CHEMISTRY 355
COURSE OUTLINE AND SUPPLEMENT
Summer 2018

Course Objectives
Materials covered in lecture courses are based on experimental data accumulated over the years which have been systematized into natural laws and theories. However, the study of chemistry (or any science) requires the learning of some laboratory techniques and methods of data acquisition. It is intended that the skills acquired in this course will be of practical value to you in your fields of interest. It is also hoped that a "hands on" experience will enable you to grasp the concepts presented in CEM 351-352 lectures more easily.

- Students will relate concepts from organic chemistry lecture courses to experiments in lab.
- Students will practice common organic chemistry laboratory techniques, including isolation, purification, synthesis, and analysis.
- Students will interpret data, draw conclusions, and recommend next steps for an experiment.
- Students will organize background information, experimental methods, results, and conclusions into written laboratory reports.

Required Materials
Lab Text: "Operational Organic Chemistry", 4th Edition.
John W. Lehman, Allyn & Bacon, Inc., Boston, Mass., 1999.
Miscellaneous: **Approved splash proof safety goggles** (ANSI 279.1 1979).
Lab coats are REQUIRED IN CEM355 LAB.
Any bound notebook with duplicate prenumbered pages **and carbon paper.**
Ballpoint pen (not a pencil).

Supplementary Readings: "Organic Chemistry," Jones or any other Organic Chemistry text book.
Microscale Organic Lab, D. W. Mayo; R. M. Pike; P. K. Trumper, 3rd ed.
Introduction to Organic Laboratory Techniques: A Microscale Approach, Pavia, Lampman, Kriz, and Engel, 2nd ed.

Course Director: Dr. Kevin Walker, Room 208 Chemistry, 353-1112, Office Hours, T 2-3 PM, or by appointment.
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Grading

Experiment	Points
Melting point calibration	60 points
Distillation	100 points
Cyclohexene	100 points
TLC	60 points
Column chromatography	60 points
Extraction	60 points
Spectroscopy	60 points
Benzopinacolone	100 points
Grignard reaction (microscale)	100 points
2-chloro-5-nitrobenzamide	100 points
Isolation of caffeine	50 points
Identification of a carbonyl compound	100 Points
Recrystallization	100 points
Azo dye	100 points
Lab Clean Up	25 points
Total	**1175 points**

Report grades will be based on format (as described in "Expectations for Laboratory Reports"), content, results (yield, melting point, boiling point, refractive index, spectra, etc.), and responses to post-laboratory questions in the discussion.

Major or continual violations of safety rules will result in dismissal from lab and a 50–point reduction in course grade (penalty).

Housekeeping violations such as leaving your bench and/or the common area dirty shall result in a 20-point penalty.

Missed Lab Policy: You can miss a maximum of 2 labs without penalty. Missing 3 or more labs results in a 50% penalty for each lab missed.
Procedure: Get results from your lab mate (or other group) and write your lab report as usual.

The **anticipated** grade distribution is:

1062 – 1175 = 4.0	975 – 1061 = 3.5	873 – 974 = 3.0
800 – 872 = 2.5	675 – 799 = 2.0	0 – 674 = 0.0

Schedule (sequence within the modules may be revised by your TA):

Date	Experiment*	Report Due
7/02 (M)	Safety discussion Check in Melting point experiment	07/07 (Saturday)
	Liquids Module	
07/03 (T)	Distillation I & II	07/07 (Saturday)
07/04 (W)	No labs (Independence Day)	
07/05 (Th)	Distillation III & IV	
07/09 (M)	Cyclohexene I	07/14 (Saturday)
07/10 (T)	Cyclohexene II $	
	Solids Module	
07/11(W)	TLC	07/14 (Saturday)
07/12 (Th)	Column Chromatography	07/21 (Saturday)
07/16 (M)	Column Chromatography	
07/17 (T)	Extraction	07/21 (Saturday)
07/18 (W)	Extraction	
07/19 (Th)	Recrystallization	07/28 (Saturday)
07/23 (M)	Recrystallization	
07/24 (T)	Recrystallization $	
	Synthesis Module	
07/25 (W)	Benzopinacolone	08/04 (Saturday)
07/25 (W)	Grignard	07/28 (Saturday)
07/26 (Th)	Grignard $	
07/30 (M)	Benzopinacolone $	08/04 (Saturday)
07/31 (T)	Multi-Step Synthesis I	08/11 (Saturday)
08/01 (W)	Multi-Step Synthesis II	
08/02 (Th)	Multi-Step Synthesis III	
08/06 (M)	Multi-Step Synthesis IV $	
08/07 (T)	Caffeine	08/11 (Saturday)
08/08 (W)	Caffeine	
08/09 (Th)	ID of Carbonyl $	08/11 (Saturday)
08/13 (M)	Azo Dye	08/18 (Saturday)
08/14 (T)	Azo Dye $	
08/15 (W)	Check out TA evaluation	N/A
08/16 (Th)	Lab Clean Up	N/A

* See Supplementary Procedures in syllabus
$ Graded product

Failure to check out before or during your last scheduled lab results in a $25.00 plus breakage fee. If you withdraw from CEM 355 early, check out early too.

Check-in Procedures 

- Record your locker number and combination in your notebook. Make sure that your name is entered beside your locker number in the T.A.'s section folder.
- Carefully check all of your apparatus, washing anything which isn't clean. A distilled water rinse will prevent water marks. Replace any chipped, cracked, broken or missing apparatus before starting any experiments. See your instructor for details on how to obtain replacement items. Give your instructor your completed inventory sheet.
- Read the safety regulations. Ask for explanations regarding any rules which you don't understand.
- Your locker may be reassigned if the check-in procedure is not followed. Be sure that all paperwork is done the first day.

Reading Assignments 

Before the second laboratory session, you are expected to be thoroughly familiar with the following sections of Lehman:
Introduction and Advice to Students (pp. 1—12)
Laboratory Safety (pp. 13—30)
Appendixes (pp. 833—879)

The contents of the operations section (pp. 585-832) will be covered during the course of the term. The sequence that these topics are covered will vary among the sections.

Before check in, you must do your safety sheet online at the bottom right of the chemistry department web page (www.chemistry.msu.edu).

Write Ups 

1. Pre-Laboratory – due at beginning of class

   Review the operations in the text that are relevant to the experiment (See Supplementary Procedures for recommended operations for each experiment) prior to arriving in the laboratory. Prepare a pre-lab write up summarizing the experiment and operations to be completed. Students are encouraged to work together, but individual copies of all laboratory work will be submitted individually. Plagiarism is taken seriously and actions will be taken according to the MSU Plagiarism Policy.

   An important laboratory skill is the making and recording of accurate observations. A well-kept notebook is an essential part of any investigation. Your notebook should be a complete description of what happened--or didn't happen--in your experiments. If someone else could repeat your work and get the identical results using only the notebook for directions, then your notes are thorough. A well-kept set of records is an essential part of any

investigation. The **carbon copies** are to be given to your instructor at the **end of each week**. Each page should be signed and dated.

2. Post Laboratory Report – due Saturday following completion of experiment

   See "Expectations for Laboratory Reports" and "Template for Laboratory Reports" on the course webpage for a description of the laboratory report format. Lab reports are due via D2L on the Saturday following completion of the experiments. Students are encouraged to work together, but individual copies of all laboratory work will be submitted individually. Plagiarism is taken seriously and actions will be taken according to the MSU Plagiarism Policy.

   You must turn in the products in order to get credit for the experiment. All products are to be turned in to your laboratory instructor in a stoppered, labeled bottle, vial, or plastic bag. The label is to have clearly written on it: notebook reference number, the compound's name, m.p. or b.p., tare weight, weight of compound, % yield, the student's name and laboratory section. Do **NOT** include your student number. The notebook reference number consists of your last name or initials, the notebook number, the page number, and a letter identifying the particular compound on that page. To give an example: ABA-II-34-D is compound D described on page 34 of notebook 2 of ABA.

   Spectra should be similarly identified. Spectra of "starting material," "recovered starting material," or even different lots of the same material are not always the same. The use of notebook reference numbers becomes increasingly important as the problems become more complex. Develop the habit now, while the problems are still simple.

Waste Disposal 

A chemical laboratory or manufacturing plant is the potential source of numerous by-products and wastes. The impact of these materials on the environment can be greatly reduced or eliminated altogether by following appropriate procedures. Not all chemical wastes are treated in the same manner.

Dispose of your hazardous chemical waste as follows:

1. **Place liquid hazardous chemical waste in the liquid hazardous waste container.**
2. **Place solid hazardous chemical waste in the solid hazardous waste container.**

Ask your TA where to place your hazardous waste if you are not sure.

The ecologically safe disposal of chemical wastes and by-products requires the cooperation of **everyone** in the lab. Place the waste materials in the appropriate bottle or jar. Remember also that hazardous reactions may occur when different types of waste are mixed. Follow the directions of this handout and your instructor carefully; read all labels twice. Know what you are discarding.

Broken glassware and stoneware belongs in the BROKEN GLASS bucket. Do not put sharp objects into the wastepaper buckets where they might injure an unsuspecting custodian.

There will be a 50–point penalty for any improper waste disposal of hazardous chemical waste.

Housekeeping 

Each student should sponge off his/her work area at the end of each lab period. All community equipment (clamps, rods, hoses, ice baths, steam bath kits, etc.) are to be returned to the proper compartments. In particular, each bench space should have a steam bath with both hoses and a **complete set of rings**. Hoarding community apparatus is grounds for an NA grade.

If the common area such as the balances and the reagent hoods is left messy, 20 points will be deducted from everyone in that particular section.

Community areas such as hoods and balances are to be kept clean. If you spill something, clean it up promptly. The lab instructors are encouraged to assign cleanup duties if needed. If you feel that the lab is not clean when you enter, tell your TA promptly. Appropriate penalties will be levied against the offenders. Remember that your housekeeping will be evaluated by the next section also!

Housekeeping violations such as leaving your bench and/or the common area dirty shall result in a 20-point penalty.

Safety Regulations

In order to avoid personal injuries and injuries to fellow students while performing experiments in your Chemistry Laboratory Courses, it is required that you read and understand the following regulations before performing any experiments. Please indicate that you have done so by signing and returning one copy to your instructor. The department reserves the right to exclude any person from the laboratory who endangers him/herself or others.

A. Personal Protection

1. Approved safety goggles (not sunglasses) must be worn at **all** times when in the laboratory. Soft contact lenses shall not be worn in the laboratory under any circumstances, even under goggles. Hard contact lenses are conditionally acceptable. Check with your instructor.

2. If you get a chemical in your eye, immediate and extensive washing with water **only** is absolutely essential to minimize damage. Use an eye wash bottle, a hose, an eye fountain or an eye cup at once. If you spill any chemical on yourself, immediately wash with large amounts of water; then notify your instructor.

3. The wearing of rubber gloves and aprons is strongly advised when working with toxic and/or corrosive substances. However, gloves must never be a substitute for neatness and careful technique. Do not use organic solvents to remove organic compounds from the skin: they will only spread the damage over a wider area. Solvents also tend to penetrate skin, carrying other chemicals along. Soap and water are more effective.

4. Do not apply ointments to chemical or thermal burns. Use only cold water.

5. Do not taste anything in the laboratory. (This applies to food as well as chemicals. Do not use the laboratory as an eating place and do not eat or drink from laboratory glassware.)Do not use mouth suction in filling pipettes with chemical reagents. (Use a suction bulb.)

6. To minimize hazard, confine long hair securely when in the laboratory. (Also, a laboratory apron is essential when you are wearing easily combustible clothing, especially synthetics. Such an apron affords desirable protection on all occasions.)Shoes or sneakers must be worn in labs at all times.

7. Exercise great care in noting the odor of fumes and whenever possible avoid breathing fumes of any kind. See also C-6.

8. No smoking in labs.

9. You are advised to obtain medical attention for cuts, burns, inhalation of fumes, or any other laboratory incurred accident. If needed, your laboratory instructor will arrange for transportation to Olin Health Center. An accident report must be completed by your laboratory instructor.

10. No earphones shall be worn in laboratories.

B. Property Protection

1. In case of fire, call the instructor at once. If you are near an extinguisher, bring the extinguisher to the fire, but let the instructor use it.

2. Know the location of all safety equipment: fire extinguisher, safety showers, fire blankets, eye washes (any water hose works in an emergency) and exits.

3. Treat all liquids as extremely flammable unless you know them to be otherwise.

4. Clean all spills promptly with water (except water-reactive substances) and paper towels. If you have any doubts about the proper clean-up procedure, ask your instructor.

5. Disposal of waste: dispose of all chemicals properly. For hazardous waste use the waste containers in your lab. Ask your instructor how to dispose of waste chemicals you are unsure about.

6. Place broken glass in the appropriate container. Do not put broken glass in the wastepaper cans.

C. Laboratory Technique

1. Read the experiment before coming into the lab. This will allow you to plan ahead so that you can make best use of your time. The more you rush at the end of a lab, the greater your chance of having an accident.

2. Perform no unauthorized experiments. Do not remove any chemicals or equipment from the laboratories. You alone will bear the consequences of "unauthorized experimentation".

3. Never work in any laboratory alone!

4. Don't force glass tubing into rubber stoppers. (Protect your hands with a towel when inserting tubing into stoppers, and use a lubricant).

5. When working with electrical equipment observe caution in handling loose wires and make sure that all equipment is electrically grounded before touching it. Clean up all puddles immediately.

6. Use hood facilities. Odors and gases from chemicals and chemical reactions are usually unpleasant and in many cases toxic.

7. View reactions from the side, keeping glass and safety glasses between you and the reactants. Do not look into the open mouth of a test tube or reaction flask. Point the open end of the tube away from you and other laboratory workers.

8. Be a good housekeeper. Order and neatness will minimize accidents.

9. Laboratory safety is the personal responsibility of each and every individual in the laboratory. Report unsafe practices.

10. Treat all chemicals as corrosive and toxic and all chemical reactions as hazardous unless you know them to be otherwise.

ANY SAFETY VIOLATION WILL RESULT IN A 50–POINT PENALTY.

Rubber Gloves 

The greatest hazards associated with chemicals generally concern swallowing or inhaling vapors, fumes, or mists. Many people consider only corrosive materials such as lye or sulfuric acid dangerous to skin. This is a false assumption. Another false assumption is that chemists build up an immunity to chemicals because they work with them every day. Skin resistance to chemicals varies. In some cases skin resistance is very good, but in others, especially lipid soluble materials, absorption through the skin can produce dangerous levels in the body. Some common chemicals which are readily absorbed through the skin in toxic amounts include:

Aniline Cyanides Mercury
Benzene 1,2-Dibromoethane Nicotine
Bis(chloromethyl) ether *N*,*N*-Dimethylaniline Nitrotoluene
Bromoform Dimethylformamide Phenol
Carbon tetrachloride Hydrazine Tetraethyl lead

As time passes, each chemist becomes more aware that the little things in the laboratory are not always simple or safe. Rubber gloves are of this class of items. The following generalizations about each glove material can be made:

Nitrile: a copolymer of butadiene and acrylonitrile. Noted for its resistance to puncture, abrasion and most chemicals, particularly petroleum solvents, oils, acids, caustics, alcohols.

Neoprene: a polymer of 2-chloro-1,3-butadiene.The standard for glove boxes, recommended for oils, greases, gasoline, DMF.**NOT** for use with aromatics and chlorinated hydrocarbons, and strong oxidizers.

Natural excellent for use with alcohols and caustics. Good for DMSO

Rubber: and aniline, also most ketones. These are the commonly seen thin tan gloves. They are rapidly destroyed by thionyl chloride and chlorosulfonic acid.

Butyl most impermeable to gases and water vapor. Best for aldehydes

Rubber: and ketones, caustics, amines. Generally good all-around protection except for aromatics, chlorinated hydrocarbons, and petroleum solvents.

PVC: polyvinyl chloride supported gloves are generally best for inorganic and organic acids, caustics.

Polyethylene: these are always disposable; excellent for acids, caustics, aldehydes and ketones.

PVA: polyvinyl alcohol supported gloves are **the** best for aromatic and chlorinated hydrocarbons, ketones, THF, but cannot be used with aqueous systems as they dissolve in water. Also not good for DMSO, DMF, pyridine.