Welcome to Chem 36!!
Organic Chem Lab

Dr. Katie Masters
# Chem 36 Info...

<table>
<thead>
<tr>
<th>Section 101:</th>
<th>Monday/Wednesday</th>
<th>1:25 – 4:25 pm</th>
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<tbody>
<tr>
<td>Section 102:</td>
<td>Tuesday/Thursday</td>
<td>1:25 – 4:25 pm</td>
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<td>Section 103:</td>
<td>Tuesday/Thursday</td>
<td>6:30 – 9:30 pm</td>
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<table>
<thead>
<tr>
<th>Faculty in Charge</th>
<th>Dr. Katie Masters</th>
<th>Dr. Jackie Bortiatynski</th>
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<tbody>
<tr>
<td></td>
<td>Director, Organic Labs</td>
<td>Director, Instrumentation</td>
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<td></td>
<td>218 Whitmore Lab</td>
<td>211D Whitmore Lab</td>
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<tr>
<td></td>
<td>Phone: 863-3319</td>
<td>Phone: 865-2772</td>
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<tr>
<td></td>
<td><a href="mailto:kmasters@psu.edu">kmasters@psu.edu</a></td>
<td><a href="mailto:jackie@chem.psu.edu">jackie@chem.psu.edu</a></td>
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<tr>
<th>Office Hours</th>
<th>Wednesday, 10:00 am – 12:00 pm</th>
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<tr>
<th>Website</th>
<th><a href="http://courses.chem.psu.edu/chem36">http://courses.chem.psu.edu/chem36</a></th>
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Required Materials


Eye Protection – Eye Protection is required at all times in lab!

Organic Lab Equipment Kit

Combination or key lock

Lab apron and rubber gloves (optional)
Lab Assignments

First Half of Semester:

5 Technique Experiments
• Recrystallization & Melting Point
• Distillation & Boiling Point
• Liquid/Liquid Extraction
• Thin-Layer Chromatography (TLC)
• Column Chromatography

Second Half of Semester:

5 Synthetic Experiments
Assigned experiments; Download from the course website.

Throughout the Semester:

Spectral Unknown Experiment
Determine an unknown’s structure

Assessment:
PreLabs (30 pts) and Final Reports (100 pts)
For each experiment you run. Ch 10 has no PreLab.
Lab Quizzes (50 pts)
Quizzes 1-6
Final Exam (100 pts)
The Lab Experiments

Chapter 4: Recrystallization and Melting Point:
Recrystallization & MP of an Unknown
Recrystallization of Phthalic Acid
MP of Spectral Unknown (Ch 10 experiment)
Decolorization with Norit

Chapter 5: Distillation & BP:
Simple or Fractional Distillation of a Known Mixture
Microscale Fractional Distillation of an Unknown Mixture

Chapter 6: Liquid/Liquid Extraction:
Separation of a Solid Mixture (acid/base chemistry)
Extraction of Caffeine from Tea
The Experiments

Chapter 7: Thin-Layer Chromatography:
  Finding a TLC Mobile Phase
  Identifying Compounds in an Analgesic Tablet

Chapter 8: Column Chromatography:
  Oxidation of Fluorene to Fluorenone
  Column Chromatography of Reaction Mixture

Chapter 9: The Synthetic Experiments:
  5 Synthetic Experiments - downloaded from web

Chapter 10: Spectral Unknown Experiment:
  Acquire mp, IR and 1H NMR spectra
  Determine the structure of an unknown
PreLab Assignments

Purpose of PreLabs & Lab Quizzes: To ensure that each student has read through and understands the theory and procedures of each experiment.

Revised PreLab Format

4 Sections:
1. Purpose of Experiment
2. Diagrams of Special Apparatus/Reactions
3. Chemical Data Table
4. PreLab Exercise

PreLabs are hand-written into the Lab Notebook. White pages are handed in for grading. The yellow copies are kept within the notebook.

NOTE: You are no longer required to do Procedure Outlines!!!!!
Download new PreLab grade sheets from website!!
PreLab Sections: Purpose of Experiment

• A concise, brief statement (one to two sentences) that describes the purpose of the experiment.
• Never use the first person!
• Do not include set-up of apparatus in this section.

Good Example:
To sublime (purify) an unknown solid and to take a melting point of the purified unknown to determine its identity.

Bad Example:
I will take a melting point.
PreLab Sections: Diagrams of Special Apparatus and/or Reaction Equations

Diagrams of Special Apparatus:
• Hand-draw and label any piece of glassware/equipment or reaction set-up that has not been used in previous experiments.
• Please draw neatly!

Reaction Equations:
• You will start including reaction equations with Chapter 8.
• Indicate limiting reagent and include the theoretical yield of the product.


**PreLab Sections: Chemical Data Table**

1. **Common Shelf Chemical Data Table:**
   - Located in notebook
   - Fill out completely and hand in with Chapter 5 PreLab

2. **Blank Chemical Data Tables:**
   - Located in notebook
   - Include chemicals for the experiment that do not appear on Common Shelf Chemical Data Table
   - Fill out all information about each chemical (see example in Ch 3).
   - Waste disposal information can be found in chapter 2 of the lab guide.

Use the *Aldrich Catalog* to find information about chemicals. When using Aldrich, pick the chemical entry that does NOT have isotopic labeling, e.g., $d$, $^{15}N$, $^{13}C$. These are more expensive and will have different FWs. Aldrich will also tell you if a chemical is flammable or toxic or an irritant.

Typically, use the last entry that gives all necessary data for the table. Use the “least pure” entry for the price.
PreLab Sections: PreLab Exercise

Answer the three assigned questions located in the lab guide.

Assignment sheet is handed out during check-in.
Lab Quizzes

Purpose: To test your understanding of theory and your preparedness for the experiments.

Quiz 1: Chapter 4  
Quiz 2: Chapter 5  
Quiz 3: Chapter 6  
Quiz 4: Chapter 7  
Quiz 5: Chapter 8  
Quiz 6: Chapter 9 and spectroscopy/spectral interpretation (Ch 10 & 11)

Please note schedule for dates of quizzes. They will be given at the beginning of lab and will take 10-15 minutes to complete.

Each are worth 50 points. Graded quizzes are not allowed to leave the lab.

Quizzes 1-5 will include questions on the specific procedures of the chapters' experiments.
Final Report Assignments

5 Sections:

I. Introduction
II. Procedure and Data/Observations
III. Results/Discussion/Conclusions
IV. PostLab Exercise
V. Reference

Attach the white in-lab notebook pages. You must have your TA initial all pages used during each lab session. Attach annotated spectral data when appropriate. Attach grade sheet to the front.

Final reports are computer-generated. Reaction schemes can be hand drawn.

Graded final reports cannot be taken out of the lab (except Ch 4).
Final Report Sections: Introduction

First part:
Theory, significance of technique, reaction, or compound

Second part:
• Brief, concise description of experiment.
• Mention techniques or analyses used.
• Mention the name(s) of compound(s), starting material or product.
• Do NOT include procedural detail!!
Final Report Sections: Procedure and Data/Observations

- Paragraph format

- Use your in-lab notebook pages (carbonless copy pages) to write this section

- Type up the procedure and include data and observations when appropriate
Final Report Sections: Results/Discussion/Conclusion

1. Presentation of results:
   - Calculations (yield, $R_f$ values, etc)
   - Highlight data

2. Discussion of results:
   - Evaluate the results
   - Compare your results to expected results
   - Talk about spectral data (if applicable)

3. Conclusions:
   - Comment on the success of the experiment
   - Make suggestions for improvements or future studies
Final Report Sections: Reference

Referencing the Lab Guide:


Referencing a Synthetic Experiment Handout:

http://courses.chem.psu.edu/chem36, “Oxidation of Borenol to Camphor, Experiment 125”
Lab Assignments: PreLabs and Final Reports

• PreLabs must be handed in BEFORE running the experiment. Please write them neatly!

• Late work is subject to a 10-point deduction the first day late and a 5-point deduction each additional day.

• See Chapter 3 (Lab Guide) for complete details on PreLabs and Final Reports.

• Course website will have new examples of final reports very soon.

• Grade sheets are to be attached to the front of the PreLabs and Final Reports; use them as a guide to include all important sections. PreLab grade sheets (revised) are on the web; final report grade sheets are at the end of the chapters.

• Graded final reports and quizzes are not to be taken out of the lab. They will be kept in a secure location until the end of the semester when they will be shredded.
Final Exam

• Worth 100 pts; cumulative

• Not so much theory, but application.

• Example question: Two layers form in a separatory funnel during extraction. You are unsure as to which layer is the organic and which layer is the aqueous. Without using densities, what can you do to determine which layer is aqueous?
Breakdown of the Chem 36 Points

5 Technique Experiment PreLabs 120 points*
5 Technique Experiment Final Reports 400 points*
5 Synthetic Experiment Prelabs 150 points
5 Synthetic Experiment Final Reports 500 points
Spectral Unknown Determination 100 points
TA Evaluation 100 points
Quizzes 300 points
Final Exam 100 points
Maximum Lab Points 1770 points

*Chapter 4 PreLab and Final Report do not count towards the final grade
Important Safety Rules

• Always wear eye protection when in the lab!

• Use gloves when appropriate; wash hands often.

• All chemistry must be done in the fume hoods.

• Your TA must be present when you are in the lab.

• If you wear shorts that are above the knee, you must wear a lab apron.

• No bare midriffs or open-toed sandals.

• Report accidents immediately to your TA.

• Keep coats and book bags on the shelves above your desk – keep them out of the aisles!
• Return all reagent and solvent bottles to their proper place on the side Shelves or in the refrigerator immediately!

• Keep your area clean. Your TA will assign one person to do a general Lab clean-up after each lab session.

• Show up prepared and on time.

• Keep the Instrument Room clean (room 206 Whitmore).

• Avoid floods.

• Keep balances clean!

• Treat everyone with respect.
Instrument Room

- NMR Spectrometer
- IR
- UV/Vis
- RI (refractive index)
- Polarimeter
- GC
- GC-MS

Chem 35 & 36 TAs will hold their office hours in this room. Instrument Room hours will be posted on the door.

Hours:
M, W, F: 1:30 pm - 5 pm
T, R: 9 am - 9:45 pm

**You are required to get all spectra signed and dated by a TA in the Instrument Room as soon as you acquire them.**
Check-In

• Look for sheets posted by the doors to 205 & 215 Whitmore – they contain all students’ desk assignments and your TA’s name.

• If your name isn’t on the list and you are registered, wait by the Stockroom and I will place you at an empty desk.
Next Lab

• Chapter 4 PreLab

• Quiz 1 – read Chapter 4 thoroughly!!