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SPECTROSCOPY in a SUITCASE

This Resource Pack is for...

...those interested in running a Spectroscopy in a Suitcase Workshop.
Teachers of Year 11-13 chemistry students and demonstrators can use this booklet as a guide to spectroscopic techniques and run a practical demonstration of Infrared Spectroscopy.

This Resource Pack includes...

Introductions on Spectroscopy, the Electromagnetic Spectrum, Mass Spectrometry, Nuclear Magnetic Resonance, Infrared Spectroscopy as well as a combinatory workshop and Infrared Spectroscopy Practical that study 8 unknown small inorganic compounds.

This Resource Pack was made by...

Eleanor Keil of The University of Southampton, with assistance from The National Crystallography Service, UK and based upon an original iteration of Spectroscopy in a Suitcase by The Royal Society of Chemistry.

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Contents

What is Spectroscopy?

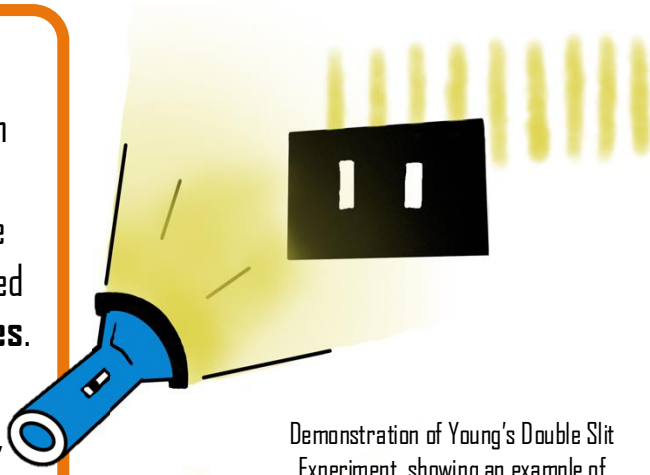
History

The word 'spectroscopy' has been in the Oxford English Dictionary for over 100 years (*spectroscopy*. Oxford Reference. DOI:10.1093/oi/authority.20110803100522408). Combining the words '**spectrum**' (meaning a full range) and '**spectre**' (as in a visible apparition) with the '**–scopy**' suffix from French, Greek and Latin origins meaning 'to study or examine', it is clear to see how 'spectroscopy' refers to the study of the electromagnetic spectrum. Despite the word being used for over a century, the definition is still being updated in the 2020's, showing how prevalent and important the field is to modern science.

Nowadays, spectroscopy describes the scientific discipline of studying how light – electromagnetic radiation – interacts with matter. Conversely, spectro**metry** describes the process of **measuring** these interactions and often will apply to analytical techniques that do not use light.

What is light?

Light has the interesting ability to be studied in multiple ways. The physics concept known as **Wave-Particle Duality** shows how light can be described as a particle; a **photon** with associated **energy**, and as a **wave**; with various **frequencies**. A fun way to prove this to students is by demonstrating Young's Double Slit Experiment, showing the interference of light waves that pass through a gap in paper, first done in 1801!



Demonstration of Young's Double Slit Experiment, showing an example of how light can behave like a wave..

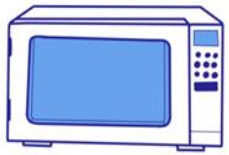
The Electromagnetic Spectrum

Electromagnetic waves travel all at the speed of light when in a vacuum (approximately 300million metres per second), and slower when passing through matter. They do not necessarily require any matter to pass through, but when passing through an electronic or magnetic field the waves vibrate transversely with an associated frequency and wavelength, shown on the next page. Different types of radiation arise from different frequencies of electromagnetic waves, including visible light.

The Electromagnetic Spectrum?

Radio	Microwave	Infrared	Visible	Ultraviolet	X-ray	Gamma

Occurrences:





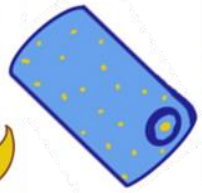

Wavelengths:

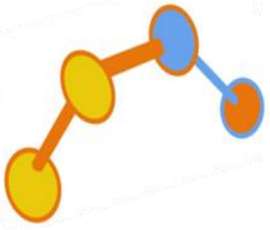
1 x 10^{3} metres AKA 1000 metres	1 x 10^{-2} metres AKA 1 cm	1 x 10^{-5} metres AKA 0.01mm	1 x 10^{-6} metres AKA 1/1000th of a mm	1 x 10^{-8} metres AKA 10 nanometres	1 x 10^{-10} metres AKA 1 Angstrom	1 x 10^{-12} metres AKA 1 picometre

Similar size to...









Frequencies:

1 x 10^{4} Hertz	1 x 10^{8} Hertz	1 x 10^{12} Hertz	1 x 10^{15} Hertz	1 x 10^{16} Hertz	1 x 10^{18} Hertz	1 x 10^{20} Hertz

Common Analytical Techniques:

NMR Spectroscopy		IR Spectroscopy		UV-Vis Spectroscopy	X-ray Crystallography	

Increasing frequency and energy

Increasing wavelength

SIAS Workshop

The objective of the SIAS Workshop is to allow students the opportunity to practice critical thinking in an analytical setting and provide them with the opportunity to prep and characterise samples using the Infrared Spectrometer that is common in Higher Education and research environments. The workshop itself includes a short presentation, a series of spectra for eight unknown compounds and the practical use of the IR. It has been designed to be easily adapted for students of many abilities and backgrounds to be all inclusive and allow students to progress their knowledge and understanding. Prior knowledge of some basic functional Groups is preferable while an understanding of the spectra is ideal.

PRACTICAL METHODOLOGY

Preparation and Set-Up:

1. Ensure the Risk Assessment/COSHH form have been read, understood and adhered to.
2. Plug in the IR and laptop to both the mains and one-another, somewhere appropriate for students to view near a safe location to display the eight compounds.
3. The IR will need the anvil mount attached and may need to replace the powder-disc mounts – do so by pressing the lock/unlock silver button.
4. Turn on and login to the laptop. Open the OPUS software once the IR has fully turned on (shows a green light) and been connected to the laptop. This may take a few minutes to fully boot up.
5. Collect a background measurement, being certain that the stage is clean.

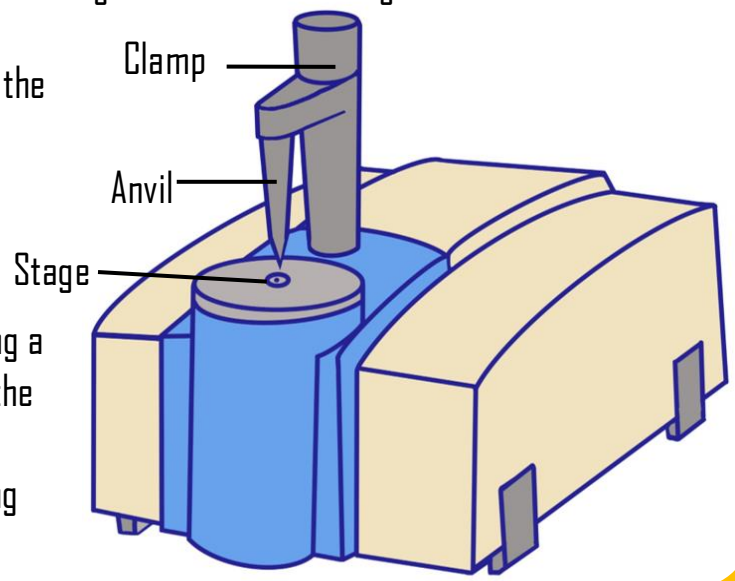
Liquid Samples:

Pipette 1-2 drops of solution onto the centre of the stage. Screw/pull lever on the clamp to secure and Collect measurement.

Solid Samples:

Repeat the procedure above, using a very small amount of powder on the end of a spatula.

Spectra can be manipulated by adjusting Axis and changing colour schemes.



Infrared Spectroscopy

Originally described as 'radiant heat', William Hershel noticed an unusual spike in temperature after attempting to measure the temperature of the red section of visible light, marking the first observation of Infrared (IR) frequencies in 1800 (W. Herschel, 1800, DOI: 10.1098/rstl.1800.0014). The frequency of IR waves are between **300Billion-430Trillion Hertz** with a wavelength similar size to that of a needle-point or a human hair.

THEORETICAL BACKGROUND

Bonds **vibrate** in many different ways when a molecule is at a temperature above absolute zero. When an IR frequency matches the natural vibrating frequency of a bond, the **energy** is absorbed, and the amplitude of the vibration is increased. This is called **resonance**, Different types of bonds have different vibrations. Vibrations are affected by:

- **Type** of vibration. (Bending vibration are lower energy and lower wavenumber than stretching vibrations.)
- **Strength** of the bond. (Tripple bonds are stronger than single bonds and absorb higher wavenumbers)
- **Mass** of the atoms. (Heavier atoms vibrate slower/at lower frequency than lighter atoms.)

AIMS

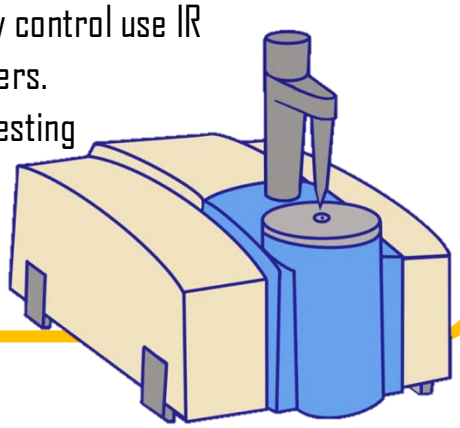
Identify the **functional groups** within a compound for **characterisation**.

METHODOLOGY

An IR spectrometer will expose the sample to the full IR spectrum and detects the absorption of the sample to detect what kind of bonds are present. This is measured using **wavenumber** **cm^{-1}**, which is the **inverse** of the **wavelength** and therefore **proportional** to the **energy** and **frequency** of the vibrating bond.

APPLICATIONS

- Any heat is the release of IR waves.
- TV remotes and automatic doors use IR detectors.
- Some snakes have organs that have the ability to sense warm-blooded prey.
- Breathalysers are limited range IR spectrometers.
- Food quality control use IR spectrometers.
- Analytical testing of paintings and artefacts.



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Mass Spectrometry

Mass Spectrometry (MS/Mass Spec) does not use the EM spectrum, hence not being called a spectro**scopic** technique. It does, however, utilise the effects **electromagnetic fields** have on ionised fragments, first discovered at the turn of the 20th century (Wein, 1989).

THEORETICAL BACKGROUND

Any flow of charge (**electrical current**) from electrons (or positively charged ions) has an associated **magnetic field**. This **electromagnetic field** will deflect when put into an external magnetic field. This is also the case for **positively charged ions**, which **deflect** differently, depending on their **mass** and their **charge**.

AIMS

Determine the structure of compounds by viewing the connectivity of their fragmentation.

METHODOLOGY

The general process of Mass Spec breaks molecules into positively charged fragments and measures the mass/charge ratio (m/z) of those fragments via the following steps:

1. **Vaporisation**: allows the sample to move freely.
2. **Ionisation**: occurs when the sample is hit with an electron gun causing the loss of an electron and fragmentation.
3. **Acceleration**: of the ions through a vacuum using an electronic field.
4. **Deflection**: occurs as heavier fragments take longer to travel through the column, than lighter ones.
5. **Detection**: Fragments then hit the detector triggering an electronic pulse that can computational be measured and produce results as a graph..

APPLICATIONS

- Airport and athletic drug testing.
- Pharmacology development.
- Astrological analysis.
- Separating Uranium isotopes during the Manhattan Project.

Note: There are many different types of Mass Spec, so the applications can vary.

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Nuclear Magnetic Resonance

Nuclear Magnetic Resonance (NMR) is the newest of the techniques in this workbook but has still provided its fair share of Nobel Prizes. NMR uses **Radio waves** to provide a source of **energy** to be absorbed by atoms, promoting the atoms to higher energy states and then measuring the energy released by the atoms as they return to the relaxed states.

THEORETICAL BACKGROUND

Protons and **Neutrons** each have '**spin**'. Just like the needle on a compass aligning with the geomagnetic field of Earth, the magnetic spin of protons and neutrons align with an external magnetic field. If a molecule has an **even** number of protons and neutrons these spins cancel out to zero. But with an **odd** number, like C^{13} or H^{1}, the atoms have a magnetic moment.

METHODOLOGY

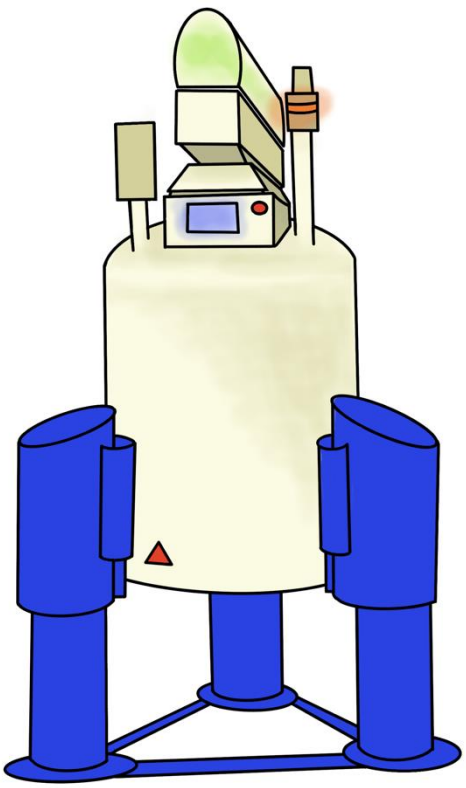
For NMR the sample is robotically placed within a liquid helium-cooled **electromagnetic field**. This causes atoms with a **magnetic moment** to **align** with the field in a relaxed state. **Radio waves** are applied and absorbed by the atoms, reaching an **excited state**, in opposite spin to the field (**resonance**). As the atoms relax back to the aligned spin state, energy is **emitted.** NMR spectrometers measure the resonance and produce a spectra, plotting the different atomic environments' **shift** in **parts per million** (ppm). This 'shift' refers to a comparison toa standardised measurement, often tetramethylsilane (**TMS**), which is set to zero.

AIMS

Locate the **position** of atoms in a molecule by determining the **functional groups** and **connectivity** present.

APPLICATIONS

- MRI body scans.
- Environmental monitoring.
- Medical diagnoses.
- Quantum state computing.



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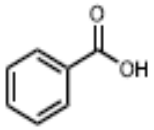
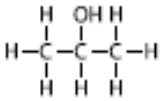
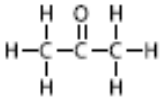
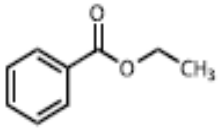
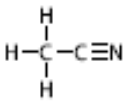
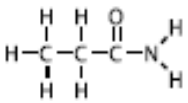
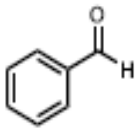
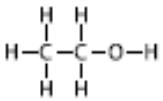
Spectroscopy in a Suitcase: Student Worksheet Answers

Sample	IR Analysis (Wavenumber and corresponding functional group)		Mass Spec Analysis (weights and possible fragments)	NMR Analysis (no. of environments, number of Hydrogens, No. of neighbours)	Chemical Structure and Name
A	2500–3000 1695	OH 2500–3000 C=O 1670–1720	Mass = 122 105 = $[M–OH]^+$ 77 = $[M–COOH]^+$	ppm / Int. ratio / n+1 12 / 1 / 1 8.2 / 2 / 2 7.6 / 3 / 5	Benzoic acid 
B	3350	OH 3000–3400	Mass = 60 45 = $[M–CH_3]^+$ 27 = $[M–CH_3 – H_2O]^+$	5 / 1 / 1 4 / 1 / + 1.2 / 7 / 2	Propan-2-ol 
C	1714	C=O 1665–1725	Mass = 58 43 = $[M–CH_3]^+$ 15 = $[M–CH_3 – COH]^+$	2 / 5 / 1	Propanone 
D	1720	C=O 1715–1750	Mass = 150 122 = $[M–CHCH_3]^+$ 105 = $[M–OCH_2CH_3]^+$ 77 = $[M–COOCH_2CH_3]^+$	8 / 2 / 3 7.4 / 3 / 3 4.3 / 2 / 4 1.3 / 3 / 3	Ethylbenzoate 
E	2252	C=N 2220–2260	Mass = 41 40 = $[M–H]^+$ 14 = $[M–HCN]^+$	2 / 2 / 1	Acetonitrile 
F	3187–3360 1660	N–H 3100–3450 C=O 1630–1690	Mass = 73 57 = $[M–NH_2]^+$ 44 = $[M–CH_2CH_3]^+$ 29 = $[M–CONH_2]^+$	6.2 / 2 / 1 2.5 / 2 / 4 1.4 / 3 / 3	Propanamide 
G	1701	C=O 1680–1725	Mass = 106 77 = $[M–COH]^+$	10 / 1 / 1 7.5 / 6 / 5	Benzaldehyde 
H	3331	OH 3000–3400	Mass = 46 45 = $[M–H]^+$ 31 = $[M–CH_3]^+$ 29 = $[M–OH]^+$ 27 = $[M–CH_2 – H_2O]^+$	5.2 / 1 / 1 3.6 / 2 / 4 1.2 / 3 / 3	Ethanol 

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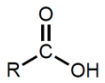
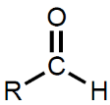
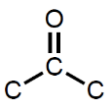
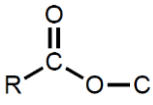
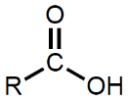
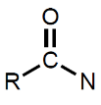
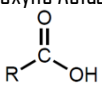
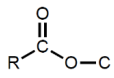
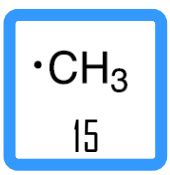
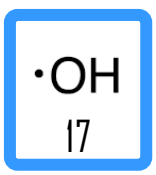
Spectroscopy in a Suitcase: Cheat-Sheet 1

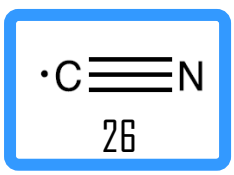
Common Functional Groups found in IR Spectra:

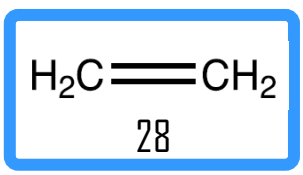
Bond	Wavenumber (cm^{-1})	Possible Functional Groups
O-H	3400-2500 (Broad)	Alcohols, R—OH Carboxylic acids,  Phenols 
N-H	3600-3100	Amides,  Amines C—N
C-H	3150-2850 (sharp)	All Organic compounds
C≡N	2260-2200	Nitriles C≡N
C=O	1750-1630 (sharp)	Aldehydes,  Ketones,  Esters,  Carboxylic Acids,  Amides 
C=C	1680-1610	Alkenes C=C
C-O	1300-1060	Alcohols, R—OH Carboxylic Acids,  Esters,  Ethers C—O—C

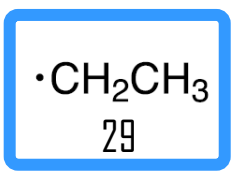
Common Ion Fragments (and their molecular weights) found in Mass Spectra:

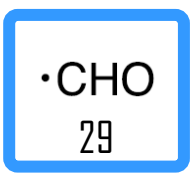


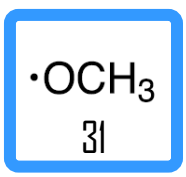


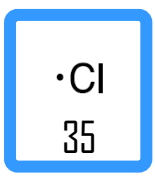


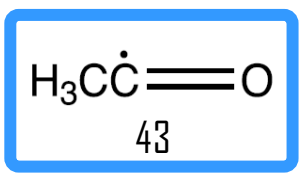


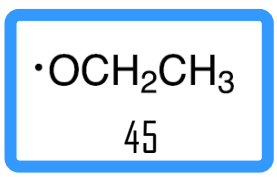


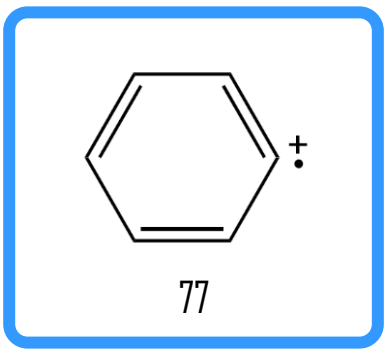


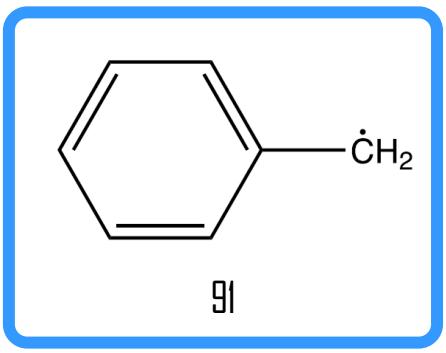


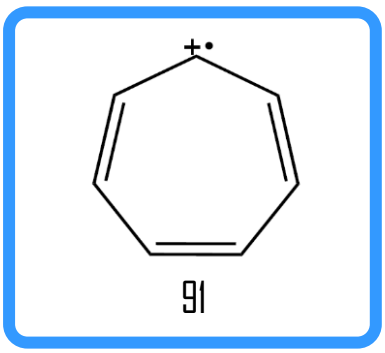












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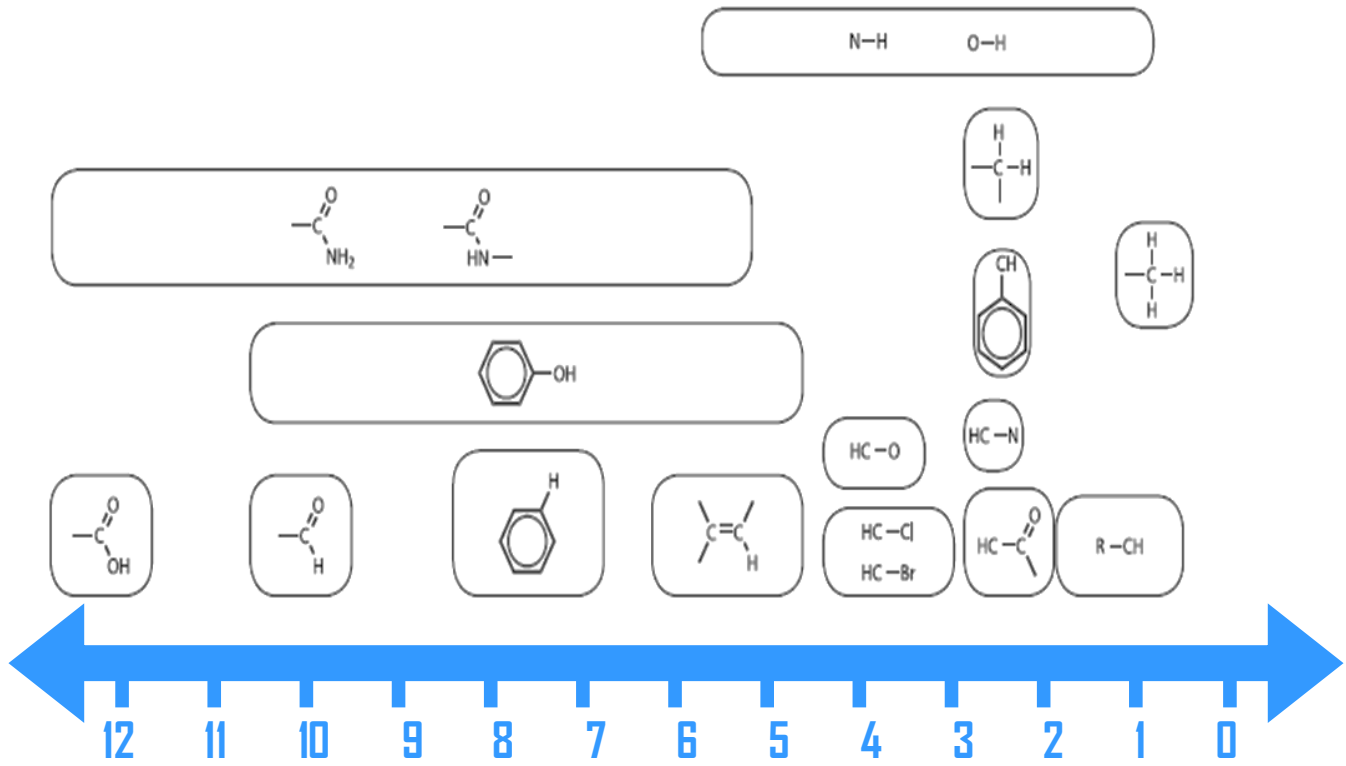
Spectroscopy in a Suitcase: Cheat-Sheet 2

Common hydrogen environments found in Proton NMR chemical shifts:



Chemical Shift (ppm) (Relative to TMS)

Downfield, De-shielding

Upfield, Shielding

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