SANTA CLARA UNIVERSITY | SUMMER TERM 2008
ORGANIC CHEMISTRY I | CHEMISTRY 31

LABORATORY SYLLABUS

INSTRUCTOR Drs. Mary Lee and Paul Gendler

TIMES Monday and Wednesday: 8 am-12:30 pm; Tuesday and Thursday: 8 am-12:30 pm
Tuesday and Thursday: 4:30-9:00 pm

- Labs begin on **June 16** and are held in DS 126
- Lab lectures are given during the first 15-20 minutes of the laboratory period.

MATERIALS

INTRODUCTION TO ORGANIC LABORATORY TECHNIQUES, A MICROSCALE APPROACH
by Pavia, Lampman, Kriz and Engel, 4th Edition, 2007

- A bound laboratory notebook
- Safety goggles

LABORATORY GUIDELINES

1) During the summer of 1994, the laboratories in Daly Science 200 were modernized and redesigned with safety as the primary concern. The main improvement in these labs has been increasing the number of hoods so that each student has a workstation in a hood, two students per hood. This is a significant safety advancement as many organic compounds are volatile and using a hood minimizes your exposure to fumes. Therefore, **virtually 100% of your chemical work should be accomplished in a fume hood**.

2) To do organic chemistry safely, you should treat all chemicals with respect; gloves should be worn unless otherwise noted by the instructor. **Safety goggles must be worn at all times**.

3) Because we are limited in space for each lab section, you may only attend your assigned laboratory time. If you miss your lab due to an excused absence, you may make-up the lab by attending another section only with the consent of **both** lab instructors.

4) Care of the organic laboratory is important because 160 students share this organic facility each year and we in the chemistry department would like to maintain this facility. It is important that you **clean your work area at the end of each lab period** as well as help with keeping the balances, instrument room and other general use areas clean. Please **inform the instructor of any spills and make sure you clean up the spill completely**.

COMMENTS

Most students enjoy organic lab; it can be challenging, stimulating and rewarding. This is partly due to the relatively unstructured nature of the lab. After the first few technique experiments, you can work at your own pace and follow whatever sequence of steps seems best for your particular project. As a result, you are left to your own initiative to a much greater extent than in most labs you have taken. If you plan your work in advance and make a real effort to understand what you are doing, then even unexpected problems that always arise can be stimulating challenges.

NOTEBOOK

Your lab notebook is the primary record of your lab work. Please look carefully at the guidelines on page 12 before making entries in your notebook. Remember that a complete, well-organized notebook is critical for someone to be able to repeat your work and serves as the primary basis for your grade in the laboratory.

SAFETY

It is important to carefully read the safety section in the lab text, pp. 5-21, and the back of your laboratory equipment check-in card before signing it. Critical points are highlighted below.

1) **SAFETY GOGGLES MUST BE WORN IN THE LABORATORY AT ALL TIMES!**
2) Bare waists, legs and shoulders and open-toed shoes are not permitted in laboratory.
3) Most organic solvents are flammable and should never be heated with an open flame. Hot plates or heating mantles are available for this purpose. The instructor's permission is required to use an open flame in the laboratory. Some solvents such as diethyl ether, t-butyl methyl ether and methanol have flash points so low that they can be ignited by the surface of a hot plate.
4) Be sure to handle organic chemicals carefully as many are toxic if absorbed through the skin or inhaled. GLOVES MUST BE WORN FOR ALL EXPERIMENTS. Disposable gloves are provided in the laboratory. Change gloves when necessary. To avoid chemical contamination of the chemistry building, do not used gloved hands to handle objects outside the laboratory.
5) Substances with noxious or toxic vapors must be handled only in the fume hoods. In general, you should perform all work in the fume hoods.
6) Neatness in carrying out lab work is related to safety. It is important that each student help keep the lab clean and organized. Allow enough time for clean-up when planning your lab activities.
7) Report all accidents and spills to the instructor.
8) Chemicals spilled on the skin must be washed of immediately with water.
9) Know the locations of the safety showers, eyewash stations and fire extinguishers

WASTE DISPOSAL

One of the most important practices in lab is the proper disposal of chemical wastes. The only substance allowed to go down the drain is uncontaminated water. The general rule is that **nothing should be poured into any sink or placed in the garbage cans**. There are containers in the lab for the various kinds of waste materials generated: aqueous waste, basic aqueous waste, acidic aqueous waste, solid organic waste, organic solvent waste (non-halogenated), halogenated organic waste, and contaminated glass. Be absolutely positive that you are putting the proper materials in the containers. Remember that anything with WATER in it is an aqueous waste and must go in one of the containers so labeled. It is also extremely important that you enter into the appropriate logbook the identity and amount of each substance added to each container. **If you are unsure of which container to use, ask one of the lab instructors**.

ACADEMIC INTEGRITY

You are expected to uphold the university policy on academic integrity. In the context of the laboratory, you may help each other understand and complete various procedures. However, **all work recorded in your notebook must be your own**. If your instructor ever tells you to include data from a fellow student, that data should be clearly referenced. Giving or receiving unauthorized aid in any form can result in course failure. See your instructor if further clarification is needed.

FORMAT

Roughly the first half of this quarter's laboratory will be devoted to learning important laboratory techniques. During the second half these techniques will be applied to separating, purifying and identifying two components in an unknown mixture. The overall scheme of the lab and some of the specific procedures to be followed are contained in this syllabus; other procedures can be found in the lab text. In addition, report forms, worksheets and this syllabus can be found on the Chem 31L ERes page: http://eres.scu.edu, (password is: lab)

Lab lectures describing important details of each experiment will be held during the regular laboratory time at the beginning of the lab. It is therefore very important that you are on time to lab so that you get the vital information to complete the experiment. It is also very important that you come prepared for lab and know exactly what you are to accomplish that day in lab. Because you may not attend other laboratory sections it is important that you make the most of your time in lab.

Laboratory Schedule:

Period No.	Lab Lecture	Experiment	Reading in Text
1	Simple and Fractional Distillation	Distillation	pp. 703-714, 715-730
	Gas Chromatography	Gas Chromatography (GC) Infrared Spectroscopy (IR)	pp. 797-817 pp. 833-847
2	Recrystallization	Recrystallization	pp. 647-668 Expt. 3, pp. 21-28
	Thin Layer Chromatography (TLC)	Thin Layer Chromatography	pp. 777-791; Expt. 5, pp. 46-48
3	Bromination of an Alkene	Bromination of Cinnamic Acid	Supplemental Experiment Procedure*
		Complete GC, IR Recrystallization, TLC	
4	Dehydration of an Alcohol	Dehydration of 4-methyl cyclohexanol, Exp. 25A	pp. 213-214
5	Extraction, Distillation and Recrystallization	Unknown Separation and Purification	pp. 669-688
6		Unknown Identification Functional Group: IR Literature Search: m.p., b,p.	 pp. 847-866 pp. 958-970, App. 1

*Supplemental Experiment Procedures can be found in DS126 or on the Chem 31L ERes page

Data, observations, conclusions and results for all experimental work should be included in your notebook. A report form is required for the unknown. This form is important for our evaluation of your work on the unknown, so fill it out carefully, in ink, and submit it with your notebook. **Notebooks are due on your last scheduled laboratory period for the quarter.** If you have any questions about when to turn in your notebook, please see your instructor. Please note that a grade can not be issued for the course unless you have completed the laboratory and turned in a notebook, so be sure to turn in your notebook on time to your laboratory instructor.

THE EXPERIMENTS

1. SEPARATION OF A BINARY MIXTURE BY SIMPLE AND FRACTIONAL DISTILLATION

Distillation is a common method for the separation of mixtures of liquids and the purification of liquids. In this experiment, we purposefully mix two liquids with very different boiling points and then practice separating them first by a simple distillation and second by a fractional distillation. The efficiency of each type of distillation will be compared through quantitative gas chromatography (GC) and a graph of the results. The identity of the liquids will be verified by infrared (IR) spectroscopy.

SIMPLE DISTILLATION

Read Technique 14, pp. 703-714, before starting this experiment.

Set up a simple distillation apparatus as shown on page 13 of the syllabus, using a 10 mL round-bottom flask containing a mixture of 3 mL hexane and 3 mL toluene. The arrangement using the air condenser should work fine. Equip the apparatus with a thermometer using the vacuum adapter to hold the thermometer in place. Make sure the thermometer bulb is positioned in the lower neck of the Hickman still as shown in Figure 14.8 on page 711. Be sure to add a boiling stone to the distilling flask. The mixture is heated by nesting the round bottom flask in the large well of an aluminum block supported by a stirring hotplate. The reservoir of the Hickman still holds a little over 1 mL of distillate. As the reservoir approaches 3/4 full, record the temperature, uncap the side port of the Hickman head and quickly remove the contents of the reservoir with a pipette. Reserve the liquid in a labeled vial. Be sure to recap the side port after removing a fraction! Continue collecting fractions in this manner until very little liquid remains in the round bottom flask. You will need to decide which fractions should be combined and used for gas chromatographic and infrared analysis. Usually, students save the first two and last two fractions. Make sure your samples are be stored in tightly stoppered, labeled containers and placed in the refrigerator. Be sure to read the sections in the syllabus on gas chromatographic and infrared analysis; the lab instructor will assist you in these two procedures. Prepare a graph of temperature versus fraction number.

FRACTIONAL DISTILLATION

Read Technique 15, pp. 715-724 before starting this experiment.

Set up the fractional distillation apparatus as shown on page 13 of this syllabus. Be sure to use a thermometer as in the simple distillation. Pack the fractionating column (the air condenser is used for this purpose) loosely with stainless steel sponge as shown in the figure. Distill a fresh 6 mL mixture of hexane and toluene in a fashion similar to that used for the simple distillation. Again you will need to decide which fractions should be combined and used for gas chromatographic and infrared analysis. Make sure your samples are be stored in tightly stoppered, labeled containers and placed in the refrigerator. Remember these analyses should be carried out as soon as possible after the samples have been collected. Graph temperature versus fraction number and compare with the plot for simple distillation.

2. GAS CHROMATOGRAPHY AND INFRARED SPECTROSCOPY

Gas chromatography (GC) is a method to separate very small samples of liquids. Importantly, it also allows one to quantify the amount of each liquid in the sample. As a result, we can use GC to determine the effectiveness of the separation achieved in our distillations.

The use of the gas chromatograph will be demonstrated, and the mole percent composition of the fractions saved for GC analysis from each of the two distillations carried out above will be determined. Be sure to read Technique 15, pp. 797-817 before starting this experiment.

Make sure that the GC is turned on and running properly and that the chart recorder is on as well. Contact your instructor if you are unsure.

Now inject 2.5-3 μL of each of the four fractions saved from your two distillations, waiting until the peaks for hexane and toluene appear for each GC trace on the recorder. Compare the four GC traces you have obtained with the hexane and toluene standard sample GC's obtained by your instructor. This will allow you to confirm which peak is associated with each compound, hexane or toluene.

The areas under the peaks should be measured manually as described on p. 811 and the percent hexane and toluene determined for each of the fractions. The relative efficiency of simple versus fractional distillation should be determined. All relevant data, calculations and conclusions must be clearly recorded in your notebook.

[The GC should be set up so that the column packing is 20% SE-30; oven temperature is 130-140°; helium gas flow of 30 mL/min; detector current of 100 milliamperes; and a chart speed of 0.5 in/min. You may be using new strip chart recorders, so other pertinent settings may be noted by your lab instructor.]

Infrared spectroscopy (IR) is a method for the identification of compounds (most often for the functional groups present in a sample). After learning how to use the IR spectrometers, acquire IR spectra for each of your saved fractions. Compare these spectra to the known spectra for pure hexane and toluene. Consider what conclusions are possible regarding identity and purity.

3. RECRYSTALLIZATION OF IMPURE SOLIDS

Recrystallization is a common method for the purification of solids. In this experiment, we will learn the basics of recrystallization by purifying a sample of acetanilide according to an optimized procedure provided in the syllabus. Subsequently, you will optimize your own procedure for the recrystallization of an impure sample of fluorene. The success of the purifications will be demonstrated by taking melting point ranges and running thin layer chromatography (TLC, in the next experiment) for both the original and the purified samples. Be sure to read Techniques 11 (pp. 650-659) and 8 (pp. 621-622) before performing any recrystallizations.

Semi-Microscale Recrystallization of Acetanilide

Obtain a small sample of impure acetanilide in a melting point capillary to determine the melting point range. This capillary may be saved and its sample's melting point determined side-by-side with that of recrystallized acetanilide. Weigh out a 150 mg sample of impure acetanilide and place it in a 10 mL Erlenmeyer flask. (Note – the sample need not be exactly 150 mg, but you should enter the exact amount you use in your notebook. For many of these technique experiments, the amounts we use are not critical, but accurately reporting the amounts is always critical.) Add 4.0 mL of water, clamp the flask to a ring stand and heat to boiling using the proper well of the aluminum block heating setup. Heat the solution until all the solid dissolves. In this case we have determined that 4.0 ml of water (the solvent) should be enough to dissolve 150 mg of sample (the solute), but ordinarily one does not know how much solvent will be necessary. If your sample is not fully dissolved, add additional small amounts of hot water until complete dissolution is reached. If any insoluble solids remain, they must be removed by filtration (ask your instructor).

Allow the flask to cool to room temperature. Then, place the Erlenmeyer flask in a small beaker of ice water to complete the crystallization.

Collect the crystals by vacuum filtration using the Hirsch funnel. With the vacuum still on, rinse the crystals with three 0.5 mL portions of ice-cold water. Use a clean micro-spatula to press the crystals as dry as possible on the funnel. Dry and weigh the crystals and determine percent recovery. You may want to wait until the next laboratory to weigh the crystals and determine their melting point (Technique 9 pp. 627-633). Save your sample of the recrystallized acetanilide for the thin-layer chromatography experiment.

Note: It can be especially difficult to remove the last traces of solvent from crystallized material, particularly if water is the solvent. Small traces of solvent can lower melting ranges substantially, so dry a sample for a melting point by working a small sample of the solid on piece of dry filter paper until the sample is a fine powder. You may also place your sample on a piece of filter paper (well-labeled) under the heat lamp. Remember that the temperature under the lamp can melt some solids, so exercise caution when using the heat lamp with unknown samples.

Semi-Microscale Recrystallization of Fluorene

Carry out the recrystallization as described in Experiment 3C (pp. 27-28) in the laboratory textbook. The eventual procedure will be identical to that used for acetanilide above, but initially you will have to determine which solvent will work best. Additionally, you will have to determine experimentally the best proportion of solvent to solute for the recrystallization. Make sure to include any observations or conclusions about the process in your notebook entries.

4. THIN-LAYER CHROMATOGRAPHY

Analysis of Recrystallized Samples

The impure and recrystallized samples of acetanilide and fluorene from the previous experiment will be analyzed by thin layer chromatography (TLC). The purified samples should show only one spot whereas the impure samples may show several. To understand this expectation, make sure you read Technique 20, 782-787, before starting this experiment. Plastic-backed TLC plates of the proper size will be available for your use in the experiment.

Place approximately 4-5 mL of developing solvent in the screw cap jar from your locker. The depth of the solvent should be approximately 0.5 cm. The jar should be capped and allowed to sit while you prepare the TLC plates.

The choice of developing solvent is one of the most important factors for successful separations by TLC. Generally, the choice is made by experimenting with different solvents or combinations of solvents until one is found that separates all the components of the mixture to be analyzed. A good general rule when testing the purity of a compound is to use a solvent in which the test compound has an Rf of 0.3 – 0.5. In this case, start by trying a 2:1 mixture of ethyl acetate:hexane as the developing solvent for the acetanilide samples and 2:1 hexane:methylene chloride as the developing solvent for the fluorene samples.

Obtain a TLC plate and spot the plate with solutions of the impure and recrystallized acetanilide samples as described on pp. 782-783. Prepare another plate in similar fashion with the fluorene samples. Remember that the spotting solution is prepared using a volatile solvent such as methylene chloride or acetone. Unlike the developing solvent, the choice of spotting solvent is not very critical; the criteria are simply that it adequately dissolves the sample and that it evaporates quickly.

After the solvent has evaporated from the spotted plate, develop it in the screw-cap jar developing chamber (Read pp. 784-785). Be sure to stop development <u>before</u> the solvent reaches the end of the plate! Observe the spots by using a UV light and also by using iodine as the visualization reagent. Calculate the Rf value for the spots detected as described on pp. 787-788.

Monitoring of a Reaction by TLC

One of the most common uses of TLC is to monitor reactions in the laboratory to assess their completion – in other words, to tell when a reaction is "done". Follow the procedure given in Experiment 5C (pp. 46-47) in the laboratory textbook but do not work with a partner. (If you wish you may help by serving as timekeepers for one another as suggested in the book, but each person should carry out his/her own reaction.) Omit the optional isolation procedure.

5. DEHYDRATION OF AN ALCOHOL: FORMATION OF 4-METHYLCYCLOHEXENE

Follow the procedure for Experiment 25A, pp. 213-214 for the microscale preparation of 4-methylcyclohexene. Take an IR spectrum of your product, but you do not have to do a more accurate boiling point determination. Fill out the report form for this experiment and answer the questions 1-3 (p. 216) on the back of the form.

6. BROMINATION OF AN ALKENE: SYNTHESIS OF 2,3-DIBROMO-3-PHENYLPROPIONIC ACID

Obtain the procedure for this experiment from the laboratory or on the Chem 31L ERes page at least a day before doing this experiment. When your product is dry, do and IR and the melting point of the crude product. For additional fun, you can do a TLC on your product and the starting cinnamic acid to determine if your reaction was successful. You will have to first determine what eluting solvent should be to obtain TLC that separates the two compounds. This could be somewhat challenging as carboxylic acids tend to “streak” up the TLC plate (meaning they leave a long spot trailing from the baseline). A small amount of ethanol or methanol added to the solvent mixture can solve this problem. Fill out the report form for this experiment and answer the questions at the end of the supplemental procedures and record your answers on the back of the report form.

7. ANALYSIS OF AN UNKNOWN MIXTURE

A solution of two organic compounds dissolved in an organic solvent will be supplied. The experiment involves the separation of the two compounds from each other and from the solvent, the purification of each component, and the identification of each component. Importantly, you will use all of the techniques learned in the previous experiments in order to complete this task.

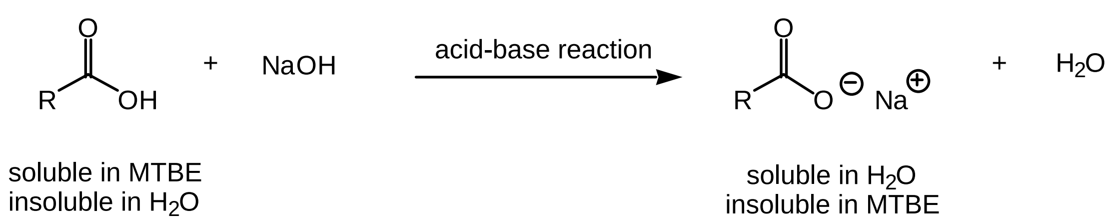
DETAILS

You will be given a test tube containing two organic compounds dissolved in the organic solvent t-butyl methyl ether (t-BuOMe, also called MTBE). One of the organic compounds in the solution will be a neutral liquid: an aldehyde, ketone, alcohol or ester; the other compound will be a solid carboxylic acid. Both compounds are soluble in t-butyl methyl ether but insoluble in water. In order to identify the components of the mixture, you will have to separate the compounds from each other and from the solvent, purify each compound, determine the physical properties of each one and identify each one by comparing its physical and chemical properties with those of a limited selection of known compounds found in the Tables in Appendix 1, pp. 958-970. In addition to being limited to compounds in the Tables, the neutral unknowns will have only a single functional group, exclusive of halogens, nitro groups, aromatic rings and carbon-carbon multiple bonds. The acids will be carboxylic acids.

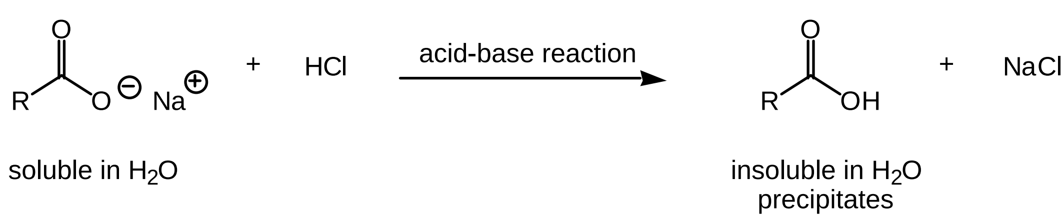
A good general description of the strategy involved can be found in the lab text on pp. 675-677.

SEPARATION OF THE ACIDIC FROM THE NEUTRAL UNKNOWN: EXTRACTION

Although both unknowns are generally soluble in t-butyl methyl ether and insoluble in water, the acidic unknown is converted to a water-soluble, t-butyl methyl ether-insoluble salt by reaction with aqueous base (5% sodium hydroxide). The procedure involves the extraction of the acidic unknown from the t-butyl methyl ether solution using aqueous base. Because t-butyl methyl ether and water are immiscible, separation of the two liquid layers can be carried out mechanically; the lower aqueous layer will contain the acidic unknown in the form of a water-soluble salt, and the upper t-butyl methyl ether layer will contain the neutral liquid. (Densities: H_2O, d = 1.000 g/mL; t-BuOMe, d = 0.740 g/mL).



After the two layers have been separated, the acid is recovered by acidification with HCl after which the carboxylic acid precipitates from water as shown below.



Before starting this part of the experiment, you should read Technique 12, pp. 669-688, especially Section 12.11 (pp. 685-686). **See Extraction Flow-Chart, page 14 in this syllabus.**

To Separate the Acid Unknown from the Neutral Unknown:

Select an unknown, making sure to record the unknown number in your notebook. Each unknown contains approximately 0.5 g of the solid carboxylic acid and 2 mL of the liquid neutral compound dissolved in about 4 mL of t-butyl methyl ether. Place the solution in your centrifuge tube and add 3 mL of 5% NaOH solution (dilute, aqueous base). If you have a solid in your unknown sample vial, wash the solid into the centrifuge tube with an extra 1 mL of 5% NaOH and continue. Cork the tube and shake gently for 30 seconds. Allow the phases to separate completely so that you can see two distinct layers. The aqueous phase should be the bottom layer. (When unsure if a phase is aqueous or organic, add a drop of water and see in which phase it dissolves.) Next, using a Pasteur pipette with a 2 mL rubber bulb attached, squeeze the bulb and insert the pipette into the centrifuge tube so that the tip touches the bottom. With experience, you should be able to judge how much to squeeze the bulb to draw in the desired volume of liquid. Pipette out the lower water layer and store it in a clean 50 mL beaker. Leave the remaining ether layer in the centrifuge tube. The above process is considered one extraction cycle.

The t-butyl methyl ether layer in the centrifuge tube must be extracted twice more using fresh 3 mL portions of aqueous base. After each extraction, combine all aqueous layers in the 50 mL beaker. A final extraction with 3 mL of water should be carried out and the rinse water added to the combined 9 mL of aqueous extract in the 50 mL beaker. The t-butyl methyl ether solution should be placed in a 10 mL Erlenmeyer flask and a few microspatulafuls of anhydrous sodium sulfate (Na_2SO_4) added as a drying agent to remove traces of water. Read over Technique 12, Section 12.9, pp. 680-683, for further details.

The combined aqueous layers (the three 3 mL NaOH portions and the 3 mL water wash) that contain the salt of the organic acid are made acidic by adding enough 5% HCl (dilute, aqueous acid) so that the solution turns blue litmus paper red, or records a pH below 7 using pH paper. The addition of the aqueous acid converts the salt of the unknown to the free acid. Because the organic acid itself (in contrast to its salt) is insoluble in water, it precipitates from the solution. You can actually use the cessation of precipitation as a guide to when acidification is complete. The solid is then isolated by vacuum filtration using the Hirsch funnel.

The liquid unknown may be separated from the t-butyl methyl ether solution by distillation. t-Butyl methyl ether has a boiling point of 55°C; the unknowns all boil above 90°C; consequently, a simple distillation is efficient enough to separate them. When the t-butyl methyl ether solution has dried (wait a minimum of 30 minutes), use a dry Pasteur pipette or a dry filter tip pipette to remove the solution from the drying agent and transfer the solution to a dry 10 mL round-bottom flask. Use simple distillation (but use a water-jacketed condenser rather than an air condenser) to remove the volatile t-butyl methyl ether. The residue in the 10 mL round-bottom flask is the neutral liquid unknown.

PURIFICATION OF THE UNKNOWNS

The liquid unknown may then be purified by simple distillation. Transfer the liquid neutral compound to an appropriate sized conical vial and begin a simple distillation of this residue. You may combine any fractions that boil at the relatively constant temperature range that will be achieved. This temperature is the boiling point of your liquid unknown; do not forget to record it in your lab notebook. The purity of the distilled liquid should then be determined by gas chromatography. You should see only one peak if the liquid is pure.

The solid unknown is to be purified by recrystallization. You will have to find an appropriate solvent (Section 11.5, p. 660). Solvent selection can be very time consuming so here are some tips. Carboxylic acids are rather polar, so for most of them a solvent like methanol, ethanol or water works well. Test your compound in test tubes with these three solvents using very small amounts of material. (Recall the procedure you used to determine the solvent for the fluorene recrystallization.) You may find you need to perform a mixed solvent recrystallization, about which you should ask your instructor. Once your solid is recrystallized and dried, check its purity by performing a TLC (ask your instructor about choices for a developing solvent) and obtain a melting point.

DETERMINATION OF PHYSICAL PROPERTIES

For the liquid unknown, the boiling point was determined during your simple distillation; the only other physical property to be determined at this point is the index of refraction. Read over Technique 24, pp. 827-831. Make sure to correct your value to the temperature used for the literature value (usually 20°C) as described in the text.

For the solid unknown, the only relevant physical property to be determined is the melting point. Proceed as in Technique 9, p.627.

At this point you should fill out an unknown purity check-sheet and submit it to your lab instructor. If your melting or boiling point range is off by more than 5°C, you will be required to recrystallize again or re-distill. Alternatively, you may be able to obtain an accurate boiling point using the microscale boiling point method described in Technique 13.2, p. 695. If your sample is less than 95% pure by GC or shows 2 or more spots on the TLC, you will also be required to re-distill or recrystallize again.

FUNCTIONAL GROUP DETERMINATION

No functional group determination is required for the solid unknown: the solid is a carboxylic acid. For the liquid unknown, however, you need to determine whether it is an aldehyde, ketone, alcohol or ester. The functional group present will be determined by *infrared spectroscopy*.

Infrared Spectroscopy

Very simply, we'll use IR in two ways: to verify the presence of a particular functional group and to verify the identity of the unknown. The presence of a particular functional group can be determined by comparing the absorption peaks in the IR spectrum of your unknown with those absorptions normally observed for a particular functional group. The usual absorptions for each functional group are described in Technique 25. To identify the compound, you should attempt to interpret the major peaks as we have in class using an IR correlation table (p. 851). Also, the unknown spectrum can be compared with that of the suspected compound in the Aldrich reference books.

Directions for running IR spectra can be found in Technique 25.3, pp. 835-836. Concentrate on the use of simple salt plates using "neat" (pure) liquid samples. Be sure to always clean the plates with hexane after each use. **NEVER CLEAN NaCl PLATES WITH WATER; THEY DISSOLVE!** For solids, we have recently purchased a new IR spectrometer that allows for the spectrum to be run directly on the solid unknown (rather than making a KBr pellet as described in your lab book). Your instructor can show you how to obtain a spectrum of your solid using this spectrometer. Check with your instructor after you have run the solid IR, to determine the quality of this spectrum as some peaks look different in a solid-phase IR spectrum.

LITERATURE SEARCH

At this stage in your analysis of the unknowns you are ready to compile a list of possible identities for each unknown. Fortunately for you, all the unknowns given to you will appear in one of the Tables in Appendix 1, pp. 958-970. There are separate tables for each functional group, and the compounds within each table are listed by increasing boiling points and increasing melting points. You should consider all compounds having b.p.'s or m.p.'s within 5°C of your experimentally determined value as possibilities. With the aid of the lab instructor or assistant, you should draw out the structures of the possible compounds. Consider these structures seriously to determine how you may differentiate between them and look each up in reference books to find out how their physical properties may differ. With the limited techniques available at this point in the laboratory, you will need to focus on differences in secondary functional groups and indices of refraction. You should also look up the reference IR spectrum for each possibility.

UNKNOWN ANALYSIS REPORT FORM

Finally, submit a completed report form along with your lab notebook at your last scheduled laboratory period. The report forms can be found in the cabinet next to the other reference materials in the front right corner of the laboratory or online at http://eres.scu.edu, Chem31L page (password is: lab). If you have any questions about how to complete the form, see your lab instructor. **It is important that you turn in the report sheet as complete as possible as we use this as the primary record of your unknown analysis.** However, any data you enter on this sheet must originally appear in your notebook.

NOTEBOOK GUIDELINES

The guidelines are derived from those drawn up by the chemistry faculty for all lab courses. Please look at them carefully before making entries in your notebook. Remember that in addition to the specifics below, the goal of the notebook is for another person to be able to repeat your lab work precisely as you did it.

Data, conclusions and results for all experimental work should be included in the notebook. A running commentary of the experimental procedure should also be included with all observations made during the experiment. The term “running commentary” is used to highlight that you should write what you are doing as you do it; you should not record what the book tells you to do, but instead what you actually did and saw.

1) The notebook must be permanently bound (not spiral) and have consecutively numbered pages.

2) Pages must never be torn out or otherwise removed from the notebook.

3) The notebook should have a title page identifying you, the course name and number, and the lab instructor. The same notebook may be used for Chem 31, 32 & 33.

4) Reserve space at the beginning of the notebook for a Table of Contents. Entries in the Table of Contents should be identified by title and page numbers for all pages containing information relevant to that title.

5) Use only indelible ink, preferably from a ball point pen, for all entries.

6) Any given page in your notebook should only include data for a single experiment. As a result, organize your notebook entries based on the experiment to which they correspond, not the date on which they were performed.

7) Each page in the notebook should be dated using an unambiguous notation like 23 June 1993. If a page includes work done for the same experiment on different days, date each entry separately.

8) If you need to continue an experiment onto another page, write the continuing page on the bottom of the initial page (cont’ on page#) and the page from which you are continuing at the top of the new one (cont’ from page#).

9) Cross out sections of pages you choose not to fill out. Do not leave blank spaces to be filled in later.

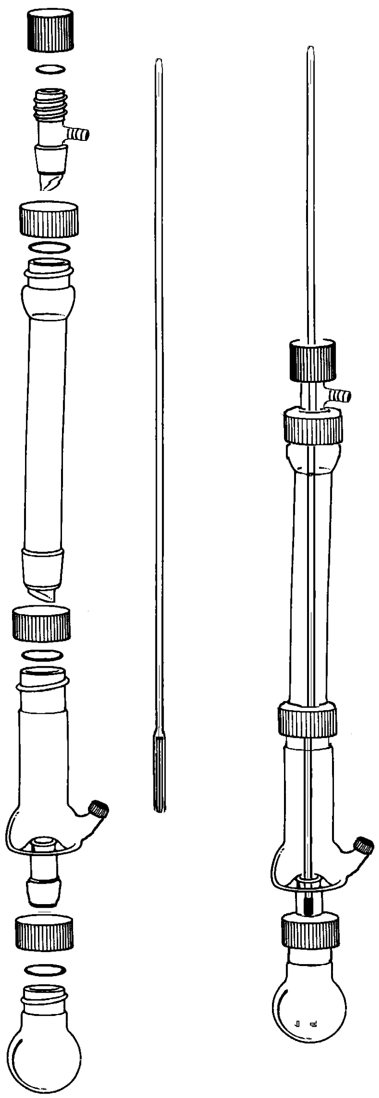
10) Graphs and spectral charts should be attached to notebook pages using glue or tape. **Each should be completely labeled**.

11) All entries should be legible and contain sufficient detail. For example:
-use proper names for instruments and glassware: 10 mL Erlenmeyer flask rather than just flask
-indicate the specific concentration of reagents used: "6M" or "0.1M" rather than just "dilute"
-indicate how precisely reagents were measured: graduated cylinder versus pipette.
-record the sequence in which chemicals were mixed, the method and length of time for heating and/or stirring; note whether a heating mantle or hot plate was used.
-completely record all observations like color changes, precipitates, gas evolution, etc.
-display any calculations in full detail for ease of verification.

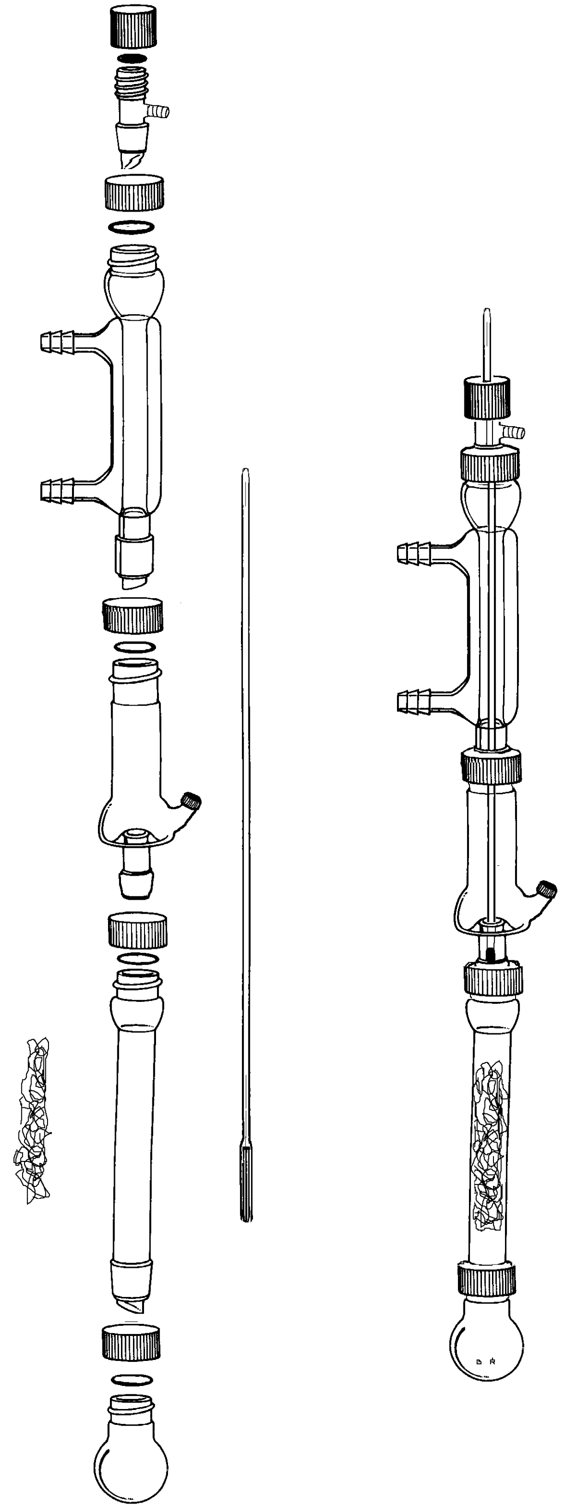
12) Draw a single line through mistakes. Erasing and over-writing are not acceptable methods for error correction.

13) At the end of each lab period sign and date your notebook and have the instructor sign it also.

Simple Distillation Setup



Fractional Distillation Setup



Extraction Flow-Chart

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