

Measurement and data processing HL

IB CHEMISTRY HL

25 Mn Manganese 54.938045	16 S Sulfur 32.065	J	6 C Carbon 12.0107	2 He Helium 4.002602	25 Mn Manganese 54.938045
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21.1 Spectroscopic identification of organic compounds

Understandings:

- Structural identification of compounds involves several different analytical techniques including IR, ^1H NMR and MS.
- In a high resolution ^1H NMR spectrum, single peaks present in low resolution can split into further clusters of peaks.
- The structural technique of single crystal X-ray crystallography can be used to identify the bond lengths and bond angles of crystalline compounds.

Applications and skills:

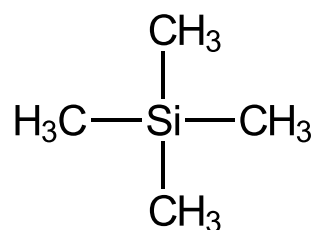
- Explanation of the use of tetramethylsilane (TMS) as the reference standard.
- Deduction of the structure of a compound given information from a range of analytical characterization techniques (X-ray crystallography, IR, ^1H NMR and MS).

Guidance:

- Students should be able to interpret the following from ^1H NMR spectra: number of peaks, area under each peak, chemical shift and splitting patterns. Treatment of spin-spin coupling constants will not be assessed but students should be familiar with singlets, doublets, triplets and quartets.
- High resolution ^1H NMR should be covered.
- The precise details of single crystal X-ray crystallography need not be known in detail, but students should be aware of the existence of this structural technique in the wider context of structural identification of both inorganic and organic compounds.
- The operating principles are not required for any of these methods.

TMS as the reference standard

- The position of the NMR signal is measured relative to the signal produced by TMS tetramethylsilane $\text{Si}(\text{CH}_3)_4$



- All the protons are in the same chemical environment therefore it gives a strong single peak at 0 ppm.
- It is non-toxic and unreactive (does not interfere with sample).
- It absorbs upfield, away from most other protons.
- It is volatile (has a low boiling point) so can easily be removed from the sample.

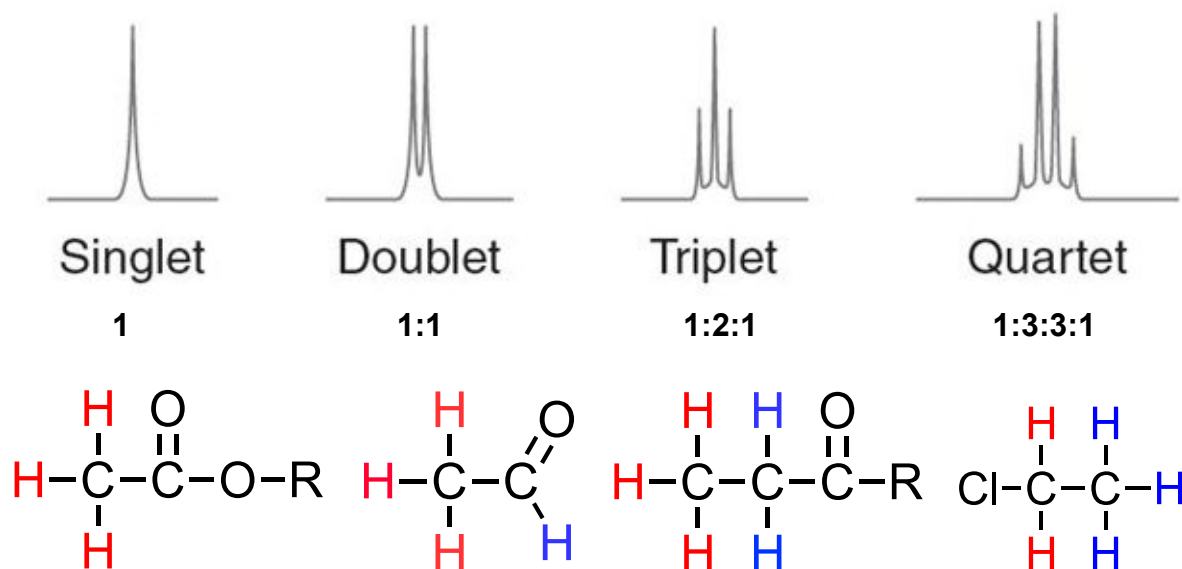
High resolution ^1H NMR

- In a high resolution ^1H NMR spectrum, what looked like single peaks in the low resolution spectrum are split into clusters of peaks.
- The number of peaks tells you about the number of protons attached to the adjacent carbon atom(s).
- The number of peaks in a cluster is one more than the number of hydrogens (protons) attached to the adjacent carbon(s).

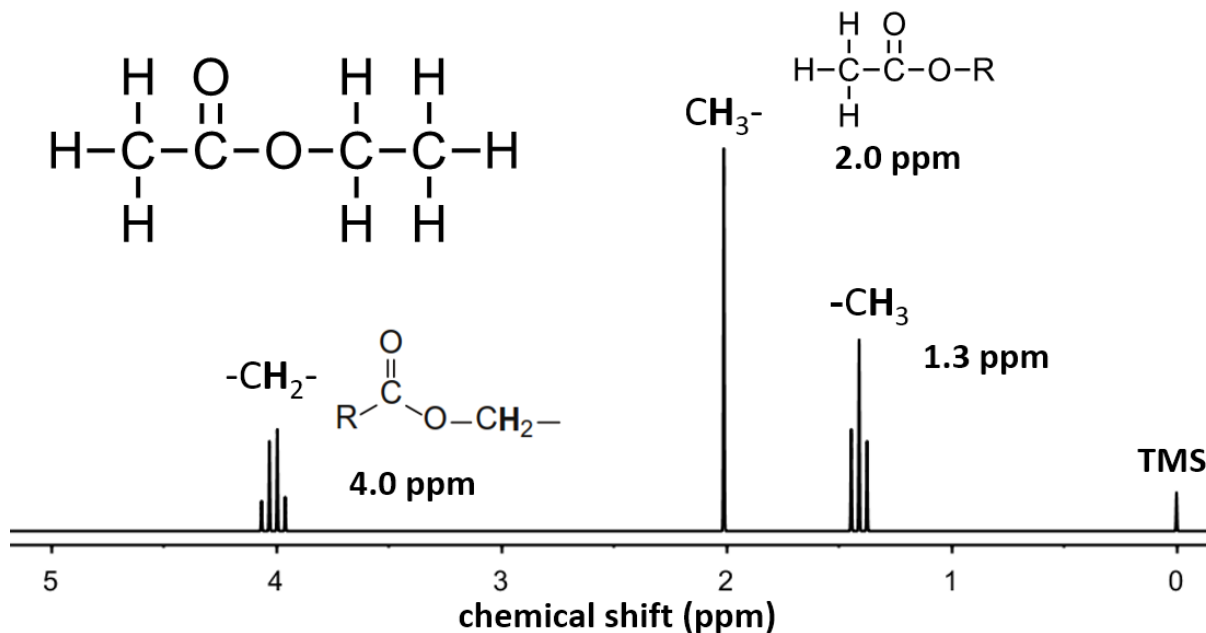
Number of adjacent protons	Number of peaks	Type of splitting	Relative intensities of peaks
0	1	singlet	1
1	2	doublet	1 1
2	3	triplet	1 2 1
3	4	quartet	1 3 3 1

Splitting patterns

- The splitting of the peaks occurs as the effective magnetic field, experienced by particular nuclei, is modified by the magnetic field produced by neighboring nuclei.
- This effect is known as spin-spin coupling.



Example: High resolution ^1H NMR spectrum for ethyl ethanoate



- Triplet at 1.4 ppm caused by two protons on adjacent carbon ($-\text{CH}_2-$).
- Singlet at 2.0 ppm caused by zero protons on adjacent carbon ($\text{C}=\text{O}$).
- Quartet at 4.0 ppm caused by three protons on adjacent carbon ($-\text{CH}_3$).

X-ray crystallography

- X-ray crystallography is a scientific method of determining the precise positions/arrangements of atoms in a crystal where beams of X-ray strike a crystal and causes the beam of light to diffract into many specific directions.
- It is used to measure bond lengths and bonds angles in a molecule.