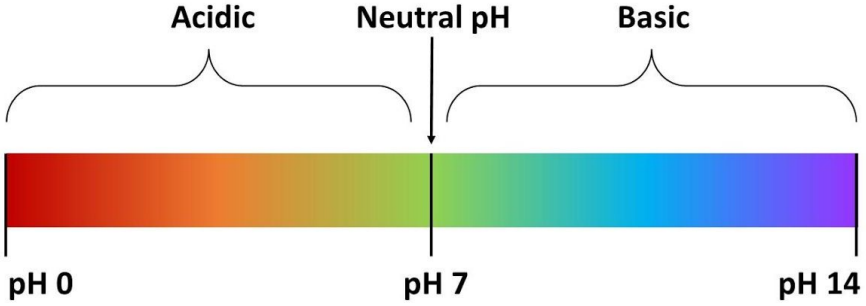
Practical 8

Analysis of Some Inorganic and Organic Unknowns

These notes are a summary of chemical tests needed for both Unit 3 (practical 8) and Unit 6 (practical 15).

Inorganic

Test for acid or base

- Use litmus paper
  - Blue litmus paper turns **red** = acid
  - Red litmus paper turns **blue** = base
- Use universal indicator and compare to a scale like the one below
  - 

Flame tests for Group I and Group II metal cations

Method:

- To clean the nichrome wire - dip the wire in a solution of concentrated HCl and then into a Bunsen burner flame.
- Dip the wire into the unknown compound and carefully place it at the top of the blue Bunsen burner flame.
- Observe the colour:
  - **Li^+ = red**
  - **Na^+ = orange/yellow**
  - **K^+ = lilac**
  - **Rb^+ = red**
  - **Cs^+ = blue**
  - **Mg^{2+} = no colour**
  - **Ca^{2+} = brick-red**
  - **Sr^{2+} = crimson red**
  - **Ba^{2+} = green**

Test for NH_4^+ ions

- Add aqueous NaOH and gently warm the mixture.
- The ammonia gas will turn moist pH indicator paper blue.
- Additionally, a pungent smell is given off.

Test for SO_4^{2-} ions

- Add acidified barium chloride solution.
- White ppt forms ($BaSO_4$).

Mixtures of anions

When carrying out the above test, it may be useful to add some HNO_3 before you add $Ba^{2+}_{(aq)}$.The carbonate anion also gives a white precipitate with barium cations, and addition of acid will remove any carbonates present in the mixture. Then, the test for sulfates can be conducted.

Halides

- Dissolve 2 spatulas of the unknown product in 10 cm^3 of water.
- Then, add a few drops of dilute nitric acid and silver nitrate solution. Observe the colour of any precipitate formed:
    - Cl^- forms a white ppt with silver nitrate which is soluble in dilute NH_3.
    - Br^- forms a cream ppt with silver nitrate which is soluble in concentrated NH_3.
    - I^- forms yellow ppt with silver nitrate which is insoluble in NH_3.
- The colours are all quite similar, so the solubility test with ammonia is a good way to confirm the identity of the halide ion present.

Halides can also be tested for by reacting a solid halide salt with conc. H_2SO_4.

- Chloride produces steamy fumes of HCl.
- Bromide produces steamy fumes, brown vapour (HBr, Br_2, SO_2).
- Iodide produces steamy fumes, purple vapours, black solid (I_2, HI, SO_2 or even solid S).

Metal cations

Testing for various metal cations using sodium hydroxide. Group I hydroxides are all soluble.

Metal cation	add some NaOH	add more NaOH
Mg^{2+}	White suspension	n/a
Ca^{2+}	White suspension, but more soluble than $Mg(OH)_2$	n/a
Sr^{2+}, Ba^{2+}	Soluble hydroxides.	n/a
Zn^{2+}	White ppt, $Zn(OH)_2$. Soluble in NH_3, colourless solution, $Zn(NH_3)_4^{2+}$	Ppt dissolves, colourless solution, $Zn(OH)_4^{2-}$
Al^{3+}	White ppt, $Al(OH)_3$	Ppt dissolves, colourless solution, $Al(OH)_4^-$
Ag^+	Dark brown ppt, Ag_2O. Soluble in NH_3, colourless solution, $Ag(NH_3)_2^+$	Ppt doesn't dissolve
Cu^{2+}	Blue ppt, $Cu(H_2O)_4(OH)_2$, soluble in excess NH_3 to form a deep blue solution, $Cu(NH_3)_4^{2+}$	Ppt doesn't dissolve
Fe^{3+}	Brown ppt, $Fe(OH)_3$	Ppt doesn't dissolve

Fe^{2+}	White/green ppt, slowly turning brown when exposed to air (oxidation to $Fe(OH)_3$)	Ppt doesn't dissolve
Cr^{3+} (green or blue)	Blue-green ppt, $Cr(H_2O)_3(OH)_3$, soluble in NH_3, $Cr(NH_3)_6^{3+}$, violet solution	Ppt dissolves, $Cr(OH)_6^{3-}$ green solution.
Co^{2+}	Blueish ppt, $Co(H_2O)_4(OH)_2$. Slowly turns pink. The precipitate dissolves in NH_3, $Co(NH_3)_6^{2+}$, light brown colour. Turns to dark brown upon standing - slow oxidation to Co(III).	Ppt doesn't dissolve
Ni^{2+}	Green ppt, $Ni(H_2O)_4(OH)_2$. Soluble in NH_3 - blue solution, $Ni(NH_3)_6^{2+}$	Ppt doesn't dissolve
Mn^{2+} (pale pink)	Off-white ppt, $Mn(OH)_2$ turns brown upon exposure to air (oxidation to MnO_2)	Ppt doesn't dissolve

Transition metal colours

- MnO_4^- = violet
- CrO_4^{2-} = yellow (could be converted to orange $Cr_2O_7^{2-}$ in acid and vice versa).

Test for bromide

- Add chlorine water to a solution of bromides
- Produces an orange solution if present.
- Some effervescence may be observed.

Organic

Haloalkanes

See core practical 5, hydrolysis followed by halide test.

Alkenes

- Add 10 drops of your compound to a test tube followed by 1 cm^3 of bromine water and shake.
- If there are any C=C bonds, the colour change occurs from orange to colourless.

Alcohols

Alcohols can be primary, secondary or tertiary. Primary and secondary alcohols can be oxidised to aldehydes and ketones respectively but tertiary alcohols cannot be oxidised.

- Add 1 cm^3 of your unknown compound to a pear shaped flask followed by 5 cm^3 of acidified potassium dichromate solution.
- Heat the mixture under distillation conditions so the product is collected.
- If there is a colour change from **orange** to **green** then the potassium dichromate has been reduced meaning a primary or secondary alcohol is present, these will have

formed aldehydes and ketones respectively. Tollen's reagent or Fehling's solution can then be used to determine if the alcohol was primary or secondary.
- If there is no colour change, a tertiary alcohol was present.

Test for CO_3^{2-} and HCO_3^- ions

- Add aqueous acid.
- If CO_3^{2-} or HCO_3^- ions are present bubbles of CO_2 gas will be observed.
- Use a delivery tube to pass the CO_2 through limewater.
- The solution will turn cloudy due to formation of $CaCO_3$ precipitate.

Carboxylic acid

- Add 1 cm^3 each of the unknown substance and ethanol into a boiling tube and heat for 5 minutes in a beaker of boiling water.
- Add a few drops of sodium carbonate solution.
- If a carboxylic acid was present there will be a sweet ester smell.

Aldehydes and Ketones

Using Tollens' solution

- Add 10 drops of the unknown compound to a test tube followed by 5 cm^3 of ethanol, 5 cm^3 of sodium hydroxide solution and warm in a water bath for 5-10 minutes.
- Then add 1 cm^3 of dilute nitric acid solution and 10 drops of silver nitrate solution.
- Aldehydes give a positive test and a silver mirror is observed.
- Ketones cannot be oxidised, so no change observed

Using Fehling's reagent

- Add 1 cm^3 of blue Fehling's reagent to 10 drops of the unknown compound and warm the mixture in a 60 °C water bath
- Aldehydes give a positive test and form a brick red precipitate.
- Ketones don't react so the solution stays deep blue.

Iodoform reaction

- Warm with iodine and sodium hydroxide.
- Yellow precipitate.
- Antiseptic smell (triiodomethane, iodoform).
- Positive test with methyl ketones (e.g. propanone), ethanal, ethanol, and methyl secondary alcohols (e.g. propan-2-ol).

Esterification test

- Gently warm an alcohol and a carboxylic acid in the presence of concentrated H_2SO_4
- Should produce a compound with a characteristic smell, (e.g. pineapples).

Solubility test:

- Compounds with groups that can form H-bonds may be soluble in water, (e.g. amines, alcohols, carboxylic acids etc.)

- Some carboxylic acids may not be soluble in water, but may be soluble in alkaline solution (due to formation of $RCOO^-$).
- Same goes with amines. Some may not be soluble in water, but will be soluble in acidic solution (e.g. formation of RNH_3^+).