EXERCISE 1

# Body in a Lab: Compound Identification

1


## INTRODUCTION

### Background

**A body has been found in the lab!**

The victim, Mr Blue, was known to have a heart condition but on the bench at the scene where the victim had been working a large bottle of concentrated acid had been upturned and spilt. All around this acid were different chemical bottles which also had been knocked over and may have mixed with the acid. A medicine bottle was also present with unknown tablets inside (Sample X - the tablets have been ground ready for analysis).

**Objective**

Try to establish cause of death by using infrared analysis to discover the functional groups present in the chemical samples collected. Decide if any of these are likely to be toxic or may have formed a lethal toxic gas on contact with the spilt acid. Establish the identity of the medicine found by library comparison of the spectra and suggest possible implications.

# METHOD

### You are provided unknown samples A – H and medicine sample X

1. Run a liquid film on all liquid samples.
2. Use the ATR attachment to run all solid samples. **(Note: Care must be taken with this expensive and fragile equipment, use only when supervised by a demonstrator).**

### Interpretation of Spectra

To interpret the spectra obtained from a sample it is necessary to refer to correlation charts and tables of infrared data. There are many different tables available for reference, one of which has been provided - Introduction page 4.

3. Using the correlation chart provided interpret your spectra and identify the functional groups present in the chemical. Record your results in the table provided.

### Identification of Unknown Compound

While IR spectroscopy is a very useful tool for identifying the functional groups in an unknown compound, it does not provide sufficient evidence to confirm the exact structure. Chemists make use of a variety of techniques in order to piece together the structure of a molecule.

4. Use your interpreted IR spectra and the mass spectra provided to determine the structure of all unknown compounds.
5. Identify any chemicals that you think may be toxic or where the functional group may possibly release a toxic gas on contact with an acid.
6. Suggest what other instrumental technique or techniques would be required to confirm the identity of the chemicals. (Your demonstrator will then be able to provide you with additional data for confirmation of analysis).
7. Identify sample X by using the library spectra.

# MATERIALS

### Infrared Spectroscopy and Identification of Functional Groups

**Chemicals**

Unknown Samples

| | | |
|---|---|---|
| A | **Benzoic Acid** | Acid |
| B | **Propan-2-ol** | Alcohol |
| C | **Propanone** | Ketone |
| D | **Ethylbenzoate** | Ester |
| E | **Acetonitrile** | Nitrile |
| F | **Propionamide** | Amide |
| G | **Benzaldehyde** | Aldehyde |
| H | **Ethanol** | Alcohol |
| X | **Acetylsalicylic Acid** | Acid |

**Apparatus**

- FTIR infrared spectrometer with ATR attachment and plate holder
- 4 sets sodium chloride plates
- Disposable pipettes and teats
- 2 x tissues
- Acetone wash bottle
- Propan-2-ol wash bottle
- 2 x micro-spatula
- Disposable gloves large
- Disposable gloves medium

**Administration**

- Poster
- Scripts
- 5 x laminated correlation charts
- Risk assessment and hazard data
- Sample spectra for IR and mass spec.
- Feedback forms
- Pens

### Please note

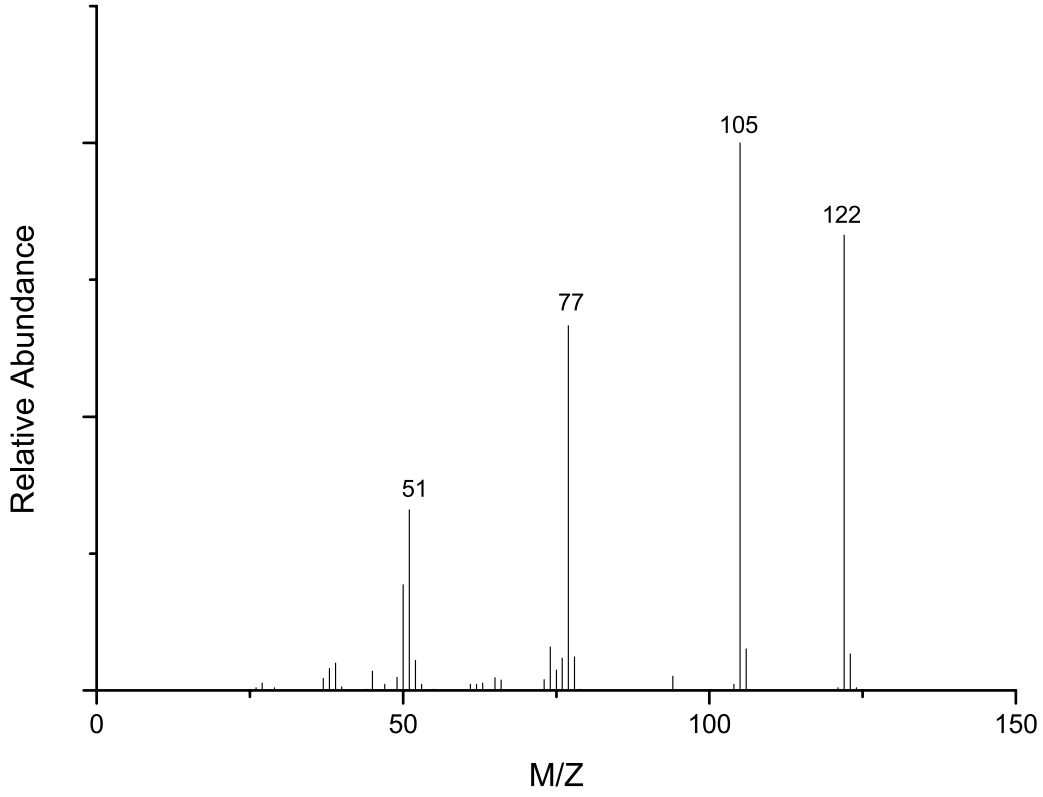
This scenario is continued in the UV section and concluded in the MS section.

# IDENTIFICATION OF UNKNOWN COMPOUNDS

Use interpreted IR spectra and mass spectra below to determine the structure of the unknown compounds

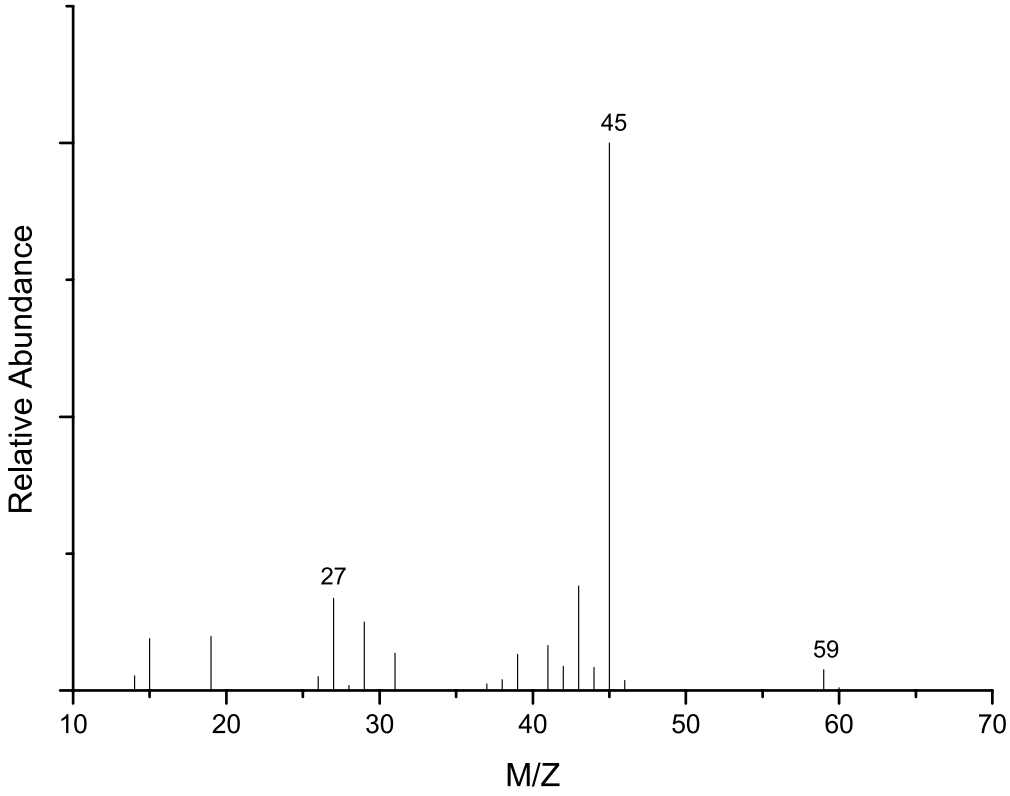
## Sample A

## Empirical Formula C7H6O2



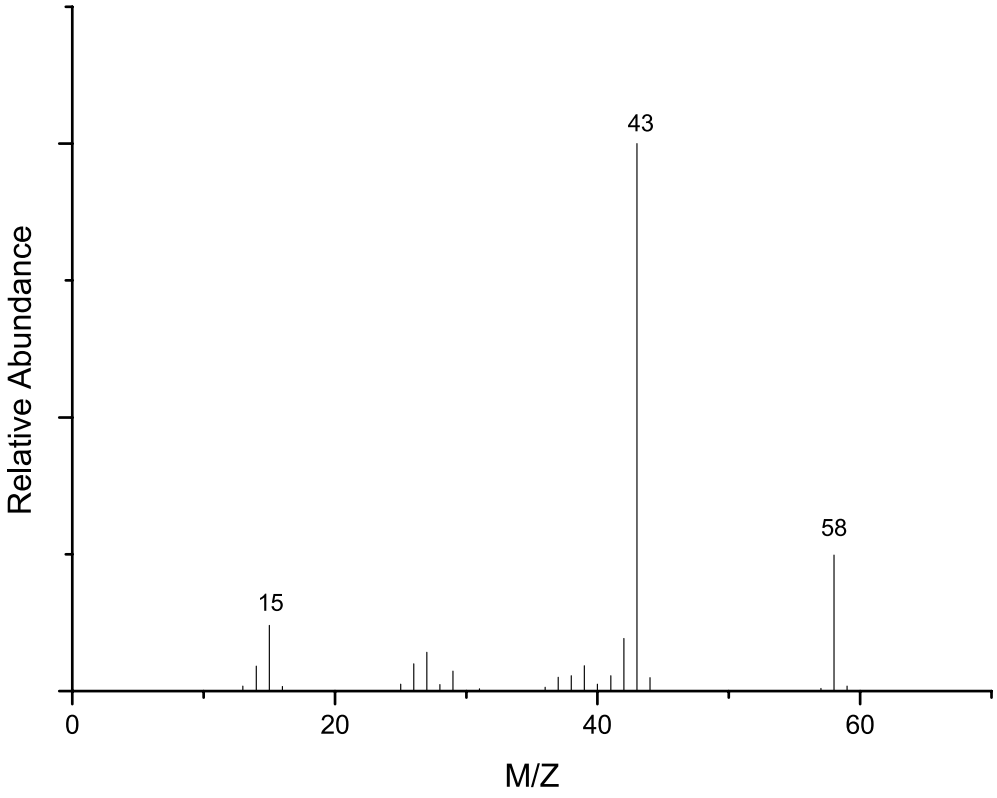

## Sample B

## Empirical Formula C3H8O



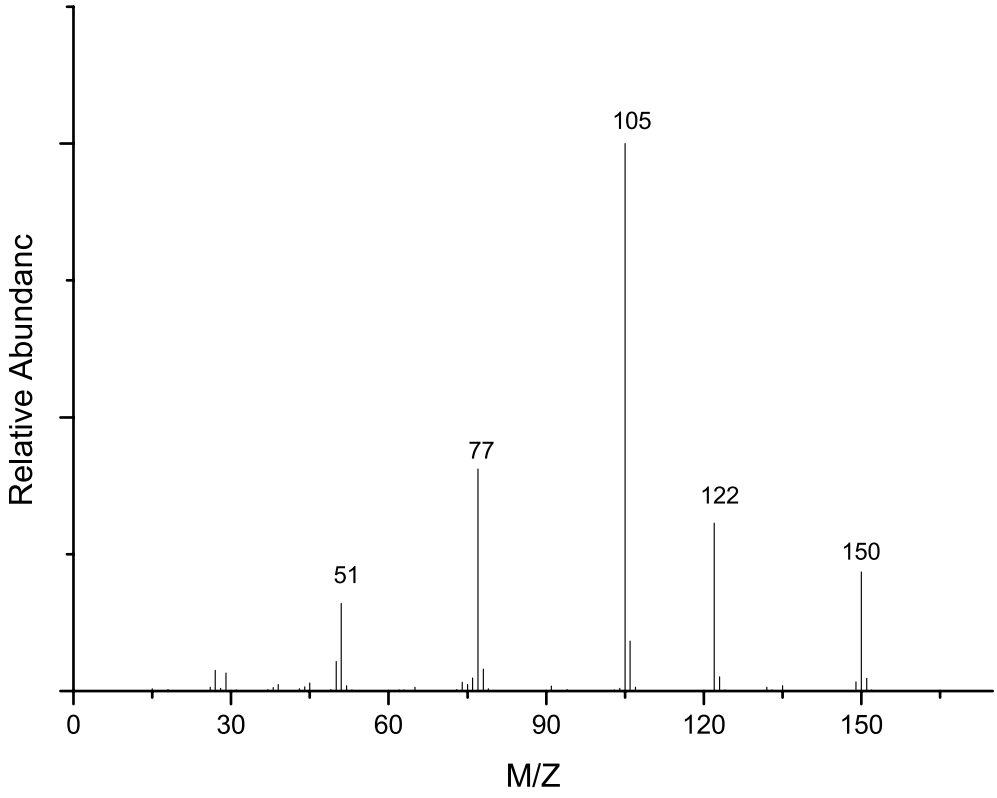
## Sample C

## Empirical Formula C3H6O



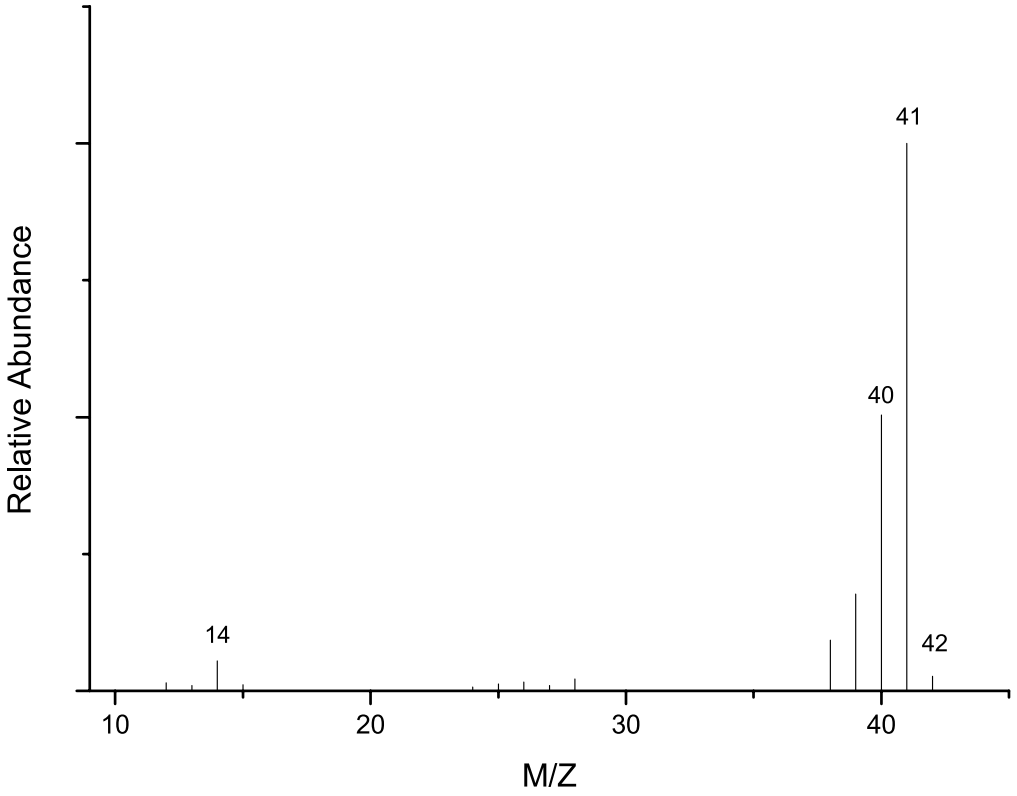

## Sample D

## Empirical Formula C9H10O2



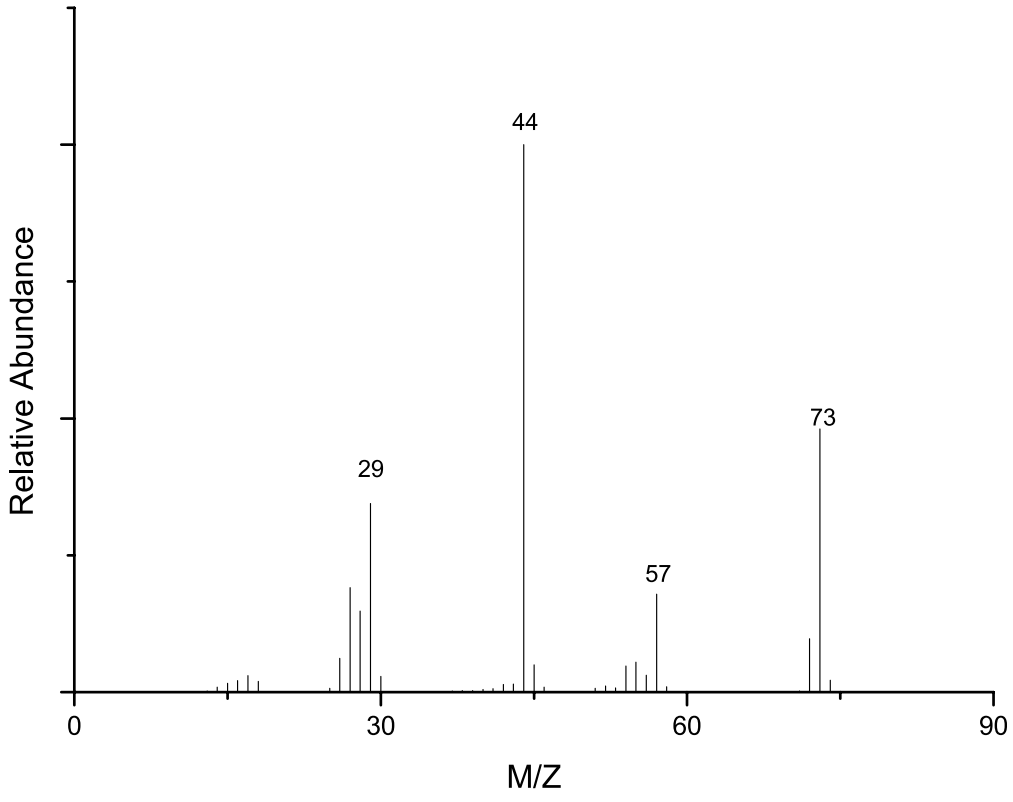
## Sample E

## Empirical Formula C2H3N



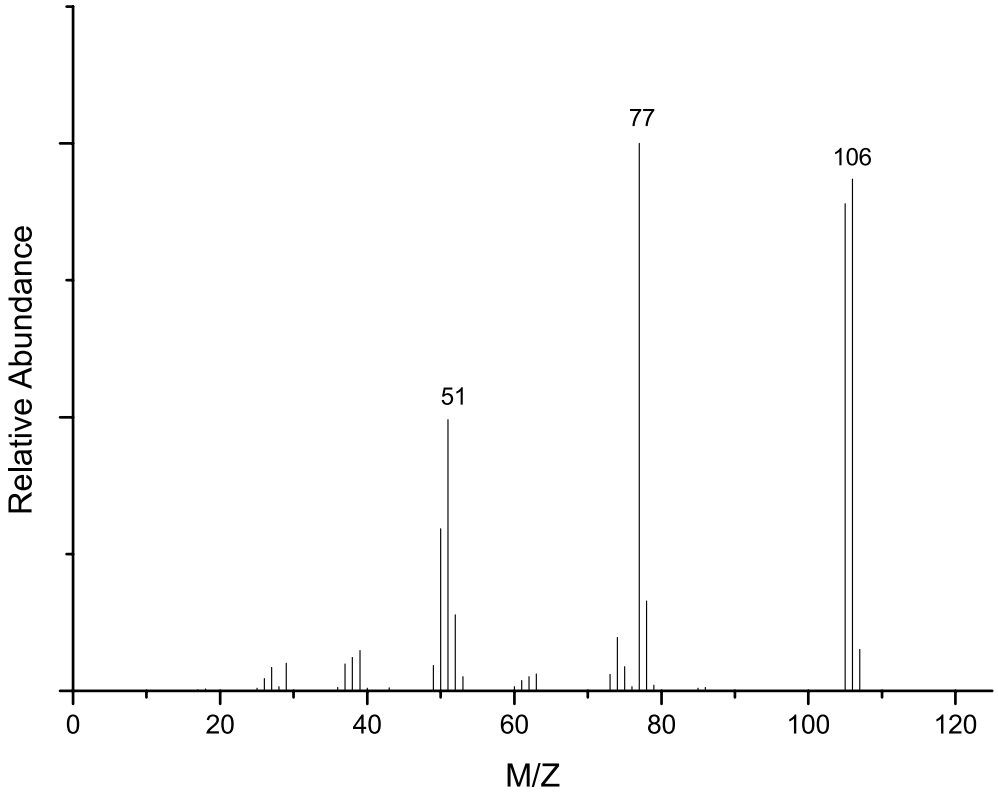

## Sample F

## Empirical Formula C3H7NO



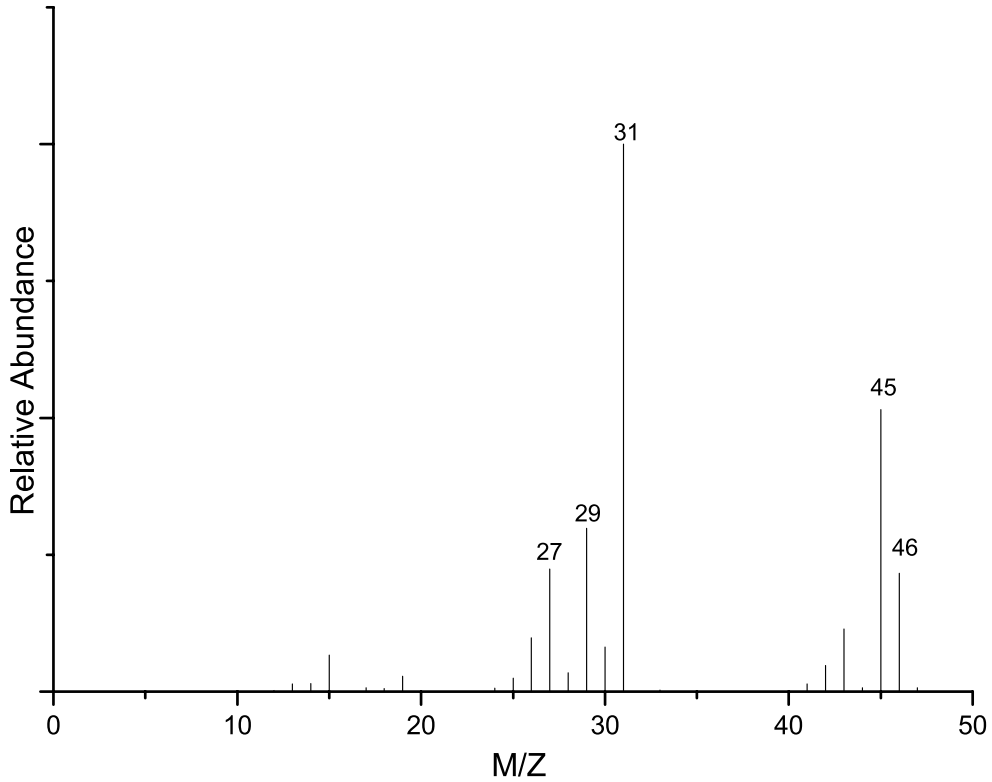
## Sample G

## Empirical Formula C7H6O



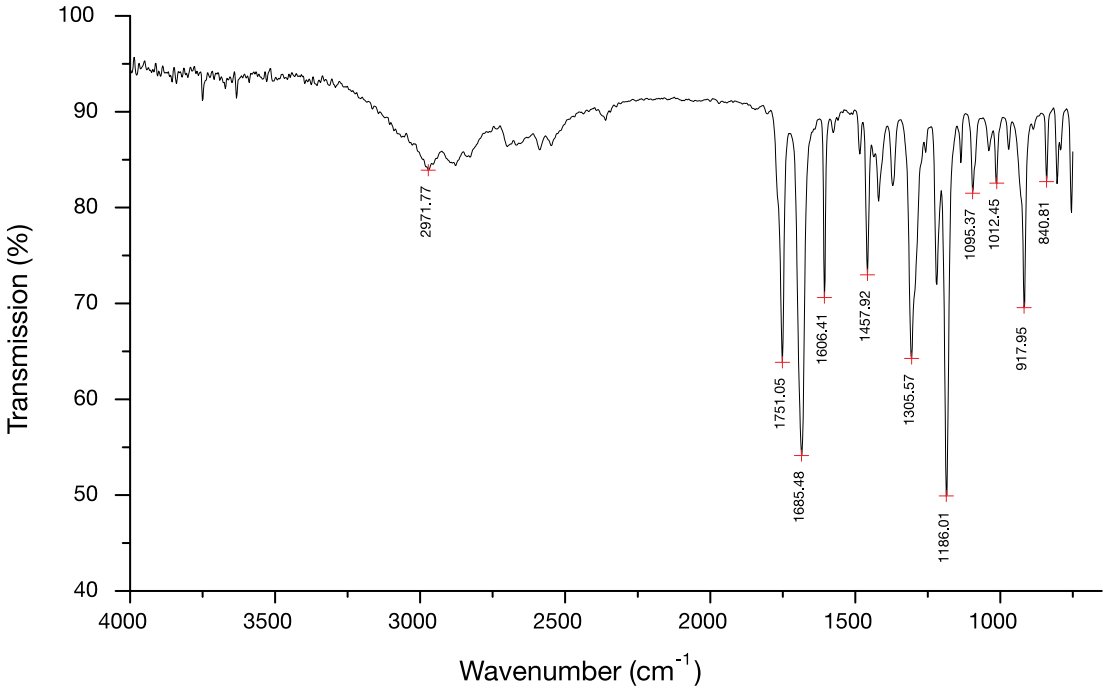

## Sample H

## Empirical Formula C2H6O



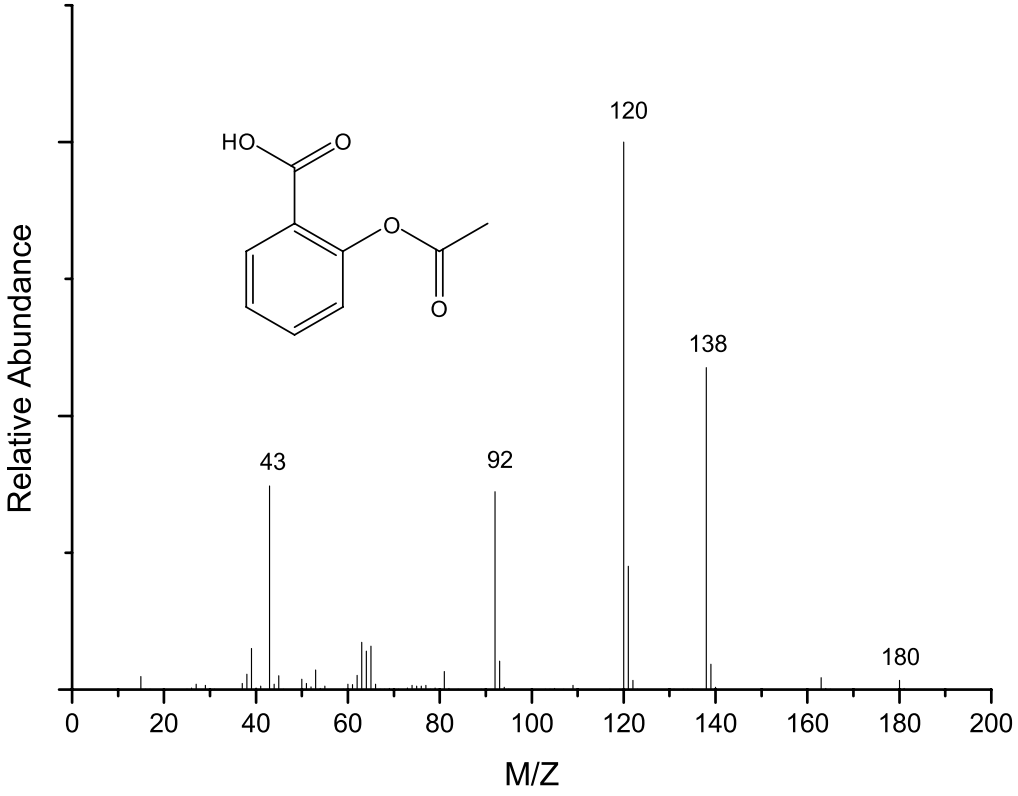
## Sample X

## Infra Red Spectrum




## Mass Spectrum

## Empirical Formula C9H8O4

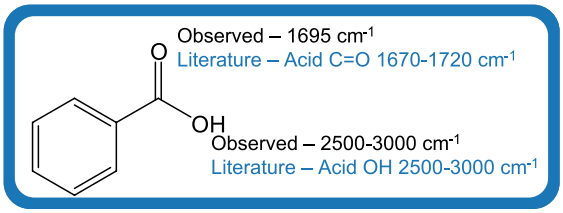


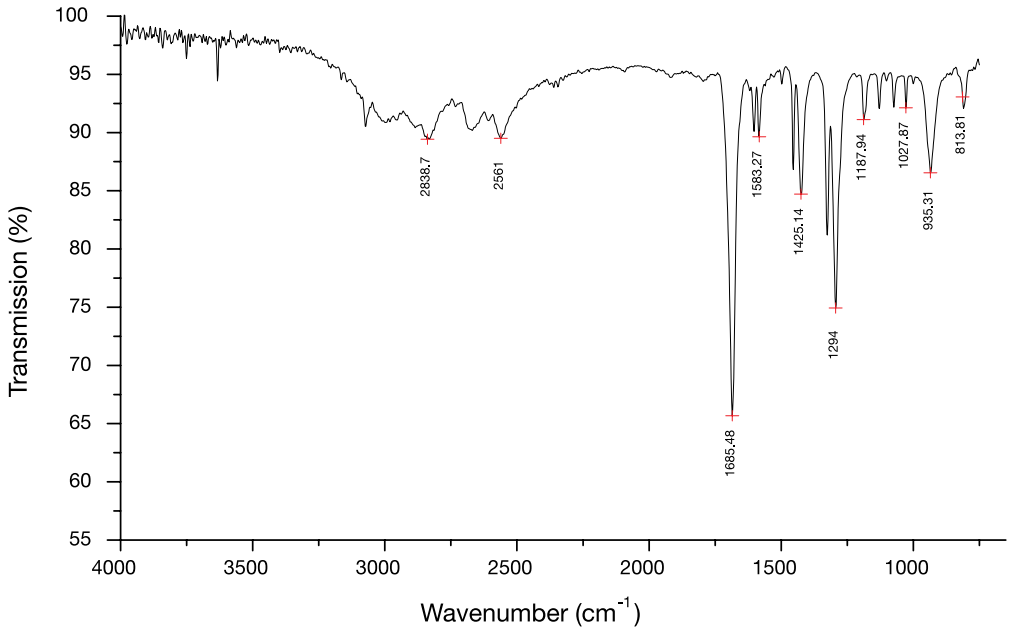
# RESULTS

| SAMPLE | IMPORTANT PEAK VALUES ($cm^{-1}$) | FUNCTIONAL GROUP AND RANGE ($cm^{-1}$) | CLASS OF CHEMICAL (acid, alcohol, Ketone, aldehyde, ester, amide, nitrile) | NAME OF CHEMICAL (Using additional information) |
|---|---|---|---|---|
| A | 2500-3000<br>1695 | OH 2500-3000<br>C=O 1670-1720 | Aromatic Acid | Benzoic Acid |
| B | 3350 | OH 3000-3400 | Alcohol | Propan-2-ol |
| C | 1714 | C=O 1665-1725 | Aldehyde or Ketone | Propanone |
| D | 1720 | C=O 1715-1750 | Ester, Aldehyde or Ketone | Ethylbenzoate |
| E | 2252 | C≡N 2220-2260 | Nitrile | Acetonitrile |
| F | 3187-3360<br>1660 | N-H 3100-3450<br>C=O 1630-1690 | Amide | Propanamide |
| G | 1701 | C=O 1680-1725 | Aromatic Aldehyde, Ketone or Ester | Benzaldehyde |
| H | 3331 | OH 3000-3400 | Alcohol | Ethanol |
| X | 2500-3000<br>1750<br>1683 | OH 2500-3000<br>Acid C=O<br>Aryl Ester C=O<br>1690-1720 | Aromatic Acid<br>*Complex sample other groups present | Acetylsalicylic Acid (Aspirin) |

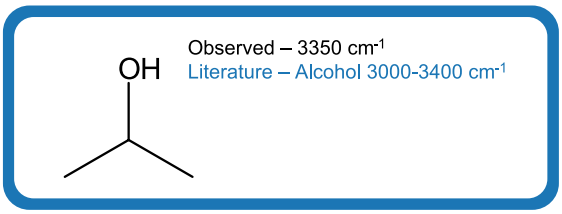
# MODEL SPECTRA

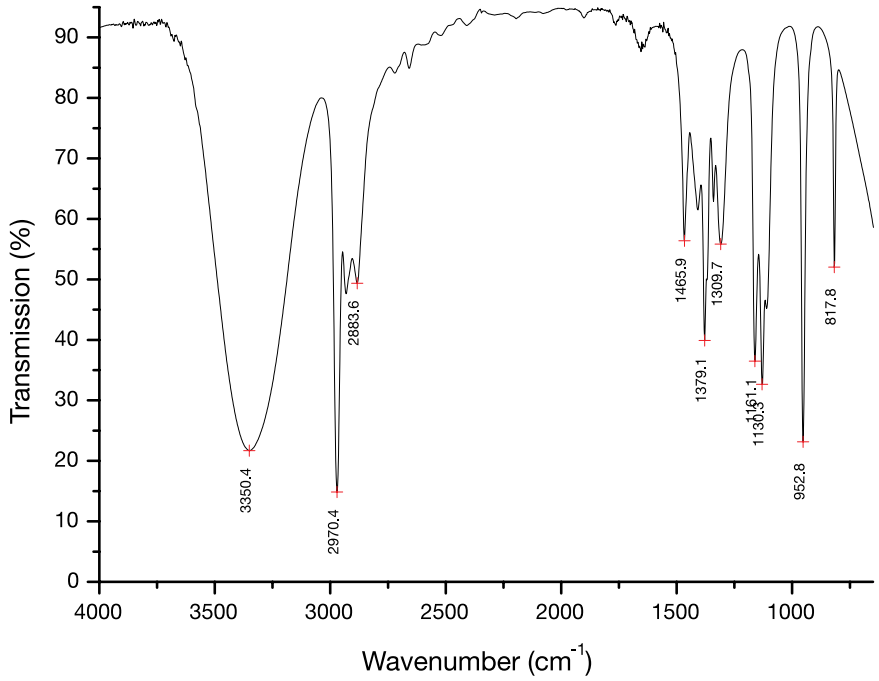
## Sample A – Benzoic Acid



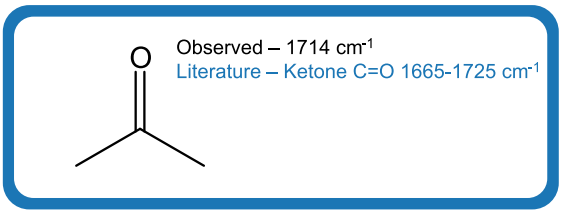



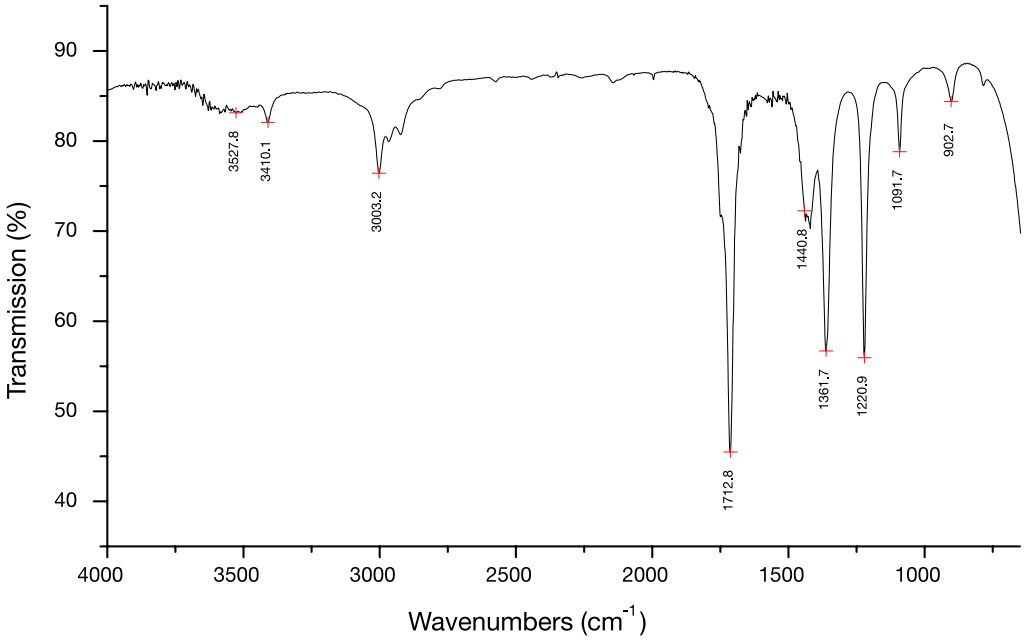

## Sample B – Propan-2-ol



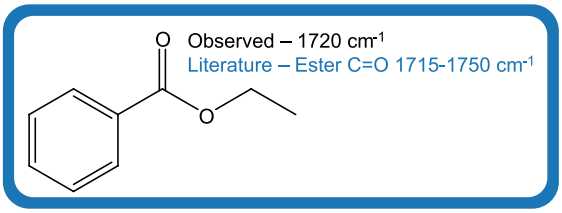



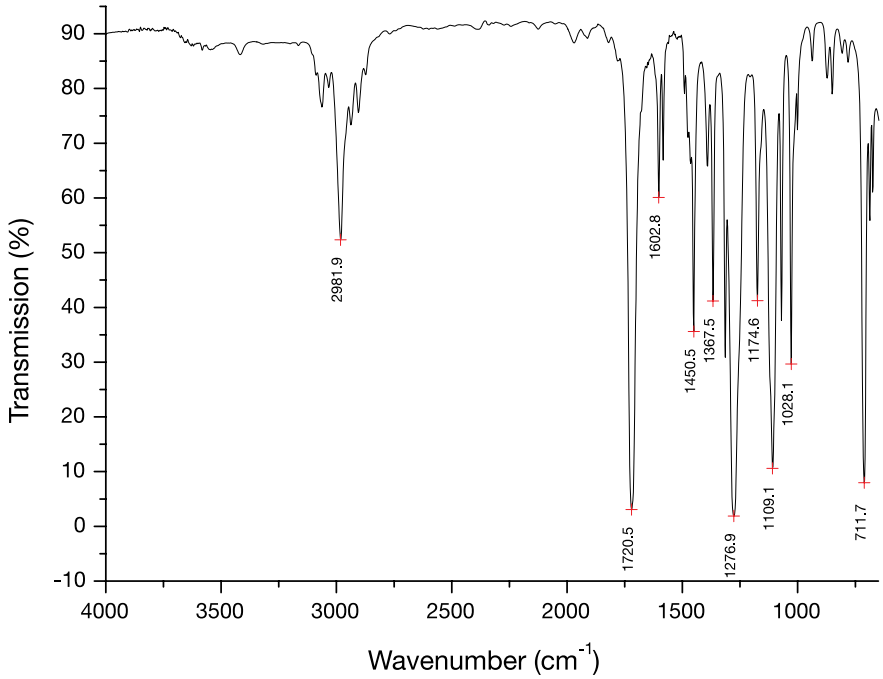
## Sample C – Propanone



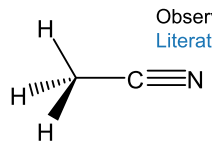




## Sample D – Ethylbenzoate

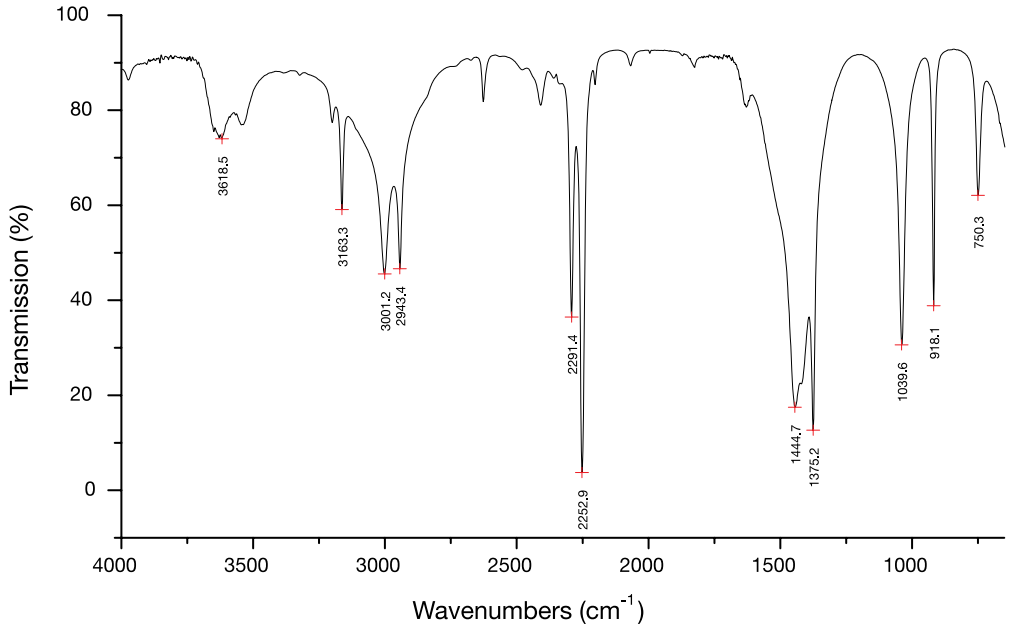


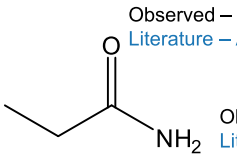
## Sample E – Acetonitrile




Observed – 2252 $cm^{-1}$
Literature – Nitrile CN 2220-2260 $cm^{-1}$

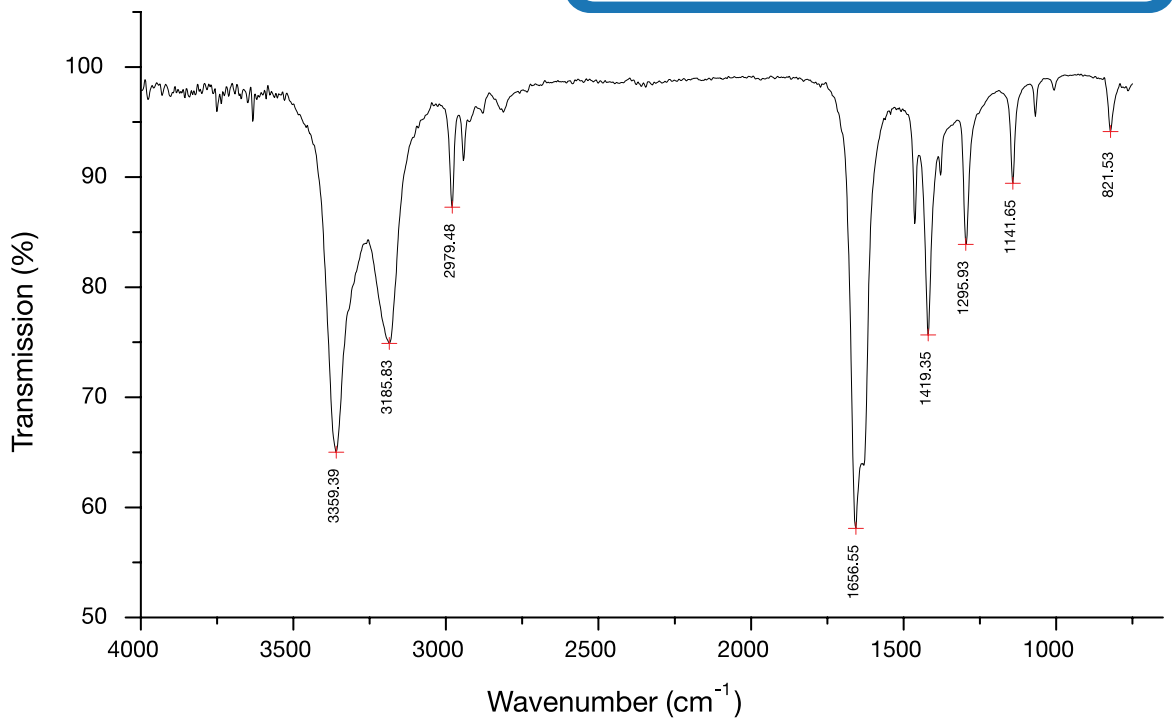



## Sample F – Propanamide

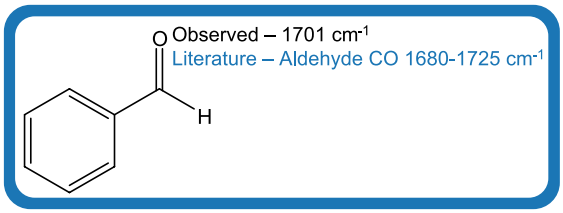


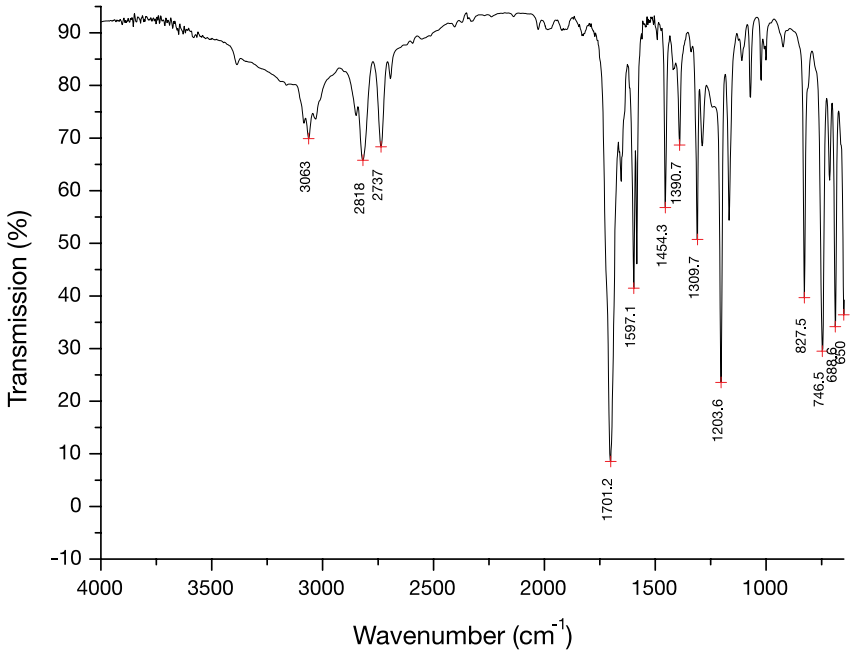

Observed – 1656 $cm^{-1}$
Literature – Amide C=O 1630-1690 $cm^{-1}$

Observed – 3187-3360 $cm^{-1}$
Literature – Amide NH 3100-3450 $cm^{-1}$

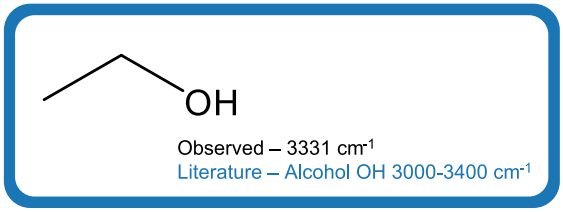


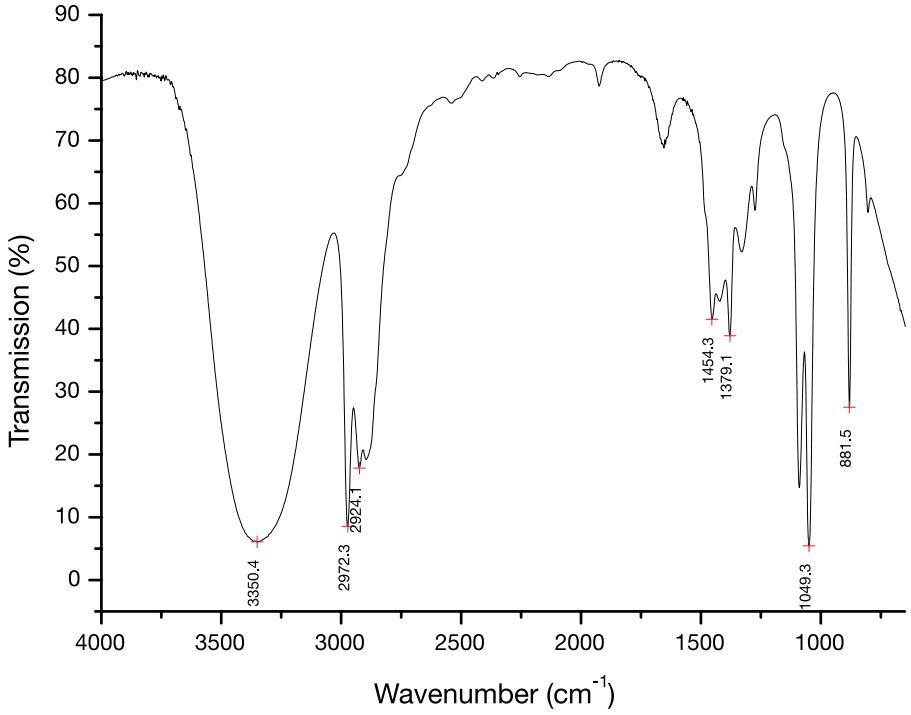
## Sample G – Benzaldehyde



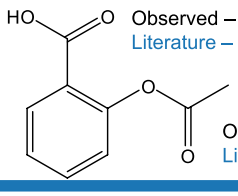

## Sample H – Ethanol



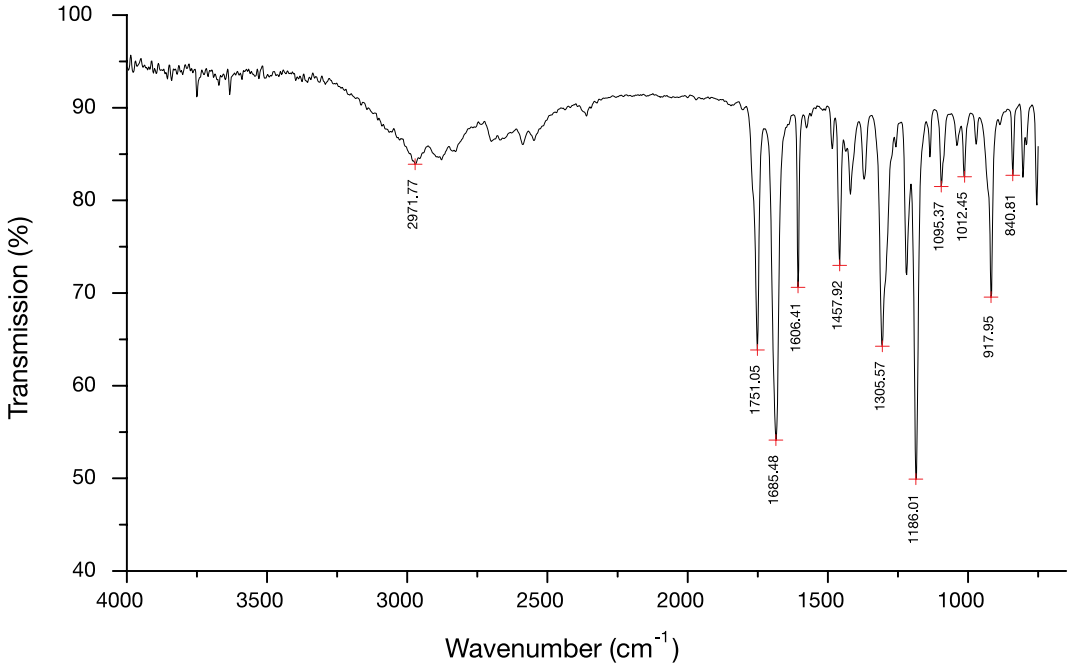
# Sample X – Acetylsalicylic Acid (Aspirin)

Observed – 1683 $cm^{-1}$
Literature – Acid C=O
1690-1720 $cm^{-1}$




Observed – 2500-3000 $cm^{-1}$
Literature – Acid OH 2500-3000 $cm^{-1}$

Observed – 1750 $cm^{-1}$
Literature – Ester C=O 1715-1750 $cm^{-1}$



# STUDENT WORK SHEET

| SAMPLE | IMPORTANT PEAK VALUES ($cm^{-1}$) | FUNCTIONAL GROUP AND RANGE ($cm^{-1}$) | CLASS OF CHEMICAL (acid, alcohol, Ketone, aldehyde, ester, amide, nitrile) | NAME OF CHEMICAL (Using additional information) |
|---|---|---|---|---|
| A | | | | |
| B | | | | |
| C | | | | |
| D | | | | |
| E | | | | |
| F | | | | |
| G | | | | |
| H | | | | |
| X | | | | |