Chem 51LB
Practical Week 10
Review

Organic Chemistry Peer Tutoring Department
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Practical Breakdown: Week 10

Week 10: Everyone

- Knowledge Check - Lab Lecture Week 10
  - multiple choice high yield topics
  - Percent yield calculations, NMR and IR spectroscopy, techniques, mechanisms

- Safety- Lab Section Week 10
  - know safety symbols and identification of incorrect safety scenarios by image

- Technique - Lab Section Week 10
  - Recrystallization
  - Know how to do calculate Percent Recovery

- Mastery
  - 2 critical thinking, 2 mechanisms, 2 spectroscopy
Safety Scenario
Safety Symbols

Make sure to know all of these symbols!

- GHS01 Explosive
- GHS04 Compressed Gas
- GHS07 Harmful
- GHS02 Flammable
- GHS05 Corrosive
- GHS08 Health Hazard
- GHS03 Oxidising
- GHS06 Toxic
- GHS09 Environmental Hazard
On the Practical

- You will be given 6 images of safety symbols and an answer bank
- You will need to match the appropriate symbol to the name of the hazard
- 4 out of 6 correct to pass
- 5 out of 6 correct for an A

Let’s do some practice!
Safety Practice

Which hazard does this symbol correspond to?

a) Carcinogen
b) Compressed gas
c) Corrosive
d) Environmental toxin
e) Explosive
f) Flammable
g) Irritant
h) Oxidizer
i) Toxic
Safety Practice

Which hazard does this symbol correspond to?

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Knowledge Check
On the Practical

- 14 question multiple choice format covering:
  - NMR/IR Spectroscopy problems
  - Percent Yield/Limiting Reagent
  - Mechanisms of reactions studied in class
  - Lab Techniques: TLC, MP, Recrystallization, Gas Chromatography

- One question from the practice exam on canvas will be on the actual exam
- Don’t need a scantron

To Pass:

- 11/14 correct
- Do the practice exam to see if you are ready!
IR Spectroscopy

Peaks you should know:

- O-H bond: broad peak from 2800-3600
- Amines
  - Primary amine: 2 bands from 3300-3500
  - Secondary amine: 1 band from 3300-3500
- C-H Bonds
  - Csp3-H: 2900-3000
  - Csp2-H: 3000-3200
  - Csp-H: 3200-3300
IR Spectroscopy

Peaks you should know:

- Aldehyde Fermi doublet: 2700-2800 and 2800-2900
- Carbonyl groups: in 1700s
  - Exceptions
    - Acid Chloride: 1800
    - Amides: 1650
- Benzene ring: usually around 1500
NMR Spectroscopy

Peaks you should know:

- **0-2.0**
  - Alkane C-H

- **2.0-3.0**
  - H neighboring carbonyl
  - H neighboring Benzene
  - H neighboring Alkene
  - H neighboring Alkyne

- **3.0-4.0**
  - H neighboring halogen
  - H neighboring ether

Neighboring means: $X - C - H$
NMR Spectroscopy

- 5.0-6.0
  - Vinyl H
- 6.0-8.0
  - Aromatic H
- ~10.0
  - Aldehyde H
- ~10.0-13.0
  - Carboxylic Acid H
Quick Review

What are the correct NMR chemical shifts for the indicated H’s?
Quick Review

What are the correct NMR chemical shifts for the indicated H’s?

- H: 5.0-6.0
- H: 6.5-8.0
- H: 10.0
Sample Spectroscopy Problem

\[ C_9H_{10}O_2 \]
Sample Spectroscopy Problem

$C_9H_{10}O_2$
Sample Spectroscopy Problem - Answer

Units of Unsaturation: 5

IR Evidence:
- Csp3-H at 2900-3000
- Csp2-H at 3000-3100
- Carbonyl at ~1720
- Possible benzene at ~1600

NMR Evidence:

<table>
<thead>
<tr>
<th>Shift</th>
<th>Integration</th>
<th>Splitting</th>
<th>Partial Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5</td>
<td>3H</td>
<td>triplet</td>
<td>CH2-CH3</td>
</tr>
<tr>
<td>4.5</td>
<td>2H</td>
<td>quadruplet</td>
<td>CH2-CH3</td>
</tr>
<tr>
<td>7.5-8.5</td>
<td>5H</td>
<td>multiplet</td>
<td>Aromatic H</td>
</tr>
</tbody>
</table>
Sample Spectroscopy Problem - Answer

Structure Answer:

Ethyl Benzoate

\[
\begin{align*}
\text{\textbf{Ethyl Benzoate}} \\
\text{\includegraphics[width=0.5\textwidth]{ethylenbenzoate.png}}
\end{align*}
\]
**Percent Yield**

Percent yield = \( \frac{\text{actual yield}}{\text{theoretical yield}} \times 100 = \_\% \)

- Actual yield = mass of product from your reaction (ideally purified)
- Theoretical yield = maximum potential mass of product that could be obtained from the reaction
  - \( \rightarrow \) Determine theoretical yield using a balanced equation for the reaction
  - \( \rightarrow \) Use equation to determine limiting reagent
  - \( \rightarrow \) PAY ATTENTION TO MOLE RATIO

**Percent Yield Error Analysis:**

- High yield (>100%): water in product or remaining impurities in product
- Low yield (<<100%): incomplete reaction, spills or other user error
Finding Limiting Reagent

- Determines the maximum amount of product that could theoretically be obtained
- Pay attention to the mole ratio of the reaction equation
Let’s practice finding limiting reagent!

Given equation: NO \,(g) + O_2\,(g) \rightarrow NO_2\,(g)

Given information about starting materials: 12.4 g of NO and 9.40 g of O_2

Which starting material is the limiting reagent and what is the theoretical yield of NO_2?

If the mass of recovered NO_2 is 17.8g, what is the percent yield?
Let’s practice finding the limiting reagent!-Answer

Balance the equation: $2 \text{NO (g)} + \text{O}_2 \text{(g)} \rightarrow 2 \text{NO}_2$

$12.4 \text{ g NO} \times \left( \frac{1 \text{ mol NO}}{30.01 \text{ g NO}} \right) \times \left( \frac{2 \text{ mol NO}_2}{2 \text{ mol NO}} \right) \times \left( \frac{46.01 \text{ g NO}_2}{1 \text{ mol NO}_2} \right) = 19.01 \text{ g NO}_2$

$9.40 \text{ g O}_2 \times \left( \frac{1 \text{ mol O}_2}{32.00 \text{ g O}_2} \right) \times \left( \frac{2 \text{ mol NO}_2}{1 \text{ mol O}_2} \right) \times \left( \frac{46.01 \text{ g NO}_2}{1 \text{ mol NO}_2} \right) = 27.03 \text{ g NO}_2$

NO is the limiting reagent and O2 is the excess reagent. The theoretical yield is 19.01g of NO2.
Let’s practice finding the limiting reagent!-Answer

Percent Yield = (Observed/Theoretical) * 100%

(17.8g/19.01g) * 100% = 93.6% Yield
Another example!

Phenol + HNO₃ → Picric acid + 3 H₂O

What is the limiting reagent?
Another example!

You can determine the limiting reagent from the image above:

3 mole of nitric acid : 1 mole picric acid

The limiting reagent is nitric acid - it will run out first
Stoichiometry

What is stoichiometry?

- The relative relationships between different measurements of quantities used in a reaction

What you should know how to do:

- Know how to convert between mass, volume, and moles
- Know how to look at a table of given information and determine how to calculate missing information
Let’s Practice Stoichiometry!

![Chemical reaction diagram]

<table>
<thead>
<tr>
<th>compound</th>
<th>phenol</th>
<th>1,2-dibromoethane</th>
<th>product</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molar Mass</td>
<td>94.11 g/mol</td>
<td>187.86</td>
<td>214.26</td>
</tr>
<tr>
<td>Density or conc.</td>
<td>2.50 g/mL</td>
<td></td>
<td></td>
</tr>
<tr>
<td>mmol</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Amount (mg)</td>
<td>438.00 mg</td>
<td>376.00 mg</td>
<td></td>
</tr>
<tr>
<td>Actual yield (mg)</td>
<td></td>
<td></td>
<td>350 mg</td>
</tr>
</tbody>
</table>
Let’s Practice Stoichiometry!

![Chemical reaction diagram]

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<td>2.00 mmol</td>
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</tr>
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<td>438.00 mg</td>
<td>376.00 mg</td>
<td>428.52 mg</td>
</tr>
<tr>
<td>Actual yield (mg)</td>
<td></td>
<td></td>
<td>400 mg</td>
</tr>
</tbody>
</table>
Percent Recovery

Percent recovery = \( \frac{\text{mass of purified product}}{\text{mass of crude product}} \times 100 \% \)

What are the crude and purified product:

- Crude product: the product which is obtained directly from the reaction
- Pure product: product that is obtained after recrystallization/purification process
Lab Technique: Recrystallization
Recrystallization Concepts

What is it?

- Recrystallization is a purification method that allows us to remove impurities from a crude compound.

What does it help you achieve?

- Recrystallization allows you to purify your compound of interest by dissolving the crude compound and then allowing it to precipitate so that most impurities remain in solution.
Recrystallization Concepts (cont.)

How does recrystallization work?

- Recrystallization is based on solubility
- 2 different solvents are used
- The compound of interest is soluble in ONE of the solvents, but not the other
- The ratio of the 2 solvents is manipulated to sequentially dissolve the compound over heat and recrystallize the compound in ice
On the Practical

You will be given an impure compound and solvents.

You will either need to:

1) Test unknown solvents to determine if they are appropriate for recrystallization of your compound
2) Or, more likely, perform a recrystallization with given solvents and a compound and:
   - Find a percent recovery
   - Write basic procedures and observations of your compounds

You will not have to do both!
Recrystallization Procedures

Imagine your compound of interest is soluble in ethanol, but insoluble in water. In this case, you would:

1. Gently heat about 20 mL of both water and ethanol in 2 separate beakers on a hot plate (make sure the ethanol does not begin boiling)
2. Place your crude compound in a small flask and add small amounts of heated ethanol until it is completely dissolves
3. Then, remove the flask from heat and add heated water dropwise until cloudiness appears
   a. This indicates the compound is beginning to precipitate from solution
4. Allow the flask to cool to room temperature and then place it in an ice bath for 15 minutes
5. Isolate your completed crystals with vacuum filtration, and weigh the product
Potential Problems

1. You added too much ethanol, and now your compound won’t precipitate
   a. How to fix: Allow some of the ethanol to evaporate and attempt precipitation again
      i. If this fails, you may need to start again
   b. How to avoid: Add ethanol very slowly and make sure to swirl with every addition so you can see the exact point at which your compound dissolves

2. You saw cloudiness in the flask, but now there is no precipitate
   a. How to fix: you may need to lightly scratch the inside of the flask to give the crystals a place to start forming
Recrystallization Practice Problem

You conduct 2 different recrystallizations with an unknown Compound A. When you use ethanol to dissolve the compound and hexanes to precipitate the compound, you succeed in isolating it. But, when you use acetone to dissolve the compound, and water to precipitate it, you do not get any solid precipitate. From this, what can we conclude for certain about Compound A?

a) It is polar
b) It participates in H-bonding
c) It is nonpolar
d) It is neither polar or nonpolar
Recrystallization Practice Problem-Answer

You conduct 2 different recrystallizations with an unknown Compound A. When you use ethanol to dissolve the compound and hexanes to precipitate the compound, you succeed in isolating it. But, when you use acetone to dissolve the compound, and water to precipitate it, you do not get any solid precipitate. From this, what can we conclude for certain about Compound A?

a) It is polar
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d) It is neither polar or nonpolar

The compound remains dissolved in the second solvent if it is polar, but precipitates if it is nonpolar. Because like-dissolves-like, we know the compound is polar. We cannot conclude anything about H-bonding without more information.
The solubility of compound B in ethanol is 7.5 g in 100 mL hexanes at 70°C, and 0.09 g in 100 mL at 0°C. What percent recovery would you expect if you dissolve 188 g of compound B in ethanol and recrystallize using the minimum amount of solvent?

A) 91%
B) 99%
C) 89%
D) 100%
Calculations Practice

The solubility of compound B in ethanol is 7.5 g in 100 mL hexanes at 70°C, and 0.09 g in 100 mL at 0°C. What percent recovery would you expect if you dissolve 188 g of compound B in ethanol and recrystallize using the minimum amount of solvent?

Answer: B) 99%

Calculations: Stoichiometry using the given solubility values

188 g Compound B * (100mL/7.5g) = 2506.6mL ethanol required at 70°C

2506.6mL ethanol * (0.09 g/ 100mL) = 2.256 g will remain dissolved at 0°C

Percent Yield: (188g - 2.256g)/188g * 100% = 98.8% recovery
Technique Reminders

1. Melting Point
   a. Impurities will cause the MP to lower and broaden
   b. The MP can never be raised

2. Gas Chromatography
   a. A large peak at the start of the spectrum is air in the product mixture
   b. Calculate percent of a product with the ratio of the area of that peak over the sum of the areas of the peaks

3. TLC
   a. The stationary phase is polar
   b. More polar solvents cause all compounds to move higher than less polar solvents
   c. Less polar compounds will move higher in all solvents than more polar compounds
Extraction Practice

Which of the following pairs of compounds could you separate with liquid-liquid extraction using saturated NaHCO$_3$?

a) 

b) 

c)
Extraction Practice - Answer

Which of the following pairs of compounds could you separate with liquid-liquid extraction using saturated NaHCO₃?

a) ![Molecules](image1)
   
   No - You would need to separate these with an acid!

b) ![Molecules](image2)
   
   Yes - Bicarbonate is a base and can deprotonate the acetic acid, but not the amide.

c) ![Molecules](image3)
   
   No - these 2 molecules have very similar pKas and bicarbonate is not strong enough to deprotonate alcohols
Mechanisms
Nucleophilic Substitution

Experiment: benzyl bromide run under reflux with one of three unknown compounds to obtain a benzyl ether product

Mechanisms: SN1 and SN2
# Nucleophilic Substitution: SN1 vs SN2

<table>
<thead>
<tr>
<th>Factors</th>
<th>SN1</th>
<th>SN2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rate Law</td>
<td>unimolecular</td>
<td>bimolecular</td>
</tr>
<tr>
<td>Number of Steps</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>Strength of Nucleophile</td>
<td>Weak (e.g. H2O, ROH)</td>
<td>Strong (e.g. -OH, -OCH3, -NH2)</td>
</tr>
<tr>
<td>Type of Alkyl Halide</td>
<td>3° &gt; 2° &gt; 1° &gt; methyl</td>
<td>Methyl &gt; 1° &gt; 2° &gt; 3°</td>
</tr>
<tr>
<td>Solvent</td>
<td>Polar protic</td>
<td>Polar aprotic</td>
</tr>
<tr>
<td>Remember!</td>
<td>Racemization of stereocenters occurs</td>
<td>Backside attack leads to inversion of stereocenter</td>
</tr>
</tbody>
</table>
SN2 Mechanism
SN1 Mechanism

You cannot have an SN1 reaction with a primary alkyl halide because it will create a primary carbocation.
Bromination of an Alkene

Experiment: Trans-cinnamic acid and Br2 are reacted to obtain different bromination products, which are then analyzed.

Mechanisms: Syn, Anti, and Syn/Anti combination
Bromination Anti-Addition
Bromination Syn-Addition
Bromination Syn and Anti-Addition
Elimination Reactions

Experiment: perform eliminations under both acidic (sulfuric acid) and basic conditions (tert-butoxide) and compare reaction results.

Mechanisms: acid-catalyzed dehydration and base induced dehydrobromination
### E1 vs E2

<table>
<thead>
<tr>
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<th>E2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of steps</td>
<td>Two</td>
<td>One</td>
</tr>
<tr>
<td>Leaving group</td>
<td>3° &gt; 2° &gt; 1°</td>
<td>1° &gt; 2° &gt; 3°</td>
</tr>
<tr>
<td>Base</td>
<td>Weak nucleophile or base (H2O, H2SO4)</td>
<td>Strong base (NaOH, K-TBu)</td>
</tr>
</tbody>
</table>
Acid-Catalyzed Dehydration

Condition: 1-butanol (E2)
Acid-Catalyzed Dehydration

Condition: 1-butanol (E1)

carbocation rearrangement

1-butene
trans-2-butene
cis-2-butene
Acid-Catalyzed Dehydration

Condition: 2-butanol (E1)
Base-Induced Dehydrobromination

Condition: 1-bromobutane (E2)
Base-Induced Dehydrobromination

Condition: 2-bromobutane (E2)

**Major product, most stable**

- trans-2-butene

**cis-2-butene**

1-butene
PASS

ALL THE TESTS