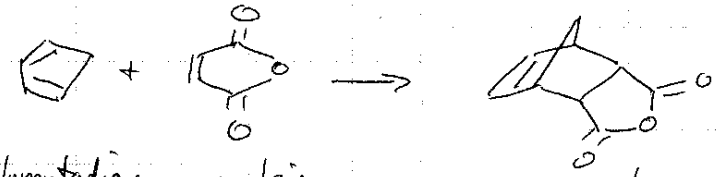
SAMPLE LAB REPORT FOR CHEM 267/268

Use this as a very general guide to follow in keeping your notebook.

EXP. NUMBER	EXPERIMENT/SUBJECT	DATE	1
	DIELS-ALDER SYNTHESIS	1/19/03	
NAME Patti Samal	LOCKER/DESK NO.	COURSE & SECTION NO.	

Synthesis of cis-Norbornene-5,6-endo-dicarboxylic Anhydride



cyclopentadiene + maleic anhydride → cis-Norbornene-5,6-endo-dicarboxylic Anhydride (Diels-Alder Adduct)

REAGENTS

	MW	Dens.	B.P.	M.P.
cyclopentadiene	66.1	0.80	41	—
maleic anhydride	98.1	—	—	53
Diels-Alder product	164.2	—	—	165

QUANTITIES

	mL	g.	moles
cyclopentadiene	0.20	0.16	0.0024
maleic anhydride	—	0.20	0.0020

MALEIC ANHYDRIDE IS LIMITING REAGENT (CYCLOPENTADIENE IS IN EXCESS)

THEORETICAL AMT OF DIELS-ALDER ADDUCT

= 0.0020 mol × 164.2 g/mol = 0.328 g

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PRELAB OUTLINE

REF: WILLIAMSON TEXT pp. 316, 317 AND CHEM 269 HANDOUT.

- MEASURE 0.20g MALEIC ANHYDRIDE INTO REACT. TUBE
- DISSOLVE IN 1mL ETHYL ACETATE THEN ADD 1mL LIGROIN (60-80° B.P.)
- ADD 0.20 mL DRY CYCLOPENTADIENE, MIX WELL,
- ALLOW TUBE TO COOL TO R.T. TO CRYSTALLIZE
- (IF CRYSTALS DO NOT FORM, SCRATCH INSIDE OF TUBE JUST BELOW SURFACE WITH GLASS STIRRING ROD. IF CRYSTALS ARE TOO FINE, REHEAT TO DISSOLVE & ALLOW TO COOL SLOWLY. ADD SEED CRYSTAL IF NECESSARY.)
- REMOVE SOLVENT BY PIPET METHOD, RINSE CRYSTALS WITH COLD LIGROIN, REMOVE SOLVENT.
- SCRAPE CRYSTALS ONTO FILTER PAPER TO DRY
- WEIGH, TAKE M.P.

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		1/9/03	
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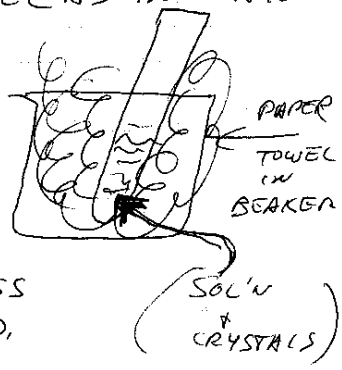
(ALL MATERIAL TO HERE IS DONE BEFORE LAB)

PROCEDURE, OBSERVATIONS, DATA.

- MALEIC ANHYDRIDE

GROSS 0.032g.
TARE 0.012g.
NET 0.020g.

ADD ~~0.020~~ 0.020g. MALEIC ANHYDRIDE TO REACT. TUBE, ADD 1mL ETHYL ACETATE TO DISSOLVE THEN ADD 1mL LIGROIN (BP 60-80), SOL'N IS SLIGHTLY YELLOW BUT CLEAR (NO PRECIPITATES). USE SYRINGE TO TRANSFER 0.20mL CYCLOPENTADIENE (DRY!) TO REACTION TUBE AND MIX WELL WITH STIRRING ROD. REACTION TUBE GETS WARM, YELLOW COLOR DISAPPEARS. PLACE WARM TUBE INTO BEAKER CONTAINING PAPER TOWEL AS INSULATION SO TUBE COOLS SLOWLY.



AFTER 10 MIN, LARGE COLORLESS PLATE-LIKE CRYSTALS APPEARED. THE TUBE WAS ALLOWED TO COOL TO ROOM TEMP. FOR COMPLETE

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CRYSTALLIZATION, SOLVENT WAS REMOVED BY THE PIPET METHOD AND THE CRYSTALS WERE RINSED WITH 1.5 mL COLD LIGROIN. THE LIGROIN WAS REMOVED, THE CRYSTALS WERE SCRAPED OUT OF THE TUBE AND ALLOWED TO DRY IN THE AIR. TO CONSTANT WEIGHT.

1ST WEIGHT crystals + filter paper 0.585g.

fil. paper 0.252g.

0.333g.

AFTER 10min 0.565g.
0.252g
0.313g.

AFTER 5min 0.565g
0.252g.
0.313g FINAL WT.

% YIELD = $\frac{0.313g.}{0.328g} \times 100 = 95\%$

m.p. 163.5-164.5° C.

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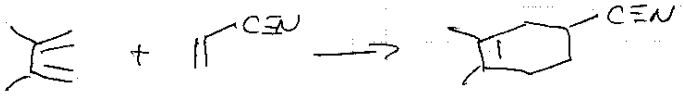
RESULTS + DISCUSSION

THE DIELS-ALDER REACTION OF CYCLOPENTADIENE AND MALEIC ANHYDRIDE PRODUCES PRODUCT IN EXCELLENT YIELD AND PURITY, AS JUDGED BY THE M.P. ADDITIONAL ANALYSES TO CONFIRM THE IDENTITY AND PURITY COULD HAVE BEEN DONE BY TLC, IR, AND NMR. (ETC, ETC, ETC. – ABOUT ½ PAGE – 1 PAGE)

ANSWERS TO ASSIGNED QUESTIONS

(1.)



(ETC, ETC, ETC.)

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