

## Syllabus, Chemistry 233 02, 04, 06-091

*Disclaimer: this syllabus is tentative and may be subject to change if circumstances beyond my control require it.*

### Contact Information

Instructor name: Buddy Glen Barnett

Room of class: CNSB 243 (pre-lab) and 242

Time of class: 2:00-5:00 p.m.

M (02), T (04), R (06)

Instructor's office: CNSB 206

Office hours: see below

Office phone: 318-342-1839 and Cell Phone 318-243-1181

Instructor's e-mail: [bbarnett@ulm.edu](mailto:bbarnett@ulm.edu)

### Course Prerequisites/Corequisites

You must have passed, or currently attend, Chem 232. If you drop Chem 232 you need **special permission** to stay in Chem 233. Please review our drop policy: [http://www.ulm.edu/chemistry/courses/drop\\_policy.html](http://www.ulm.edu/chemistry/courses/drop_policy.html)

### Course Description

You will attend ten laboratory sessions, consisting of pre-lab, quiz and laboratory experiment. In addition, you will attend check-in (today), check-out, and a final exam. See attached pages for details. **You are expected to work alone!**

### Course Topics, Objectives and Outcomes

Please review the attached list of experiments for specific topics covered in this course. These topics refer to the laboratory text (Williamson, Macroscale and Microscale Organic Experiments). You will carry out ten chemistry experiments targeting ten specific topics crucial to the understanding of organic chemistry, evaluate their results by completing written laboratory reports, and demonstrate your understanding of the practical and theoretical aspects of these experiments. See attached experiment list for specifics!

### Instructional Methods and Activities

All experiments have to be carried out exclusively by you; no partner is required or permitted. See attached experiment list for specifics!

Dress code: No exposed skin from the waist down, no ultra-thin fabrics. You must wear closed, sturdy, heeled shoes that protect your feet adequately (tennis shoes are a good choice). You must wear eye protection **whenever** you are in the lab. Make sure your clothing and hairstyle is compatible with a lab environment. If you violate the dress code you will be sent home. You can easily avoid this situation by keeping some spare shoes and pants handy.

### Evaluation and Grade Assignment

Tests, quizzes: Each quiz will cover mostly material from the **previous** pre-lab, as well as the book chapter corresponding to that day's experiment. Also, expect one simple, applied question relating to **today's** experiment. Bring a calculator for your quizzes and final! Quiz dates are attached.

Readings: You are expected to read your hand-outs and any assigned book chapters for the day's experiment **in advance**. Be prepared to answer questions related to the hand-outs. If you have any questions as to which material you will have to review it is your responsibility to ask.

Homework assignments: Lab reports are due one week after each experiment and have to be completed at home if necessary. I expect all lab reports to be entered into a carbon copy lab notebook.

Grading system: Your grades will be determined based on the following:

|  |                   |
|--|-------------------|
| 10 quizzes (20 pts each)                       | 200 points        |
| 10 experiments (lab reports worth 10 pts each) | 100 points        |
| 1 final  | <u>100 points</u> |
|  | 400 points total  |

Grades will be assigned based on the usual scale (A, 100-90%; B, 89-80% etc.). There will be no curving and you cannot drop any grades.

Mid-term grades: ULM requires that I post mid-term grades for you by March 6. These are merely designed to give you feed-back on your standing in class. **Mid-term grades indicate a student's status at mid-semester only and do not indicate the final performance outcome of a student!**

### **Class Policies and Procedures**

Textbooks and materials: Williamson's *Microscale and Macroscale Organic Experiments* and any material covered by the hand-outs. Other required material: Model Kit, calculator, eye protection, proper dress code, carbon copy lab notebook.

Attendance policy: You must be present for the entire duration of all pre-labs and experiments. Any unexcused absences will result in 0 points earned for the missed report and quiz.

Make-up policy: You may miss one experiment as a consequence of an excused absence, but generally must make this up on the assigned day (see attachment). Any misses require a written excuse (e.g. from your physician).

Academic integrity: If you cheat, you will get zero points on the exam or quiz. This may happen to you even if you are a cooperating "benefactor": it is in your interest not to cooperate. If you are caught cheating on the final you will be 100 points short and usually will not be able to pass.

Course evaluation policy: Please complete the on-line course evaluation!

Student Services: Please note that the following ULM student services are available to you: Student Success Center (<http://www.ulm.edu/cass/>), Counseling Center (<http://www.ulm.edu/counselingcenter/>), Special Needs (<http://www.ulm.edu/counselingcenter/special.htm>).

Discipline, course specific policies: If your cell phone rings you may face a penalty (typically, a point loss). Only **simple** (non-programmable) calculators are allowed or needed during exams.

Office hours and review sessions: Office hours are intended to discuss problems with your schedule, allow you to see your quizzes, etc. You may attend my 233 review sessions if you have any problems with the theory. In practice, there usually is plenty of time to discuss your questions in lab.

Posting of exam results: Quiz results will be posted Moodle..

Lab reports will be due the following lab, your grade will be based on the number of labs offered to you.

**Come prepared when we meet again! By next time, you should** a) follow the dress code, b) have your goggles or safety glasses, c) be prepared for your first quiz remember a calculator, d) bring your carbon copy lab note book, e) bring your lab text book (Williamson's), f) bring or remember your combination.

Lab Schedule for  
Organic Chemistry 233

| #       | M | T | R | Chapter          | Title                                   |
|---------|---|---|---|------------------|---|
| 0       |   |   |   | 1 & 2 (p 1 & 23) | Introduction, Safety, Grading, Check-in |
| 1       |   |   |   | 17 (p 333)       | S <sub>N</sub> 1 & S <sub>N</sub> 2     |
| 2       |   |   |   | 48 (p 661)       | Diels-Alder                             |
| 3       |   |   |   | 28 (p 431)       | Electrophilic Aromatic Substitution     |
| 4       |   |   |   | Notes            | Nucleophilic Aromatic Substitution      |
| 5       |   |   |   | 54 (p 709)       | Oxidation of Benzoin to Benzil          |
| 6       |   |   |   | 55 (p 716)       | Hydride Reduction of Carbonyls          |
| 7       |   |   |   | 40 (p 554)       | Fischer Esterification                  |
| 8       |   |   |   | Notes            | Acetanilide                             |
| 9       |   |   |   | Notes            | Nitrile Hydrolysis                      |
| 10      |   |   |   | 37 (p 521)       | Dibenzalacetone                         |
| 11      |   |   |   |                  | Check-out (Make-up Lab: if needed)      |
| Final   |   |   |   | All              | Final Exam                              |
| Make-up |   |   |   | 47 (p 653)       | Orange II                               |

### **EXPERIMENT 1: S<sub>N</sub>1 vs S<sub>N</sub>2**

*Note: Silver nitrate is extremely sensitive to traces of halides, all test tubes must be thoroughly rinsed with d.i. water!*

1. Place 0.1 ml (that corresponds to 2-3 drops, not more!) of each of the alkyl halides in a test tube.
2. To each, rapidly add 1 ml of 18% NaI/acetone solution.
3. Note the time of the first formation of any precipitate.
4. If no precipitate has formed in 5 minutes, place the tubes in a 50°C water bath for six minutes noting the time of the first formation of any precipitate.
5. Rinse the tubes with ethanol and repeat step 1.
6. To each, rapidly add 1 ml of 1% AgNO<sub>3</sub>/ethanol solution.
7. Note the time of the first formation of any precipitate.
8. If no precipitate has formed in five minutes, place the tubes in a 50°C water bath for six minutes noting the time of the first formation of any precipitate.
9. Don't forget to return your test tube rack!

The following compounds will be handed out: chlorobenzene, 3-chloro-1-propene, 1-chloro-2-methylpropane, 2-chloro-2methylpropane, 1-chlorobutane, 2-chlorobutane.

### **EXPERIMENT 2: DIELS-ALDER**

1. Combine 200 mg of anthracene, 110 mg of maleic anhydride, and 2.5 ml of xylene into a 5 ml round-bottomed flask. *Make sure your equipment is dry!*
2. Attach a reflux condenser and heat the mixture for 30 minutes. Wrap your condenser with damp piece of paper towel and check periodically so your solvent does not evaporate!
3. Cool the solution to room temperature with running tap water and then to 0 °C with an ice bath.
4. Isolate the crystals by filtration and determine the melting point, actual and percent yield during the next lab.

### EXPERIMENT 3: ELECTROPHILIC AROMATIC SUBSTITUTION

1. Combine 0.6 ml of concentrated sulfuric acid and 0.30 grams of methyl benzoate in a reaction tube. Mix well.
2. Cool the solution to 0 °C and add dropwise a mixture of 0.2 ml of concentrated sulfuric acid and 0.2 ml of concentrated nitric acid while in the ice bath. Use a stirring rod or thermometer to mix the solutions after each drop.
3. Warm the mixture to room temperature after addition and, after fifteen minutes, pour the reaction onto about 2.5 grams of ice.
4. Isolate the solid by filtration. *Make sure your product is solid, this may require seeding!* Using a pipette, wash the product on the filter with 1 ml of water followed by approx. 0.2 ml of methanol. **No recrystallization should be performed.**
5. Isolate the crystals by filtration and determine the melting point, actual and percent yield during the next lab.

### EXPERIMENT 4: NUCLEOPHILIC AROMATIC SUBSTITUTION

1. Place 3 ml of ethanol, 0.2 grams of 1-chloro-2,4-dinitrobenzene, and 0.2 ml of aniline in a 5 ml round bottomed flask, and reflux for 30 minutes. Wrap your condenser with damp piece of paper towel and check periodically so your solvent does not evaporate. Significant solvent losses should be compensated for by adding ethanol to restore the original volume. Towards the end solids may precipitate, this is normal.
2. Cool the solution in ice, and vacuum-filter the red crystals. This is somewhat challenging, you will have to use a bent spatula to dislodge the solids in your flask and return the filtrate several times to recover most of your product.
3. Recrystallize the product from approximately 12 to 14 ml of ethanol.
4. Determine the melting point, actual and percent yield during the next lab.

### **EXPERIMENT 5: OXIDATION OF BENZOIN TO BENZIL**

1. Combine 1.0 gram of benzoin and 3.5 ml of concentrated nitric acid in a 50-ml Erlenmeyer flask.
2. Place an aspirator tube in the opening of the flask and heat in a boiling water bath for 11 minutes.
3. While hot add 20 ml of water to the reaction and allow it to cool to room temperature.
4. Filter the solid, wash it on the filter with 1-2 ml water, and recrystallize the solid from ethanol and water: (heat with 5 ml of ethanol until all dissolves, then add water **dropwise** until the solution barely becomes cloudy when hot).
5. Collect the solid and allow it to dry until next lab period to determine the weight, yield, and melting point. (Average yield: 90%)
6. Save the product. It will be used as the starting material for **experiment 6**.

### **EXPERIMENT 6: HYDRIDE REDUCTION OF CARBONYLS**

1. Dissolve 0.5 grams of benzil in 5 ml of 95% ethanol with gentle heating.
2. Cool the solution to room temperature with tap water.
3. Add 0.1 grams of sodium borohydride.
4. After 10 minutes, add 5 ml of water, heat to boiling, and add another 10 ml of water.
5. Cool in an ice bath and filter.
6. Determine the melting point and yield of the compound next lab period. (Average yield: 90%)

### **EXPERIMENT 7: FISCHER ESTERIFICATION Benzocaine, ethyl-*para*-aminobenzoate**

1. Combine 0.27 grams of p-aminobenzoic acid, 3.0 ml of 100% ethanol, and **0.5 ml** of sulfuric acid in a 5 ml round-bottomed flask fitted with a reflux condenser. *Make sure your equipment is dry!*
2. Heat the reaction to a gentle reflux using an aluminum block for 45 minutes. Wrap your condenser with a damp piece of paper towel and check periodically so your solvent does not evaporate. Significant solvent losses should be compensated for by adding ethanol to restore the original volume.
3. Cool the solution to room temperature and pour the reaction into 10 ml of cold 10% Na<sub>2</sub>CO<sub>3</sub> with stirring (foaming!).
4. After standing in an ice bath, a solid should form which can be collected by vacuum filtration.
5. Allow the solid to dry until next lab period to determine the melting point, the actual, and percent yields.

### **EXPERIMENT 8: ACETANILIDE**

1. Combine 1.2 ml of aniline, 30 ml of water and 1.2 ml of hydrochloric acid in a 50 ml Erlenmeyer flask. You may see white fumes forming and some solids precipitating. This is normal. **Stir until oil becomes missible.**
2. Dissolve 1.42 g of sodium acetate in 6 ml of water in a separate container.
3. Add 1.7 ml of acetic anhydride to the anilinium hydrochloride solution, and immediately add the sodium acetate solution.
4. Swirl until precipitation begins, then allow standing at room temperature for 10 minutes.
5. Cool the slurry in an ice bath for 10 minutes and vacuum filter.
6. Recrystallize the solid from water and collect by vacuum filtration.
7. Allow the crystals to dry until the next lab to determine melting point, actual, and percent yield.

## EXPERIMENT 9: NITRILE HYDROLYSIS

1. In a 5-ml round bottom flask (long neck), place 2.5 ml of 10% sodium hydroxide, 0.25 ml of benzonitrile, and a stirring bar.
2. Fit the flask with a reflux condenser and heat the solution to boiling for 40 minutes. Wrap your condenser with damp piece of paper towel and check periodically that the volume does not change. Every 10 minutes, place a wet strip of pH paper in the top of the condenser to monitor ammonia evolution (pH should change to basic).
3. Allow the flask to cool and transfer the solution to a 10-ml Erlenmeyer flask.
4. Cool the flask in an ice bath and add 2.0 ml of concentrated hydrochloric acid.
5. Collect the solid by vacuum filtration and recrystallize it from 3 ml of water.
6. Allow the solid to dry until next lab period to determine the melting point, the actual, and percent yields. (Average yield: 70%)

## EXPERIMENT 10: DIBENZALACETONE

1. Combine 2 ml of 3 M sodium hydroxide, 0.212 g of benzaldehyde, and 1.7 ml of a 3.7 % (w/v) solution of acetone in ethanol in a reaction tube (one of the small tubes in your kit, **not** a regular test tube!).
2. Place a septum on the tube and shake the solution vigorously.
3. Shake the mixture intermittently for the next 30 minutes.
4. Cool the slurry in an ice bath and vacuum filter.
5. Wash the solid three times with 3 ml each of water.
6. Allow the solid to dry until next lab period to determine the melting point, the actual, and percent yields.

**MAKE-UP: Orange II (1-p-Sulfobenzeneazo-2-naphthol, sodium salt)  
(handed out as needed)**

**MAKE-UP:**

**Orange II (1-p-Sulfobenzeneazo-2-naphthol, sodium salt)**

**Diazotization of amines, azo dyes)**

**(modified from Williamson, Ch. 47)**

Needed :

Sulfanilic acid monohydrate

anhydrous sodium carbonate

sodium nitrite

hydrochloric acid

2-naphthol

sodium hydroxide solution, 3M

sodium chloride

Note: use disposable pipettes to measure volumes. They are graduated, total volume usually 1 mL (verify this volume!).

1. *Preparation of diazotized sulfanilic acid:* In a 10 mL beaker or Erlenmeyer flask with magnetic stirring, combine 0.24 g sulfanilic acid monohydrate, 2.5 mL water, and 0.07 g anhydrous sodium carbonate. Heat mixture to boiling or until all is dissolved. Chill in an ice bath, then add 0.2 g of sodium nitrite and stir for 3 min with a glass rod. Add 1.5 mL water, then 0.25 mL concentrated hydrochloric acid. Keep in ice bath. Stir for 3 more minutes.

2. *Preparation of Orange II:* In a 50 mL beaker, combine 1 mL of 3M sodium hydroxide and 0.18 g 2-naphthol. Swirl until a solution has formed. Transfer *to* (not the other way around!) this solution, with stirring, the solution prepared in step 1. Stir the resulting orange paste for 2 min, then heat on a hot plate with stirring to dissolve everything. Add 0.5 g sodium chloride to the hot solution, stir for an additional 2 min, chill in an ice bath and collect product by vacuum filtration.

## CHEMISTRY 233--Desk and Kit Checklist

Student Name (typed) \_\_\_\_\_

Student ID \_\_\_\_\_

Section # \_\_\_\_\_ Room # \_\_\_\_\_ Desk # \_\_\_\_\_ Combination \_\_\_ - \_\_\_ - \_\_\_

All replacement parts for the drawer are found in Room 122.

| Item  | Quantity |
|---|----------|
| Beaker, 250 ml                              | 1        |
| Beaker, 100 ml                              | 2        |
| Beaker, 50 ml                               | 2        |
| Cork, fits 50 mL E. flask                   | 2        |
| Flask, Erlenmeyer, 50 ml                    | 2        |
| Organic Kit*                                | 1        |
| Stirring rod, 6 inch                        | 1        |
| Test Tubes, 15 x 125 mm (or multiple sizes) | 8        |
| Thermometer                                 | 1        |

Organic Kit\*: Consult page 10 in the Organic Lab textbook (Williamson) for a listing of the equipment. All replacement parts for the kit are found in Room 240A.

### Shared equipment:

|   |                    |
|---|--------------------|
| 1 | Hot plate          |
| 1 | Heating block      |
| 1 | Rod                |
| 1 | 90° clamp          |
| 3 | Three prong clamps |
| 1 | Ring clamp         |
| 1 | Lab jack           |
| 1 | Plastic bowl       |
| 1 | Test tube rack     |

## SAFETY AGREEMENT

I, \_\_\_\_\_ (Print Name), have read, understand, and agree to the laboratory Safety Rules. I, therefore, release the Department of Chemistry at ULM and my instructor from any responsibility for accidents occurring while not following those rules.

\_\_\_\_\_ Signed \_\_\_\_\_ Dated