CH 220C
ORGANIC CHEMISTRY LABORATORY
Fall, 2012

Section **Page**

1. GENERAL INFORMATION

REGISTRATION PROBLEMS

If you are having difficulty in completing your registration for the laboratory, consult Supplement A for the procedure to follow to resolve any problems.

PRE- and CO-REQUISITES

Pre- and co-requisites for CH 220C listed in the Course Schedule. *Important:* Because the lecture and laboratory courses are co-requisites of each other, dropping one of them requires that you drop the other as well, *unless* the drop occurs during the final **2** laboratory periods of the term.

Pre- and co-requisites will be checked and students not meeting the requirements *must* drop the course.

REQUIRED or RECOMMENDED COURSE MATERIALS

A. *Experimental Organic Chemistry,* 5th edition, by J. C. Gilbert and S. F. Martin, Saunders College Publishing, 2010 (Required).

B. Lab Notebook: A "carbon copy" notebook with quadrille-ruled pages with page numbers. The recommended notebook will be sold by Student Affiliates of the American Chemical Society at the beginning of each term (Required).

C. Turnitin.com Access Code

D. One Combination Lock: These must be sturdy combination locks. UT Chem locks are available at the University Co-op. You MUST have them for check-in. Otherwise you will *not* be allowed to check into the laboratory until you have obtained the required locks. See p. 7 or this document for information regarding your responsibility for your equipment (Required).

E. UT ID card: Bring your UT ID card to every laboratory session. It is needed to obtain items from the stockroom.

2. SAFETY INFORMATION

If you are pregnant now or become pregnant during the semester, you **must** immediately consult with the coordinator about your eligibility to continue in this course.

CLOTHING

Street Clothes: Shorts or short skirts *cannot* be worn in the laboratory **at any time**, *with or without a lab coat*. Your shirt must at least have short sleeves and cover your torso, *i.e.*, tank tops are *not* permitted.

Note: If you wear these to laboratory you will be sent home to change. On hot days you may wish to bring a pair of jeans or sweatpants to change into before entering the lab.

Shoes: Closed-toe shoes *must* be worn, i.e. sandals or clogs are not allowed.

Protective clothing: It is strongly recommended that you wear a lab coat or apron in the laboratory at all times.

SAFETY GOGGLES

The clear safety goggles provided by the department *must be worn at all times* in the lab. *If your vision is corrected,* wear your glasses *under* the clear safety goggles. Safety glasses *are not permitted* as substitutes for the goggles**.** *Not* wearing goggles in the laboratory will result in your expulsion for the remainder of the period.

RADIO AND TAPE PLAYERS

Radio, MP3 and tape or CD players are not allowed in lab.

JEWELRY

It is strongly recommended that you *not* wear rings, bracelets, or watches to the lab. Such items can trap chemicals next to your skin, thereby worsening the effects of burns or allergic reactions. Also NOTE that the solvents used in this course may permanently mar the synthetic materials contained in watchbands and crystals!

3. ATTENDANCE

LAB LECTURE

Attendance at the laboratory lecture before your regularly scheduled laboratory period is *required*. This lecture provides some of the general "How To's of Organic Chemistry", and helpful hints on performing the experiments. It also correlates the laboratory experiments with the topics being covered in the lecture section. Most of this information *is not written* down anywhere; the only way to get it is from the lecture. Material discussed in the laboratory Lecture is also one of the primary sources of material for the quizzes in the course. Finally, there may be a quiz at the end of every lab lecture.

LABORATORY

Attendance will be taken at all laboratory sessions. You will not receive credit for any experiment scheduled for a laboratory period for which you have an unexcused absence. If you have an excused absence, you must obtain a makeup permit before you will be permitted to work in any of the labs outside of your own regularly scheduled laboratory period.

In some cases, data will be collected by groups of two or more students. Zero credit will be given if you are not present or did not participate in the experimental work. You are NOT PERMITTED to use data collected by others in the group work if you are not present at the time the work is done.

MAKE-UP POLICY

Makeup Only *excused* absences may be made-up. Excused absences are those that are beyond your control, such as major emergencies and illness. Written verification of an excuse must be submitted for all absences, e.g., a doctor's note). To be eligible for a make-up laboratory period, you must request a permit for the make-up from Dr. Fjetland **within one week** of the excused absence.

A specific make-up day will be established for each experiment and that is the only day upon which the make-up work may be performed. You must have a make-up permit and must be on time to be eligible for the make-up lab.

Excuses will *not* be granted for the following occurrences, among others: oversleeping, trips not connected with official University activities, the need to study for another class, conflict with an exam. It is your responsibility to register for a laboratory section that avoids recurring conflicts that are not of an emergency nature.

There will be no exceptions to this policy!

Make-up reports are due when stated by Dr Fjetland.

4. LABORATORY PROTOCOL

ASSIGNED READING

Prior to each laboratory lecture, you should read the assigned pages of your text or of any hand-out associated with the experiments you are to perform.

PRE-LAB QUIZZES

A pre-lab will be given the first five minutes of wet lab. The questions will be based upon the procedure and the theory of the experiment. It is strongly recommended so that you can use it to study for the quizzes. If you miss the quiz, you miss it. **There will be no makeup quizzes given**.

LAB NOTEBOOK

The laboratory notebook is a critical record of your accomplishments in the laboratory so you should treat it accordingly by making careful and complete entries in it. Your lab notebook must be written in ink. If an error is made, draw a single line through the error and then continue. Note that the *original* pages in your laboratory notebook should never be removed; rather, turn in the *carbon copies* of these pages as directed. The pages should be sequentially numbered and your name should appear at the upper right-hand corner of each page. Leave the first 2 pages of the notebook blank for future use as a TABLE OF CONTENTS. This notebook is the last line of defense if there are any problems with grades, which means **DON'T THROW IT OUT.**

Your notebook will be turned in at the end of the semester and be graded on organization, neatness and thoroughness. You need not use consecutive pages in your laboratory notebook for each experiment, but this may have an effect on your notebook score. However, if you are unable to use consecutive pages, carefully reference on which page(s) additional entries for a particular experiment are to be found.

Note: All preliminary and final write-ups and Post-Lab exercises are to be done by yourself. Data interpretation and analysis are your individual responsibility and must also be done by yourself. Otherwise, the actions of the cheating policy (see Section **9**) are applicable.

There is only 1 format for the laboratory notebook consisting of the following:

1. **Heading:** Use a new page of the notebook to start the entries for the experiment. Provide information that includes your name, the date, the title of the experiment, and a reference to the place in the laboratory textbook or other source where the procedure may be found. See page 7 of your laboratory textbook for an example.

2. **Introduction:** Give a brief introduction to the experiment in which you clearly state the purpose(s) of the experiment. This should require no more than ¼ of a page.

3. **Main Reaction(s):** Write a balanced equation giving the main reaction(s) for the conversion of the starting material(s) to product(s). If you are conducting a preparative type experiment, such as the synthesis of cyclohexene, the reaction that converts cyclohexanol to cyclohexene is given along with all the catalysts and conditions required (see page 10 of your textbook for an example). If you are conducting an investigative experiment, no general reaction is required.

4. **Table of Chemical Data:** Set up a table chemical data as an aid in summarizing the amounts and properties of all reagents,catalysts and product(s). Also include those chemicals used in the cleanup of the products such as solvents.

   The following information needs to be included into the table:

   a. Name of compound
   b. Molecular formula of the compound
   c. Molecular weight of the compound
   d. Melting point and boiling point of the compound
   e. Density (d) of the compound
   f. Color of the compound
   g. Solubilities of the compound
   h. Hazards of the compound
   i. Symptoms of exposure
   j. Treatments for exposure to the compound

5. **Procedure:** Reference the source and page of the procedure and then give a summary of what you will be doing that day. Also include any changes that have been made

6. **Data and Results:** Any observations that you make during the experiment belong here. This includes things like the color of the solution when mixed, how the reaction proceeded and what happened when you added a reactant. This section also includes the observed melting point, weight, and percent yield of the product. You must also put any and all spectra, TLC or other type of data in this section.

7. **Discussion and Conclusion:** Discuss the theory behind the experiment performed and give a detailed mechanism of the reaction if one exists. Then write a conclusion stating whether or not the experiment demonstrated the principles and if not, why the data were inaccurate. This section should be started on a fresh page and should be no longer than 2 pages in length.

8. **Post-Lab Exercises:** Assigned post-lab exercises for a particular experiment are to be answered in your lab notebook in this section. They need to be placed after the conclusion section.

For more information on specifics of each section of the notebook, consult your TA.

SAMPLES

All samples obtained in the laboratory must be submitted in a properly labeled, sealed container. The label *must* include:

a. Date
b. Name of compound
c. MP measured
d. Weight or volume of compound
e. Percent yield of compound
f. Your name, laboratory day and time

Full credit for Final Reports require that you turn in all products produced in the course of your experimental work.

CHEMICALS

All reagents should be in your laboratory prior to the start of the laboratory period. Dry reagents are kept in the cupboards and liquid reagents are kept in the hoods. Any unknowns you may need can be obtained from the Stockroom (NHB). You will need to obtain an unknown card from your TA to pick up the unknown sample.

DUE DATES FOR REPORTS

The Preliminary Report includes **sections 1-5 from above** and is to be completed before the beginning of the laboratory specified in the Work Schedule. The TA will check and grade it to make sure that you have completed the preliminary report. It is your responsibility to make sure that the TA has checked and signed your preliminary report. If you haven't completed the preliminary report when you walk into lab, you will not be allowed in until it is completed. Then you must complete the lab in the time remaining, and you will be deducted 50% credit.

The Final Report is complete when **sections 6-8 from above** are completed. Any other assignments, as specified separately on the **Work Schedule** for the laboratory, are to be submitted separately. Your TA will sign the data section of your notebook after you have completed the experiment. It is your responsibility to make sure that your TA has signed your data section.

The Final Report is due on the date listed in the work schedule. All Final Reports are to be turned in to your TA at the beginning of the wet laboratory on the due date. Anything turned in after that time will be graded as late. Late Final Reports will receive 50% credit up to one week late. After one week, no credit will be accorded for the report. The Final Report discussions will be turned in typed. The typed discussion will be submitted to the Turn-It-In website to be checked and printed. The printed report will then be turned into your TA along with your data section and your post lab questions. Information regarding Turn-It-In can be found on the webpage. **Please ensure that you have printed the Turn-It-In report in the correct format, or it will not be accepted. Directions on how to print your report are on the webpage.**

Make-up labs: All papers due at the missed laboratory period will be due at your next regularly scheduled laboratory period. Final reports for experiments completed in the make-up laboratory are due as directed by the instructor. The same penalties as given above for late submissions also apply here.

5. ORIENTATION

IN-LAB INFORMATION

On the first day of laboratory you will receive a form that lists various safety-related items that you are to locate in the room. It is important that you complete the form accurately to demonstrate your knowledge of the locations. The completed form is to be submitted as directed by your instructor.

LIBRARY INFORMATION

There is a variety of sources in the Chemistry Library (WEL 2.132) that you may need to consult during this course. To acquaint with these sources, on the first day of laboratory you arel assigned an exercise for which you must supply the information listed.

The sources that contain the information are briefly discussed below. There are synonyms for some of the compounds, so if you fail to find the substance listed under the name you were provided, check for synonyms. Printed sources are located in the Chemistry Library on the Handbook Table, which is in the reference area in the front part of the library.

Information Required	References to Consult
A. Name of Compound:	1,2,3,4,5
B. Other Common/IUPAC Name of Compound:	1,2,3,4,5
C. Structural Formula:	2,3,4,5
D. MW: (i.e. molecular weight):	2,3,5
E. MP/BP:	1,3,4,5
F. Density (d):	1,2,3,5
G. Color:	1,2,3,4,5
H. Solubilities:	2,3,5
I. Hazards:	1,3,4,5
J. Treatment for Exposure:	1,4,5

1. Material Safety Data Sheets (MSDS)

MSDS are available from various sites on the Web. They are created by chemical manufacturers and can contain a variety of different data; no two are the same, and many different MSDS can exist for the same chemical. Good starting points are: http://www.utexas.edu/business/oehs/msds/ and http://www.lib.utexas.edu/chem/internet.html#msds. The Organic Labs page (http://www.cm.utexas.edu/CH210C/) provides access to abbreviated forms of relevant MSDS.

2. CRC Handbook of Chemistry and Physics

Located on the Handbook Table, in many editions. Find the table in the CRC called "Physical Constants of Organic Compounds," and look up your compound by name. *Remember that a particular compound may go by many different names, so check synonyms*! Older CRCs are quite different from newer ones in the way they are indexed and arranged; if you don't find your compound in one, try another edition. The newest editions have useful formula, synonym and structural formula indexes after the Table itself. Abbreviations used in the Table are defined at its beginning. Do not use the index in the back of the CRC to find compound information. **The CRC Handbook is now available on the Web.** The link can be found on http://www.lib.utexas.edu/chem/internet.html.

3. Merck Index

Also on the Handbook Table. This book contains information about common organic, inorganic, and biological substances, and has a Synonym Index in the back.

4. Sigma-Aldrich Library of Chemical Safety Data

Two large black volumes located on the Handbook Table. Look up your compound name or its molecular formula (if you know it) in the Index in the back of Vol. 2. The entry will provide a structural formula as well as some physical data and hazard information.

5. Dictionary of Organic Compounds

Also on the Handbook Table. This set of books contains information about common organic, inorganic, and biological substances, and has separate Synonym and Formula Index volumes.

6. OTHER USEFUL WEB RESOURCES

Chemfinder:	http://chemfinder.cambridgesoft.com/
NIST Chemistry WebBook:	http://webbook.nist.gov/chemistry/
Lange's Handbook of Chemistry:	http://www.knovel.com/knovel2/Toc.jsp?BookID=47

6. CHECK-IN

At the assigned laboratory period you will check into a drawer and hall locker that contain all your equipment. A copy of the checklist is attached (see Section **14**). Anything that is missing or broken can be replaced free of charge during the CHECK-IN period. After this time, you will be responsible for all equipment and glassware. At check-out, drawers and lockers will be checked by the TA for broken or missing items, which you must be replace. *Be aware that you are responsible for the safe storage of your equipment. Lost or stolen glassware will be reported to the UT Police and investigated by them.*

To replace broken glassware or equipment, go to the stockroom with your student ID and purchase the needed replacements. You will be sent a bill from the University for any such items. Be very careful with your ground-glass kits. Each kit has a total replacement value of $165.

You are required to check out of your drawer upon completing the semester or dropping the course (see below). A $25 penalty plus a charge for missing equipment will be assessed if you fail to check out.

7. DROP PROCEDURE

The last date on which you may drop the course without academic penalty is **September 14th, 2012.**

To drop the course *on* or *before* to this date, check out of your drawer and hall locker during your regular laboratory Period (see Section **13**).

To drop the course *after* this date, a) obtain a drop form the office of the Dean of your College; b) deliver the form to the Lower Division Course Office, WEL 2.212, and obtain a check-out slip from that office that is to be completed by your TA when you check out of the laboratory; c) check out of the laboratory during your regular laboratory period and have the check-out slip signed by your TA; d) return the signed slip to the Lower Division Chemistry Office for further processing of your drop.

8. GRADING PROCEDURE

GRADING SCALE

This laboratory course uses the +/- grading scale. This grading scale has the following distribution:

Grade	GPA	Grade GPA Recommended % Range
A	4	92-100%
A-	3.67	90 - 92%
B+	3.33	88 - 90%
B	3	82 - 88%
B-	2.67	80 - 82%
C+	2.33	78 - 80%
C	2	72 - 78%
C-	1.67	70 - 72%
D+	1.33	68 - 70%
D	1	62 - 68%
D-	0.67	60 - 62%
F	0	Below 60%

FINAL GRADE DETERMINATION

Each laboratory section will be graded on an individual curve, and distributions will be posted periodically. TAs will be provided common guidelines for evaluation of reports and laboratory Technique. Final grades will be determined by a computerized procedure. The final laboratory letter grade will be calculated as follows:

a. A class curve may be established, and a letter grade will be assigned on the basis of the final total score and this curve, if any.
b. To earn a C- or better in the course you must complete all assigned work and turn in all required reports.

REGRADES AND CORRECTIONS

Once an assignment has been returned, you will have one week to get an error corrected. After the week has passed, no regrades or corrections will be made on that assignment. The only exception to this is the correction of an error in the entry of the grade on the computer or an error in addition.

POINT DISTRIBUTION

What	Points
Preliminary Report	10 ea
Heading	1
Introduction	1
Reactions	1
Table of Chemical Information	4
Procedure	3
Final Report	100 ea
Data and Results	20
Discussion and Conclusion	40
Technique	25
Post-Lab Exercises	15
Quiz	50 ea

9. POLICY ON CHEATING FOR THE DEPARTMENT OF CHEMISTRY AND BIOCHEMISTRY

The University of Texas at Austin expects honesty and integrity to be the ordinary way of life in all student activities. Plagiarism, or the use of another person's statements without giving proper credit, is dishonest and is regarded as cheating. Although group study and projects are often appropriate, it is expected that individual assignments and examinations will be the private efforts of the particular student.

The following are considered examples of cheating:

1. Copying raw data for a laboratory without actually participating in acquisition of the raw data.

2. Inventing data.

3. Filling in parts of laboratory reports that require the raw data for calculations or interpretation before the data are collected.

4. Holding discussions so thorough that they result in identical laboratory reports, homework assignments, and computer programs.

5. Allowing anyone to copy any laboratory report, homework assignment, test, or computer program, either now or in the future.

6. Gaining access to, having in your possession at any time, or using old laboratory reports for any purpose. If you have questions regarding the format of any laboratory write-up, you should consult your TA or AI.

A student detected cheating beyond any reasonable doubt in the preparation of any individual assignment is subject to disciplinary action. See the General Information Bulletin.

10. TA OFFICE HOURS

TAs will hold office hours in Welch 2.304 in one of the stalls at times posted at this office, or on the web page. Feel free to consult with any TA and to ask questions concerning either laboratory or lecture material. It is Departmental policy that undergraduate students are not permitted in research laboratories. If you wish to consult your TA outside of his/her scheduled office hours, use e-mail and your TA will contact you.

11. FACULTY COURSE COORDINATOR

Dr. Conrad Fjetland
Office: NHB 1.128
Phone: 232-7676
Email: crfjetland@cm.utexas.edu
Office hours: M, Th 3-4 or by appointment

12. COURSE WEB PAGE

A web page has been developed for the course. Among other things, it provides the course syllabus, a listing of office hours, and links to MSDS information and web pages, if any, for the various lecture sections associated with the lab sections. The URL is http://courses.cm.utexas.edu/cfjetland/ch210c/.

13. CHECK-OUT INSTRUCTIONS

1. Wash all chemical residues off of your glassware. There is a waste bottle provided for this purpose.

2. Discard all trash, *i.e.,* corks, Parafilm™, papers, gloves, *etc.*

3. Dispose of all chemical samples in the appropriate containers.

4. Return *all* extra equipment and items not specified on the equipment list. This includes glass stirring rods. There is a dishpan in the hood for such items.

5. Check *all* of your equipment *before* coming to the storeroom to purchase any missing items. This will minimize the number of times you have to seek assistance from the storeroom personnel and will help speed the check-out process. Feel free to check the dishpan in the hood for the missing items you need *before* going to the storeroom to replace them.

6. Have your TA check your equipment only *after* you have replaced any missing items.

14. HINTS TO MINIMIZE FRUSTRATIONS IN ORGANIC CHEMISTRY

Organic chemistry is one of the most exciting and challenging courses you will encounter at UT-Austin. The course encompasses a broad range of topics including petrochemicals, polymers, pharmaceuticals, and life-sustaining biochemical processes. Organic chemistry can bring immense pleasure and numerous rewards. Yet it may also foster frustrations, most of which involve time constraints. You may often feel overwhelmed by the sheer volume of material to be learned and the amount of work accompanying the demands of the laboratory. These are legitimate concerns. Much information is indeed covered, and considerable time is required both in and out of the laboratory itself.

There tend to be two major gripes that students have concerning the lab:

A. Keeping a laboratory notebook and preparing for experiments require too much time.

Good science practices dictate that certain documentation be present in your laboratory book. Because we believe in teaching good science, this problem cannot be changed. With practice, you should become more efficient at preparing your laboratory book. A pointer: If you can't find the necessary information (physical data, hazards) in a reasonable amount of time, don't worry about it.

B. Students feel rushed during the laboratory period.

To a certain extent, this is true. Most organic experiments involve several steps and techniques, one or more of which is often laborious and time-consuming. This is the nature of the beast. Nonetheless, organic experiments can be fun, especially if you can minimize frustrations. Fortunately, we have more control over time constraints during lab. Handling these problems is merely a matter of time management--making the most efficient use of your time in lab. To that end, the following suggestions should prove helpful:

1. *Come to laboratory prepared.* This point can't be overemphasized. People who know what they are doing before starting are far more efficient than people who must constantly refer back to a procedure to find out what they are going to do next. Advance preparation also lets you find any ambiguous points in the procedure. You can then ask to have these clarified during laboratory lecture.

2. *Start the experiment as soon as possible.* This is usually not a problem. But you should be aware that you don't have time to stop for a soft drink or to chit-chat on the way to the lab, if it occurs immediately following the laboratory lecture.

3. *Familiarize yourself with the location of frequently used chemicals and equipment in the lab.* You will save time by not constantly having to ask where things are.

4. *Make the most efficient use of "dead time."* Many organic experiments have a stirring or reflux period during which there is nothing to do but wait. This time should be used for cleaning glassware and getting chemicals and/or apparatus ready for the subsequent steps. If there are qualitative tests assigned, they may be performed during such periods. These tests should not be done before starting the main reaction.

5. *Don't presume that every reaction will work perfectly (or even at all).* Often, these "tried and true" reactions fail to proceed the way the book describes. Even professional organic chemists with years of experience can't get every reaction to work for them, despite the fact that the reaction may be cited extensively in the scientific literature.

6. *Clean your glassware before you leave lab.* Like your pots and pans at home, laboratory apparatus is far easier to clean just after it is used rather than a week later. You will then be ready to start the next week's experiment without delay.

7. *Remember that your TAs and AI are here to help you.* If you have any problems or feel your frustration level rising, please don't hesitate to talk to us. Here's to a successful, enjoyable semester!

16. WORK SCHEDULE

LAB REPORT DUE DATE SCHEDULE*

Report	Due	Report	Due	Report	Due
Distillation	**Period 4**	Substitution	**Period 8**	Luminol	**Period 12**
Extraction	**Period 6**	Grignard	**Period 9**	Dehydrbromination	**Period 12**
Stereochemistry	**Period 6**	Aldol Condensation	**Period 10**	Relative Rates	**Period 12**
Arenes	**Period 7**	9-Fluorenone	**Period 10**	Nitration	**Period 13**
Stilbene	**Period 7**	Methyl Benzoate	**Period 11**	Methyl Orange	**Period 13**

* All reports are due at the beginning of the period.

EXPERIMENTS

REQUIRED PRE-LAB PREPARATION! Read about the techniques listed at the start of each experiment in preparation for working in the laboratory.

Period 1	**Lab Lecture: 8-29, 8-30**	**Wet Lab: 8-31, 9-4, 9-5**

Due Today
Reading Assignments (due by lab lecture) CH 1, Parts 1–11 of General Information in Syllabus, Secs. 2.7, 3.1and 3.3
What Are We Doing Today? (In Wet Lab)
CHECK-IN THERMOMETER CALIBRATION and MELTING POINTS (*Investigative*) (Procedure, Sec. 2.7, p. 38, Sec. 3.3, pp. 117–118, Part A) **You do not need to have a write-up for the Thermometer calibration. Store the data in your notebook.**

Notes for Melting Point Calibration

- You will be given your own apparatus and three samples with known melting points. Take the melting point for each recording the difference for each in your notebook. Make note of this difference on the apparatus as well.

Period 2	Lab Lecture: 9-6, 9-10	Wet Lab: 9-7, 9-11, 9-12

Due Today
Reading Assignments (due by lab lecture) Secs. 2.14, 4.1-4.4, 6.1 and 6.4
What Are We Doing Today? (In Wet Lab)
FRACTIONAL DISTILLATION AND GAS CHROMATOGRAPHY (*Investigative*) (Procedure, p. 141-142, Procedure, GC, TBA)(**Post Lab Questions:** pp. 143-144, Problems 6,7,13 pp. 208-209, Problem 2) **You do not need a procedure for the GC in the pre-lab writeup and there is no writeup for the Melting Point.**

Notes for Fractional Distillation

- You will be given an unknown mixture of two solvents from the table below.

Solvent	Boiling Point (°C)
Acetone	56.5
Methanol	64.7
Hexane	68.8
Cyclohexane	80.7
Heptane	98.4
Ethyl Benzene	136.2
Toluene	110.6

- Set up the apparatus for the fractional distillation as pictured in Fig. 2.39, p. 59. Use 30-mL of the unknown mixture that is provided. Be sure to insulate your apparatus with cotton wrapped in aluminum foil. This insulation should include the stillpot as described in the laboratory lecture.

- Prior to performing the distillation, prepare a graph in your notebook for plotting the head temperature *vs.* the *cumulative* volume of distillate obtained. During the distillation, look for plateaus, collect three fractions, A, B and C, and record their respective volumes. Measure the amount of residual liquid in the distillation flask (if any) so that a % composition of distilled liquid can be calculated. Also record the boiling points of A and C.

Notes for Gas Chromatography

- Each person is to shoot fractions A, B and C. Your TA will shoot the original sample and provide the necessary data. Identify what you think are the correct solvents by their respective boiling points. Then shoot those two solvents on the GC to confirm that identity.
- Use the data from your traces to calculate the percent compositions of the original mixture and fractions A, B and C. Include these calculations in your Final Report. Record the results a table having the following form.

Sample	Boiling range (°C)	Volume (mL)	% Composition
Fraction A	58-60	8	90.5:9.5
Fraction B	60-102	7	NA
Fraction C	102-110	15	14.7:85.3
Mixture	NA	30	30.2/69.8

- In the "Conclusions" section of your final report, determine the percent composition of the unknown mixture by two different methods. Method one is by using the temperature *vs*. volume graph. Method 2 is by using the GC data for each individual fraction collected. Compare the results from each method with the original sample GC data supplied by your TA. Then compare the results with the rest of the lab section.

Period 3	Lab Lecture: 9-13, 9-17	Wet Lab: 9-14, 9-18, 9-19

Due Today
Reading Assignments (due by lecture) Secs. 2.7, 2.21, 3.1-3.3, 5.1–5.3, Supplement A and Supplement B
What Are We Doing Today? (in wet lab)
EXTRACTION AND RECRYSTALLIZATION: DAY 1 (*Investigative*) (Procedure, extraction, miniscale Supplement A, Procedure, recrystallization, Supplement B) (**Product Analysis: MP, % Recovery) (Post Lab Questions:** pp. 167-171, Problems 6,8,10,11)

- Obtain 1 g of the unknown mixture that is a 1:1:1 ratio
- Before coming to lab, prepare a table in your notebook that has the headings and entries shown below.

Compound	Initial Amt. (g)	Crude Amt (g)	Recovery (%)	MP Crude (°C)	Purified Amt. (g)	Recovery (%)	MP Purified (°C)
Acid							
Base							
Neutral							

- For the recrystallizations in this experiment, recrystallize each crude sample in the appropriate solvent provided by the TA.

Period 4	Lecture: 9-20, 9-24	Wet Lab: 9-21, 9-25, 9-26

Due Today
Reading Assignment (due by lecture) Secs. 7.1, 7.3-7.5
What Are We Doing Today? (In wet lab)
EXTRACTION AND RECRYSTALLIZATION: DAY 2 (STEREOCHEMISTRY:ISOMERIZATION OF DIMETHYL MALEATE (*Preparative)* and ANALYSIS OF CARVONES (*Investigative*) (Procedure, miniscale, pp. 220-221, Procedure, p. 225-226 Part 1, Procedure, polarimetry, TBA) **(Product Analysis: MP, % Yield) (Post Lab Questions:** pp. 222-223, Problems 2,4,6 pp. 226-227 Problem 8)

Notes for Stereochemistry

- Use *great* care in handling the bromine solutions, as specified in the "Safety Alert!".
- For the sample to be kept in the dark, wrap the tube in aluminum foil and leave it on the bench-top.
- Test the odor and determine the optical rotation of the carvone samples provided.

Period 5	Lecture: 9-27, 10-1	Wet Lab: 9-28, 10-2, 10-3

Due Today
Reading Assignment (due by lecture) Secs. 9.1, 9.3, 10.1, 10.4 and 10.6
What Are We Doing Today? (In wet lab)
FREE-RADICAL CHAIN REACTIONS: BROMINATION OF ARENES (*Investigative*) (Procedure, pp. 326-328) **(Product Analysis: Rates of Reactivity) (Post Lab Questions:** pp. 328-329, Problems 2,4,8) ADDITION REACTIONS OF ALKENES: BROMINATION OF (*E*)-STILBENE (*Preparative*) (Procedure, miniscale, pp. 377-378) **(Product Analysis: % Yield, MP) (Post Lab Questions:** p. 381-382, Problems 1,5,8) **These are to be written as two separate reports.**

Notes for Bromination

- Save your product for Period 10 dehydrobromination.

Period 6	Lab Lecture: 10-4, 10-8	Wet Lab: 10-5, 10-9, 10-10

Due Today
Reading Assignment (Due by lecture) Secs. 14.1–14.4 and Sec. 25.9
What Are We Doing Today? (In wet lab)
NUCLEOPHILIC SUBSTITUTION: PREPARATION OF 1-BROMOBUTANE (*Preparative*) (Procedure, miniscale, pp. 467-468) **(Product Analysis: IR, % Yield, Halide Tests)** (**Post Lab Questions:** pp. 470-472, Problems 2, 5, 9, 12) ALKYL HALIDE CLASSIFICATION TESTS (*Investigative*) (Procedure, pp. 869-871) **This is 1 report**

Notes for Substitution

- *Note:* Despite what it says in the textbook, the 1-bromobutane layer obtained during the work-up of the reaction mixture is *not* cloudy.

- Conduct the reaction at ½ scale.

Notes for Alkyl Halide Tests

- Prepare a table in your notebook that has the headings that follow. The results for each test that you perform on each compound are to be entered in this table.

Compound	Sodium Iodide	Silver Nitrate

- Perform the tests on your product and on the other compounds provided *in the hood!* Put all used *and* unused reagents in the waste bottle. Rinse your test tubes once with acetone and put the rinse liquid in the waste bottle. Then wash your glassware at the sink.

- In the "Conclusions" section of your Final Report, explain the reactivities of each compound in terms of its structure and suggest possible structures for products of any positive tests that you observed.

Notes for Next Week

- **WASH ALL GLASSWARE NEEDED FOR PERIOD 7'S EXPERIMENT THIS WEEK SO THAT THEY HAVE A WEEK TO DRY.**

Period 7	Lab Lecture: 10-11, 10-15	Wet Lab: 10-12, 10-16, 10-17

Due Today
Reading Assignments Secs. 19.1–19.4
What Are We Doing Today?
ORGANOMETALLIC REACTIONS: PREPARATION OF BENZOIC ACID (*Preparative*) (Procedures, miniscale, pp. 643-645, and pp. 655-656) **(Product Analysis: IR, % Yield, MP)** (**Post Lab Questions:** pp 660-663, Problems 3, 13, 15, 19)

Notes for Grignard

- All glassware *must* be completely dry! (Place the glassware in the oven for 20 minutes to dry. It is not necessary to flame-dry. Lightly grease the ground joints before placing the glassware in the oven. After drying, assemble the glassware while it is still hot.
- Plan to add 1 or 2 crystals of iodine, *no more* than that, *prior* to beginning to add 2-bromopentane to the magnesium. You will receive additional instructions for initiating the reaction from your AI.
- You will perform this reaction at ½ scale.

Period 8	Lab Lecture: 10-18, 10-22	Wet Lab: 10-19, 10-23, 10-24

Due Today
Reading Assignment (due by lecture) Secs., 18.1 and 18.3 and 17.1, 17.2, 17.4
What Are We Doing Today? (In wet lab)
ALDOL CONDENSATION: PREPARATION OF TRANS-*p*-ANISALACETOPHENONE (*Preparative*) (Procedure, microscale, pp. 620-621) **(Product Analysis: IR, MP, % Yield)** **(Post Lab Questions:** pp. 622-623, Problems 2, 5, 10) REDUCTION OF CARBONYL COMPOUNDS: PREPARATION OF FLUORENOL (*Preparative*) (Procedure, microscale, pp.583-584) **(Product Analysis: IR, MP, % Yield)** (**Post Lab Questions:** pp. 584-585, Problems 2, 3, 6, 11) **These are to be written as two Separate Reports**

Period 9	Lab Lecture: 10-25, 10-29	Wet Lab: 10-26, 10-30, 10-31

Due Today
Reading Assignment (due by lecture) Secs. 20.1,20.2, Supplement C
What Are We Doing Today? (In wet lab)
ESTERIFICATION: PREPARATION OF METHYL BENZOATE (*Preparative*) (Procedure, miniscale, Supplement C) **(Product Analysis: IR, % Yield) (Post Lab Questions:** pp. 676-677, Problems 1, 5, 7)

Period 10	Lab Lecture: 11-1, 11-5	Wet Lab: 11-2, 11-6, 11-7

Due Today
Reading Assignment (due by lecture) Secs. 11.1,11.2, 20.1 and 20.4
What Are We Doing Today? (In wet lab)
CHEMILUMINESCENCE: SYNTHESIS OF LUMINOL (Procedure, microscale, pp. 694 and pp. 695) **(Product Anlysis: Did it Glow)** **(Post Lab Questions:** pp. 695-696, Problems 1, 2, 4) ALKYNE FORMATION: DEHYDROBROMINATION OF *MESO*-STILBENE DIBROMIDE(*Preparative*) (Procedure, microscale, pp. 406-407) **(Product Analysis: % Yield, MP) (Post Lab Questions:** pp. 408-409, Problems 2,4,6) **These are to written as two separate reports**

Notes for Dehydrobromination

- Use the meso-stilbene dibromide that you prepared from period 5. If you did not make enough, some will be provided.

Period 11	Lab Lecture: 11-8, 11-12	Wet Lab: 11-9, 11-13, 11-14

Due Today
Reading Assignment (Due by lecture) Secs. 15.1, 15.4 and 15.5
What Are We Doing Today? (In wet lab)
ELECTROPHILIC AROMATIC SUBSTITUTION: NITRATION OF BROMOBENZENE (*Preparative*) (Procedure, microscale, pp.516-517) **(Product Analysis:% Yield, MP)** **(Post Lab Questions:** pp. 519-520, Problems 2, 5, 6, 9) ELECTROPHILIC AROMATIC SUBSTITUTION: RELATIVE RATES OF REACTION (*Investigative*) (Procedure, pp. 525-526 (part A)) **(Product Analysis: Relative rates of Reaction)** **(Post Lab Questions:** pp. 527-528, Problems 1, 3, 9) **These are to be written as two Separate Reports**

Notes for Rates of Reaction

- Predict the order of reactivity before you come to lab and have that prediction in your notebook.
- Do the rate determination on the following compounds: phenol, 2-bromophenol, 3-bromphenol, 4-bromophenol, anisole, acetanilide, benzoic acid and diphenyl ether.

Period 12	Lab Lecture: 11-15, 11-26	Wet Lab: 11-16, 11-27, 11-28

Due Today
Reading Assignment (due by lecture) Supplement D
What Are We Doing Today? (In wet lab)
PREPARATION OF A DYE: METHYL ORANGE (*Preparative*) (Procedure, Supplement D) **(Product Analysis: Dyeing analysis, pH affect) (Post Lab Questions:** None**)**

Period 13	Lab Lecture: 11-29, 12-3	Wet Lab: 11-30, 12-4, 12-5

Due Today
Reading Assignment (due by lecture) None
What Are We Doing Today? (In wet lab)
CHECKOUT

16. SUPPLEMENTS

A. Acid-Base Extraction (Miniscale)

To prepare for this experiment, study the detailed instructions for using a separatory funnel.

Obtain from your instructor a 1 g of the unknown mixture (it is 1:1:1). Using an Erlenmeyer flask, dissolve the mixture in about 30 mL of ethyl acetate. Transfer the solution to the separatory funnel and the extract it sequentially with three 10–mL portions of 6 *M* hydrochloric acid. Combine the three aqueous acidic layers from the extractions in an Erlenmeyer flask labeled "acidic extract."

Extract the remaining organic layer in the separatory funnel with three 10 mL portions of 6 M NaOH. Combine the three aqueous basic layers from the extractions in an Erlenmeyer flask labeled "basic" extract.

Transfer the organic layer into an Erlenmeyer flask labeled "neutral" extract. Dry the sample with Na_2SO_4.

While the organic solution is drying, cool both of the aqueous extracts in an ice-water bath. Neutralize the "acidic extract" with 6 *M* sodium hydroxide and add a little excess base to make the solution distinctly basic to pH paper. Neutralize the "basic extract" with 6 *M* hydrochloric acid and add a little excess acid to make the solution distinctly acidic to pH paper. Upon neutralization, a precipitate should form in each flask.

Collect the precipitates separately by vacuum filtration. Wash each of the precipitated solids on the Büchner funnel with *cold* distilled water. Collect the filtrate and label them appropriately.

Separate the "organic solution" from the sodium sulfate by gravity filtration and remove the solvent by simple distillation. Discontinue the distillation when only a small amount of material remains in the distillation pot. Allow the pot to cool and then attach it to the water aspirator to remove the last small amount of the solvent. Be sure to have a clean Büchner flask as an aspirator trap. Gently swirl the liquid in the pot to expose a greater surface area and to facilitate vaporization. The pot can also be warmed mildly with the warmth of your hands.

Transfer the resulting solid residue to the third vial and allow it to air-dry in the same way.

After the samples have been dried, re-weigh each of the vials to obtain the weight of the crude solids, and determine the melting point of each of them. The reported melting points are given below.

	Compounds	**MP (°C)**
Acidic	benzoic acid	122–123
	2-methylbenzoic acid	103–105
	2-chlorobenzoic acid	138–140
	salicylic acid	158–160
Basic	4-nitroaniline	149–151
	2-methyl-4-nitroaniline	131–133
	3-nitroaniline	112–114
Neutral	9-fluorenone	82–85
	anthracene	216–218
	fluorene	114–116
	phenanthrene	101–103

Continue the experiment as directed to determine a suitable recrystallization solvent for each solid obtained.

16. SUPPLEMENTS (CONT.)

B. Recrystallization of Acidic or Basic Products

Recrystallization as a purification technique involves the following:

Selecting an appropriate solvent.

The following criteria must be met for a solvent to be used in a recrystallization:

1. The compound should be soluble (approx. 1 g in 20 mL) in *hot* solvent, but insoluble in *cold* solvent.

2. The impurities present in the compound need to either be completely insoluble in the solvent or be completely soluble in the solvent (at all temperatures).

3. The solvent should be volatile enough that it can be easily removed from the crystals.

4. The boiling point of the solvent should be lower than the melting point of the crystals, otherwise the crystals could melt before they dissolve in the solvent and form an oil. This event is called "oiling out" of the solid, and makes the crystals much more difficult to isolate.

Dissolving the crystals.

Place the solid in a suitable container. Add a small volume of solvent and bring it to a boil. After the solvent starts boiling, add small amounts of fresh solvent until all of the solid dissolves. It is important that you use a minimum amount of solvent.

Forming the purified crystals.

Allow the hot solution to cool SLOWLY to room temperature. Cooling the solution too rapidly (by placing it in an ice-water bath) causes crystals to be formed too rapidly and may possibly lead to entrapment in the crystals not only of solvent but also of other impurities. If no crystals form after an appropriate amount of time, several measures can be taken. For example you may seed the solution by adding a crystal or two of the original compound. Also, you may use a glass rod to scratch the side of the container at the air-liquid interface. If all else fails, place the container in an ice-water bath.

Isolating the purified crystals.

Isolate the crystals obtained from the recrystallization by vacuum filtration using a Hirsch or Büchner funnel depending upon the volume of crystals obtained. Then rinse the purified crystals with a small amount of COLD solvent.

Drying the crystals.

The purified crystals are usually allowed to air-dry in a watch glass or a vial.

PROCEDURE

Note: Each lab will be split into groups of 3.

Place about 20 mg of solid in each of 6 test tubes. Label each test tube according to the solvent you will be testing and add about 0.5 mL of solvent to the solid in the test tube.

Label each of the solids in the test tubes as either soluble or insoluble at room temperature. If the solid is insoluble, heat the mixture and again label it as either soluble or insoluble.

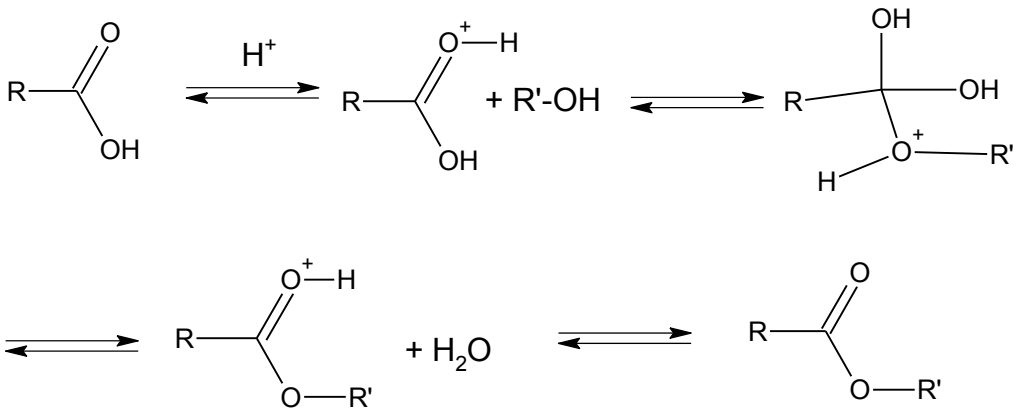
For the solids that are insoluble at room temperature *but* soluble at a higher temperature, let the solutions cool and examine the crystals to see which solvent appears to give the best yield of solid and the highest quality crystals.

Once your group has determined the solvent for your solid, check with your TA who will then inform the rest of the class which solvent you selected. You should then recrystallize all three of the acidic and basic products using the specified solvents.

16. SUPPLEMENTS (CONT.)

C. Fischer Esterification: Synthesis of Methyl Benzoate

Esters are important functional groups that can be synthesized in a number of different ways. Many esters have pleasant odors and are often used in foods and perfumes. One method to synthesize and ester is by combining an alcohol and a carboxylic acid. The problem with this method is that the alcohol is not a strong enough nucleophile to attack the carbonyl carbon of the carboxylic acid. Fischer overcame this problem by adding a strong acid to the reaction, which protonates the carbonyl oxygen generating a better electrophile. Because each step in the mechanism is reversible, care must be taken to avoid reversing the reaction.



We will be synthesizing methyl benzoate from methanol and the benzoic acid that we made previously. Methy l benzoate is commonly used in the perfume industry and the food industry as a flavor additive.

APPARATUS

A 100 mL round bottom flask, a condenser, a stirbar, an aluminum heating block and a hot plate.

PROCEDURE FOR THE FORMATION OF METHYL BENZOATE

Synthesis

Place 1.0 g of benzoic acid and 25 mL of methanol in a 100-mL round-bottomed flask, cool the mixture in ice, pour 1.5 mL of concentrated sulfuric acid slowly and carefully down the walls of the flask, and then swirl to mix the components. Attach a reflux condenser, add 3 boiling chips, and reflux the mixture gently for 1 hr. with the set-up shown.

Isolation and Purification

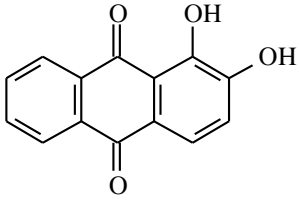
Cool the solution, decant it into a separatory funnel containing 25 mL of water, and rinse the flask with 25 mL of diethyl ether (Use wet ether found in a supply bottle in each hood). Add this ether to the separatory funnel, shake thoroughly, and drain off the water layer, which contains the sulfuric acid and the bulk of the methanol. Extract the ether in the separatory funnel with 25 mL of water followed by 25 mL of 10% sodium bicarbonate to remove unreacted benzoic acid. Again shake, with frequent release of pressure by inverting the separatory funnel and opening the stopcock, until no further reaction is apparent; then drain off the bicarbonate layer into a beaker. If this aqueous material is made strongly acidic with hydrochloric acid, unreacted benzoic acid may be observed. Wash the ether layer in the separatory funnel with saturated sodium chloride solution, and dry the solution over anhydrous calcium chloride in a 125-mL Erlenmeyer flask. Add sufficient anhydrous calcium chloride so that it no longer clumps together on the bottom of the flask. After 10 min, decant the dry ether solution into a dry 50-mL Erlenmeyer flask, wash the drying agent with an additional 5 mL of ether, and decant again.

Remove the ether by evaporation in the hood. When evaporation is complete, add 2 to 3 spatula tips full of anhydrous calcium chloride to the residual oil and air dry for about 5 min longer.

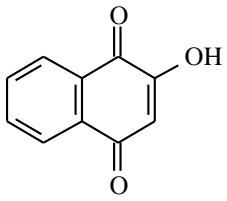
16. SUPPLEMENTS (CONT.)

D. Preparation of Methyl Orange

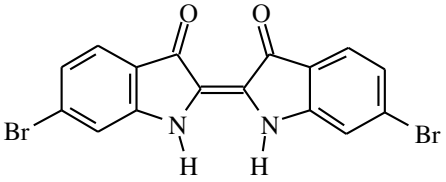
Humans have been using dyes since prehistoric times. Madder, which is today called alizarin, is extracted from the roots of the madder plant and is an example of one of the oldest known dyes; it ranges from brilliant red to orange in color. A similar dye to alizarin is called henna, which has long been used in dyeing human hair red and is derived from the leaves, seeds, and thorns of the henna plant. Another example of an ancient dye is indigo, which is light blue to navy in color and is obtained from the leaves of the indigo plant, *Indigofera tinctoria*; it has been in use in Asia for more than 4000 years.



Alizarin

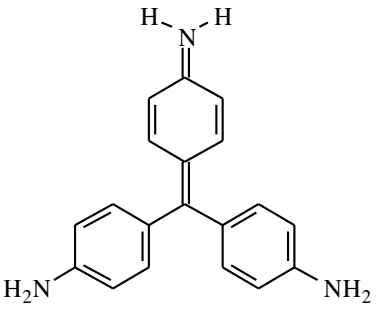


Henna

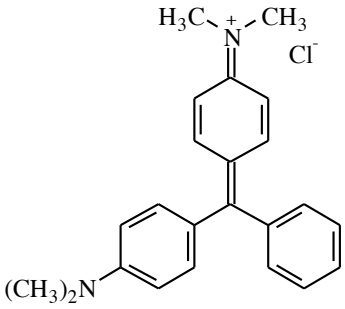


Indigo

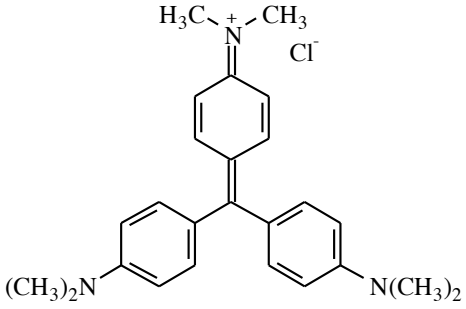
Plants were the main source of dyes until the middle of the 19th century, although other living organisms, insects, for example, provided dyes. In the case of plants, roots, leaves and berries could simply be boiled in water to extract the dyes. Today, dyes are made synthetically rather than being isolated from natural sources. The first such dye, Perkin's Mauve, was accidentally discovered by William H. Perkin, an English chemist, when he attempted the synthesis of quinine from allyltoluidine, His discovery led to the production of pararosaniline, malachite green and crystal violet, all of which are in a group classifed as triphenylmethyl dyes.



Pararosaniline

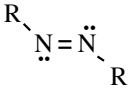


Malachite Green

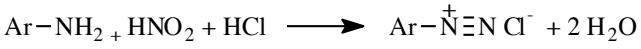


Crystal Violet

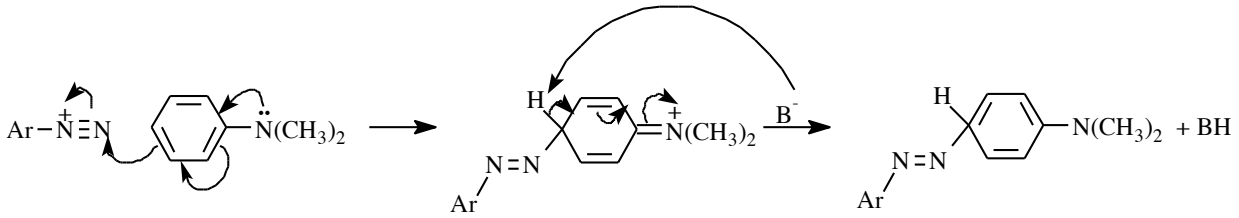
Today, the most common dyes are the azo dyes, which are formed from a coupling reaction between aryl diazonium salts and phenols and other aromatic amines. They are called azo dyes because they contain a nitrogen-nitrogen double bond, is the so-called an azo group. The reaction first involves reaction between nitrous acid and an aromatic amine to produce an aryl diazonium salt, a process called diazotization. Next, the phenol or aromatic amine is added to the salt in a reaction called diazonium coupling.



Azo group (in *anti* form)

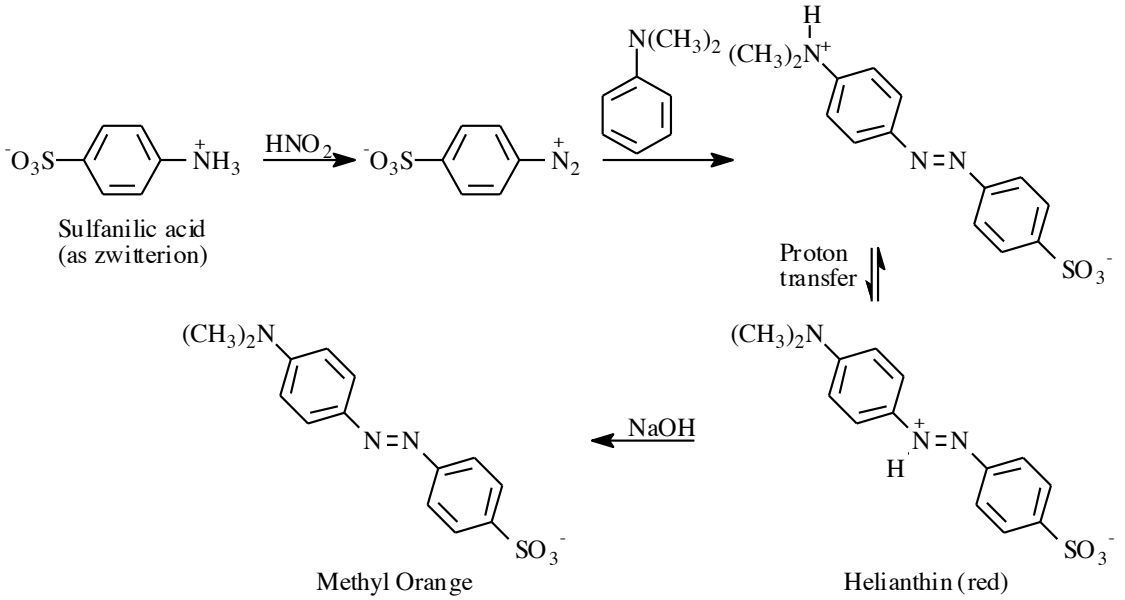


Diazotization



Diazonium Coupling

In the following experiment, you will be synthesizing methyl orange, which is commonly used as an acid-base indicator. It is synthesized from *N,N*-dimethylaniline and sulfanilic acid. The initial product is a bright red intermediate called helianthin, which yields methyl orange after addition of base. After synthesizing the dye, you will test its ability to color cotton. You will also determine if it can be used to indicate the *pH* of different solutions.



APPARATUS

Three 10 x100-mm test tube, a 5 mL conical vial, a heating block, a hotplate for heating, a spinvane, a 25-mL Erlenmeyer flask, a 30 mL beaker, Hirsch funnel and filter flask for filtration.

PROCEDURE FOR THE FORMATION OF METHYL ORANGE

Diazotization of Sulfanilic Acid

Add 1.25 mL of water to a 5 mL conical vial. Next add 120 mg of sulfanilic acid and 35 mg of sodium carbonate. Using a heating block and hot plate, heat the solution to boiling until all is dissolved. Allow the solution to cool to room temperature. Once it has cooled, add 50 mg of sodium nitrite and stir the mixture with a glass rod until the solids are dissolved. Now cool the vial in ice, and with stirring, add 0.75 g of ice and 0.125 mL of *concentrated* HCl. Once the white solid has precipitated, set the vial aside for use later in the reaction.

Formation of Methyl Orange

To a 25-mL Erlenmeyer flask, add 75 mg of *N,N*-dimethylaniline, a spinvane and 65 mg of acetic acid and stir the mixture to achieve homogeneity. Add the diazotized sulfanilic acid slurry from above, rinsing the last of the solid out of the test tube with a few drops of water. Stir the mixture thoroughly. When a stiff paste has formed, add 1 mL of 3 *M* aqueous NaOH. Heat the solution to the boiling point, stirring constantly to avoid bumping. When most of the solid has dissolved, allow the solution to cool to room temperature, and then cool it in ice for 10 min. Collect the crystalline product by vacuum filtration with a Hirsch funnel and rinse it with 3 mL of saturated sodium chloride solution.

Dyeing Test

To a 30-mL beaker, combine 50 mg of recrystallized methyl orange, 0.5 mL of 1 *M* aqueous sodium sulfate, 15 mL of water and 5 drops of 1 *M* aqueous sulfuric acid and mix thoroughly. Bring the solution close to the boiling point and place a small piece of cotton into the solution for 5 min. Remove the cotton, allow it to cool and then rinse thoroughly with tap water at the sink. Allow the cotton to dry and then compare it to undyed cotton.

Indicator Test

Place a few crystals of methyl orangeinto three test tubes. Next add a few drops of 0.5 *M* aqueous HCl to one tube and a few drops of 0.5 *M* aqueous NaOH to a second test tube until the color changes. To the third test tube, add a few drops of water. Record the different color changes you observe for each solution.