

**Chemistry 332**  
**General Organic Laboratory**  
**Spring 2011**

**Instructor:** Dr. Dmitry Kadnikov (dkadnikov@niu.edu)

**TA:**

**Office Hours:**

**Meeting:**

**Location:** FR307

**TEXT:** Gilbert, John C, Martin, Stephen F, "Experimental Organic Chemistry", 4<sup>th</sup> Edition  
Before coming to the first class meeting make sure you have read:

- (a) First Aid in Case of Accident (opposite title page of the text)
- (b) All of chapter 1, with special emphasis on sections 1.8 and 1.9 (dealing with safety considerations)

**SAFETY:**

Safety is the number one priority in the organic laboratory, since many, if not all, organic chemicals are toxic. **ALL STUDENTS MUST WEAR EYE PROTECTION AND CLOSED-TOE SHOES AT ALL TIMES during the class. Long sleeves and leg coverings are also recommended.** Goggles are available free of charge at the chemistry stockroom, and they must be the type approved by the Chemistry Department. No food or drinks are permitted in the laboratory. Waste chemicals must be properly disposed of. Notify TA of any glassware breakage, chemical spills, or emergencies immediately. Your lab area, lab equipment, etc. must be cleaned prior to leaving the lab. Refer to the textbook for more comprehensive discussion of safety. The Department of Chemistry has a zero tolerance policy against safety violations, and points will be deducted for violating safety rules (see the section on grading).

Pregnant students should consult with their doctors regarding the risks of being enrolled in this and other laboratory classes.

**Class Attendance and General Advice:**

Regular attendance is essential for a successful and pleasant experience in the organic chemistry laboratory. Lab starts promptly at 9:30 am, so be on time. You have to attend every lab session unless you have a university-sanctioned excuse, of which you have to inform your TA in advance. If for some unforeseen reason you will not be able to attend lab, please see me as soon as possible or let Dr. Kadnikov know. **There will be no make-up labs**, and absence during a lab will result in a ZERO (0) for that experiment. Also note that some labs require multiple periods, so you must attend all of them to receive credit for the experiment.

**Prelab Preparation:**

You should read all the material in the textbook pertaining to the lab before showing up to class. There are no formal prelab questions to do for the laboratories, but there are post-lab questions, which you will answer after you have completed the experiment.

## Laboratory Notebooks

During the laboratory, your data should be recorded into a laboratory notebook (in ink), and **the data pages must be signed by the TA prior to your departure from lab**. Laboratory notebooks must be kept neat and must have **duplicate, numbered pages**. Regular paper notebook pages or composition notebooks are not acceptable. Lab reports should be in your own writing, and copied lab reports will receive a grade of zero. Even though you may conduct your experiments in pairs, the lab reports, including all calculations and answers to questions, should be prepared individually. The reports should be written neatly and legibly in black or blue ink. Pencils, white out, colored pens (other than black or blue) are not permitted or acceptable for your notebooks. Lab reports are due the following lab period after an experiment is completed, and multiple period labs will be due the next lab period after completion, as indicated in the tentative schedule. The following information should be included, of which parts 1-4 should be done before coming to lab, parts 5 and 6 during lab, and parts 7 and 8 after lab:

- 1) Name and Date
- 2) Experiment Title, e.g. "Recrystallization of Sulfonylamine."
- 3) Purpose. A brief summary of what you are trying to accomplish and/or learn from this experiment, (not just a restatement of the title). List methods, etc.
- 4) Data. You might want to include all the vital information that you should know prior to coming to lab. For example, if you are recrystallizing sulfonylamine, you would want to draw out the chemical structure and list the important physical properties, such as the melting point. The mechanisms of reactions (if you are running a reaction) must be shown in the lab notebooks and can be obtained from the lab manual. Include equations and quantities of materials needed.
- 5) Procedure. You are expected to write out a general procedure that you performed during the lab on that day. Also you should record your observations, such as color changes, formation or disappearance of a precipitate, evolution of heat or gas, etc.
- 6) Results. This is the location where you would record weights of the products, melting point ranges, etc. Theoretical and percent yields should be calculated if they pertain to the experiment. **Obtain your TA's signature at this point.**
- 7) Conclusions. You should write a brief but informative conclusion to the lab stating the results obtained and discussing the possible reasons for those results. You should mention possible errors, and how they could be avoided in the future. This may improve your technique for later experiments.
- 8) Answers to post-lab questions.

## **Report on spectroscopy**

There will be two classes on using the spectroscopic techniques to characterize organic molecules (IR on Tue, 3/1 and NMR on Tue, 3/8). During these classes you will learn the basics of each method and practice analyzing the spectra. After each of the classes you will use the techniques you have learned to characterize actual reactions you will have performed in the lab (IR for Exp.16.2 on Thu 3/3, and  $^{13}\text{C}$  NMR for Exp. 17.4 on Thu, 3/10). In your report you will summarize your knowledge on how IR and  $^{13}\text{C}$  NMR spectroscopy could be used to characterize chemical reactions. The details on preparation of the report will be announced in class.

## **Quizzes and exams**

There will be five pre-lab quizzes (see the tentative schedule) on the past material, mainly concerning experiments performed since the previous quiz. Lateness to a quiz will result in a zero for that quiz. There will be no make-up quizzes. There is no midterm exam, but, during the last class period, there will be a written final exam, consisting of multiple choice and short answer questions.

## **Grading**

Your grade in organic laboratory is largely based on the work done performing the assigned experiments and understanding the techniques/procedures. The lab reports will be graded based on the quality of your data, presentation of the results, and answers to post-lab questions. You have to prepare your report individually, and violation of this rule will result in zero points for both laboratory partners. Safety is an important part of any laboratory work; therefore, points might also be deducted for the following safety violations:

GOGGLES ARE NOT WORN AT ANY TIME (except pre-lab discussion)	Minus 20 points
CHEMICAL SPILL IS NOT CLEANED (at the balance, at your work area, in fume hoods)	Minus 10 points
FOOD/DRINKS IN LAB	Minus 5 points
IMPROPER WASTE DISPOSAL	Minus 10 points
DISPOSING OF GLASS IN WASTE BASKET (Use the "Glass Waste" container for broken glass)	Minus 5 points

The course grade will be assigned based on your point totals from the lab experiments and reports, quizzes, and the final. The point breakdown is as follows:

Lab Reports (22 at 40 points each)	880 points
Quizzes (5 at 15 points each)	75 points
Report on spectroscopy	15 points
Final Exam	130 points
Total	1100 points

Approximate Grading Scale:	A 92 – 100%
	B 83 – 91%
	C 74 – 82%
	D 65 – 73%

## Tentative Schedule of Experiments

The experiments will be done at the “miniscale” level.

Date	Experiment	Lab Due
Thu 1/20	Check In and Safety (Sect. 1.8, 1.9, 1.11)	
Tue 1/25	Expt. 3.2B and 3.3B Recrystallization and Melting Point of Benzoic Acid	Thu 1/27
Thu 1/27	Expt. 4.3 Simple Distillation	Tue 2/1
Tue 2/1	Expt. 4.4 Fractional Distillation	Thu 2/3
Thu 2/3	<b>Quiz 1</b> Expt. 5.3A One-Base Extraction	Tue 2/8
Tue 2/8	TLC of Drugs (pages 175-180 and Handout)	Thu 2/10
Thu 2/10	Expt. 15.3 Friedel-Crafts Acylation of <i>m</i> -Xylene	Tue 2/15
Tue 2/15	Expt. 15.5 Relative Rates of Aromatic Substitution	Thu 2/17
Thu 2/17	<b>Quiz 2</b> Expt. 14.4 Preparation of 1-bromobutane	Tue 2/22
Tue 2/22	Expt. 14.5 Preparation of 2-chloro-2-methylbutane	Thu 2/24
Thu 2/24	Expt. 25.9 Identification of primary, secondary, and tertiary chlorides and bromides	Tue 3/1
Tue 3/1	Expt. 8.2 Infrared Spectroscopy	Thu 3/3
Thu 3/3	<b>Quiz 3</b> Expt. 16.2A Oxidation of Cyclododecanol to Cyclododecanone	Tue 3/8
Tue 3/8	Expt. 8.3 <sup>1</sup> H and <sup>13</sup> C Nuclear Magnetic Resonance	Tue 3/22
Thu 3/10	Expt. 17.4 Reduction of 9-Fluorenone	Thu 3/24
Tue 3/22	Expt. 17.4 continued	
Thu 3/24	Expt. 19.4A Grignard Reagents	Thu 3/31
Tue 3/29	<b>Report*</b> Expt. 19.4A continued	
Thu 3/31	Expt. 10.3B Dehydration of 4-Methylcyclohexanol	Tue 4/5
Tue 4/5	Expt. 10.6 Bromination of ( <i>E</i> )-Stilbene	Thu 4/7
Thu 4/7	<b>Quiz 4</b> Expt. 12.3B Diels-Alder Reaction	Tue 4/12
Tue 4/12	Expt. 20.2 Fisher Esterification: Benzocaine	Thu 4/14
Thu 4/14	Expt. 18.3 Synthesis of <i>trans-p</i> -Anisalacetophenone	Thu 4/19
Tue 4/19	Expt. 18.4 Preparation of 4,4-Dimethyl-2-cyclohexen-1-one	Tue 4/26
Thu 4/21	Expt. 18.4 continued	
Tue 4/26	<b>Quiz 5</b> Expt. 22.3 Polymers: Nylon	Thu 4/28
Thu 4/28	Check Out	
Tue 5/3	Optional review	
Thu 5/5	FINAL EXAM	

\* See In the report on spectroscopy