Name _____ Partner Name _____

TA Name _____ Section Letter _____ Day _____ Time _____

Experiment 6 Worksheet – Diels-Alder [4+2] Cycloaddition Reaction in Water

Use as reference for notebook preparation – submit on Canvas this individually after lab

A. Experimental Purpose and Cycloaddition Reaction Scheme

B. Reagent Table

Refer to the procedure for amounts and safety table for hazards; find the chemical properties on Wikipedia!

Name	Volume	Density	Mass	MW	mmol	Equiv*	Boiling / melting point	Hazards
9-anthracenemethanol	-	-						
N-ethylmaleimide (NEM)	-	-						
Water								
Cycloaddition Product						-		
Toluene						-		
Diethyl ether					-	-		

*** Equiv** = molar equivalents of reaction components with respect to the limiting reagent

- reagent equivalents: divide the mmol of reagent by the mmol of citrals

- solvent: divide the mmol of limiting reagent by the volume of water (mL)

C. Procedure Diagrams - on as many pages as needed

- All **labeled equipment, chemical names** with **amounts**, **transfers**, **cleanup & safety** notes
    - Help w diagrams: Slugs@home Exp 6 website & class notes

1. **Reaction setup** – all equipment and chemicals (name, structure, and amount)
2. **Reaction workup** – flow chart / diagrams with all containers labeled and all solution transfers shown
3. **Recrystallization** – all equipment involved, preparation and solution transfers
4. **Analysis** – NMR, IR, and UV-vis sample preparation; sketches of spectra, identifying key signals

D. Partner Agreement / Accountabilibuddy Contract: Both students in the pair get the same lab report grade. There is also the option to submit individual reports – please do what works best for you and your partner. Split up partner assignments in part **(a)** and schedule a time to collaborate after lab in part **(b).**

(a) *Students are encouraged to work on report together during lab. The assignments below indicate who will put together or type the* ***final responses****.*

Name		
Abstract		
In-Lab Questions		

(b) "DO" Date: _____ = when / how you'll meet or exchange work to discuss & proofread, at least 1-2 days before the DUE date

E. Data & Analysis

Mass of 9-anthracenemethanol _____ mg Theoretical yield of adduct _____ mg

Theoretical Yield Calculation:

Miscellaneous notes & observations – ex. Suspected sources of product loss

Empty RBF mass _____ g After rota-vap: mass of RBF & crude product _____g

Crude product mass (actual yield) _____g

Percent Yield = [(actual yield) / (theoretical yield)] x 100% _____% Yield of Adduct

9-anthracenemethanol IR

Functional Group	Bond	Expected Wavenumber Range (cm^{-1})	Observed Wavenumber (cm^{-1})

NEM IR

Functional Group	Bond	Expected Wavenumber Range (cm^{-1})	Observed Wavenumber (cm^{-1})

Product IR

Functional Group	Bond	Expected Wavenumber Range (cm^{-1})	Observed Wavenumber (cm^{-1})

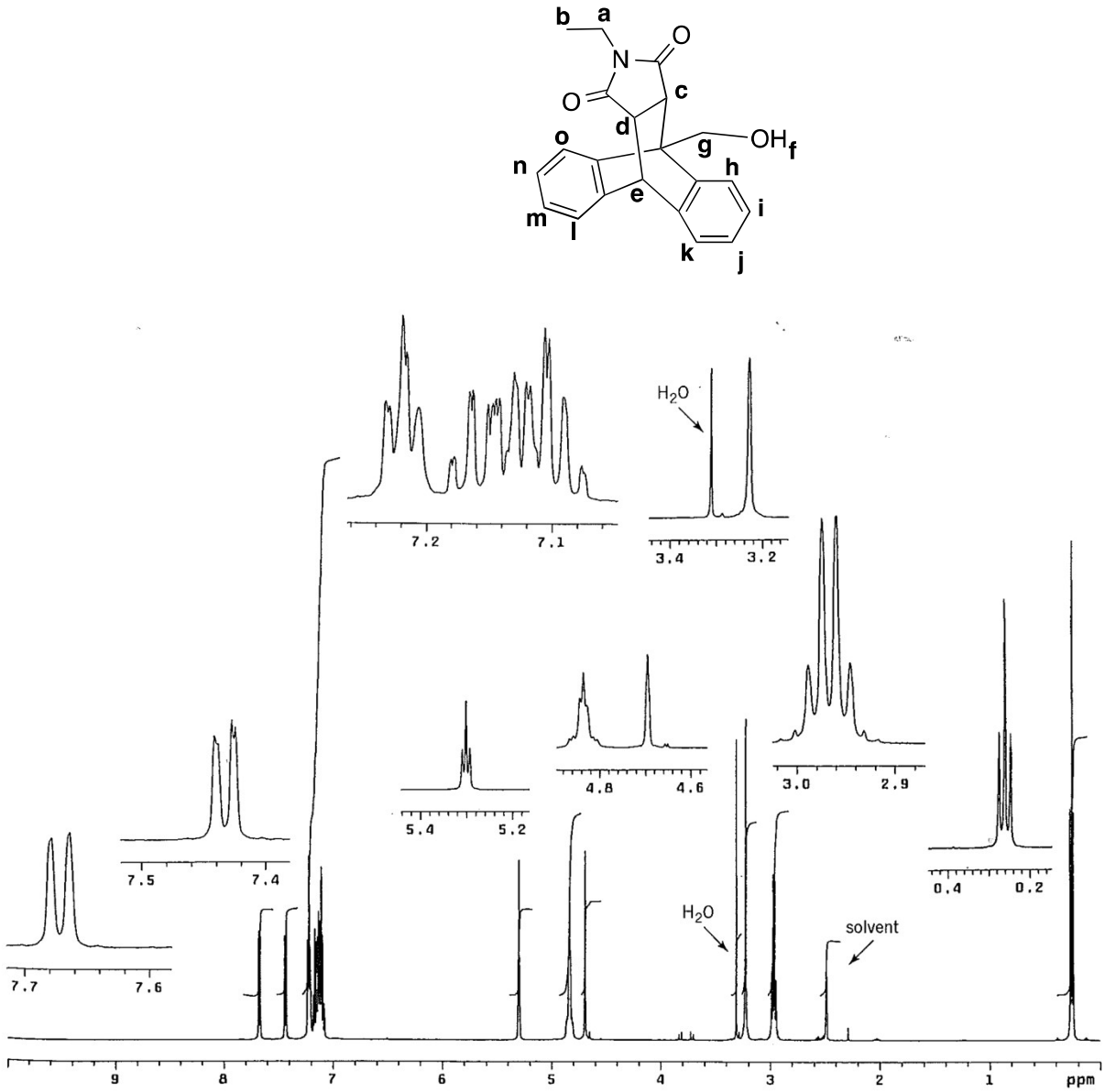
UV-vis Data

9-anthracenemethanol

Expected λ_{max}	Observed λ	Absorbance

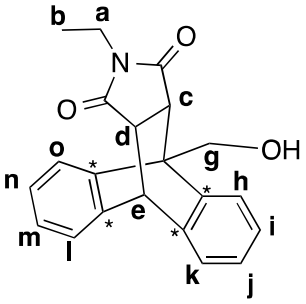
Product

Expected λ_{max}	Observed λ	Absorbance



ure 19.13 500-MHz ^{1}H-NMR spectrum of 9-anthracenemethanol-NEM adduct in DMSO-d_6.

Signal (a-o)	Integration	Splitting	Expected Chemical Shift	Observed Chemical Shift
				0.4
				3.0
				3.2
				4.7
				4.8
				5.3
	5H (overlap)			7.1
				7.2
				7.4
				7.7



Use these designations for unlabeled carbons...
* = aromatic C's without H's
Amide C=O
4° (between c-g)

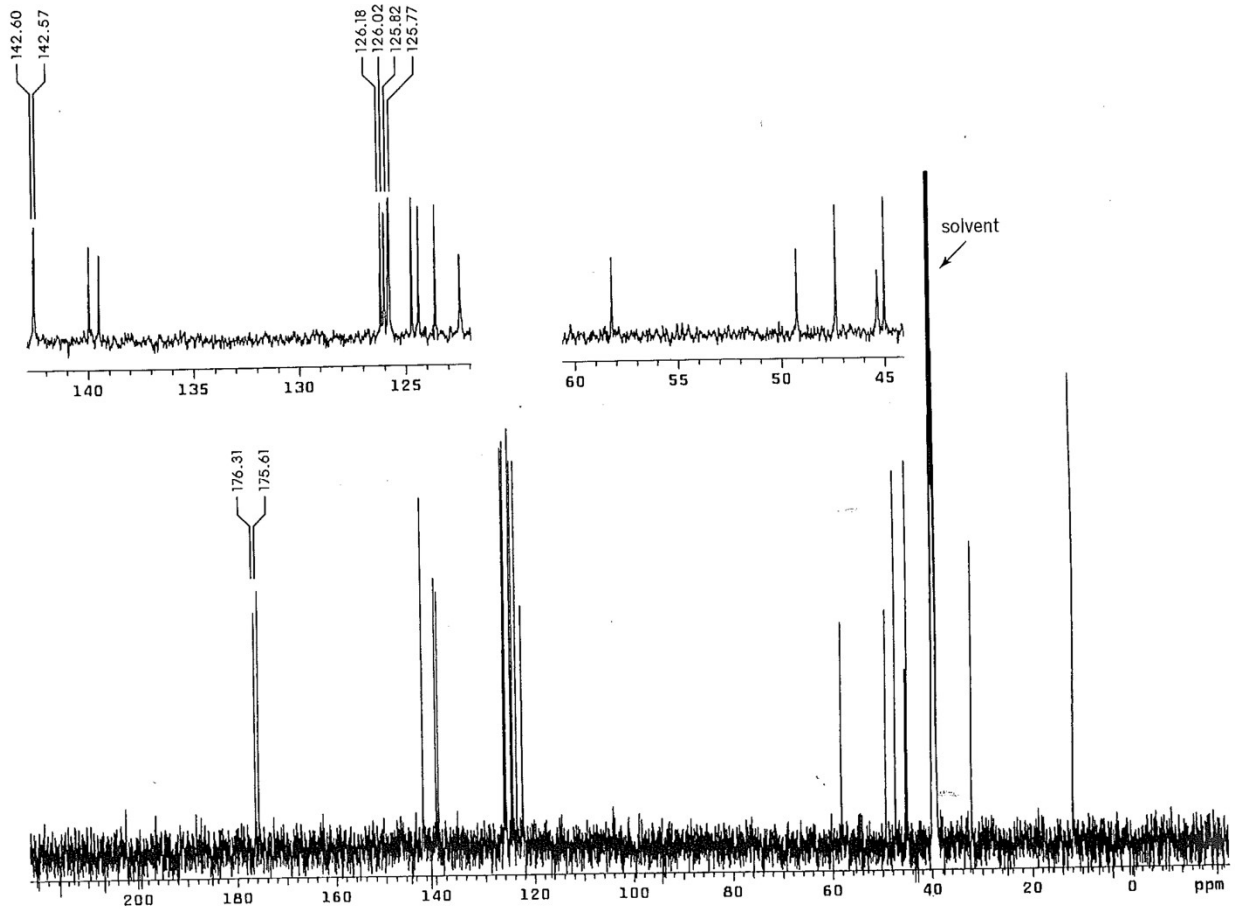


Figure 19.14 125.7-MHz ^{13}C-NMR spectrum of 9-anthracenemethanol-NEM adduct in DMSO-d_6.

Signal	Observed Shift (ppm)	Expected Shift
	10	
	32	
	46	
	45 & 47	
	50	
	57	
	123-125 (4 signals)	
	125-126 (4 signals)	
	139-142 (4 signals)	
	175 & 176	