Chemistry Skill P

The purpose of this experiment is to identify an unknown organic compound, henceforth compound X. It is known that the functional group present in compound X is one of alkene, ester, aldehyde, ketone, alcohol, phenol or carboxylic acid. By performing a series of experiments, it should be possible to determine which is the case.

Attached is a flow chart, outlining the process necessary to identify the functional group in compound X. Red text denotes an observation, green text a test to be carried out, and black text the conclusion to be drawn from the results. Additionally, the event that no change is observed, and hence no reaction has taken place, is simply denoted by "N/R". Below, one can find detailed descriptions of how to perform the reactions

In the vast majority of cases, organic compounds are in the liquid state at room temperature, and this an assumption I have made of compound X during the ensuing instruction.

Safety Precautions

As we are working with an unknown substance, it is important to take safety in to account. There are some general safety points that are necessary for all tests, and they are as follows:

- Lab coats are to be worn at all times, to protect skin from exposure to chemicals.
- Eye protection is to be worn throughout, to protect eyes from chemicals.
- Bags, and other possible obstructions, should be kept away from the working area, to prevent tripping, etc.
- Glassware should be handled with care, so as to prevent breakages. In the event that glass is broken, it should be cleaned up immediately, and carefully, along with any spillages.
- More generally, spillages should be dealt with immediately, and in accordance with any precautions necessary for the chemicals being used.
- Waste should be disposed of in waste beakers, contained within fume cupboards.
- Care should be taken when heating apparatus, and Bunsen burners should not be left unattended.

Safety precautions specific to each reaction will be included within the instructions.

Add Sodium Metal

Chemicals: Sodium metal.
Equipment: Boiling tube, retort stand, boss, clamp, tweezers, scalpel, splint, source of ignition.

Method: Place the boiling tube, containing roughly 2cm^3 compound X, in the jaws of the clamp, and secure it in place. Using the tweezers, remove a small sample of sodium from it's storage under oil. If necessary, use the scalpel to reduce its size further. Your sample of sodium should be no more than 0.05g in mass. Again using the tweezers, add your sample of sodium to the boiling tube, and watch for effervescence.

Safety: Sodium is a highly reactive metal, and as such should be handled with care, and kept away from water, or any other oxidising agent. The reaction of sodium with the hydroxyl group is exothermic, and could heat glassware, hence care is needed when handling it after a reaction has taken place. Sodium metal is also flammable and corrosive, and as such, gloves are a necessary precaution.

Add Sodium Carbonate Solution

Chemicals: Sodium Carbonate, Ethanol, Distilled Water.
Equipment: Boiling tube.

Method: Put a few drops of compound X into 1cm^3 of ethanol, in the boiling tube. Follow this with approx. 5cm^3 of distilled water and 2cm^3 of sodium carbonate solution. Watch for effervescence.

Safety: Sodium Carbonate solution is an irritant. Contact with skin and eyes should be avoided, should it occur, the affected area should be washed thoroughly under water.

Warm with ethanol and a drop of conc. sulphuric acid

Chemicals: Ethanol, Concentrated Sulphuric Acid (1 mol dm^{-3}), Distilled Water.
Equipment: Boiling tube, 2x100cm^3 beaker, Bunsen burner, Tripod, Gauze.

Method: Fill both beakers with water. Take the first and heat it with the Bunsen burner until the water is approaching boiling point. Take the boiling tube and add to it 2cm^3 each of ethanol, compound X, and the concentrated sulphuric acid. Place the boiling tube in the beaker full of hot water, and wait for several minutes. Waft the air above the boiling tube towards your nose, and try to detect the sweet smell of an ester.[i] If it is not immediately obvious, take the contents of the boiling tube and empty it into the second beaker, containing cool water. The insolubility of the esters, when compared with the acid and alcohol, may result in the smell being more easily detectable.[ii]

Safety: Sulphuric acid at a concentration of 1 mol dm^{-3} is corrosive, and irritant. Great care should be taken to avoid exposing the eyes to contact with sulphuric acid, as it causes burns and has an affinity for water.[iii]

Add Aqueous Bromine

Chemicals: Aqueous bromine solution.
Equipment: Boiling Tube, Bung.

Method: Fill the boiling tube half full with aqueous bromine solution, and add one or two drops of compound X. Put the bung on, and gently mix the solution. Watch for decolourisation of the mixture, and a white precipitate, depending on the outcomes indicated on the flowchart.

Add neutral iron chloride solution

Chemicals: neutral iron (III) chloride solution.
Equipment: Boiling tube.

Method: Add 1cm^3 of compound X to the boiling tube, followed by a few drops of the iron chloride. Watch for the formation of a brightly coloured, often purple complex.[iv]

Warm with acidified potassium dichromate solution

Chemicals: Potassium dichromate, sulphuric acid, water.
Equipment: Boiling tube, 250cm^3 beaker, Bunsen burner, tripod, gauze.

Method: Fill the beaker with roughly $150cm^3$ of water, and heat using the Bunsen burner until it is approaching boiling. Take the boiling tube and add to it several drops of the compound X, followed by roughly $5cm^3$ of 1 mol dm^{-3} sulphuric acid. Add a few drops of potassium dichromate, stir with a glass rod, and leave for a few minutes. Look for a change in colour from orange to green.

Safety: Sodium dichromate is toxic, and can be a danger to the environment. It must be disposed o f properly, and not simply washed down the sink.

Add phosphorous pentachloride

Chemicals: Phosphorous pentachloride.
Equipment: Boiling tube, UI paper.

Method: Add a few drops of compound X to the boiling tube, ensuring there is no water present. Follow this by adding the tip of a spatula measure of phosphorous pentachloride to the boiling tube, and watch for the evolution of misty fumes[v]. If fumes are being evolved, place the UI paper inside the boiling tube, and look for a change in colour to red.

Safety: Hydrogen Chloride, the gas evolved during this reaction, is irritant to the lungs, and should not be inhaled. The gas is denser than air, and so shouldn't rise up out of the boiling tube, but if in doubt the boiling tube should be placed in a fume cup board and left to ventilate. It is advisable to keep the room well ventilated whilst performing the reaction.

Add to zinc chloride in aqueous hydrochloric acid

Chemicals: Aqueous zinc chloride, hydrochloric acid (1 mol dm^{-3}).
Equipment: Boiling tube.

Method: Add a $1cm^3$ of zinc chloride, $2cm^3$ of hydrochloric acid and a few drops of compound X to the boiling tube. Watch for the transparent solution quickly turning cloudy from the formation of a chloroalkane precipitate.[vi]

Safety: Hydrochloric acid is harmful and corrosive at 1 mol dm^{-3}, and as such should be treated with care. Zinc chloride

Add dilute acidified potassium manganate

Chemicals: Dilute potassium manganate (VII), sulphuric acid (.1 mol dm^{-3}).
Equipment: Boiling tube.

Method: Add a few drops of the potassium manganate to $1cm^3$ of sulphuric acid. Note the purple/pink colour, then add a few drops of compound X and watch for the decolourisation.

Safety: The mixture of sulphuric acid and potassium manganate can be explosive. Potassium is a strong oxidising agent, and if mixed with hydrochloric acid gives off chlorine gas. Potassium manganate should be disposed of carefully.

Add 2,4-DNP

Chemicals: 2,4-Dinitrophenylhydrazine.
Equipment: Boiling tube.

Method: Add $0.5cm^3$ of compound X to a boiling tube containing $5cm^3$ of Brady's reagent. Gently shake the tube to mix the two. Watch for the formation of a brightly coloured (usually orange/yellow) crystalline precipitate. Scraping the edge of the test tube with a glass rod can sometimes help to encourage crystallisation.

Safety: Brady's reagent is highly poisonous, toxic and explosive. Gloves should be worn when working with it, to prevent contact with skin, and caution should be exercised at all times.

Warm with Tollen's reagent

Chemicals: Silver nitrate, sodium hydroxide (1 mol dm^{-3}), 1 mol dm^{-3} ammonia solution, water
Equipment: $250cm^3$ beaker, boiling tube, Bunsen burner, tripod, gauze.

Method: Fill the $250cm^3$ beaker with roughly $150cm^3$ of water, and heat to near boiling using the Bunsen burner, tripod and gauze. Add roughly $5cm^3$ depth of silver nitrate solution to a *clean* boiling tube, and place this in the water bath. Upon adding a drop of sodium hydroxide, you should see a brown precipitate form. Now slowly add drops of the aqueous ammonia until all the brown precipitate has dissolved. Without delay, add several drops of compound X to your solution, and watch for the formation of a silver mirror around the inside of the boiling tube, or a grey precipitate. Either of the two is a positive result.

Safety: Silver nitrate is corrosive, and should be handled with care. Ammonia and sodium hydroxide are both bases, and contact with skin should be avoided. It's important to take when disposing of the substances.

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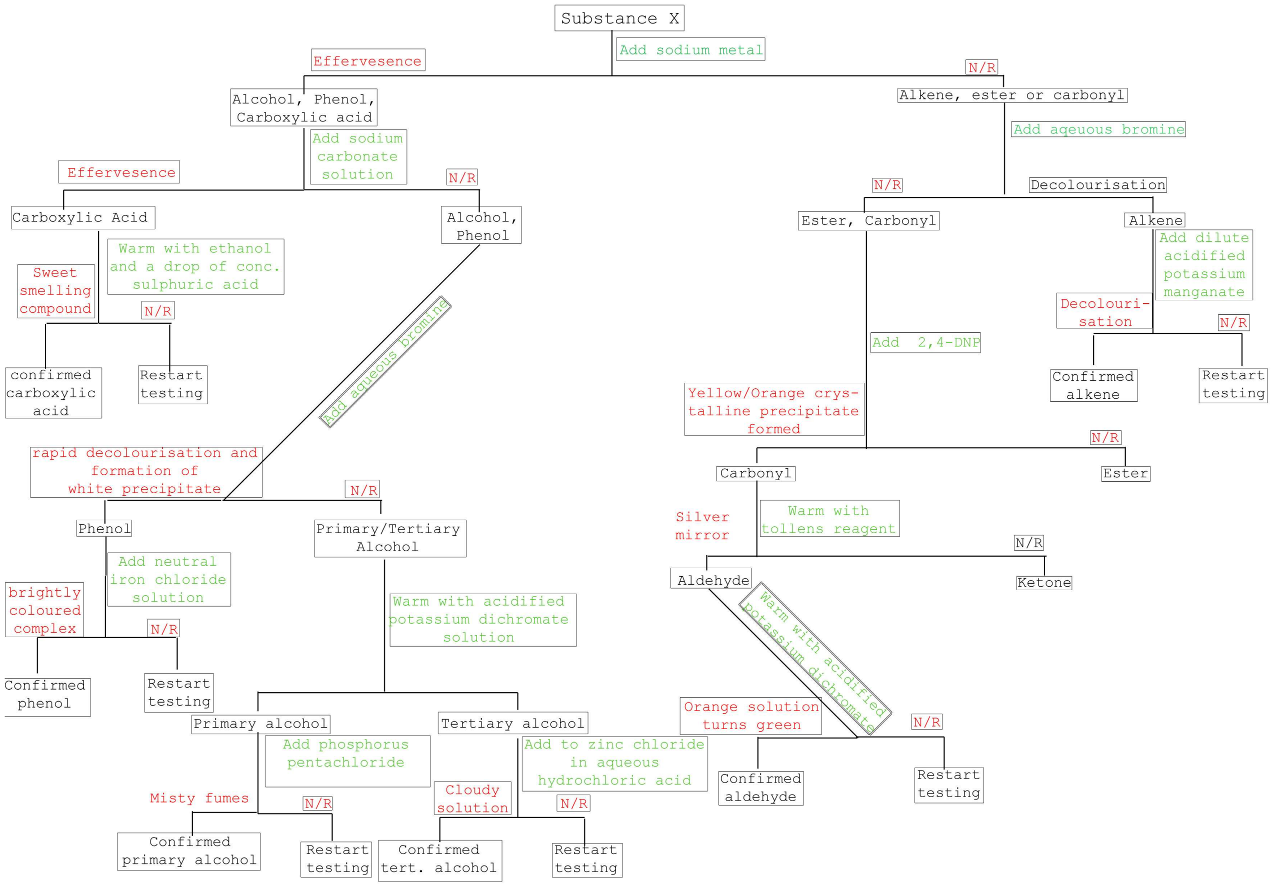
i Hills Road Study Pack CRS4 pg. 41
ii http://www.chemguide.co.uk/organicprops/esters/preparation.html
iii CLEAPSS Hazcard 098
iv Hills Road Study Pack CRS4 pg. 43
v Hills Road Study Pack CRS4 pg. 43
vi
http://jchemed.chem.wisc.edu/JCEsoft/CCA/CCA5/MAIN/1ORGANIC/ORG11/TRAM11/C/0362502/MOVIE.HTM



Substance X
Add sodium metal
Effervesence
N/R
Alcohol, Phenol, Carboxylic acid
Alkene, ester or carbonyl
Add sodium carbonate solution
Add aqueous bromine
Effervesence
N/R
Carboxylic Acid
Alcohol, Phenol
N/R
Decolourisation
Ester, Carbonyl
Alkene
Warm with ethanol and a drop of conc. sulphuric acid
Sweet smelling compound
N/R
confirmed carboxylic acid
Restart testing
Add aqueous bromine
Add dilute acidified potassium manganate
Decolouri-sation
N/R
Confirmed alkene
Restart testing
Add 2,4-DNP
Yellow/Orange crys-talline precipitate formed
N/R
Carbonyl
Ester
rapid decolourisation and formation of white precipitate
N/R
Phenol
Primary/Tertiary Alcohol
Silver mirror
Warm with tollens reagent
N/R
Aldehyde
Ketone
Add neutral iron chloride solution
brightly coloured complex
N/R
Confirmed phenol
Restart testing
Warm with acidified potassium dichromate solution
Warm with acidified potassium dichromate
Orange solution turns green
N/R
Primary alcohol
Tertiary alcohol
Add phosphorus pentachloride
Add to zinc chloride in aqueous hydrochloric acid
Confirmed aldehyde
Restart testing
Misty fumes
N/R
Cloudy solution
N/R
Confirmed primary alcohol
Restart testing
Confirmed tert. alcohol
Restart testing