

University of Rhode Island; Department of Chemistry
CHM 226: Organic Chemistry Laboratory
Odd Section Numbers – Beapre 245
Even Section Numbers – Beapre 260

Course Instructor Prof. Matt Kiesewetter
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Office Hours By appointment.

Textbook/Materials A carbon copy laboratory notebook must be used to record all data.

Experimental procedures and supplemental information will be provided to you via Sakai, but the textbook for the accompanying lecture course may be useful for preparing laboratory reports.

Safety goggles/glasses, lab coat, purple nitrile gloves. RAM account and card to purchase items in the chemistry stockroom. MNova and Chemdraw software (free downloads though URI Chemistry).

Access to the internet. **You are responsible for checking the Sakai site, coming to lab prepared to conduct the right experiment and checking your URI email regularly.**

Course Goals CHM 226 is an introduction to conducting organic research. The ultimate goal of the course is to provide students a basis by which they will be able to conduct a chemical reaction that they have never performed before. The 'real world' of organic chemistry involves performing reactions and making compounds that are unknown. The virtually-unlimited diversity of chemical structure originates at the fountainhead of a handful of lab and analytical techniques. By conducting specific named reactions and targeted technique instruction, this course will introduce students to a variety of skills that they may implement in their professional development and careers. You are about to embark on one of the most empowering courses in the undergraduate curriculum after which you will have the tools required to synthesize almost any chemical compound, including those that are unknown to human kind. Also, this material is on the MCAT.

Grading Evaluations are directly related to work performed in lab, and attendance is required. There are no makeup labs. All graded work should be performed individually.

Student grades will be based on written lab reports (11 reports, 100 points each); lab technique and citizenship (100 pts); and a lab practicum (100 pts).

Lab report format varies depending on the experiment, but each experiment has a report which is worth 100 points. The lowest lab report grade will automatically be dropped. The type of lab report varies depending on the experiment, see

Experiment List for the type of report required. The requirements and grading rubric for each report type is given below.

Lab technique and citizenship are important parts of safely conducting chemistry experiments in a shared space. Students will receive a demerit for exhibiting poor technique and/or citizenship. Each demerit will be documented and will result in the loss of 20 pts, so behave and be safe. Any demerits beyond 5 (100 points worth), will be deducted from the course points.

Demerit-worthy violations include:

1. Students engage in unsafe practices.
2. Students maintain an untidy work space and does not return the lab to its starting condition.
3. Students fail to follow the lab safety policies.
4. Being late to lab; important safety information is discussed at the beginning of the lab period.

No makeup labs are given for any reason.

Lab Practicum: The lab practicum is a combined written and hand-on exam. Any technique, method, experiment or mechanism discussed or used in CHM 226 is fair game for the exam.

Regrading: Students may request a re-grade on any evaluation for up to 1 week from when the evaluations are returned in lab. Requests for regrades must 1) be made in writing, 2) include the assignment being regraded, 3) clearly state the issue being disputed and 4) include a BRIEF rationale for overturning the initial grade. Regrade requests are to be submitted to Prof. Kiesewetter via the Department Office or his office. The whole assignment is subject to regrading.

Cheating or plagiarism on a graded assignment will result in a zero for that evaluation and referral to the Dean and possible failure of the course. Students are expected to follow the University policy of ACADEMIC HONESTY and all other University policies.

Due Dates

Lab reports are due at the beginning of the lab period one week after the experiment is completed. Some weeks will have more than 1 report due, so plan ahead.

All lab reports are due at the beginning of the lab period. No late lab reports will be accepted; late, in this context, is 10 minutes after the beginning of the lab period. Reports are to be performed alone with your own data unless instructed otherwise. Reports should be type written and use Chemdraw (or similar) for figures. Spectra can be assigned by hand.

Absenteeism

This is a laboratory course; there is no substitution for a missed lab. If a student must be absent for a legitimate reason, the missed experiment will be counted as their dropped grade. Further absences resulting in a missed experiment will result

in a grade of zero for that experiment. Students that miss more than 1 experiment will be advised to drop the course. If a student misses one day of a multiple day experiment, they should not expect to be able to finish the experiment in the other day(s). Excused absences must be cleared by Prof Kiesewetter 1 week in advance, minimum.

Students will not be admitted to the lab if they are 10 minutes late to lab; important safety information is discussed at the beginning of the lab period. That experiment will be scored as a zero.

Equipment

Students are responsible for the equipment in the drawer assigned to them on the first day of class. Any broken equipment must be replaced at the student's expense, this includes any shared/departmental equipment. Any student who has an unpaid bill with the chemistry stockroom will have a hold placed on their account which will prevent the student from registering in the future. Drawers must be checked out at the end of the semester or if a student chooses to drop the course. Improper or missed checkout results in a \$10 charge and a hold.

Experiment List

- 1- General Techniques[†]
- 2- ChemDraw, ACS Publications, SciFinder[†]
- 3- Recrystallization Techniques[†]
- 4- Bromination Experiment*
- 5- Chromatography[†]
- 6- NMR Lab[†]
- 7- Acylation of Ferrocene*
- 8- Suzuki**
- 9- Distillation[†]
- 10- Diels-Alder Experiment*
- 11- Reducing Benzil*
- 12- Dipeptide Synthesis**

[†]Post lab questions (plus notebook)

* Brief Lab Report for this experiment

** Formal Lab Report for this experiment

Experiment Schedule

week	day	Even Section Numbers	Odd Sections Numbers
1/23	T/W	Check-in and course policies	Check-in and course policies
	R/F	1-General Techniques	2- ChemDraw, ACS Publications, SciFinder
1/30	T/W	2- ChemDraw, ACS Publications, SciFinder	1-General Techniques
	R/F	3- Recrystallization Techniques	3- Recrystallization Techniques
2/6	all	4-Bromination Experiment	4-Bromination Experiment
2/13	T/W	5-Chromatography	6-NMR Lab
	R/F	5-Chromatography	5-Chromatography
2/20	T/W	6-NMR Lab	5-Chromatography
	R/F	7-Acylation of Ferrocene	7-Acylation of Ferrocene
2/27	T/W	7-Acylation of Ferrocene	7-Acylation of Ferrocene
	R/F	8-Suzuki	8-Suzuki
3/6	all	8-Suzuki	8-Suzuki
3/20	T/W	9-Distillation	9-Distillation
	R/F	10-Diels-Alder	10-Diels-Alder
3/27	T/W	10-Diels-Alder	10-Diels-Alder
	R/F	11- Reducing Benzil	11- Reducing Benzil
4/3	T/W	11- Reducing Benzil	11- Reducing Benzil
	R/F	12- Dipeptide Synthesis	12- Dipeptide Synthesis
4/10	all	12- Dipeptide Synthesis	12- Dipeptide Synthesis
4/17	T/W	12- Dipeptide Synthesis	12- Dipeptide Synthesis
	R/F	Lab Practicum	Lab Practicum
4/24	T/W	no lab	no lab
	R/F	Check-out day (last day of lab)	Check-out day (last day of lab)

T = Tuesday; W = Wednesday; R= Thursday; F = Friday

University Schedule

Feb 13 – Last day to drop class w/o designation

Feb 20 – President's Day (no class)

March 6 – Last day to drop a class

March 13-17 – Spring Break (no class)

Post Lab Questions (100 pts each)

1. *Post lab questions* (50 pts): answer all questions posed at the end of the lab experiment
2. *Lab notebook* (50 pts): copies of lab pages are provided and entries are made according to the guidelines.

Brief Lab Report (100 pts each)

These reports should be type written and use ChemDraw/MNOVA but are of a less formal nature than the *Formal Lab Reports*. No external referencing is usually required, but if it is done/required it must be done properly. Evaluation will be based on correctly and completely providing these sections:

1. *Descriptive Title/Purpose* (5 pts): a title/purpose of the experiment is given
2. *Reaction scheme* (10 pts): a reaction scheme and mechanism is given
3. *Analysis of Data* (20 pts): relevant data (including spectra) are provided in table or list form, assigned and analyzed
4. *Yield* is calculated (10 pts)
5. *Post lab questions* (30 pts): answer all questions posed at the end of the lab experiment
6. *Lab notebook* (25 pts): copies of lab pages are provided and entries are made according to the guidelines.

Formal Lab Report (100 pts each)

Reports should be type written and use ChemDraw (or similar) for figures. Spectra can be assigned by hand. Evaluation will be based on correctly and completely providing the sections below. A more detailed template is available on Sakai.

1. *Title and abstract with reaction scheme* (10 pts): Should include a ChemDraw reaction scheme and a short description of the experiment.
2. *Introduction* (15 pts): Give some historical perspective, applied use or recent development for the reaction. Needs to include properly-formatted references. This section is not about YOUR experiment, but puts your experiment in perspective.
 - a. *Reaction mechanism* (10 pts): correct reaction mechanism is drawn by you in ChemDraw.
3. *Experimental Section*:
 - a. A procedure is given (10 pts)
 - b. Relevant data in table or list form and a ChemDraw reaction is shown (10 pts).
4. *Discussion and Conclusion* (10 pts): give your observations, relevant spectra are provided and assigned, give suggestions for improving the experiment/yield/etc and justify them, yield is calculated
5. *Post-lab questions answered* (25 pts).
6. *Lab notebook* (10 pts): copies of lab pages are provided and entries are made according to the guidelines.

Keeping a Laboratory Notebook

Students must acquire a carbon copy laboratory notebook and maintain it according to the rules below. Students will be required to hand in a copy of their lab book entries for each experiment; hand in all pages. These copies should be affixed to the lab report. In short, a lab notebook should contain enough information that a person 'skilled in the art' could reproduce your experiments.

An example lab page is on Sakai.

Before coming to lab:

1. Name the experiment.
2. Place a chemical drawing of the reaction being performed at the beginning of the entry, if applicable.
3. For each reagent used, clearly give 1) the desired amount; 2) the amount used in grams/mL/etc; 3) (m)mol; and 4) (m)M; 5) formula weight and density (if applicable). These data will help as you go to write up experiments and helps trace errors later.
4. Write down a purpose or hypothesis statement. It can be difficult to remember why you did something 6 months after you do it.

During/after each experiment and in general:

1. Give a short synopsis of the experimental procedure and reference any literature that you are following (this can often be done before lab as well). Update your notebook with observations.
2. Errors should be crossed out with a single line and the new entry provided above/beside
3. Each experiment should receive its own page (or more than one page).
4. All spectra/data should be numbered (physically on the paper) and saved to the spectrometer/instrument so that they reference a specific notebook page. The sample should be numbered in the same format. Use the format: 'your initials'-'notebook number'-'page number' (ex. MK-1-19). The type of data collected and what was analyzed should be clearly labeled in the notebook.
5. Multiple analyses should be given letters. Example: If MK-1-19 is the crude material, the purified material could be called MK-1-19b. Consider giving complicated purifications their own notebook page/experiment.
6. Cross reference your pages, if applicable. For experiments that use more than one page, clearly label at the bottom of the first page where the second page starts (it may not always be on the next page).
7. Date your entries.
8. Calculate yield and give brief conclusions. Think about it as giving yourself pointers for 'next time' you do that reaction. Eg. "My yield may have been low because I [...]. Next time I would [...]."