Edexcel Chemistry A-level

Topic 19 - Modern Analytical Techniques II

Flashcards

What does NMR stand for?

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Nuclear Magnetic Resonance

What are the basic principles of NMR?

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You can find the structures of complex molecules by placing them in a magnetic field and applying EM waves of radio frequency to them. If radio waves of the right frequency are absorbed, the nuclei flips from parallel to applied magnetic to field to anti-parallel. This energy change can be monitored and recorded. Uses the resonance of nuclei with spin.

How would you carry out NMR spectroscopy?

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Dissolve the liquid sample in suitable solvent, put in a tube along with a small amount of TMS and put the tube into an NMR machine. The sample is spun to even out any imperfections in the magnetic field and the spectrometer is zeroed against the TMS. Radiation with different radio frequencies but a constant magnetic field is applied to the sample and any absorptions (due to resonance) are detected

Give one use of NMR?

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MRI scans

What kind of nuclei does NMR work with (and examples)?

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Those with an uneven number of nucleons, meaning they will spin e.g. ^{1}H, ^{13}C

What percentage of carbon atoms are ^{13}C?

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1% - but modern instruments are sensitive enough to detect this

What defines the resonant frequency of a ^{13}C atom?

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The chemical environment that it is in; the amount of electron shielding it has.

What graph is produced by NMR spectroscopy?

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Energy absorbed against chemical shift

What is chemical shift? What is its symbol? What are its units?

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The resonant frequency of the nuclei, compared to that of a ^{1}H atom in TMS.

Symbol □

Parts per million (ppm)

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0-200ppm

What means ^{13}C atoms show a different chemical shift value?

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What kind of environment leads to a greater chemical shift?

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A C atom next to more electronegative atom has a greater chemical shift.

Summarise what these mean for ^{13}C NMR:

Number of signals?
Chemical shift?
Area under peak?
Splitting?

Summarise what these mean for ^{13}C NMR:

Number of signals: One signal for each carbon environment (each set of inequivalent ^{13}C atoms)

Chemical shift: Greater □ from atoms closer to electronegative atoms or C=C

Area under peak: no meaning

Splitting: there is no splitting for ^{13}C NMR

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What is the integration trace?

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A stepped line that makes it easier to measure the area under the curve (height of line = area under that peak)

What is TMS (name and structure)?

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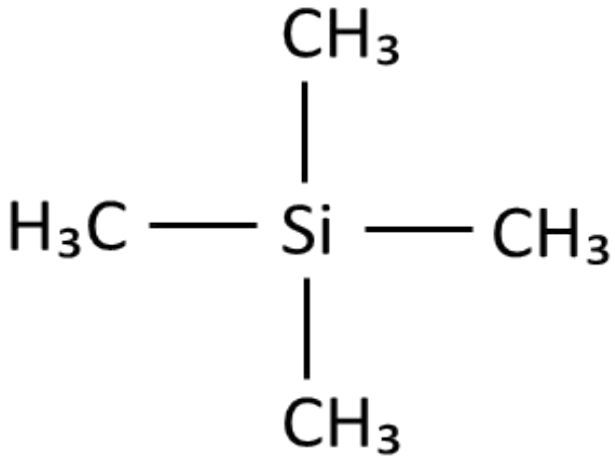
Tetramethylsilane



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Why is TMS used?

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Can be added to sample to calibrate the NMR equipment. It provides a peak at exactly □ = 0ppm. It is the reference point against which all □ are measured

What are other advantages of using TMS?

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Inert, non-toxic, easy to remove from the sample (as relatively volatile)

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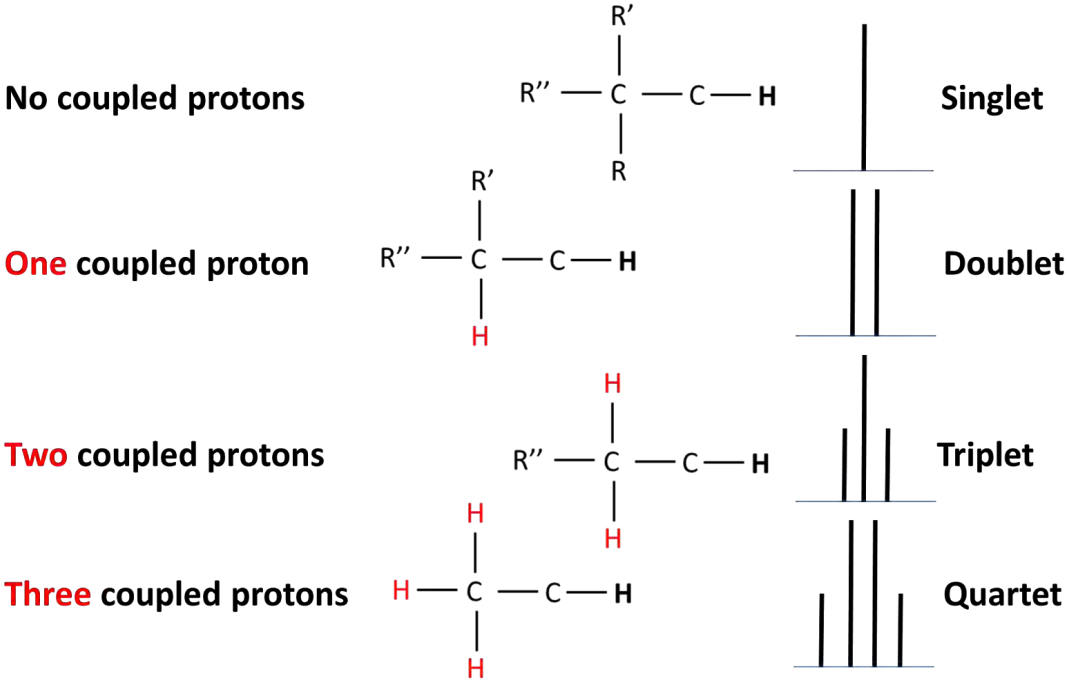

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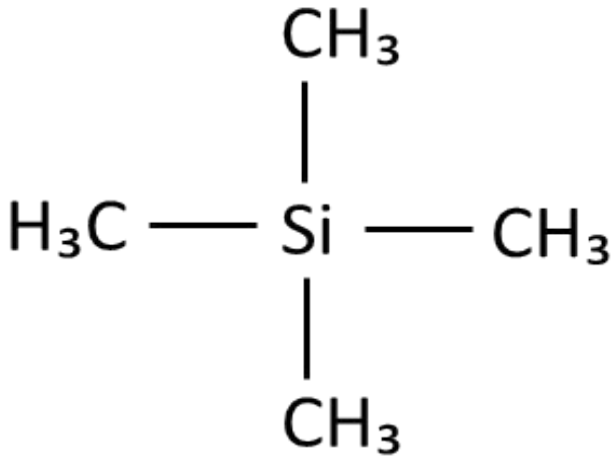

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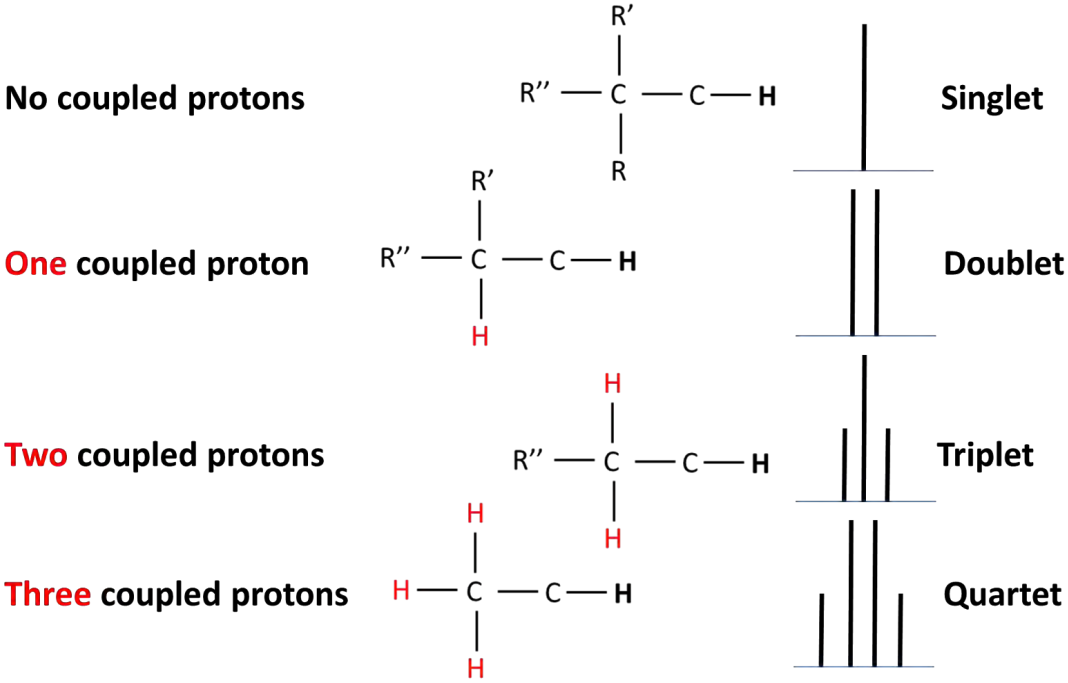

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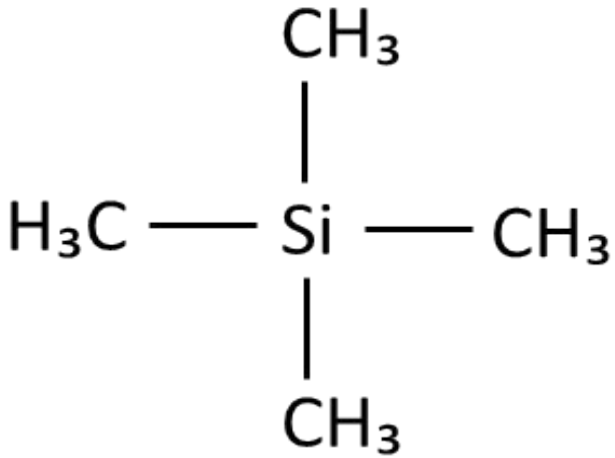

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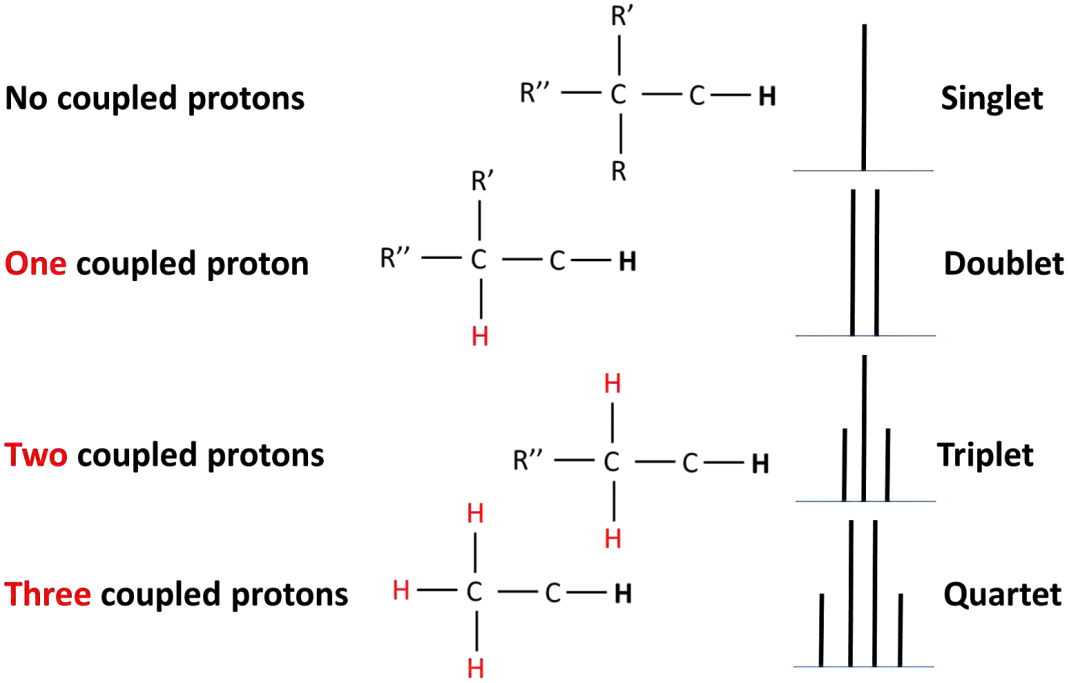

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What are the basic principles of all kinds of chromatography?

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A family of separation techniques that depend on the principle that a mixture is separated if it is dissolved in a solvent and this mobile phase is passed over a solid (the stationary phase).

What is the mobile phase?

What is the mobile phase?

Carries the soluble components of the mixture

What relationship between a sample and the mobile phase makes the sample move faster?

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More soluble components / components with more affinity to the solvent move faster

What does the stationary phase do?

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Holds back components of the mixture that are attracted to it.

What is the relationship between a sample and the stationary phase that makes the sample move slower? What kind of bonding does this often involve?

What is the relationship between a sample and the stationary phase that make the sample move slower? What kind of bonding does this often involve?

More affinity for the stationary phase means that a component moves slower; often attracted by hydrogen bonding

How are substances separated by chromatography?

How are substances separated by chromatography?

If suitable stationary/mobile phases are chosen, the balance between affinity for the mobile phase and affinity for the stationary phase is different for each component of the mixture. Thus, they move at different rates and are separated over time.

Why will different substances show different R_f values?

Why will different substances show different R_f values?

They are bonded differently and have different polarities - more polar bonds mean longer retention time or smaller R_f value, since hydrogen bonding/dipoles are attracted more strongly to the stationary phase

What does TLC stand for?

What does TLC stand for?

Thin Layer Chromatography

What is the stationary phase in TLC?

What is the stationary phase in TLC?

Plastic/glass/metal sheet or “plate” coated in silica (SiO_2) or alumina (Al_2O_3)

What are the advantages of TLC over paper chromatography?

What are the advantages of TLC over paper chromatography?

Runs faster

Smaller amounts of a mixture can be separated

TLC plates are more robust that paper

How can you observe colourless spots?

How can you observe colourless spots?

Shine UV light on them.

Or spray with a developing agent (e.g. ninhydrin turns amino acid spots from colourless to purple, so they can be seen) (heating needed with ninhydrin)

How do you calculate the R_f value?

How do you calculate the R_f value?

Measure the distance from the initial line (that the mixture was spotted onto) to the solvent front, and the distance from the initial line to the spot.

Calculate R_f using: Rf = distance moved by spot ÷ distance moved by solvent front

What does R_f value stand for?

What does R_f value stand for?

Retention factor; a measure of the rate of movement of a component through the chromatography apparatus; a ratio between the rate of movement of the solvent and that component

How could you confirm the identity of a substance from its R_f value?

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Compare your R_f value to accepted values R_f for that substance run in the same solvent and set-up; if they match, then identity is confirmed

What is column chromatography?

What is column chromatography?

Column packed with silica, alumina or resin has solvent run through it downwards

What is the stationary phase in column chromatography?

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Silica, alumina or resin packed into a column

What is the mobile phase in column chromatography? What is it also known as?

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Solvent added at the top and runs down the column; called “eluent”

Draw a diagram of column chromatography

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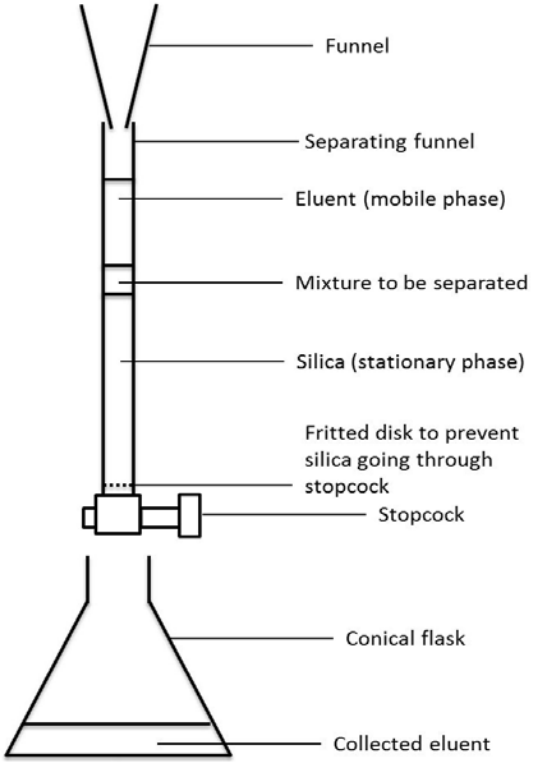

What are the advantages of column chromatography?

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More than one eluent can be used, which leads to better separation

Fairly large amounts can be separated and collected after separation

Draw a diagram for gas-liquid chromatography

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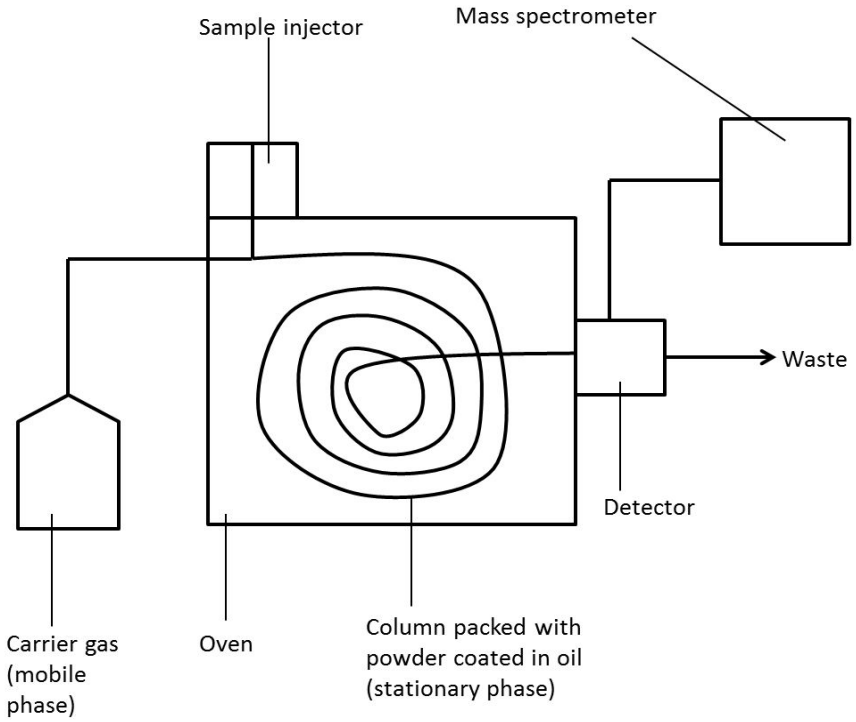

What is the stationary phase in gas-liquid chromatography?

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Powder, coated with oil. Packed into a long, thin, capillary tube (100m long, 0.5mm diameter). Coiled and placed in an oven, the temperature of which can be varied

What is the mobile phase in gas-liquid chromatography?

What is the mobile phase in gas-liquid chromatography?

Carrier gas, inert e.g. N_2 or He

What do you measure in gas-liquid chromatography?

What do you measure in gas-liquid chromatography?

Retention time; different components of the mixture take different amounts of time to move through

What are the advantages of GLC?

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Very sensitive; GC can detect minute traces of substances in foodstuffs, and link oil pollution on beaches to the specific tanker the oil came from

What are GLC's uses?

What are GLC's uses?

Test athletes' and horses' blood and urine for drugs

How can you use GC or GCMS to identify substances?

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Match Gas Chromatograph to that of a known substance under the same conditions; retention time should exactly match. Substance's identity can be confirmed by mass spectrometry, NMR or infrared spectroscopy.

How does GCMS work?

How does GCMS work?

Gas Chromatography is run, retention time is recorded, then mixture is run through a Mass Spectrometer. Fragmentation pattern/molecular ion peak confirms identity.

Will an alcohol or an aldehyde have a shortest retention time by column chromatography?

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Aldehyde has shortest retention time, since it has a less polar bond than an alcohol. It therefore adsorbs less strongly to the stationary phase, so moves down the column at a quicker rate. Force of attraction between stationary phase and aldehyde is less

Complete this question

A sample of the element barium is made up of four isotopes. The data below were taken from a mass spectrum of this sample.

Mass/charge ratio	% abundance
135	9.01
136	10.81
137	12.32
138	67.86

Calculate the relative atomic mass of the sample, giving your answer to **one** decimal place.

(2)

Answer

% abundance = (135 x 9.01 + 136 x 10.81 + 137 x 12.32 + 138 x 67.86) / 100 **(1)**

= 137.4 **(1)**

ignore units

Allow TE for one slip in transfer of data from question

Correct answer scores **(2)**

What is the symbol of molecular ion?

What is the symbol of molecular ion?

M^+

The molecular mass of the molecular ion is equal to what?

The molecular mass of the molecular ions is equal to what?

Relative molecular mass of the compound

What is the m/z value of CH_3^+?

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15

What is the m/z value of OH^- from alcohol?

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17

What is the m/z value of $C_2H_5^+$?

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