Assessed practical   Debbie Siobhan Warner   November 2002

Determining the identity of an organic unknown

When supplied with a organic unknown there are many ways of determining its identity and it is important to use as much information as can be gained to work out the identity of an unknown as many are very similar in physical properties, chemical properties and/or chemical make up.
In this experiment I will be using a flow chart to identify the functional group that the unknowns contain, and once I have suggested this I will then use spectra given to me to determine what the identity of the organic unknown is.

I have been told that the organic unknown, will one of the following functional groups:

Alcohol's

Probably the most common of these functional groups is the "-OH" group, which is known as the **hydroxyl group**. It is **NOT** the hydroxide ion, OH^{1-}, as it does not have a charge. The dash in front of the OH stands for a single covalent bond, which is what will be formed between the oxygen and a carbon atom. An aliphatic hydrocarbon that has one hydroxyl group attached to a carbon is called an alcohol. The simplest alcohol is methyl alcohol, or methanol.

The molecular formula is usually written as CH_3OH, because it gives more of a picture of the actual structure than does CH_4O. This way of writing the formula becomes more important as the number of carbons increases. Take the case of ethyl alcohol, or ethanol. The parent compound is ethane, so the formula is C_2H_6O, but C_2H_5OH gives specific information that the compound is an alcohol and not any other compound.

Phenol

Colourless solid, partially miscible in the cold water, soluble in organic solvents. Differ in many ways to aliphatic alcohols. Like alcohols contain -O-H group but also a benzene ring.
The difficulty in substituting the –OH group is due to the stabilisation caused by the overlap of the p-orbital of the oxygen atom with the bonding in the ring. The situation is similar to that in chlorobenzene. Phenols have many chemical properties
i) Acid-base properties
Phenol is a stronger acid than water whereas aliphatic alcohols are weaker. The polarity of the hydroxyl bond facilitates the loss of a proton and the formation of a phenoxide ion and the delocalisation in the phenoxide ion also stabilises it as compared with RO^- or OH^- ions. If electron-withdrawing groups (-Cl) are substituted into the benzene ring the polarity of the O-H bond is increased still further giving still stronger acids.

Aldehydes

A new class of substituted hydrocarbons arises when an oxygen atom is double bonded to the carbon at the end of the chain. In this case there are two less hydrogen atoms, so instead of three end hydrogens, there is the C=O and only one hydrogen.

The simplest aldehyde is, formaldehyde, CH_2O. Its IUPAC name is methanal. It has an -al ending as opposed to the -ol ending that alcohols have. These compounds show the generic formula, H-R=O.

Ketones

A different class of organic compounds results if the C=O occurs somewhere along the chain other than on the end carbon. The simplest ketone has three carbons. It has the common name acetone, and is in most fingernail polishes and removers. It is sometimes called dimethyl ketone, but is more properly called propanone. Break apart the name to see how the name propanone gives a better picture of the compounds formula than does acetone.

- First of all, the propan- indicates that the parent hydrocarbon is propane, and thus has three carbons.
- Second, the ending -one goes along with the ending of the name of the class of compounds to which it belongs, ketones.

The generic formula for ketones is R-C=O(-R').

Carboxylic acids

Carboxylic acids have a more complex functional group, and if you look at it, you can see both the C=O of the aldehyde and the -OH of the alcohol. The organic, or carboxyl group, is -C=O(-OH), often written as COOH, or even CO_2H. Organic acids may have more than one carboxyl group. The simplest organic acid is methanoic acid, CHOOH, or formic acid, to use the older name. Ants inject formic acid into their victim whem they bite them.
The next in line is, of course, ethanoic acid, CH_3COOH. Notice the ending, -oic, to the IUPAC name, and -ic, to the common name. There are some very important organic acids, and one of the most important is ascorbic acid, better known as Vitamin C. The generic formula is R-(COOH)x
Why are these compounds acids? Well, they must be able to produce at least one hydrogen ion when they are put into solution, since that is the general definition for an acid. Even though these organic acids may contain quite a few hydrogen atoms in the molecule, only select hydrogens are able to be "ionized" or turned into hydrogen ions. These "select" hydrogens are those in the carboxyl group (-COOH) The presence of one or more of these groups, therefore, causes the compound to belong to the organic acids.

Ester

An ester results when there is an oxygen atom between two carbons in the chain. The simplest is dimethyl ester, which has the same molecular formula as ethanol. The way the formula is written to show the ester, rather than the alcohol, is CH_3OCH_3. There are two other esters of interest, ethylmethyl ester and diethyl ester. You should be able to write the formulas for each of these. Esters are represented as R-O-R', where R and R' can be the same or different hydrocarbon units.

Method

Apparatus
· 10 cm^3 measuring cylinder
· Teat pipette
· Test tubes
· Electric hotplate

Reagents
· Bromine water
· 2,4-dinitrophenylhydrazine
· Tollens' reagent
· Aqueous sodium carbonate
· Lime water
· Acidified potassium dichromate
Only small volumes of the unknown compound need to be used in each test as they are in a pure form. Therefore I will only use $3cm^3$ of each reagent and the unknown compound.

Test for presence of alcohol: An oxidation reaction must take place. Mix equal volumes ($3cm^3$) of dilute sulphuric acid and potassium dichromate solution. Then add the same volume of the unknown to it. If the solution turns from orange to green then a primary or secondary alcohol group is present.
Test for presence of a carbonyl group: A condensation reaction takes place. 2,4-dinitrophenylhydrazine solution is added to the sample, if the solution turns into an orange precipitate then a carbonyl compound is present.
Test to distinguish between a ketone and an aldehyde: An oxidation reaction takes place. Add a small volume of sodium hydroxide solution to $3cm^3$ of silver nitrate solution until a slight precipitate forms, than add ammonia solution dropwise until the precipitate dissolves. Then add a few drops of the unknown sample and warm in a water bath. If a silver mirror forms, then the compound has an aldehyde functional group.
Test for presence of carboxylic acid: An acid / base reaction occurs. Aqueous sodium carbonate solution is added to the sample. If effervescence occurs and the gas evolved is carbon dioxide (test gas by bubbling into lime water) then a carboxylic acid group is present.
Test for presence of phenol: A substitution reaction occurs. Bromine water is added to the sample and if a phenol group is present then the bromine water decolourises and a white precipitate is formed.
Test for ester group: Esters have a distinct fruity smell. There is no simple test to carry out for identifying an ester, so I will determine the sample is an ester by negative results from the previous tests.

Saftey
Safety is imperative when carrying out experiments in the lab, this is even more true when the identity of a compound is unknown as it could have any dangerous properties, gloves should be used when handling substances and goggles worn, long hair tied back and cuts covered.

Flow chart

Results of organic tests

All organic tests proved negative, apart from the test for an alderhyde although this test did not produce a silver mirror some dark precipitate was formed indicating the likeliness of the presence of an aldehyde.

Analysis of spectropic data

Conclusion

I conclude that the organic unknown is an aldehyde this is because of the results found from the chemical tests performed , the specific aldehyde I believe the unkown to be is benzaldehyde. This is because not only does its physical properties fit those described in text books it also has the right structural formula to fit with the spectropic data provided.

benzaldehyde

benzaldehyde or benzenecarbonal , C_6H_5CHO, colourless liquid aldehyde with a characteristic almond odour. It boils at 180°C, is soluble in ethanol, but is insoluble in water. It is formed by partial oxidation of benzyl alcohol, and on oxidation forms benzoic acid. It is called oil of bitter almond, since it is formed when amygdalin, a glucoside present in the kernels of bitter almonds and in apricot pits, is hydrolysed, e.g., by crushing the kernels or pits and boiling them in water; glucose and hydrogen cyanide (a poisonous gas) are also formed. It is also prepared by oxidation of toluene or benzyl chloride or by treating benzal chloride with an alkali, e.g., sodium hydroxide. Benzaldehyde is used in the preparation of certain aniline dyes and of other products, including perfumes and flavourings.

Evaluation

Overall I feel this experiment went very well as I was able to determine both the functional group present and then with the aid of specropic data the exact compound. I carried out the experiment safely following all guideline set in my method. The only test that could have been performed more accurately was the test for an alderhyde that did not produce a silver mirror.