Organic Chemistry
Lab 1
Experiment Packet
Fall Quarter 07
BASTYR UNIVERSITY

COURSE INFORMATION FOR STUDENTS

Fall Quarter '07

<table>
<thead>
<tr>
<th>COURSE NUMBER</th>
<th>BC3124</th>
</tr>
</thead>
<tbody>
<tr>
<td>COURSE TITLE</td>
<td>Organic Laboratory Lab 1</td>
</tr>
<tr>
<td>INSTRUCTOR</td>
<td>Tess Cebrian</td>
</tr>
</tbody>
</table>
| CLASS TIME    | Weeks 1,5,7,10: Friday, 8:00-11:50 Section A and 1:00 -4:50 Section B  
                Weeks 2,4,6,8: Friday 8:00-11:50 Section C and 1:00 -4:50 Section D  
                Weeks 3: Friday All sections – Library research  
                Week 9: Thanksgiving holiday |
| CREDITS       | 1 each |
| STUDENT ADVISING HOURS | 12-1 Friday: by appointment |
| PHONE         | (W)(206) 587-4075  (Fax)206 587-3837  (E-mail)tcabas@sccd.ctc.edu |

Website: www.chemsccc.org

Students are responsible for knowing and adhering to Academic Policies and Procedures as outlined in the Student Handbook.

*Listed are the major areas to cover. Please see Course Syllabus Instructions for more details on content*

** There is no lab make up. Only excused absences will be exempted from 20% point deduction. Absences due to illness must have a doctors note to be excused.

1. Table of Contents

Course Overview and Objective
Requirements
Evaluation
Lab Schedule
Lab Report Format
Sample Lab Report

2. Syllabus Introduction

3. Course Overview

- Course Description

The course is designed as a practical application of the theories learned in lecture. Experiments are picked to have relevancy to the ND, Nutrition, exercise and
herbal science programs. Experiments include techniques of simple, fractional and steam distillation, esterification of fatty acids, and isolation of natural product. The experiments will vary in degree of difficulty. As the quarter progresses, the experiments will be more challenging but also more interesting and relevant to your program. It is important to come to lab prepared, on time and with a positive attitude. Remember the definition of “experiment.” Although the experiments have been tried and are known to work, sometimes they do not. Failure of an experiment is just as important a learning tool as one that works. It allows you to examine the procedure more thoroughly to figure out what went wrong. A degree of enthusiasm and the willingness to learn and hard work are the key to a successful lab experience. There is ample time to complete the experiments and at times to restart the experiment as needed.

- **Major Course Competencies**

  - Learn organic chemistry lab techniques.
  - Develop data analysis skill.
  - Apply theories learned from lecture to interpret data gathered from the experiments.
  - Learn to write a complete, clear and concise lab report.

- **Organization & Requirements**

  1. Completion of 3 experiments, 2 exercise/data sheet, 2 formal lab reports, and a research paper are required to complete the course.
  2. On week 3, October 12, there will be no lab. It is a day assigned for you to do research on your paper, Biodiesel, an alternative to fossil fuel. Write a 3 page paper on the biodiesel. Include in the paper a realistic experiment proposal that can be
  3. There is no lab make up. An unexcused absence will be a deduction of 20% from your total points.
  4. Experiments will be performed in pairs (no more) unless otherwise instructed.
  5. **A lab notebook (composition type- not binders or spiral type) is required for the course.** All notes and record keeping during the experiment will be done in the lab notebook not in loose pieces of paper. Write in pen only. Record all data on the lab notebook. Cross out unwanted data. Do not erase or block-out data. Transfer data (if included in the experiment procedure) to the printed data sheet in the handout to be included in the lab report. There have been many data sheets misplaced or lost. Having the data recorded in the notebook reduces the probability of loosing important data.
  6. The pre-lab assignment for each experiment is to prepare a pictorial flow chart of the experiment in your lab notebook. The purpose of this exercise is to allow you to understand the procedure clearly. **Pre-lab assignments will be checked off at the beginning of each lab period.** You will not be allowed to start the lab until prelab assignment is completed.
  7. Lab reports and post lab questions are due the following week after the completion of the experiment (the week that you do not have lab). Lab reports can be a collaborative work between partners or individual reports. Please do not turn in identical reports with different names. **All hard copy reports must be in by 1:00 PM.** An electronic copy can also be emailed to tcabas@scccd.ctc.edu as “word” attachment. A 10-point deduction per week will be applied for late report.
  8. Compliance to the laboratory rules is essential for the safety of class.
• **Prerequisite Knowledge**
  General chemistry techniques.

• **Instructional Materials and Resources**
  Hand-outs

4. **GRADING**

• **Evaluation Standards with Criteria for Passing and Remediation**

  EVALUATION:
  Your lab grade will be based on the following
  Prelab assignment 10pts each
  Formal lab reports/post lab question (50)
  and Laboratory performance(50 pts) 100pts each
  Notebook 10pts each

  **Grading will be as follows:**
  95-100% A
  90-94.9 A-
  87-89.9 B+
  83-86.9 B-
  80-82.9 B
  77-79.9 C+
  73-76.8 C

Laboratory performance will be an evaluation of lab technique, lab preparedness, lab etiquette; which includes cleaning after oneself, keeping track of lab glassware, putting away chemicals, effort given to make for a successful experiment, and most important, following safety instructions. Attendance of lab lecture during the first half hour of lab is required. You will not be allowed to perform the lab if not present for lab lecture. Courtesy to lab instructor and classmates is expected.

5. **Study Strategies & Class Participation Expectations**

• A positive attitude and willingness to learn is essential to be successful.
• Download the lab lecture from www.chemsccc.org. Click on the Bastyr University link and download appropriate lecture notes.
• You must come to lab prepared and on time. Read the experiments ahead of time.
• Complete your pre-lab assignment, the pictorial flowchart of your experiment.
• Write down questions that come up as you read the experiment. Ask questions!
• Remind yourself of the definition of "experiment" when things are not working out well with your experiment.
• Although good results are the ultimate goal, determining why an experiment did not work and understanding the experiment is equally or more valuable.

6. **Syllabus Chapters/Materials (if applicable)**

All experiments are Handouts.
Laboratory Safety

Organic Lab 1
-Experiment with Models, Fermentation
-Isolation of Clove Oil
-Synthesis of Isoamyl Acetate
-Preparation of Soap

8. FORMAL LAB REPORTS
Word process the formal lab reports. Lab reports will contain the following information. Follow the format on the sample lab report.

Introduction - Provide a short informative paragraph about the chemical being evaluated and how it is used. Include information that describes the practical application of the compound or procedure in question and a statement of the objective. There may be more than one objective for each experiment.

Data (include calculations) Graphs, tables, spectra and chromatograms are part of the data and must also be included in the report.

Result and Discussion - Describe the procedure briefly and the results of your experiment. Example: If you separated compounds, tell how it was done. If you synthesized a compound, tell how it was made and in what yield. Support your statements with your data. Data report in the data sections should be referred in the result and discussion. Explain the data/results and what they imply. Discuss unusual observations, errors and anything that will better your understanding of the principles involved. Make suggestions that would solve the stated problems. Make this section brief and concise.

Conclusion - A brief statement to answer the problem posed in the objective.

Sample lab report - See attached.

9. Lab Schedule

<table>
<thead>
<tr>
<th>Week</th>
<th>Date</th>
<th>Section</th>
<th>Experiment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9/28</td>
<td>A,C</td>
<td>Experience with Models</td>
</tr>
<tr>
<td>2</td>
<td>10/5</td>
<td>B,C</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>10/13</td>
<td>A,B,C,D</td>
<td>Library Research</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Biodiesel, Alternative to Fossil Fuel</td>
</tr>
<tr>
<td>4</td>
<td>10/19</td>
<td>C,D</td>
<td>Isolation of Clove Oil</td>
</tr>
<tr>
<td>5</td>
<td>10/26</td>
<td>AB</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>11/2</td>
<td>C,D</td>
<td>Synthesis of Isoamyl Acetate</td>
</tr>
<tr>
<td>7</td>
<td>11/9</td>
<td>A,B</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>11/16</td>
<td>C,D</td>
<td>Preparation of Soap</td>
</tr>
<tr>
<td>9</td>
<td>11/23</td>
<td></td>
<td>Thanksgiving Holiday</td>
</tr>
<tr>
<td>10</td>
<td>11/30</td>
<td>A,B</td>
<td>Preparation of Soap</td>
</tr>
</tbody>
</table>
Recrystallization of Benzoic Acid.

Introduction.
Benzoic Acid is an aromatic carboxylic acid with the structure, C₆H₅OH. Benzoic Acid occurs in nature in free and combined forms. Most berries contain appreciable amounts, about 0.05%. It is used mainly as a preservative in foods, juices; dyes; as a mordant in calico printing and for curing tobacco. Pharmaceutically, it is used as an antifungal agent.

In this experiment, benzoic acid will be purified by recrystallization. The appropriate solvent in which the compound is not soluble at room temperature but soluble in at elevated temperature will be determined. The impurity is separated by filtration if present in large amount. If present in minute amount, it is separated by its infinite solubility in the solvent as the pure compound is crystallized. The yield and purity of the recrystallized benzoic acid will be measured.

Data.

**Solvent Determination:**
- Ethanol – soluble at room temperature
- Hexane – insoluble at room temp and at elevated temperature
- Water – insoluble at room temp and soluble at elevated temperature

**Yield**
- Mass of impure benzoic acid. 5.256 g
- Mass of recrystallized benzoic acid 2.785 g
- % recovery 53 %

**Melting Point**
- Recrystallized benzoic acid 119.0-123.5º C
- Literature 122-123º C

Results and Discussion
Three solvents were tested; water, ethanol and hexane. Of the three, water fit the criteria of a good solvent for recrystallization, the benzoic acid was not soluble at room temperature but completely dissolved when heated. Water was used to recrystallize the impure benzoic acid.

Benzoic acid was recrystallized from water with a percent recovery of 53%. The original sample was heterogeneous mixture containing yellow, white and tan particles. The resulting solution appeared dark orange initially and a very light orange-yellow after decolorization. The recrystallized product appeared as a light yellow crystalline solid with a melting point of 119.0-123.5º C.

Pure benzoic acid is a white crystalline solid with a melting point of 122-123º C. The recrystallized benzoic acid in this experiment is slightly colored and has a lower and broader melting point range. These observations both indicate that this sample was not completely purified. Although not entirely pure, the recrystallized benzoic acid
was much lighter than the original sample. The low percent recovery was due to the loss of approximately $\frac{1}{4}$th of the total volume on the lab bench and from many transfers involved in the experiment. To increase the purity of the benzoic acid, it could be recrystallized again.

**Conclusion**

Water proved to be the best solvent to recrystallize the benzoic acid. The yield was 53% recovery. The melting point (119.0-123.5°C) of the recrystallized product was slightly depressed indicating some impurity in the sample.
LOCATIONS:

A,B,C,D, I, J - HOODS
E,P – INCUBATORS
F,H – DISHWASHING SINKS
G – ALL GLASSWARE AND METALWARE
K,L – EYEWASH STATION
M - FIRE BLANKET
N-MISCELLANEOUS SUPPLIES (PIPETS, PENS, RULERS)
O-FIRE EXTINGUISHER
R- FIRST AIDE KIT
SAFETY RULES FOR ORGANIC CHEMISTRY

1. Experiments can only be done during scheduled time only. Hood space is limited.
2. Organic chemistry lab involves working with chemicals that are carbon base. Organic chemicals can be one or all of the following:
   a. Volatile
   b. Flammable
   c. Toxic or carcinogenic
   d. Respiratory irritants
3. Modes of exposure to organic chemicals are:
   • inhalation
   • absorption through the skin
   • ingestion
4. Minimize your exposure.
   A. Inhalation
      a. Keep your time in the lab at a minimum by coming to lab prepared. Read experiments ahead of time. Ask questions during the lab lecture.
      b. Use the hoods.
   B. Absorption through the skin
      a. Wear gloves.
      b. Wear goggles at all times.
   C. Ingestion:
      a. Don’t eat or drink in the lab.
6. Know the health hazard of your reagents. MSDS are available on-line.
7. Dress appropriately for lab. Tie back long hair. The following are clothing are not allowed in lab:
   a. open toed shoes or sandals
   b. halter tops, tops that exposes the midriffs-stomach area, short shorts or mini skirts.
   c. Loose sleeves.
8. If pregnant or trying to be pregnant - consider taking the lab at a later time.
9. Dispose of all organic waste into waste jars.
10. Keep flammables away from open flame. If heating is required as much as possible use water baths. Baths are heated with a hot plate.
11. Read your labels very carefully. Inadvertently mixed chemicals can cause serious explosion.
12. Excess chemicals are considered waste. DO NOT RETURN CHEMICALS BACK TO THE REAGENT BOTTLE.
13. Prevent contamination by pouring our liquids into your own container. Don’t use droppers in the reagent bottle. Use a clean, dry spatula to get solids out of the reagent bottle.
14. No food or drinks are allowed in the lab.
15. Dispose of broken glass in the broken glass bucket.
16. Rinse chemicals off skin for at least 15 minutes with cold water.
17. Know the locations of the safety equipment, such as the fire extinguisher, eye wash station, and emergency shower.
Organic Lab I
Experiment 1

Molecular Geometry:
Experience with Models

Objective:
To become familiar with the three-dimensional aspects of organic molecules.

Materials:
Molecular models (ball and stick). A black sphere with four holes represents carbon, hydrogen by a white sphere with one hole, and chlorine by a green sphere with one hole.

Background:
Organic compounds are extremely numerous—in fact, there are approximately $2 \times 10^6$ known organic compounds. The chemical and physical properties of these compounds depend upon what elements are present, how many atoms of each element are present, and how these atoms are arranged in the molecule. Molecular formulas often, but not always, permit one to distinguish between two compounds. For example, even though there are eight atoms in both $\text{C}_2\text{H}_6$ and $\text{C}_2\text{H}_5\text{Cl}$, we know immediately that these are different substances on the basis of their molecular formulas. Similarly, inspection of the molecular formulas $\text{C}_2\text{H}_6$ and $\text{C}_3\text{H}_8$ reveals that these are different compounds. However, there are many substances that have identical molecular formulas but are completely different compounds. Consider the molecular formula $\text{C}_2\text{H}_6\text{O}$. There are two compounds that correspond to this formula: ethyl alcohol and dimethyl ether. While the molecular formula gives no clue as to which compound one may be referring, examination of the structural formula immediately reveals a different arrangement of atoms for these substances:

\[
\begin{align*}
\text{H} & \quad \text{H} \\
| & \quad | \\
\text{H} & \quad \text{C} & \quad \text{C} & \quad \text{O} & \quad \text{H} \\
| & \quad | \\
\text{H} & \quad \text{H} \\
\end{align*}
\]

Ethyl alcohol

\[
\begin{align*}
\text{H} & \quad \text{H} \\
| & \quad | \\
\text{H} & \quad \text{C} & \quad \text{O} & \quad \text{C} & \quad \text{H} \\
| & \quad | \\
\text{H} & \quad \text{H} \\
\end{align*}
\]

Dimethyl ether

In addition, when molecular models (ball-and-stick type) are used, trial and error will show that there are just two ways that two carbons, six hydrogens, and one oxygen can be arranged without violating the usual valances of these elements. (Remember, carbon has a valance of 4, oxygen 2, and hydrogen 1.) Compounds that have the same molecular formula but different structural formulas are termed isomers. This difference in molecular structure results in differences in chemical and physical properties of isomers. In the case of ethyl alcohol and dimethyl ether, whose molecular formula is $\text{C}_3\text{H}_6\text{O}$, these differences are very pronounced (see Table 37.1). In other cases, the differences may be more subtle.
TABLE 1  Properties of Ethyl Alcohol and Dimethyl Ether

<table>
<thead>
<tr>
<th>Property</th>
<th>Ethyl Alcohol</th>
<th>Dimethyl Ether</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boiling point</td>
<td>78.5°C</td>
<td>-24°C</td>
</tr>
<tr>
<td>Melting point</td>
<td>-117°C</td>
<td>-139°C</td>
</tr>
<tr>
<td>Solubility in H₂O</td>
<td>Infinite</td>
<td>Slight</td>
</tr>
<tr>
<td>Behavior toward sodium</td>
<td>Reacts vigorously, liberating hydrogen</td>
<td>No reaction</td>
</tr>
</tbody>
</table>

The importance of the use of structural formulas in organic chemistry becomes evident when we consider the fact that there are 35 known isomers corresponding to the formula C₉H₂₀! For the sake of convenience, condensed structural formulas are often used. The structural and condensed structural formulas for ethyl alcohol and dimethyl ether are

**Structural formulas**

<table>
<thead>
<tr>
<th>Ethyl alcohol</th>
<th>Dimethyl ether</th>
</tr>
</thead>
<tbody>
<tr>
<td>H H</td>
<td>H H</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>H -C–C–O–H</td>
<td>H–C–O–C–H</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>H H</td>
<td>H H</td>
</tr>
</tbody>
</table>

**Condensed structural formulas**

CH₃CH₂OH     CH₃OCH₃

A short glance at these formulas readily reveals their difference. The compounds differ in their connectivity. The atoms in ethyl alcohol are connected or bonded in a different sequence from those in dimethyl ether.

You must learn to translate these condensed formulas into three-dimensional mental structures and to translate structures represented by molecular models to condensed structural formulas. Naming alkanes, the IUPAC way.

1. The longest continuous chain of carbon atoms is the parent chain. If there is no longest chain because two or more chains are the same longest length, then the parent chain is defined as the one with the most branches. (The idea here is to keep the name simpler. More branches numbered from the parent chain means fewer parentheses needed later.)

2. Carbon atoms connected to the parent chain but not part of it are parts of branches. To avoid ambiguity, branches are numbered based on the carbon number of the parent chain at the point of attachment to the parent chain.

3. The general idea in naming organic compounds is to always aim for the smallest
numbers possible. Whenever two or more possibilities exist, which is usually the case, because there are two ends you can start numbering from on all acyclic chains, "smallest numbers" means smallest at the first difference. Thus:

- Between 3-ethyl-4,8-dimethylnonane and 7-ethyl-2,6-dimethylnonane, the first results in the lower first-different number.
- Between 2,3,8-trimethylnonane and 2,7,8-trimethylnonane, the first results in the lower first-different number.

4. Basically, what this means is that if you start checking from both ends of the chain, stepping toward the center of the chain one carbon at a time counting 1, 2, 3..., then the branch you get to first sets all the other numbers for the name alphabetically. If two branches are reached at the same time, then the "winning" branch is the one that is first alphabetically. If the branches are the same, then you have to keep stepping in toward the center until a difference is found. If no difference is found, then it doesn't matter which end you number from.

5. Once all the numbers for the branches are determined, the branches are named using -yl, and ordered alphabetically. If branches themselves are branched, then the complete name of the branch (with numbers) must be determined at this time. It is the complete name of the branch which is alphabetized. Thus, for example:

- (2,2-dimethylbutyl) comes before ethyl
- isopropyl comes before sec-butyl
- If a halogen is a substituent, it is named first before the alkyl group.

6. Finally, when more than one of the same branch is present, the prefixes di, tri, tetra, etc. for simple branches. The name is constructed by separating numbers with commas and adding hyphens before and after sets of numbers so that they don't run into words. Branches with numbers are set off with parentheses so that it is clear that the numbers only refer to that branch. Other than that, there is no punctuation and there are no spaces in the names.

PROCEDURE:
Construct models of the molecules as directed and determine the number of isomers by trial and error as directed below. **Answer all questions that are bolded at the end of each section.**

A. Methane
Construct a model of methane, CH₄. Place the model on the desktop and note the symmetry of the molecule. Note that the molecule looks the same regardless of which three hydrogens are resting on the desk. All four hydrogens are said to be equivalent. Now, grasp the top hydrogen and tilt the molecule so that only two hydrogens rest on the desk and the other two are in a plane parallel to the desk top (see Figure 1). Now imagine pressing this methane model flat
onto the desktop. The resulting imaginary projection in the plane of the desk is the conventional representation of the structural formula of methane:

![FIGURE 1 Model of methane.](image)

Replace one of the hydrogen atoms with a chlorine atom to construct a model of chloromethane (or methyl chloride), CH₃Cl. Replace a second hydrogen atom by a chlorine atom to make dichloromethane, CH₂Cl₂. Convince yourself that the two formulas

\[
\begin{align*}
\text{H} & \quad \text{Cl} \\
\text{Cl} - \text{C} - \text{Cl} & \quad \text{and} \quad \text{Cl} - \text{C} - \text{H} \\
\text{H} & \quad \text{H}
\end{align*}
\]

represent the same three-dimensional structure and are not isomers. Replace another hydrogen to make CHCl₃, chloroform (or trichloromethane). Finally, make CCl₄, carbon tetrachloride.

**Question:** Write the structural formula and name each of the chloromethanes.

**B. Ethane**

Make a model of ethane, C₂H₆, from your model of CH₄ by replacing one of the hydrogens with a CH₃ unit; the —CH₃ unit is called the methyl group. Note that the hydrogens of ethane are all equivalent. Replace one of the hydrogens in your ethane model with a chlorine ball. Does it matter which hydrogen you replace? How many compounds result? Now examine your model of C₂H₅Cl and note how many different hydrogen atoms are present. If another hydrogen of C₂H₅Cl is replaced by a chlorine atom to yield C₂H₄Cl₂ how many isomers would result?

**Questions:**

1. **Does the formula, C₂H₄Cl₂, distinguish the possible molecules corresponding to this formula?**

2. **Write the condensed structural formula for C₂H₅Cl. Are there isomers for C₂H₅Cl?**

3. **Write the condensed structural formulas for all isomers of C₂H₄Cl₂ and assign the IUPAC names to each.**
C. Propane
From your model of ethane, construct a molecular model of propane, C₃H₈, by replacing one of the hydrogen atoms by a methyl group, —CH₃. Examine your model of propane and determine how many different hydrogen atoms are present in propane. If one of the hydrogens of propane is replaced with chlorine, how many isomers of C₃H₇Cl are possible?

Question:
1. Write the condensed structural formulas of the isomers of C₃H₇Cl and give their names.
2. How many isomers are there corresponding to the formula C₃H₆Cl₂. Write condensed structural formulas and IUPAC names for these isomers. By this stage in this experiment, you should realize that a systematic approach is most useful in determining the numbers of isomers for a given formulas.

3. Determine the number of isomers with the formula C₃H₅Cl₃. Write the condensed structural formulas and IUPAC names of all isomers.

D. Butane
The formula of butane is C₄H₁₀. From your model of propane, C₃H₈, construct all of the possible isomers of butane by replacing a hydrogen atom with a methyl group, —CH₃.

Questions:
1. How many isomers of butane are there? Give their structural formulas and IUPAC names.
2. There are four isomers corresponding to the formula C₄H₉Cl. Write their structural formulas and name them.
3. Write and name all of the isomers of C₄H₈Cl₂. Use your models to help answer this question.

E. Pentane
Questions:
1. Use your models in a systematic manner to determine how many isomers there are for the formula C₅H₁₂. Write their condensed structural formulas and name them.
2. Write and name all isomers for the formula C₅H₁₁Cl.

F. Cycloalkanes
Cycloalkanes corresponding to the formula CₙH₂ₙ exist. Try to construct (but do not force too much in your attempt) models of cyclopropane, C₃H₆; cyclobutane, C₄H₈; cyclopentane, C₅H₁₀; and cyclohexane, C₆H₁₂.

Questions:
1. Although cyclopropane and cyclobutane exist, would you anticipate these to be highly stable molecules?

G. Alkenes
Using two curved connectors for bonds, construct a model of ethene (ethylene), C₂H₄. Note the rigidity of the molecule; note that there is no rotation about the carbon-carbon double bond as there is in the case of carbon-carbon single bonds such as in ethane or propane.

Questions:

1. How many isomers are there corresponding to the formulas C₂H₅Cl? C₂H₂Cl₂?
2. Give the structures and names of all the isomers.
Organic Lab
Experiment 2

Isolating Clove Oil from Cloves Using Steam Distillation

INTRODUCTION
Simple and fractional distillations are carried out on miscible mixtures. Ideal mixtures follow Raoult’s law: The total vapor pressure of the system is determined by adding together the products of the vapor pressure and the respective mole fraction of each compound. For a two-compound system, this relationship is shown in Equation 1, where \( P_T \) is the total vapor pressure, \( P_1^\circ \) and \( P_2^\circ \) are the vapor pressures of pure compounds 1 and 2, and \( X_1 \) and \( X_2 \) are the respective mole fractions.

\[
P_T = P_1^\circ X_1 + P_2^\circ X_2 \quad \text{(Eq.1)}
\]

Distillation can also be performed on mixtures in which the two compounds are not miscible. This process is called codistillation. When one of the compounds is water, the process is called steam distillation.

When two immiscible liquids are distilled, the total vapor pressure \( P_T \) above the liquid is equal to the sum of the vapor pressures of each compound. This relationship, known as Dalton’s law, is shown in Equation 2.

\[
P_T = P_1^\circ + P_2^\circ \quad \text{(Eq.2)}
\]

The respective mole fractions are not included in this equation because, in an ideal situation, each liquid vaporizes independently of the other. When \( P_T \) is equal to atmospheric pressure of 760 torr, compounds 1 and 2 begin to codistill, with each compound contributing to \( P_T \).

Consider water as compound 1. The vapor pressure of pure water at its boiling point of 100 °C is 760 torr. Because compound 2 also contributes to \( P_T \), the mixture will distill at a temperature less than 100 °C. The actual distillation temperature will depend on the vapor pressure of compound 2. Steam distillation offers an advantage in that volatile compounds that are unstable or have high boiling points can codistill with water at relatively low temperatures. This process avoids decomposition that might occur at the normal boiling point of the compound of interest. For example, eugenol, the major compound of clove oil, boils at a relatively high temperature of 254 °C. Steam distillation avoids this high temperature and results in the distillation of eugenol at a temperature slightly less than 100 °C.

In practice, steam distillation is usually carried out by one of two methods. In the first method, an excess of water is added to the compound of interest in a distilling flask. The mixture is then heated to the boiling point. The resulting vapor is condensed and collected in a receiving flask. The compound of interest is then separated from the water, often by extraction. In the second method, steam is bubbled into the compound of interest to effect the distillation. In this experiment, you will use the first method because it is easier to set up.
Clove oil belongs to a large class of natural products called the **essential oils**. Many of these compounds are used as flavorings and perfumes and, in the past, were considered to be the "essence" of the plant from which they were derived. Cloves are the dried flower buds of the clove tree, *Eugenia caryophyllata*, found in India and other locations in the Far East. Steam distillation of freshly ground cloves results in clove oil, which consists of several compounds. Eugenol is the major compound, comprising 85-90 percent. Eugenol acetate comprises 9-10 percent. These structures are shown in Figure 1.

![Figure 1: Structures for (a) eugenol and (b) eugenol acetate](image)

**Qualitative Tests**

Eugenol contains a carbon-carbon double bond and an aromatic hydroxyl group called a phenol. These functional groups provide the basis for simple chemical tests used to characterize the clove oil. A solution of bromine ($\text{Br}_2$) in dichloromethane decolorizes as $\text{Br}_2$ reacts with the double bond to form a colorless compound, as shown in Equation 3. A positive test is the disappearance of the red $\text{Br}_2$ color.

\[
\text{CH}_2=\text{CH}_2 + \text{Br}_2 \rightarrow \text{BrCH}_2\text{CH}_2\text{Br}
\]

A potassium permanganate ($\text{KMnO}_4$) solution can oxidize a double bond at room temperature to form a 1,2-diol with the simultaneous reduction of $\text{Mn}^{7+}$ in $\text{KMnO}_4$ to $\text{Mn}^{4+}$ in manganese dioxide ($\text{MnO}_2$), as shown in Equation 4. A positive test is the disappearance of the purple $\text{KMnO}_4$ and the appearance of $\text{MnO}_2$ as a muddy brown precipitate.

\[3 \text{CH}_2=\text{CH}_2 + 2 \text{KMnO}_4 + 4 \text{H}_2\text{O} \rightarrow 3 \text{HOCH}_2\text{CH}_2\text{OH} + 2 \text{MnO}_2 + 2 \text{KOH}\]

Phenols ($\text{Ar-OH}$) react with the $\text{Fe}^{3+}$ ion in iron(III) chloride ($\text{FeCl}_3$) to give complexes that are blue, green, red, or purple, as shown in Equation 5. The color may last for only a few seconds or for many hours, depending on the stability of the complex.

\[6 \text{ArOH} + \text{Fe}^{3+} \rightarrow 3\left[\begin{array}{c} \text{Ar}\text{O} \\ \text{Ar}\text{O} \\ \text{Ar}\text{O} \end{array}\right] + 6 \text{H}^+ \]
In this experiment, you will steam distill clove oil from freshly ground cloves. Following the distillation, clove oil and water will be present in the receiving flask. Because clove oil will be a minor fraction of the distillate, the clove oil must be extracted from the water into an organic solvent such as dichloromethane. Removing the dichloromethane layer leaves clove oil as the product.

### Reagents and Properties

<table>
<thead>
<tr>
<th>Substance</th>
<th>Quantity/pair</th>
<th>Molar Mass (g/mol)</th>
<th>Boiling Point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cloves, ground</td>
<td>5 g</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dichloromethane</td>
<td>21 mL</td>
<td>84.29</td>
<td>40</td>
</tr>
<tr>
<td>eugenol (product)</td>
<td></td>
<td>164.20</td>
<td>254</td>
</tr>
<tr>
<td>sodium chloride, sat. solution</td>
<td>10 mL</td>
<td>58.44</td>
<td></td>
</tr>
<tr>
<td>sodium sulfate, anhydrous</td>
<td>0.5 g</td>
<td>142.04</td>
<td></td>
</tr>
<tr>
<td>methanol</td>
<td>16</td>
<td>32.04</td>
<td>64.7</td>
</tr>
<tr>
<td>1% Ferric Chloride Solution</td>
<td>1 mL</td>
<td>162.21</td>
<td></td>
</tr>
<tr>
<td>potassium permanganate, 0.05M</td>
<td>1 mL</td>
<td>158.04</td>
<td></td>
</tr>
<tr>
<td>Acetone</td>
<td>.5 mL</td>
<td>58.08</td>
<td>56</td>
</tr>
</tbody>
</table>

### Equipment

- Bunsen burner
- 10, 100 mL graduated cylinders
- Marking pen
- 4 pasteur pipets with latex bulb
- 4 test tubes (qual tubes)
- Steam Distillation apparatus

### Part 1. Conducting Steam Distillation

1. Weigh 5 g of ground cloves and record the mass.
2. Use a powder funnel to transfer the ground cloves to a 250-mL round-bottom flask. Add 40 mL of deionized or distilled water and a boiling chip to the flask. Mix well by swirling. Mark the level of the mixture on the side of the flask with a permanent marker.
3. Add 30 mL of water to a 100-mL round-bottom flask. Mark the level of the water on the side of the flask. Then discard the water from the flask.
4. Assemble the steam distillation apparatus shown in Figure 2. Grease all glass joints lightly. Use the 250-mL round-bottom flask as the boiling pot. Use the 100-mL round-bottom flask as the receiver. Pour 25 mL of water into the addition 30-mL separatory funnel. Connect the IN- hose to the
bottom hose connector and the OUT hose through the top hose connector. Tape the hoses in place. Start the flow of water through the condenser.

5. Adjust a Bunsen burner flame to lessen the hot central cone. Heat the boiling pot by waving the flame back and forth under the pot. Do not heat the mixture too rapidly. The clove oil tends to foam when rapidly heated. The burner flame can easily be added and withdrawn to control the heating rate but maintain boiling. Maintain a distillation rate of approximately one-drop every 3-5 s.

6. Add water to the 250-mL round bottom flask as needed to keep the water level at the mark. Stop the distillation when approximately 30 mL of distillate has been collected.

Part 2. Extracting the Clove Oil

**Caution:** Dichloromethane is a strong irritant. This portion of the experiment must be done in a fume hood. Clove oil (eugenol) is irritating. Prevent eye, skin, and clothing contact.

1. Allow the receiver to cool to room temperature. Transfer the distillate from the receiver into a 125-mL separatory funnel using a pipet. Add 10 mL of saturated NaCl solution.

2. Dismantle the condenser from the distilling head. Significant amounts of clove oil will adhere to the condenser and the sides and neck of the receiving flask. Using a dropper pipet, carefully rinse the condenser and the inside neck of the receiving flask with 5 mL of dichloromethane. Swirl the flask gently to dissolve the remaining clove oil. Transfer this dichloromethane to the distillate in the separatory funnel using a pipet.

3. Cap the separatory funnel and gently swirl the contents for several seconds. Vent the separatory funnel frequently. After the pressure has been vented, shake the contents vigorously to thoroughly mix the two layers.

4. Swirl the separatory funnel. At the same time, gently tap the outside of the separatory funnel with your index finger to force into the bottom layer any droplets of dichloromethane that are adhering to the sides of the funnel.

5. Allow the layers to separate. Remove the stopper and drain the lower dichloromethane layer into a 50-mL Erlenmeyer flask, making certain that none of the aqueous layer is transferred to the flask.

USE A 250 mL ROUND BOTTOM FLASK FOR A BOILING POT AND A 100 mL boiling flask for a receiving flask. Stopper the three way connector instead of using a thermometer.
6. Rinse the condenser and the receiver with a second 5-mL portion of dichloromethane. Transfer the rinsing to the separatory funnel. Repeat the extraction of the aqueous layer.

7. Drain the second dichloromethane extract from the separatory funnel and combine it with the first one in the 50-mL Erlenmeyer flask. Repeat the rinsing and extraction process with a third 5-mL portion of dichloromethane. Combine the third extract in the same 50-mL Erlenmeyer flask.

Part 3. "Drying the Dichloromethane layer"

*Caution:* Anhydrous sodium sulfate (Na$_2$SO$_4$) is irritating and hygroscopic. Do not inhale and ingest this compound. **This portion of the lab must be done in the fume hood.**

1. Add approximately 0.5 g increments of anhydrous Na$_2$SO$_4$ to the Erlenmeyer flask containing the dichloromethane extracts until the anhydrous Na$_2$SO$_4$ no longer clumps. Parafilm the flask. Allow the extracts to dry for 5 min.

2. Weigh a clean, dry 50-mL round boiling flask to the nearest 0.001 g and record the mass. Using a pipet, transfer the dried dichloromethane into the preweighed boiling flask, making certain that no Na$_2$SO$_4$ is transferred with the solution. Use three additional 2-mL portions of dichloromethane to rinse the Na$_2$SO$_4$ and ensure complete transfer of the clove oil to the boiling flask.

3. Add a (1-2, no more) boiling chip. Label the beaker with your name using a permanent marker. Cover the boiling flask with parafilm and give the flask to your instructor to evaporate using a rotary evaporator. When all of the dichloromethane has evaporated, cool the boiling flask. Weigh it to the nearest 0.001 g and record the mass. Subtract the mass of the empty flask to obtain the mass of the clove oil.

Part 4. Analyzing Clove Oil by flammable Chemical Tests

*Caution:* Clove oil (eugenol) is irritating. Methanol is flammable and toxic. Keep away from flames or other heat sources. Prevent eye, skin, and clothing contact.

1. Dissolve the clove oil in 10 mL of methanol*.

2. Obtain four test tubes and label them 1-4. Label tubes 1 and 2 "control". Add 1 mL of methanol to each of the four tubes.

3. Using a Pasteur pipet, add 5 drops of the methanol-clove oil solution prepared in step 1 to test tube 3 and 10 drops into test tube 4. Gently swirl each tube.

**Testing with Potassium Permanganate**

*Caution:* Potassium permanganate (KMnO$_4$) is corrosive and oxidizing. Prevent eye, skin, and clothing contact. Do not inhale or ingest KMnO$_4$.

4. Using a Pasteur pipet, add three drops of 0.05M KMnO$_4$ to test tubes 1 and 3 and record your observations.

**Testing with Iron (III) Chloride**

*Caution:* Iron(III) chloride (FeCl$_3$) is toxic and corrosive. Prevent eye, skin, and clothing contact. Do not inhale or ingest FeCl$_3$.

5. Using a Pasteur pipet, add two drops of 1% FeCl$_3$ solution to test tubes 2 and 4. Record your observations.
Post-Laboratory Questions

Complete the following reactions, giving the correct structure for each organic product.

(a) eugenol + \text{KMnO}_4 \rightarrow
(b) eugenol + \text{FeCl}_3 \rightarrow
Simple esters tend to have pleasant odors. In most cases the characteristic flavors and fragrances of flowers and fruits are due to the compounds with the ester functional group. An exception is the essential oils. Although the odors and flavors can be due to single esters, more often the aroma is due to a complex mixture in which a single ester predominates. Some esters and their odors are listed Table 1 below.

**TABLE 1**

<table>
<thead>
<tr>
<th>Pure Compound</th>
<th>Odor</th>
<th>Pure Compound</th>
<th>Odor</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-propyl acetate</td>
<td>pear</td>
<td>isobutyl propanoate</td>
<td>rum</td>
</tr>
<tr>
<td>isopentyl acetate</td>
<td>banana</td>
<td>n-octyl acetate</td>
<td>orange</td>
</tr>
<tr>
<td>methyl salicylate</td>
<td>wintergreen</td>
<td>methyl anthranilate</td>
<td>grape</td>
</tr>
<tr>
<td>benzyl acetate</td>
<td>peach</td>
<td>isobutyl propanoate</td>
<td>floral</td>
</tr>
<tr>
<td>ethyl propanoate</td>
<td>fruity</td>
<td>propyl propanoate</td>
<td>pineapple</td>
</tr>
<tr>
<td>butyl benzoate</td>
<td>balsamic</td>
<td>methyl benzoate</td>
<td>prune</td>
</tr>
<tr>
<td>ethyl benzoate</td>
<td>fruit</td>
<td>geranyl acetate</td>
<td>rose</td>
</tr>
</tbody>
</table>

Food and beverage manufacturers often use esters as additives to enhance flavor or odor of a dessert or beverage. Many times such flavors or odors do not have a natural basis. The “juicy fruit” flavor, isopentyl acetate is an example. Instant pudding that has the flavor of rum does not contain rum in its ingredients; this flavor is duplicated by the combination of ethyl formate and isobutyl propanoate. The natural flavor and odor are not exactly duplicated, but give the essence of the flavor it tries to duplicate.

A single compound is rarely used in good-quality imitation flavorings. A formula for imitation pineapple flavor is listed in Table 2 below.

**TABLE 2**

<table>
<thead>
<tr>
<th>Pure Compound</th>
<th>%</th>
<th>Essential Oils</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ally caproate</td>
<td>5</td>
<td>Oil of sweet birch</td>
<td>1</td>
</tr>
<tr>
<td>Isoamyl acetate</td>
<td>3</td>
<td>Oil of spruce</td>
<td>2</td>
</tr>
<tr>
<td>Isoamyl isovalerate</td>
<td>3</td>
<td>Balsam Peru</td>
<td>4</td>
</tr>
<tr>
<td>Ethyl Acetate</td>
<td>15</td>
<td>Volatile mustard oil</td>
<td>1</td>
</tr>
<tr>
<td>Ethyl Butyrate</td>
<td>22</td>
<td>Oil cognac</td>
<td>5</td>
</tr>
<tr>
<td>Terpinyl propionate</td>
<td>3</td>
<td>Conc. orange oil</td>
<td>4</td>
</tr>
<tr>
<td>Ethyl crotonate</td>
<td>5</td>
<td>Distilled oil of lime</td>
<td>2</td>
</tr>
<tr>
<td>Caproic acid</td>
<td>8</td>
<td></td>
<td>19</td>
</tr>
<tr>
<td>Butyric acid</td>
<td>12</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acetic Acid</td>
<td>5</td>
<td></td>
<td>81</td>
</tr>
</tbody>
</table>

Although the fruity tastes and odor of esters are pleasant, rarely are they used in perfumes and scents applied to the body. The ester group is not as stable to perspiration as essential oils. Esters on contact with sweat will undergo hydrolysis, giving organic acids. These acids, unlike their precursor esters, generally do not have a pleasant odor.
Esters have the general formula, $R\text{COOR}$. The reaction below depicts the Fischer esterification reaction for the synthesis of an ester. A Fischer esterification is the acid-catalyzed synthesis of an ester from an alcohol and a carboxylic acid. In this reaction, the acid and alcohol reactants are in equilibrium with the ester product.

$$
\begin{align*}
\text{Acid} & \quad + \quad \text{Alcohol} & \quad \overset{\text{H}_2\text{SO}_4}{\rightleftharpoons} & \quad \text{Ester} \\
\quad & \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad \quad
2. Weigh a clean and dry 10 mL graduated cylinder and record its mass. In the hood, place approximately 5.0 mL of isopentyl alcohol into the graduated cylinder and reweigh it. Calculate the mass of the alcohol (mass of alcohol and graduated cylinder – graduated cylinder).

3. Transfer the isopentyl alcohol to the 25 or 50 mL boiling flask. Without cleaning or washing the graduated cylinder, add approximately 7.0 mL of acetic acid (MW = 60 g/mole and density = 1.06 g/mL) and add the acid to the alcohol already in the boiling flask.

4. Using a calibrated dropper pipet, add 1 mL of concentrated sulfuric acid to the alcohol/carboxylic acid mixture and mix. Add one boiling chip to the reaction mixture.

Safety caution: Both Acetic Acid and Sulfuric Acid are very corrosive. If spilled on the skin, rinse immediately with cold water for 15 minutes. Neutralize acid spills first with baking soda before wiping.

5. Assemble the reflux apparatus, greasing the joints with vacuum grease. Start the water circulating and bring the mixture to boil. Heat, using a hot plate, under reflux for 60 minutes. While refluxing calculate the theoretical yield of the reaction using the mass of the isopentyl alcohol. Remember the acetic acid is used in excess.

Part B Extraction

1. In the hood carefully clamp a 125 mL separatory funnel to a ring stand using a two prong clamp. Be sure that the stopcock is closed. Disassemble the reflux apparatus and transfer the reaction mixture (use a dropper, don’t pour) to a 125 mL separatory cylinder. Avoid transferring the boiling chip. Grease the stopper lightly.

2. Add 10 mL of distilled water; stopper the funnel and mix, venting frequently. Allow the layers to separate and unstopper the funnel.

3. Drain the lower aqueous layer into a beaker. Extract the organic layer with 5 mL of 5% sodium bicarbonate. Again drain the lower aqueous layer.

4. Extract the organic layer with another 5 mL sodium bicarbonate and drain the lower aqueous layer.
Part C Drying
1. Transfer the crude ester to a clean, dry 50 mL Erlenmeyer flask and add approximately 1.0 gram of anhydrous sodium sulfate. Mix.
2. Cover the flask with parafilm and let sit for 10 minutes while you prepare the distillation apparatus.

3. If the mixture does not appear "dry" - the drying agent is clumping and/or the solution is cloudy - add another 0.5 gram of anhydrous sodium sulfate and mix. Let it sit for another 10 minutes.

Part D Distillation
1. Assemble a distillation apparatus using a 50 mL boiling flask as the distillation flask.
2. Preweigh a clean and dry 50 mL Erlenmeyer flask to function as the collection flask.
3. Immerse the collection flask in a beaker of ice to ensure condensation and reduce the odor.
4. Collect the distillate between 120-142°C, isopentyl acetate's boiling point. Stop when the temperature of the distillate falls below 120 or above 142 °C.
5. Weigh the product and calculate the percentage yield of the ester.

Post Laboratory Questions
1. One method of favoring the formation of an ester is to add excess acetic acid. Suggest another method, involving the right hand side of the equation that will favor the formation of the ester.
2. Why is the mixture extracted with sodium bicarbonate? Give an equation and explain its relevance.
Introduction

A soap is the sodium or potassium salt of a long-chain fatty acid. The fatty acid usually contains 12 to 18 carbon atoms. The source of the fatty acids is either from animal fats or vegetables which are esters of carboxylic acids. They have a high molecular weight and contained alcohol and glycerol. Chemically, these fats and oils are called triglycerides.

Solid soaps usually consist of sodium salts of fatty acids, whereas liquid soaps consist of the potassium salts of fatty acids. A soap such as sodium stearate consists of a nonpolar end (the hydrocarbon chain of the fatty acid) and a polar end (the ionic carboxylate).

Because "like dissolves like" the nonpolar end (hydrophobic or water-hating part) of the soap molecule can dissolve the greasy dirt, and the polar or ionic end (hydrophilic or water-loving part) of the molecule is attracted to water molecules. Therefore, the dirt from the surface being cleaned will be pulled away and suspended in water. Thus soap acts as an emulsifying agent, a substance used to disperse one liquid (oil molecules) in the form of finely suspended particles or droplets in another liquid (water molecules).

Treatment of fats or oils with strong bases such as lye (NaOH) or potash (KOH) causes them to undergo hydrolysis (saponification) to form glycerol and the salt of a long-chain fatty acid (soap).

Natural fatty acids are rarely a single type in any given fats or oil. In fact, a single tryglyceride molecule in a fat may contain three different acid residues (R₁COOH, R₂COOH, R₃COOH), and not every triglyceride in the substance will be identical.

The fats and oils that are most common in soap preparations are lard and tallow from animal sources and coconut, palm and olive oils from the vegetable sources.
length of the hydrocarbon chain and number of double bonds in the carboxylic acid salt of the carboxylic acid portion of the fat or oil determine the properties of the resulting salt. For example, the salt of a saturated long chain acid make a harder, more insoluble soap. Chain length also affects solubility.

Tallow is the principal fatty material used in making soap. The solid fats of cattle are melted with steam and tallow layer formed at the top is removed. Soap makers usually blend tallow with coconut oil and saponify this mixture. The resulting soap contains mainly the salts of palmitic, stearic and oleic acids from the tallow and the salts of lauric and myristic acids from coconut oil. The coconut oil is added to produce a softer, more soluble soap. Lard differs from tallow in that lard contains more oleic acids.

Pure coconut oil yields a soap that is very soluble in water. It is so soft that it will lather even in salt water. Palm oil contains mainly two acids, palmitic and oleic acid, in equal amount. Saponification of this oil yields a soap that is an important constituent of toilet soaps. Olive oil contains mainly oleic acid. It is used to prepare Castille soap.

<table>
<thead>
<tr>
<th>Acid</th>
<th>Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Palmitic acid</td>
<td>CH₃(CH₂)₁₄COOH</td>
</tr>
<tr>
<td>Stearic acid</td>
<td>CH₃(CH₂)₁₆COOH</td>
</tr>
<tr>
<td>Oleic acid</td>
<td>CH₃(CH₂)₇CH=CH(CH₂)₇COOH</td>
</tr>
<tr>
<td>Lauric acid</td>
<td>CH₃(CH₂)₁₀COOH</td>
</tr>
<tr>
<td>Myristic acid</td>
<td>CH₃(CH₂)₁₂COOH</td>
</tr>
</tbody>
</table>

Table 1
Structure of Acids Commonly Found in Soap

Toilette soaps generally have been carefully washed free of any alkali remaining from saponification. As much glycerol as possible is usually left in the soap and perfumes and medicinal agents are sometimes added. Soft soaps are made by using potassium hydroxide, yielding potassium salts. They are used in shaving creams and liquid soaps.

Because soaps are salts of strong bases and weak acids, they should be weakly alkaline in aqueous solution. However, a soap with free alkali can cause damage to skin, silk, or wool. Therefore, a test for basicity of the soap is quite important.

Soap has been largely replaced by synthetic detergents during the last two decades, because soap has two serious drawbacks. One is that soap becomes ineffective in hard water; this is water that contains appreciable amounts of Ca²⁺ or Mg²⁺ salts.

\[
2C_{17}H_{35}COO^-Na^+ + M^{2+} \rightarrow [C_{17}H_{35}COO^-]_2 M^{2+} \downarrow + 2Na^+
\]

\[ M = (Ca^{2+} \text{ or } Mg^{2+}) \]

The other is that, in an acidic solution, soap is converted to free fatty acid and therefore loses its cleansing action.
Procedure
Preparation of a soap
1. Measure 23 mL or 23 grams of a vegetable oil into a 250-mL Erlenmeyer flask.
2. Add 10 mL of ethyl alcohol (to act as a solvent) and 20 mL of 25% sodium hydroxide solution (25% NaOH). While stirring the mixture constantly with a glass rod, the flask with its contents is heated gently in a boiling water bath.
3. A 600-mL beaker containing about 200 mL of tap water and a two boiling chips can serve as a water bath (Fig. 1).

Caution: Alcohol is flammable!

4. After being heated for about 20 min, the odor of alcohol will disappear, indicating the completion of the reaction. A pasty mass containing a mixture of the soap, glycerol, and excess sodium hydroxide is obtained.
5. Use an ice-water bath to cool the flask with its contents. To precipitate or "salt out" the soap, add 150 mL of a saturated sodium chloride solution to the soap mixture while stirring vigorously. This process increases the density of the aqueous solution; therefore, soap will float out from the aqueous solution.
6. Filter the precipitated soap with 4 ply cheese cloth and wash it with 10
mL of ice cold water three times. Observe the appearance of your soap and record your observation on the Report Sheet.

**Properties of a soap**

*Emulsifying Properties.*
1. Shake 5 drops of mineral oil in a test tube containing 5 mL of water. A temporary emulsion of tiny oil droplets in water will be formed.
2. Repeat the same test, but this time add a small piece of the soap you have prepared before shaking. Allow both solutions to stand for a short time.
3. Compare the appearance and the relative stabilities of the two emulsions.
4. Record your observations on the Report Sheet.

*Hard Water Reactions.*
1. Place about one-third spatula full of the soap you have prepared in a 50-mL beaker containing 25 mL of water.
2. Warm the beaker with its contents to dissolve the soap.
3. Pour 5 mL of the soap solution into each of 5 test tubes (nos. 1, 2, 3, 4, and 5).
4. Test no. 1 with 2 drops of a 5% solution of calcium chloride (5% CaCl$_2$), no. 2 with 2 drops of a 5% solution of magnesium chloride (5% MgCl$_2$), no. 3 with 2 drops of a 5% solution of iron(III) chloride (5% FeCl$_3$), and no. 4 with tap water. The no. 5 tube will be used for a basicity test, which will be performed later.
5. Record your observations on the Report Sheet.

*Alkalinity (Basicity).*
1. Test soap solution no. 5 with a wide-range pH paper.
2. What is the approximate pH of your soap solution? Record your answer on the Report Sheet.
Turn in completed data sheet before leaving. No lab report for this experiment.
Name
Partner:

REPORT SHEET

Preparation

Describe the appearance of your soap.

Hard Water Reaction

No. 1 +
CaCl_2

No. 2 +
MgCl_2

No. 3 +
FeCl_3

No. 4 + tap water

Alkalinity

pH of your soap solution (no. 5)
POST-LAB QUESTIONS

1. When you made soap, first you dissolved vegetable oil in ethanol. What happened to the ethanol during the reaction?

2. What are the two main disadvantages of soaps versus detergent?

3. Soaps that have a pH above 8.0 tend to irritate some sensitive skins. Was your soap good enough to compete with commercial preparations.