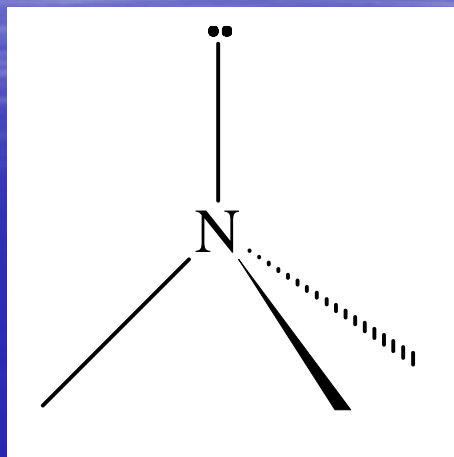


Amine: Acetylation of Aniline with Acetic Anhydride

苯胺和乙酸酐的乙酰化反應

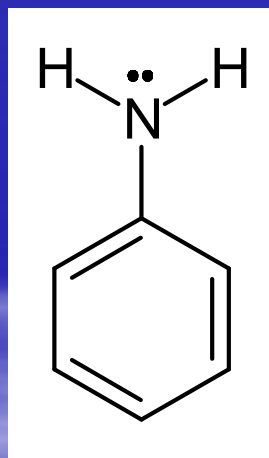
1. Amine:



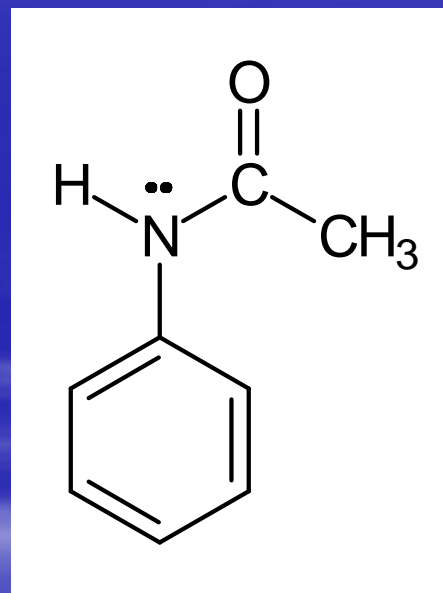
basicity:	<	<	<
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nucleophilicity:	<	<	?
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2. Amine and Amide:



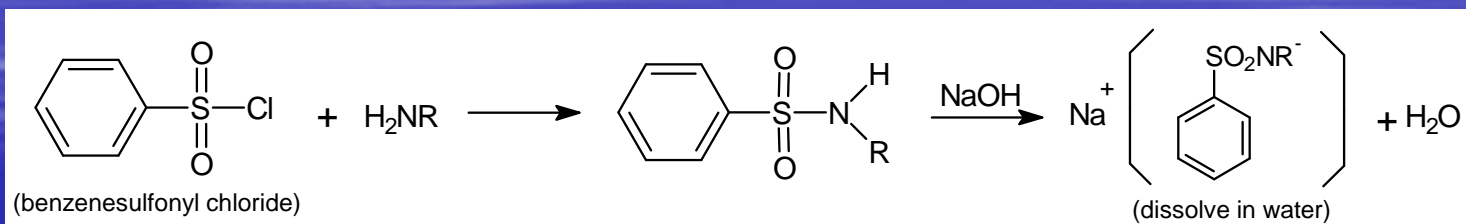
base



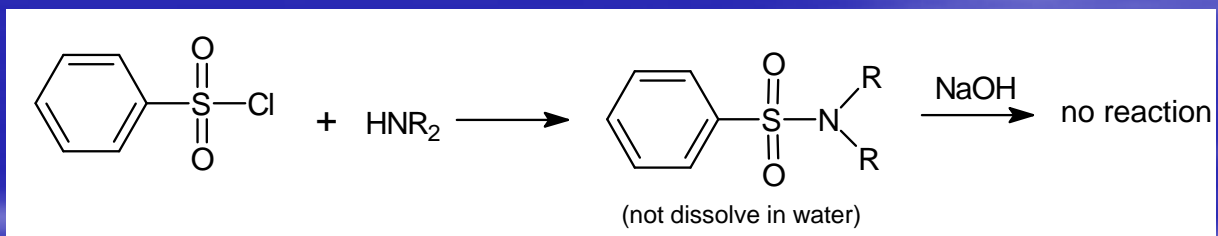
weak acid

3. Hinsberg test:

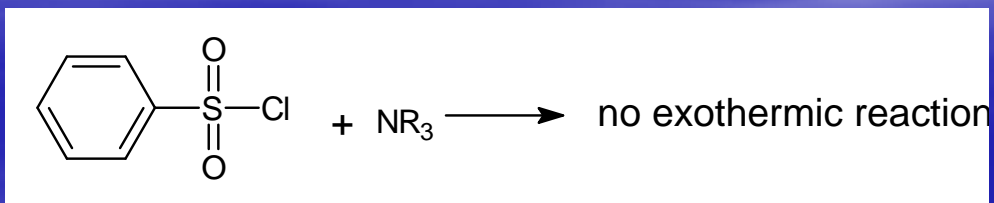
1°-amine:



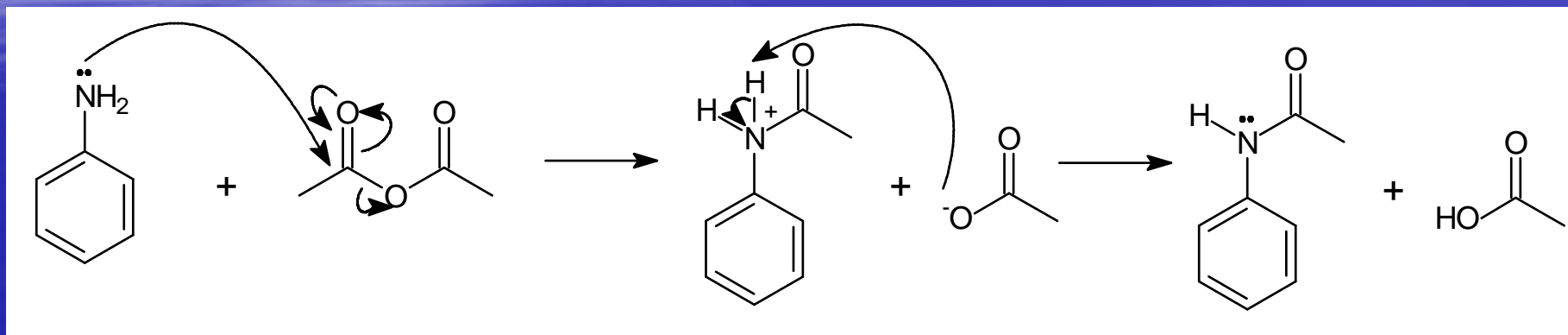
2°-amine:



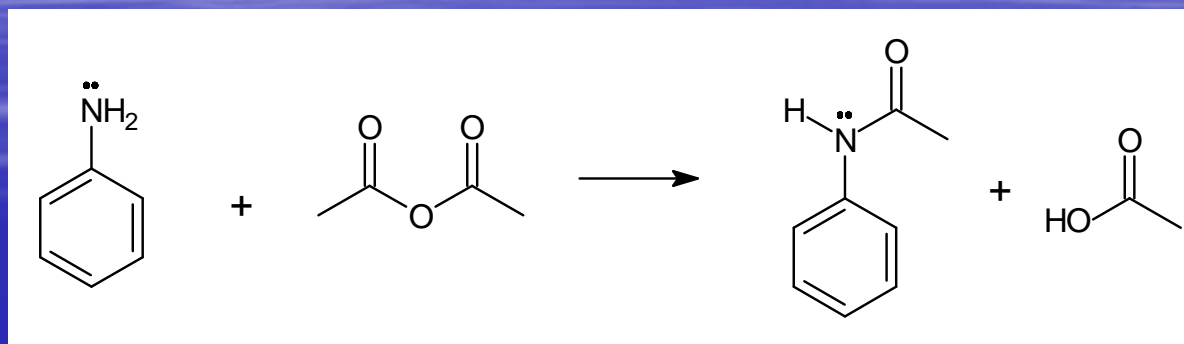
3°-amine:



4. Mechanism of acetylation of aniline with acetic anhydride:



Reaction equation:



0.31g aniline + 10mL H₂O in a 50ml Erlenmeyer flask

↓(ice-water bath)

stir for 2 min

↓

add 0.5mL acetic anhydride (Ac₂O) slowly

↓

warm the mixture in a water bath until it dissolve

↓

add 0.02g active charcoal

filter (water bath)



cool it to r.t. and then ice-water bath for 2 min



suction filter



recrystallize with H₂O



suction filter (wash with cold water)



dry



weight



calculate the % yield (in result report)

Hinsberg test

3d amine (10mg solid) in a test tube



add 4d benzenesulfonyl chloride (in hood)



alkaline \longrightarrow no exothermic reaction \longrightarrow *3°-amine*

↓(exothermic reaction)

cool



the residue + 1mL 10% NaOH_(aq)

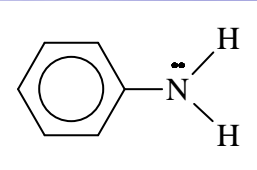
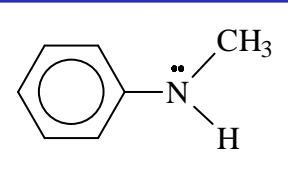
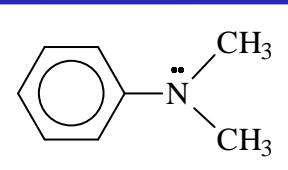


shake \longrightarrow not dissolve \longrightarrow *2°-amine*

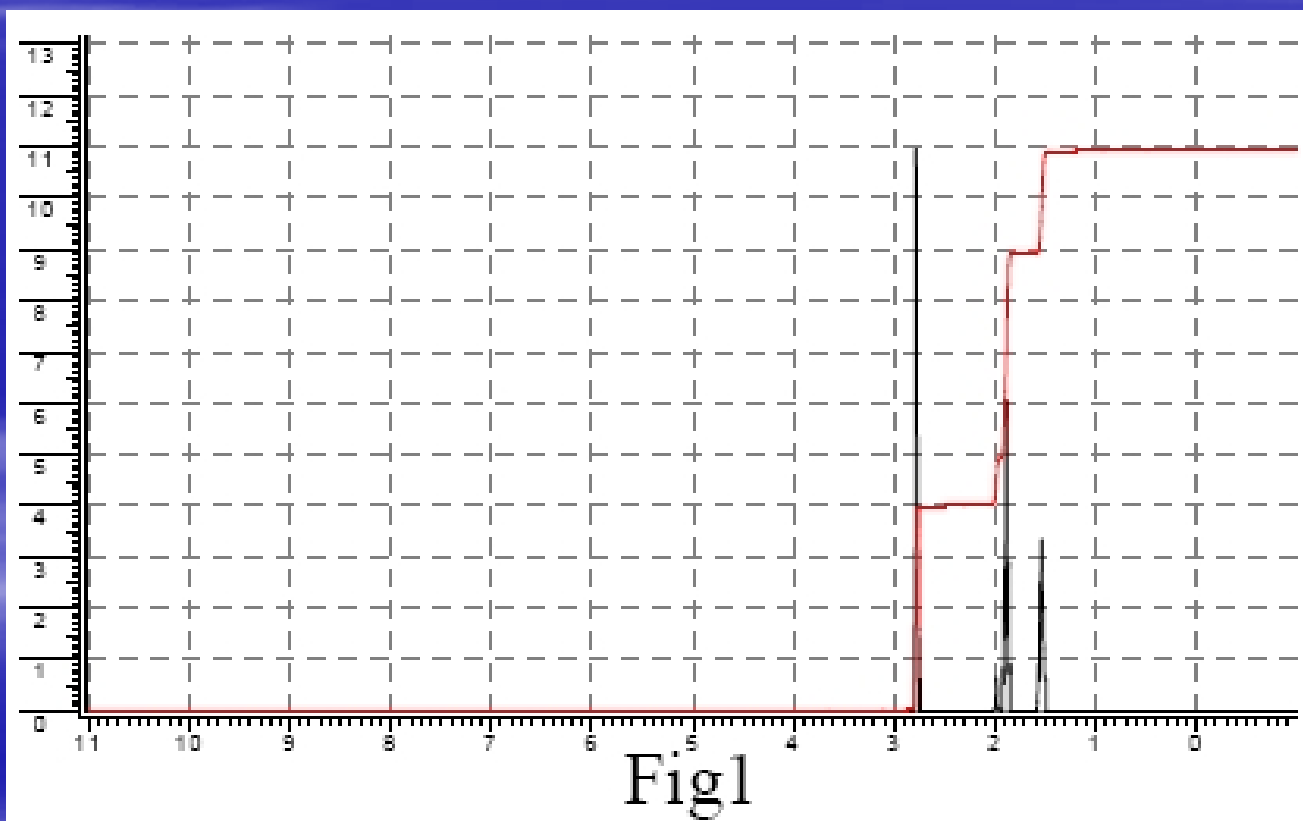
↓(dissolve)

1°-amine

Hinsberg test result

Amines	Structures	Exothermic reaction	Dissolve in NaOH _(aq)	Result
Aniline		+	+	1°-amine
N-methyl aniline		+	—	2°-amine
N,N-dimethyl aniline		—	—	3°-amine
Unknown 1				
Unknown 2				

(1) 實驗中, unknown 1 的分子式為 $C_5H_{11}N$
 1H -NMR 光譜如下, 試問其結構式為何?



(2) 實驗中, unknown 2 的分子式為 C_7H_9N
 1H -NMR 光譜如下, 試問其結構式為何?

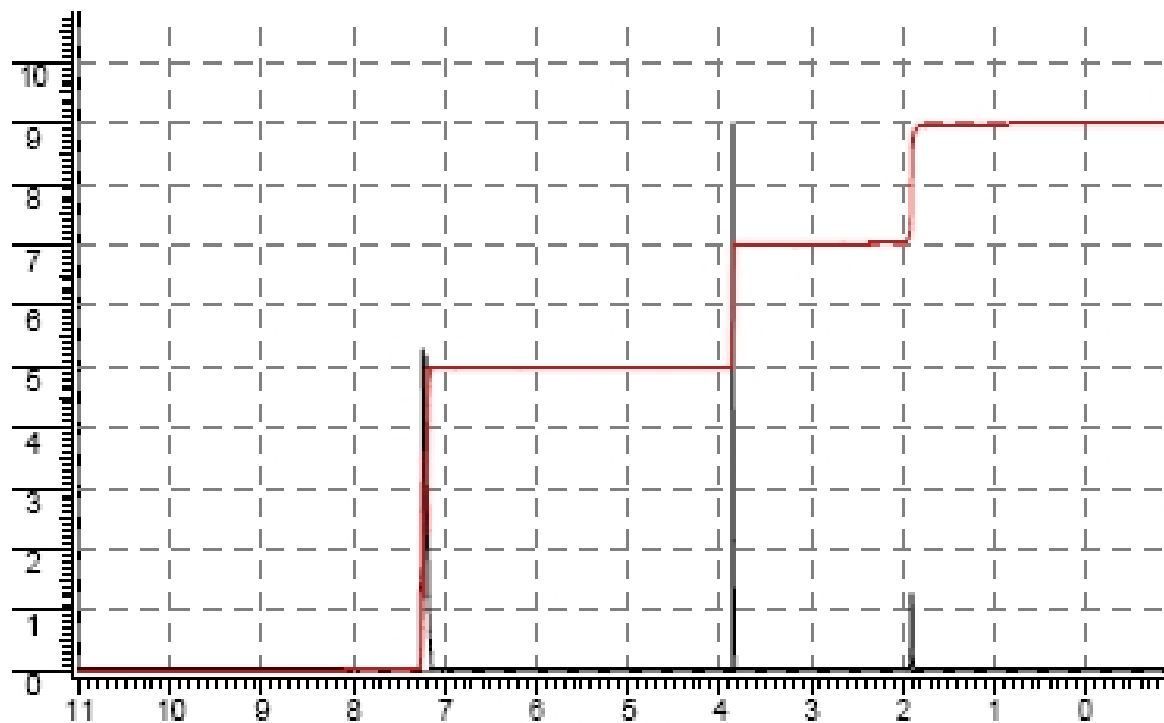


Fig2

Check out

1. 繳交產物並告知產物淨重。
2. Homework 1,2 : 請畫出 unknown 1 , 2 的結構式。