Identification of an unknown organic

I have been supplied with an unknown organic compound containing one of the following functional groups:

- Alkene
- primary alcohol
- tertiary alcohol
- aldehyde
- ketone
- carboxylic acid
- ester
- phenol

By completing a series of chemical tests, the functional group can be established and therefore what the organic compound is. Complete the following tests in this order in accordance with the flowchart and safety procedures, to identify the functional group.

1. Test to identify a C=C bond

We will start with the bromine test. This particular test identifies phenols and the presence of unsaturated C=C bonds found in alkenes.

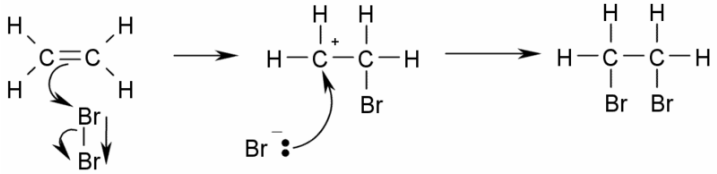
Equipment required:

- Bromine water
- Unknown organic
- Pipette
- Test tube

Procedure: Add the bromine water drop by drop to 1cm^3 of the unknown compound a test tube and shake.

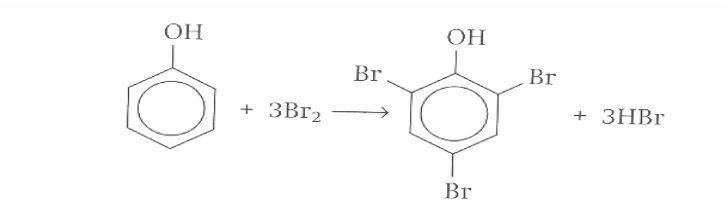
Outcome for an alkene: The bromine water has a distinct orange/brown colour; an alkene will decolourise the bromine producing a clear liquid.

Explanation: The C=C double bond in alkenes consists of a sigma bond and a π bond, which increases its reactivity. The bromine atom becomes polarised when close to a region of negative charge such as the π bond in the alkene, in this reaction it acts as an electrophile. The π bond breaks and the electron pair form a new covalent bond to the bromine atom. The bromide ion and carbocation produced then react to form a dibromoalkane. This is an electrophilic addition reaction. The mechanism is shown below:



Outcome for a phenol: A phenol will also decolourise bromine water, but will also produce a white precipitate of 2, 4, 6 – tribromophenol.

Explanation: The oxygen atom in the hydroxyl group has a lone pair of electrons, this lone pair can move onto the delocalized benzene ring, therefore increasing electron density (especially at the 2, 4 and 6 positions). This results in the ring being more susceptible to electrophilic attack which is why it reacts so readily with bromine. This is an electrophilic substitution as the hydrogen atoms are substituted for bromine atoms. The the reaction is shown below:



If the test gives a negative result and a phenol or alkene is not identified, continue onto test number 2.

2. Test to identify a carbonyl group (aldehydes and ketones)

Both aldehydes and ketones contain a c=o carbonyl group. Hydrazines such as 2, 4-dinitrophenylhydrazine (DNPH) identify the carbonyl group in aldehydes and ketones.

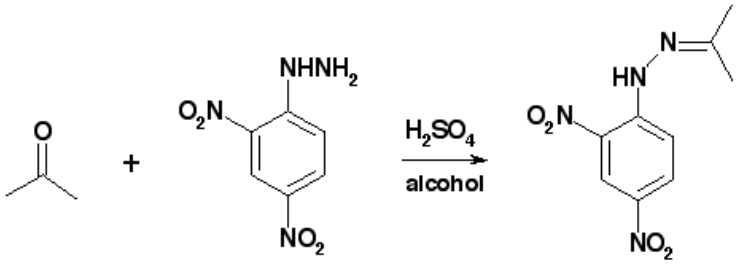
Required equipment:

- 2,4-dinitrophenylhydrazine
- Unknown compound
- Ethanol
- Pipette
- Test tube

Procedure: Add a few drops of the unknown compound to $2cm^3$ of ethanol. Add this to an excess of 2, 4 – DNPH (about $5cm^3$). If crystals do not form add a little $2mol\ dm^{-3}$ sulphuric acid, warm the mixture then cool in ice.

Outcome: If a carbonyl group is present then a distinctive crystalline precipitate called a hydrazone will be formed, usually bright red, orange or yellow.

Explanation: The reaction involves nucleophilic addition across the double bond of the C=O in the carbonyl group, the - NH_2 group adds to the C=O carbonyl group forming a N=C double bond with the elimination of a H_2O molecule. The reaction is shown below:



If the test gives a positive result then the compound must be an aldehyde or a ketone, continue onto test number 3.

If the test gives a negative result then continue onto test number 4.

3. Test to distinguish between an aldehyde and ketone

After confirming the compound contains a carbonyl group, a positive result to the following test will confirm an aldehyde and a negative will confirm a ketone.

Required equipment:

- Silver nitrate solution
- Dilute ammonia solution

} (Tollen's reagent)

- Unknown compound
- Water bath
- Test tube
- Pipette

Procedure: Add dilute ammonia solution to $3cm^3$ of silver nitrate solution until the silver oxide precipitate dissolves. The solution now contains $[Ag(NH_3)_2]^+$ ions. Add 10 drops of the unknown compound and warm the mixture in a water bath.

Outcome for an aldehyde: An aldehyde will produce a silver mirror on the inside of the test tube.

Explanation: Aldehydes are easily oxidised to acids. When the silver nitrate is mixed with ammonia to form Tollen's reagent, the complex ion [Ag (NH3) 2]+ is formed. This is reduced to silver during the process and the aldehyde is oxidised to a salt of the corresponding carboxylic acid. The reaction is shown below:

Outcome for a ketone: A ketone will not react, and no mirror will be produced.

Explanation: A ketone has no available hydrogen atom on the carbonyl carbon that can be oxidized - unlike an aldehyde.

4. Test to identify primary/secondary alcohols

The following test will identify a primary or secondary alcohol; in this case a positive result will show a primary alcohol.

Procedure: Add potassium dichromate to sulphuric acid, and add a few drops of the unknown compound to the test tube heat.

Required equipment:

- Potassium dichromate
- Sulphuric acid
- Unknown compound
- Pipette
- Bunsen burner

Outcome for a primary alcohol: A primary alcohol will give a colour change from orange to green.

Explanation: Primary alcohols are oxidised to form carboxylic acids, the hydrogen in the –OH group is lost and the single bond between the carbon and oxygen is replacedwith a double bond. The chromium (VI), which is orange, is reduced to chromium (III), which is green. The reaction is a redox reaction and is shown below:

5. Test to identify a carboxylic acid

Carboxylic acids are identified as such in the lab by their acidic properties. The following test will confirm that the compound is acidic and therefore a carboxylic acid.

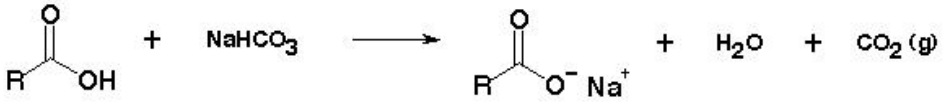
Required equipment:

- Sodium bicarbonate solution
- Unknown organic
- Test tube
- Pipette

Procedure: Slowly add 1cm^3 of sodium bicarbonate solution to the same amount of the unknown compound.

Outcome: Bubbles of carbon dioxide will be produced.

Explanation: Carboxylic acids react with a bases to form a carboxylate salt (where the hydrogen of the –OH group is replaced with a metal), carbon dioxide gas and water. The release of carbon dioxide bubbles in the solution confirms an acid. The neutralisation reaction is shown below:



If the test gives a negative result and a carboxylic acid is not identified, continue onto test number 6.

6. Test to identify a secondary/tertiary alcohol

The following test will identify a secondary and tertiary alcohol; in this case a positive result will indicate a tertiary alcohol.

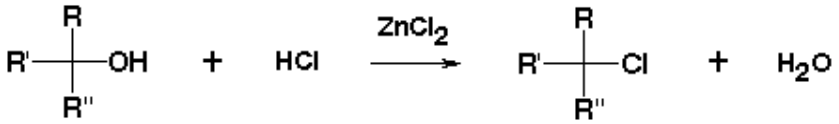
Required equipment:

- Lucas' reagent (solution of ZnCl in conc. HCl)
- Unknown compound
- Test tube
- Pipette

Procedure: Add 1cm^3 of the unknown to 10cm^3 of Lucas' reagent at room temperature. Put a bung in the tube, shake vigorously and allow the mixture to stand.

Outcome: The mixture will become cloudy or form an insoluble layer almost instantly due to the production of a chloroalkane.

Explanation: The reaction is a substitution in which the chlorine replaces the hydroxyl group of the alcohol.



If the test gives a positive result, the compound is a tertiary alcohol. If the test gives a negative result then the compound must be an ester

Safety – Risk analysis

Gloves, goggles and lab coats should be worn at all times throughout the experiment and long hair tied up. Take care if using Bunsen burner and any glassware. Be cautious with all chemicals used:

- Do not inhale any vapours
- Keep them
- If they come into contact with skin wash the area thoroughly with water
- Make sure not to rub face or eyes
- Clean up any spillages immediately

Follow the same safety precautions below for the unknown compound.

Risk	**Cause**	**Safety precautions**
Irritation to eyes and skin	Bromine water 2, 4 DNPH Sulphuric acid Potassium dichromate Ammonia solution (Tollen's reagent) Lucas' reagent	Avoid contact with skin and eyes. Wear eye protection and gloves; wash with cold water in the event of contact.
Fire/explosion hazard	Dry 2, 4 DNPH is extremely flammable and can be explosive. Ethanol is very flammable Tollen's reagent may explode on storage.	Make sure the solid doesn't dry out. Avoid working near naked flames. Neutralise it with dilute nitric acid and dispose of it immediately/
Harmful if inhaled	Bromine water gives off bromine vapour	Avoid inhaling any vapours and work in a well ventilated area

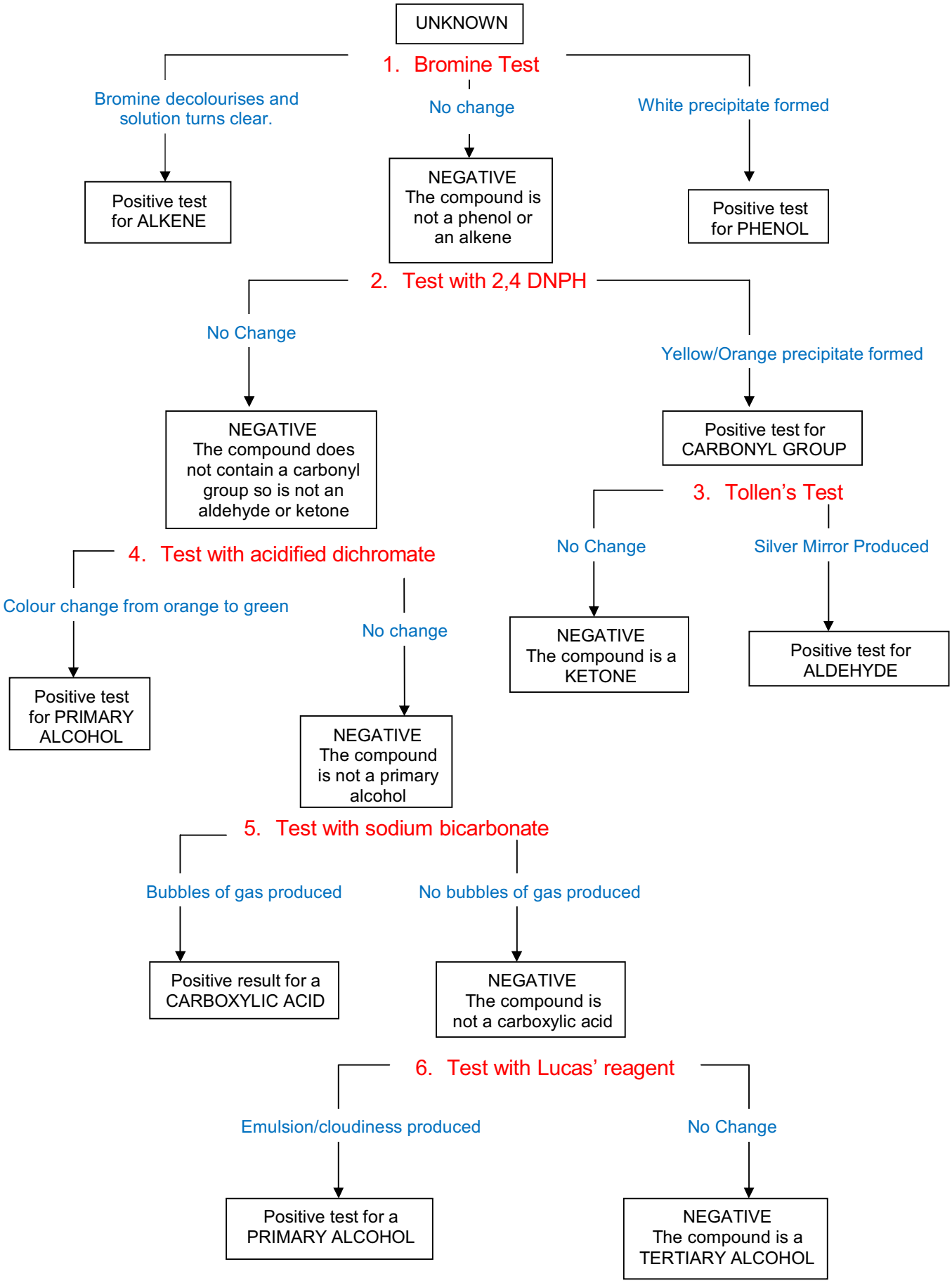
References

http://www.wellesley.edu/Chemistry/chem211lab/Orgo_Lab_Manual/Appendix/ClassificationTests

http://www.umsl.edu/~orglab/experiments/UNKEXP.html

http://www.chemguide.co.uk/organicprops/acids/acidity.html

http://en.wikipedia.org/wiki/Bromine_test


UNKNOWN
1. Bromine Test
Bromine decolourises and solution turns clear.
No change
White precipitate formed
Positive test for ALKENE
NEGATIVE The compound is not a phenol or an alkene
Positive test for PHENOL
2. Test with 2,4 DNPH
No Change
Yellow/Orange precipitate formed
NEGATIVE The compound does not contain a carbonyl group so is not an aldehyde or ketone
Positive test for CARBONYL GROUP
3. Tollen's Test
No Change
Silver Mirror Produced
NEGATIVE The compound is a KETONE
Positive test for ALDEHYDE
4. Test with acidified dichromate
Colour change from orange to green
No change
Positive test for PRIMARY ALCOHOL
NEGATIVE The compound is not a primary alcohol
5. Test with sodium bicarbonate
Bubbles of gas produced
No bubbles of gas produced
Positive result for a CARBOXYLIC ACID
NEGATIVE The compound is not a carboxylic acid
6. Test with Lucas' reagent
Emulsion/cloudiness produced
No Change
Positive test for a PRIMARY ALCOHOL
NEGATIVE The compound is a TERTIARY ALCOHOL