Analytical Techniques Map

Cambridge International AS & A Level Chemistry · Topic 37 Analytical techniques

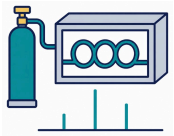
1 Thin-layer chromatography 37.1

- Stationary: **silica** or alumina
- Mobile: the **solvent**
- **Rf** = spot ÷ solvent front
- Polar spots held back: **low Rf**



2 Gas chromatography 37.2

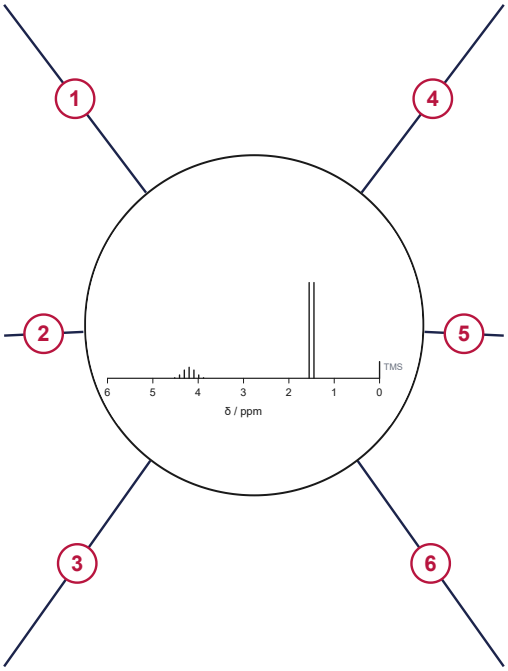
- Unreactive carrier gas; liquid on a solid
- **Retention time** identifies
- More interaction: **held longer**
- **Peak area** ÷ total: % composition



3 ^{13}C NMR 37.3

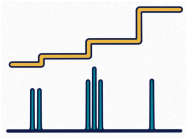
- One peak per **carbon environment**
- **Symmetry**: equivalent carbons share a peak
- δ from TMS (0 ppm)
- C=O 160–220; C–O 50–90





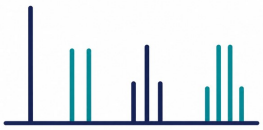
4 ^{1}H shifts and areas 37.4

- Peaks: **proton environments**
- δ: what the H is near (O, C=O, ring)
- **Peak areas**: ratio of H
- Solvent $CDCl_3$; TMS at δ = 0



5 Splitting 37.4

- **n + 1 rule**: n H on adjacent C
- Singlet, doublet, triplet, quartet
- CH_2CH_3: quartet + triplet
- Equivalent H do not split



6 D_2O exchange 37.4

- Shake the sample with **D_2O**
- O–H and N–H swap H for **D**
- Their peak **vanishes**
- Identifies the O–H or N–H peak



Which technique tells you what

use them together: each answers a different question

Technique	What you read	What it tells you
TLC	Rf	compare with a standard; purity
Gas chromatography	retention time, peak area	identity and percentage composition
^{13}C NMR	number of peaks, δ	carbon environments, C=O or C–O
^{1}H NMR	peaks, δ, areas	proton environments and how many H
^{1}H NMR splitting	n + 1 lines	H on the adjacent carbon
D_2O shake	a peak vanishes	which peak is O–H or N–H

Worked examples

Rf value

- spot 2.7 cm; front 6.0 cm
- 2.7 ÷ 6.0
- **Rf = 0.45**

Percentage composition

- areas 15, 45, 40
- total 100
- **45% of B**

Read the splitting

- CH_3CHCl_2
- CH_3: doublet; CH: quartet
- **areas 3 : 1**

Mass spectra are in Topic 22; NMR and chromatography return in Topic 36 to confirm a synthesis.

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