Organic Chemistry Lab II
CHE-243

Lab Manual

Prepared by Ralph Fleming, 2017

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Notice

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www.chemistry-solutions.com

Rowan College at Burlington County
Spring 2017

STEM Division
Syllabus: Organic Chemistry Lab II, CHE243

One Credit

Co-requisites: CHE241 Class Meeting Days: Three hours, Once per week per section,
Location: LAB SB212

Instructor(s) Ralph Fleming
Contact Information: rfleming@rcbc.edu cell: 609-354-2179
Office Hours: Mondays 10:00AM to Noon

SECTION 1:

Course Description:
This is the second semester of a two semester transfer organic chemistry lab course, for one credit each. The primary objective of the course is the hands on synthesis, purification, and characterization of organic compounds. Students prepare a research quality notebook of their laboratory work.

Laboratories:
Three hours in the laboratory per week. Laboratory preparation information, laboratory safety information, laboratory procedures, and data are entered for each experiment in a laboratory notebook per procedures outlined in this syllabus. An analysis and an abstract are written for each experiment.

Required Text and other Materials

Laboratory Text: On Blackboard for course

Required Material: Lab coat, ¾ length (available in College Bookstore).

Course Learning Outcomes
Upon completion of the course, students will be able to:
- Demonstrate safe and effective work habits in the laboratory.
- Synthesize and purify organic compounds.
- Operate laboratory equipment with proper technique.
- Construct laboratory reports of their experiments.

General Education Outcomes.

Written and Oral Communication: Communication
* Students will conduct investigative research which demonstrates academic integrity, originality, depth of thought, and mastery of an approved style of source documentation.

Scientific Knowledge and Reasoning: Science
* Students will demonstrate critical thinking skills the analysis of scientific data

Core Course Content

1. A typical sequence in oxidation reactions
2. Friedel-Crafts synthesis
3. EAS reactions
4. Esterification reactions
5. Grignard reactions
6. Intellectual property in the pharmaceutical industry

See RCBC Hand Book http://www.rcbc.edu/publications

SECTION 2: Course Structure

Goals: to introduce the student to:

1. the techniques used in synthesis of organic molecules
1. multi-step synthesis in the preparation of organic molecules
1. safe laboratory practices
1. the structure and writing of a research quality laboratory notebook.

Rationale:
Further study in the fields of biology, biotechnology, medicine, and chemistry requires the student to use his/her knowledge of the basic structure of organic molecules and organic reaction mechanisms to analyze situations and experiences that he/she encounters in the other courses and work experiences in these fields.

Course and Classroom Policies

Preparation

It is important that you come prepared for every experiment. Prepared means that you have completed the required sections of your journal such as the Physical Constants Sheet and CHP before you enter the Lab. Your instructor prior to you starting you lab work will check the lab journal. Deductions will be made for incomplete work. If you are not prepared, you will not be able to start the lab until you are prepared and your journal is complete. If you arrive late and /or not prepared there is a grade penalty and possibility forfeiture of lab period.

Attendance/ Grade Policy

1. chronically arriving late to lab 10% grade penalty per instance

2. missing lab, no makeup, zero (0) grade for that part of the experiment.
3. missing lab, makeup in another section, 10% grade penalty for the experiment.
4. missing lab, no makeup, use instructor data for report, 15% grade penalty for experiment.
5. late notebook, 1 week, 20% grade penalty for the experiment

Behavior Policy

1. Smart phone are permitted as calculators and timers.
2. Texting and phone conversations are not permitted during the lab period.
3. Students can leave the lab for emergency communications.

	Points (%)
Experiment 9	22.50
Experiment 10	22.50
Experiment 11	22.50
Experiment 12	22.50
Intellectual Property	10.00
TOTAL	100.00

Criteria for Grade Determination

Assessment Methods

Academic Honesty:

Students are expected to comply with all college policies regarding academic honesty. Acts of cheating, plagiarism or other acts of academic dishonesty are grounds for failure of the course, suspension from the college, or both. The lab journal represents your work in the course. To copy someone else's lab book information is academic dishonesty. Do not share your lab books or information. Any information obtained from another source other than yourself must be documented as to the source. See RCBC Hand Book http://www.rcbc.edu/publications

Grading: Total grade is based on the content and evaluation of the laboratory notebook per:

Grading Rubric

Grading Rubric

PRELAB exp 9		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	5	
CHP	2		EXPER CALCS	7	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	1		ANALYSIS	15	
Data Table	1		Abstract	15	
Tech: TM, Saf, Prep	7		Tech:TM,Prep	10	
Flow sheet	2		IR	5	
Exp procedure	2				
total	25		*total*	75	

PRELAB exp 10		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	10	
CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
TECH	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		*total*	75	

PRELAB exp 11		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	10	
CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
prep/tech	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		*total*	75	

PRELAB exp 12		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	10	
CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
Tech	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		*total*	75	

Intellectual Property: Report:10 %, Presentation: 90%

For further information on grading, See: http://www.chemistry-solutions.com/

Course Schedule or Calendar

See: http://www.chemistry-solutions.com/

Week 1 : Introductions, Safety, Review
Weeks 2-4: Experiment 9

Weeks 5-7: Experiment 10
Weeks 8-10: Experiment 11
Weeks 11-13: Experiment 12
Week 14: Intellectual Property

Intellectual Property: Report:10 %, Presentation: 90%

For further information on grading, See: http://www.chemistry-solutions.com/

Course Schedule or Calendar

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Week 1 : Introductions, Safety, Review
Weeks 2-4: Experiment 9
Weeks 5-7: Experiment 10
Weeks 8-10: Experiment 11
Weeks 11-13: Experiment 12
Week 14: Intellectual Property

SECTION 3: College Resources

College Policies

In order for students to know their rights and responsibilities, all students are expected to review and adhere to all regulations and policies as listed in the College Catalog and Handbook. These documents can be accessed at rcbc.edu/publications . Important policies and regulations include, but are not limited, to the following:

- College Attendance Policy
- Grading Standards
  - Withdraw (W) and Incomplete Grades (I & X)
  - Withdrawal date for this semester **(will vary)**
- Student Code of Conduct
  - Academic Dishonesty/Plagiarism and Civility
- Use of Communication and Information Technology

Office of Student Support and Disability Services

RCBC welcomes students with disabilities into the college's educational programs. Access to accommodations and support services for students with learning and other disabilities is facilitated by staff in the Office of Student Support (OSS). To receive accommodations, a student must contact the OSS, self-identify as having a disability, provide appropriate documentation, and participate in an intake appointment. If the documentation supports the

request for reasonable accommodations, the OSS will provide the student with an Accommodation Plan to give to instructors. For additional information, please contact the Office of Student Support at 609-894-9311, ext. 1208, disabilityservices@bcc.edu, or rcbc.edu/studentsupport .

Educational Technology Statement

Rowan College at Burlington County (RCBC) advocates the use of technology to enhance instruction. Students should assume that classroom and online technology will be used throughout their coursework at RCBC, as it will most certainly be used in their future education and careers. The College provides on-campus facilities for the convenience of the RCBC community. Various college departments, including the Office of Information Technology and the Office of Distance Education, provide technology training and assistance to faculty and students.

Student Success Services

RCBC offers a variety of free services for its students including those listed below. Descriptions of these services, as well as many others, can be found in the College Catalog and Handbook and on the RCBC website at rcbc.edu/publications .

- Academic Advisement (rcbc.edu/advising)
- Career Services (rcbc.edu/careers)
- Educational Opportunity Fund (EOF) (rcbc.edu/eof)
- Financial Aid (rcbc.edu/financialaid)
- International Students Office (rcbc.edu/international)
- Library/Integrated Learning Resource Center (ILRC) (rcbc.edu/library)
- Office of Veteran Services (rcbc.edu/vets)
- Student Support Counseling (rcbc.edu/cpit)
- Tutoring Center (rcbc.edu/tutoring)
- Test Center (rcbc.edu/testcenter)
- Transfer Services (rcbc.edu/transfer)

Grading

Course and Classroom Policies

Preparation

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5

PRELAB exp 9		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	5	
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Exp procedure	2				
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CHP	2		EXPER CALCS	8	
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TECH	5		Tech	5	
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PRELAB exp 11		GRADE	POSTLAB		GRADE
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CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
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RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
prep/tech	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		***total***	75	

PRELAB exp 12		GRADE	POSTLAB		GRADE
ATTENDANCE			ATTENDANCE		
GP	2		EXPER PRO	10	
CHP	2		EXPER CALCS	8	
Physical Constants	4		Flow sheet	8	
PRELIMINARY CALCS	4		DATA TABLE	10	
RX Equation	3		ANALYSIS	14	
Data Table	1		Abstract	15	
Tech	5		Tech	5	
			IR	5	
Exp procedure	4				
total	25		***total***	75	

ORGANIC CHEMISTRY LABORATORY

KEY ELEMENTS TO CONSIDER IN REPORTS:

ABSTRACT:
Does it have the proper scope and depth for an abstract?
Would someone reading this abstract know what was done? Why it was done? What is the conclusion? In other words, does the abstract include the objective of the research, results, and a concluding statement?
Does it include data and facts and avoid subjective terms?
ANALYSIS:
It is comprehensive?
Does it propose reasonable explanations for yield and purity results in synthesis experiments?
When applicable, does it address the mechanism of the reaction?
When applicable, does it address side products? Purification techniques?
EXPERIMENTAL PROCEDURE:
Does it include enough detail that someone to reproduce their work and obtain the same results? Does it contain key observations, dates, and conditions? , Does it contain any modifications to, or elements not explicit ion the General Procedure?
SPECTRA/CHROMATOGRAMS:
Are all required spectra/chromatograms present?
Are they clearly labeled?
Do they contain required sample preparation and instrument data?
Are key elements identified and compared to standards?
DATA TABLES:
Are they comprehensive and neat?
Do they contain the proper units when required?
Does the data table serve to help the reader understand the results and conclusions?
FLOW SHEETS:
Does it accurately diagram what was done in the experimental procedure?
Are all reagents, side products, and final products accounted for?
CALCUALTIONS:
Are all required calculations present? There should be one example of each unique calculation.
Are they accurate, clearly labeled, and include the proper units?
TECHNIQUE (LAB):
Does the student work safely, wear PPE, keep lab clean, and dispose of waste properly?
Does the student effectively use instrumentation and lab equipment?
TECHNIQUE (LAB BOOK)
Is the report neat? Does it follow the protocol for keeping research quality notebooks?

GRADING OF LABORATORY LAB NOTEBOOK: ANAYSIS, ABSTRACT, TECHNIQUE
CHE-241, CHE-243

The **Analysis** section of the notebook is evaluated based on the following:

1. Form: format, header, title, paragraphs are used to separate points.
2. Function: the writing is readable, organized, logical order, free of mistakes, proper grammar, and punctuation. Avoid informal wording, addressing the reader directly, and jargon, or slang. Use concrete terms. Do not use pronouns.
3. Content: all parts of the experiment should be discussed and analyzed. Key elements reported, (percent yield, purity, IR, MP, RI, relative comparison to Literature or standard values, note compounds, structures, impurities, and any relationships). Do not include procedures.
4. Conclusion/summary: A strong affirmative conclusion or summary statement. Identify structures or compounds.

The **Abstract** section of the notebook is evaluated based on the following:

1. Form: Abstract format: citation, header, author, and title. Written in the past tense. A single paragraph, no more than three. Be concise.
2. Function: the writing is readable, organized, logical order, free of mistakes, proper grammar, and punctuation. Avoid informal wording, addressing the reader directly, and jargon, or slang. Use concrete terms. Do not use pronouns.
3. Content: Key elements reported, (percent yield, purity, IR, MP, RI, relative comparison to Literature or standard values, note compounds, structures, impurities, and any relationships).
4. Conclusion/summary: A strong affirmative conclusion or summary statement. Identify structures or compounds.

An abstract is a concise summary of the completed experiment. In a minute or less a reader can learn the rationale behind the experiment, the general procedure, pertinent results, and the conclusions of the experiment.

The Technique evaluation: Laboratory notebook preparation, time management, neatness, safety, general understanding due to preparation, lab techniques.
(1/8/2011)

9A

Suggestions, comments for: _____ Exp #: _____

_____Well documented experiment

_____ Incomplete documentation of experiment, missing:
_____

_____

product

Analysis.

_____ Definitive, concise.

_____ Incomplete

_____ Need a defining statement on the chemistry of experiment.
_____ missing explanation of data and /or missing data for analysis
_____Need a strong summary conclusion

Other :_____
Abstract.

_____ Definitive, concise.

_____ Incomplete

_____ Not an abstract of experiment
_____ Need a defining statement on the chemistry of experiment.
_____ Give data and analysis.
_____ Need a strong summary conclusion based on objectives
Other :_____
File; suggestionsgradenote2015

Code of Academic Integrity

SEARCH: Registered Web Pages (CU Search) GO

Principle

Absolute integrity is expected of every Cornell student in all academic undertakings. Integrity entails a firm adherence to a set of values, and the values most essential to an academic community are grounded on the concept of honesty with respect to the intellectual efforts of oneself and others. Academic integrity is expected not only in formal coursework situations, but in all University relationships and interactions connected to the educational process, including the use of University resources. While both students and faculty of Cornell assume the responsibility of maintaining and furthering these values, this document is concerned specifically with the conduct of students.

A Cornell student's submission of work for academic credit indicates that the work is the student's own. All outside assistance should be acknowledged, and the student's academic position truthfully reported at all times. In addition, Cornell students have a right to expect academic integrity from each of their peers.

I. GUIDELINES FOR STUDENTS

A. General Responsibilities

1. A student shall in no way misrepresent his or her work.

2. A student shall in no way fraudulently or unfairly advance his or her academic position.

3. A student shall refuse to be a party to another student's failure to maintain academic integrity.

4. A student shall not in any other manner violate the principle of academic integrity.

B. Examples of Violations

The following actions are examples of activities that violate the Code of Academic Integrity and subject their actors to proceedings under the Code. This is not a definitive list.

1. Knowingly representing the work of others as one's own.

2. Using, obtaining, or providing unauthorized assistance on examinations, papers, or any other academic work.

3. Fabricating data in support of laboratory or field work.

4. Forging a signature to certify completion of a course assignment or a recommendation to graduate school.

5. Unfairly advancing one's academic position by hoarding or damaging library materials.

6. Misrepresenting one's academic accomplishments.

C. Specific Guidelines for Courses

1. Examinations. During in-class examinations no student may use, give, or receive any assistance or information not given in the examination or by the proctor. No student may take an examination for another student. Between the time a take-home examination is distributed and the time it is submitted by the student for grading, the student may not consult with any persons other than the course professor and teaching assistants regarding the examination. The student is responsible for understanding the conditions under which the examination will be taken.

2. Course Assignments. Students are encouraged to discuss the content of a course among themselves and to help each other to master it, but no student should receive help in doing a course assignment that is meant to test what he or she can do without help from others. Representing another's work as one's own is plagiarism and a violation of this Code. If materials are taken from published sources the student must clearly and completely cite the source of such materials. Work submitted by a student and used by a faculty member in the determination of a grade in a course may not be submitted by that student in a second course, unless such submission is approved in advance by the faculty member in the second course. If a student is submitting all or part of the same work simultaneously for the determination of a grade in two or more different courses, all faculty members in the courses involved must approve such submissions.

3. Academic Misconduct. A faculty member may impose a grade penalty for any misconduct in the classroom or examination room. Examples of academic misconduct include, but are not limited to, talking during an exam, bringing unauthorized materials into the exam room, and disruptive behavior in the classroom.

a. The faculty member must promptly notify the student of the reason for the imposition of a penalty for academic misconduct and the degree to which his
or her grade will be affected.

b. Academic misconduct is not a violation of academic integrity. The student may, however, seek review by the Academic Integrity Hearing Board on the basis either that the finding of guilt is arbitrary and capricious or that the penalty for academic misconduct is excessive or inappropriate to the circumstances involved. ("Arbitrary and capricious" describes actions which have no sound basis in law, fact, or reason or are grounded solely in bad faith or personal desires. A determination is arbitrary and capricious only if it is one no reasonable mind could reach.)

D. Principles for Computer Use and Network Systems

The use of computers and network systems in no way exempts students from the normal requirements of ethical behavior in the Cornell University community. Use of a computer and network system that is shared by many users imposes certain additional obligations. In particular, data, software and computer capacity have value and must be treated accordingly.

Although some rules are built into computer and network systems, such restrictions cannot limit completely what students can do. In any event students are responsible for their actions whether or not rules are built in, and whether or not they can circumvent them.

Standards of behavior include:

Lab Journal Protocol, Instructions, References

11

The Laboratory Notebook

prepared by Ralph Fleming

Background

Documentation, a system of records, is essential in any laboratory. Documents are a record of what was done, when, how, why, and by who. If a researcher is working on a potential patent or their work is being review by governing agencies, proper documentation is essential.

The Food and Drug Administration, FDA, overseas: the production and distribution of foods and pharmaceuticals, animal testing, clinical trials, medical devices, and over the counter drugs. The policy of the FDA is that if it is not written it was not done. The FDA specifies what shall be recorded and how data shall be recorded in The Code of Federal Regulation, CFR, Title 21, Part 211.194 Laboratory Records. (This part can be found at the fda.gov web site)

The ISO 9000 product quality system, Quality System, ISO 4.2.1 General and Quality Planning 4.2.3 requires documentation as means of ensuring that products conform to specified requirements.

General

The laboratory notebook or journal is the primary form of documentation in the research laboratory. A laboratory notebook is a legal document. It can be used to establish a patent claim, to assign credit for a discovery, to document data for publication, and document historical data. It is critical that the laboratory notebook be used properly and consistently. Since the laboratory notebook has legal significance (grading significance for students), the following formatting rules, guidelines and specifications apply:

Reference: Basic Biotechnology Laboratory Methods in the Regulated Work Place, Madison Area Technical College, Lisa Seidman, NSF ATA grants,

Laboratory Notebook Protocol

The book specification: Composition Book, 9 1/2in. x 7 in. , or 8 ½ in x11in, bound (sewn), ruled pages

- 1st , Enter your name , class , and book volume on the out side cover for identification.
- 2nd, Number every page of book, right side only
- 3rd, Label the top of the first page of the book as : **Table of Content**
- All entries are in chronological order by date
- All entries are in pen: black
- All entries are made direct to book (no scrap paper or other notebooks)

- Entries are make on right hand pages only, left hand pages are for class notes, calculations, instructions
- State sources for all literature values or outside sources of information.
- Every right page is date when the entry is made
- Every right page has the Experiment # , Experiment name , and date as a header
- Every right page at the end, lower right designates continuance or end
- Every right page is initialed when completed
- The Table of Contents shall reflect all entry pages by Exp. # and Name with page numbers and dates of the work.
- Never rip out any pages
- Cross out, do not erase or white out mistakes. Use a single strike through line and initial the cross out.
- Do not leave blank page or blank areas on a page, draw a diagonal line through the unused portion or pages.
- tables may be drawn in or taped in from a lab manual or spread sheet. Note: proper credit must be given to the author. Include date of entry.
- Hand outs with procedures , charts, reference data, etc. may be taped in with the referenced author , book, etc. and date of entry.
- Spreadsheets may used and be taped in from student developed spread sheets. Note: must have typed on spreadsheet: name, date, citation. Note: proper credit must be given to the author. Include date of entry.
- Use APA format for all citations

Entry Protocol for Organic Chemistry Laboratory Experiments, Che 241, 243

Organic students will obtain from the bookstore:

- **Instructional Supplements for : Organic Chemistry Lab Che-241 or Che-243, BCC,** This will contain a course syllabus and the Experiments.
- **3, bound laboratory notebooks.**

1st, students should familiarize themselves with **Laboratory Note book Protocol** and prepare their books per the **Laboratory Note book Protocol.**

Specific Organic Laboratory Note book Protocols:

- Bound, 8 ½" x 11" sewn composition notebooks either lined or quad ruled are used.
- All entries are made in black ink.
- A Table of Contents on the first page of the notebook lists each experiment by number, name and lab book page number.
- All right side pages in the notebook are numbered consecutively, in the top right corner, starting with page 101. Notebook 2 will start with page 201, etc.
- Right side pages are used for all entries of record; left side pages may be used for rough notes, sketches, etc. The left side pages are **never** used for recording data, procedures or observations.
- Every page is dated in the top right corner. Additional entries made at later dates on the same page are dated at the beginning of the entry.
- Each experiment is titled with the experiment number and the experiment name. Each section of the experiment has a separate header within the experiment
- The order of the section is:
    1. Purpose
    2. General Procedure
    3. Reaction Equation
    4. Preliminary Calculations
    5. Physical Constants Table
    6. Flow Sheet
    7. Data Table
    8. Experimental Calculations
    9. Diagram
    10. Experimental Procedures
    11. Supplements, IR Spectrograms/chromatograms
    12. Analysis
    13. Abstract
- All experimental entries and procedures are made **directly** into the laboratory notebook **as the work is done**.

- Use consecutive, right side pages; do not skip whole pages. However, a page with a title on it, to be completed later, is not a skipped page.
- When an experiment (or an individual section) is continued to another non-consecutive page—use exp. # (section) continued to page... and exp. # (section) continued from page....
- Do not make entries for different experiments on the same page.
- If a mistake is made line through the erroneous entry, leaving it legible, explain briefly and make new entry. **Never erase or tear page out of the notebook.**
- Give sources for all literature values or information cited.

2nd, student should read the experiment to be started completely.

3rd Prepare a list of chemicals to be used in the experiment. All chemicals involved in the experiment are to be listed in your laboratory notebook appendix, starting with the last page and working toward the front.

(In the Appendix of your journal, back of book)

- **Chemical Hazards and Precautions Appendix** (CHP) - all chemicals involved in an experiment are to be listed, with hazards and precautions, in an appendix:

  a. Start the appendix on the last page of the notebook, and work forward, using both right and left side pages.
  b. Number each chemical in the appendix, sequentially for each book; this is the CHP reference number. Example: the first chemical listed in Lab Book 1 would be numbered 1-1.
  c. List the CAS number from the Chemical Identification & Hazard List given to you.
  d. List the NFPA rating as : H-2, F-3, R-1. Notations such as oxy or w are included.
  e. For any chemical with any NFPA rating of 2 or greater, or if the rating sheet indicates - look up and record **all** hazards and precautions.
  f. List spill and waste code letters. Tape comment pages in the back of the lab book. Extra pages are available in lab.
  g. List flash points.
  h. Reference the source of the additional information on the chemicals, i.e. Merck, HCP, Lange, MSDS, NJ fact sheet, etc.
  i. Physical constants may be included for convenience.
  j. A chemical needs to be listed only once; when used in a subsequent experiment, the original entry may be given as the CHP reference number in the experiment Physical Constant Sheet.

CHP APPENDIX

CHP #	Compound	CAS #	NFPA Rating	S	W	Flash Pt.	Reference
11-1	Benzoic Acid	65-85-0	H-2, F-1, R-U	n	n	121°C	MSDS
11-2	Methanol	67-56-1	H-1, F-3, R-0	f	h	12°C	BOTH
11-3	Concentrated Sulfuric Acid	7664-93-9	H-3, F-0, R-2	a	h	208°C	BOTH
11-4	Methyl Benzoate	93-58-3	H-0, F-2, R-0	f	n	83°C	MSDS
11-5	Water	7732-18-5	N/L	N/L	N/L	100°C	NONE
11-6	Diethyl Ether	60-29-7	H-2, F-4, R-1	f	h	-45°C	BOTH
11-7	Sodium Carbonate	497-19-8	H-2, F-0, R-0	n	h	N/L	MSDS
11-8	Sodium Chloride	7647-14-5	H-1, F-0, R-0	n	n	N/L	MSDS
11-9	Anhydrous Magnesium Sulfate	7487-88-9	H-1, F-0, R-0	h	h	N/L	MSDS
11-10	Concentrate Hydrochloric Acid	7647-01-0	H-3, F-0, R-0	a	h	N/L	MSDS

Hazards & Precautions

11-1 **Benzoic Acid:** Warning! Causes eye irritation. May cause irritation to skin and respiratory tract. May form combustible dust concentration in air.

11-2 **Methanol:** Poison! Danger! Vapor harmful. May be fatal or cause blindness if swallowed. Flammable liquid and vapor.

11-3 **Concentrated Sulfuric Acid:** Warning! Causes irritation to skin and eyes and is harmful when ingested.

11-4 **Methyl Benzoate:** Warning! Harmful if swallowed or inhaled. Causes irritation to eyes and skin. Combustible liquid and vapor.

11-5 **Water:** N/L

11-6 **Diethyl Ether:** Danger! Extremely flammable liquid and vapor. Harmful if swallowed, inhaled or absorbed through skin.

11-7 **Sodium Carbonate:** Danger! May cause eye burns. Harmful if swallowed or inhaled. Causes irritation to skin and respiratory tract.

11-8 **Sodium Chloride:** Causes eye irritation.

11-9 **Anhydrous Magnesium Sulfate:** Caution! May be harmful if swallowed.

11-10 **Concentrated Hydrochloric Acid:** Poison! Danger! Corrosive. Liquid and mist cause severe burns to all body tissue. May be fatal if swallowed or inhaled. Inhalation may cause lung damage.

HS

Dec 1, 2011

EXPERIMENT ENTRIES, The write-up for each experiment includes:

Pre-Laboratory Write-up: This is the **information needed to begin work on the experiment in the lab and must be completed and signed by the instructor or instructional assistant before the student may begin work**. This includes, in the order given:

1. **Purpose**: The reason for doing the experiment.
2. **General Procedure**: A synopsis of the directions from the experiment sheet. This covers all experimental points in brief, sufficient to do the experimental work.
3. **Reaction Equations**: For preparative experiments, equations for all the chemical reactions taking place in the experiment, including main and side reactions. (See Additional Comments). Reactions for purification steps need not be included.
4. **Preliminary Calculations Work Page**: **All** preliminary calculations made involving limiting reagent, theoretical yield, weights, moles, mole ratios, etc., on one page, or pages if necessary.
5. **Physical Constant Sheet**: This form is obtained in the lab and taped into the notebook. All chemicals involved in the experiment are included: CHP numbers and all physical constants necessary in the course of the experiment are given. Limiting reagent, theoretical yield, weights, moles, mole ratios, etc., are included for preparative experiments. (See Additional Comments)
6. **CHP Appendix**: Complete for all chemicals in the experiment (back of book).

The pre-write-up must be written in or word processed, no "cut and pasting" from Experiment Sheets is allowed. The Physical Constant Sheet must be taped in. All pre-write-up sections must precede the first experimental entry.

Laboratory Write-up: The information **obtained and used during the laboratory session. The laboratory write-up must be signed prior to leaving the lab**. This includes, in the order given:

1. **Data Table**: This should be filled in as experimental results are obtained. Each set of data should be identified. DO NOT TYPE DATA TABLE OR RECOPY DATA
2. **Experimental Calculations Work Page**: Includes **all** experimental calculations made during the experimental work and/or involving results, on one page, or pages if necessary.

3. **Flow Sheet**: This is a schematic of the purification procedure which is developed from the general procedures and physical constants for all preparative experiments. (See Additional Comments).
4. **Experimental Procedure**: An exact and detailed record of what is done and observed in the course of the experiment. This is written as the lab work is done. It need not be in sentence form but must be legible and sequential. A diagram of any new apparatus used in the experiment is included. (See Additional Comments).
5. **Spectrographs/chromatograms**: Any such run on the course of the experiment, or given to the student as part of the experiment, are taped in the lab book, properly identified.

Post-Laboratory Write-up: Through CHE 241 the topics to be included in this section will be specified in the experiment sheet. In CHE 243 it will be up to the student to identify and include the necessary information.

The **Analysis** is a statement of the conclusions, which may be arrived at from the results obtained: i.e., a comparison of methods used; yield and purity of a product; etc. Experimental numbers and the corresponding literature values are included in the statement to support the conclusions made.
The analysis only will be graded on the writing. However, in this section as in the entire experiment, the **content** is the major consideration in the grading.
As a guide for the student when writing the analysis, the following information is provided: Each topic paragraph in the analysis should include:
1. a statement of results, including experimental numbers
2. a comparison to literature values or theoretical values
3. a conclusion drawn from the above

The last paragraph is a summary containing the conclusions drawn in the topic paragraphs, without explanation. As for any good writing, it is suggested that the student begin with an outline, to organize the work and make sure nothing is omitted, and write at least one draft before writing the final copy in the lab notebook.

The **Abstract** for you experiment will be a paragraph or two stating the objective, discussing the experiments purpose, the chemistry involved, the procedure and techniques used, the results of the experiment, and a summary or conclusion on the results based on the objective.
An abstract is a brief statement of the essential thoughts of a book, article, body of work, or research.

General Guidelines

EXPERIMENTAL PROCEDURE AND OBSERVATIONS:
Experimental procedure and observations are always written in detail as the work is done. Written procedures and observations should contain sufficient detail that another person could duplicate the work precisely, and know if the results are the same. When work is being recorded properly, a small amount of work is done and then recorded, one step at a time. In the rush to get "work" done the student forgets to write what is done; this is counterproductive. The student will find that by writing as the work is done, over all time is saved, steps will be in sequence and details are not forgotten. When a large amount of work is done before recording, more time is taken, steps are out of sequence and important details are left out. All observations of importance are noted. Used correctly, a laboratory notebook is your memory.

TECHNIQUE EXPERIMENTS AND NEW PROCEDURES:
The first time a new technique or procedure is done, it is written out in detail in the experimental section, including safety precautions, start up, shut down, etc. Thereafter when the procedure is used it is referred to as "standard procedure for ...", noting any variations from the standard, which may affect the results, i.e. "pot" size and heat source for all distillations.

DIAGRAMS:
A diagram of a new apparatus, which is assembled from pieces, is included. Diagrams need not be beautiful, indeed, it is a waste of time to draw a perfect diagram. However, the diagram must be large, and **every** piece must be labeled.

USE OF INSTRUMENTS:
Any time an instrument is used, if there is more than one of that particular instrument, the instrument number is recorded. The first time an instrument is used, directions for use are recorded in detail. However, if an instrument manual is given the student, the pertinent page numbers may be referred to, instead. Thereafter, "standard procedure" is used, noting any items that vary, i.e., "thin film on salt plates" or "sealed cell" for all IR spectra; instrument settings for all melting points, etc. The sequential spectrum number for IR and NMR spectrums is recorded. If an instrument has a log book, it is always signed when the instrument is used.

PREPARATIVE EXPERIMENTS:

The planning of such an experiment is as important as the actual manipulation. A Pre-laboratory write-up is done outside the laboratory period and must be checked by the instructor or instructional assistant before an experiment is begun. If this is not completed before the student comes into the laboratory, valuable lab time is wasted. For the first preparative experiment, a slide-tape presentation is given in independent study, and must be completed as well as a pre-write-up before the experiment is begun.

All preparative experiments consist of two sections, the reaction and then the separation and purification of the product.

1. **Equation For Main Reaction(s):** The first thing to do is choose the reagents and write the balanced equations for the expected reaction.
2. **Side Reactions**: Because of the complexity of most organic molecules, the desired reaction is seldom the only one that actually occurs. Those other reactions are called "side reactions". Write equations for any significant side reactions.
3. **Mole Quantities And Theoretical Yield**: The proper quantities of reagents are usually indicated in the procedure given. The quantity of the most valuable reagent is chosen to give the desired quantity of product. The amounts of other reagents used are then usually greater than that indicated by the main reaction, to allow for loss by side reactions or to act as the solvent. Determine which is the limiting reagent by calculating the number of moles of each reagent used and comparing the ratio of moles used with the theoretical mole ratio indicated by the main equation. Then calculate the number of moles and how many grams of product would be formed from the limiting reagent if there were no side reactions and if the main reaction went to completion (this is the "theoretical yield"). See slide-tape presentation for further details. This information is recorded on the Physical Constant Sheet.
4. **Physical Constants And Solubilities**: A knowledge of the physical properties of the reagents is important in carrying out the reaction, and of the product, side products and secondary products in the isolation of the main product from the reaction mixture. These usually are found in a handbook. Record such physical constants and solubilities as are important for the experiment on the Physical Constant Sheets.
5. **Separation And Purification Of Product**: Frequently, the isolation of the desired product from the other products and excess reagents in the reaction mixture requires more thought and knowledge than does the carrying out of the reaction itself. A procedure for the isolation should be planned before the reaction is run. The steps can be shown in a flow sheet which indicates what happens to each substance present in each step of the isolation. Flow sheets will be provided for the first few experiments, with an increasing number of omissions to be supplied by the student.

6. **Results**: The product(s) is(are) collected in prelabelled, preweighed bottles (liquids in narrow mouth glass bottles, solids in wide mouth glass bottles) and reweighed to determine the gram yield. Certain tests are run on the product for identification and determination of purity, either during (boiling point) or following (melting point, refractive index, etc.) final purification. Characterization of the product is done by Infrared spectroscopy. NMR standards may be provided. These tests are included in the experimental procedure and the results entered in the Data Table.

Experiments 9, 10, 11, 12

EXPERIMENT IX

CYCLOHEXANONE

REFERENCES: Introduction to Organic Laboratory Techniques, 3rd Ed., Pavia, Lampman and Kriz
Steam Distillation, p. 582-587
NMR Spectroscopy, pp. 713-728

Macroscale and Microscale Organic Experiments, Williamson
Oxidation: Cyclohexanol to Cyclohexanone, pp. 285-287 and 289-290.

OBJECTIVES: The student should be able to:

1. Run a synthetic reaction which requires temperature control.
2. Separate the reaction products by steam distillation.
3. Determine the purity of the product by refractive index and infrared spectroscopy.
4. Calculate the mole, gram, and volume amounts of reactants given the moles of reactants, or the moles of products.
5. Calculate the moles of reactants given the volumes of the reactants and the w:v percentage or molarity of the solution.
6. Identify the product from the NMR spectrum.

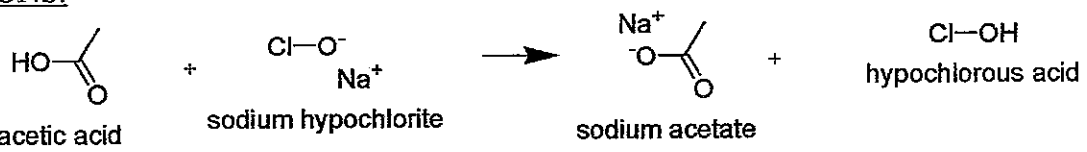
INTRODUCTION:

Cyclohexanone is prepared by oxidation from cyclohexanol. The product and any unreacted alcohol are separated from the inorganic reactants and products by direct (in situ) steam distillation of the reaction mixture. The procedure of purification does not separate the alcohol and ketone well; however, the purity of the ketone can be determined by infrared spectroscopy and refractive index.

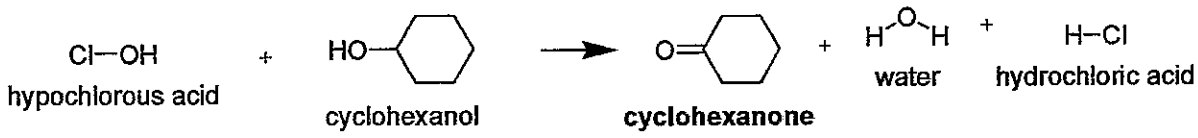
Since the exact concentration of commercial bleach varies, it is necessary to analyse the reaction mixture for unreacted hypochlorite and to reduce any excess to chloride ion with bisulfite.

EQUATIONS:

9A



9B



REAGENTS:

Reactants: Cyclohexanol and
Sodium hypochlorite (commercial bleach solution - 5.25% NaClO or 0.75 M)

Initiator: Glacial acetic acid - 6 ml [1]

Calculate the amount of cyclohexanol required to yield 0.100 moles of cyclohexanone, assuming a 100% yield. Use this theoretical amount of cyclohexanol.

Use 155 ml of commercial bleach solution; calculate the mole quantity used. Calculate the mole ratios of the reactants used.

When calculating amounts of reactants, round final masses to 1 decimal place and final volumes to whole numbers.

APPARATUS:

A 300 ml round-bottom flask with thermometer well
A 250 ml dropping funnel (Separatory funnel used for reagent addition)

DIRECTIONS:

Add the cyclohexanol and glacial acetic acid to the 300 ml flask. Insert an alcohol thermometer in a neoprene themometer adaptor into the side well so the bulb is in the liquid. Add the entire volume of bleach to the dropping funnel and position above the flask with the spout of the funnel in the neck of the flask. (Hood)

The bleach is added dropwise to the flask via the dropping funnel with constant vigorous swirling. The rate should be fast enough to cause the temperature of the mixture to increase slowly. Optimum temperature for the reaction is between 40 ^{o}C and 50 ^{o}C; this range can be maintained by increasing the addition rate or cooling in an ice bath. After all of the bleach solution has been added the reaction is allowed to stand with occasional swirling for 20 minutes.

To test for unreacted hypochlorite, add 3 drops of the aqueous layer of the reaction mixture to one well of a drop plate and add 2 drops of starch/KI solution. The presence of hypochlorite is seen by the blue-black color of iodine/starch complex. If hypochlorite is present, add 1 ml portions of a saturated sodium bisulfite solution to the reaction mixture, mix well and retest until a negative test is obtained.

The reaction mixture is transferred to a 500 ml round bottom flask, rinsing the reaction flask twice with 25 ml portions of water and adding the rinses to the 500 ml flask. Using a simple distillation apparatus the mixture is direct steam distilled until the distillate in the condenser is a single phase and the liquid remaining in the pot is clear. The aqueous layer is saturated with sodium chloride (add 5 gm portions and swirl until the mixture is saturated). The cyclohexanone is separated, and the aqueous layer extracted with ether (10 ml of ether is probably enough).

The combined cyclohexanone and ether extract are dried over anhydrous magnesium sulfate and the magnesium sulfate filtered off. The solution is placed in a 125 ml suction flask and the ether evaporated off by use of a suction set up. The remaining liquid is transferred to a distilling flask and distilled. Cyclohexanone is collected at 140 - 155 ^{o}C. The refractive index and infrared spectrum of the product are determined. From the refractive index, boiling point, and infrared spectrum the product is identified. From the refractive index and infrared spectrum the product purity is determined.

All physical constants are reported.

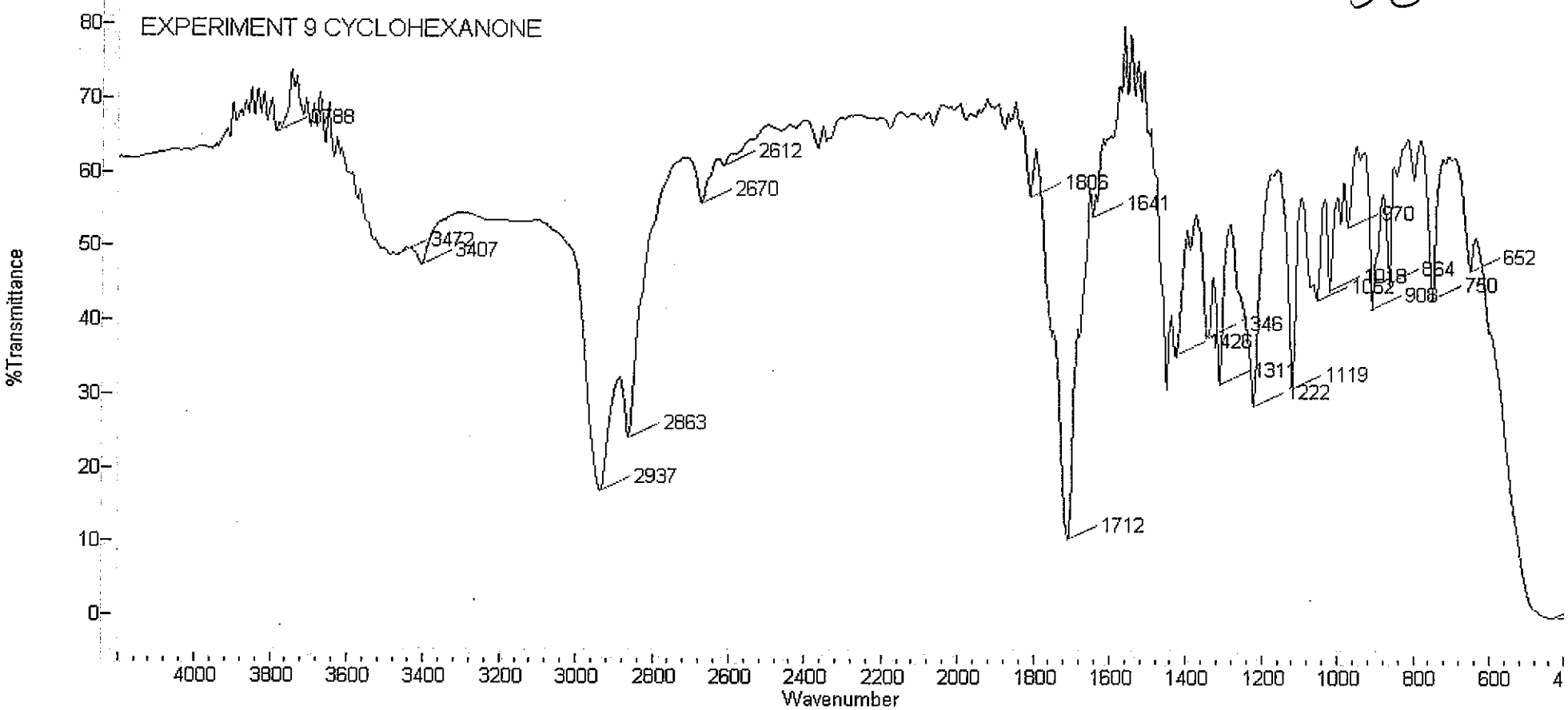
Note 1:

> The glacial acetic acid serves as an intiator in the reaction, by reacting initially with the sodium hypochlorite to form hypochlorous acid. Once the reaction mixture is acidic from the generation of hydrochloric acid in the main reaction, the hypochlorous acid continues to be generated from sodium hypochlorite.

RF
Revised 1/13

Varian Resolutions Pro

58



	Name	Spectrum	Hitlist
15	EXPERIMENT 9 CYCLOHEXANONE		Hits = 0
16	cyclohexanol(7)		Hits = 0

35

EXPT 9 OXIDATION OF CYCLOHEXANOL

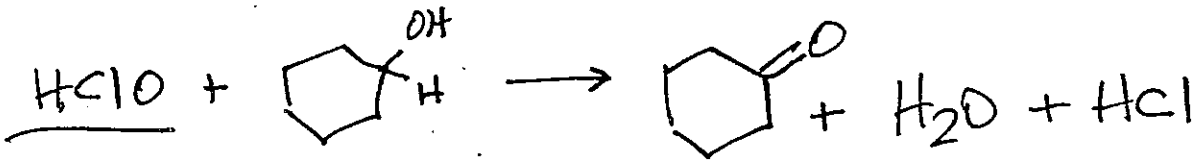
Reference

30 ②

$NaClO + HOAc \rightarrow HClO + NaOAc$

(BLEACH) INITIATOR HYPOCHLOROUS ACID

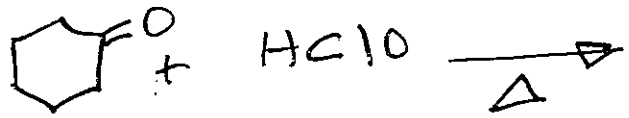
RXN



ONCE SOME HCl PRESENT, FURTHERS INITIATES

$NaClO + HCl \rightarrow NaCl + HClO$

EXCESS NaClO AND HClO AT END MUST BE NEUTRALIZED OR CAN CAUSE SIDE RXN



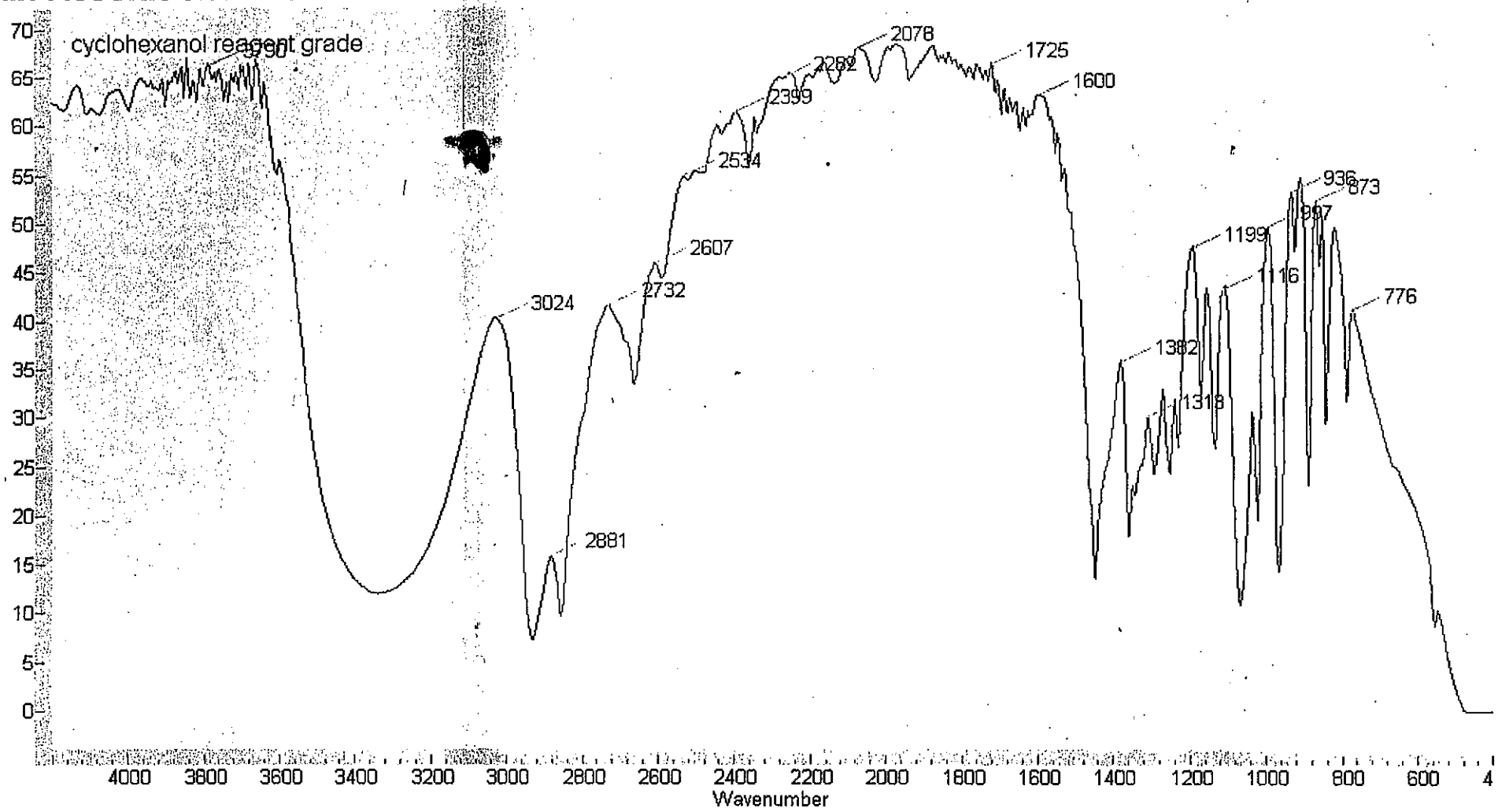
ADIPIC ACID

$HOOC(CH_2)_4COOH$

USE $NaHSO_3$

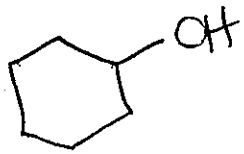
$NaHSO_3 + HClO \rightarrow HCl + NaHSO_4$

Varian Resolutions Pro



2/5/08 TPS

NEAT ON SALT PLATE

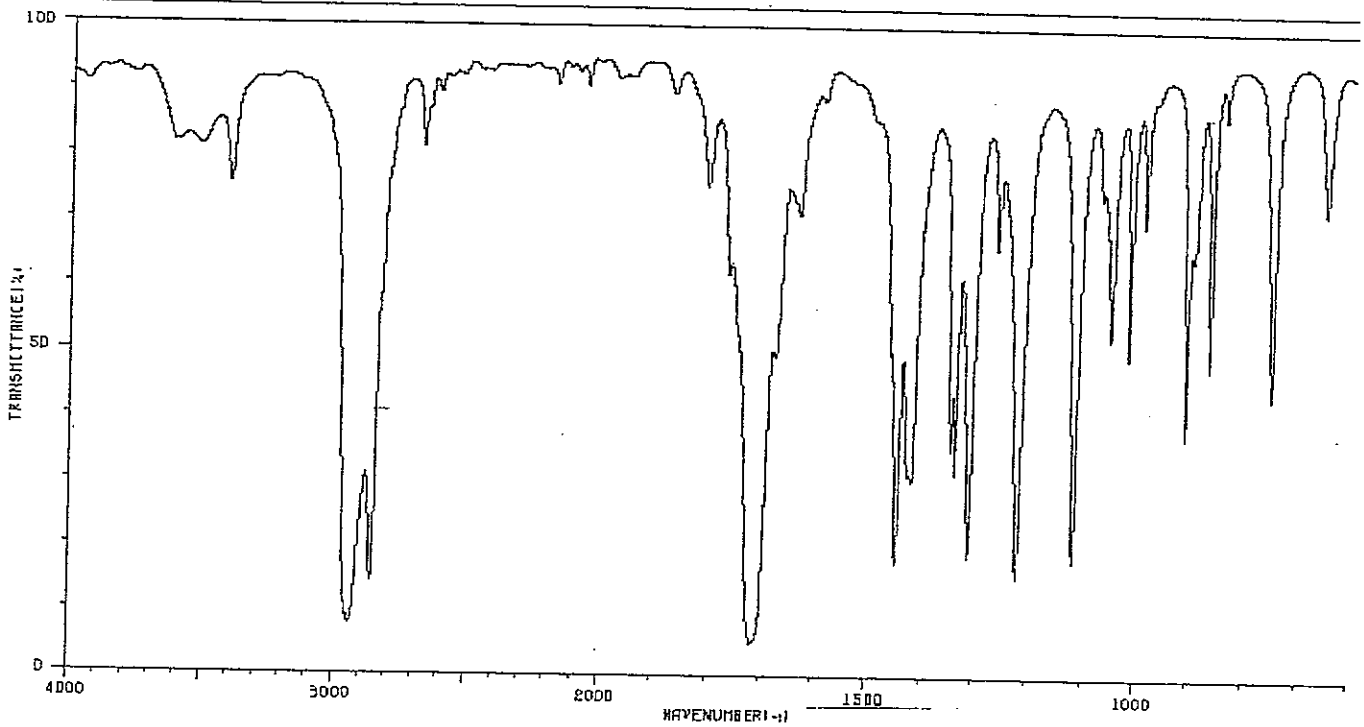


27

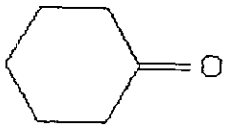
HIT-NO=1070	SCORE= ( )	SDBS-NO=571	IR-NIDA-05262 : LIQUID FILM

CYCLOHEXANONE

$C_6H_{10}O$



3610	79	2611	86	1460	17	1222	16	896	62
3515	79	1870	86	1429	30	1119	17	864	46
3407	72	1808	72	1422	28	1073	72	639	84
2941	7	1766	60	1347	34	1052	50	750	42
2864	13	1716	4	1338	30	1018	47	652	70
2870	79	1677	47	1311	17	951	68	490	33
2654	84	1634	68	1265	64	909	36		



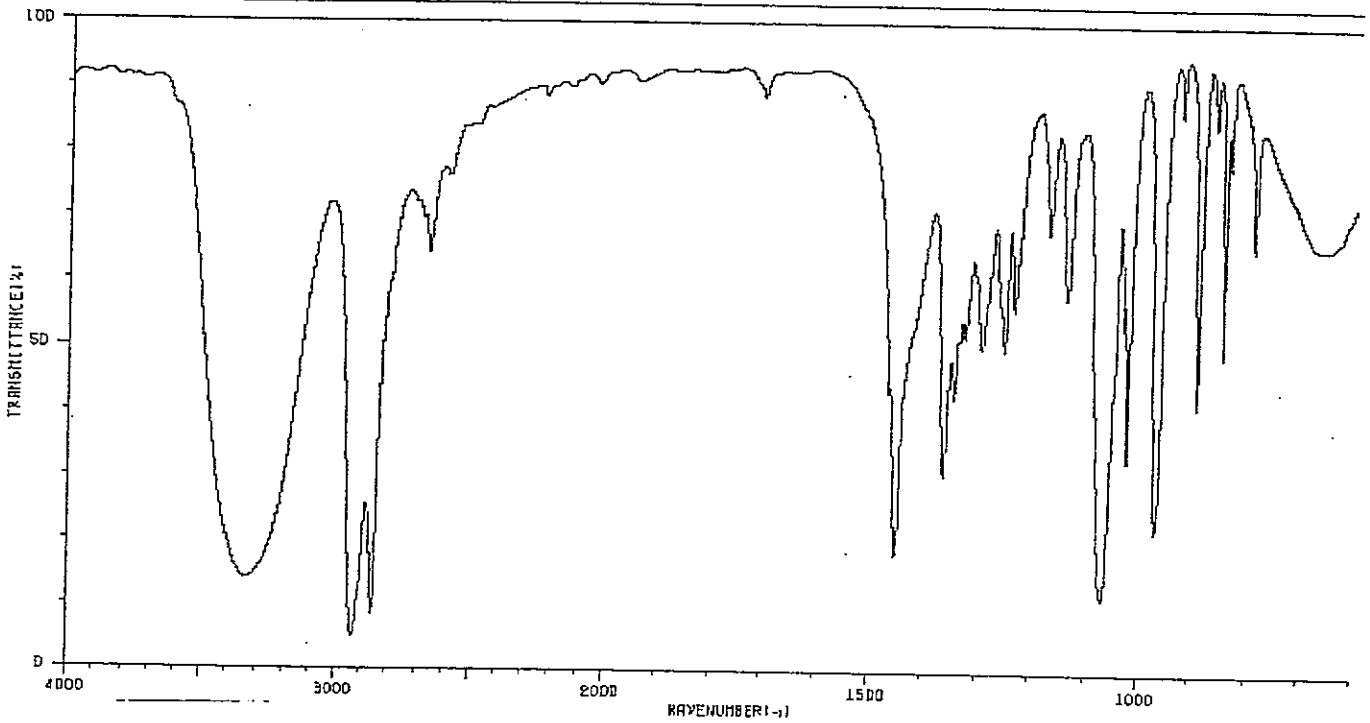
/SDBS/sdbs/OWA/sdbs_SEA.GRE_FRAME_SEA

28

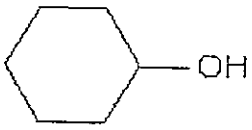
HIT-NO=1077	SCORE= [ ]	SDBS-NO=581	IR-NIDA-09018 : LIQUID FILM

CYCLOHEXANOL

$C_6H_{12}O$



3331	13	1704	86	1266	47	970	21	667	64
2932	4	1467	42	1238	53	926	84	557	53
2855	8	1452	17	1174	86	890	38	482	81
2686	68	1363	29	1140	55	863	81		
2666	62	1346	41	1068	11	845	47		
2588	74	1329	50	1034	52	835	74		
2233	84	1298	49	1025	32	789	64		



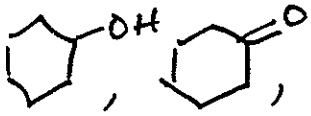
EXPT NINE

1/30/00
TPS

29

FLOW SHEET SAMPLE

RXN FLASK



, H_2O, HAc, $NaHSO_4$, Na^+Cl^-

NaClO/HClO SHOULD BE NEUTRALIZED

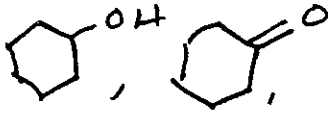
DISTILL

POT

H_2O, HAc

NaCl, OTHER IONS

DISTILLATE



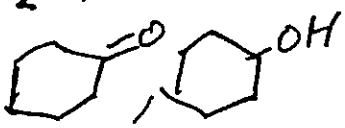
TRACE H_2O

SAT'D NaCl SEPARATE

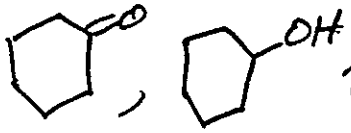
AQUEOUS

H_2O, NaCl

TRACE



ORGANIC



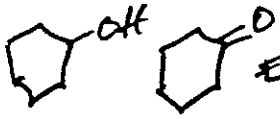
TRACE H_2O

ET_2O EXTRACT

AQUEOUS

H_2O, NaCl

ORGANIC

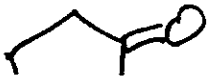


ET_2O

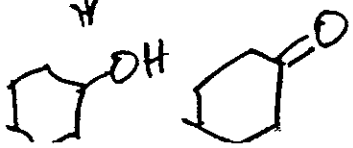
TRACE H_2O

Combine

1. DRY $MgSO_4$
2. FILTER
3. EVAPORATE ET_2O



DISTILL 140°-155°C



CHEMISTRY 243

31

EXPERIMENT X

Friedel-Crafts Reaction - 1,4-Di-t-butyl-2,5-dimethoxybenzene

REFERENCES: Organic Experiments, 7th Ed., Fieser and Williamson, p. 359

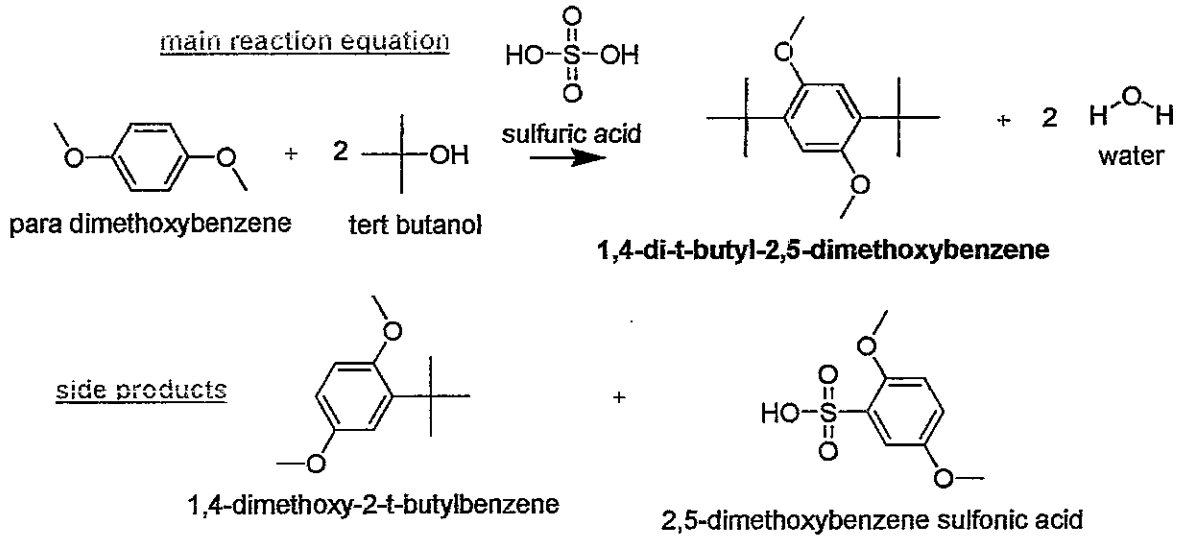
Introduction to Organic Laboratory Techniques, 3rd Ed., Pavia, Lampman and Kriz
Preparing an NMR Sample, pp. 667-671

OBJECTIVES: The student should be able to:

1. Remove an acid from the reaction mixture.
2. Recrystallize a solid product.
3. Calculate the mole and gram amounts of reactants given the grams of product.
4. Identify the product from its melting point, IR and NMR spectra.

INTRODUCTION: This preparation is an example of a Friedel-Crafts reaction occurring on an activated benzene molecule, hydroquinone dimethyl ether. The usual Friedel-Crafts reagents, aluminium chloride and an alkyl chloride are not used to produce the alkyl carbonium ion, the electrphilic reagent in this reaction. Instead a tertiary alcohol and sulfuric acid will be used to produce the electrophilic reagent.

REACTION EQUATIONS:



REAGENTS:

Reactants:	Hydroquinone dimethyl ether (1,4-dimethoxybenzene); t-butyl alcohol
Solvent:	Acetic acid, 20 ml
Catalysts:	Conc. Sulfuric acid, 20 ml;

Calculate the amounts of the reactants required to yield 10.0 grams of 1,4-di-t-butyl-2,5-dimethoxybenzene assuming a 100% yield.

Use this theoretical amount of 1,4-dimethoxybenzene.

Use a 30% excess of t-butyl alcohol.

When calculating amounts of reactants, round mole values to 4 decimal places, final masses to 1 decimal place and final volumes to whole numbers.

APPARATUS:

125 ml Erlenmeyer flask; 50 ml Erlenmeyer flask; 125 ml dropping funnel

DIRECTIONS:

Hydroquinone dimethyl ether[1], t-butyl alcohol and acetic acid are placed in a 125 ml erlenmeyer flask and dissolved on a steam bath. The flask is then cooled in an ice bath until the temperature is 0-3 ^{o}C. The concentrated sulfuric acid are placed in a 50 ml erlenmeyer flask and the flask is placed in the ice bath. A 125 ml dropping funnel is positioned over the 125 ml erlenmeyer flask containing the hydroquinone dimethyl ether solution in the ice bath and the chilled sulfuric acid solution is poured into the dropping funnel. The chilled sulfuric acid is added in slow drops over a period of 15-20 minutes to the hydroquinone dimethyl ether solution. The flask containing the hydroquinone dimethyl ether solution is swirled constantly while remaining in the ice bath. The solid reaction product should have separated at this time and the temperature of the reaction mixture should have risen to 20-25 ^{o}C. Swirl the mixture at 25 ^{o}C for about 5 more minutes and then cool in ice.
(Hood)
Add enough ice to decompose the excess sulfuric acid and then fill the flask with water, cool, and filter the crystals by suction using two filter papers. Apply only very gentle suction to avoid breaking the filter paper while filtering the acid solution and 200 ml of water. Wash with water and then turn on the suction full force. Press down the crystals and allow them to drain well. Turn off the suction and add 10 ml of a cooled solution of methanol to the crystals and apply suction. Repeat this procedure two more times.

The crystals are dried and recrystallized from methanol. The melting point of the product is determined, and IR spectrum are run. The product should be turned in, and all physical constants reported.

NOTES:

1. Grind the hydroquinone dimethyl ether in a mortar and pestle before weighing.

2. Fuming sulfuric acid is highly corrosive and irritating and must be handled with extreme care. A face shield, long sleeved clothing or lab coat, apron and double gloves will be worn while working with this chemical.

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EXPERIMENT_

PCS

EXPT 10

CHP #	COMPOUND	
X	1,4-dimethoxy benzene	START MTL
X	+ BuOH	
X	acetic acid	
X	H_2SO_4	
X	~~[illegible]~~	Delete
X	1,4 dimethoxy 2,5 di t butyl benzene	PRODUCT
X	1,4 dimethoxy 2 t butyl benzene	SIDE PROD.
X	2,5 dimethoxy benzoic acid	SIDE PROD.
X	methanol	
X	H_2O	
	.5	
X	~~CH_2Cl_2~~	Delete
X	~~[illegible]~~ A or C	DELETE

a) Limiting r
b) Theoretica
c) eoretica

5
5
1.
1.5
1.
.5
2
2
1
1
5
5

34

Calculations

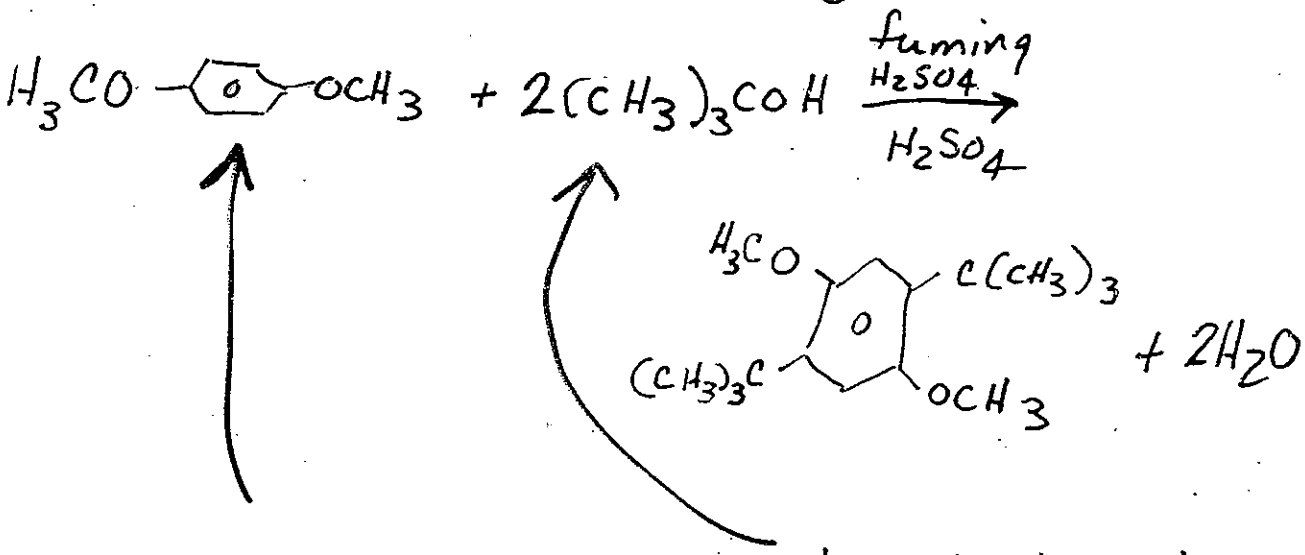
35M

wk 4

Excesses

Ex 10 - 30% excess of t-butyl alcohol

xn eq



Ratio: 1 1,4 dimethoxybenzene : 2 t-butyl alcohol

theoretical moles: .0399 : .0798 ← (.0399 x 2)

100%

excess - .0798 x 1.3 = .1037 moles → 1.0 – 100%
.3 – 30% excess
1.3 (130%)

Easiest if done on mole level

If done at gram level you must back calculate to get new mole amt.

For Your Information:

Exp 10 Solubilities

	MP	Solubility H_2O	M
1,4 dimethoxy 2,5 di-t-butyl benzene	104-106°C	i	sh
1,4 dimethoxy 2 t-butyl benzene		i	s
2,5 dimethoxy benzene sulfonic acid		s	

Ex 10 Melting Points

1,4-dimethoxy benzene	57°C
1,4 dimethoxy 2,5 di-t-butyl benzene	104-106° C

Exp 10 Note:

IR of Product -

Doublet around 1370 & 1390 is characteristic of 2 or 3 methyl groups on the same carbon

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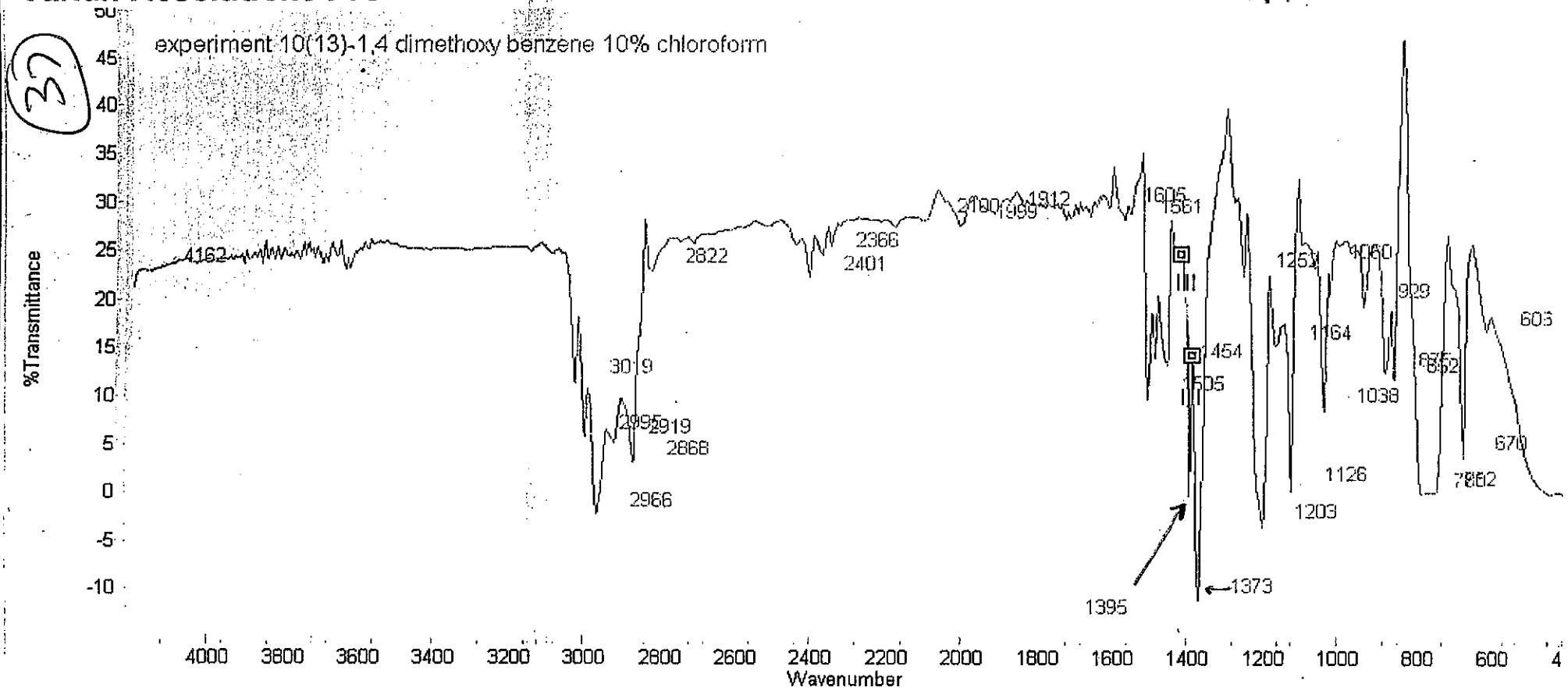
DIFFERENTIAL – CHLOROFORM

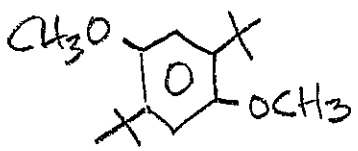
37

experiment 10(13)-1,4 dimethoxy benzene 10% chloroform



	Name	Spectrum	Hitlist	Created Scan Scans	Created Scan Resolution
1	rebecca		Hits = 0	8	4
2	experiment		Hits = 0	8	4

– 10% SOLUTION IN CHLOROFORM
.025 MM CELL



EXPERIMENT XI

ESTERIFICATION - The Preparation of Methyl Benzoate

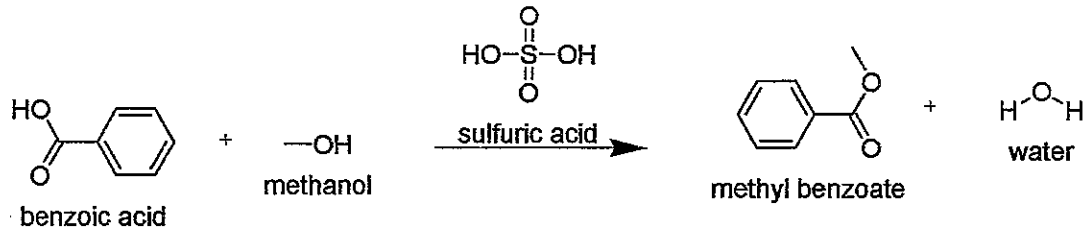
REFERENCE: Introduction to Organic Laboratory Techniques, 3rd Ed., Pavia, Lampman and Kriz
Vacuum Distilation, pp. 558-562, 590-592

OBJECTIVES: The student should be able to:

1. Write up a step by step procedure for the reaction using information on the handout sheet, laboratory manuals and The Handbook of Chemistry and Physics.
2. Extract the reaction product by use of the properties of polarity and acidity.
3. Recover the unreacted benzoic acid.
4. Distill the product by vacuum distillation.
5. Calculate the amounts of reactants given the grams of product and the expected percent yield.
6. Identify the final product from physical properties, IR and NMR spectra.

REACTION EQUATION:

main reaction equation



REAGENTS:

Reactants: Benzoic acid; methanol

Catalyst: conc. H_2SO_4, 4 ml

Calculate the amounts of the reactants required to produce 21.8 grams of methyl benzoate assuming a 80% yield.

Use this theoretical amount of benzoic acid.

Use a 518% excess of methanol.

When calculating amounts of reactants, round moles to 2 decimal places, final masses to 1 decimal place and final volumes to whole numbers.

PRODUCT ISOLATED: Methyl benzoate[1]

REACTANT RECOVERED: Benzoic acid[2]

APPARATUS: 250 ml standard taper round bottom flask and narrow condenser
500 ml separatory funnel

REACTION: Sulfuric acid should be added slowly with swirling to a well dispersed mixture of benzoic acid in methanol.

REACTION TIME: Approximately 1 hour at reflux temperature.

PURIFICATION:

Use is made of the solubility differences of the compound in water and in ether. The reaction mixture is added to a couple of hundred ml of water in a separatory funnel and the whole extracted with approximately 50 ml of ether, retaining the ether layer. The ether may first be used to rinse the reaction flask. Further separation of the organic components is accomplished by extraction of the ether layer with an aqueous solution of base[3] (10% sodium carbonate, approx. 50 ml), retaining both layers.
IN HOOD → LEAVE OPEN, SHAKE, VENT

CAUTION: Pressure of CO_2, which develops must be released often; shake very gently.

Proceed with the purification of both the ether and aqueous layers.

Wash the ether with a saturated sodium chloride solution. The ether solution now contains only low boiling solvents, some water and the ester. Add anhydrous magnesium sulfate to the ether solution until dry and gravity filter the ether through fluted filter paper to remove any water present. The ether is then removed by vacuum evaporation. The residue is transferred to a distilling flask, and the final product is distilled by a low pressure distillation.

The aqueous layer is acidified, causing the precipitation of solid benzoic acid. Use concentrated hydrochloric acid, adding slowly with swirling, until no more solid seems to form. Check for acidity with litmus paper.

CAUTION: Much frothing occurs and the reaction vessel should be a large beaker.

Collect the benzoic acid by vacuum filtration. Dry and weigh the recovered benzoic acid. When a reactant is recovered, the amount recovered is subtracted from the initial amount of benzoic acid used and the theoretical yield of the product, methyl benzoate, is recalculated.

Turn in both ester and acid, with percent yield and percent recovery calculated, respectively. For the product, methyl benzoate, all physical constants should be reported and an IR spectrum and a NMR spectrum should be run.

NOTES

1. Liquid substances are obtained by distillation as a final step. The material is collected over a boiling range of at least five degrees. With such a high boiling substance the boiling range may start as much as ten degrees below the reported boiling point.
2. Solid substances are separated from a solvent in which they are only slightly soluble by suction filtration and dried before the weight and melting point are determined. Water is more difficult to remove from solids filtered from it and the best procedure is to spread it on a watch glass to dry, or leave it in the Buchner funnel, until the next period. However benzoic acid sublimes easily and should not be left completely uncovered for a long time.
3. A check for completeness of extraction should be made here; be sure the aqueous layer after extraction is still basic.

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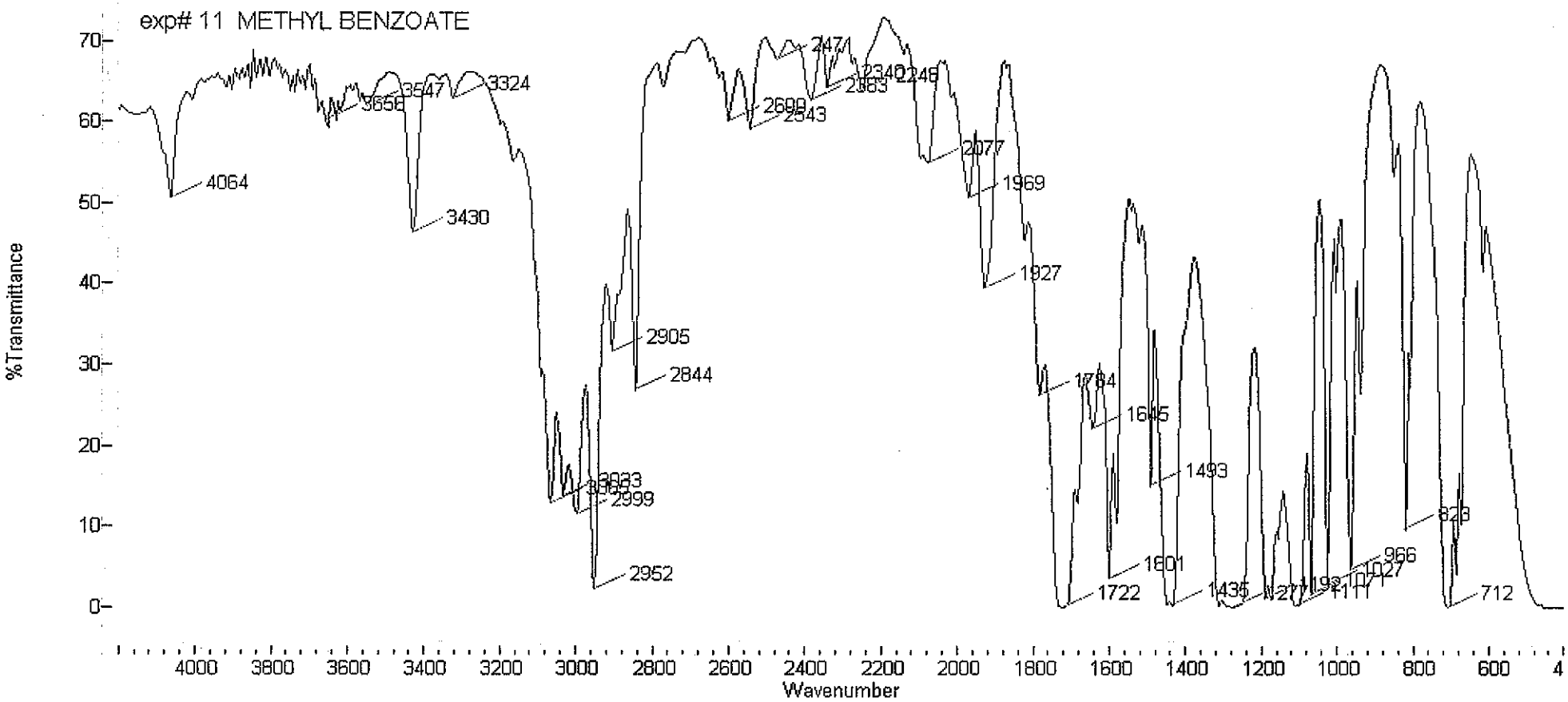
60



	Name	Spectrum	Hitlist
4	exp# 11 METHYL BENZOATE		Hits = 0
5	afatbeexp11MeBen		Hits = 30

14

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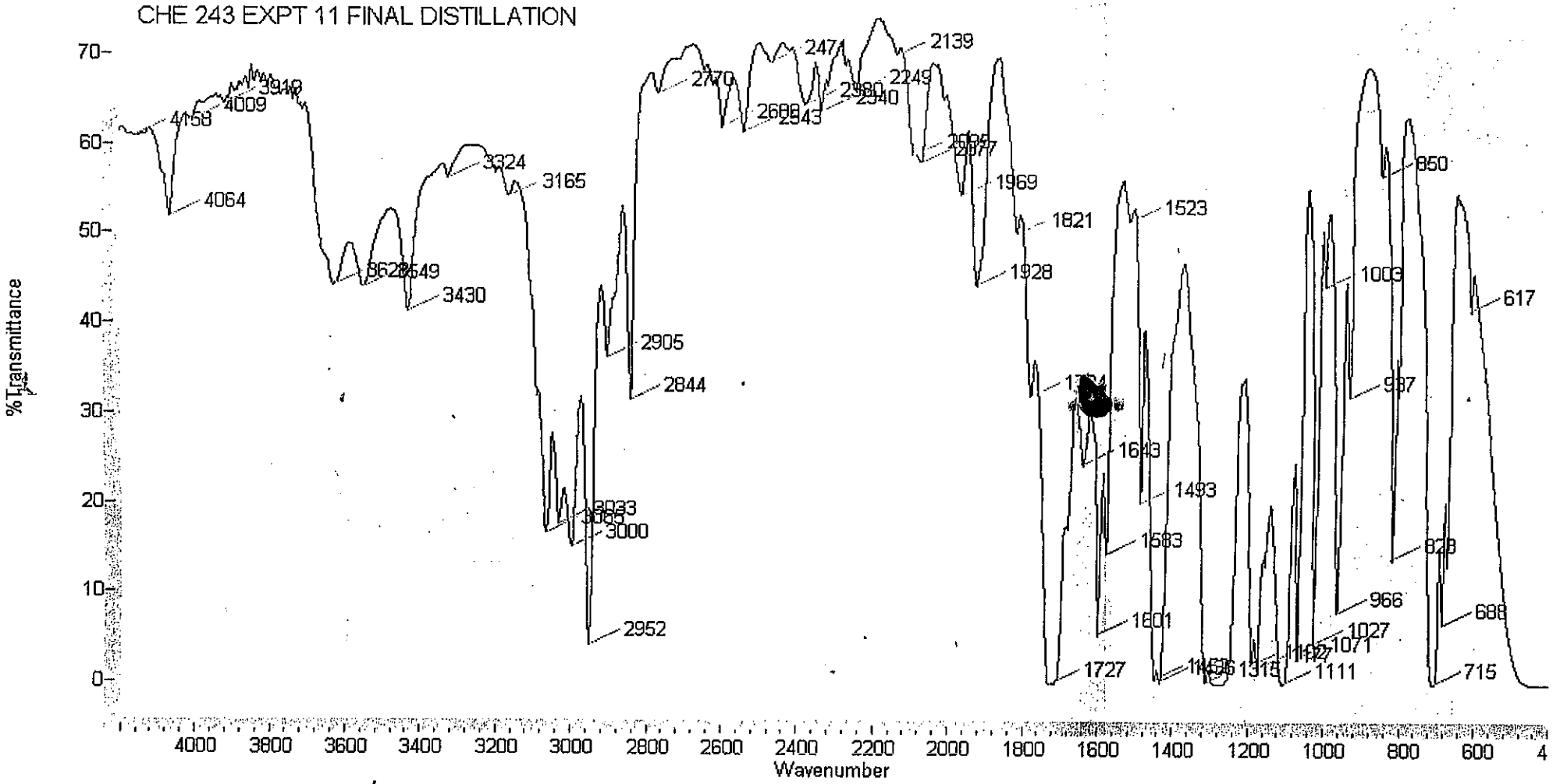
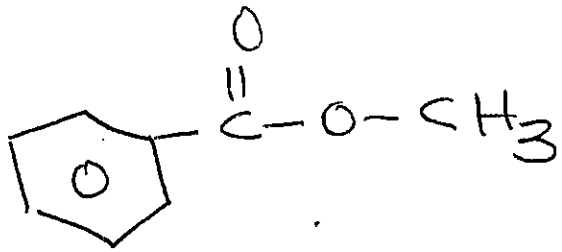
CHE 243 EXPT 11 FINAL DISTILLATION



3/18/08
TPS

THIN FILM ON
NaCl PLATES

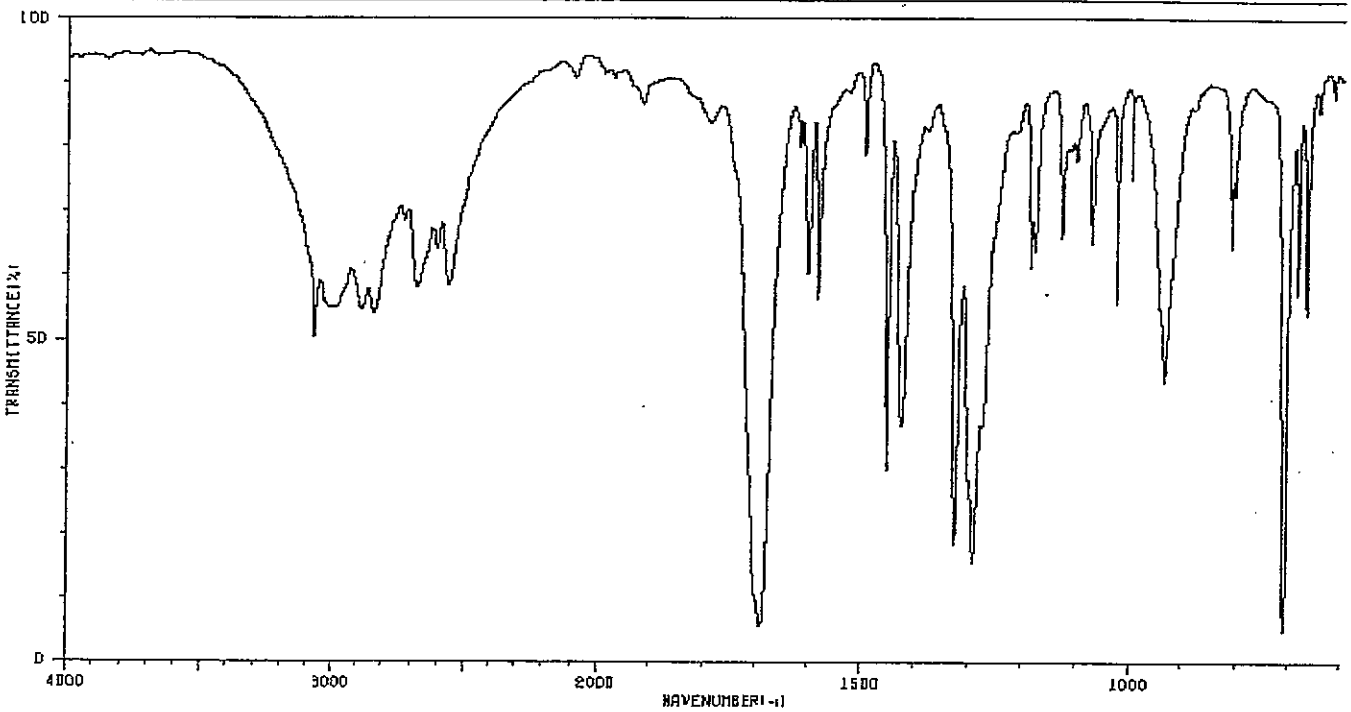


43

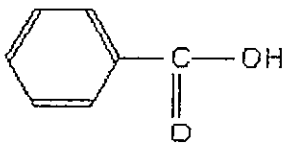
HIT-NO=1081	SCORE= ( )	SDBS-NO=673	IR-NIDA-63340 : KBR DISC

BENZOIC ACID

$C_7H_6O_2$



3073	49	2678	67	1426	35	1112	77	936	42
3012	53	2607	62	1327	17	1107	77	812	62
2998	53	2564	57	1294	14	1102	74	805	70
2986	63	1689	6	1187	58	1074	62	708	4
2886	52	1603	58	1180	60	1028	53	685	55
2838	52	1585	53	1129	84	1001	72	667	52
2726	66	1464	28	1118	77	943	60	664	58

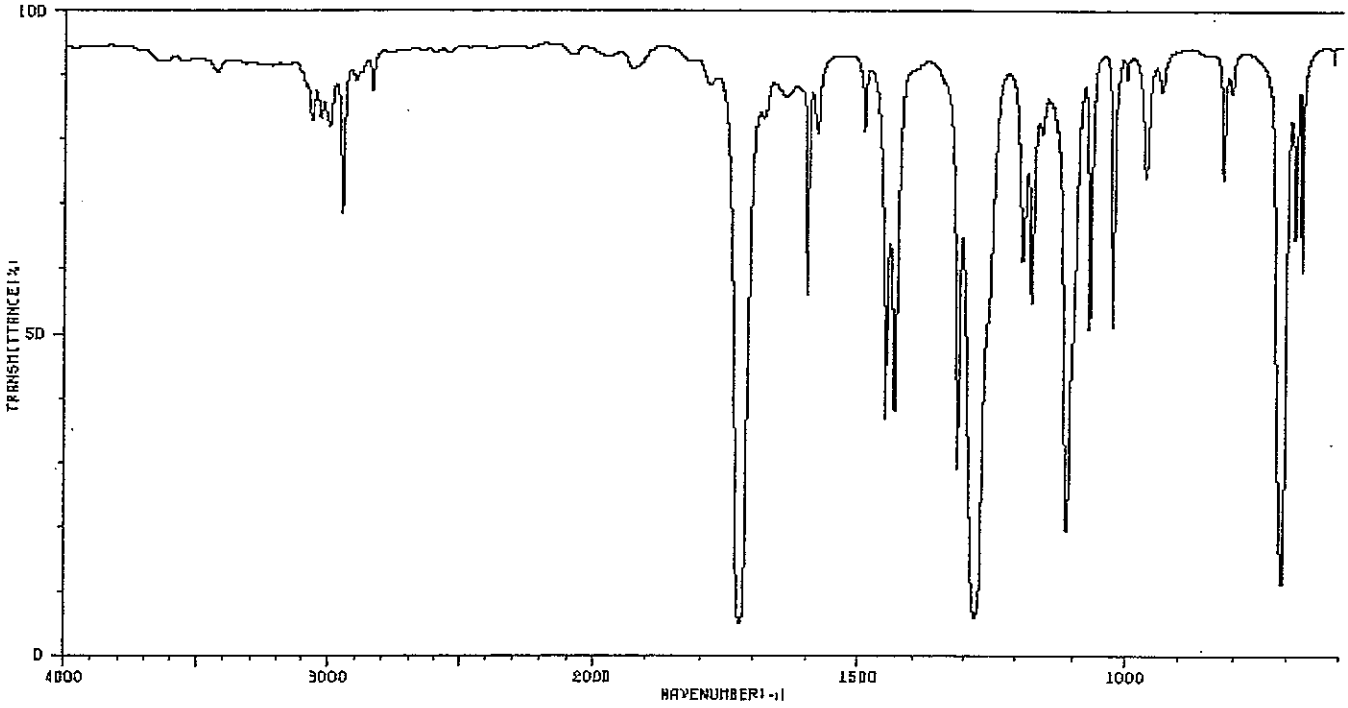


44

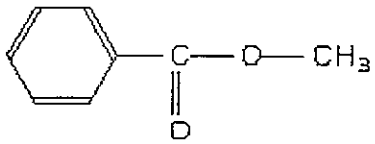
HIT-NO=1155	SCORE= ( )	SDBS-NO=725	IR-NIDA-00428 : LIQUID FILM

METHYL BENZOATE

$C_8H_8O_2$



3066	79	1724	4	1316	27	1029	49	688	62
3034	79	1644	84	1279	5	1003	86	676	58
2999	79	1602	55	1193	58	966	72	483	86
2953	66	1582	79	1177	62	937	84		
2907	86	1493	79	1160	77	823	70		
2845	84	1453	35	1112	18	808	84		
1784	84	1436	36	1072	49	710	10		



45

EXPERIMENT XII

THE GRIGNARD REACTION - The Synthesis of 5-Methyl-5-Nonanol

REFERENCES: Morrison and Boyd, 5th Ed., pp. 883, 653-659, 97-99. A discussion of the chemistry of the Grignard reaction.
Adams, Johnson and Wilcox, pp. 191-195, 388
Fieser and Williamson, Organic Experiments, 7th Ed., pp. 319-328
Pavia, Lampman and Kriz, 3rd Ed., pp. 226-227. A discussion of the precautions that must be observed when running a Grignard reaction.
Organic Synthesis, Col. Vol. II, p. 179

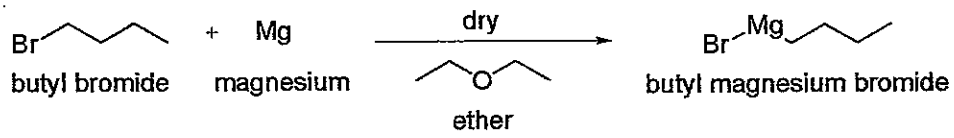
OBJECTIVES: The student should be able to:

1. Set up the apparatus for the reaction
2. Dry a reaction flask for use in the Grignard reaction
3. Prepare a Grignard reagent
4. React the Grignard reagent with an ester
5. Extract the product from the reaction with ether
6. Distill the product by vacuum distillation
7. Calculate the amounts of reactants needed given the appropriate information
8. Identify the product by physical constants, IR and NMR spectra

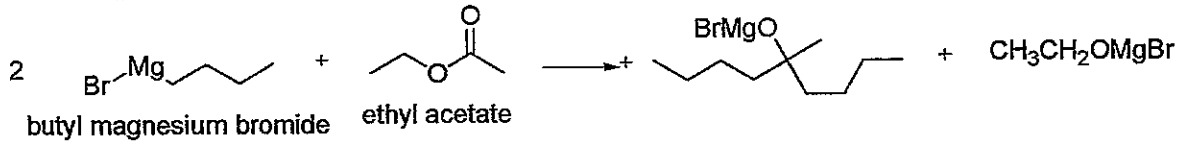
REACTION EQUATIONS:

46

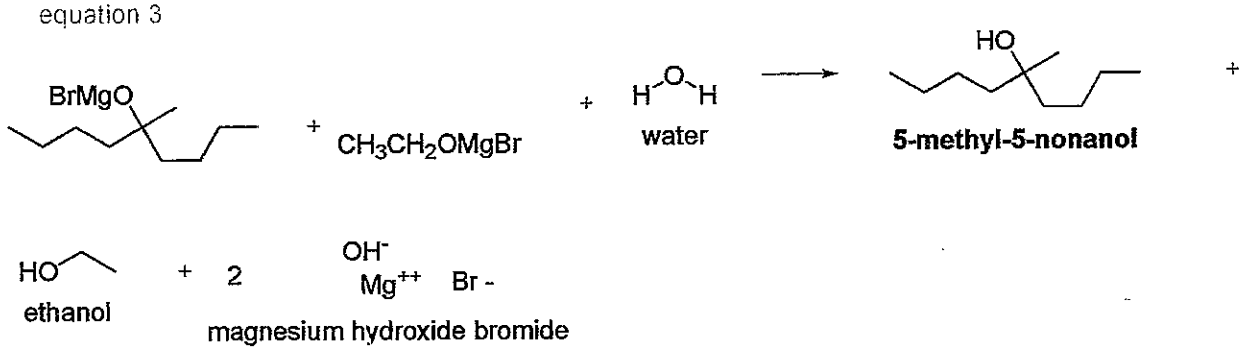
equation 1



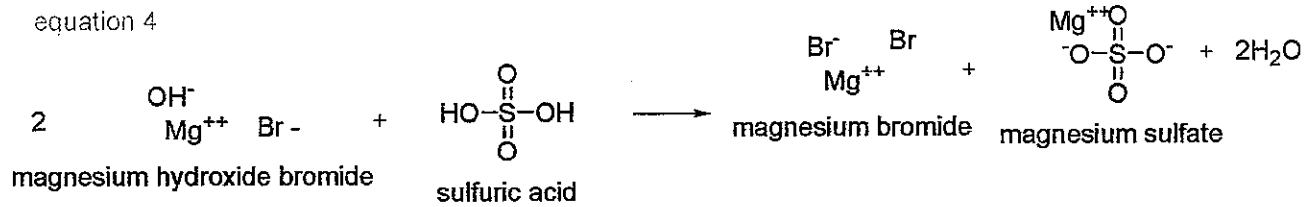
equation 2



equation 3



equation 4



Some of the side products arise from reaction of the Grignard reagent with water, oxygen, carbon dioxide; others are products of the Wurtz reaction of n-butyl bromide with magnesium, and the dehydration of the final product.

REAGENTS:

Reactants: 1. Magnesium, 1-bromobutane, ethyl acetate
2. Conc. H_2SO_4

Solvents: 1. ether (3 additions)
2. water

Calculate and use the amount of these reactants required to produce 14 g of 5-methyl-5-nonanol assuming an 80% yield.

When calculating amounts of reactants, round mole values to 2 decimal places, final masses to 1 decimal place and volumes to whole numbers.

PHYSICAL CONSTANTS:

5-Methyl-5-Nonanol

Boiling point - 195 °C at 760 Torrs

n_D^{20} - 1.4346

Solubilities:

H_2O - insoluble
ether - soluble

5-Methyl-4-nonene

Approximate Boiling point - 160 °C at 760 Torrs

APPARATUS:

A 500 ml 3-necked round-bottom flask is equipped with a stirrer with a rubber seal, a reflux condenser with a drying tube, and a 125 ml dropping funnel. All glassware must be clean and acetone dried. This must be assembled and checked out the period before running the Grignard reaction.
(see instructor diagram)

DIRECTIONS:

1. In the hood the magnesium is placed in the flask and the flask gently heated with a soft flame (a flame that shows no inner cone, yet is not smoky). This will evaporate absorbed moisture from the surface of the magnesium and the glass. When the flask has cooled, 85 ml of anhydrous ether

is added to the magnesium through the dropping funnel. The n-butyl bromide is dissolved in 40 ml of anhydrous ether and put into the dropping funnel. About 5 ml of the halide solution is allowed to flow into the flask from the funnel, and a starter solution added. The reaction generally begins within a few minutes as evidenced by a cloudy appearance, and the start of gentle refluxing. Once the reaction begins the stirrer is started and the remainder of the halide solution is added at such a rate that gentle refluxing takes place. A pan of ice may be necessary to moderate the reaction.

After the n-butyl bromide solution has been added (it should take 30 - 40 minutes) the cooling bath is removed and stirring is continued 15 minutes longer, after which only a small residue of magnesium remains. The acetate ester dissolved in 20 ml of anhydrous ether is added through the dropping funnel at such a rate that the ether refluxes gently. Once refluxing begins, this may or may not require cooling in ice. The addition of the ester/ether solution should take 30 minutes. The cooling bath is removed and stirring continued for 10 minutes.

2. The flask is then cooled in a ice bath and, with rapid stirring, 17 ml of H_2O is added through the dropping funnel at such a rate that rapid refluxing occurs. Following this, 7 ml of concentrated sulfuric acid in 67 ml of water is added. After the acid is added the two layers become practically clear. The liquid layers are transferred to a separatory funnel and the layers separated. The ether layer is washed once with a 10% sodium hydroxide solution, once with water, and once with a saturated sodium chloride solution. The ether is then dried over anhydrous magnesium sulfate, filtered through fluted filter paper and the ether removed by vacuum evaporation. The residue is transferred to a distilling apparatus with a distilling cow and vacuum distilled. The product should be turned in, the physical constants reported (refractive index) and an IR spectrum of the product run.

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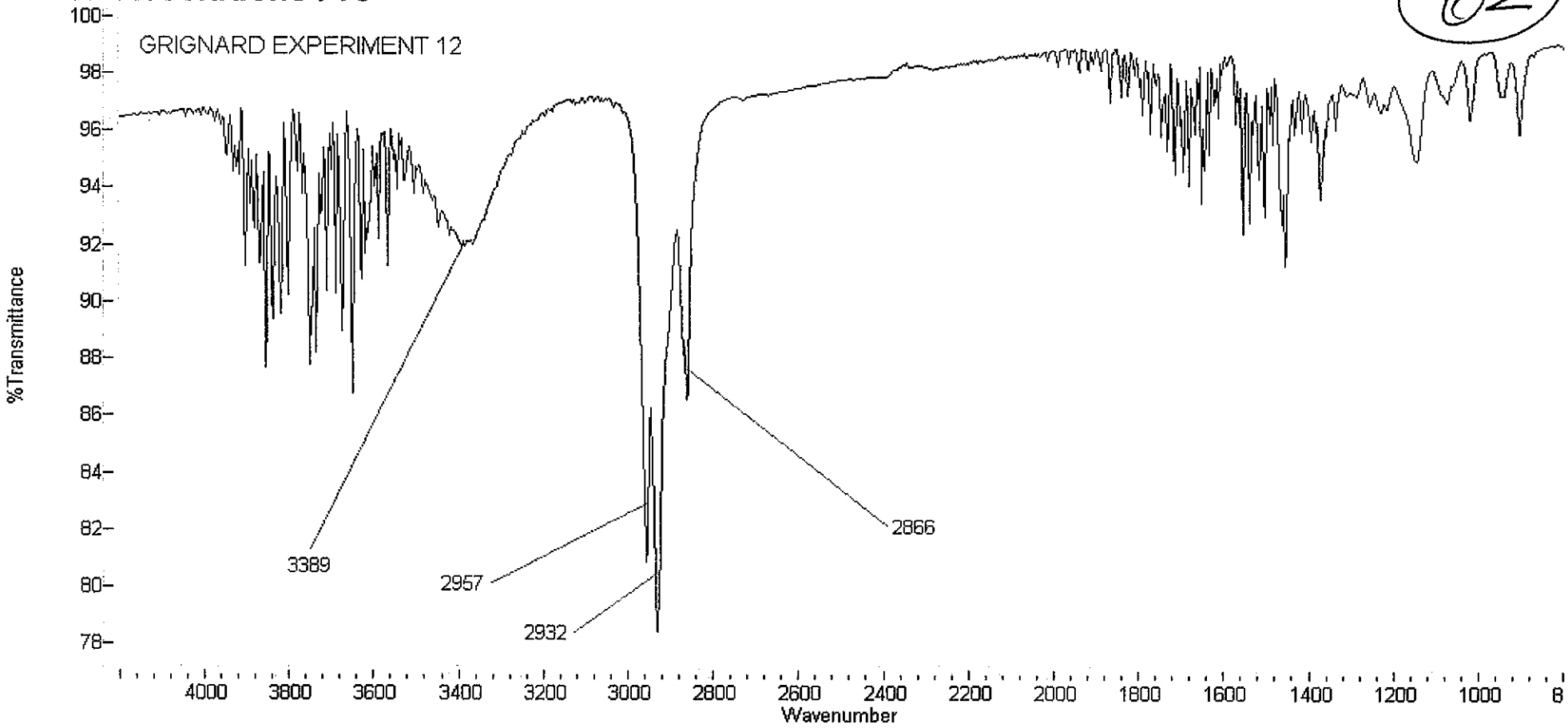
(102)



	Name	Spectrum	Hitlist
12	GRIGNARD EXPERIMENT 12		Hits = 0
13	experiment 12(7)		Hits = 0

(44)

Exp 12 Side Products 50

Butane
Butanol
Pentanoic acid
octane
5 methyl 4 nonene ≈ 160°C

Write rxn equations for these products!

51

Intellectual Property Website and Reference Websites

Intellectual Property Website
http://www.ciese.org/pathways/rwlo/rwlos/2500/CHEMISTRY%20INTELLECTUAL%20PROPERTY/overview.html

Websites

Abstracts
http://owl.english.purdue.edu/handouts/pw/p_abstract.html
http://faculty.concord.edu/boggess/abstract.html
http://mason.gmu.edu/~gandryko/690abstractadv.html
://www.asu.edu/duas/wcenter/abstract.html
://leo.stcloudstate.edu/hizwrite,abstract.html

Course
www.chemistry-solutions.com

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www.inchem.org
www.chemfinder.com
www.jtbaker
www.msds.com
http://phychem.ox.ac.uk/msds/
www.merck.com

52A

Websites

Abstracts

http://owl.english.purdue.edu/handouts/pw/p_abstract.html

http://faculty.concord.edu/boggess/abstract.html

http://mason.gmu.edu/~gandryko/690abstractadv.html

://www.asu.edu/duas/wcenter/abstract.html

://leo.stcloudstate.edu/hizwrite,abstract.html

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www.inchem.org

www.chemfinder.com

www.jtbaker

www.msds.com

http://phychem.ox.ac.uk/msds/

www.proitech.com

www.scorecard.org/chemicalprofiles

www.skylighter.com

www.merck.com

www.cdc.gov

www.state.nj.us/health/eoh

Sitting Marshall Handbook of Toxic Chemicals, 4th ed. Vol 2

http://ptel.chem.ox.ac.uk/msds/

Websites

Abstracts

http://www.wikihow.com/Write-a-Scientific-Abstract

http://owl.english.purdue.edu/

http://writingcenter.unc.edu/handouts/abstracts/

http://www.ncbi.nlm.nih.gov/pmc/articles/PMC3136027/

http://www.scribd.com/doc/9668146/5-Steps-To-Write-the-Abstract-of-Scientific-Writing#scribd

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www.jtbaker

www.msds.com

http://phychem.ox.ac.uk/msds/

www.merck.com

www.cdc.gov

www.state.nj.us/health/eoh

Sitting Marshall Handbook of Toxic Chemicals, 4th ed. Vol 2

Appendix A
Physical Constant Sheet
Reference information on physical constants

54

PHYSICAL CONSTANT SHEET - INFORMATION AND EXPLANATIONS

CHP #:

The CHP number from the student's CHP appendix must be entered for all compounds.

COMPOUND:

All reagents used in the main reaction, all products from the main reaction, all side products from any side reactions, and all compounds used in purification and characterization are entered. If a compound is used in purification as a water solution it should be so designated.

M.W. g/mole:

The molecular weight is needed for all reagents for which it is necessary to convert grams used to moles used or vice versa. It is also needed for the main product in order to calculate the theoretical yield.

AMOUNT USED, gm./ml, MOLES:

A. For all reactants used the moles specified in the experiment, or calculated, are entered in the MOLES column.
B. For a solid reactant the grams to be weighed, to 1 decimal place, is entered in the gr./ column.
C. For a liquid reactant the mls to be measured, in whole mls, is entered in the /ml column..
D. For all solvents (and catalysts) used in the reaction the volumes (or grams) specified in the experiment are entered.
E. Amounts of compounds or solutions used in purification need not be entered.

MOLE RATIO, Theory & Used

A. The MOLE RATIO Theory for each reactant and the main product is determined from the balanced reaction equation.
B. The MOLE RATIO Used is calculated for each reactant from the AMOUNTS USED/MOLES: each mole value is divided by the smallest mole value and the ratio entered.

55

C. Example: for the reaction: $Mg + 2\ HCl \longrightarrow MgCl_2 + H_2$

the MOLE RATIO Theory is: Mg = 1; HCl = 2; $MgCl_2$ = 1

if 0.5 moles Mg and 1.2 moles HCl are actually used,

the MOLE RATIO Used is Mg = .5/.5 = 1; HCl = 1.2/.5 = 2.4

the appropriate column entries would be:

	AMT. USED MOLES	MOLE RATIO Theory	Used
Mg	0.5	1	1
HCl	1.2	2	2.4
$MgCl_2$		1	

M.P. and B.P.:

The melting point and boiling point are entered for:

A. all reagents used in the main reaction so the student may determine whether the compound is a solid or a liquid.
B. the main product.
C. side products and secondary products if their solubilities are similar to the solubilities of the main product.
D. purification solvents in which the main product is soluble.

The melting point and boiling point are not generally needed for:

A. any purification compound used as a water solution.
B. any drying agent.

DENSITY, g/ml:

A. DENSITIES OF COMPOUNDS - the density of a liquid is needed when it is a reagent and the mls used must be calculated from the grams used, or when the liquid will be worked with in a separatory funnel. The densities of most liquids are found in handbook citations as d_4: the density at (^{o}C) relative to the density of water at $4^{o}C$. Since this is usually about room temperature this density may be used as is in the DENSITY g/ml column.

B. DENSITIES OF SOLUTIONS - When a water solution will be used in a separatory funnel, it is necessary to have the solution density in the PCS, not the density of the solid. However these are seldom found directly in most handbook citations.

For saturated solutions, the Merck Index, Miscellaneous Tables Section has a Table of Saturated Solutions; it gives the concentrations in g/100 ml (%w/v - see Solubilities below); g/100 g (%w/w - see Solubilities below) and specific gravity (density) of most saturated solutions.

For a less than saturated solution, the density may be approximated using the above table. As an example, find the density of a 10% w/w solution of sodium hydroxide. The density of a saturated (50.8% w/w) solution is 1.51 g/ml. The difference between this density and the density of water, 1.00 g/ml, is .51 g/ml and is due to the 50.8 gms of NaOH. A 10% solution is 10/50.8 or approximately 1/5 the concentration in the saturated solution. So an increase in density for a 10% solution would be approximately 1/5 times .51 which equals .1 g/ml [(1/5)x .51 = .1]. Therefor the density of the 10% solution is approximately 1.1 g/ml.

Water solutions provided for use in the lab will be % w/w.

Most water solutions of 10% or less concentration may be taken to have a density of 1.

SOLUBILITY:

A knowledge of the solubilities of the compounds used is necessary in the purification of all products. Generally for a liquid product the solubilities of a compound in a polar solvent (water) and a non-polar solvent (ether) is needed for ALL compounds in the experiment. Sometimes the solubility in another solvent is needed, i.e. ethanol if a solid product is to be recrystallized from ethanol.

A. Definition of Solubility: the limit of solubility will be taken as 3 grams solute/100 ml solvent: a substance is soluble in a solvent if 3 grams or more dissolves in 100 ml of the solvent. However, few references give solubility as g/100 ml solvent so the following guide lines are to be used.

1. The Handbook of Chemistry and Physics (63rd Edition and earlier) uses a solubility classification which may be used directly in the PCS:

   i - insoluble s - soluble
   $\mathcal{S}$ - slightly soluble v - very soluble
   (less than 3g/100ml but not insol)
   m - miscible - refers to two liquids which are mutually soluble in all proportions.

   Temperature effect on solubility is noted by:

   i^c - insoluble in cold solvent
   v^h - very soluble in hot solvent

   If no temperature is given, it is assumed to be room temperature.

2. The Merck Index and other references give solubility information in other ways. Unless otherwise noted in the reference, solubility data is assumed to be at room temperature.

   a. Soluble in (solvent); used in the PCS as: s.
      Freely soluble in (solvent); used in the PCS as: v.
      Slightly soluble in (solvent); used in the PCS as: $\mathcal{S}$
      Insoluble in (solvent); used in the PCS as: i.

   b. Solubility as (for example) 10g/160ml H_2O

      This must be converted to g/100ml, i.e. 10g/160ml x 100 ml = 6.25 g/100ml and used in the PCS as 6.25 g/100ml, s.

      Thus a solubility given as 3.75g/150ml converts to 2.5g/100ml which by our definition is slightly soluble and is used in the PCS as $\mathcal{S}$.

   c. soluble in x parts - this means that 1 part solute is soluble in x parts solvent, both solute and solvent being in either grams or mls. This is usually used when a solute is very soluble in the solvent: soluble in 5 parts water , 1/5 --> 20/100.

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d. x %, in (solvent)

1. w/w - x grams are soluble in 100 grams of solution for the solvent cited.

2. w/v - x grams are soluble in 100 mls of solution for the solvent cited.

3. v/v - x mls are soluble in 100 mls of solution for the solvent cited.

These do not give the solubility of the solute in terms of solvent, but in terms of solution, and are not easily converted. However this method is usually used only for fairly soluble solutes and as an approximation, any percent citation of 3% or greater may be taken as soluble.

3. If no solubility data is given for a particular solvent - the solute is assumed to be insoluble in that solvent. This is particularly true for inorganic, ionic compounds in non-polar solvents; they are insoluble.

REFRACTIVE INDEX, n_d :

For liquids, the refractive index is entered for the main product and any reagents or side products with solubilities and boiling points similar to the main product.

LIMITING REAGENT, and THEORETICAL YIELDS

A. Limiting Reagent: Using the example from Section V-D above, by comparing the MOLE RATIOS Theory and Used it is found that the HCl is in excess, i.e. 2 moles are needed, 2.4 moles are used. Therefore Mg is the limiting reagent.

	AMT. USED MOLES	MOLE RATIO Theory	Used
Mg	0.5	1	1
HCl	1.2	2	2.4
$MgCl_2$		1	

B. Theoretical Yield, moles: is found by comparison of the Mole Ratio Theory and Amt. Used Moles for the limiting reagent and the main product. In the above example the Mole Ratio Theory is 1:1, therefor for every 0.5 moles of limiting reagent used, 0.5 moles of main product should be formed, and 0.5 is the theoretical yield in moles.

C. Theoretical Yield, grams: is calculated by multiplying the theoretical yield in moles times the molecular weight of the main product.

EXPERIMENT _____

TABLE OF PHYSICAL CONSTANTS

CHP #	COMPOUND	M.W g/mol	AMT. USED		MOLE RATIO		M.P. ° C	B.P. ° C	DENSITY g/mL	SOLUBILITY			N_D^T
			g/ml	Moles	Theory	Used				H2O	ETHER	ALC	

a) Limiting Reagent_____ b) Theoretical Yeild (mol) _____ c) Theoretical Yield (g) _____

Appendix B
Refractive Index Calculations
Electro thermal Melting Point Apparatus Instructions
Thermal Scientific MEL-TEMP 3.0 Instructions
I.R. Varian 2000 FT_IR Instructions
G.C. Varian 3900 GC Instructions
Distillation Calculations
Pressure/Temp Conversion Scale

Appendix B:

Refractive Index Calculations
Electro thermal Melting Point Apparatus Instructions
Thermal Scientific MEL-TEMP 3.0 Instructions
G.C. Agilent 7820A GC
I.R. Agilent Cary FTIR Instructions

REFRACTIVE INDEX N_D^{20}

Refractive indices must be compared at the same temperature

1. Measure your experimental value (N_D^T)exp, record the temperature.
2. If it is at a different temperature than reference, then you must apply correction factor.

EXAMPLE:

(N_D^{20})REF: = 1.5932 @ 20 degreees C
(N_D^T)exp = 1.5918 @ 23 degrees C

Add .00045 for every degree exp Temperature is above reference Temperature.

Subtract .00045 for every degree exp Temperature is below reference Temperature.

Corrected (N_D^T)exp = 1.5918 + (23 °C – 20 °C)(0.00045/°C)

Corrected (N_D^T)exp = 1.5918 + 0.0013 = 1.5931

PURITY STANDARDS:

IF Corrected (N_D^T)exp is +/- 0.001 of (N_D)REF then "HIGH PURITY"

IF Corrected (N_D^T)exp is +/- 0.002 of (N_D)REF then "SATISFACTORY PURITY"

IF Corrected (N_D^T)exp is > +/- 0.002 of (N_D)REF then "TECHNICAL GRADE PURITY"

TPS REV 12APRIL2006

ELECTROTHERMAL MELTING POINT APPARATUS INSTRUCTIONS

Turn Power switch on at back left side of power supply.

Preheat apparatus for 30 mins prior to use.

For even heating, 3 capillary tubes must be placed in instrument, even when only one contains a sample.

1. Enter a Set Point (2 to 5 degrees below the expected Melting point): Press the Up key { ▲ } to arrive at the closest value.
EX: To get to a set point of 107°C press the { ▲ } 11 times to 110° C, then press the { ▼ } 3 times to get to 107°C

Note- The Up key { ▲ } moves up in 10 degree increments.
The Down key { ▼ } moves down in 1 degree increments.

Press {Clear} (lower right of key pad) of a mistake is made.

2. Press GO TO key { ⤴ } to go to the Set Point.
When the Set Point is reached the apparatus will beep 3 times.

3. Press GO TO { ⤴ } again to initiate the temp. ramp of 1°C per minute. Watch for the melting point.

4. When the first signs of liquid formation occur, press the GO TO key { ⤴ }, this stores this temperature in memory and continues heating. When a completely clear liquid forms, (total melt), press { ⤴ } again.

5. Press {Clear} once.

To review stored points, press { ⤴ }. The display will show the temp of each successive stage as it was entered. The first depression of the { ⤴ } key gives the first temp, the second depression of the { ⤴ } key gives the second temp.

**Temps must be recorded in the lab notebook at this point. (After the {Clear} key is pressed all temps are erased).

6. After temps have been reviewed, press {Clear} key once. The current set point will be displayed. If this is the desired set point, press the GO TO { ⤴ } key. If a new set point is needed, press the { ▲ }/{ ▼ } keys as previously explained.

**Do not insert new samples until temp has stabilized at new set point.

After insertion, allow tubes and samples to stabilize for approximately 2 minutes before continuing.

When all trials are complete press {Clear} key 4 times.

If no one else will be using the Melting point apparatus please turn Power off using the switch on the rear of the power supply.

Dispose of the dirty capillary tubes in receptacles provided. Return clean, spare tubes to capillary tube dispensers.

Thermo Scientific MEL-TEMP 3.0

Operation

• Switch the power ON.
• The upper display, marked ACTUAL, indicates the temperature of the capillary chamber.
• The lower display reads the previously selected "start temperature".
• The START °C LED (Light Emitting Diode) is lit to identify the meaning and units of data displayed in the lower display.
• The capillary chamber will heat or cool to the "start temperature".
• Whenever the heater or fan is on, the corresponding LED on the control panel will light.

Performing a Melting Point Determination

Set Test Parameters

1. Press [SET]. Use the UP and DOWN ARROW to select Start (start temperature).

2. Repeat for End (end temperature) and rAtE (heating rate).

3. Press [SET] to exit the set mode.

Perform Test

1. Insert charged capillaries when the "Insert Capillaries" indicator flashes and 3 audible beeps sound.
• The capillary openings are visible directly below the 1/2 inch round hole in the top of the instrument. They are also visible by looking through the viewing glass about an inch behind, and just above, the inner lens.
• Note that it is not necessary to wait for

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the flashing "Insert Capillaries" light before inserting charged capillaries. The light and audible beeps assure you that the oven temperature has not changed more than 0.5°C for at least two minutes.

• You can start your test any time before or after you hear the three beeps or notice the flashing "Insert Capillaries" light.

2. Press [RAMP/COOL] to begin ramping. You can change the ramp rate any time while you are ramping by pressing the UP or DOWN ARROW key.

3. Press [STO/RCL] to store the onset of the melting range in MEM1. Two audible beeps will sound and "Store" will briefly be displayed, ensuring that the data has been stored.

4. Press [STO/RCL] to store end of melting range in MEM2. Two audible beeps will sound and "Store" will briefly be displayed, ensuring that the data has been stored.

5. Press [RAMP/COOL] to cool oven back to Start. the fan will run until the start temperature is reached.

6. The "Insert Capillaries" indicator will flash and 3 audible alarms will sound when the unit is ready for the next test.

Recall Results

You can recall previously stored temperatures any time that the instrument is not ramping.

1. Press [STO/RCL] to recall MEM1. The "MEM1" light will illuminate. Repeat for MEM2.

2. Press [STO/RCL] or wait 60 seconds to exit the recall mode.

Operating Procedures - Aligent 7820A GC System

Angela Foy, 2016

Procedure 1: Starting the GC

1. Turn gas on all the way
2. Turn on machine/Turn on computer
3. Username: BCC Password: BCC
4. Click desktop icon: Aligent Lab GC (online); allow software to load

Procedure 2: Loading a Simple Run

This will store all data in the previous user's folder and chromatograms will contain the sample name of the last sample run.

1. Start the GC and load the software according to Procedure 1
2. Go to bottom left, click on Method and Run Control (if a save window opens, do not save changes)
3. Go to drop-down box at top left of screen; select the method temp ramp 3.M
4. Wait for the **ready lights** (top left of computer monitor and display panel of the GC unit). Both must say 'ready' before injecting sample
5. Inject sample and push start button on the machine
6. Wait for sample to run; a chromatogram will open when complete
7. Print using default printer: HP LaserJet P3010 Series UPD PCL6 (Copy 2)

Procedure 3: Loading an Advanced Run

This allows the user to name the sample and store chromatogram data in a chosen folder.

1. Start the GC and load the software according to Procedure 1
2. Go to bottom left, Click on Method and Run Control (if a save window opens, do not save changes)
3. Go to drop-down box at top left of screen; select the method temp ramp 3.M
4. Go to top left, click on run control and select sample info --a window will open
5. Next to **Subdirectory**, select the folder to save your data
6. Next to **Sample Name**, enter the name of your sample (e.g. EXP7 FINAL PROD_JEN SMITH)
7. Next to **Vial/Location**, enter a number one (1)
8. Click Run Method (system status **ready** will change to **Run in Progress/Waiting for Injection**)
9. Inject sample and push start button on the machine

10. Wait for sample to run; a chromatogram will open when complete
11. Print using default printer: HP LaserJet P3010 Series UPD PCL6 (Copy 2)

Procedure 4: Labeling Peaks

1. Start the GC and load the software according to Procedure 1

2. Go to bottom left, click on Data Analysis
   (If a save window opens, do not save changes)

3. On top left, double click the instructor folder containing the chromatogram you would like to edit. Under this folder, double click Single Runs.
   (If 'no signals available' window opens, click Ok)

4. The top center of the screen should now display a table, listing chromatograms by date and time. Double click within this table to open a chromatogram.

5. Go to top, click Calibration and select New Calibration Table – a 'new calibration table' window will open, click Ok.
   (An 'overwrite existing calibration table?' window will open, click Yes)

6. Go to **Calibration Table** at bottom of screen – In the **compound column**, enter the compound name for each peak you wish to label. Click Ok when finished to save changes.
   (If 'delete lines with zero amounts?' window opens, click No)
   (If 'some compounds have no name' warning window opens, click Ok)

****Note:** Additional compound names cannot be added later without overwriting the existing calibration table.

7. The chromatogram on the screen should now be showing labeled peaks. Go to top of screen, click Report and select Print Report. A preview window will open.

8. Print using default printer: HP LaserJet P3010 Series UPD PCL6 (Copy 2)

Agilent Cary 630 FTIR

Turning On the Instrument To turn on the instrument: 1 Press the power button of the Cary 630 system and hold for two (2) seconds. Initially, the light will flash between red and green while the firmware is being loaded. This process should take less than 15 seconds. When the LED light illuminates solid green, the instrument is now ready to use. 2 The spectrometer must have a warmup period of 5 minutes before reliable analysis may begin. When finished using the instrument, press the green power button again to turn off the system. The LED light will turn red.

Power switch LED status The power button contains a two-colored LED. The color displayed on the LED can indicate the state of the system. The table below lists the colors of the LED and the status of the instrument.

Turning Off When you have finished using the spectrometer, press the green power button to turn off the system. The LED light will turn red.

The basic steps for analyzing a sample with all sampling accessories are:

1 Clean the sample interface
2 Collect a background spectrum.
3 Collect a sample spectrum.

NOTE To ensure accuracy of the measurement, it is recommended that the system is configured in the software to collect a background spectrum before every sample is analyzed. This provides a baseline profile of the system conditions with no sample loaded on the instrument. By collecting a background automatically before each sample measurement, negative effects of changes in the environment can be avoided.

In the sample loading and cleaning mode, the sample press should be in the highest position, so the sample press tip is well above the diamond sampling window (see Figure 5). It is in this position that the sample mounting area can be easily accessed for loading the sample into place and for cleaning the sampling surfaces before the next sample is analyzed.

In the sample analysis mode, the sample press is lowered such that the tip is in contact with a solid sample. In this position, contact is made between the sample and the infrared energy emitting from the diamond window.

NOTE If the sample is a liquid or paste, the sample press does not need to be used at all.

When in the sample analysis mode, the ATR sample pathlength is fixed based on the number of reflections in the ATR. The alignment of the accessory is also pre-set at the factory, and so there is no optical or mechanical adjustment required.

Cleaning To clean the Diamond ATR accessory in preparation for analysis: 1 Open the sample press by rotating the top knob counterclockwise until the top of the press travel is reached (see Figure 5). 2 Clean the sample press tip using Kimwipes (or a suitable substitute such as cotton swabs) and a suitable solvent, such as acetone, methanol, ethanol or isopropyl alcohol. 3 Clean the diamond sampling window.

Collecting a background spectrum for instructions on collecting a background using the software, refer to the Agilent MicroLab FTIR Software User's Guide.

CAUTION To ensure an accurate background spectrum is collected, perform a visual inspection of the diamond sampling window, looking for any haze or film present from measuring the previous sample. If a film is observed, repeat the cleaning procedure on Page 27 until the sampling window is clear of any residue.

To load a sample and collect a spectrum using the Diamond ATR accessory: 1 Turn the sample press knob in a counterclockwise direction to open the sample press so that the tip is elevated slightly from the diamond sampling window (see Figure 5). 2 Place a small amount of material to be measured on the diamond crystal. The crystal is the clear, circular shaped material held in place by the surrounding metal disk. 3 Ensure that the sample covers the entire surface area of the diamond crystal.

In the case of a volatile sample, larger amounts of sample can be applied without concern of leakage or damage to the instrument. However, using the smallest amount of sample possible will ease in the cleaning process.

CAUTION The Diamond ATR accessory windows are made from type IIa synthetic diamond, which is extremely chemically-resistant. However, it is still possible the diamond crystal or metal mounting plate can be damaged by extreme samples. Only measure samples with a pH between 1 and 14. Do not leave extremely acidic samples on the metal mounting plate for an extended time.

CAUTION Although diamond is a very hard material, the ATR window is relatively thin (0.5 mm or less) and can crack under extreme pressure conditions. Ensure that the sample makes contact with the entire surface area of the diamond and not just a point within the diamond. Avoid using the sample press on samples that may be sharp or pointed.

If the sample is a liquid or paste, the sample press does not need to be used at all. In this case, you are now ready to proceed with the analysis.

This Manual: http://www.manuallib.com/file/2630539

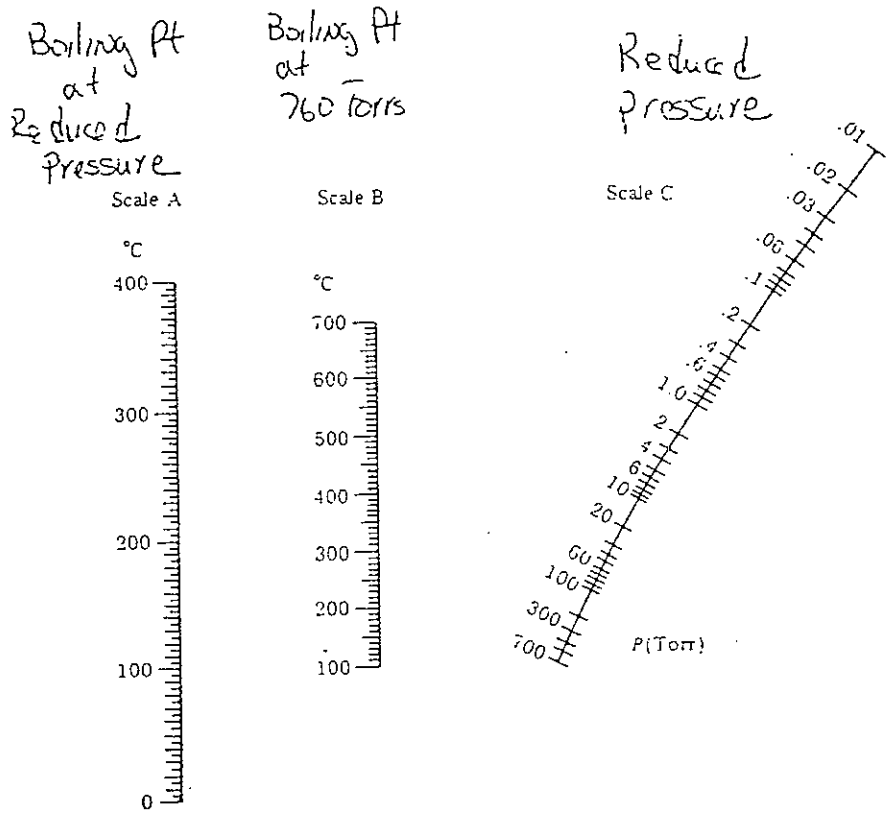
Analyzing Samples

Agilent Cary 630 FTIR Spectrometer User's Guide 29

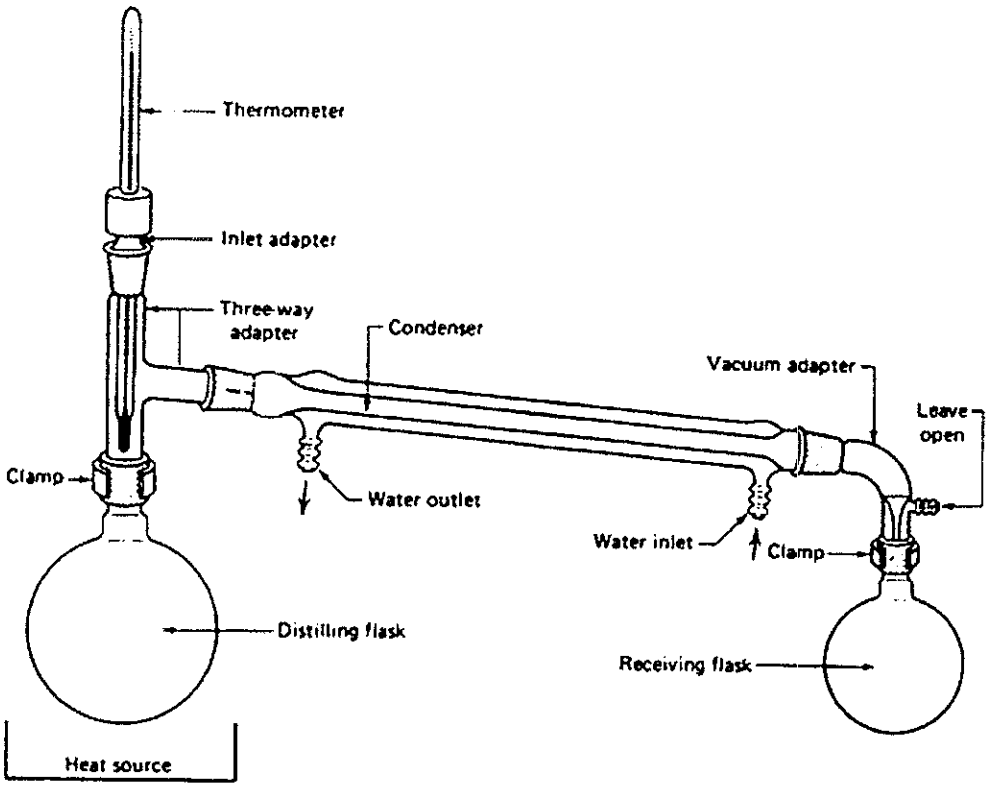
If the sample is a powder or solid, the sample press must be in contact with the sample. To make contact, rotate the sample press knob in a clockwise direction until it reaches the detent (click-in) position. The sample press arm uses a slip clutch mechanism, whereby the maximum applied pressure cannot be surpassed — once maximum pressure has been reached, the slip clutch mechanism will click and not allow any extra pressure to be exerted on the sample.

Agilent Manuel. Manual Part Number G8043-90001 Edition First edition, September 2011 Printed in USA Agilent Technologies,

Reduced Pressure Temperature Conversion Scale

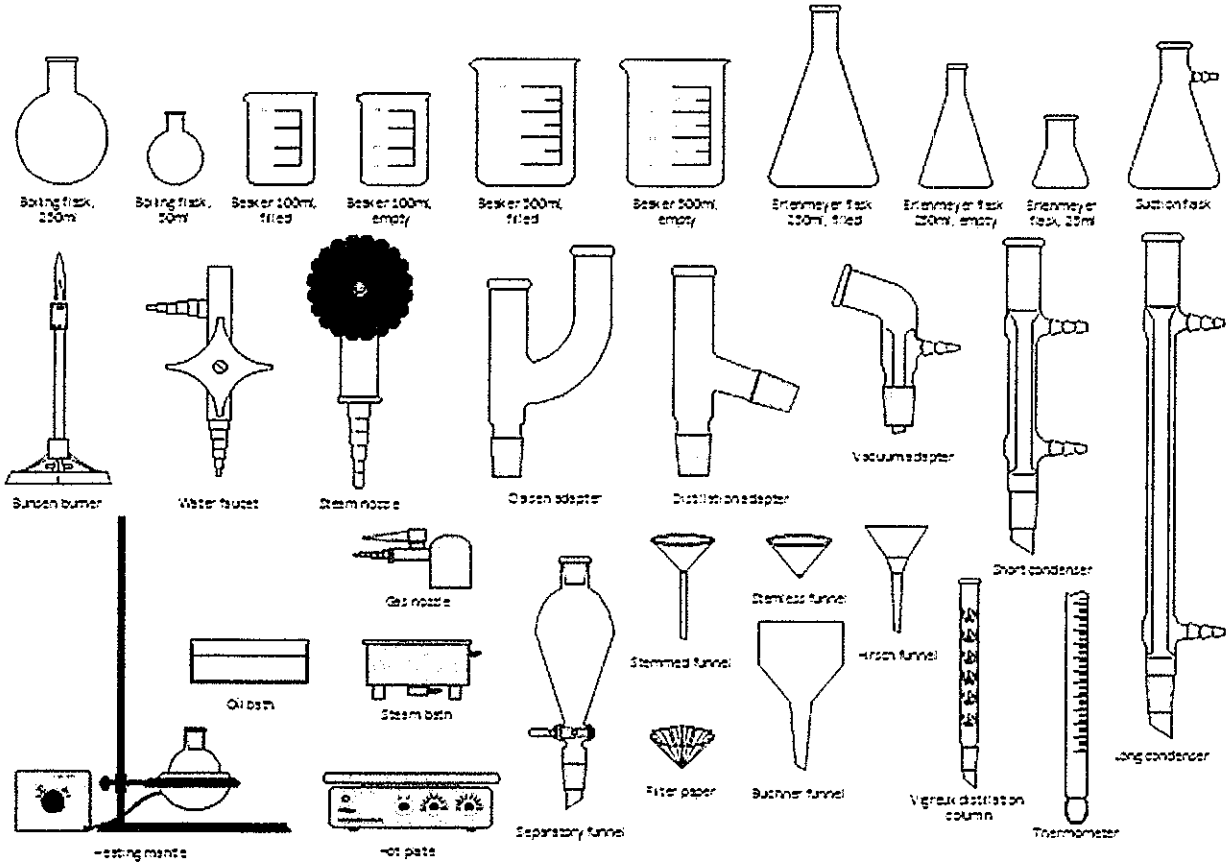


Simple Distillation Apparatus



Reference:

http://www.bing.com/images/search?q=Simple+Distillation+Apparatus+Set+Up&view=detailv2&&id=F596C74357B7E2368F45433D407D742101D88117&selectedIndex=7&ccid=D4%2fGM5r2&simid=608004418928642004&thid=OIP.M0f8fc6339af63f3bb10db08499105ab3o0&ajaxhist=0



Reference:

http://www.bing.com/images/search?q=Lab+Distillation+Equipment&view=detailv2&&id=2BEFBF3F354BF7D0FD8EED23E6A16C57E19DE636&selectedIndex=68&ccid=9P%2bV4I8n&simid=608032757122861673&thid=OIP.Mf4ff95e08f27743f8d0c48d5ffcedad6o0&ajaxhist=0

Appendix C
Infrared Spectroscopy Information

INFRARED SPECTROSCOPY

The Infrared spectrum is most usually a qualitative tool. It is used to: 1) identify a compound by comparison to a standard spectrum, and 2) to identify structural functional groups using a correlation chart.

Information from the experimental IR spectrum of a final product is included in the Laboratory Notebook in the Data Table and Analysis and in the Result Cards for each experiment. The IR spectrum is part of the experimental procedure and is taped in the Lab Notebook, properly labeled.

Using 1-butanol (IR spectrum on back) as an example, the entries should be in the following form:

DATA TABLE

A. Experimental spectrum matches standard for 1-butanol

Functional group ID:

Compound	Vibration	Wave number, cm-1	Inten.	Correlation Chart, cm-1
alkane	C-H stretch	2950, 2850	s	3,000 - 2850
alkane	CH3 bend	1380	m	1450 & 1375
alkane	CH2 bend	1460	m	1465
alcohol, free	O-H stretch	3600	m	3650 - 3600
alcohol, H-bonded	O-H stretch	3400	s(broad)	3400 - 3200
alcohol, 1o	C-O stretch	1060	s	1050

B. no peaks attributable to an impurity noted

ANALYSIS: (for example):

Since the experimental spectrum matches the standard spectrum for 1-butanol the product is identified as 1-butanol.

CH stretches at 2950, 2850 (3,000 - 2850), CH3 bend at 1380 (1450 & 1375), and CH2 bend at 1460 (1465) indicate an alkane. OH stretches at 3600 and 3400 (3650-3600, 3400-3200) and CO stretch at 1060 (1050 - 1o) indicate a 1o alcohol. This agrees with the known structure of 1-butanol.

LABELLING THE SPECTRUM:

The IR spectrum is to be labeled as: Exp. # product, phase, concentration, method, date, name. Example: Exp 1 product, liquid, neat, salt plates, date, name.

RUNNING AN IR SPECTRUM:

Follow the directions in the instrument manual provided.

For liquid compounds with a boiling point above 100 oC, run a neat sample on salt plates. For liquid compounds with a boiling point at or below 100 oC, run a neat sample in a sealed cell. For solid compounds soluble in chloroform, run a 10% solution in chloroform. For solid compounds not soluble in chloroform, run a mull.

The spectrum should be plotted with peak positions printed.

IDENTIFICATION OF COMPOUND:

The experimental IR spectrum is compared to the standard IR spectrum for the compound, found in the lab spectra book, and matched over all. Every peak in the Standard IR should be in the experimental IR and the relative intensities should be the same. There should be no extraneous peaks in the experimental IR. This tells you that your experimental compound is, in the case of the example used, 1-butanol and not any other 1o aliphatic alcohol, all of which would have the same major functional group peaks.

In the data table, Part A is the statement: "experimental IR matches standard spectrum"

Compare the experimental IR spectrum to the standard run by the same method.

STRUCTURE DETERMINATION OF PRODUCT:

The Correlation Chart gives the most common primary peak positions of functional group absorption for most compounds; peak position can shift slightly due to variation in structure, some peak position can shift significantly; see Pavia pg. 702 - 712 for additional explanation.

When determining structure, look for the most prominent peak in the region of functional group absorption. Do not try to identify all peaks, many compounds have fine structure which is not identified peak by peak.

Be careful with aliphatic CH2 and CH 3 bends; for the CH3 bend the peak around 1380 is critical. Above 1400, a peak may be either: if the compound has more CH2 groups than CH 3 groups and there is one peak between 1440 and 1470, it is probably a CH2 bend; If there are 2 peaks the lower peak is a CH3 bend and the higher is the CH2 bend.

If a compound has both alipahatic and aromatic components, the 1500-1400 region is difficult; aliphatic CH2/CH3 bends and aromatic C=C stretches may overlap. In this case do your best but, if necessary, peaks may be identified as:

alkane CH2 bend	1460, 1430	1465
aromatic C=C stretch		1500, 1400

For experimental products, you know what the structure should be & look for peaks that should be in the IR, i.e. for an aliphatic alcohol look for CH stretches, CH2 and CH3 bends, OH stretch and CO stretch. Do not identify peaks from functional groups which are not part of the known structure. For an aliphatic alcohol peaks would not be identified for aromatic groups, carbonyl groups, etc. even though there are peaks in the experimental IR that may match those positions.

IDENTIFICATION OF IMPURITIES

When the experimental spectrum is compared to the standard spectrum, if impurities are present there will be significant peaks present in the experimental spectrum which do not appear in the standard spectrum. In order for an impurity to be detected in the spectrum, the impurity must have functional groups which produce peaks in regions in which the major product does not have peaks; i.e. an alcohol impurity could be detected in a ketone, but an alkane could not be detected in an alkyl halide.

If the IR spectrum shows an impurity the Data Table part A should say "the experimental spectrum matches the standard spectrum with the exception of impurities listed in B", and the non matching peaks identified in Part. B in the same format as before.

CES/AEV
Revised 8/95

INFRARED CORRELATION CHART

To be used in conjunction with Pavia, Lampman, and Kriz
Appendix 3, pp. 699-712

TYPE OF VIBRATION	WAVE NUMBER (CM^{-1})	INTENSITY
C-H stretch		
alkane	3000-2850	s
alkene	3100-3000	s
alkyne	3300	s
aldehyde	2900-2800	w
	2800-2700	w
aromatic	3150-3050	s
C-H bend		
alkane: -CH	1340	w
-CH2	1465	m
-CH3	1450 and 1375	m
alkene: -CH oop	1000-650	m
aromatic: -CH oop		
no sub.	670	s
mono sub.	770-730, 710-690	s
o sub.	770-735	s
m sub.	900-860, 810-750, 725-680	m, s, m
p sub.	860-800	s
1,2,4,5 sub.	900-860	m
C=C stretch		
alkene	1680-1600	m-w
aromatic	1600 (2 peaks)	w
	1500-1400 (2 peaks)	m
C ≡ C stretch	2250-2100	w
C-O stretch		
phenol	1230	s
alcohol 1°	1050	s
2°	1100	s
3°	1150	s
ether: -aliphatic	1060-1150	s (broad)
-aromatic	1270-1230	
carboxylic acid	1250	s
ester	1300-1000 (2 peaks)	s
anhydride	1300-900	s

TYPE OF VIBRATION	WAVE NUMBER (CM^{-1})	INTENSITY
O-H stretch		
alcohol & phenol:		
free	3650-3600	s
H-bonded	3400-3200	s (broad)
carboxylic acid	3300-2500	s(v.broad)
O-H bend oop		
carboxylic acid	950-900	m(broad)
alcohol	750-650	m (broad)
C=O stretch		
aldehyde	1740-1720	vs
ketone	1725-1705	vs
carboxylic acid	1725-1700	vs
ester	1750-1730	vs
acid chloride	1810-1750	vs
amide	1700-1640	vs
anhydride	1810 and 1760	vs
C-Cl stretch	800-600	s
overtone	1500-1470	m
C-Br stretch	600-500	s
overtone	1200	m
C-I stretch	500	s
overtone	1150	m
N-H stretch		
amide, unsub.	3500-3100 (2 peaks)	m (sharp)
monosub.	3500-3100 (1 peak)	m (sharp)
amine, 1°	3500-3300 (2 peaks)	m (sharp)
2°	3500-3300 (1 peak)	m (sharp)
N-H bend		
amine	1640-1560	m (broad)
amide	1640-1550	m (broad)
C-N stretch		
amine: -aliphatic	1220-1020	m
-aromatic	1350-1250	s

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Quick Procedures for Infrared Analysis

Infrared spectra: It is important to remember that the absence of an absorption band can often provide more information about the structure of a compound than the presence of a band. Be careful to avoid focusing on selected absorption bands and overlooking others. Use the examples linked to the table to see the profile and intensity of bands. Remember that the absence of a band may provide more information than the presence of an absorption band.

Look for absorption bands in decreasing order of importance:

1. the C-H absorption(s) between 3100 and 2850 cm^{-1}. An absorption above 3000 cm^{-1} indicates C=C, either alkene or aromatic. Confirm the aromatic ring by finding peaks at 1600 and 1500 cm^{-1} and C-H out-of-plane bending to give substitution patterns below 900 cm^{-1}. Confirm alkenes with an absorption at 1640-1680 cm^{-1}. C-H absorption between 3000 and 2850 cm^{-1} is due to aliphatic hydrogens.

2. the carbonyl (C=O) absorption between 1690-1760cm^{-1}; this strong band indicates either an aldehyde, ketone, carboxylic acid, ester, amide, anhydride or acyl halide. The an aldehyde may be confirmed with C-H absorption from 2840 to 2720 cm^{-1}.

3. the O-H or N-H absorption between 3200 and 3600 cm^{-1}. This indicates either an alcohol, N-H containing amine or amide, or carboxylic acid. For $-NH_2$ a doublet will be observed.

4. the C-O absorption between 1080 and 1300 cm^{-1}. These peaks are normally rounded like the O-H and N-H peak in 3. and are prominent. Carboxylic acids, esters, ethers, alcohols and anhydrides all containing this peak.

5. the CC and CN triple bond absorptions at 2100-2260 cm^{-1} are small but exposed.

6. a methyl group may be identified with C-H absorption at 1380 cm^{-1}. This band is split into a doublet for isopropyl(*gem*-dimethyl) groups.
7. structure of aromatic compounds may also be confirmed from the pattern of the weak overtone and combination tone bands found from 2000 to 1600 cm^{-1}.

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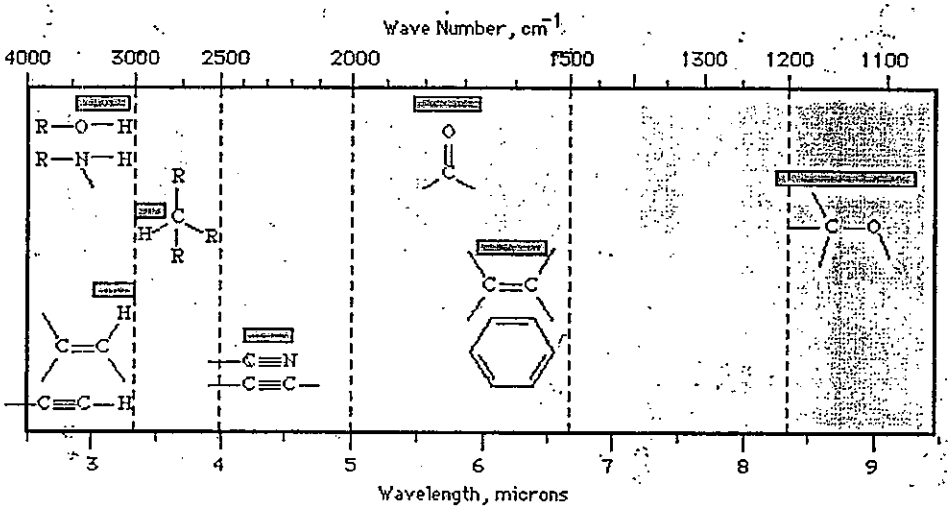
Infrared Absorbances for Common Functional Groups

Return | Spectroscopy Home

Use the Back Arrow to return to a spectroscopy problem



- Alcohols and amines display strong broad O-H and N-H stretching bands in the region 3400-3100 cm^{-1}. The bands are broadened due to hydrogen bonding and a sharp 'non-bonded' peak can often be seen at around 3400 cm^{-1}.

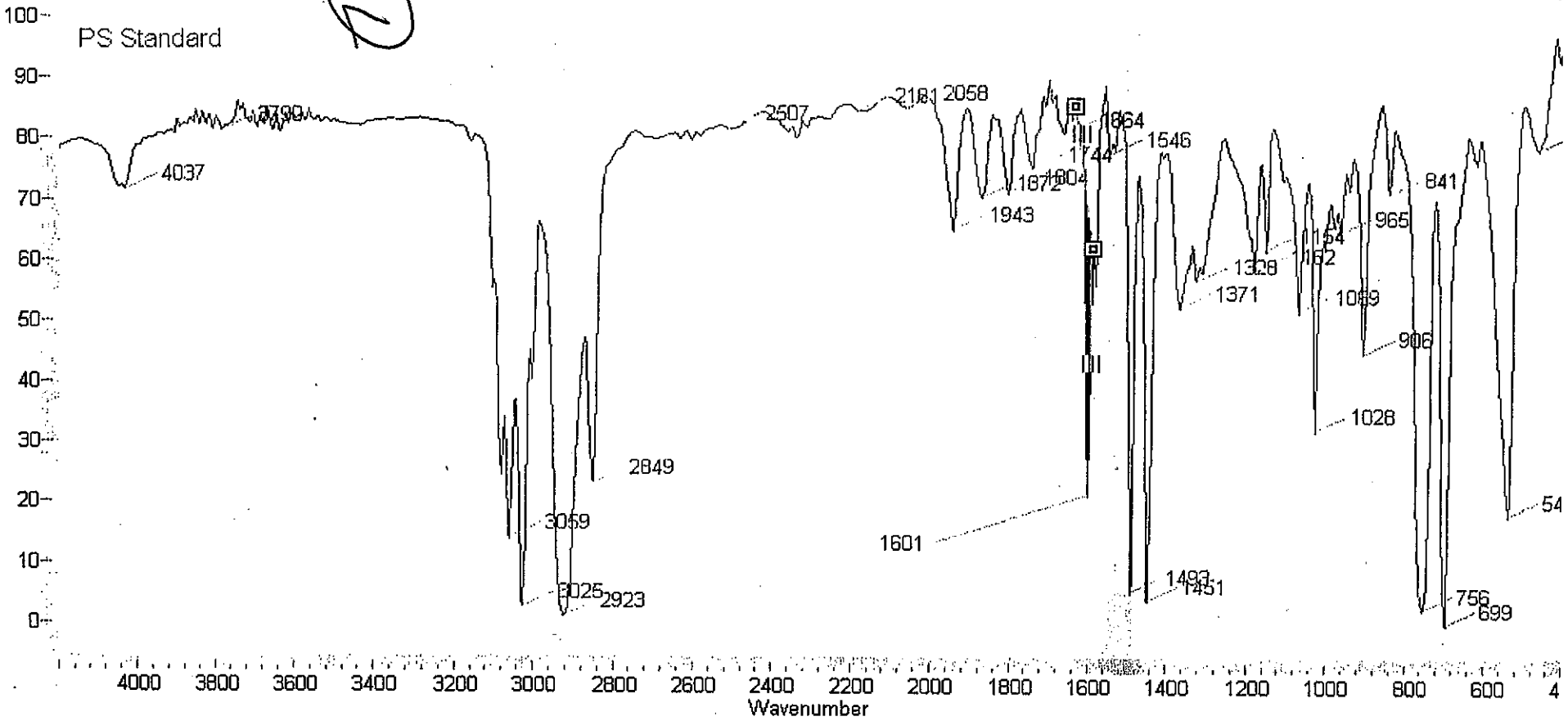
- Alkene and alkyne C-H bonds display sharp stretching absorptions in the region 3100-3000 cm^{-1}. The bands are of medium intensity and are often obscured by other absorbances in the region (i.e., OH).

- Triple bond stretching absorptions occur in the region 2400-2200 cm^{-1}. Absorptions from nitriles are generally of medium intensity and are clearly defined. Alkynes absorb weakly in this region unless they are highly asymmetric; symmetrical alkynes do not show absorption bands.

- Carbonyl stretching bands occur in the region 1800-1700 cm^{-1}. The bands are generally very strong and broad. Carbonyl compounds which are more reactive in nucleophilic addition reactions (acyl halides, esters) are generally at higher wave number than simple ketones and aldehydes, and amides are the lowest, absorbing in the region 1700-1650 cm^{-1}.

- Carbon-carbon double bond stretching occurs in the region around 1650-1600 cm^{-1}. The bands are generally sharp and of medium intensity. Aromatic compounds will typically display a series of sharp bands in this region.

'arian Resolutions Pro



	Name	Spectrum	Hitlist	Created Scan Scans	Created Scan Resolution
1	PS Standard		Hits = 0	8	4

Appendix D
CHP
Chemical Hazards and Precautions
Safety Information

CHEMICAL IDENTIFICATION & HAZARD LIST

Student CHP #	1 COMPOUND	2 CAS. NO.	3	4	5 PEL	6 OT	H	F	R	7	8	9 Fl. Pt.	S	W
	Acetic acid	64-19-7	*	MSDS	10 ppm		2	2	1			43°C	a	h
	Acetic acid, ethyl ester	141-78-6	*	BOTH	400 ppm	3.9 ppm	1	3	0				f	h
	Acetic anhydride	108-24-7	*	BOTH	5 ppm	0.13ppm	2	2	1	w			d	h
	Acetanilide, o-bromo	5326-87-4								NL			n	n
	Acetanilide, p-bromo	103-88-8								NL			n	n
	Adipic acid	124-04-9	*	BOTH			2	1	0				a	n
	Aniline	62-53-3	*	BOTH	2 ppm	1.1 ppm	3	2	0				d	h
	Aniline, o-bromo	615-36-1								NL			n	n
	Aniline, p-bromo	106-40-1		MSDS						NL			d	h
	Benzene, 1,4 dimethoxy	150-78-7		MSDS						NL			n	n
	Benzene, 1 ,4-dimethoxy-2 -t-butyl									NL			n	n
	Benzene, 1 ,4-dimethoxy-2 ,5-di-t-butyl									NL			n	n
	Benzene, nitro	98-95-3	*	BOTH	1 ppm	<.018 ppm	3	2	0				d	h
	Benzene sulfonic acid , 2,5-dimethoxy									NL			a	h
	Benzoic acid	65-85-0	*	MSDS			2	1	U				n	n
	Benzoic acid, methyl ester	93-58-3	*	MSDS			0	2	0			83°C	f	h
	Butane	106-97-8	*	NJFS			1	4	0				f	h
	Chloroform	67-66-3	*	BOTH	2 ppm	200 ppm	2	0	0	C			c	h
	Hypochlorous acid	7790-92-3								NL			a	h
	Iron	7439-89-6		MSDS			1	3	0		MS		f	h
	Iron(II) oxide	1345-25-1								NL			n	n
	Magnesium	7439-95-4	*	BOTH			0	1	1	w			f	h
	Magnesium bromide	7789-48-2								NL			n	n
	Magnesium hydroxy bromide									NL			n	n
	Magnesium sulfate	7487-88-9		MSDS			U	0	0		*		n	n
	Methanol	67-56-1	*	BOTH	200 ppm	100 ppm	1	3	0			12°C	f	h

Student CHP #	1 COMPOUND	2 CAS. NO.	3	4	5 PEL	6 OT	H	F	R	7	8	9 Fl. Pt.	S	W
	5-Nonanol, 5-methyl	33933-78-7								NL			f	h
	Nonene	10405-85-3								NL			f	h
	Nujol (Mineral oil)	8012-95-1					U	1	0				n	n
	Octane	111-65-9	*	BOTH	300 ppm	48 ppm	0	3	0			13°C	f	h
	Pentanoic acid	109-52-4	*	MSDS			2	1	0				a	h
	Sodium acetate	127-09-3		MSDS						NL			n	n
	Sodium bisulfite	7631-90-5	*	BOTH	5 mg M^3		3	0	0				d	h
	Sodium carbonate	497-19-8		MSDS						NL			n	n
	Sodium hypochlorite	7681-52-9	*	BOTH						NL	MS		d	f
	Sulfuric acid , fuming	8014-95-7	*	BOTH	1 mg M^3		3	0	2	ox			a	h

NMR Solvents and Reference Standards:

	Silanor-C Chloroform d_1 Tetramethylsilane	 865-49-6 75-76-3	*	MSDS	2 ppm					C			c	h
	Silanor-A Acetone d_6 Tetramethylsilane	 666-52-4 75-76-3	*	MSDS									f	h
	Silanor-DMSO Dimethylsulfoxide Tetramethylsilane	 2206-27-1 75-76-3		MSDS									d	h
	Deuterium Oxide	7789-20-0		MSDS						NL			n	n
	Sodium 2,2-dimethyl-2-silapentane (DSS)	2039-96-5								NL			n	n
	Tetramethylsilane (TMS)	75-76-3	*	MSDS						NL	MS		f	h

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NOTES:

(1) Compounds are listed alphabetically by IUPAC names, as found in the Handbook of Chemistry and Physics, CRC. This list includes all chemicals found in the CHE 231 experiments.

(2) CAS. NO. - Chem Abstracts Service Number Identification - as required by the New Jersey Worker and Community Right to Know Act - Given for all chemicals unless none is available.

(3) * - Identified as on Right to Know Hazardous Substance List, December 1989, New Jersey Worker and Community Right to Know Act.

(4) NSFS - New Jersey Fact Sheet is available in laboratory;; MSDS - Material Saftey Data Sheet is available in laboratory;
BOTH - Both of the above are available

(5) PEL - OSHA Permissable Exposure Limits - airborne exposure limit averaged over an 8 hour day. These exposure limits are for air levels only. When skin contact also occurs you may be overexposed even though air levels are less than the limits listed.

(6) OT - Odor Threshold - airborne concentration at which odor is noticed.

H,F,R National Fire Protection Assocication (NFPA) Ratings are taken from the MSDS where available, or from Instant Warning System for Dangerous Materials, @1980, Science Related Materials Inc.

Chemicals are rated at four hazard levels from 0 - no hazard to 4 - extreme hazard (u -unrated), based on the hazard presented in a fire fighting situation, in four hazard classes:

Health - H - (Blue in NFPA diamond) ; Flammability - F - (Red in NFPA diamond)
Reactivity - R - (Yellow in NFPA diamond); Special notice - see (7) below (White in NFPA diamond)

(7) NFPA Special Notice: C - suspected carcinogen - use only in Designated Area; ~~w~~ - water reactive;
oxy - oxidizing agent; exp - explosive - heat or shock sensitive; pol - polymerizes under normal conditions
NL - NFPA not listed;

(8) Look up hazard - Some chemicals pose hazards in the laboratory which are not critical in a fire fighting situation. These chemicals must be looked up in spite of a low NFPA rating. Look up hazard in:
* - Merck Index or other source; NJ - New Jersey Fact Sheet; MS - Material Safety Data Sheet

(9) Fl.Pt. - Flash point is listed when the flash point is not found in the Merck Index.

S,W Spill and Waste codes: See Spill & Waste Codes - Explanation

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CHP APPENDIX

CHP #	Compound	CAS #	NFPA Rating	S	W	Flash Pt.	Reference
12-1	Magnesium	7439-95-4	H-0, F-1, R-1	f	h	N/L	BOTH
12-2	1-Bromobutane	109-65-9	H-2, F-3, R-0	f	h	18°C	MSDS
12-3	Ethyl Acetate	141-78-6	H-1, F-3, R-0	f	h	-4°C	MSDS
12-4	Concentrated Sulfuric Acid	7664-93-9	H-3, F-0, R-2	a	h	208°C	BOTH
12-5	Anhydrous Ether	60-29-7	H-2, F-4, R-1	f	h	-45°C	BOTH
12-6	Water	7732-18-5	N/L	N/L	N/L	100°C	NONE
12-7	5-Methyl-5-Nonanol	33933-78-7	N/L	f	h	N/L	MSDS
12-8	5-Methyl-4-Nonene	N/L	N/L	N/L	N/L	N/L	NONE
12-9	Sodium Hydroxide (10% Sol.)	1310-73-2	H-3, F-0, R-1	b	h	N/L	BOTH
12-10	Saturated Sodium Chloride	7647-14-5	H-1, F-0, R-0	n	n	N/L	MSDS
12-11	Magnesium Sulfate	7487-88-9	H-1, F-0, R-0	h	h	N/L	MSDS
12-12	Acetone	67-64-1	H-1, F-3, R-0	f	h	-18°C	BOTH
12-13	Ethanol	64-17-5	H-0, F-3, R-0	f	h	13°C	BOTH
12-14	Magnesium Bromide	7789-48-2	H-1, F-0, R-0	n	n	N/L	MSDS
12-15	Butane	106-97-8	H-1, F-4, R-0	f	h	N/L	NJFS
12-16	Butanol	71-36-3	H-1, F-3, R-0	f	h	37°C	BOTH
12-17	Pentanoic Acid	109-52-4	H-2, F-1, R-0	a	h	95°C	MSDS
12-18	Octane	111-65-9	H-0, F-3, R-0	f	h	13°C	BOTH
12-19	Magnesium Hydroxy Bromide	N/L	N/L	n	n	N/L	NONE

Hazards & Precautions

12-1 **Magnesium:** Warning! Flammable Solid. Dangerous when wet. Highly reactive. May cause irritation to skin, eyes and respiratory tract. May ignite spontaneously on contact with water or damp materials.

12-2 **1-Bromobutane:** Danger! Highly flammable. May cause central nervous system depression. May cause eye and skin irritation. May cause respiratory and digestive tract irritation.

12-3 **Ethyl Acetate:** Warning! Flammable liquid and vapor. Harmful if swallowed or inhaled. Affects Central Nervous System.

12-4 **Concentrated Sulfuric Acid:** Causes irritation to skin and eyes and is harmful when ingested. Wear appropriate clothing and goggles.

12-5 **Anhydrous Ether:** Danger! Extremely flammable liquid and vapor. Harmful if swallowed, inhaled or absorbed through skin. Affects Central Nervous System.

12-6 **Water:** N/L

12-7 **5-Methyl-5-Nonanol:** N/L

12-8 **5-Methyl-4-Nonene:** N/L

12-9 **Sodium Hydroxide (10% Sol.):** Causes burns and irritation to eyes and skin; causes blindness; ingestion can be fatal.

12-10 **Saturated Sodium Chloride:** Can affect the body if it is inhaled or if it comes in contact with the eyes or skin. It can also affect the body if swallowed.

12-11 **Magnesium Sulfate:** Caution! May be harmful if swallowed.

12-12 **Acetone:** Danger! Extremely flammable liquid and vapor. Vapor may cause flash fire. Causes irritation to skin, eyes and respiratory tract. Affects Central Nervous System.

12-13 **Ethanol:** Highly flammable! Vapor/air mixture are explosive. Harmful if swallowed, inhaled or poured in eyes.

12-14 **Magnesium Bromide:** Caution! May cause skin irritation. Causes respiratory and digestive tract irritation and eye irritation.

12-15 **Butane:** Extremely Flammable! Vapor/air mixture are explosive. Harmful if swallowed, inhaled or poured in eyes.

12-16 **Butanol:** Can cause eye and skin irritation. Swallowing large amounts may be harmful.

12-17 **Pentanoic Acid:** Caution! Causes skin irritation and digestive and respiratory tract irritation. Causes severe eye irritation and possible eye injury.

12-18 **Octane:** Danger! Flammable liquid and vapor. Harmful or fatal if swallowed. Harmful if inhaled.

12-19 **Magnesium Hydroxy Bromide:** N/L

SPILL & WASTE CODES - EXPLANATION

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ALL SPILLS ON THE BODY - FLUSH WITH WATER for a minimum of 15 minutes, WASH WITH SOAP SOLUTION - NOTIFY LABORATORY INSTRUCTOR IMMEDIATELY

a. **Acids**:
- solids - scoop up solid and place in acid waste container
- liquids & solutions, either:
  1. neutralize with Sodium bicarbonate, scoop up solid and place in acid waste container
  2. use Acid Spill Kit

b. **Bases**:
- solids - scoop up solid and place in waste container
- liquids & solutions, either:
  1. neutralize with 6M HCl, absorb on paper towels, rinse towels in Sodium bicarbonate solution before discarding
  2. use Caustic Spill Kit
  3. absorb on spill wipe or spill pillow, bag, label and turn in

c. **Carcinogens**: absorb on spill wipe or spill pillow, bag, label and turn in. Place paper towels used to do final clean-up in waste bag also.

d. **Oxidizers/Toxics/Corrosives**:
- solids - scoop up solid and place in waste container
- liquids & solutions - absorb on spill wipe or spill pillow, bag, label and turn in

f. **Flammable liquids**, either:
1. absorb on spill wipe or spill pillow, bag, label and turn in
2. use solvent spill kit

h. **Hazardous waste**: place all waste in labeled waste container

m. **Heavy metal waste**: place all waste in labeled waste container, including at least two container rinses

n. **Nonhazardous waste**:
- solids - brush up and dispose of
- liquids - flush with water down drain

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CHE 236, 223, 227

CARCINOGEN SAFETY TRAINING

CARCINOGEN commonly describes any agent that can initiate or speed the development of malignant or potentially malignant tumors, malignant neoplastic proliferation of cells, or cells that possess such materials.

An OSHA Select carcinogen means any substance that meets one of the following criteria:

* It is regulated by OSHA as a carcinogen.

* It is listed under the category "known to be carcinogens" in the National Toxicology Program (NTP) "Annual Report on Carcinogens" (latest edition)

* It is listed under Group 1, "carcinogenic to humans" by the International Agency for Research on Cancer Monographs (IARC).
* It is listed under Group 2A or Group 2B by IARC or under the category "reasonably anticipated to be carcinogens" by NTP, and causes statistically significant tumor incidences in experimental animals according to any of the following criteria:
    a. after inhalation exposure of 6-7 hours per day, 5 days per week, for a significant portion of a lifetime, to doses of less than 10 mg/m3
    b. after repeated skin application of 300 mg/kg of body weight per week
    c. after oral doses of less than 50 mg/kg of body weight per day

(See page 3 for explanation of NTP and IARC categories)

The use of OSHA Select Carcinogens are regulated under the OSHA Lab Standard as Highly Hazardous Chemicals. They must be used in designated areas, and all persons using them must receive chemical-specific training in the health hazards, the handling requirements and the protective equipment required.

CHE 236, 223, 227

CARCINOGEN: Chloroform CAS No. 76-66-3
NTP: Group 2 - Reasonably anticipated to be carcinogens
IARC: Group 2B - sufficient evidence of carcinogenicity in experimental animals
See Fisher MSDS for additional health and safety information.

SMT HANDLING REQUIREMENTS:

Areas and Methods of Use:

Chloroform is used only in the designated areas in Rooms 140D and 146A. In these areas, chloroform is used only in the posted hoods; closed cells and sample tubes containing chloroform may be moved to adjacent instrumentation. In the hoods, chloroform is used only within the confines of the hood liner trays.

All equipment in which chloroform is used: glassware, dropper bulbs, and pipettes, is left in the hood. Disposables such as Kimwipes, droppers and pipette tips are placed in the labelled containers provided in the hood. Other equipment which is used only in these hoods should not be removed.

Personal Protective Equipment

When chloroform is being used, persons actively using it must wear the gloves, lab aprons, and chemical splash goggles supplied. In addition, long sleeves, or lab coats, must be worn, so that no gap exists between sleeve cuff and glove cuff.

Persons in the general area who are not actively using the chloroform must wear chemical splash goggles at all times.

Spills

Spills of chloroform are absorbed either with spill wipes or spill pillows. These are then bagged, sealed and labelled. The sealed bag is left in the hood for pick-up as hazardous waste, and the college Chemical Safety Officer notified.

Storage and Waste Handling

Chloroform is stored in the posted storage areas in each room, in secondary containers sufficient to contain the volume stored in case of breakage.

Used and excess chloroform is placed in labelled bottles, which are kept in the storage area until they are full or are picked up for disposal.

Disposal of waste chloroform, contaminated equipment and disposables is the responsibility of the Laboratory Safety Officer or the college Chemical Safety Officer, or their designate.

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The following is quoted from the Seventh Annual Report on Carcinogens, Summary 1994, NTP.

NTP CATEGORIES:

1) Known to be carcinogens:

There is sufficient evidence of carcinogenicity from studies in humans which indicates a causal relationship between the agent and human cancer

2) Reasonably anticipated to be carcinogens:

A. There is limited evidence of carcinogenicity from studies in humans which indicates that causal interpretation is credible, but that alternative explanations, such as chance, bias or confounding, could not be excluded, or

B. There is sufficient evidence of carcinogenicity from studies in experimental animals which indicates that there is an increased incidence of malignant tumors: (a) in multiple species or strains, or (b) in multiple experiments (preferably with different routes of administration or using different dose levels), or (c) to an unusual degree with regard to incidence, site or type of tumor, or age at onset. Additional evidence may be provided by data concerning dose-response effects, as well as information on mutagenicity or chemical structure.

IARC CATEGORIES:

Group 1: The agent is carcinogenic to humans: Sufficient evidence of carcinogenicity to humans is necessary. Sufficient evidence is considered by IARC to be evidence that a causal relationship has been established between exposure to the agent and human cancer.

Group 2A: The agent is probably carcinogenic to humans: This category generally includes agents for which there is limited evidence of carcinogenicity in humans and sufficient evidence of carcinogenicity in experimental animals. On occasion IARC may classify an agent in the category solely on the basis of limited evidence of carcinogenicity in humans or of sufficient evidence of carcinogenicity in experimental animals in view of supporting evidence from other relevant data.

Group 2B: The agent is possibly carcinogenic to humans: This category generally includes agents for which there is limited evidence of carcinogenicity in humans in the absence of sufficient evidence of carcinogenicity in experimental animals. It may also be used when there is inadequate evidence of carcinogenicity in humans or when human data are nonexistent but there is sufficient evidence of carcinogenicity in experimental animals. In some instances, agents by be included for which there is inadequate evidence or no data in humans but limited evidence of carcinogenicity in experimental animals together with supporting evidence from other relevant data.

Organic Students

Product label Instructions

All products are turned in to your Instructor

Labeled as follows:

1. Experiment number, (Exp. 3)
2. Chemical compound name
3. CAS #
4. student name
5. gram yield
6. melting point range (if applicable)
7. boiling point range (if applicable)
8. uncorrected refractive index(if applicable)

■■■

ORGANIC CHEMISTRY LAB
Student Contract

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-[] PAG

I have read, I am familiar with, I understand, and I agree to the terms and the conditions of:

ORGANIC CHEMISTRY LAB
Student Contract

I will maintain the document and refer to the document during the term.

Name_____

Signed_____

Date_____

Course_____ Section_____

Term_____