Analytical Techniques

A-Level Chemistry ▪ Topic 37

A-Level Chemistry

What we will cover

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

NMR uses a strong magnet

1 Chromatography

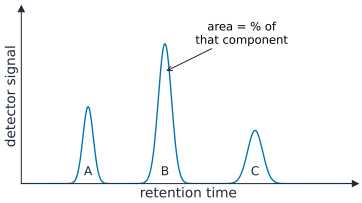
Thin-layer chromatography

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.

2 NMR

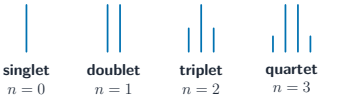
NMR basics

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



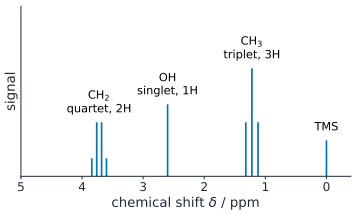
singlet $n=0$ | **doublet** $n=1$ | **triplet** $n=2$ | **quartet** $n=3$

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

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

- $0 \rightarrow$ **singlet** 单峰; $1 \rightarrow$ doublet
- $2 \rightarrow$ triplet; $3 \rightarrow$ quartet

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

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 ⇒ 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 that disappears 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)