

References

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 D. H. Williams, I. Fleming, *Spectroscopic Methods in Organic Chemistry*, McGraw Hill, London, UK.
 E. Breitmaier, W. Voelter, *Carbon 13 NMR Spectroscopy*, 3rd ed., VCH, New York, NY, 1987.
 K. Wüthrich, *NMR of Proteins and Nucleic Acids.*, Wiley, New York, N. Y., 1986.

Computer Programs for Help in Spectral Interpretation

Name	Type of Program	Host Application	Spectroscopy covered
<u>ChemDraw</u> : NMR spectrum simulation, Rule based spectrum prediction, first order multiplets			
ChemDraw	CAI	PC, Mac	H, ¹³ C-NMR
<u>NMR Spectrum Simulator</u> : Rule based spectrum prediction, first order multiplets			
Spectrum Simulator	CAI	Mac	H-NMR
<u>¹H Chemical Shift Prediction</u> : Rule based spectrum prediction			
H1pred.html	Simulation	web	H-NMR
<u>Upsol Assemble</u> : Automatically generates all possible molecular structures (www.upstream.ch)			
Assemble2	Structure generator	PC	Any
<u>IRHelper</u> : Spectral Interpretation guidance			
IRHelper.html	CAI	web	IR
<u>IR Tutor</u> : IR tutorial, Spectral interpretation examples, animated normal modes			
IRtutor	CAI	PC, Mac	IR
<u>SpectraDeck</u> : Interpreted Spectral Database			
SpectraDeck	CAI	Mac HyperCard Stack	H-NMR, C-NMR, IR, MS
<u>MS Interpreter</u> : NBS Mass Spectral Interpretation, fragment prediction			
MS Interpreter	Simulation, Helper	Varian Saturn PC	MS
<u>TIMS</u> : Mass Spectral Interpreter, fragmentation prediction, spectral comparison			
TIMS	Simulation	Mac	H-NMR
<u>First Order Multiplet Maker</u> : First Order Multiplet prediction			
Jmm.html	Simulation	web	H-NMR
<u>Sweet J</u> : J coupling calculator, Karplus relations			
Sweet J ppc	Helper	Mac	H-NMR
<u>Spin-Spin Splitting Simulation</u> : Second order multiplets			
JD.html	Simulation	course web site	NMR
<u>Fragment Finder</u> : Formulas from molar mass			
Fragment.html	Helper	web	MS
<u>Formula Finder</u> : Formulas from molar mass			
Formula.html	Helper	web	MS
<u>Distance Geometry and MM2</u> : Molecular Modeling Program			
Dgmm2.html	Molecular Modeling	web	H-NMR J _{AB}
<u>MOE</u> : Molecular Modeling Program			
MOE	Molecular Modeling	Schupf PC, Mac	H-NMR J _{AB}

CAI= computer aided instruction

Mac= Macs in Keyes 203 and filerserver1: /Chemistry/CH255J

Web= NMR course Web page

Grading

900-1000 A	800-899 B	700-799 C	600-699 D
<u>unknown 1</u>	40		
<u>unknown 2</u>	60		
<u>unknown 3</u>	100		
<u>drivers license</u> (See below)	300	^1H , ^{13}C , probe tuning, 90° pulse determination, SW set, DEPT	
<u>water suppression</u>	50	Watergate on glucose	
<u>molecular modeling</u>	40	WF pp. 96-101 pulmericin, Fig. 3.29	
<u>MO chemical shift calculation</u>	40	Spartan on camphor or β -ionone using GIAO approach	
<u>analysis of a complex spin-spin pattern</u>	60	JD.html or nmrsim	
<u>matched filter and sine-bell apodization</u>	40	data work up	
(try different BF's on a noisy spectrum. Find the BF for a "matched filter" and try sine-bell apodization.)			
<u>T₁ determination and partially relaxed spectra</u>	90		
<u>HMOC</u>	40		
<u>HMBC</u>	50		
<u>phase sensitive COSY</u>	80	(double quantum filtered)	
<u>1D-gradient NOE</u>	70	(camphor, beta-ionone or one of your unknowns)	
<u>1D-gradient TOCSY</u>	70		
<u>NOESY</u>	120	camphor	
<u>TOCSY</u>	120	camphor, limonene, or betulin	
<u>HMOC-TOCSY</u>	120	limonene or betulin	
<u>GC/MS</u>	40		
<u>Chromatoprobe MS</u>	60	on solid unknown or candy	
<u>direct infusion MS and MS/MS</u>	100	unknown or transition metal complex	
<u>LC/MS</u>	100	on unknown or tea infusion	
<u>Mirror rate optimization in FT-IR</u>	60	choose 4 cm^{-1} and 8 cm^{-1} resolution and optimize mirror rate	
<u>KBr pellet IR sample prep</u>	30		
<u>CHN analysis</u>	50		

• some of the above techniques are combined in the projects below:

unknown monosacharride	200		
unknown dipeptide	300		
assign resonances in N-[2-(dimethylamino)ethyl]-N-methylguanidinium			100
+ guest-host complex NOESY			250
^{31}P of phosphate complex with N-[2-(dimethylamino)ethyl]-N-methylguanidinium			250
assign resonances in cimetidine	250		
assign resonances in pinene	300		
assign resonances in dipeptide from NutraSweet	300		
assign resonances in catechin, ^1H and ^{13}C	400		
assign resonances in betulin (from birch bark)	500		

Quizzes and homework will also add to the point total

Bruker Avance 400 MHz NMR
License Activities

Take a 1D Proton
Manual shim
Tune the proton probe
Narrow SW and set O1
Retake 1D Proton
Find proton high-power 90° pulse
Do a DEPT135

You can work on an unknown or just the ethylbenzene or menthol sample