TABLE OF CONTENTS

Note: This document is internally linked. You can click on page numbers, URLs, etc. to go to the location.

Preface 1
Equipping the Organic Chemistry Laboratory 4
Waste Management Guidelines 9
Laboratory Equipment and Supplies 13

Chemicals and Supplies for Each Experiment

Answers to Questions

Experiment 1 Solubility 18
Experiment 2 Crystallization 21
Experiment 3 Extraction 23
Experiment 4 Chromatography 26
Experiment 5 Simple and Fractional Distillation 30
Experiment 6 Infrared Spectroscopy and Boiling-Point Determination 32
Experiment 7 Acetylsalicylic acid 34
Experiment 8 Acetanilide 36
Experiment 9 Acetaminophen 38
Experiment 10 TLC of Analgesic Drugs 39
Experiment 11 Isolation of Caffeine 44
Experiment 12 Isopentyl Acetate (Banana Oil) 47
Experiment 13 Methyl Salicylate (Oil of Wintergreen) 50
Experiment 14 Isolation of Eugenol from Cloves 52
Experiment 15 Spearmint and Caraway Oil: (+)- and (-)-Carvones 54
Experiment 16 Isolation of Chlorophyll and Carotenoid Pigments from Spinich 59
Experiment 17 Ethanol from Sucrose 61
Experiment 18 An Introduction to Molecular Modeling 63
Experiment 19 Computation Chemistry 64
Experiment 20 Reactivity of Some Alkyl Halides 65
Experiment 21 Nucleophilic Substitution Reactions: Competing Nucleophiles 68
Experiment 22 Hydrolysis of Some Alkyl Halides 73
Experiment 23 Synthesis of n-Butyl Bromide and t-Pentyl Chloride 74
Experiment 24 4-Methylcyclohexene 78
Experiment 25 Phase Transfer Catalysis: Addition of Dichlorocarbene to Cyclohexene 80
Experiment 26 Methyl Stearate from Methyl Oleate 82
Experiment 27 Preparation of Soap 86
Experiment 28 Preparation of a Detergent 87
Experiment 29 Gas Chromatographic Analysis of Gasolines 89
Experiment 30 Chromic Acid Oxidation of Alcohols 91
Experiment 31 Chiral Reduction of Ethyl Acetoacetate; Optical Purity Determination 96
Experiment 32 Nitration of Aromatic Compounds Using a Recyclable Catalyst 98
Experiment 33 An Oxidation-Reduction Scheme: Borneol, Camphor, Isoborneol 100
<table>
<thead>
<tr>
<th>Experiment</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>34</td>
<td>Multistep Reaction Sequence: The Conversion of Benzaldehyde to Benzilic Acid</td>
</tr>
<tr>
<td>35</td>
<td>Tetraphenylcyclopentadiene</td>
</tr>
<tr>
<td>36</td>
<td>Triphenylmethanol and Benzoic Acid</td>
</tr>
<tr>
<td>37</td>
<td>Resolution of (\alpha)-Phenylethylamine and Determination of Optical Purity</td>
</tr>
<tr>
<td>38</td>
<td>The Aldol Condensation: Preparation of Benzalacetophenones (Chalcones)</td>
</tr>
<tr>
<td>39</td>
<td>Preparation of an (\alpha,\beta)-Unsaturated Ketone via Michael and Aldol Condensation Reactions</td>
</tr>
<tr>
<td>40</td>
<td>Enamine Reactions: 2-Acetylcyclohexanone</td>
</tr>
<tr>
<td>41</td>
<td>1,4-Diphenyl-1,3-Butadiene</td>
</tr>
<tr>
<td>42</td>
<td>Relative Reactivities of Several Aromatic Compounds</td>
</tr>
<tr>
<td>43</td>
<td>Nitration of Methyl Benzoate</td>
</tr>
<tr>
<td>44</td>
<td>Benzocaine</td>
</tr>
<tr>
<td>45</td>
<td>(N,N)-Diethyl-(m)-Toluamide: The Insect Repellent “OFF”</td>
</tr>
<tr>
<td>46</td>
<td>Sulfa Drugs: Preparation of Sulfanilamide</td>
</tr>
<tr>
<td>47</td>
<td>Chromatography of Some Dye Mixtures</td>
</tr>
<tr>
<td>48</td>
<td>Preparation and Properties of Polymers: Polyester, Nylon, and Polystyrene</td>
</tr>
<tr>
<td>49</td>
<td>The Diels-Alder Reaction of Cyclopentadiene with Maleic Anhydride</td>
</tr>
<tr>
<td>50</td>
<td>Photoreduction of Benzophenone and Rearrangement of Benzpinacol to Benzopinacolone</td>
</tr>
<tr>
<td>51</td>
<td>Luminol</td>
</tr>
<tr>
<td>52</td>
<td>Carbohydrates</td>
</tr>
<tr>
<td>53</td>
<td>Analysis of a Diet Soft Drink by HPLC</td>
</tr>
<tr>
<td>54</td>
<td>Isolation of Casein and Lactose from Milk</td>
</tr>
<tr>
<td>55</td>
<td>Identification of Unknowns</td>
</tr>
<tr>
<td>56</td>
<td>Preparation of a C-4 or C-5 Acetate Ester</td>
</tr>
<tr>
<td>57</td>
<td>A Separation and Purification Scheme</td>
</tr>
<tr>
<td>58</td>
<td>Isolation of Essential Oils from Allspice, Cloves, Cumin, Caraway, Cinnamon or Fennel</td>
</tr>
<tr>
<td>59</td>
<td>Friedel-Crafts Acylation</td>
</tr>
<tr>
<td>60</td>
<td>The Analysis of Antihistamine Drugs by Gas Chromatography-Mass Spectrometry</td>
</tr>
<tr>
<td>61</td>
<td>Carbonation of an Unknown Aromatic Halide</td>
</tr>
<tr>
<td>62</td>
<td>The Aldehyde Enigma</td>
</tr>
<tr>
<td>63</td>
<td>Synthesis of Substituted Chalcones: A Guided-Inquiry Experience</td>
</tr>
<tr>
<td>64</td>
<td>Michael and Aldol Condensation Reactions</td>
</tr>
<tr>
<td>65</td>
<td>Esterification Reactions of Vanillin: The Use of NMR to Determine a Structure</td>
</tr>
<tr>
<td>66</td>
<td>An Oxidation Puzzle</td>
</tr>
</tbody>
</table>

**Answers to Problems in the Techniques Section**

<table>
<thead>
<tr>
<th>Technique</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Laboratory Safety</td>
</tr>
<tr>
<td>2</td>
<td>The Laboratory Notebook, Calculations, and Laboratory Records</td>
</tr>
</tbody>
</table>

© 2005 Brooks/Cole, a division of Thomson Learning, Inc.
| Technique 3 | Laboratory Glassware: Care and Cleaning | 220 |
| Technique 4 | How to Find Data for Compounds: Handbooks and catalogs | 221 |
| Technique 5 | Measurement of Volume and Weight | 222 |
| Technique 6 | Heating and Cooling Methods | 223 |
| Technique 7 | Reaction Methods | 224 |
| Technique 8 | Filtration | 226 |
| Technique 9 | Physical Constants of Solids: The Melting Point | 227 |
| Technique 10 | Solubility | 227 |
| Technique 11 | Crystallization: Purification of Solids | 228 |
| Technique 12 | Extractions, Separations, and Drying Agents | 231 |
| Technique 13 | Physical Constants of Liquids: The Boiling Point and Density | 234 |
| Technique 14 | Simple Distillation | 235 |
| Technique 15 | Fractional Distillation, Azeotropes | 236 |
| Technique 16 | Vacuum Distillation, Manometers | 239 |
| Technique 17 | Sublimation | 239 |
| Technique 18 | Steam Distillation | 240 |
| Technique 19 | Column Chromatography | 241 |
| Technique 20 | Thin-Layer Chromatography | 243 |
| Technique 21 | High-Performance Liquid Chromatography (HPLC) | 244 |
| Technique 22 | Gas Chromatography | 245 |
| Technique 23 | Polarimetry | 246 |
| Technique 24 | Refractometry | 247 |
| Technique 25 | Infrared Spectroscopy | 248 |
| Technique 26 | Nuclear Magnetic Resonance Spectroscopy | 249 |
| Technique 27 | Carbon-13 Nuclear Magnetic Resonance Spectroscopy | 250 |
| Technique 28 | Mass Spectrometry | 250 |
| Technique 29 | Guide to the Chemical Literature | 250 |

Correlation of Experiments with Lecture Topics | 252
PREFACE

*Introduction to Organic Laboratory Techniques: A Small Scale Approach (Second 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.

This edition of the Small Scale book includes stand-alone technique experiments. However, we are aware of many schools that use our textbook supplement our book with their own technique experiments. Because of this, and because our teaching philosophy has evolved for the past twenty-five years, we have included five new technique experiments in this book: Crystallization, Extraction, Chromatography, Distillation, and Infrared Spectroscopy and Boiling Point Determination (Experiments 2-6). We have also included an introductory experiment on solubility (Experiment 1). These six experiments emphasize understanding of and proficiency in performing the techniques.

The new experiments are listed in the Preface of the Textbook. These include,
along with the six technique experiments listed above, 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 have provided experiments that introduce students to computer-interfaced experiments and to molecular modeling. If you are interested in implementing any of these new experiments, we recommend that you test them beforehand to make sure that your system and software are compatible with the procedures that we have outlined.

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.

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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E-Mail: pavia@chem.wwu.edu

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E-Mail: lampman@chem.wwu.edu
We have also set up a special electronic mail address for questions and comments. This address is:

plke@chem.wwu.edu

We also encourage you to visit our home page at:

http://lightning.chem.wwu.edu/dept/staff/org/plkhome.html
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 cannot 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!).

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.

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 613 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 °C are required. The stirrer/hot plate units should provide a temperature range of about 60 to above 250 °C. Reaction mixtures which boil at less than about 100 °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 and one polarimeter for every 20 students. The FT-infrared instruments increase the through-put of students in the laboratory. We usually recommend that students determine the infrared spectrum using the dry film method, if possible. We have available two of the hand press units shown on page 879 for KBr pellets. NMR spectroscopy is a valuable addition to the modern chemistry laboratory. The availability of both proton and carbon NMR increases student interest especially when solving unknowns.

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 (Figure 7.3, page 626). 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 on page 776 of the Textbook.

Sublimation Equipment

It is suggested that the laboratory be supplied with microscale sublimation equipment such as that shown on page 784 of the Textbook (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.

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

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

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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)
Side arm test tube, 20 x 150 mm
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
Microchromatographic column (optional)
NMR tube stored in PVC tube (optional)

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

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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 Chromatograph, 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)

C. Community Supplies

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

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 (see page 574 and 575 of the Textbook)
Experiment 1
SOLUBILITY

TIME ESTIMATE: Parts A-D (3 hours); Part E (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 13 mL

SPECIAL NOTES

In Part A, it is very important that students follow the instructions carefully for stirring the mixtures. The spatula shown on page 591 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. 12 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)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Water</th>
<th>Methyl alcohol</th>
<th>Hexane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzophenone</td>
<td>Insoluble</td>
<td>Soluble in about 25 sec</td>
<td>Soluble in about 60 sec</td>
</tr>
<tr>
<td>Malonic acid</td>
<td>Soluble in about 10 sec</td>
<td>Soluble in about 10 sec</td>
<td>Insoluble</td>
</tr>
<tr>
<td>Biphenyl</td>
<td>Insoluble</td>
<td>Partially soluble</td>
<td>Soluble in about 40 sec</td>
</tr>
</tbody>
</table>

Part B (expected results)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Water</th>
<th>Hexane</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-Octanol</td>
<td>Insoluble</td>
<td>Soluble</td>
</tr>
<tr>
<td>1-Butanol</td>
<td>Partially soluble</td>
<td>Soluble</td>
</tr>
<tr>
<td>Methanol</td>
<td>Soluble</td>
<td>Insoluble</td>
</tr>
</tbody>
</table>

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

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.

**Experiment 2**

**CRYSTALLIZATION**

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

CHEMICALS AND SUPPLIES PER 10 STUDENTS:

**Part A**

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

- 95% Ethyl alcohol 250 mL

- Filter paper for Büchner 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.