

Analytical Techniques

A-Level Chemistry • Topic 37

A-Level Chemistry



Analytical Techniques

What we will cover

- 1 Thin-layer chromatography
- 2 Gas/liquid chromatography
- 3 NMR basics
- 4 Splitting & the $n + 1$ rule
- 5 A proton NMR spectrum



NMR uses a strong magnet

1 Chromatography

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Chromatography

Thin-layer chromatography

The **baseline** 基线 is the starting line where the spots are placed; the **solvent front** 溶剂前沿 is the highest level the solvent reaches. The **mobile phase** 流动相 is the solvent that travels up the plate, the **stationary phase** 固定相 the coating it travels through.

A spot of mixture travels up a plate. Components that adsorb more strongly to the stationary phase move less.

$$R_f = \frac{\text{distance of the spot}}{\text{distance of the solvent front}}$$

R_f is always between 0 and 1. Identify a component by matching R_f with a known standard on the same plate.

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Gas/liquid chromatography

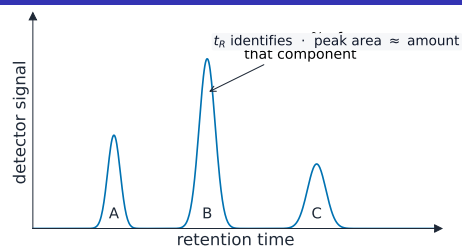
In **GLC** 气液色谱 a carrier gas sweeps the mixture over a liquid stationary phase.

Each component peaks at its own **retention time** 保留时间; the peak **area** gives its percentage in the mixture.

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Gas/liquid chromatography, in detail



In **GLC** 气液色谱 a carrier gas sweeps the mixture over a liquid stationary phase.

2 NMR

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└ NMR

NMR basics

carbon-13 NMR

each carbon **environ-ment** 化学环境 gives one peak

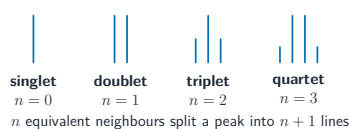
proton NMR

chemical shift 化学位移 & peak areas give the proton ratio

NMR 核磁共振 studies nuclei in a strong magnetic field; the number of peaks = the number of environments.

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Splitting and the $n + 1$ rule



Splitting 裂分: n equivalent neighbouring protons split a peak into $n + 1$ lines.

- 0 → **singlet** 单峰; 1 → doublet
- 2 → triplet; 3 → quartet

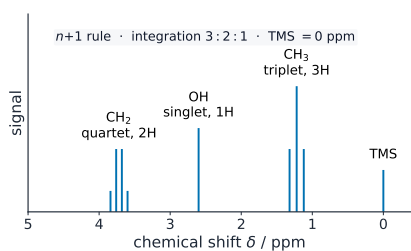
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A proton NMR spectrum

Ethanol: a CH_3 triplet, a CH_2 quartet & an OH singlet (areas 3:2:1).

- **TMS** 四甲基硅烷 is the zero reference
- a **deuterated solvent** 氘代溶剂 gives no signal
- D_2O removes the O–H/N–H peak

A proton NMR spectrum, in detail



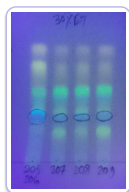
Ethanol: a CH_3 triplet, a CH_2 quartet & an OH singlet (areas 3:2:1).

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└ NMR

Practical points

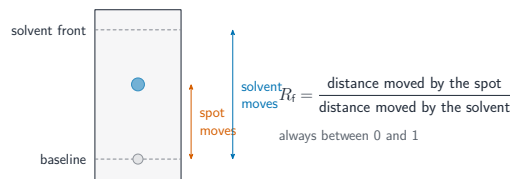
- **tetramethylsilane** 四甲基硅烷 (TMS) is the standard, set at a chemical shift of 0.
- a **deuterated solvent** 氘代溶剂 (such as CDCl_3) is used so that the solvent itself gives no proton signal.
- shaking the sample with D_2O makes the O–H and N–H peaks disappear (their hydrogen is swapped for deuterium), which identifies those protons.

Thin-layer chromatography, continued



A real TLC plate under UV light: each glowing spot is a separated component of the mixture

Thin-layer chromatography, continued (2)



Thin-layer chromatography: the R_f value is how far the spot moved divided by how far the solvent front moved

Exam tips

- In ^{13}C NMR the number of peaks equals the number of carbon environments – use symmetry to count them.
- In ^1H NMR use chemical shift (data booklet), integration (ratio of H's) and splitting (the $n + 1$ rule): a triplet + quartet means an ethyl group.
- TMS is the reference ($\delta = 0$); a D_2O shake removes O-H and N-H peaks.
- Combine IR, mass spectrum and NMR to deduce a structure, stating which evidence gives which feature.

3

Recap

Worked example – reading an NMR spectrum

$\text{C}_3\text{H}_8\text{O}$ gives three ^1H peaks: 6H doublet, 1H septet, 1H singlet that vanishes with D_2O . Identify it.

- Three peaks $\Rightarrow$ three proton environments; the areas 6 : 1 : 1 give the H in each.
- The singlet that vanishes with D_2O is an O-H (it exchanges, and does not split).
- 6H as a doublet: two equivalent CH_3 next to one H ($n + 1 = 2$).
- 1H as a septet: that H has six neighbours ($n + 1 = 7$).
- $(\text{CH}_3)_2\text{CH} - \text{OH}$: propan-2-ol.

Peaks = environments; area = how many H; splitting = $n + 1$ neighbours. A peak lost with D_2O is O-H or N-H.

Topic 37 – key takeaways

1 TLC

R_f = spot distance / solvent distance

2 GLC

retention time; peak area = percentage

3 NMR

peaks = chemical environments

4 Splitting

n neighbours give $n + 1$ lines

Check yourself

- 1 What does the R_f value compare?
- 2 What does a GLC peak area tell you?
- 3 What does the number of NMR peaks tell you?
- 4 Three neighbouring protons split a peak into how many lines?



Answers 1. spot distance vs solvent distance 2. the percentage of that component
3. the number of carbon environments 4. four (a quartet)