

CAIE Chemistry A-level

37: Analytical Techniques

(A-level only)

Definitions

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Definitions and Concepts for CAIE Chemistry A-level Analytical Techniques

Baseline: The pencil line where the samples are placed at the start in paper chromatography. The R_f values should be measured from the baseline to the sample and the solvent front.

Carbon-13 NMR spectroscopy: A type of NMR spectroscopy which analyses ^{13}C nuclei. The number of peaks in the spectrum shows the number of different environments and the chemical shifts represent the environments present.

Chemical shift: The shift of a carbon or proton environment relative to the standard tetramethylsilane (TMS). This chemical shift value (δ) depends on the molecular environment of the proton/carbon atom.

Chromatography: A technique used to separate and identify components in a mixture. There are several different types of chromatography, including thin-layer chromatography and gas chromatography. Separation during chromatography depends on the balance between each compound's solubility in the mobile phase and retention by the stationary phase.

Coupling: The interaction between adjacent non-equivalent protons during NMR spectroscopy.

Deuterated solvent: A solvent, such as CDCl_3 , in which all of the hydrogen atoms have been replaced by deuterium (^2H). Deuterated solvents are used so there is no solvent peak on the ^1H NMR spectrum.

Doublet: A signal on a ^1H NMR spectra made up of two peaks, indicating that there is one adjacent non-equivalent proton.

Elemental analysis: A sample is analysed to determine the proportion of elements that make up the compound present. This is done by converting a known amount of an unknown sample into simple known compounds.

Environment: In NMR spectroscopy, a proton or carbon environment considers the atoms/groups that are adjacent to that hydrogen/carbon atom. If two hydrogen/carbon atoms are surrounded by different groups of atoms, they are in different environments.

Equivalent protons: Protons that are in the same environment.



Gas chromatography (GC): A type of chromatography in which a column is packed with a solid (or a solid coated in a liquid) and gas is passed through the column under pressure at high temperature. Mass spectrometry may then be used to analyse the components separated by GC.

Mobile phase: A fluid that moves through a chromatography system to separate a mixture. This may be a gas (GC) or a liquid (TLC). The different components of the mixture have different affinities to the mobile phase, causing them to be separated.

Nuclear magnetic resonance (NMR): A technique that uses the absorption of electromagnetic radiation by an atomic nucleus in an external magnetic field to analyse the structure of a compound. Typically, either ^{13}C or ^1H nuclei are analysed. ^{13}C NMR spectra are generally simpler than ^1H NMR spectra.

N+1 rule: If there are n equivalent protons on the carbon atom adjacent to the one to which the given proton is attached, the multiplicity would be n+1.

Proton NMR spectroscopy: A type of NMR spectroscopy which analyses ^1H nuclei. The number of peaks on the spectrum shows the number of proton environments and the chemical shifts represent the environments. The area under each peak shows the relative number of protons in each environment. The splitting pattern can be used to work out the number of adjacent protons.

Quartet: A signal on a ^1H NMR spectra made up of four peaks, indicating that there are three adjacent non-equivalent protons.

Retention time: The time taken for a sample to travel through the GC column. The recorded retention time can be compared with standards to identify the substances in the mixture.

R_f value: In TLC, R_f values can be calculated as shown below then compared with standards to identify the substances present in a mixture.

$$R_f = \text{Distance moved by substance} \div \text{Distance moved by solvent}$$

Solvent front: In TLC the solvent front is the distance the solvent moves up the chromatographic plate from the baseline.

Splitting pattern: Protons are influenced by other protons that are bonded to adjacent carbons, this causes peaks to split. If there are no adjacent protons the peak does not split and forms a singlet, if there is one adjacent proton the peak forms a doublet and if there are two adjacent protons the peak forms a triplet ect. The splitting follows the n+1 rule.





Stationary phase: A substance through which the mobile phase moves in chromatography. This is typically a solid, although a solid coated in a liquid may be used in gas chromatography (GC).

Tetramethylsilane (TMS): The standard for chemical shift measurement in NMR spectroscopy.

Thin Layer Chromatography (TLC): A technique used to separate mixtures. The stationary phase is a thin layer of alumina or silica fixed to a metal or glass plate. The plate is dotted with the mixture and placed in a beaker of solvent which is allowed to travel up the plate. The mixture separates due to the components having different solubilities in the mobile phase.

Triplet: A signal on a ^1H NMR spectra made up of three peaks, indicating that there are two adjacent non-equivalent protons.

