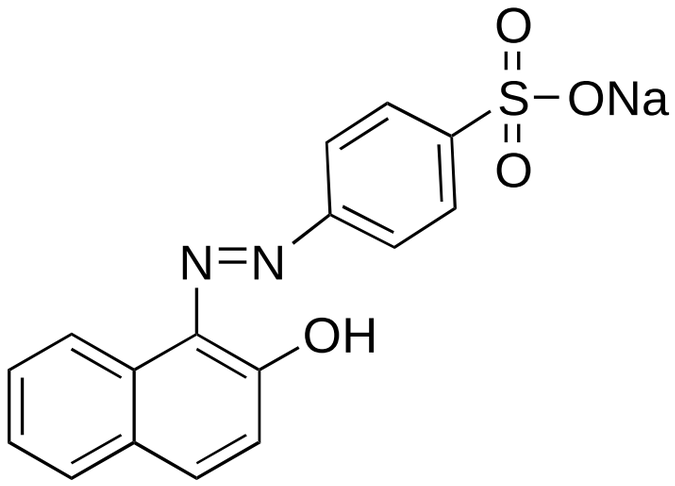
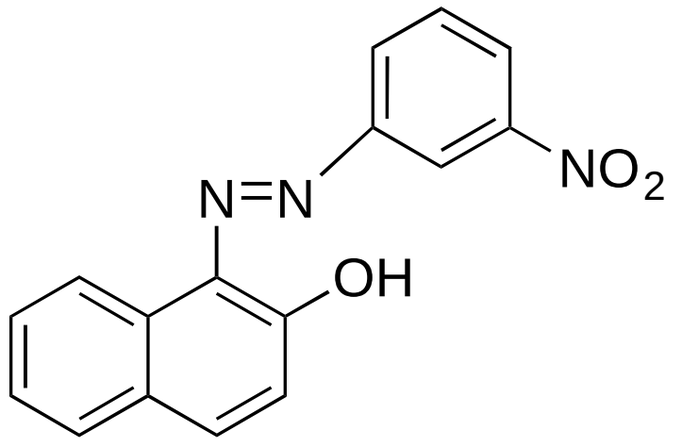
Week 12

Azo Dye Synthesis, Orange II



4-[(2-hydroxy-1-naphthalenyl)azo]benzene
Sulfonic acid monosodium salt



3-[(2-hydroxy-1-naphthalenyl)azo]nitrobenzene

Mechanism of Orange II Formation

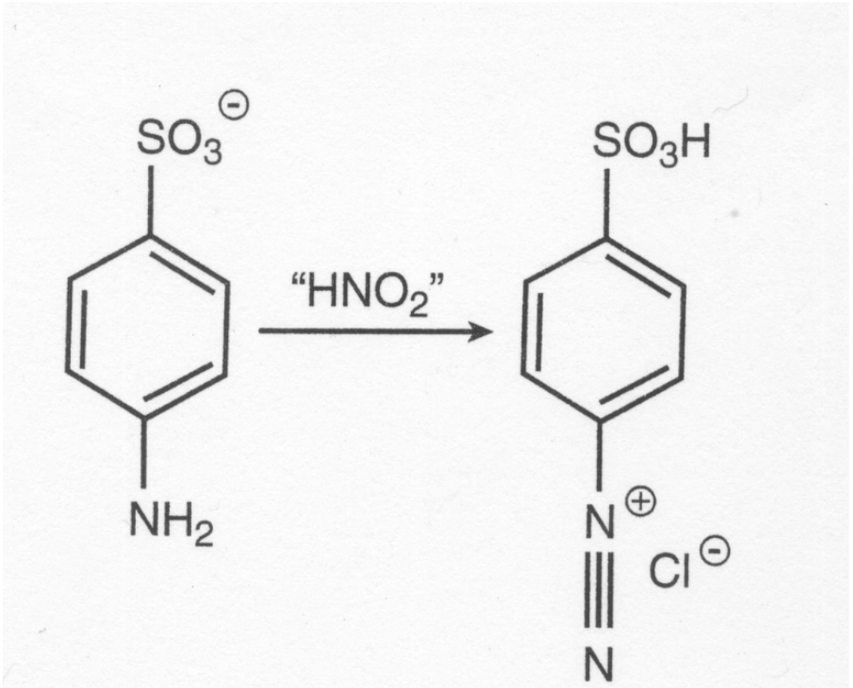
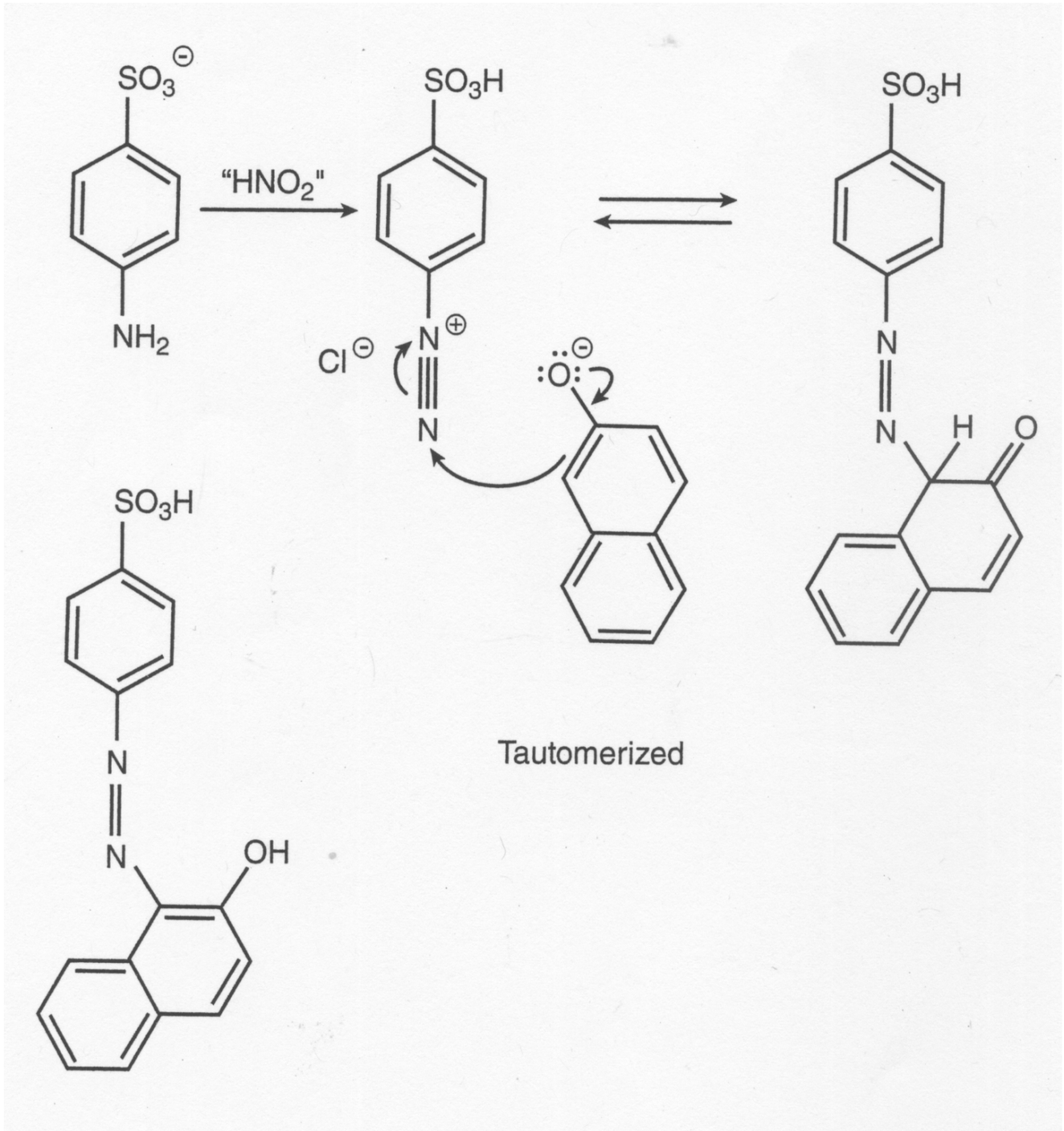
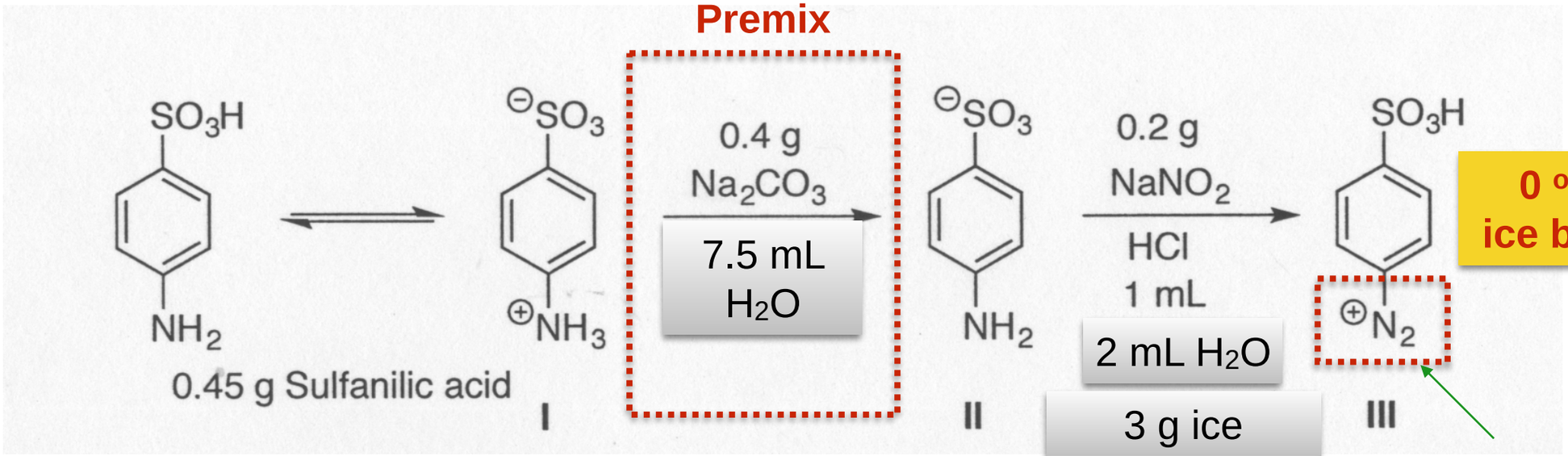


Figure 13.2. Diazotization of sulfanilic acid.



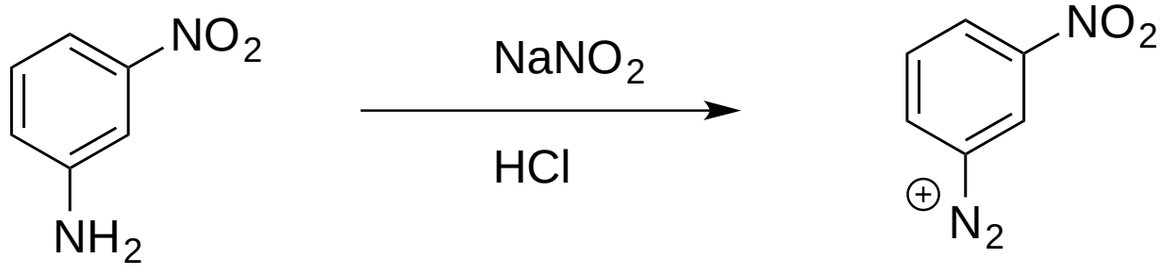
Synthesis of Orange II

$NaNO_2$
Sodium nitrite
+
HCl
↓
"HNO_2"



One of the best leaving groups!
Keep it cold not to eliminate!

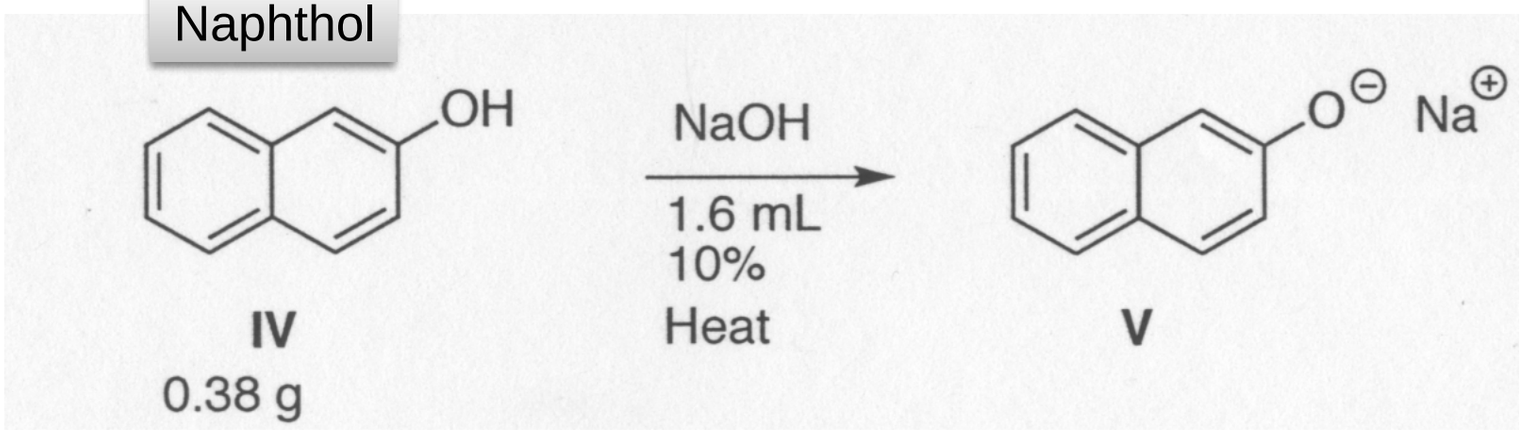
1. Heat Erlenmeyer over steam bath to dissolve
2. Cool to RT, then add $NaNO_2$, stir to dissolve
3. Pour the pale yellow solution slowly into a 125 mL Erlenmeyer with H_2O, HCl, ice. Keep it COLD!



No need for Na_2CO_3.
Already free amine, no acidic proton

Synthesis of Orange II

Naphthol



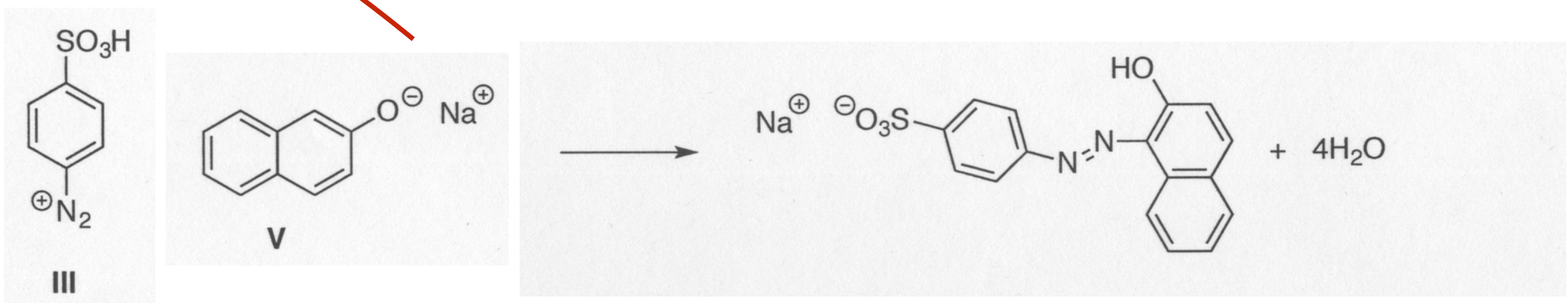
IV
0.38 g

V

1. Heat flask over steam bath to dissolve
2. Cool in ice bath

Add the naphthol solution DROPWISE into the diazonium solution and stir SLOWLY and CAREFULLY!

Orange II will crystallize! Collect and recrystallize with EtOH.
Don't get it on you, you will be graded on how dean your hands, bench are.



III

V

After you are done, you need to Check Out!!!

General Reminders

Bring a calculator to CEM255 Final Exam

Final Exam: Thursday, December 5, 2019
5:00 – 5:50 PM
Rm 115 Wells Halls

10 Extra Point Lab Clean Up
Friday, December 6, 2019
10 AM – Noon
1 hour each person

CEM 255 Final Exam Study Guide

1. Bring a calculator to CEM255 final exam.

2. Study the discussion part of every experiment you have performed.

3. Make sure you know the answers to the questions at the end of each of the experiments that you have performed this semester.

4. Know how to calculate empirical formula/molecular formula from elemental percent composition.

5. There will be at least one ^{1}HNMR question, where you have to come up with the structure of the compound from a proton–NMR spectrum.

6. There will be question(s) about theoretical yield and percent yield.

7. Although, you should not have to memorize the experimental procedures, you have to know the reasons for some "particular steps". For instance: What is the reason for chilling the diazonium salt in an ice bath while preparing sodium naphthoxide during the azo-dye experiment?
Answer: because N2 is world's greatest leaving group, thus diazonium salts would decompose at a fast rate at room temperature.

Calculate Empirical Formula From Elemental Analysis

Determine the empirical formula of Compound **A** based on the elemental analysis provided below. Elemental analysis of Compound **A**:

			Ratio
% C = 60.86 (AM = 12)	⟶	5.07 moles/2.17	2.34
% H = 4.38 (AM = 1)	⟶	4.38 moles/2.17.	2.02
% O = 34.76 (AM = 16)	⟶	2.17 moles	1

$C_{2.34}\ H_{2.02}\ O$

x 2 ⟶ $C_{4.68}\ H_{4.04}\ O_2$

x 3 ⟶ $C_{7.02}\ H_{6.06}\ O_3$

$C_7\ H_6\ O_3$