CHEM 322 Laboratory Schedule—Spring 2013

Week of		Experiment
Feb.	11	Isolating Clove Oil—TECH 722, Handout Setup of Diels-Alder Reaction--Handout
	18	Day 2 of Diels-Alder Reaction
	25	Nitration of Methyl Benzoate—REAC 716
Mar.	4	Aldehydes and Ketones—ANAL 728, Handout
	11	Reduction of Vanillin—Handout
	18	Fischer Esterification—SYNT 713
	25	Spring Break – No Lab
Apr.	1	Aspirin—SYNT 745
	8	Grignard Reaction—Handout
	15	Aldol Condensation—SYNT 720
	22	Qualitative Analysis/Unknowns—Handout

CHEM 322 (Spring 2013) Lab Reports and Notebooks

Lab safety: All students must wear appropriate personal protective equipment during all labs: safety goggles, gloves (provided), long pants/skirt, and close-toed shoes. Lab safety will be taken *very seriously*. You will be penalized if you neglect safety:

· For first offense of the semester: Lose 20% of the points for that lab. ·
For second offense of the semester (not per lab) and beyond: Immediate dismissal from lab and loss of all points for that lab.

1. All students must use notebooks with self-duplicating pages. The UD bookstore carries Hayden-McNeil Student Lab Notebook with carbonless duplicate sets.
Please see the example Lab Notebook pages on-line.

2. *Before the lab starts*, you must have the following sections complete in your notebooks:

Title
Balanced reaction equation and mechanism (if applicable)
Reagent Table with the following columns:

Compound Name	Structure	F.W.	Amount	mmol	Equivalents	Physical Properties

Procedure (use the two-column format for procedure/observations, as in the sample lab report provided). *You should be able to perform the experiment by following the procedure written in your notebook.*

You will not be allowed to begin the experiment until all of the above sections are complete and checked by your TA.

3. *As you perform the lab*, the following sections of your lab report must be updated faithfully.

· -Amount in the reagent table: leave room to record the exact amount you used, with correct significant figures. This means you should leave room in your table to record this data, and that you should carry your notebook to the scale to immediately record your exact mass in it (and **not** on scrap pieces of paper, your hand, etc.)

· mmol and equivalents in the reagent table for each reactant or catalyst. 1 mmol = 1/1000 of a mole, and is a more convenient number to use for the small-scale reactions you will be performing. Equivalents are obtained by dividing the mmol of each reactant or catalyst by the lowest number of mmol reactant. By doing this, the equivalents reflect the mole ratio that the materials are combined in.

· -Observations. It should be clear which step an observation pertains to. Also, if any deviations to your pre-written procedure, you must record that as well.

4. *At the end of the lab period*, your duplicate pages will be torn out and handed to your TA before you leave.

5. Your complete lab write-up, including post-lab questions, is due at the start of your next lab period. You will turn in only the duplicate pages. In addition to the sections above that were already handed in, your report should include:

Calculations (yield, etc.) if applicable
Results & Discussion
Post-lab Questions

In the results and discussion section: State any pertinent data and discuss the data. How well did the experiment go compared to what is discussed in the book. Did you get a good yield? If not, why? Were your melting points sharp (1-2 degrees) or did the compound melt over a broader range of temperature; why? Were there any particularly difficult steps in the procedure. If so, can you make suggestions as to make them easier. Included in your discussion should be an evaluation of how well the laboratory worked, what significant sources of error are present, and potential improvements. For example, measurement errors would normally not be mentioned unless you believe that you made an error larger than indicated in sigfigs. Human errors such as "I spilled some of my product" should obviously be recorded. Of particular interest are problems you can identify that potentially can be attenuated by a change in procedure. For example: if your reactant is volatile, but the reaction mixture is boiled in a beaker, the reaction may be improved by performing it in a reflux apparatus to prevent evaporation of reactant.

The points for each lab will be assigned as follows:

Pre-lab Preparation	10 pts
Participation	15 pts
Observations	15 pts
Calculation	10 pts
Results & Discussion	15 pts
Post-lab Questions	15 pts
Selected Section*	20 pts
Total	**100 pts**

* One unannounced section will be carefully graded for quality for each lab.

Missed Labs: All laboratories must be attended. If you miss a lab for a reason that your Dean will vouch for, then your grade will be prorated. All other absences will result in a grade of zero for that day.

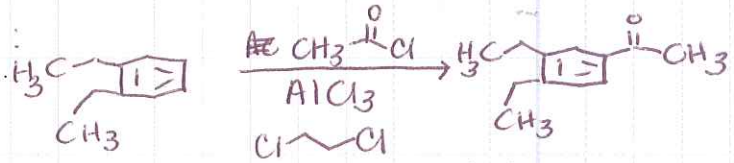
Late Lab Reports: Labs not turned in on time will be penalized 10% of the lab grade per day.

EXP. NUMBER	EXPERIMENT/SUBJECT	DATE
11	Friedel-Crafts Acylation	2/5/11

NAME	LAB PARTNER	LOCKER/DESK NO.	COURSE & SECTION NO.
Mary Watson	Richard Heck		322-020

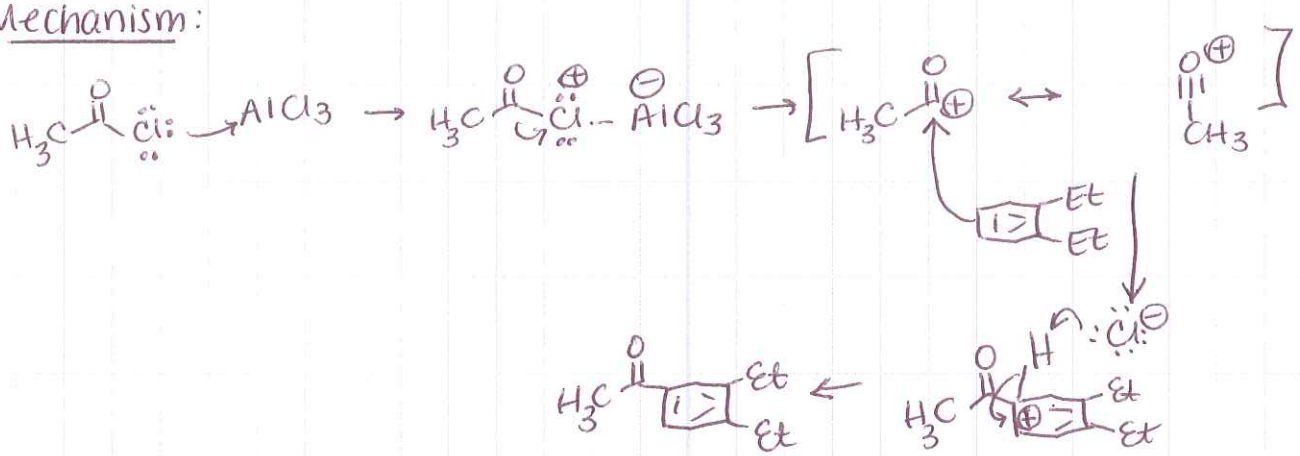
Title: Friedel-Crafts Acylation of o-Diethylbenzene

Reaction:

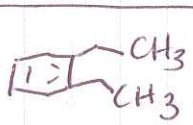
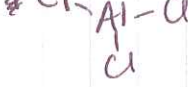
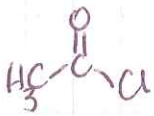


o-diethylbenzene 0°C 3,4-diethylacetophenone

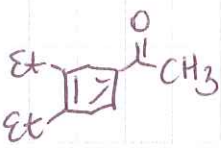
Mechanism:



Reagent Table:

Compound Name	Structure	mw(g/mol)	Equiv	mol	amt	Comment
o-diethylbenzene		134.22	1.0	0.0373	5.0g, 5.7ml	d=0.889g/ml (liquid)
aluminum trichloride		133.34	1.14	0.0427 ~~0.0407~~	5.7 ~~3.2~~g	
acetyl chloride		78.50	1.09	0.0407	3.2g, 2.9ml	d=1.104g/ml (liquid) (lachramator)
1,2-dichloroethane		Solvent			30mL+30mL	(carcinogen)

Expected Product:

3,4-diethyl aceto-phenone		176.25	1.0	0.0373	6.57g	

SIGNATURE	DATE	WITNESS/TA	DATE

EXP. NUMBER	EXPERIMENT/SUBJECT	DATE
11		

08

NAME	LAB PARTNER	LOCKER/DESK NO.	COURSE & SECTION NO.

[PRE-LAB QUESTIONS WOULD GO HERE.]

Procedure

1. Combine 5.7g $AlCl_3$ and 30 mL dichloroethane in 200mL round-bottom flask w/ stir-bar.
2. Cool suspension to 0°C using ice/water bath.
3. In 2nd flask, combine 5g/5.7mL o-diethylbenzene, AcCl (~~3~~ 2.9mL), and 30mL dichloroethane.
4. Add ~~2nd~~ solution in 2nd flask to cooled flask in portions. (Exotherm may occur.)
5. After addn is complete, stir 1h @ 0°C.
6. Pour reaction mixture into 50mL ice-water.
7. Extract product w/ 50mL diethyl ether.
8. Organic layer: wash w/ 1N $NaHCO_3$ (30mL), then H_2O (30mL), then brine (30mL).
9. Dry organic layer w/ $MgSO_4$.
10. Filter. Concentrate on rotovap.
11. Mass product.
12. TLC: Product & o-diethylbenzene.
    ~~If~~ Eluent: hexanes.
    Visualize: UV lamp

Observations

$AlCl_3$ did not dissolve ⇒ suspension.

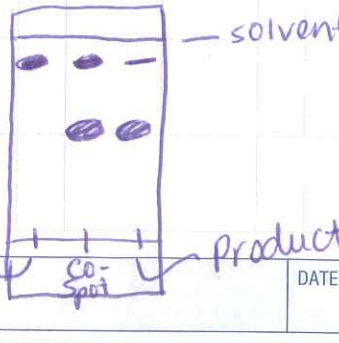
Light yellow solution (homogeneous)

3:45 - 4:45 pm.

Exotherm observed; all ice melted quickly.

Bumped on rotovap. Lost some material on rotovap trap.
m = 4.08g (61%)
yellow oil.

TLC:



SIGNATURE	DATE	WITNESS/TA	DATE

EXP. NUMBER 11	EXPERIMENT/SUBJECT	DATE	09
NAME	LAB PARTNER	LOCKER/DESK NO.	COURSE & SECTION NO.

Results & Discussion

The Friedel-Crafts acylation of o-diethylbenzene proceeded in 61% yield to give 4.08 g of ~~#~~ 3,4-diethylacetophenone. The yield was lower than the theoretical yield probably because the solution of product bumped on the rotovap and some product was lost to the dirty bump trap. Next time, the bump trap will be cleaned prior to use, so any "bumped" product can be recovered.

By TLC ~~a~~ analysis, a small amount of o-diethylbenzene product was mixed with the final product. The ratio of product : starting material cannot be quantified by TLC. To quantify this ratio, I could have used ^{1}H NMR. Remaining starting material, or incomplete reaction, may also be a reason for the low yield.

[POST-LAB QUESTIONS WOULD GO HERE.]

SIGNATURE	DATE	WITNESS/TA	DATE