UNIT 13 – ANALYTICAL METHODS

OCSL: Chapter 1 & Chapter 2

VTOC: Spectroscopy
- Mass Spectrometry
- Ultraviolet-Visible Spectroscopy
- Infrared Spectroscopy
- Nuclear Magnetic Resonance Spectroscopy

UCalg: **Spectroscopy** Theoretical Background

Spectroscopic methods
- Infra red (IR)
- Nuclear magnetic Resonance (NMR)
- Ultra-violet / visible (UV-VIS)
- Mass Spectrometry (MS)
- Getting structures from spectra
- H NMR sketching problems
- Spectra Problems
- Interactive Spectroscopy Problems

UCDavis:
- Section 4.1: Introduction to molecular spectroscopy
- Section 4.2: Infrared spectroscopy
- Section 4.3: Ultraviolet and visible spectroscopy
- Section 4.4: Mass Spectrometry
- Section 4.5: Problems for Chapter 4
- Section 5.1: The origin of the NMR signal
- Section 5.2: Chemical equivalence
- Section 5.3: The NMR experiment
- Section 5.4: The basis for differences in chemical shift
- Section 5.5: Spin-spin coupling
- Section 5.6: ^{13}C-NMR spectroscopy
- Section 5.7 : Determining unknown structures
- Section 5.P: Problems for Chapter 5

I will not be testing on differences in diastereotopic hydrogens. You may come across them in the websites.

Skills:

13A. Know the relationship between wavelength, frequency, wavenumber, and energy of light.

13B. Know the relationship between extent of conjugation and peak wavelength absorbance, HOMO-LUMO energy difference and color of organic molecules.

13C. Given a MS, identify and know the significance of the base peak, M+, and M+1 peak. Be able to distinguish between spectra containing chlorine, bromine or neither based on presence of and relative ratio of the M+2 peak

13D. Given an IR spectrum and table of absorptions, identify the functional group of a molecule.

13E. Given a structure predict the number of ^{1}H and ^{13}C peaks in its NMR spectrum.

13F. Given a table predict the approximate chemical shift of different ^{1}H and ^{13}C peaks.

13G. Predict integration values of ^{1}H spectra.

13H. Predict splitting patterns of protons in a structure.

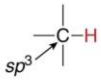
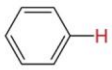
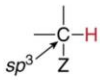
13I. Use number of peaks, chemical shift, integration and splitting data to match a structure to its NMR spectrum

13J. Know the basic workings of UV-VIS, mass spec, HRMS, GCMS, IR, and NMR and what information can be gained from them.

Table 13.2 Important IR Absorptions

Bond type	Approximate $\tilde{\nu}$ (cm^{-1})	Intensity
O–H	3600–3200	strong, broad
N–H	3500–3200	medium
C–H	~3000	
• C_{sp^3}–H	3000–2850	strong
• C_{sp^2}–H	3150–3000	medium
• C_{sp}–H	3300	medium
C≡C	2250	medium
C≡N	2250	medium
C=O	1800–1650 (often ~1700)	strong
C=C	1650	medium
	1600, 1500	medium

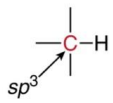
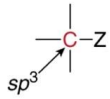
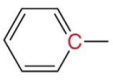
Table 14.1 Characteristic Chemical Shifts of Common Types of Protons

Type of proton	Chemical shift (ppm)	Type of proton	Chemical shift (ppm)
	0.9–2		4.5–6
• RCH_3 • R_2CH_2 • R_3CH	~0.9 ~1.3 ~1.7		6.5–8
	1.5–2.5		9–10
—C≡C–H	~2.5		10–12
	2.5–4	RO–H or R–N–H	1–5

Z = C, O, N

Z = N, O, X

Table 14.5 Common ^{13}C Chemical Shift Values

Type of carbon	Chemical shift (ppm)	Type of carbon	Chemical shift (ppm)
	5–45		100–140
	30–80		120–150
—C≡C—	65–100		160–210

Z = N, O, X