

## SUPPORTING INFORMATION

### Academic Research Inspired Design of an Expository Organic Chemistry Lab Course

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#### 1. KAIST Department of Chemistry's Laboratory Safety and Work Instructions

1) Wear approved eye protection at all times while in the laboratory.

2) Wear shoes at all times. (No one will be admitted without them.)

3) Eating, drinking, and smoking are strictly prohibited in the laboratory at all times.

4) Know where to find and how to use all safety and first-aid equipments.

5) Consider all chemicals to be hazardous unless you are instructed otherwise. *Dispose of chemicals as directed by your instructor.*

6) If chemical come into contact with your skin or eyes, wash immediately with copious amounts of water and then consult your laboratory instructor.

7) Never taste anything. Never directly smell the source of any vapor or gas.

8) Perform in the fume exhaust hood any reactions involving skin-irritating or dangerous chemicals, or unpleasant odors.

9) Never point a test tube that you are heating at yourself or your neighbor-it may erupt like a geyser.

10) Do not perform unauthorized experiments.

11) Clean up all broken glassware immediately.

12) Always pour acids to water, not water into acid, because the heat of solution will cause the water to boil and the acid to spatter. "Do as you oughter, pour acid into water."

13) Avoid rubbing your eyes unless you know that your hands are clean.

14) **NOTIFY THE INSTRUCTOR IMMEDIATELY IN CASE OF AN ACCIDENT.**

15) Many common reagents-for example, alcohols, acetone, and especially ether-are highly flammable. Do not

use them anywhere near open flame.

16) Observe all special precautions mentioned in experiments.

17) Learn the location and operation of fire-protection devices.

18) Whenever possible, use hot plates in place of Bunsen burners.

#### 2. KAIST Department of Chemistry's Basic Instruction for Laboratory Work

1) Read the assignment before coming to the laboratory.

2) Work independently unless instructed otherwise.

3) Record your results directly onto your report sheet or notebook. **DO NOT RECOPY FROM ANOTHER PIECE OF PAPER.**

4) Work conscientiously to avoid accidents.

5) Dispose of excess reagents as instructed by your instructor. **NEVER RETURN REAGENTS TO THE REAGENT BOTTLE.**

6) Do not place reagent-bottle stoppers on the desk; hold them in your hand. Your laboratory instructor will show you how to do this. Replace the stopper on the same bottle, never on a different one.

7) Leave reagent bottles on the shelf where you found them.

8) Use only the amount of reagent called for; avoid excesses.

9) Whenever instructed to use water in these experiments, use distilled water unless instructed otherwise.

10) Keep your area clean.

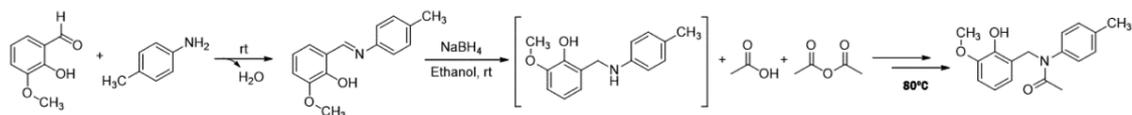
11) Do not borrow apparatus from other desks. If you need extra equipment, obtain it from the stockroom.

12) Do not weigh hot or warm objects. Object should be at room temperature.

13) Do not put hot objects on the desktop. Place on a wire gauze or heat-resistant pad.

## 3. Example of Experiments and Results PPT Slides

## Exp4. Solid-Solid reaction of reductive amination

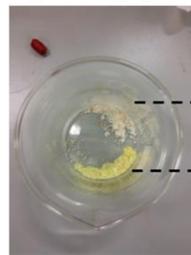


Molecular name	o-Vanillin	p-Toluidine	Imine	Sodium borohydride	Ethanol	Amine	Acetic acid	Acetic anhydride	Product
Molar weight [g/mol]	152.15	107.16	241.29	37.83	46.07	243.30	60.05	102.09	285.34
n [mmol]	5.0	5.0	5.0/5.0	2.6	-	-/5.0	35.0	21.2	5.0/5.0
mass [g]	0.76	0.535	1.20/1.21	0.1	-	-/1.22	2.10	2.16	1.36/1.43
density [g/ml]	-	-	-	-	-	-	1.05	1.08	-
volume [ml]	-	-	-	-	15	-	2	2	-
equivalent [eq]	1	1	1.0/1	0.53	(0.33M)	-/1.0	7.0	4.2	0.95/1
			Yield : 100%						Yield : 95%
Meting point [°C]	40-42	41-46							
	Pale yellow	White	Bright Orange			Yellow			White

## Exp4. Solid-Solid reaction of reductive amination

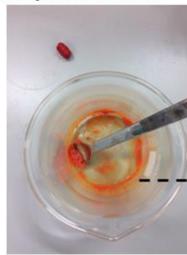
- 1:26pm reaction preparation  
 1:32pm reaction start  
 - color : bright orange  
 1:40pm add 10ml ethanol  
 - slightly dissolved  
 1:47pm add sodium borohydride  
 - bubble(H<sub>2</sub>)  
 - color : clear orange  
 - all dissolved  
 2:00pm add 2ml acetic acid  
 2:04pm add 2ml acetic anhydride  
 2:06pm heating  
 2:24pm add 75ml water  
 - white solid precipitation  
 2:35pm ice bath  
 2:40pm vacuum filter  
 - white solid  
 3:00pm NMR sampling

## 1:26pm reaction preparation



Low melting point => Impurity => Lowering of melting point => Melting, direct reaction  
 - Ecofriendly, no solvent  
 - Fast reaction

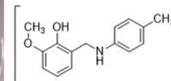
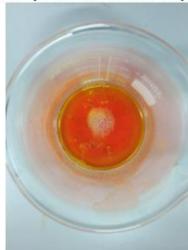
## 1:32pm reaction start



## 1:40pm add 10ml ethanol



## 1:47pm add sodium borohydride (reductive amination)

H<sub>2</sub> bubble

clear orange solution

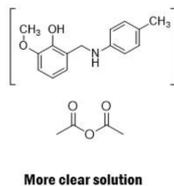
All reactants are dissolved

## Exp4. Solid-Solid reaction of reductive amination

2:00pm add 2ml acetic acid



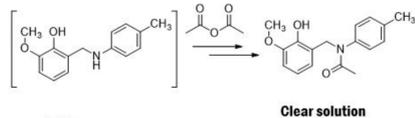
+ add 2ml acetic anhydride



2:06pm heating (15min)



Amide formation

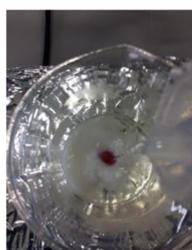


80°C

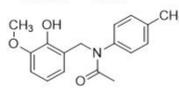
2:24pm add 75ml water



Strongly stirring



White precipitation

Solubility  
Ethanol >> Water

2:35pm ice bath

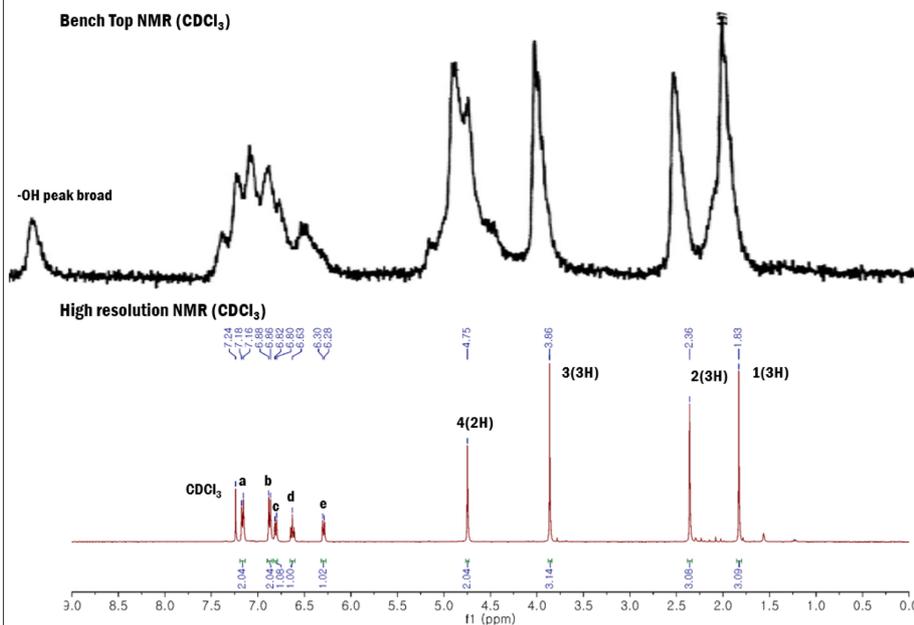
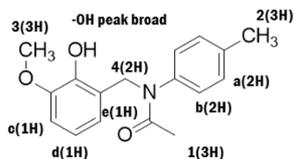


Crystallization

2:40pm vacuum filter

Washing by water  
Air dryWhite solid  
1.36g (Yield : 95%)

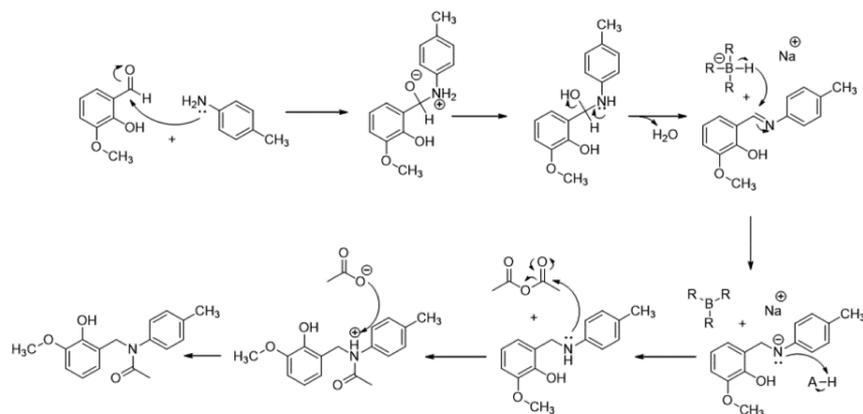
## Exp4. Solid-Solid reaction of reductive amination

Bench Top NMR (CDCl<sub>3</sub>)High resolution NMR (CDCl<sub>3</sub>)

Reference

<sup>1</sup>H-NMR (300MHz, CDCl<sub>3</sub>, δ): 1.85 (s, 3H), 2.37 (s, 3H), 3.88 (s, 3H), 4.76 (s, 2H), 6.33 (dd, 1H, J=7.5, 1.8 Hz), 6.63 (t, 1H, J=8.1Hz), 6.81 (dd, 1H, J=7.8, 1.8), 6.88 (d, 2H, J=8.7Hz), 7.15 (d, 2H, J=7.8Hz).

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**Exp4. Solid-Solid reaction of reductive amination****Post-Laboratory Question****<Mechanism>****- role of acetic acid**