

## ORGANIC CHEMISTRY LABORATORY

**Laboratory Coordinator:** Dr. Ingmar Bauer. Office Hours: Wednesday 9:20- 10:30, Thursday 11:00-12:30, Chemieneubau, 128 and by appointment. Telephone: 463-32489 or 353-2500. E-mail [ingmar.bauer@chemie.tu-dresden.de](mailto:ingmar.bauer@chemie.tu-dresden.de)

**Required Text:** D.L. Pavia, G.M. Lampman, G.S.Kriz, Jr. and R.G. Engel, Introduction to Organic Laboratory Techniques, A Microscale Approach, edition 3 – BU custom edition, W.B. Saunders, 2000.

**Required Equipment:** To reduce the risk of eye injury approved goggles or safety glasses with side shields or must be worn AT ALL TIMES in lab. Students ignoring this rule will receive a 10% reduction in their grade and then will be asked to leave the laboratory if the infraction continues. Contact lenses may **never** be worn in the lab due to safety hazards. Students should own their own safety glasses. A lab coat is required. Closed-toe shoes, long pants, and long-sleeved shirts should be worn to lab. Your shirt and pants, or a lab coat, must cover you completely from neckline to shoes. Safety information in the text should be read prior to the first lab and the safety. **All work should be recorded in a laboratory notebook with consecutively numbered pages and the facility for making carbon copies.** Students are responsible for having copies of all work submitted. Appropriate notebooks are available in the stationery department of the Boston University Bookstore. These notebooks have two copies of each page, one in white and one in yellow. You will turn in the white copy.

**Collaboration:** Students are allowed and encouraged to talk about the laboratories together and to collaborate on understanding the material. **However, all work in preparing the prelabs, observations and reports should be done individually.** No joint preparation of laboratory write-ups is allowed. Any student discovered copying from another student's lab, either past or present, will be taken before the academic conduct committee as will the student whose lab was being copied. Do not "help" other students by handing them your completed lab. You will be held responsible if they copy it.

**Attendance:** Students must always attend the scheduled lab session. Punctuality is crucial as special instructions may be given at the start of lab. Attendance will be taken at the start of the lab and short quizzes may be given. Lab is an integral part of the course. Anyone not completing the laboratory portion of the course will receive an incomplete in the course.

**Grading:** The laboratory grade comprises 25% of the overall course grade. The grade for each lab is usually based on 35 points for pre-lab preparation, 45 points for experimental skill and ability to record observations about the experiment and 20 points for post-lab reports to be submitted for each of six experiments. These percentages will sometimes change slightly if there are pre-lab rather than post-lab questions. Part of your skills grade will be based on results (e.g. ability to obtain product) and part will be based on your technique (adherence to safety rules, cleanliness and organizational skills) as assessed by your teaching fellow. Sometimes quizzes will be given at the start of lab and will count as part of the pre-lab preparation grade. Quizzes will be announced on the lab information sheet. The grade for the end-of-semester lab exam will count as much as one lab. Thus each student will have seven grades.

**Make-up Labs:** Students are expected to perform all experiments. To account for absences which may result from illness, religious holidays, death in the family, etc., one make-up lab will be offered.

Admission to make-up lab requires Dr. Bauer's approval. To receive full credit a student must document the reason for their absence (for example a doctor's note or a note from a funeral home). Please note: having scheduled exams in another subject is not a valid excuse.

A student who does not have a documented excuse will receive a maximum grade of 70. Any student requesting more than one make-up lab a semester must present a letter from the Director of Dresden Programmes validating their excuse. **If you cannot attend the scheduled make-up lab notify Dr. Bauer immediately.**

**Lab Reports: COME TO LAB PREPARED!**

**Lab information sheet** - Be sure to get a lab information sheet before each lab. These will contain important directions as well as possible pre-lab and post-lab questions. They will be handed out at the prelab lectures.

**Report Format:** Lab reports consist of three sections: I. A **Pre-lab** to be recorded in the lab notebook **before** coming to lab. II. **Data and Observations** recorded while performing the experiment: III. **Results and Discussion** to be written (typed or in the lab notebook) after lab. Parts I and II will be turned in directly to the teaching fellow at the end of each lab session. Part III will be due one week later.

Part III should be turned back at the next lab session. **Be sure to put your teaching fellow's name on all reports.** Late reports will lose half credit for the report only.

**I. Pre-Lab:** In organic chemistry a critical part of the lab write-up is done before performing the experiment. The goal of advance preparation is to develop a good understanding of the required operations, a sense for their proper timing, and familiarity with the reactions, substances, and instruments involved. The Pre-Lab should include:

- 1) the **main reaction** under study (if any).
- 2) a list of **safety hazards** specific to the lab you are doing. **ALL SAFETY HAZARDS SHOULD BE INCLUDED.** Examples of hazards are flammable liquids, corrosive acids, and highly reactive materials. These hazards will often be mentioned in the lab text or on the prelab information sheet. However it is up to you to check on **all chemicals used.** Please describe hazards in your own words, do not include long printouts or xeroxes from books. The Merck index and MSDS sheets (available online) are helpful sources. Also note that goggles and gloves are precautions, not hazards. Use your own judgment. While ethanol is listed as being toxic (and can kill you), it is not a toxicity hazard in lab given that you can ingest more than you will be working with in lab without permanent harm. It is however a flammability hazard.
- 3) a **separation and purification scheme** (if appropriate). This is not the same as the procedure (see 4), but is an outline of where the reactants and products are (in solution, in the precipitate, etc.) Look at page 27 of the text- note that it does not include amounts or directions for the purification steps. You will need a separation and purification scheme for experiment one.
- 4) Instructions for the **experimental procedure** in your own words. Provide as much or as little detail as required so that **the lab can be executed exclusively from pre-lab notes.** Try to outline the procedure from the text. Do not simply rewrite the text. This procedure should include information on safety hazards and drawings of equipment needed. It should be written on the left half of the page. The right half will be used to record data and observations next to the appropriate step of the procedure.
- 5) **pre-lab questions** if assigned

The pre-lab should be complete enough that **it should not be necessary to refer to the lab text during lab.** The presence of the lab text on the lab bench will be taken as an indication of poor preparation and will result in grade reduction.

**II. Data and Observations:** A most important skill is the ability to record clearly the phenomena observed in the course of the experiment since these frequently are a sign of important chemical events. Examples of observations to be noted are color changes, the evolution of heat, the liberation of gas, the physical state and appearance of starting materials, intermediate reaction mixtures, and crude and purified products, etc. Set up your lab notebook so that observations can be recorded on the same page as the portion of the experimental procedure which is being observed (see pre-lab item 4).

Data will be largely quantitative in nature and should be recorded with appropriate units. Examples include melting and boiling point ranges, quantities of materials used and products obtained, percent yields, reaction times, spectroscopic absorption maxima, R<sub>f</sub> values, retention times, etc. All information should be recorded in your lab notebook.

**Carbon Copies** of all data and observations recorded in lab should be kept.

You will be handing in the originals to your tf at the end of every lab.

**III. Calculations, Results and Discussion:** It is a good practice to collect results neatly in tabular form. This is especially true if the raw data was recorded hastily or sloppily. The conclusion of the lab report should consist of a discussion of the major observations and results in terms of the chemistry involved. Your results should be compared to the expected results. What did you learn from doing this experiment? How do you know you produced the right product? How have you shown it to be pure? What problems did you run into? If you synthesize or isolate a product you should always calculate percentage yield. This section of the lab should rarely exceed one page.

Any post-lab questions should also be included.

#### **PRELAB LECTURE DATES**

You should attend the prelab lectures. Note well: The prelab lectures are not repeated.

**Prelab lectures will start in October**

## LABORATORY SCHEDULING INFORMATION

Lab Times:

Wednesdays, 1:00 – 4:30 pm - room 149, Chemieneubau

**Labs will start in October**

### CH 203 - LAB SCHEDULE

Session	Title of Experiment	Experiment Number	Techniques
1	<b>1. Introduction</b>	Safety and Preparation	p. 2-45
	Isolation of an Analgesic From a Pill	<b>7</b> , p. 96	Filtration, p. 545, 551-3, Melting point - sections 6.1 (p. 576) 6.2, 6.5, 6.7
2	<b>2. Preparation of Acetyl salicylic acid (TLC test of purity)</b>	<b>8</b> , (semi-microscale) p. 100-105	Heating & Cooling p. 521-5 Reaction Methods, p. 529-34 Crystallization, p. 558-575
	and TLC of Analgesics	<b>10</b> , p. 114	TLC p. 669-77, p. 698, p. 703-711
3	<b>3. Isolation of Chlorophyll and Carotenoids from spinach TLC</b>	<b>15</b> , p. 158	TLC column chromatography p. 669-685
4	<b>4. Synthesis of isopentyl acetate</b>	<b>12 B</b> , (semi-microscale) p. 128-135	reflux, p. 530-33 distillation, p. 617-27 extraction and drying p. 695-605, 607-10 IR -appendix 3
5	<b>5. Spearmint and Caraway oil (GC and polarimetry)</b>	<b>14</b> , p.143	gas chromatography technique 15 polarimetry handout technique 17
	and Synthesis of soap	<b>43</b> , p. 356	
6	<b>6. Synthesis of Methyl Stearate</b>	<b>25</b> , p. 239	IR, hydrogenation crystallization p. 558-566
	Make-up lab (all experiments)		
	<b>Lab exam 1</b>		

**Be sure to read the relevant technique sections and the essays preceding the experimental procedure prior to coming to lab.**

Labs may only be made up with prior permission from Dr. Bauer. LAB MAKE-UP FORMS - or email with all information - MUST BE TURNED IN EARLIER THAN NOVEMBER 17<sup>TH</sup>. If you cannot make up a lab on this date, contact Dr. Bauer. Make-up labs are reserved for students with valid medical or academic excuses.