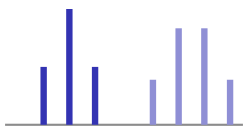
Analytical Techniques

A-Level Chemistry ▪ Topic 37

A-Level Chemistry



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

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.

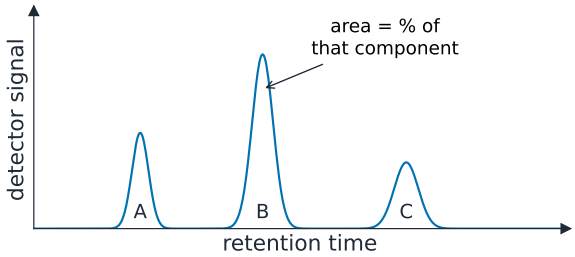
$$R_f = \frac{\text{distance of the spot}}{\text{distance of the solvent front}}$$

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

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.



t_R identifies · peak area ≈ amount

2 NMR

NMR basics

carbon-13 NMR

each carbon **environment** 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.

Splitting and the $n + 1$ rule

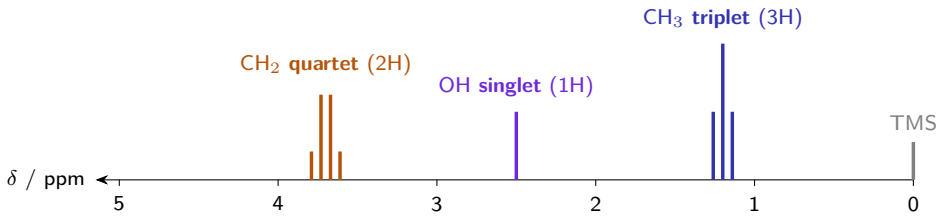
singlet $n = 0$ **doublet** $n = 1$ **triplet** $n = 2$ **quartet** $n = 3$

n equivalent neighbours split a peak into $n + 1$ lines

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

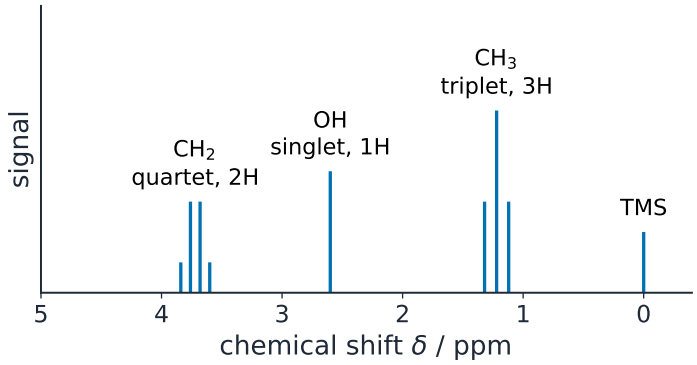
- 0 → **singlet** 单峰; 1 → doublet
- 2 → triplet; 3 → quartet

A proton NMR spectrum



Ethanol: peak **areas** $3{:}2{:}1$ count the H; a peak splits into $n{+}1$ by its **neighbouring** H.

A proton NMR spectrum, in detail



n+1 rule · integration 3 : 2 : 1 · TMS = 0 ppm

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

Practical points

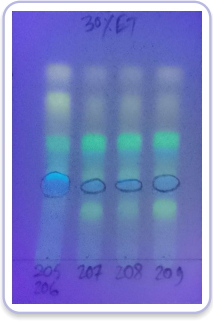
TMS 四甲基硅烷
the standard,
set at $\delta = 0$

deuterated solvent 氘代溶剂
e.g. $CDCl_3$: the solvent gives **no** proton signal

shake with D_2O
O−H and N−H peaks **disappear**

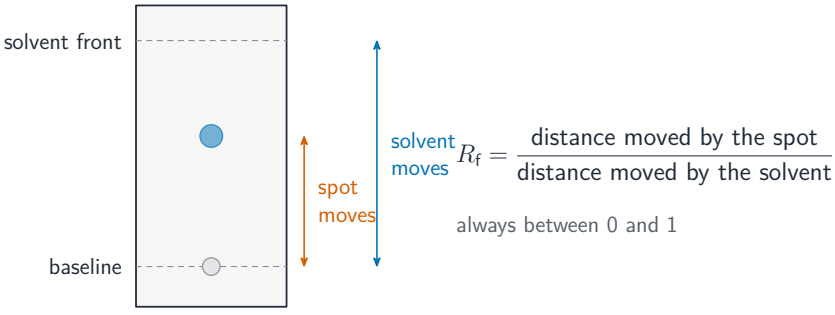
D_2O swaps the O−H/N−H hydrogen for deuterium, which gives no signal: the vanished peak **identifies** those protons.

A TLC plate under UV light



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 ^{1}**H 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

C_3H_8O 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 ($n+1=7$).
- $(CH_3)_2CH-OH$: 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

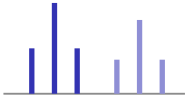
peaks = chemical environments

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)