

**CHEM 201**  
**PURIFICATION AND CHARACTERIZATION**  
**LABORATORY I**

**AY 2012-2013**

## Table of Contents

<b>Calendar</b>	2
<b>Laboratory Overview</b>	3
<b>Grading/Reports Overview</b>	4
<b>Strategies for Chemistry Lab</b>	5
<b>Cycle 1: Check-In, Moodle, Safety, ACS Journals</b>	6
<b>Faculty- Student Meeting</b>	7
<b>JOC Summary</b>	7
<b>Pure Sample</b>	8
<b>Solvent Impurity</b>	13
<b>Weighing and Filtration</b>	17
<b>Projects</b>	
Liquid-liquid Extractions	23
Fractional Distillation and Gas Chromatography	29
Acid-Base Extractions	36
Recrystallization	44
Sublimation	53

## Calendar

## LABORATORY OVERVIEW

This semester is focused on purification and analysis techniques used in chemistry laboratories.

- The first 3 cycles you will be introduced to IR,  $^{13}\text{C}$  NMR, and  $^1\text{H}$  NMR spectroscopy, setting up samples for analysis, weighing and filtering.
- You have five small projects to complete this semester. Each project involves purifying a mixture using a specific technique and then analyzing your results for yield and purity.

**In cycle 1**, you will be introduced to the chemistry lab, complete the pure sample unknown lab, and complete spectroscopy workbook pages on short hand structure drawings and IR spectroscopy.

Safety quiz on Moodle must be completed before cycle 2.

**In cycle 2**, you will complete the solvent impurity unknown lab, interpret your pure  $^{13}\text{C}$  NMR and  $^1\text{H}$  NMR spectra, and work on  $^{13}\text{C}$  NMR and  $^1\text{H}$  NMR spectroscopy workbook pages.

Pure sample IR analysis is **due** at the beginning of lab. You must also show that you have your pure  $^{13}\text{C}$  NMR and  $^1\text{H}$  NMR spectra (print and process them); you must have printed the *JOC* and *Organometallics* solvent impurity NMR papers and put them into the lab manual; completed the Solvent Impurity prelab.

**In cycle 3**, you will complete your weighing and filtration unknown lab, and work on combined spectroscopy workbook pages. Your pure sample lab report is **due** at the beginning of the lab period.

**In cycle 4**, your solvent impurity and weighing and filtration lab reports are **due** at the beginning of the lab period.

**In cycle 6**, Faculty - student meetings; your JOC summary is **due**.

**In cycles 4-5; 7-11**, you will complete five projects. You can choose to do the projects in almost any order, with the following restriction:

The liquid-liquid extraction needs to be completed before acid-base extraction.

**In cycle 8**, your first two lab reports are **due** at the beginning of the lab period.\*\*

**In cycle 12**, your final three lab reports are **due** at the beginning of the lab period.\*\*

\*\* Two bonus points for turning a lab report in one cycle (or more) early.

## GRADING/REPORTS OVERVIEW

### Preparation, Safety and Clean-up

For every lab period, you will receive **up to 2 points** for **each** of the following things:

1. Completing the Moodle prelab exercises, **due before the beginning of lab.**
2. Flow-chart/plan for your lab procedure for that day; **due at beginning of lab.**
3. Physical constants and safety information; **due at beginning of lab:**
  - For safety information, see [www.msdssearch.com](http://www.msdssearch.com) for MSDS (Material Safety Data Sheets) links.
  - Solubility data is easy to find in the CRC Handbook of Chemistry and Physics or the Merck Index (Reference section of either library) or the Dictionary of Organic Chemistry (available in the Reference section at Clemens library).
  - Densities, melting points/boiling points and structures can often be found at ChemBioFinder Database <http://ChemBioFinder.com> or Chemexper Chemical Directory <http://www.chemexper.com/>
4. Laboratory Attitude: efficient, organized, independent
5. Lab Notes
6. Lab Bench and drawer cleaned up before leaving

Points will not be awarded after the end of the lab period. Plan accordingly and make sure you are already *cleaning up at least fifteen minutes before* the end of the period.

### Introduction to Spectroscopy Reports- more details in *Report Format tab*

1. Lab report forms included in the manual.
2. All data attached with analysis in a neat table (may be handwritten; spectral tables **must** be on the spectrum for easy comparison with data). *Examples in report format*
3. A list of references used in American Chemical Society (ACS) style. This includes a reference for the *experimental procedure* (Padias, lab manual or journal article), references for *previously reported physical constants* and/or *spectral databases* and references on any *background information* used in the introduction.

### Project Reports- more details in *Report Format tab*

The 5 reports for the projects will be in the same format:

1. Lab report forms included in the manual.
2. All data attached with analysis in a neat table (may be handwritten; spectral tables **must** be on the spectrum for easy comparison with data). *Examples in Report Format*
3. An experimental written in the style of the American Chemical Society (ACS). See *Report Format tab*.
4. A list of references used American Chemical Society (ACS) style. This includes a reference for the *experimental procedure* (Padias, lab manual or journal article), references for *previously reported physical constants* and/or *spectral databases* and references on any *background information* used in the introduction.