

Syllabus, Chemistry 2033-60249, -60251 Spring 2012

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

Contact Information

Instructor name: Dr. Thomas Junk

Room of class: CNSB 244 (pre-lab) and 240

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

Mo (-60249), Tu (-60251)

Instructor's office: CNSB 112

Office hours: see below

Office phone: 342-1830

Instructor's e-mail: junk@ulm.edu

Website: <http://www.ulm.edu/~junk/home.html>

My schedule	Mo	Tu	We	Th	Fr
8:00-8:50 a.m.	2032-60247		2032-60247		2032-60247
9:00-10:00 a.m.	Office hr		Office hr		Office hr
10:00-10:50 a.m.		Office hr		Office hr	
2:00-5:00 p.m.	2033-60249	2033-60251	2031-60245		
5:00-6:00 p.m.			2032 Review		

Course Prerequisites/Corequisites

You must have passed, or currently attend, Chem 2032. If you drop Chem 2032 you need **special permission** to stay in lab. Please review our drop policy: 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 (no partners)!**

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 are compatible with a lab environment. If you violate the dress code you will be sent home. You can easily avoid this situation by keeping a set of spare shoes, socks and pants in your drawer.

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 **the same day'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 grade will be based on 520 points total:

10 quizzes (15 pts each, 2 can be dropped except safety)	120 points
10 experiments (lab reports worth 25 pts each)	250 points
1 final	150 points

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 labs.

Mid-term grades: ULM requires that I post mid-term grades for you by March 8. 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 your final grades!**

Class Policies and Procedures

Textbooks and materials: Klein's *Organic Chemistry* will help you with the theory. Procedures contained in this syllabus and pre-labs will cover practical aspects. 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 150 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 2033 review sessions if you have any problems with the theory. In practice, there usually is plenty of time to discuss your questions during lab period.

Posting of exam results and answer keys: Quiz results will be posted next to my door, according to your student ID number. Copies of quizzes will be posted.

Cancellations: Cancellations are rare. Cancelled labs should be treated as labs that were never scheduled: If your lab is cancelled, your next quiz will cover the last pre-lab before the cancellation, your 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! Please a) follow the dress code, b) have your goggles or safety glasses, c) be prepared for your first quiz, d) bring your carbon copy lab note book, e) bring or remember your combination, f) print the procedures.

Lab Schedule
Organic Chemistry 2033

#	M	T	R	Chapter	Title
	1/23	1/24	1/26	(pre-lab)	Introduction, Safety, Grading, Check-In
1	1/30	1/31	2/2	(pre-lab)	Thin Layer Chromatography
2	2/6	2/7	2/9	8	Elimination
3	2/13	2/14	2/16	13 (Fig. 13.11)	Alcohol Oxidation
4	2/27	2/28	3/1	13 (Fig. 13.11)	Borohydride Reduction
5	3/5	3/6	3/8	17	Diels Alder
6	3/12	3/13	3/15	19	Electrophilic Aromatic Substitution
7	3/19	3/20	3/22	19	Nucleophilic Aromatic Substitution
8	3/26	3/27	3/29	21	Nitrile Hydrolysis
9	4/2	4/3	4/5	21	Esterification
10	4/16	4/17	4/19	22	Aldol Condensation
Final	4/23	4/24	4/26		Final Exam
Make-Up	4/30	5/1	5/3	(hand-out)	Check-out/make-up

Note 1: The chapters listed refer to the lecture textbook, David Kleins's *Organic Chemistry*.

Note 2: You will find an abundance of information about the concepts covered on check-in day and for experiments 1–6 on the internet. Several links are provided below as examples, there are many more to be found without difficulty.

Safety review, examples of links (there are many!)

<http://orgchem.colorado.edu/safety/labsafety.html>

http://go.hrw.com/resources/go_sc/gen/HN2SAFET.PDF

Thin Layer Chromatography, examples of links (there are many!)

<http://www.files.chem.vt.edu/chem-ed/sep/tlc/tlc.html>

<http://www.wpi.edu/Academics/Depts/Chemistry/Courses/General/tlc.html>

CHEMISTRY 2033--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 12 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

EXPERIMENT 1: Thin Layer Chromatography (TLC)

1. Obtain 2 TLC plates. Draw a **light** pencil line about 1 cm from the end of each chromatographic plate. (**Be careful to press lightly, otherwise you'll scrape though the coating**)
2. Spot one plate with your 4 known standards (acetaminophen, aspirin, caffeine, and ibuprofen) and a mixture of all four as a fifth spot. Spot the other plate with the 5 unknown commercial painkillers and a mixture of all four as a sixth spot.
3. Use a capillary tube for each standard and unknown solution. After spotting each standard or unknown **clean the capillary tube with acetone before inserting it into a new sample**. Make each spot as small as possible, preferably less than 0.5 mm in diameter. Examine the plate under the ultraviolet (UV) light to see that enough of each compound has been applied; if not, add more.
4. Prepare a developing chamber by using a large beaker as the chamber, a half-piece of filter paper inside, and evaporating dish to cover. Pour the eluting solvent, a 99:1 mixture of ethyl acetate:glacial acetic acid, into the beaker to a depth of approximately 1 cm. Place the prepared TLC plates in the developing chamber.
5. After the solvent has risen to near the top of the plate (about 1 cm from the top), remove the plate and mark the solvent front with a pencil. Keep the plates in the hood until the majority of the eluting solvent has evaporated from the plates. Examine the plate under UV light to see the components as dark spots against a bright green-blue background.
6. Outline the spots with a pencil and note anything distinctive about any of the compounds. The spots should then be visualized by putting the plates in an iodine chamber. The iodine chamber is pre-made and contains a few crystals of iodine in the bottom of a capped jar. More than 2 plates can be placed in the iodine chamber at one time. Remove the plates when a definite change in appearance takes place on your plates. Note which compounds stained with iodine and to what intensity. The iodine stains will dissipate over time.
7. Calculate the R_f values for each spot. Unknowns can be identified using R_f values.

You need to print the remaining procedures, to be found in the complete syllabus at <http://www.ulm.edu/~junk/coursestaught.html>

EXPERIMENT 2: CYCLOHEXENE (E1)

1. Combine 20.0 mL of cyclohexanol, 5.0 mL of phosphoric acid, and a magnetic stir bar in a 50-mL round bottom flask. (Note: cyclohexanol can freeze if the lab is cold. If it does, just warm the flask with hot water)
2. Assemble a simple distillation apparatus and distill the cyclohexene with stirring. Submerge the receiving flask in an ice bath.
3. Transfer the distillate to a separatory funnel secured with a ring clamp, allow the layers to separate, and remove the aqueous layer.
4. Wash with 15 mL of a saturated sodium chloride solution, allow the layers to separate, and remove the aqueous layer.
5. Transfer the organic layer to an Erlenmeyer and add ~5 g of sodium sulfate. Stopper well and let stand for ~ 5 min.
6. Carefully decant the liquid into a round bottom flask closed with a **well-greased** stopper and determine the percent yield. The remaining solid is waste – spent drying agent!

EXPERIMENT 3: OXIDATION OF BENZOIN TO BENZIL

1. Combine 1.0 gram of benzoin and 3.5 ml of previously prepared solution of ammonium nitrate, copper acetate, and 80% by volume glacial acetic acid/water solution in a 10 ml round bottom flask.
2. Attach an air condenser and heat on an aluminum plate to reflux for at least 30 minutes using a magnetic stir bar.
3. Remove the air condenser and pour the contents into a small beaker. Add 5 ml of cold water and place in ice bath. Collect crystals by vacuum filtration.
4. Recrystallize the solid using about 5 ml of 95% ethanol. Heat until all solid dissolves. Remove from heat.
5. Cool to room temperature, and place beaker in an ice bath to allow crystals to form.
6. Collect the solid by vacuum filtration (use ice cold water to rinse crystals), and allow it to dry until next lab period.
7. Determine the weight, yield, and melting point. Perform TLC on starting material and product.

8. Test product for unreacted benzoin. Dissolve about 100 mg of product in about 0.1 ml 95% ethanol. Add one drop 10% sodium hydroxide. Observe.
9. Save the product. It will be used as the starting material for experiment 6.

EXPERIMENT 4: 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. **Sodium borohydride is hygroscopic and moisture sensitive. Keep the storage bottle closed!**
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 5: 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 6: 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. Recrystallize from an equal volume of methanol.
5. Isolate the crystals by filtration and determine the melting point, actual and percent yield during the next lab.

EXPERIMENT 7: 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 8: 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. If it does, add water. 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 9: FISCHER ESTERIFICATION

1. Combine 0.27 grams of p-aminobenzoic acid, 3.0 ml of 100% ethanol, and 0.2 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 with a hot water bath for 45 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.
3. Cool the solution to room temperature and pour the reaction into 10 ml of cold 10% Na_2CO_3 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 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, 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.