BALL STATE UNIVERSITY
COLLEGE OF SCIENCES AND HUMANITIES

Instructor: Wei Shi wqshi@bsu.edu FB-504

Office Hours: Wed (1:00–3:00 pm) or by appointment. Contact me if none of these hours fit your schedule.

Lab Hours: Mon and Fri at 2:00 –4:50 pm, FB-556

Course Goal

Built upon what you have learned from Chem 231/232 and Chem 241/242, this course is intended to help expand your knowledge and experience in a variety of experimental and spectroscopic techniques. The successful student will leave with good capability of working more independently in organic chemistry laboratories to synthesize, isolate/purify and characterize organic molecules. To this end, we particularly emphasize (1) the knowledge and skills in acquisition and interpretation of NMR (both 1D and 2D) spectra; and (2) the column chromatography technique for purification.

For Chem 530 only:

Besides all the work required for 430, students in 530 will take some extra responsibilities to assist in smooth running of this lab course, especially helping train other students on how to run 1D and 2D NMR (and collect IR spectra if deemed necessary)

Attendance

Full performance in the laboratory requires regular and punctual class attendance. Absences and late arrivals will negatively impact on your performance, especially for the final practical exam on reaction. Alternative accommodations can, and likely will be made when there are extenuating circumstances:

- Adequate paperwork clarifying Ball State University representation activities must be presented no later than 3 days before the event.
- In the event of an unforeseeable event (illness, bereavement, etc), please provide a copy of some form of documentation (a medical certificate in the case of illness, or a brief note or e-mail message in other events).

Required Supplies

- A composition lab notebook, not spiral bound, and a pen
- Safety Goggles
- Lab Coat (with Name Tag via sticker, ink, sewing, or other visible idea)
- Clothing which covers all skin including the ankles
- Flat soled shoes that cover the entire foot (no ballet flats, sandals, etc.)

- A lock
- *(optional but highly recommended) The Organic Chem Lab Survival Manual: A Student's Guide to Techniques 10th Edition by James W. Zubrick (online editions and previous editions OK)*
- *(optional but highly recommended) Organic Chemistry, David Klein, Wiley*

Lab Notebook

Although it won't be graded, **your lab notebook is the only thing you may refer to during your lab practical exam**. I would be happy to answer any questions you may have about how to make effective lab notes. For easy indexing, the first page of your notebook should contain a table of contents, pages must be numbered. Professionally, everything in a lab notebook must be written using blue or black ink, **NOT PENCIL**.

Title

Experimental Objective/ Reaction Scheme

Table of Reagents – *Whenever applicable, each reagent should be listed with molecular weight, the mass or volume, density, mmoles and melting/boiling point. Leave space to fill in the actual quantity measured out in lab. The theoretical calculations for the product(s) should be included as well. Solvents and chemicals used in the work-up are not required. A good rule of thumb is If you can set up the reaction without the chemical in question, it isn't needed in the table.*

Procedure – *The use of experimental hand-outs in lab is strongly discouraged. It would be more time efficient for you to have a detailed procedure written up in your notebook before coming to lab. I recommend using bullet points and leaving spaces between each instruction to make the procedure easy to follow and to allow for changes in the procedure. Think carefully about the questions embedded in the procedure if any. Although you are not required to answer them in the pre-lab notebook, it is important to help you become an independent thinker along the way. Ask if you are not sure about your answers.*

Observations and Data – *Physical observations such as color changes, precipitation, vigorous boiling, etc. should be recorded, as well as any data such as masses of product, MP/BP. Original processed NMR spectra must be included as spectroscopic data.*

Sample code for NMR: your initials_experiment #_date_sample code (optional), e.g. SW_Exp1_210120_A.

Carefully read the experimental handout to determine what is necessary for notebook preparation. Data and observations should be entered directly into the notebook at the time you made the measurement or observation. Do not erase, scribble over, or use correction fluid in your notebook. Rather, mark a single line through any inaccurate or mistaken entry. Remember, your book will be evaluated on the basis that it is a *working* document. You should not be writing observations on pieces of scrap paper or on worksheets. Spiral bound books are NOT acceptable.

Worksheets

For all the labs except for exp 9, you will be assessed by a worksheet. Worksheets will encompass experimental data and likely some questions pertaining to the lab. I would encourage you to type your answers in the worksheets except for structures that you may draw by hand if you don't yet know how to use ChemDraw.

Quizzes and Exams

There will be two quizzes on NMR, one for 1D and one for 2D. At the end of the semester, there will be a written lab final in which you will be tested on organic chemistry theories and lab techniques as well as spectroscopic knowledge.

There will also be two lab practical exams, one focusing on separation and the other on reaction. The separation exam is exploratory and more of an open-book style over multiple lab periods. On the other hand, in the reaction practical exam, you need to finish a 2-step synthesis experiment independently within a limited amount of time over three lab periods, including acquisition and interpretation of NMR (both 1D and 2D) spectra. During the practical finals, you are allowed to refer to your lab notebook, but nothing else. You also need to independently acquire and print out your NMR spectra within 35 mins.

Make-up Quizzes and Exams

Make-up quizzes and exams may be provided only if you contact me **BEFORE** the test has started. Otherwise, the exam will count zero points.

Final Grade

Point distribution for the final grade is listed below. See the separate lab schedule for more details. There are also 10 bonus points available for this course. See below for detailed breakdowns. No other extra points exist.

Safety	5 points
Lab worksheets/reports	230 points
2 NMR assignments	40 points
2 NMR quizzes	30 points
Synthesis design project	25 points
Practical final (separation)	30 points
Practical final (reaction)	40 points
Final exam (written)	100 points
Total Points	**500 points**

Bonus points

Course evaluation	10 points
Total	**10 points**

❖ **Letter grade (tentative):**

A (≥ 93%)	**A- (92.9–90%)**	
B+ (89.9–87%)	**B (86.9–83%)**	**B- (82.9–80%)**
C+ (79.9–77%)	**C (76.9–73%)**	**C- (72.9–70%)**
D+ (69.9–67%)	**D (66.9–63%)**	**D- (62.9–60%)**
F (< 60%)		

- 5% point deductions will occur as a result of a skipped checkout.

- 10% point deductions will occur as a result of late reports after 24 hours; 20% point deductions will occur as a result of late reports after 72 hours; 50% point deductions will occur as a result of late reports after a week. After graded assignments are returned, late reports will not be accepted, and you will receive a zero score.
- 20% point deductions will occur when adequate safety precautions are not followed during lab (wear goggles, and adequate clothing in the lab).
- 100% point deductions will occur as a result of copied material for pre-lab quizzes, MOWs, Worksheets, and Reports.

Additional Considerations: It is important to understand the purpose of each step in each experiment. If you understand why you are doing what you are doing before you walk into the lab, you are more likely to successfully complete the experiment with extra time to start working on something else. Please ask all questions related to lab expectations, assignments, course material, lab procedures, and lab report expectations throughout the semester. It will be beneficial for both you and your classmates.

Posting of Grades

The points and scores will be posted on Canvas as soon as possible. You should double check to make sure that the actual score posted and the score achieved in the exam are identical.

Lab Safety

The number one priority in this laboratory course is safety – personal and environmental. You therefore should be aware of your own safety, that of others in the lab, and that of people outside of the lab. You must exercise all care in the handling (including the disposal of) of chemicals, and of equipment.

For this course, there are 11 bonus points associated with a safety quiz and a safety contract. You must complete both of them in order to conduct an experiment in the lab. If the safety quiz is not completed before the first experiment, attendance in the lab is not permitted.

Laboratory Risk Statement

Chemical Use: Students enrolling in this course will be working with a variety of chemicals, some of which could be irritating or hazardous. Proper and accepted good laboratory practice will always be utilized to minimize or eliminate any adverse contact. Individuals are required to wear flat-soled shoes that cover the entire foot (no ballet flats, sandals, flip flops or high heels) and clothes plus lab coat should fully cover your body from shoulders to feet. However, individuals with sensitive medical conditions (including pregnancy) should take precautions such as wearing additional protective garments, delaying enrollment, or not enrolling in this course. Chemical Safety Data Sheets can be searched on Risk and Safety Solutions by logging in with your BSU credentials.
Please consult with your instructor and health care professional if you have any concerns.

Personal Safety: Chemistry is a discipline that requires an awareness and appreciation of safety issues. You must exercise care in the handling (including the disposal of) of chemicals, and of equipment.

1. Safety Goggles and Lab Coats are **MANDATORY**, and must be worn **AT ALL TIMES** in the laboratory. You need to purchase full coverage goggles, not glasses - Safety or hardened-glass spectacles are not an acceptable substitute, but may be worn under goggles.

2. You must wear appropriate clothing and footwear: Closed-toe shoes or sneakers are appropriate, open-toed sandals, flip-flops and the like are not. The legs must be covered at all times while in the lab.
3. Clothing/Skin Protection, in the form of appropriate attire and gloves, are encouraged. In any event, it is advisable that you not wear your "best" in the lab. Latex and/or nitrile gloves will be made available during the labs.
4. You should avoid contact with chemicals and solvents (including inhalation) as much as possible. Gloves should be available in the lab for handling of material. You should discard material in the waste containers provided, not wash them down the sink.
5. Inform yourself. Read all of the required reading material before coming to the lab. You should be aware that Material Safety Data Sheets (MSDS) of all materials used in the lab are available upon request.
6. Concentrate. It is important to stay focused in the lab, particularly toward the end of the period, when avoidable accidents may happen. Chemistry is one of those disciplines where a momentary lapse can cause harm.

Environmental Safety: Be aware of what is going on around you, and take personal responsibility for your safety. **SAFETY IS EVERYONE'S RESPONSIBILITY**, not just the instructors.

Organic Chemistry Lab Rules

- Students may only enter the stockroom with permission from the stockroom staff.
- Students may only take the amount of chemicals necessary to complete the lab and no more.
- Students may only take the necessary amount of acetone to clean glassware.
- **Never** use the same pipette on two different chemicals. Never cross contaminate chemicals. Lids must remain on chemical containers.
- Waste chemicals, and particularly organic compounds, should not simply be washed down the sink. Solid and liquid organic waste containers will be provided in the lab. Do not overfill waste containers.
- The broken glass containers are **only** for broken glass. No gloves, transfer pipettes, weight boats, etc.
- If glassware is broken in lab, ask stockroom for brush and pan and put broken glassware in the broken glass container.
- Clean up your own mess. If you make a mess, it is your responsibility to clean it up. This applies to your hood as well as the rest of the lab. This also includes things like putting the tops on bottles and jars (particularly those containing drying agents), as well as wiping up spills of liquids; or dribbles on the lips and sides of bottles. All chemicals must be disposed of properly. If the student does not know how to properly dispose of a chemical, he or she can ask their instructor or the stockroom staff.
- Inform the stockroom or instructor when waste jug is **almost** full or if acetone is needed.
- Lids must remain on chemicals that can be evaporated away.
- Students are responsible for returning any equipment borrowed during lab to the correct location after lab is completed.

Need Extra Help?

If you need extra help, I will strongly encourage you to come to my office hours, which will provide you direct and specific help you may need.

Return of Worksheets, Reports, Quizzes and Exams

The worksheets, reports, quizzes and exams will be graded as early as possible and returned to you usually in the following lab. Feedback on wrong calculations of total scores or any other grading inquiry on the test should be provided on site. It remains the responsibility of the student to ensure that the total score of the exam is correct. The final exam will not be returned, but you may schedule an appointment to review.

University Statement

We are committed to ensuring that all members of the community are welcome, through valuing the various experiences and worldviews represented at Ball State and among those we serve. We promote a culture of respect and civil discourse.

If you need course adaptations or accommodations because of a disability, please contact the instructor of record as soon as possible. Ball State's Disability Services office coordinates services for students with disabilities; documentation of a disability needs to be on file in that office before any accommodations can be provided. Disability Services can be contacted at 765-285-5293 or dsd@bsu.edu.

"Freedom of Expression" Statement

In this course, we are committed to fostering a learning environment that values intellectual diversity, encourages free expression, and promotes open inquiry. As members of the Ball State Community, we treat each person in the Ball State community with civility, courtesy, compassion, and dignity and respect and learn from differences in people, ideas, and opinions. Please review Ball State University's Statement on Freedom of Expression, the resources on Ball State's Freedom of Expression webpage, and Ball State's Beneficence Pledge.

Academic Misconduct

Academic misconduct will not be tolerated under any circumstances. In all instances of cheating, this professor pursues the harshest penalties available. Please read the Code of Student Conduct in the Student Handbook. If you do not own a copy, please access it online at: https://www.bsu.edu/about/administrativeoffices/student-conduct/policiesandprocedures/studentcode.

Inclement Weather

Class will be held regardless of weather conditions except in cases when the University is closed.

======= Quotations to Contemplate =======

"The only guarantee for failure is to stop trying." – John C. Maxwell

"Judge a person by his[her] questions rather than by his[her] answers." – Voltaire

"You cannot teach a person anything, you can only help him[her] to find it for him[her]self." – Galileo Galilei

"Learning and teaching should not stand on opposite banks and just watch the river flow by; instead, they should embark together on a journey down the water." - Loris Malaguzzi

"A teacher can but lead you to the door; learning is up to you." - Chinese proverb

"The person who knows "how" will always have a job. The person who knows "why" will always be his boss." - Diane Ravitch

Note: This schedule may be amended as deemed necessary.

Date	Experiment	Format (pts)	Due date*
8/18 (1)	• Introduction • Safety Quiz & lab check-in • NMR lecture • Glassware cleaning • Oil bath temperature calibration • Synthesis design project	1D Assignment (16) Synthesis (30)	8/25 12/8
8/22 (5)	• 2D NMR • NMR training & practice • Oil bath temperature calibration (Cont'd)	2D Assignment (24)	9/26
8/25 (1)	• Exp 1: Structural elucidation of pure unknowns • NMR training & practice (Cont'd)	Wksht for 1 (15)	9/5
8/29 (5)	• 1D NMR quiz (40 min) • Exp 2: Recrystallization and vacuum filtration • Exp 1: Structural elucidation of pure unknowns (Cont'd) • NMR training & practice (Cont'd)	Quiz (14) Wksht for 2 (15)	9/12
9/1 (1)	Labor Day, no class		
9/5 (5)	• Exp 3: Liquid-liquid extraction and TLC • Exp 2: Recrystallization and vacuum filtration – NMR	Wksht for 3 (15)	9/19
9/8 (1)	• Exp 4: Column chromatography (group 1) • Exp 5: Acetylation of alcohols (group 2) • Exp 3: Liquid-liquid extraction and TLC (Cont'd) • Practical exam 1 (open)	Wksht for 4 (40) Wksht for 5 (20) 30	10/10 10/3 11/7
9/12 (5)	• Exp 4: Column chromatography (group 1) • Exp 5: Acetylation of alcohols (group 2)		
9/15 (1)	• Exp 4: Column chromatography (group 2/1) • Exp 5: Acetylation of alcohols (group 1)		
9/19 (5)	• Exp 4: Column chromatography (group 2/1) • Exp 5: Acetylation of alcohols (group 1)		
9/22 (1)	• Exp 4: Column chromatography (group 2) • Exp 6: Identification of an unknown product	Wksht for 6 (30)	10/17
9/26 (5)	• Exp 4: Column chromatography (group 2) • Exp 6: Identification of an unknown product • Exp 7: Acetylation of Glucose ($HClO_4$)	Wksht for 7 (30)	10/24
9/29 (1)	• 2D NMR quiz (30 min) • Exp 6: Identification of an unknown product • Exp 7: Acetylation of Glucose ($HClO_4$)	Quiz (16)	
10/3 (5)	• Exp 8: Multistep Synthesis – Step 1 • Exp 7: Acetylation of Glucose ($HClO_4$)	Wksht for 8 (65)	11/24
10/6 (1)	Fall Break, no class		

Date	Experiment	Format (pts)	Due date*
10/10 (5)	• Exp 8: Multistep Synthesis – Step 1/2		
10/13 (1)	• Exp 8: Multistep Synthesis – Step 2		
10/17 (5)	• Exp 8: Multistep Synthesis – Step 2/3		
10/20 (1)	• Exp 8: Multistep Synthesis – Step 3/4		
10/24 (5)	• Exp 8: Multistep Synthesis – Step 3/4		
10/27 (1)	• Exp 8: Multistep Synthesis – Step 4		
10/31 (5)	• Exp 8: Multistep Synthesis – Step 4		
11/3 (1)	• Exp 8: Multistep Synthesis – Step 4		
11/7 (5)	• Exp 8: Multistep Synthesis – Step 4 • Practical exam 1 due		
11/10 (1)	• Practical Exam (2-step synthesis, group 1)	40	
11/14 (5)	• Practical Exam (2-step synthesis, group 1 & 2)		
11/17 (1)	• Practical Exam (2-step synthesis, group 1 & 2)		
11/21 (5)	• Practical Exam (2-step synthesis, group 2)		
11/24 (1)	• Exp 9: BSA labeling	No wksht for 9	
11/28 (5)	Thanksgiving Break, no class		
12/1 (1)	• Exp 9: Click chemistry		
12/5 (5)	• Exp 9: SDS-PAGE analysis • Lab check-out		
12/8 (1)	• Synthesis design project due • Lab final – written	100	
		500 (total)	

* Your lab notebook will not be graded, but would be the only thing you may have with you in your practical exam.