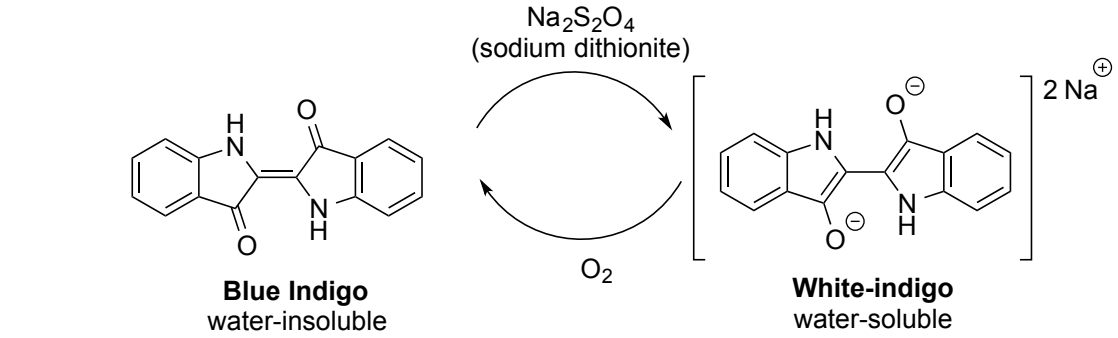
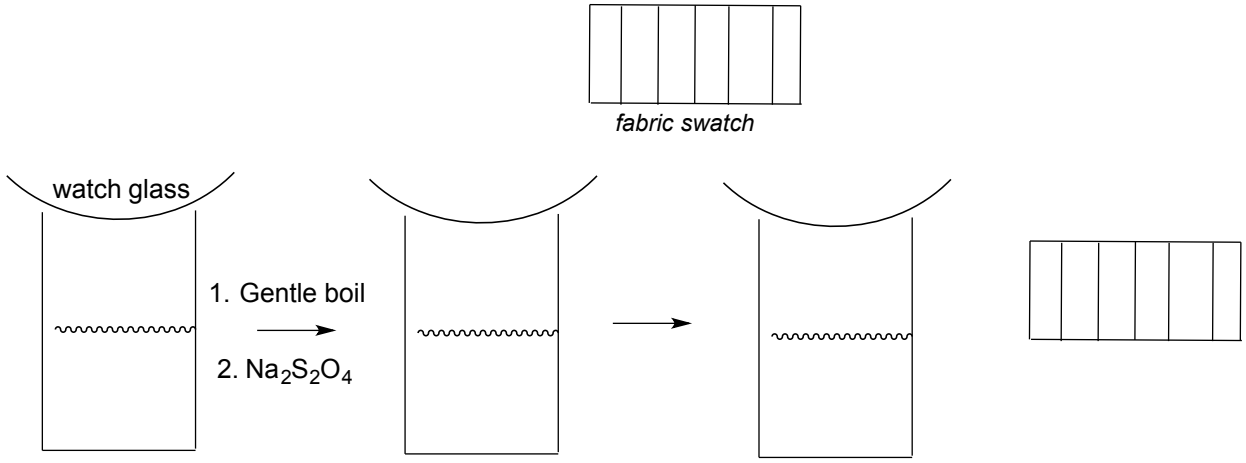
CHEM 8M, Lecture 7
Exp 4, Day 2 – Synthesis of & Vat Dyeing with Indigo
Prep for NMR Problem Set – ^{1}H NMR Splitting

Part D.2. Vat Dyeing with Indigo





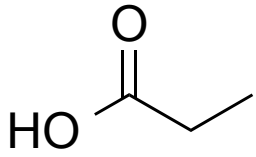
^{1}H NMR = "Proton NMR"

Relate the terms below to propanoic acid…

- Chemical Equivalence

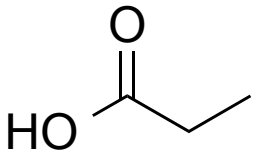
- Integration

- Chemical Shifts



Splitting aka ^{1}H-^{1}H Coupling and the "n+1 Rule"

Non-equivalent signals (neighboring proton nuclei) effect each other's peak shape.



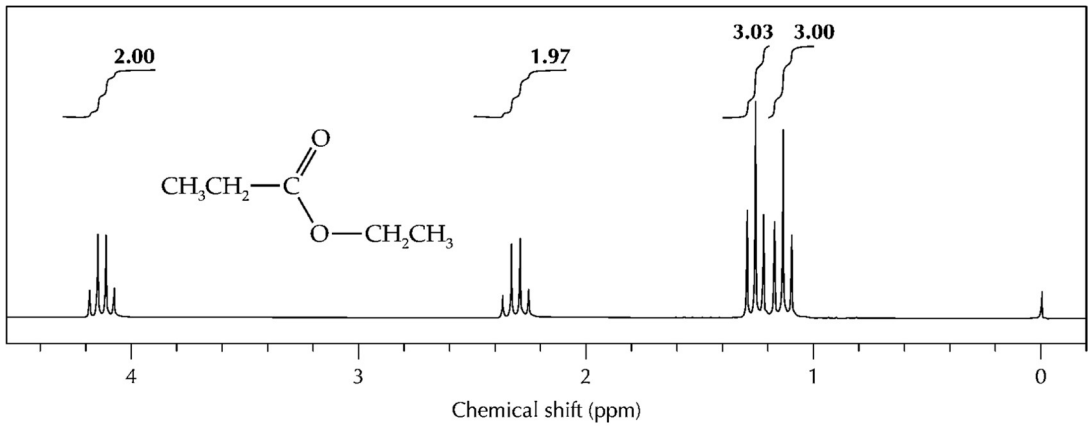
The "n+1 rule" and splitting patterns

n (# of H neighbors)	**n+1** (splitting or multiplicity)	**Splitting Pattern**
0	1	Singlet
1	2	Doublet
2	3	Triplet
3	4	Quartet
4	5	Pentet
5	6	Sextet
6	7	Septet

^{1}H spectrum of **ethyl propanoate** has two **2H quartets** (**B**&**C**) and two **3H triplets** (**A**&**D**).

- Signal **D** is a **3H triplet** at **1.3 ppm**: "δ 1.1 (3H triplet)"
  - All **D** nuclei spin *against B field* @ **1.3 ppm**
  - **D** has **2** neighbors (signal **C, n = 2**) that are *50 / 50 with & against B field*
  - The *central* chemical shift of **D** remains the same (1.3 ppm)
  - The random spins of **C** effect the *peak shape* of signal **D**
    - **n = 2; n+1 = 3, triplet**

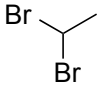
Signal **D** without splitting Signal **D** with splitting

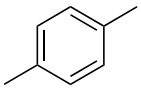


*Explain the splitting pattern of **C,*** "δ 4.1 (2H quartet)"

Preparing for NMR Problem Set

Predict # signals, integration, splitting, and chemical shift (range and calculated)

	Signal	Integration	Splitting	Chemical Shift Range (ppm)	Calculated Chemical Shift (ppm)*
					

	Signal	Integration	Splitting	Chemical Shift Range (ppm)	Calculated Chemical Shift (ppm)*
					

Structural Elucidation - Propose a structure from chemical formula, IR, & ^{1}H NMR

*Process: calculate degrees of unsaturation, use IR to identify possible functional group(s), draw structure fragments based on signal information (2H doublet = C**H$_2$**CH), then put the fragments together in the final structure. Check your work by calculating chemical shifts of the proposed structure.*

C_3H_6O; **IR** 1720 cm^{-1}, 2900 cm^{-1}; **^{1}H NMR** – δ 2.0 ppm (3H singlet)

$C_5H_{12}O$; **IR** 3300 cm^{-1}, 2900 cm^{-1}; **^{1}H NMR** – δ 4.0 ppm (1H broad singlet), δ 3.5 ppm (2H triplet), δ 1.6 (1H nonet), δ 1.5 ppm (2H quartet), δ 0.9 (6H doublet)

Next week in lab: Indigo Synthesis, Work on Experimental Methods & NMR Problem Set

Next week in lecture: More ^{1}H NMR Analysis