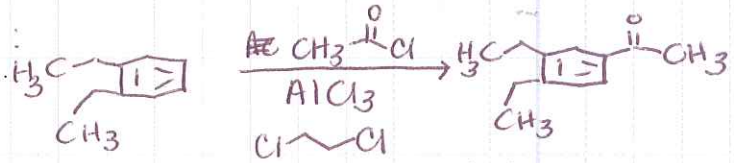
EXP. NUMBER	EXPERIMENT/SUBJECT	DATE
11	Friedel-Crafts Acylation	2/5/11

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

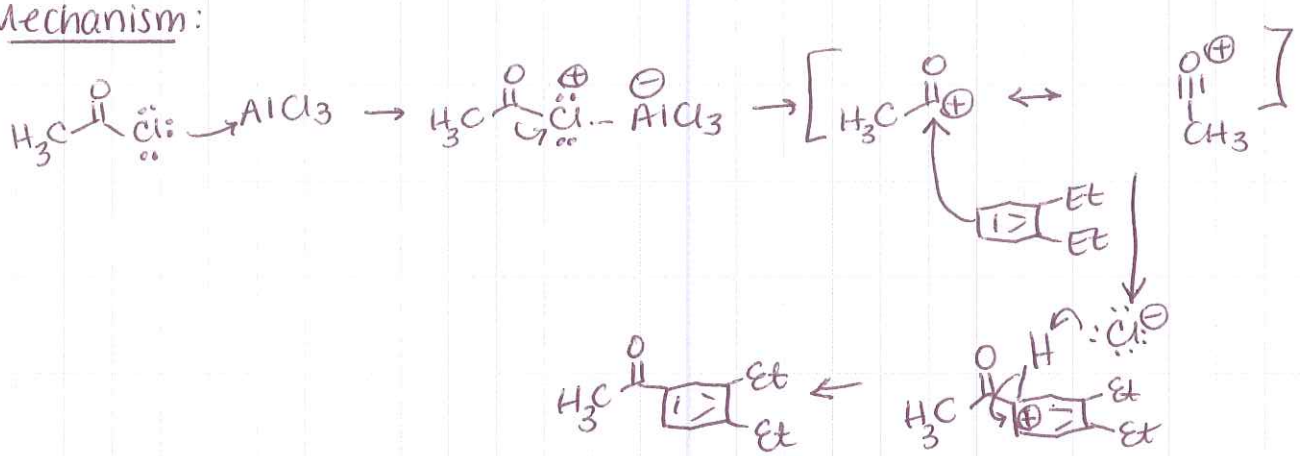
Title: Friedel-Crafts Acylation of o-Diethylbenzene

Reaction:

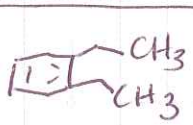
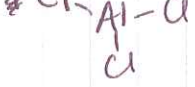
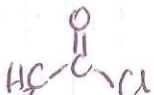


o-diethylbenzene — 0°C — 3,4-diethylacetophenone

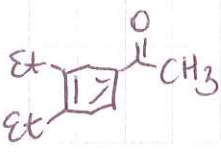
Mechanism:



Reagent Table:

Compound Name	Structure	mw(g/mol)	Equiv	mol	amt	Comment
o-diethylbenzene		134.22	1.0	0.0373	5.0 g, 5.7 ml	d = 0.889 g/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	3.2 g, 2.9 ml	d = 1.104 g/ml (liquid) (lachramator)
1,2-dichloroethane		Solvent			30 mL + 30 mL	(carcinogen)

Expected Product:

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

SIGNATURE	DATE	WITNESS/TA	DATE

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11			08
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[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.

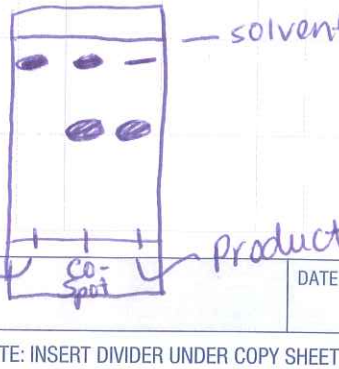
Light yellow solution (homogeneous)

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:



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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 ^1H 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