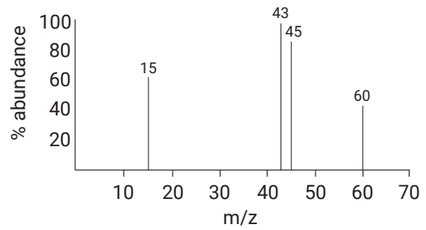
2.8 – Instrumental analysis

1. Mass spectrometry

In a mass spectrometer an electron is knocked off a molecule of a gaseous sample producing a positive ion, **M^+** (the molecular ion). This will produce the peak with the highest m/z value and gives us M_r for the molecule. Some molecules are also split forming fragments (smaller parts of the molecule). Deducing M_r and analysing the peaks produced by the fragments gives information about the structure of the substance, allowing it to be identified.



The mass spectrum of ethanoic acid, CH_3COOH

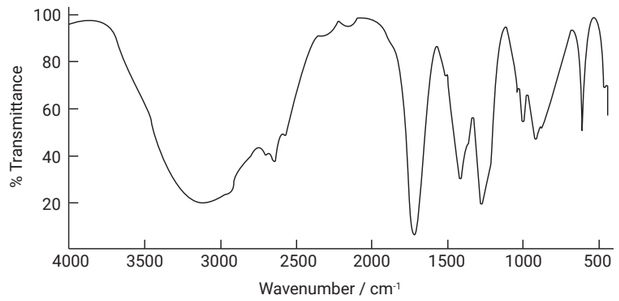
Interpreting the spectrum

1. The peak with the largest m/z value has m/z of 60. This is due to the molecular ion, **M^+**. M_r (CH_3COOH) is 60 so the peak at 60 is due to **CH_3COOH^+**.
2. The peak at m/z 45 (M_r 45) is due to **$COOH^+$**.
3. The peak at m/z 43 (M_r 43) is due to **CH_3CO^+**.
4. The peak at m/z 15 (M_r 15) is due to **CH_3^+**.

Note: When assigning species to a peak you must remember to add a positive charge to all species.

2. Infrared spectroscopy (IR)

Organic molecules absorb energy from the electromagnetic spectrum. In an infrared (IR) spectrometer, a range of IR radiation of different energies is passed through the sample. This IR energy causes increased vibrations and bending of bonds. The spectrum produced shows the energies that are absorbed. The wavenumber where absorption happens is characteristic of that particular bond so helps to identify the functional group present. When using an IR spectrum to deduce the structure of a substance, a lot of the troughs that are shown can be ignored. Look for the presence of expected functional groups or the absence of others to aid identification.



The IR spectrum of ethanoic acid, CH_3COOH

Interpreting the spectrum

Use the infrared absorption values from the data booklet.

1. Absorption at 1650-1750 cm^{-1} due to C=O
2. Absorption at 1000-1300 cm^{-1} due to C–O
3. Absorption at 2500-3200 cm^{-1} due to O–H (carboxylic acid)

Note: Write the wavenumbers as the ranges that are found in the data booklet not as a specific number.

3. Nuclear magnetic resonance spectroscopy (NMR)

Energy is absorbed changing the **spin** of atomic nuclei within a magnetic field. The absorption of energy causes **resonance** and depends on the environment around the atom. Each absorption appears at a different place on the spectrum and is shown as a chemical shift value, δ.

Environment: The nature of the surrounding atoms or groups in a molecule.

Chemical shift: A measure of the difference in parts per million from the energy of a standard absorption.

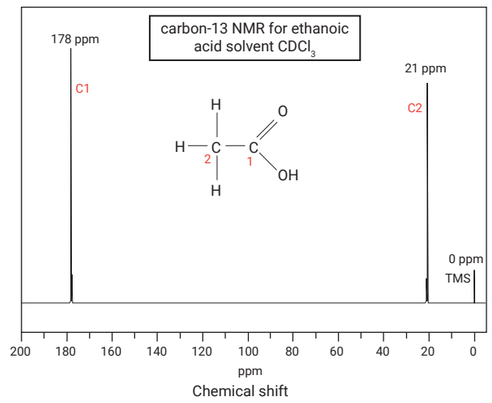
^{13}C spectroscopy

All organic compounds contain a small percentage of the isotope ^{13}C. These atoms absorb energy and produce a ^{13}C NMR spectrum.

The spectrum gives **two** types of information:

- The number of peaks gives the number of different carbon environments.
- The chemical shifts of the peaks give the type of carbon environment.

The ^{13}C NMR spectrum of ethanoic acid, CH_3COOH



Interpreting the spectrum

Use the ^{13}C NMR chemical shift values from the data booklet

1. Two peaks: two different carbon environments
2. Peak C1 at δ 170 due to **C**=O (carboxylic acid)
   Peak C2 at δ 21 due to **C**–C

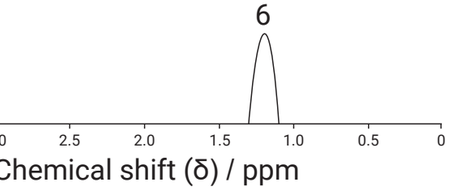
^{1}H spectroscopy (low resolution)

This can also be called proton NMR spectroscopy. These atoms absorb energy and produce a ^{1}H NMR spectrum.

The spectrum gives **three** types of information:

- The number of peaks gives the number of different proton environments.
- **The relative peak areas give the ratio of the number of protons in each environment.**
- The chemical shifts of the peaks give the type of proton environment.

The NMR spectrum of 2-bromopropane



Interpreting the spectrum

Use the ^{1}H NMR chemical shift values from the data booklet.

1. Two peaks: two different hydrogen environments
2. Ratio of peak areas is 1:6 so there must be one hydrogen in one environment and six hydrogens in the other.
3. Peak at δ 1.2 due to –C**H_3**
   Peak at δ 3.8 due to –C**H**Br

4. Identification questions

Questions often give a number of spectra and other information about an unknown compound and ask for the compound to be identified. When answering this type of question, always refer to the inference you can make from every piece of evidence. An inference you make from a mass spectrum could, for example, be confirmed by a piece of evidence from an NMR spectrum. This means that you can be more certain of your overall conclusion and gain the maximum number of marks!