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PREFACE

Introduction to Organic Laboratory Techniques: A Small Scale Approach (Third Edition) continues our dedication to the teaching of the organic chemistry laboratory. As we have gathered experience with microscale techniques in the organic laboratory through the development of experiments and methods for the microscale versions of our textbook, we have discovered that students *can* learn to do careful work in the organic laboratory on a small scale. They do not have to consume large quantities of chemicals, and they do not have to work with very large flasks to learn the standard laboratory techniques. Furthermore, we recognize that many instructors do not wish to abandon the traditional-scale approach to their courses, and many colleges and universities cannot afford to convert all of their glassware to microscale.

In the traditional approach to teaching this subject, the quantities of chemicals used were on the order of 5-100 grams. The approach used in this textbook differs from the traditional laboratory in that nearly all of the experiments use smaller amounts of chemicals (1-10 grams). However, the glassware and methods used in this approach are identical to the glassware and methods used in traditional-scale experiments. The advantages of the small-scale approach include improved safety in the laboratory, reduced risk of fire and explosion, and reduced exposure to hazardous vapors. This approach decreases the need for hazardous waste disposal, leading to reduced contamination of the environment.

In this edition we have devoted considerable effort toward improving the safety of all of the experiments. Technique Chapter 1, "Laboratory Safety," places strong emphasis on the safe use and disposal of hazardous chemicals. We have included information on Material Safety Data Sheets (MSDS) and Right-to-Know laws. We have continued to update and improve instructions for the handling of waste products that are produced in the experiments. We recommend that virtually all waste, including aqueous solutions, be placed into appropriate waste containers.

The new experiments are listed in the Preface of the Textbook. These include several "green" chemistry experiments and some project-based experiments. In the latter experiments, students must either solve a significant problem or they must generate all of part of the experimental procedure. A Green Chemistry essay has been added and some of the experiments have been modified to make them more "green." We have significantly increased the number of unknowns listed in Appendix 1. We also offer an alternative way of solving unknowns using mainly spectroscopy.

We hope that this instructor's manual will assist you in preparing solutions, chemical reagents, supplies, and equipment necessary for each experiment that you choose to do. The lists of chemicals and equipment required for each experiment are based on the amount required for ten students. For chemicals, the amounts indicated include at least a 25% excess. At the end of the manual we have included a section that correlates the experiments with topics presented in standard organic lecture courses.

The time required for each experiment is given in laboratory periods. It is assumed that a laboratory period is about three hours in length. For laboratory periods that are either shorter or longer, appropriate adjustments must be made.

The technique chapters of the textbook are designed to stand independently from the experiments. You may have a favorite experiment that you like to do in your course. If this is the case, you can freely add your experiment and still take advantage of the technique chapters in the textbook. Since both standard-scale and microscale techniques are described in the technique chapters, you may even add some microscale experiments and still be able to refer your students to the appropriate sections in these chapters for information on each technique.

The publishers have made available videos to accompany the Textbook. These videos can be studied by students in advance of the laboratory session. They show the various steps for assembling an apparatus and carrying out a technique. See the Preface to the Textbook for more information. Students may purchase access to the website: www.cengagebrain.com

A new feature of the Instructor's Manual is the inclusion of some laboratory practical exams that test students on two basic organic laboratory techniques: crystallization and extraction. You may find these exams to be a useful way of evaluating student technique. The idea is to have students perform techniques without the textbook and without looking over another student's shoulder for help!

If you encounter problems with any of the experiments in the Textbook or if you need help in setting up your laboratory, please contact us. We would also like to hear from you if you have any suggestions for improvements in techniques or in any of the experiments.

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EQUIPPING THE ORGANIC CHEMISTRY LABORATORY

This section includes some suggestions for equipping a macroscale laboratory using a reduced scale approach. In addition, this section provides information for all instructors on some of the laboratory requirements for doing the experiments found in the textbook.

Although some experiments involve microscale techniques, they do not require special equipment. These experiments can be conducted with flasks, beakers, test tubes and other simple equipment.

Dispensing and Measuring Liquids

Where possible liquid reagents and solvents should be stored in a hood in small glass or plastic bottles. To avoid waste, the exact amount of liquid should be transferred to the student's container by one of the methods described below. Students should not pour an approximate amount of liquid into one container and then measure the required volume, leaving some excess liquid behind which must be discarded.

When accuracy is not important, one-piece polyethylene transfer pipets or calibrated Pasteur pipets shown in Technique 5, Figures 5.6B and 5.6C, provide an efficient method for delivering liquids, especially solvents to be used for extractions or crystallizations. We tape a test tube to the bottle containing the liquid in order to hold the pipet. It is easy for students to use one of these pipets to transfer liquid to a graduated cylinder for more careful measurement. If one of these techniques is used for measuring a limiting reagent, students need to be strongly encouraged to weigh the liquid following transfer.

Dispensing pumps (Technique 5, Figure 5.2) may be used to deliver larger amounts (more than 0.5 mL) of liquid. They are especially useful in dispensing solvents or non-limiting reagents. *Care must be taken to ensure that the tip is filled with liquid and that no air bubbles are observed in the tubing.* These units easily lose their "prime" especially with more volatile solvents. We have observed that some solvents swell the plastic plunger such that it can not be pulled up easily. If this happens to you, remove the solvent from the unit. After drying out thoroughly, the unit can be used again (with another solvent!). We have found that waste has been significantly reduced with dispensing pumps. They can be set individually for a particular solvent. Students simply raise the pump and dispense exactly the right amount of solvent thus eliminating waste.

If you plan to do some microscale experiments, it is useful to have available several adjustable 100-1000 μL automatic pipets for dispensing very small amounts (less than 1 mL) of liquids used as limiting reagents. They are also useful for measuring densities of liquids. The automatic pipet is very accurate

with aqueous solutions, but it is not as accurate with organic liquids. With all limiting reagents, it will be necessary to obtain the weight in order to determine accurately the amount of substance used. Since most of the errors that occur in the laboratory may be attributed to "sloppy" transfers, you should give a thorough demonstration of how to use the automatic pipet. They should be cautioned about not allowing the plunger to snap back rapidly. The automatic pipet should be placed near the appropriate reagent and supported in a vertical holding device. Automatic pipets should never be used with corrosive or caustic liquids.

A graduated 1 or 2-mL one-piece polyethylene pipet should be used to dispense small amounts or corrosive or caustic liquids, such as sulfuric acid, hydrochloric acid, or sodium hydroxide. Alternatively, a graduated pipet and pipet pump can be used. When using graduated pipets, we prefer the pipet pump shown in Technique 5, Figure 5.3B in the Textbook. The top of the pipet fits more securely in a pipet pump of this style than in a pump similar to the one shown in Figure 5.3A. To avoid contamination of the stock reagent and to minimize waste, we provide a graduated pipet and pipet pump with the reagent for community use. A pint bottle is a convenient container for holding the pipet when it is not in use.

For most procedures one of the above methods will work well to deliver the volumes of liquid required in the experiments in the Textbook. Even when more than 2 mL of liquid is required, we prefer to use one of the pipet methods for transferring the liquid, rather than having the students pour an approximate amount of liquid from the original container into their own.

The instructor should place the appropriate measuring device with each reagent and solvent. In most cases, the device will be a one-piece polyethylene pipet, a graduated pipet with a pipet pump, or dispensing pump. *The person who prepares the laboratory for an experiment should read the procedure in order to determine which device is appropriate.*

Dispensing Solids and Weighing Reagents

Four top-loading balances that read to 0.01 or 0.001 gram are required for a class of 20 students. The balances should be used with draft shields to improve accuracy. It is convenient to store solids in containers near the balances. To avoid the possibility of contamination, we provide a community spatula with the reagent.

Evaporating Solvents

Ideally, students should remove solvent by heating at a low temperature and by directing a stream of nitrogen or air through a Pasteur pipet into a flask in order to evaporate a solvent. This procedure gives a student complete control of

the evaporation process, but only works well in a laboratory with many individual hoods. In laboratories where there are only a few hoods it becomes necessary to have a permanent community evaporation station assembled in the hood. A community station may consist of aluminum heating blocks on hot plates (see Lodwig, S.N. "The Use of Solid Aluminum Heat Transfer Devices in Organic Chemistry Laboratory Instruction and Research, "*Journal of Chemical Education, 66* (1989): 77). Hot plates with containers filled with small pebbles or sand may also be used to heat the samples. The station is equipped with multiple outlets using Y-connectors and screw clamps with flexible tubing. In this way, several students can evaporate solvents using one air or nitrogen source.

The N-EVAP evaporator is a commercially available unit which is useful for larger classes. Several commercial models are available from Organomation Associates, Inc., 266 River Road West, Berlin, MA 01503-1699. Phone: (888) 838-7300. These units consist of an electrically heated water-bath container and a gas manifold equipped with blunt-end, stainless-steel needles. The holders are made with 6, 12, 24, 36 or 45 positions and will accept a variety of containers including test tubes and Erlenmeyer flasks. The 12 position model provides an exceptionally efficient means of evaporating solvents in a lab with 20 students.

Rotary Evaporators

It is becoming more common to equip the laboratory with rotary evaporators to avoid some environmental problems with evaporating solvents in the hood. We suggest systems that have coolers attached to the evaporators to improve solvent recovery thereby making the laboratory a greener environment for students and instructors. You may want to continue evaporating small amounts of solvents, approximate 10 mL or less, as indicated in the above section. Use a rotary evaporator for larger amounts, about 25 mL or more. The use of a rotary evaporator can create a large student backup unless reserved for evaporating large volumes of solvent.

A “rotacool” model rotary evaporator is available from Heidolph-Brinkmann for about $10,000. It is equipped with vacuum pump, condensation cooler and rotacool circulating chiller. This is a very efficient system that does a great job of collecting even the most volatile solvents. If you can afford to buy a rotary evaporator with a cooler, you won't regret it!

Heating Mantles and Hot Plates

For most applications involving reflux and distillation, we recommend that you use a heating mantle equipped with a temperature controller, such as the one shown on page 610 of the Textbook (Thermowell mantle with Powermite controller). These mantles employ a ceramic heating shell with electric heating coils embedded within the shell. The ceramic shell protects the mantle from

damage caused by chemical spills. A 100-mL mantle will heat 25, 50, and 100 mL flasks, and should be sufficient for most experiments in this Textbook.

Hot plates are very useful for heating solvents required for crystallization. In some cases, reactions mixtures need to be stirred as well as heated. For this reason, we suggest purchasing combination stirrer/hot plate units. If the hot plate is used for refluxing a mixture in a round-bottom flask, it is best to use a hot plate with an aluminum top and an aluminum block as a heating source. *You should not use hot plates with ceramic tops unless you are certain that the tops will withstand high temperatures without cracking.* The holes that have been drilled in the aluminum block easily support and accommodate smaller round-bottom flasks and a thermometer. The aluminum block is especially useful when temperatures above 200 ^{o}C are required. The stirrer/hot plate units should provide a temperature range of about 60 to above 250 ^{o}C. Reaction mixtures which boil at less than about 100^{o}C can usually be heated under reflux with only a hot plate without an aluminum block.

If you wish to monitor the temperature of aluminum blocks, we recommend that you not employ mercury thermometers, especially those inserted in the aluminum blocks. Glass thermometers break too easily. We suggest that you use metal dial thermometers rather than mercury thermometers. They are sufficiently accurate for monitoring the temperature of the aluminum blocks.

Melting Point Apparatus

Four electrically-heated melting point apparatus should be provided for a class of 20 students (Mel-Temp or Electrothermal). A Thomas-Hoover Uni-Melt apparatus should be considered if the class is determining micro boiling points. This device has a rapid temperature response. The Mel-Temp or Electrothermal units are less expensive and more serviceable alternative, but the temperature response is not as rapid and micro boiling point determinations may be more difficult to perform. You should try several different melting point units before buying them to see which one is the best for you.

Gas Chromatographs

At least two gas chromatographs should be provided for every 20 students, if students are expected to perform their own injections. Conditions for running samples on the Gow-Mac 69-350 or Hewlett Packard 5890 gas chromatographs are given in this Textbook. If students are expected to collect samples from a chromatograph, Gow-Mac models 69-350 or 580 can be equipped with a convenient sample collection device. Gow-Mac instruments should be equipped with an 8-foot column packed with Carbowax 20M and an 8-foot column containing 20% DC-710. Columns required for the Hewlett Packard chromatographs are given in the Textbook or in this manual.

Spectrometers/Polarimeters

The laboratory should have at least one FT-infrared spectrometer for every 20 students. The FT-infrared instruments increase the through-put of students in the laboratory. If you can afford it, an FT instrument with ATR (attenuated total reflectance) accessory is highly recommended. Use of this accessory makes analysis of solids totally trivial! Otherwise, you will need to make do with determining a spectrum using the dry film method or with a KBr pellet. For conventional IR spectroscopy we have available two of the hand press units for solids. NMR spectroscopy is important in the modern organic chemistry laboratory and you should make this available to your students, if possible. The availability of both proton and carbon NMR increases student interest especially when solving unknowns. The laboratory should be equipped with one polarimeter for use by the class.

Centrifuges

Several experiments or experimental techniques require the use of a centrifuge. They are very useful for breaking emulsions. One or two "clinical" centrifuges are adequate for 20 students. They should hold 15 mL centrifuge tubes.

Vortex Mixer

Extractions can be carried out conveniently in a 15-mL centrifuge tube. Although the tube can be stoppered and shaken to mix the layers, mixing can be accomplished efficiently with a vortex mixer. This method eliminates the problems of pressure buildup and leakage. One mixer easily serves 20 students.

Syringes and Rubber Septa

In some experiments a syringe is used to add reagents to a reaction mixture. A 1 or 2-mL glass or plastic syringe should be provided to allow use with organic solvents without contamination occurring. The plastic syringes are readily available and are much cheaper and durable than glass syringes. Disposable hypodermic needles may be used for most applications. We recommend 1 1/2- or 2-inch needles (21 or 22 gauge). When the experiment is completed, they should be saved for reuse.

Plastic Joint Clips ("Blue Clips")

It is essential that the lab be supplied with plastic joint clips to secure the 19/22 ground glass equipment (Technique 7, Figure 7.3). Breakage is dramatically reduced when they are used to secure equipment. At least 3 should be included in each laboratory locker.

Monometers

Several monometers should be available in the laboratory for use in vacuum distillations. A simple U-tube manometer is shown in Technique 16, Figure 16.9 of the Textbook.

Sublimation Equipment

It is suggested that the laboratory be supplied with microscale sublimation equipment such as that shown in Technique 17, Figure 17.2 A or B. This apparatus is equipped with 14/10 joints and can be used to perform all sublimation procedures described in this Textbook. We suggest 5 complete units as part of the community equipment.

Washing Glassware and Equipment

A plastic dishpan provides a convenient container in which to soak and wash dirty glassware. You may want to consider buying an ultrasound cleaner (sonicator) cleaner for the laboratory. Especially dirty glassware can often be effectively cleaned with one of these devices. There are some disadvantages: they are noisy and students often forget to retrieve their glassware.

WASTE MANAGEMENT GUIDELINES

These guidelines are intended for schools where the chemistry department is responsible for its own waste management. Although most of this information should apply to your situation, specific waste management practices will depend on the size of your program, other hazardous wastes generated on your campus, and state and local regulations. This information may not cover everything you need to know; however, it can help you get started or may provide some new ideas that will improve your existing waste management program.

To get started, you need to determine who regulates hazardous waste in your state. The U.S. Environmental Protection Agency (EPA) has ultimate responsibility for regulating hazardous waste in all 50 states plus the District of Columbia, Puerto Rico, and the Virgin Islands. Many states have been delegated the authority to regulate their own hazardous waste by the EPA. States which have the authority to regulate their own hazardous waste must have regulations that are as strict as the federal laws. If you operate in a state that has a hazardous waste regulating agency, then you must follow the regulations for your state rather than the federal regulations. The EPA has a home page (http://www.epa.gov) and ten regional offices that can help you find out if there is a state program in your area.

Region	States in the Region	Telephone Number
1	ME, NH, VT, MA, RI, CT	617-565-3423
2	NY,NJ,PR,VI	212-637-5000
3	PA,DE,DC,MD,VA,WV	800-438-2474
4	KY,TN,NC,SC,MS,AL,GA,FL	800-241-1754
5	MN,WI,IL,MI,IN,OH	800-621-8431
6	NM,TX,OK,AR,LA	214-665-2200
7	NE,KS,IA,MO	913-551-7000
8	MT,ND,WY,SD,UT,CO	800-227-8917
9	CA,NV,AZ,HI	415-744-1500
10	WA,OR,ID,AK	800-424-4372

You must obtain a Resource Conservation and Recovery Act (RCRA) site identification number if your campus does not already have one. This number identifies your site and all the waste generated there. This identification number is obtained through the agency that regulates hazardous waste in your state. You must supply this number to waste disposal firms when you ship waste off site, and it identifies your site on your annual hazardous waste report.

We collect all chemical waste generated in student laboratories, and we make a serious attempt to teach students that waste management is important. Therefore, students do not dispose of any chemical materials down the drain or in the trash. We find that labeling waste containers with the experiment name and a list of the chemicals that should be placed in the container greatly increases the chances that students will put wastes into the correct containers. We will use our "Isolation of Caffeine from Tea" experiment to give an example. The students generate an aqueous layer contaminated with methylene chloride. Unfortunately, the small amount of methylene chloride that dissolves in water renders the entire aqueous solution hazardous waste. The waste bottle would be labeled as follows:

Isolation of Caffeine from Tea
Hazardous Waste
Aqueous layer contaminated w/ methylene chloride
Suspect Human Carcinogen

Note that "Hazardous Waste" must be included on the label, as required by law. Also, the primary hazard of the waste, the last entry on this label, is required by law. Refer to the material safety data sheet (MSDS) for the primary or most hazardous constituent of the waste to determine an appropriate warning.

Wastes collected from student labs are consolidated by waste type or treated, if it is safe and legal to do so. We find that all wastes we generate fit into one of the following categories:

Nonhazardous Solids such as paper, tea bags, and corks are disposed of with the ordinary trash.

Broken Glassware is disposed of in a container designated for this purpose. When the container is full, it is packaged securely and disposed of with the ordinary trash.

Organic Solids with halogens are consolidated with our halogenated organic solvents, and those without halogens are consolidated with our non-halogenated organic solvents.

Inorganic Solids such as alumina and drying agents are accumulated together and disposed of as hazardous waste.

Non-Halogenated Organic Solvents such as alcohols, toluene, hexane, and diethyl ether are disposed of as hazardous waste. Intentional evaporation or drain disposal of these materials is illegal. However, evaporation of these solvents as part of the workup in an experiment *is legal*, since the material is not yet waste and the evaporation is a legitimate part of the procedure.

Halogenated Organic Solvents such as dichloromethane (methylene chloride), chloroform, and carbon tetrachloride are disposed of as hazardous waste. Intentional evaporation or drain disposal of these materials is illegal. However, evaporation of these solvents as part of the workup in an experiment is legal, since the material is not yet waste and the evaporation is a legitimate part of the procedure.

Inorganic Acids without heavy metals or halogenated solvent contamination are neutralized and discharged to the sewer. A log of these treatment activities is maintained.

Inorganic Bases without heavy metals or halogenated solvent contamination are neutralized and discharged to the sewer. A log of these treatment activities is maintained.

Aqueous Solutions Contaminated with Halogenated Solvents are disposed of as hazardous waste. Intentional evaporation or drain disposal of these materials is illegal.

Aqueous Solutions with Heavy Metals may either be treated to remove the heavy metal or disposed of as hazardous waste. If you treat these wastes, you

must test the pH and metal levels before discharge of the treated waste to the sewer to confirm successful treatment. In most states, the water may be legally evaporated to reduce the waste volume, and the remaining metal sludge treated as hazardous waste. The original amount of waste including water must be reported on your annual hazardous waste report.

Most states allow some forms of treatment by the waste generator without the need for special permits. Before you treat a waste you must make sure that your regulators allow the treatment practice. Prior to waste treatment, all of the constituents of the waste, such as heavy metal, solvent content, and low or high pH must be determined. You also need to contact your local sewer district to find out if they have limits on what may be discharged to their system. In many cases a material may not be considered hazardous waste by the EPA or a State Environmental Regulatory Agency, but is restricted from disposal to the sanitary sewer. Treatment and discharge of waste is not recommended if you are on a septic system.

If you elect to treat waste, you are required to test the treated waste for each constituent that made the untreated waste hazardous before you discharge it to the sewer. For example, if you treated an aqueous waste that contained silver, barium, and chromium by precipitating the metals, you would have to check the barium, silver, and chromium levels of the treated waste before discharge to the sewer. Because of this burden, we limit our treatment to neutralization of non-heavy-metal-bearing aqueous wastes that have a low or high pH. Also, remember that intentional evaporation of solvents, and dilution and drain disposal of hazardous wastes not only violates EPA regulations but is also harmful to the environment.

Maintain a log of all wastes treated on site. At a minimum this log should include: a description of the waste, the amount of waste treated, the name of the person treating the waste, the treatment method, and the treatment date. Hazardous wastes that are treated on site must be "counted" and reported on your annual hazardous waste report.

Maintain a waste generation log, which includes the total amount of waste treated and generated. At a minimum this log should include: date, description of the waste, amount, and identity of generator. This log must be included in the annual hazardous waste report that is described below.

We recommend that you limit the amount of waste you accumulate not only to simplify your regulatory requirements, but also to minimize the risk of leaks and spills. In most states, by accumulating less than 55 gallons of each type of waste you simplify the storage and record keeping requirements associated with waste storage. Larger waste accumulation areas must be inspected weekly and equipped with emergency response supplies. Waste must be stored in a secure

(locked) area, segregated by type, capped when not in use, and provided with secondary containment (several bottles of the same type of waste can be placed in a tray or individual bottles may be stored in pails). We recommend hazardous waste shipments at intervals as dictated by your operation to limit the amount of waste stored.

At smaller schools you may find that annual waste shipments are a good management practice. At larger schools shipments each semester, quarterly, or even monthly may be required. At Western Washington University, the motor pool and the physical plant operations generate far more waste than the chemistry department. You may find it worthwhile to coordinate your waste disposal with other departments or operations within your school.

If you elect to ship your own waste, you must learn and follow all of the mandated procedures. As a simpler alternative, there are private contractors who will consolidate, treat, package, and ship your waste for you. However, this alternative does not keep you from having to keep good records.

Contact your local fire department to find out about requirements concerning hazardous material storage. Often these agencies require chemical inventory and storage information about your site so that they can respond appropriately in the event of an emergency.

Establish written hazardous waste management procedures for your campus and communicate these procedures to those involved with waste handling. Also, assure that the person on your campus who signs manifests has received Department of Transportation training on hazardous material shipping.

Retain copies of all manifests and land disposal restriction certifications, sometimes known as "land bans", of waste sent off site for disposal. Manifests can be thought of as the shipping papers for hazardous waste shipments. Land disposal restriction certifications accompany manifests and document disposal and treatment restrictions based on the characteristics of the waste being sent for disposal.

Complete an annual hazardous waste report for all hazardous waste activities on your campus. This report is required by law and must be submitted to the agency that regulates hazardous waste in your area. The report summarizes your hazardous waste activities for the previous calendar year. To complete this report you will need: your RCRA site identification number, copies of all manifests for the past year and your treatment and generation logs.

LABORATORY EQUIPMENT AND SUPPLIES

A. Individual student glassware and equipment contained in the locker

1. Organic Chemistry Kit (19/22 joints)

500 mL 3-Neck round bottom flask
250 mL Round-bottom flask with side tubulation
25, 50, and 100 mL Round-bottom boiling flasks
Stoppers (2)
Thermometer adapter
Rubber thermometer holder
Bleed tube (ebulliator tube)
Claisen head
Distilling head
Vacuum adapter
Condenser
Fractionating column (packed with steel wool)
125 mL Separatory funnel, Teflon stopcock

2. Other individual glassware

Beakers; 50 mL (2), 100 mL (2), 250 mL (2), 400 mL (1)
Graduated cylinders; 10 mL and 100 mL
Drying tubes (2)
Evaporating dish, size 00
Erlenmeyer flasks; 25 mL (2), 50 mL (2), 125 mL (1), 250 mL (1), 500 mL (1)
Filter flask; 125 mL
Aspirator trap bottle (part of community equipment)
Conical funnel (stemless), 50 mm
Powder funnel
Büchner funnel; size 0
Büchner funnel; size 2A (optional)
Hirsch funnel, plastic preferred
Test tubes or culture tubes; 10 x 75 mm (6); 16 x 100 mm (5); 15 x 125 mm (3)
Thermometer, non-mercury; 360°
Watch glasses; 50 mm (2) and 100 mm (2)
Small ground glass bottles for submitting liquid samples (2)
Small vials for submitting solid samples to the instructor (2)
4 oz. Screw cap bottles (2)
Centrifuge tubes, glass, screw cap with Teflon liner; 15 mL (2)
or Centrifuge tubes, polypropylene, screw cap, 15 mL (2), VWR 20171-010

Centrifuge tubes, plastic (no screw cap), 15 mL (2)
2 mL Glass or plastic syringe (Luer lock and Teflon plunger tip preferred)
Needles to fit syringe

3. Individual equipment

Plastic joint clips to fit 19/22 joints (3)
Condenser clamp, 3-prong with holder
Utility clamps (2)
Screw clamp
Dropper bulbs, latex, 2 mL (4)
Rubber policeman
Stirring rod
Neoprene adapters, nos. 2, 3, and 4
Rubber serum bottle stopper to fit over 19/22 joint
Brushes, small and large
Microburner and chimney (optional)
Wire gauze (optional)
Test tube holder
Spatula
Stir bar
Test tube block
Rubber tubing
Pressure tubing
Scorer or file
Safety glasses
Aluminum blocks (optional)
Metal thermometer to measure temperature when accuracy is not required (optional)

B. Community Equipment

The following equipment should be available in the laboratory or nearby. (Numbers in parentheses indicate requirements for 20 students)

Hot plate/stirrer (20)
Ring stands (40)
Iron rings to hold separatory funnels (20)
Heating mantles with controllers, two sizes (20 each)
Wooden blocks to support glassware
Automatic pipets; 100 - 1000 μL (optional)
Dispensing pumps; 2 and 5 mL sizes (optional)
Pipet pumps (optional)
Sponges (10)
Screw cap bottles for chromatography (40)

Ice buckets (5)
Filter flasks; 500 mL (optional)
Separatory funnels, 500 mL (optional)
Microscale sublimation apparatus, with 14/10 joints (optional, 5)
Melting point apparatus (4)
Top-loading balances with draft shields, 0.001 g (4)
Refractometer (1)
Polarimeter (1)
Centrifuges (2)
Gas Chromatographs, Gow-Mac, model 69-350
Equipment required for optional collection of liquids from Gow-Mac chromatographs: metal adapter for collection of samples (2), 1 mL conical reaction vials with 5/5 joint (2) and collection tubes with 5/5 joint (2)
Vortex mixer (optional, 1)
Infrared Spectrometer (2)
Potassium bromide hand press (2)
Salt plates for infrared spectroscopy (2 pairs)
NMR spectrometer
Ovens (2)
Glass working bench with burners and supply of glass tubing
Matches or gas lighters for burners
Scissors (2)
Handbook of Chemistry and Physics (mounted on board)
Handbook of Tables for Organic Compound Identification
Merck Index (mounted on board)
Rotary Evaporators (recommended)

C. Community Supplies

1. Chemicals and supplies
The following materials should be available at all times on the side shelves or desks.

Gloves, disposable
Pasteur pipets; 5 3/4-inch and 9-inch sizes
Graduated one-piece polyethylene transfer pipets
Applicator sticks
Decolorizing carbon, pelletized and powdered
Sample vials for submitting products
Filter paper to fit Büchner and Hirsch funnels
Filter paper for gravity filtrations
Stopcock grease
Glycerol in dropper bottle
Boiling stones, inert such as corundum

Corks, assorted
pH paper
Red and blue litmus paper
Copper wire
Capillary tubes, sealed on one end for melting points
Capillary tubes, open on both ends for TLC chromatography
Glass wool
Cotton
Labeling tape
Soap
Celite (Filter Aid)
Rock salt
Anhydrous magnesium sulfate (powdered)
Anhydrous calcium chloride (4-20 mesh)
Anhydrous sodium sulfate (granular)

2. Acids and bases
The solutions and reagents should be placed in one area of the laboratory on a chemically resistant surface.
Acids need to be separated from bases.
Sodium hydroxide solutions; 5%
Sodium bicarbonate solution, 5%
Hydrochloric acid solutions; concentrated and 5%
Sodium chloride solution, saturated
Nitric acid, concentrated
Ammonium hydroxide, concentrated
Sulfuric acid, concentrated

3. Common solvents
These solvents should be placed in a hood during use and stored in a special cabinet at other times (see below).

Hexane
Petroleum ether (various boiling ranges)
Acetone
Methanol
Toluene
Methylene chloride
95% Ethanol (5 % water)
Diethyl ether
Carbon tetrachloride and methylene chloride, kept in a hood near the infrared spectrometer, with a Pasteur pipet attached.

4. Test reagent shelves
We usually keep the reagents and known compounds for

Experiment 55 (qualitative analysis) in a designated area of the laboratory at all times. The noxious chemicals are kept in a hood.

D. Safety

Storage cabinet for flammable organic solvents
Fire extinguishers
Eye wash fountains
Showers
Fire blankets
Solvent waste containers (see individual experiment)

E. Safety References and MSDS sheets (Technique 1)

ORGANIC LABORATORY TECHNIQUES PRACTICAL EXAMS

Some instructors may desire to test students on two very basic organic laboratory techniques: crystallization and extraction. We call this a laboratory practical exam. With the technique exam, you can determine who really has the best technique and who the leaders and followers are in your laboratory course. Students do their work without a textbook in front of them. They are prohibited from looking at what other students are doing.

Organic lab practical exam advice for instructors

You may desire to give this test to students at the end of the first organic laboratory course. Each student will be required to purify a compound either by acid-base extraction or crystallization. These students have completed Experiment 2 (Crystallization) and Experiment 3 (Extraction). They have also completed Experiment 4 (A Separation and Purification Scheme).

Several days before the exam they are given the handout titled "Organic Lab Practical Exam Instructions for Students". At this point they don't know if they will be doing an extraction or crystallization. Therefore, they must prepare for both possibilities. On the test day, they are given either the sheet titled "Extraction" or "Crystallization" and a sample of an impure compound. They have three hour to complete this assignment, but most students are done after two hours.

We make up the samples for crystallization by mixing thoroughly 12 g of urea and 0.53 g of trans-cinnamic acid. Each student is given 1.0 g of the mixture. We don't tell them the actual weight and they are told not to weigh it. For the extraction, dissolve 3.0 g of fluorene and 0.75 g of benzoic acid in 60

mL of methylene chloride. Each student is given 4.0 mL of this solution. Students should use a centrifuge tube to perform the extraction procedure.

Because of the way this test is designed, students do not know how much of the compound they start with or the melting point. Therefore, it is impossible for them to change their data to get a better grade.

Organic lab practical exam instructions for students

For this lab practical exam you will be given an impure sample of a compound to be purified either by acid/base extraction or crystallization. You are to carry out this task without the aid of other students or any written or electronic resources.

When you arrive in lab, you will be given an impure sample and an instruction sheet that will inform you whether the compound is to be purified by acid/base extraction or crystallization. You will be told the structure of the compound, but not the melting point.

Extraction. On the instruction sheet for the acid/base extraction purification you will be told the structure of the neutral compound, the approximate weight of the compound, and what organic solvent it is dissolved in (the compound and impurity will already be dissolved in an appropriate solvent to do the extraction). The volume of this solution will be 4.0 mL. Therefore, you should use a centrifuge tube to perform the extraction. You will also be told whether the impurity is an organic acid or base (amine) and how much 1*M* NaOH or 1 *M* HCl you should use for the extraction step. You do not have to isolate the acid or base impurity. You must decide whether to use NaOH or HCl to extract the impurity.

Your goal will be to separate the neutral compound from the acid or base impurity, isolate it in a pure form, and determine the weight recovered and the melting point. You will **not** know exactly how much of the neutral compound is in the original sample or the melting point.

Crystallization. For the crystallization purification, you will be told the structure of the impure compound and the approximate weight of the sample. You will also be given three suggestions as to which solvent could be used for crystallization. One of these solvents will be suitable for crystallizing this compound. The compound will be too soluble in one of them and not soluble enough in the third solvent. Your goal will be to determine the best solvent, purify your sample by crystallization, and determine the weight recovered and melting point. You will **not** know the exact amount of the compound that you start with or the literature melting point of the compound

General comments and grading procedures. You may **not** use your textbook, handbooks, or any other resources (written or electronic) while completing this exercise. You may not talk to other students and you should refrain from looking at the set-ups used by other students. This exam will be worth 20 points and will be based on the weight recovered of the purified material and the purity based on melting point. You will also be graded on how your sample looks and whether or not the sample is dry. You may have a second sample, but it will cost you 2 points. If you take a third sample, this will cost you an additional 3 points.

Organic lab practical exam instructions and report sheet for crystallization

_____ Name

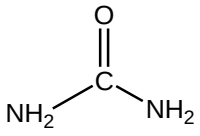
_____ Tube Number

You may **not** use your textbook, handbooks, or any other written material while completing this exercise. You may not use any electronic resources. However, you may take down notes on this sheet of paper. You may not talk to other students during this exercise and you should refrain from looking at the set-ups used by other students. If you have questions during the lab, **ask your instructor.**

There will be a 2-point subjective grade. The subjective grade will be determined by the instructor's assessment of such things as whether or not you talk to other students or if you obviously look at what other students are doing. You may have a second sample, but it will cost you 2 points.

Instructions. You will be given a sample of impure urea that has a weight between 0.8 – 1.2 g. **Do not weigh the sample**. You should crystallize the entire sample**. Write down the number of the tube and your name in the space above.**

The structure of urea is:



Urea can be crystallized from one of the three following solvents: 95% ethyl alcohol, water, or hexane. You may determine which solvent to use either by experimentation or by making an educated guess.

After crystallizing the sample of impure urea, determine the weight and melting point of the dry crystals. The melting point should be between 120 - 140° C. Turn in this sheet and your sample in a vial labeled as follows: your name, the name of the compound, weight of sample, and melting point. You

will be graded on the recovery and purity, as determined by appearance and melting point. Record the recovery and melting point below:

Recovered weight of sample_____

Melting point of sample _____

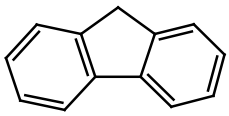
Organic lab practical exam instructions and report sheet for extraction

_____ Name

You may **not** use your textbook, handbooks, or any other written material while completing this exercise. You may not use any electronic resources. However, you may take down notes on this sheet of paper. You may not talk to other students during this exercise and you should refrain from looking at the set-ups used by other students. If you have questions during the lab, **ask your instructor.**

There will be a 2-point subjective grade. The subjective grade will be determined by the instructor's assessment of such things as whether or not you talk to other students or if you obviously look at what other students are doing. You may have a second sample, but it will cost you 2 points.

Instructions. You will be given 4.0 mL of a methylene chloride solution containing fluorene (a neutral compound) and an **acid** impurity. The weight of fluorene will be between 0.15 - 0.25 g. Your goal is to isolate the neutral compound and determine its weight and melting point. To remove the impurity, you should extract the methylene chloride solution with two 2-mL portions of either 1.0*M* NaOH or 1.0*M* HCl. After drying the organic layer, evaporate off the methylene chloride. Weigh the solid and determine the melting point. The melting point should be between 105-125 °C.

Fluorene: 

Turn in this sheet and your sample in a vial labeled as follows: your name, the name of the compound, weight of sample, and mp. You will be graded on the recovery and purity, as determined by appearance and melting point. Record the recovery and melting point below

Recovered weight of sample _____

Melting point of sample _____

Experiment 1

SOLUBILITY

TIME ESTIMATE: Parts A-D (3 hours); Part E (1 hour); Part F (1 hour)

CHEMICALS AND SUPPLIES PER 10 STUDENTS:

Part A

Benzophenone (Grind up the flakes into a powder)	0.5 g
Malonic acid	0.5 g
Biphenyl	0.5 g
Methyl alcohol	40 mL
Hexane	40 mL

Part B

Methyl alcohol	13 mL
1-Butanol	13 mL
1-Octanol	13 mL
Hexane	40 mL

Part C

Ethyl alcohol	13 mL
Diethyl ether	13 mL
Methylene chloride	25 mL
Hexane	13 mL

Part D

Benzoic acid	1.2 g

Ethyl 4-aminobenzoate		1.2 g
1M NaOH		25 mL
1M HCl		25 mL
6M NaOH		6 mL
6M HCl		6 mL
Litmus paper		

Part E

1. Acetone 25 mL

   Hexane 13 mL

2. We give each pair of students two mixtures. Each mixture contains 2 mL of each liquid and about 0.1 g of the dissolved solid. There are many possible combinations of substances to use. The mixtures we have used contain one of the following combinations of solid and liquids (the solid is listed first): fluorene, methylene chloride, water; triphenylmethanol, diethyl ether, water; salicylic acid, methylene chloride, 1M NaOH; ethyl 4-aminobenzoate, diethyl ether, 1M HCl; naphthalene, hexane, water; benzoic acid, diethyl ether, 1M NaOH; *p*-aminoacetophenone, methylene chloride, 1M HCl. The mixtures containing ethyl 4-aminobenzoate and *p*- aminoacetophenone should be made up fresh on the same day as the lab, otherwise the solutions become colored.

3. Tetraphenylcyclopentadienone 0.3 g

   Methyl alcohol 13mL

Part F

Self-Assembled Monolayer Demonstration Kit
Available from Asemblon, Inc., 15340 N.E. 92nd St., Suite B, Redmond, WA 98052; phone 425-558-5100; asemblon.com. Instructions for filling the Asemblon pen with the thiol are provided with the kit.

If done as a demonstration, one kit is sufficient. Otherwise one kit for every 4 students works well.

Butane to fill the torch for erasing the gold slide

Methyl alcohol	10 mL
Acetone	10 mL

CAS Registry numbers:

Benzophenone	119-61-9
Malonic acid	141-82-2
Biphenyl	92-52-4
Hexanes	73513-42-5
Methyl alcohol	67-56-1
1-Butanol	71-36-3
1-Octanol	111-87-5
Ethyl alcohol (ethanol), 95%	64-17-5
Diethyl ether	60-29-7
Methylene chloride	75-09-2
Benzoic acid	65-85-0
Ethyl 4-aminobenzoate	94-09-7
Acetone	67-64-1
Fluorene	86-73-7
Triphenylmethanol	76-84-6
Salicylic acid	69-72-7
Naphthalene	91-20-3
p-Aminoacetophenone	99-92-3
Tetraphenylcyclopentadienone	479-33-4

SPECIAL NOTES

In Part A, it is very important that students follow the instructions carefully for stirring the mixtures. The bigger spatula shown in Figure 12.10 on page 697 of the Textbook is very effective in achieving consistent stirring from one mixture to another.

We have found that some students have difficulty performing Critical Thinking Application #2 (p. 9 of the Text) on the same day that they complete the rest of this experiment. Many students need time to assimilate the material in this experiment before they can complete this exercise successfully. One approach is to assign Critical Thinking Applications from several technique experiments (for example, Experiments 1 - 3) on a laboratory period following

the completion of the individual technique experiments. This provides an effective way of reviewing some of the basic techniques.

Part A (expected results)

Compound	Water	Methyl alcohol	Hexane
Benzophenone	Insoluble	Soluble in about 25 sec	Soluble in about 60 sec
Malonic acid	Soluble in about 10 sec	Soluble in about 10 sec	Insoluble
Biphenyl	Insoluble	Partially soluble	Soluble in about 40 sec

Part B (expected results)

Compound	Water	Hexane
1-Octanol	Insoluble	Soluble
1-Butanol	Partially soluble	Soluble
Methanol	Soluble	Insoluble

Part F

In Part F, a butane torch is used to erase the gold slide. It is best that this be done by the instructor. When erasing the slide, keep the torch moving and do not hold it in one spot on the slide. Heating one spot for too long can cause the slide to shatter.

ANSWERS TO QUESTIONS

1. a) yes
   b) no
   c) yes
   d) no
   e) no
   f) yes
   g) no

2. a) miscible
   b) miscible
   c) miscible
   d) immiscible
   e) miscible
   f) miscible

3. Ibuprofen is a carboxylic acid which is converted to a water-soluble salt in 1.0M NaOH.

4. Thymol has a phenolic OH group which is acidic. In 1.0M NaOH, thymol is converted into a water-soluble salt.

5. Cannibinol is only slightly soluble in methyl alcohol because the large hydrocarbon component of cannibinol negates the fact that they belong to the same family.

6. When you write on the slide, a monolayer of thiols is deposited on the slide. The OH groups will be located at the top surface of the coated area. Since hydroxyl groups are hydrophilic, water is attracted to the part of the slide that was written on. The rest of the surface is coated with hydrocarbons from the air and is hydrophobic, so the water rolls off and does not stick.

7. Water adheres to the gold surface immediately after flame cleaning because the gold surface is a high-energy surface that attracts water molecules. Within several minutes, the surface becomes coated with hydrocarbons from the air, making the surface hydrophobic.

8. A methyl group on the end of the thiol molecule would make the surface nonpolar or hydrophobic.

9. Heating the slide with a butane torch burns off the thiols.

10. When you write on a glass surface with a crayon or wax pencil, you are transferring a thick layer of material rather than a monolayer. You can even see this difference since you can see the film made by the wax, but you cannot see the monolayer after it forms on the gold slide.

11. This is caused by the very high surface tension of water that allows water molecules to span the gap of the hole in these letters.

Experiment 2

CRYSTALLIZATION

TIME ESTIMATE: Parts A and B (3 hours), Parts C (about 1hour)

CHEMICALS AND SUPPLIES PER 10 STUDENTS:

Part A

Impure sulfanilamide (5% fluorenone the impurity) 10 g
Grind thoroughly to make homogeneous.

95% Ethyl alcohol 250 mL

Filter paper for □üchner funnel

Melting point capillary tubes

Waste container for non-halogenated organic wastes.

Part B

The appropriate solvent for crystallizing the impure fluorene is methyl alcohol. Fluorene is too soluble in toluene and insoluble in water at all temperatures.

Impure fluorene (5% fluorenone as the impurity) 10 g
Grind thoroughly to make homogeneous.

Methyl alcohol 300 mL

Toluene 25 mL

Waste container for non-halogenated organic wastes.

Part C

Acetylsalicylic acid	5 g
Benzoic acid	5 g
Benzoin	5 g
Dibenzoyl ethylene	5 g
Succinimide	5 g