#### A2 – Assessed Practical (Skill P)

# **Introduction.**

#### Alcohols.

'Alcohols are a family of organic compounds in which the –OH functional group is attached to a hydrocarbon chain', pg. 296 *Chemistry for Advanced level, Third edition.*A substance can be tested for the presence of this functional group by selective oxidation. 'A solution of acidified potassium dichromate ions is often used as the oxidising agent. In the reaction the orange dichromate (VI) ions are reduced to green chromium (III) ions' pg 308, *Chemistry for Advanced Level, Third Edition*,

$$Cr_2O_7^{2-} + 14H^+ + 6e^- - 2Cr^{3+} + 7H_2O$$

The overall equation is:

#### Ketones.

Oxidation of secondary alcohols produces ketones, e.g. propanone and contains a C=O bond, where the carbon atom is also bonded to a CH<sub>3</sub> group. 'The basic method of identification of an unknown carbonyl compound is to make a solid derivative and then measuring the melting point of the derivative. The original carbonyl compound is then identified by reference to tables of melting temperatures of 2,4- dinitrophenylhydrazone derivatives.' Pg.536 *Chemistry for Advanced Level, Third Edition*.

## Aldehydes.

Primary alcohols produce aldehydes, e.g. ethanal, these contain a C=O group and here the carbon atom is bonded to a hydrogen. 'Aldehydes (but not ketones) are oxidised by Ag<sup>+</sup> ions in alkaline solution. The Ag<sup>+</sup> is reduced to metallic silver. When an aldehyde is warmed with Tollen's reagent (a solution of Ag<sup>+</sup> ions in aqueous ammonia), metallic silver is formed. In a clean test tube a silver mirror will be formed on the inside of the test tube. Ketones give no reaction to this test.'pg. 539 *New Understanding of Chemistry For Advanced Level*.

## Carboxylic acids.

The carboxylic acid consists of a C=O bond, where the carbon is also attached to an OH group. The evolution of carbon dioxide from sodium hydrogen carbonate is used as a test to distinguish carboxylic acids from weaker acids such as phenols.

'The reaction can be summarised using this equation:

$$2RCO_2H_{(aq)} + Na_2CO_{3(s)}$$
 —  $2RCO_2Na_{(aq)} + CO_{2(g)} + H_2O_{(l)}$ ' pg.697 A-Level Chemistry, E.N.Ramsden.

# Esters.

Esters also contain a C=O bond but here the carbon is attached to another oxygen, which is bonded with another carbon. 'Ester can be identified by using phenolphthalein indicator and refluxing with dilute acid or alkali. There should be a colour change from colourless to pink and after warming in water if ester is present the colour should change from pink back to colourless.' Pg.618 *A-Level Chemistry, E.N.Ramsden*.

#### Phenol.

Phenols are compounds containing a hydroxyl group attached to an aromatic ring, for example 2-methylphenol.

'Phenol produces a purple complex when mixed with iron (III) chloride in which the phenol is acting as a ligand. This is often used as a test for a phenol' pg.521 *Chemistry for Advanced Level, Third Edition.* 

#### Task.

The task is to identify an unknown substance through a series of tests that should systematically eliminate all other possible substances.

It is to be assumed that you are provided with an organic unknown containing one of the following groups:

- ➤ Alcohol
- ➤ Aldehyde
- > Ketone
- > Carboxylic acid
- > Ester
- > Phenol

Each test should be dependent on the result of the previous test.

# Apparatus.

- ➤ 100ml water
- > Phenolphthalein indicator
- ➤ Electric kettle
- > Sodium hydrogen carbonate
- ➤ Silver nitrate solution
- ➤ 6 test tubes
- ➤ 2M sodium hydroxide solution
- ➤ 2M ammonia solution
- ➤ 1 semi-micro test tube
- > 5 drops of 2,4 dinitrophenylhydrazine
- > 5 drops of potassium dichromate (VI)
- > 3 drops of dilute sulphuric acid
- > 5 drops of ferric chloride
- > Pipette
- > Small amount of unknown substance
- ➤ 2 large beakers
- > Test tube rack

# Procedure.

Carry out these following tests on the unknown substance and write down observations.

1) To five drops of ferric chloride add two drops of the substance followed by 2 drops of water. If there is no reaction move onto step two. If a reaction is observed note down any colour change, in this case if the solution turns a black-purple colour this indicates that there is a phenol present.

- 2) Mix a few drops of the substance with about 2-3cm<sup>3</sup> of water. Add a drop of phenolphthalein indicator, shake to mix, then add sodium hydroxide solution drop by drop, until the indicator just changes colour. Allow the solutions to stand in a beaker of hot water for a few minutes. If the solution remains a pink colour then no reaction has taken place so you should proceed to step three. If however, the solution turns colourless after standing this indicates that an ester is present.
- 3) Add a few drops of the substance to about 2-3cm³ of water and mix well. Add a little sodium hydrogen carbonate. Test for the identity of any gas released. If there is no gas released then continue with the following test. If the test indicates carbon dioxide is present then the unknown substance is a carboxylic acid.
- 4) To about 0.5cm³ of silver nitrate solution in a test tube add one drop of 2M sodium hydroxide solution followed by 2M ammonia solution drop by drop, with shaking, until the initial precipitate just dissolves. Pour this solution into a semi-micro test tube, add one drop of the substance and heat in water. If you observe a silver mirror on the inside of the test tube the substance is an aldehyde, if there is no silver mirror then no reaction has taken place so move onto the next test.
- 5) To five drops of a solution of 2,4 dinitrophenylhydrazine in a test tube, add one drop of the substance. Shake, and allow to stand for two or three minutes. If you observe a yellow/orange precipitate the unknown substance is a ketone, and if there is no change proceed with the following test.
- 6) To five drops of potassium dichromate (VI) in a test tube add 3 drops of dilute sulphuric acid followed by 2-3 drops of the substance and warm. This is the final test so there should be a colour change from orange to green and this indicates that the unknown substance contains the functional group –OH, which means it is an alcohol.

#### Prediction.

I predict that to find the functional group of the unknown organic substance the following flow chart indicating the sequence of the tests, will show which functional group is present. (Flow chart is in the appendix).

#### Variables.

Pressure and temperature must be kept constant throughout this experiment, as any change in either of these will have an effect on the results. The experiment will have to take place at room temperature and pressure (25°c and 100kPa). When conducting the silver mirror test care must be taken to avoid leaving the substance under light as this will have an effect on the results.

# Risk assessment.

- Phenolphthalein can cause skin contamination, always wear rubber gloves when handling this.
- Sodium hydroxide is corrosive and is an irritant. Handle with care by wearing rubber gloves and protective eyewear.
- Potassium dichromate (VI) is very toxic if inhaled, handle with care.
- Ferric chloride is irritating to eyes and skin so rubber gloves and eyewear are required.

# Appendix.

# *Test 1.*

This is the ferric chloride test and if reacted with phenol a violet complex is formed.

# *Test 2.*

Using phenolphthalein indicator and reflux with sodium hydroxide.

#### Test 3.

Sodium hydrogen carbonate with water, and testing for any gas released.

#### Test 4.

Silver mirror test.

# <u>Test 5.</u>

2,4 dinitrophenylhydrazine with one drop of water.

#### Test 6.

Potassium dichromate (VI) added to dilute sulphuric acid.