# Identification of Unknown Organic Compounds

## Introduction

The identification and characterization of the structures of unknown substances are an important part of organic chemistry. Although it is often possible to establish the structure of a compound on the basis of spectra alone (IR, NMR, etc.), the spectra typically must be supplemented with other information about the compound: physical state and properties (melting point, boiling point, solubility, odor, color, etc.), elemental analysis, and confirmatory tests for functional groups.

In this experiment you will carry out several qualitative tests that will allow you to identify functional groups in organic molecules. You will then apply what you have learned by characterizing unknown organic compounds in terms of their functional group and solubility behavior. The functional groups you will examine include amines, alcohols, carboxylic acids, alkenes, alkanes, and alkyl halides.

## Solubility and Functional Group Tests

Each functional group has a particular set of chemical properties that allow it to be identified. Some of these properties can be demonstrated by observing solubility behavior, while others can be seen in chemical reactions that are accompanied by color changes, precipitate formation, or other visible effects.

### 1. Solubility Tests

The solubility of an organic compound in water, dilute acid, or dilute base can provide useful information about the presence or absence of certain functional groups. A flowchart showing the sequence of solubility tests along with the appropriate conclusions is shown in **Figure 1**.

*Solubility in water*: Most organic compounds are not soluble in water, except for low molecular-weight amines and oxygen-containing compounds. Low molecular-weight compounds are generally limited to those with fewer than five carbon atoms.

- Carboxylic acids with fewer that five carbon atoms are soluble in water and form solutions that give an acidic response (pH < 7) when tested with litmus paper.

- Amines with fewer than five carbons are also soluble in water, and their solutions give a basic response (pH > 7) when tested with litmus paper.

- Ketones, aldehydes, and alcohols with fewer that five carbon atoms are soluble in water and form neutral solutions (pH = 7).

*Solubility in NaOH*: Solubility in 6M NaOH is a positive identification test for acids. A carboxylic acid that is insoluble in pure water will be soluble in base due to the formation of the sodium salt of the acid as the acid is neutralized by the base.

*Solubility in HCl*: Solubility in 6M HCl is a positive identification test for bases. Amines that are insoluble in pure water will be soluble in acid due to the formation of an ammonium chloride salt.

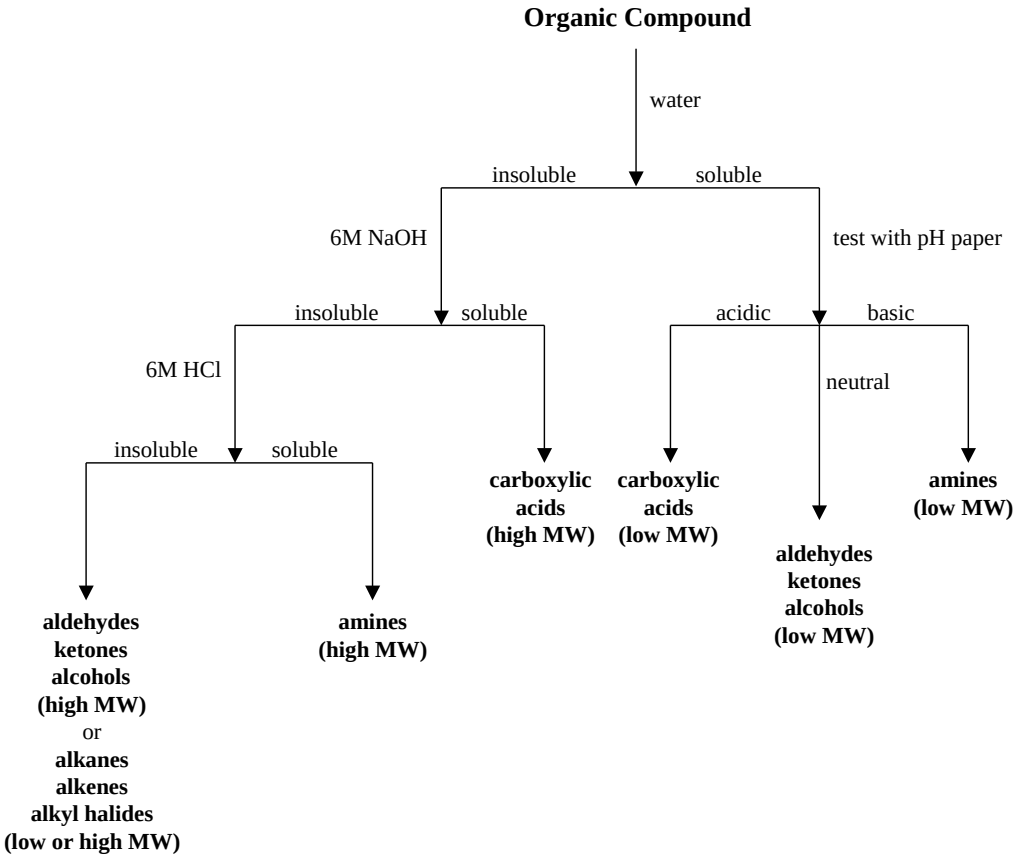



**Figure 1.** The Solubility Test Flowchart

## 2. Silver Nitrate Test for Alkyl Halides and Carboxylic Acids

The reaction of an alkyl halide with silver nitrate in ethanol will result in the formation of a white or yellow silver halide precipitate that is insoluble in nitric acid. This reaction quite often proceeds slowly, and occasionally slight warming is necessary.

$$RX + AgNO_3 \longrightarrow RONO_2 + AgX(s)$$

It is important to check if the precipitate is soluble in dilute nitric acid. Carboxylic acids form insoluble silver salts that precipitate, but these dissolve in nitric acid whereas the silver halides do not.

### 3. Beilstein Test for Halides

Halogens can be detected easily and reliably by the Beilstein test. It is the simplest method for determining the presence of a halogen, but does not differentiate among chlorine, bromine, and iodine. A positive Beilstein test results from the production of a volatile copper halide when an organic halides is heated with copper oxide. The copper halide imparts a blue-green color to the flame.

### 4. Lucas Test for Alcohols

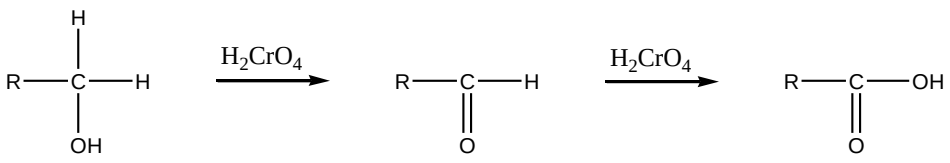
This test depends on the appearance of an alkyl chloride as an insoluble second layer when an alcohol is treated with a mixture of hydrochloric acid and zinc chloride (Lucas reagent):

$$ROH + HCl \xrightarrow{ZnCl_2} RCl + H_2O$$

Primary alcohols do not react at room temperature; therefore, the alcohol is seen simply to dissolve. Secondary alcohols will react slowly, whereas tertiary, benzylic, and allylic alcohols react instantly.

### 5. Chromic Acid Test for Alcohols

This test is based on the reduction of chromium(IV), which is orange, to chromium(III), which is green, when an alcohol is oxidized by the reagent. A change in color of the reagent from organce to green represents a positive test. Primary alcohols are oxidized by the reagent to carboxylic aicds; secondary alcohols are oxidized to ketones. Tertiary alcohols are not oxidized at all by the reagent. Hence, this reaction can be used to distinguish tertiary alcohols form primary and secondary alcohols. Unlike the Lucas test, this test can be used with all alcohols, regardless of molecular weight and solubility.

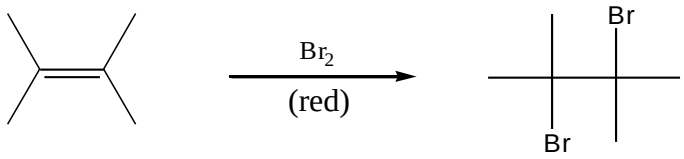


primary alcohol

secondary alcohol

## 6. Bromination Test for Alkenes

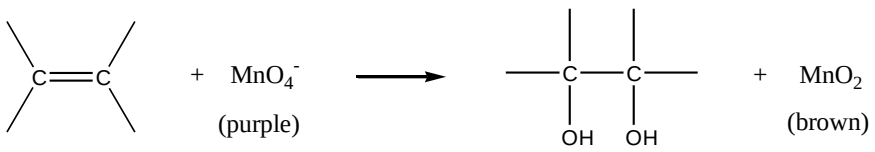
Bromine readily adds across the carbon-carbon double bond of an alkene to produce a dibromoalkane. A red solution of bromine in methylene chloride will quickly become colorless as the addition reaction with the alkene takes place. The general equation describing the test is:




(colorless) (colorless)

## 7. Potassium Permanganate Test for Alkenes

This test is positive for double and triple bonds but not for aromatic rings. It depends on the conversion of the purple ion $MnO_4^-$ to a brown precipitate of $MnO_2$ following the oxidation of an unsaturated compound.



Other easily oxidized compounds also give a positive test with potassium permanganate solution. These include aldehydes, some alcohols, phenols, and aromatic amines.

Name(s) _____ Date _____

# Report Sheet: Identification of Unknown Organic Compounds

## A. Characterizing Known Compounds

Carefully record your observations. Accurately describe each positive test and include factors such as reaction time, color change, precipitate formation, and the need for heating, stirring, or shaking.

### 1. Solubility Tests

Indicate which compounds are soluble in water, and for those that are soluble, indicate whether the solution is acidic, neutral, or basic.

### 2. Silver Nitrate Test

| |
|---|
| Observations for 1-bromobutane: |
| Observations for other compounds: |
| What happened when $HNO_3$ was added to each precipitate? |

### 3. Beilstein Test

| |
|---|
| Observations for unknown: |
| Observations for benzoic acid |

### 4. Lucas Test

| |
|---|
| Observations for 1-butanol: |
| Observations for 2-butanol: |
| Obsercatuibs for t-butyl alcohol: |

### 5. Chromic Acid Test

| |
|---|
| Observations for 1-butanol: |
| Observations for 2-butanol: |
| Obsercatuibs for t-butyl alcohol: |

### 6. Bromination Test

| |
|---|
| Observations with cyclohexene: |
| Observations with cyclohexane: |

### 7. Permanganate Test

| |
|---|
| Observations with cyclohexene: |
| Observations with toluene: |

## B. Characterizing Unknown Compounds

Unknown numbers: (a) _____ (b) _____

| Test | Observations | Conclusions |
|---|---|---|
| Solubility in water and pH of the solution | (a) | (a) |
| | (b) | (b) |
| Solubility in 6M NaOH (if needed) | (a) | (a) |
| | (b) | (b) |

| | | |
|---|---|---|
| Solubility in 6M HCl (if needed) | (a) | (a) |
| | (b) | (b) |
| Silver nitrate test | (a) | (a) |
| | (b) | (b) |
| Beilstein test | (a) | (a) |
| | (b) | (b) |
| Lucas test | (a) | (a) |
| | (b) | (b) |
| Chromic Acid test | (a) | (a) |
| | (b) | (b) |
| Bromination Test | (a) | (a) |
| | (b) | (b) |

| | | |
|---|---|---|
| Permanganate Test | (a) | (a) |
| | (b) | (b) |

Indicate the functional group present in the unknown:

(a) Unknown number _____: _____

(b) Unknown number _____: _____

From its solubility in water, is the unknown of low molecular weight?
(Answer *yes*, *no*, or *test was indecisive*.)

(a) Unknown number _____: _____

(b) Unknown number _____: _____

3. What class of organic compound with less than five carbon atoms dissolves in water to form a slightly acidic solution? Draw an equation to represent this reaction.

4. What class of organic compound with less than five carbon atoms dissolves in water to form a slightly basic solution? Draw an equation to represent this reaction.

5. What class of organic compound with more than five carbon atoms is insoluble in water but readily dissolves in base? Draw an equation to represent this reaction.

6. Draw the reaction between cyclohexene and bromine in $CCl_4$. what type of reaction is this?

7. What type of reaction is the reaction between an alkyl halide and alcoholic silver nitrate? How would you expect the structure of the alkyl halide to affect the rate of this reaction?

8. You have now proposed a functional group present in your unknown. Name two methods that could be used to confirm your conclusion.

http://a-s.clayton.edu/cclower/CHEM2412L/Organic%20Functional%20Groups%20Lab.doc