Identification of an Organic Unknown (Skill P)

Equipment:
Test tubes
Pipette
Water Bath
Sodium Bicarbonate
Methanol
Bromine Water
2, 4 DNPH
Ethanol
Silver Nitrate solution
Sodium Hydroxide solution
Ammonia solution
Potassium Dichromate solution
Sulphuric acid

General safety:

- In the event of getting a substance in my eye I will wash it out with cold water immediately. Safety goggles will be worn for all of the experiments.
- If irritation is felt on my skin due to having contact with a substance I will wash the area of skin immediately with cold water.
- If a spillage of any of the substances used occurs, I will notify those around me straight away. If the substance is not considered harmful I will clear it up personally, if it is hazardous I will inform a technician.
- In the event of swallowing a harmful substance, medical help would be received be urgently requested.
- Highly flammable substances must be kept at least 1 metre away form an open flame.
- When tidying up at the end of my experiments it will be important to check those solutions that are safe to pour down the sinks and those that need to be handled with more care.

1) Test for an Alkene or Phenol

The reaction of an alkene with Bromine water is an electrophilic addition reaction; the alkene will open up its double bond and form bonds between its carbon atoms and the electrophilic Bromine atoms. This produces dibromoalkane.
The OH group on Phenol is electron releasing, because the oxygen p-orbital is delocalised, therefore the electron density of the delocalised ring is increased. It makes the substitution of Bromine with the Hydrogen atom much easier compared with, for example, Benzene. Substitution usually takes place at the 2, 4 and 6 positions.
Using a pipette add 6 drops of the unknown compound to a test tube comprised of 25cm3 Bromine water. Then shake the test tube so that the compound and the bromine water are thoroughly mixed together.
Result: If the Bromine water decolourises then an alkene or Phenol is present and the electrophilic addition reaction has taken place. If no reaction follows and the Bromine water remains a brown colour then the unknown compound still has not been identified.

Hazards:
Bromine water can be toxic and corrosive, so gloves must be worn and care taken. Extreme care must be taken when dealing with Phenol, direct contact with skin can cause severe burns, so gloves must be worn at all times. It is also very harmful if inhaled as it can burn the respiratory tract and may cause breathing problems, consequently the Phenol must be handled through in a fume cupboard.

2) Test for Phenol

Iron(III) ions form a strongly coloured complex with phenol.
Add 2 crystals of Iron(III) chloride to a test tube containing approximately 25cm3 of the solution containing either Phenol or an alkene, and stir with a glass rod.
Result: If an intense violet-purple solution is formed then the compound present is Phenol, consequently in this case, if no change occurs then it is an Alkene.

Hazards: When handling Iron(III) chloride gloves must be worn, as it is highly corrosive and can cause burns to the skin. Again I am aware of the dangers of Phenol.

3) Test for a Carbonyl group

Place 5 drops of Ethanol to a test tube containing 1 cm3 of the unknown compound then add methanol drop wise until the solution is clear. Once this is achieved add 2cm3 of 2,4 DNPH, using a pipette, to the solution and agitate for 1 minute, before leaving to stand for 5 minutes in a water bath .
Result: The formation of a bright orange precipitate indicates the presence of a carbonyl compound, which will be an Aldehyde or a Ketone. If no change occurs then an unknown functional group still remains.

Hazards: 2, 4 DNPH is explosive and toxic, so must be handled in a fume cupboard whilst wearing gloves. Also I am aware that ethanol is highly flammable, so it will be kept away from any open flames.

4) Test for an Aldehyde

Aldehydes are easily oxidised to acids, whereas Ketones cannot be oxidised, as there is no place for the oxygen from the oxidising agent to attach on to.
When the silver nitrate is mixed with ammonia to form Tollen's reagent, the complex ion [Ag (NH3) 2]+ is formed.

[Ag (NH3) 2]+ + e- Ag (s) + 3NH2

This is reduced to silver during the process of oxidation. Tollen's reagent is the oxidising agent.
Into a test tube, place 2cm3 of Silver nitrate solution and add 1 drop of dilute Sodium Hydroxide (NaOH). Add ammonia solution drop wise until the initially formed brown precipitate of silver oxide is re-dissolved. Then add 5 drops of Methanol to the solution and 5 drops of the unknown compound. Warm the test tube gently in water bath for 1 minute and leave to stand for 5 minutes.
Result: If a silver mirror is formed in the test tube, this indicates the presence of an Aldehyde, if no change occurs then a Ketone is present.

Hazards: Silver nitrate solution, sodium hydroxide solution and ammonia solution are all corrosive so I shall wear gloves when handling them. Methanol is highly toxic so if ingested or inhaled it can cause a wide range of harmful effects, breathing in the vapour can also be harmful to the respiratory tract so use of Methanol will be carried out in a fume cupboard.

5) Test for a Primary Alcohol

For the potential oxidation of the unknown compound, Potassium Dichromate(VI) acidified with dilute Sulphuric acid will be used. If oxidation occurs, the orange solution containing the Dichromate(VI) ions is reduced to a green solution containing Chromium(III) ions.

The electron-half-equation for this reaction is

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

Add 3 drops of the unknown substance, using a pipette, to a test tube containing 1cm3 of Potassium Dichromate(VI) solution acidified with 1cm3 of dilute Sulphuric acid. The test tube would then be warmed in a water bath for 3 minutes.

Result: A colour change to Green would highlight that an Alcohol was present, in this case a primary alcohol. No colour change would suggest that the unknown compound was an Ester or carboxylic acid.

Hazards: Potassium Dichromate(VI) is toxic, so care should be taken when handling this. It is also highly flammable, being aware of this I will keep it away from any danger of flames. Also Sulphuric acid is extremely corrosive, so gloves should be worn.

6) Test for a Tertiary alcohol

The Lucas test is used to distinguish between primary, secondary and tertiary alcohols.
Add 3 drops of Lucas reagent ($ZnCl_2/HCl$) to a test tube containing 25cm3 of the solution containing the unknown alcohol.
Result: A tertiary alcohol reacts rapidly and immediately, giving an insoluble white layer (alkyl chloride).
$(CH_3)_3C\text{-}OH + HCl + ZnCl_2 \rightarrow (CH_3)_3C\text{-}Cl + H_2O$

A primary alcohol does not react at all.
$CH_3CH_2\text{-}OH + HCl + ZnCl_2$

Hazards: The Lucas reagent can cause pain and redness to the skin, therefore gloves must be worn during the experiment. Likewise, goggle must be worn as getting the reagent in your eye can be dangerous.

7) Test for a Carboxylic acid

Using a Pipette add approximately 6 drops of the unknown compound solution and mix with 3 drops of methanol, to this then add 2 crystals of $NaHCO_3$ and shake the test tube vigorously.
Result: Solubility will be indicated by a colour change, or the evolution of Carbon dioxide. In this case a Carboxylic acid is present.

To test for the evolution of Carbon dioxide, and thus the presence of a Carboxylic acid, the limewater test can be used. Lime water is a solution of calcium hydroxide

(slaked lime). If Carbon dioxide is bubbled through it, a solid precipitate of Calcium Carbonate is formed.
Calcium Carbonate is chalk or limestone, it is this that makes the lime water cloudy.

calcium hydroxide + carbon dioxide calcium carbonate + water.
$Ca(OH)_2(aq) + CO_2(g) \rightarrow CaCO_3(s) + H_2O(l)$

Equipment: Bunsen burner
Bung
Rubber tube

Hazards: Sodium Hydrogen Carbonate may cause skin irritation if constantly exposed to it, so gloves should be used. It is also Moisture sensitive so should be kept in a closed container, before and during the experiment. As mentioned earlier I am aware as to the dangers of Methanol.

If all the tests prove a negative result then the unknown organic compound should be an Ester. All experiments should be carried out at least 3 times to be completely certain however.