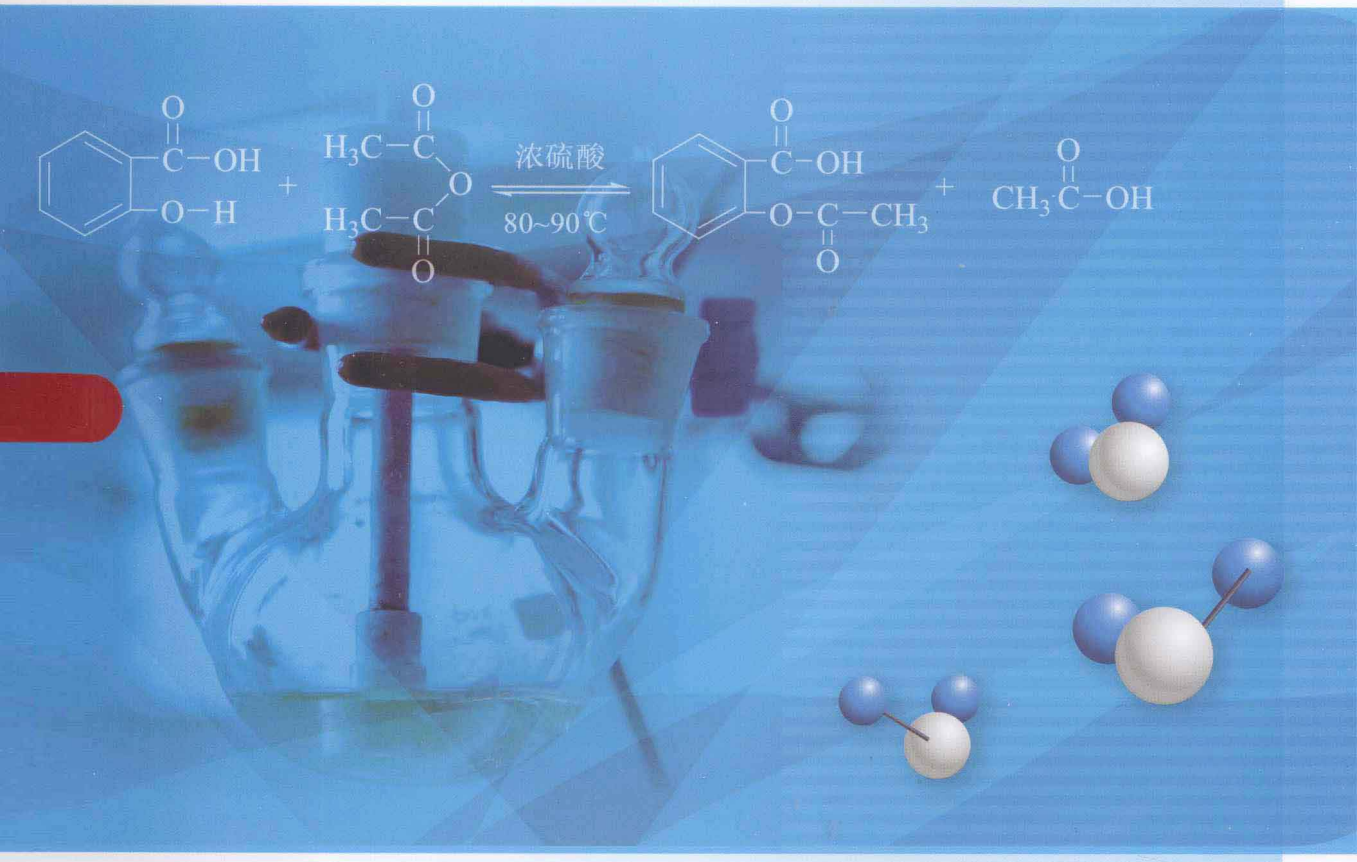
高等学校“十二五”规划教材

有机化学实验

Experimental Organic Chemistry

（英汉双语教材）

陈彪　魏永慧　编



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·北京·

本书是编者在总结多年来有机化学实验的教学经验及近年来实施双语教学实践的基础上，参考了国内外出版的同类教材，采用英汉两种语言，编写的与有机化学双语课程配套使用的《有机化学实验》教材。

全书包括：有机化学实验的一般知识，有机化学实验的基础知识和基本操作，有机化学综合实验（包括合成及分离提取两大部分），附录及参考文献。

本书可使学生在掌握有机化学实验基本原理、基本技能的同时，提高英语实际应用能力和水平，适用于化学、医学、药学及相关专业的学生使用，也可作为有机化学及相关专业硕士研究生的实验参考书。

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前　言

为了适应高等教育事业的快速发展，满足21世纪高等教育教学改革发展的需要，全面落实教育部提出的本科教学质量工程，进一步提高学生动手能力、实践技能及英语水平，提高教育教学和人才培养质量及人才的国际竞争能力，我们在总结多年来有机化学实验的教学经验及近年来实施双语教学实践的基础上，参考了国内外出版的同类教材，注重实验的小量化、绿色化，采用以英文为主、中英文相对照的方式，编写了这本与有机化学双语课程配套使用的《有机化学实验》英汉双语教材。

本书在编写过程中努力体现“反映特色，加强基础，注意交叉，够用为度，注重能力培养”的现代课程建设理念。

书中对基本操作和实验方法用英语作了较为详细精炼的描述。为了加强基本实验技能的训练，加深学生对实验原理和实验操作的理解，在有关章节中均附有较为详细的注释、思考题，以便于教学或学习。本书中多数的合成、提取与分离实验是编者多年来教学研究、科学研究与实践所形成的较成熟的实验，教学效果良好，对某些毒性较大、内容陈旧的传统实验项目进行了改革和更新。

在内容和结构安排上，从有机化学实验的基本知识入手，采取循序渐进的方式，主要包括有机化学实验基础知识、基本操作、综合实验三大部分。教材中的综合实验可加深学生对本门课程教学内容的全面了解和掌握，有利于学生综合素质和创新能力的培养。

本书在编写过程中参考了部分国内外出版的同类教材，在此深表感谢。

由于编者水平有限，书中难免有疏漏、欠妥之处，敬请各位专家、老师和读者批评赐教。

编者

2013年2月

目　录

Chapter 1 Introduction of Experimental Organic Chemistry

Experimental organic chemistry is an integral and basic part of the organic chemistry course. With the coming of new techniques, this course is being directed towards the development of small-scale experiments, high-efficient operations and the use of environment-friendly chemicals. The purpose of this course is to provide an opportunity to observe the reality of compounds and reactions, learn something of the operations and techniques that are used in experimental organic chemistry and in other areas, and further understand the basic principles of organic chemistry. Students should get into the habit of "preparation (pre-lab)-experiment-recording (in-lab)—summary (post-lab)", and rigorous scientific attitude, and work style.

1.1 Basic Rules for the Organic Chemistry Lab

In order to ensure all experiments go smoothly and laboratory safety is observed, students must abide by the following rules when entering into an organic lab:

① Familiarize yourself with the safety rules for lab work and learn about how to correctly use water, power, gas, hood, fire extinguisher and so on. Get to know what to do in the event of experimental accidents. Everyone, before doing the experiment, should be well prepared, understand the hazardous nature and safe usage of chemicals and promote safety consciousness. The experimental instruments and equipment must be used with care, adhering to their operating procedures. Report all abnormal conditions to your instructor to minimize the operational hazards.

② Before doing an experiment, check all glass equipment. During the experiment, use it carefully and skillfully; after the experiment, clean it up and keep it in order.

③ In the experiment, keep your experimental area and the whole lab tidy, operate with care, and adhere to the experimental procedures as well as reagent specifications and dosage required in every experiment. If you want to make any change, ask your instructor to get authorization. Never leave an ongoing experiment unattended.

④ Before using chemicals, read their labels carefully. Use them only as required in the experiment. Cover the stopper of the container immediately after use, and avoid the stoppers being confused as well as the chemicals being contaminated. Don't leave a mess for someone else to clean up. Don't change the position at random of normal reagents and common instruments in the lab such as balances, desiccator, refractometer and so on.

⑤ Your full attention must be given to what you are doing during the experimental period. Don't be careless or clown around in lab. You may hurt yourself or other people. Don't

speak loudly or eat or drink in the lab.

⑥ In-lab or post-lab, all kinds of solid or liquid waste should be placed in various authorized containers.

⑦ Before leaving lab, check carefully whether water, power and the gas are switched off safely, and wash your hands thoroughly with soap and water.

1.2 Common Lab Equipment and Apparatus

1.2.1 Lab Equipment

A typical set of lab equipment including glassware with standard-taper ground glass joints and non-glass equipment is shown in Figure 1.1～Figure 1.4.

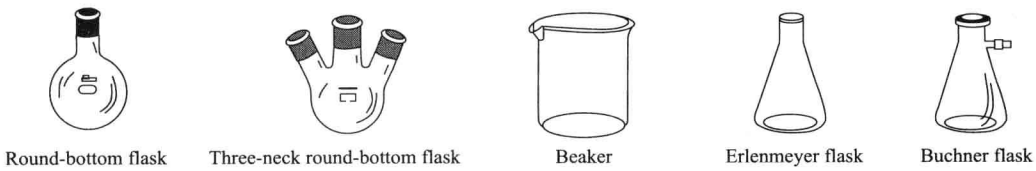
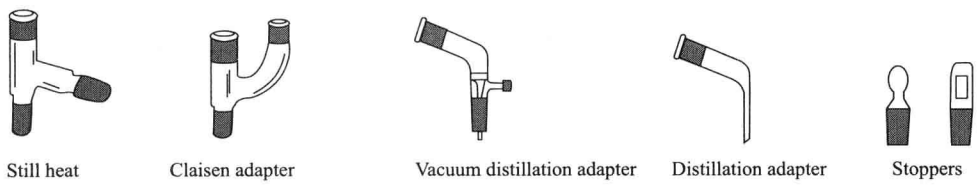


Figure 1.1 Lab Equipment (Ⅰ)



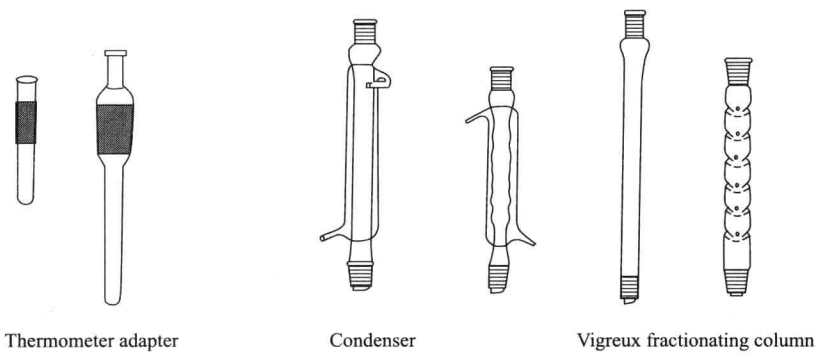


Figure 1.2 Lab Equipment (Ⅱ)

Round-bottom flask is used for distillation, reflux; three-neck round-bottom flask is used for more complicated reaction set-ups (two-neck flask are also available); Beaker is used for heating or mixing; Flask is used for titration, crystallization, preparation; Buchner flask is used for collecting the filtrate.

Still heat is used for distillation; Various adapter is used for distillation, vacuum distillation; Condenser is used for distillation; Air condenser is used for distillation with high boiling liquids; Vigreux fractionating column is used for fractional distillation.

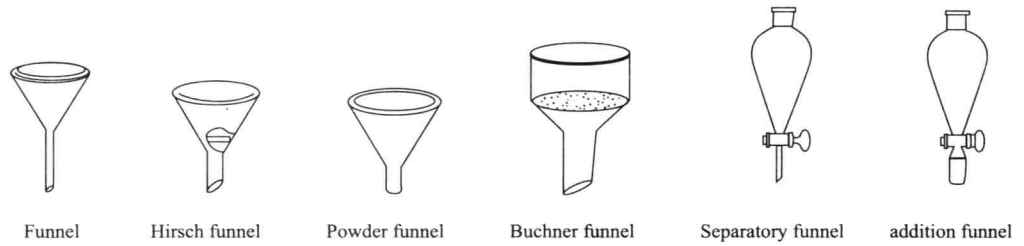


Figure 1.3 Lab Equipment (Ⅲ)

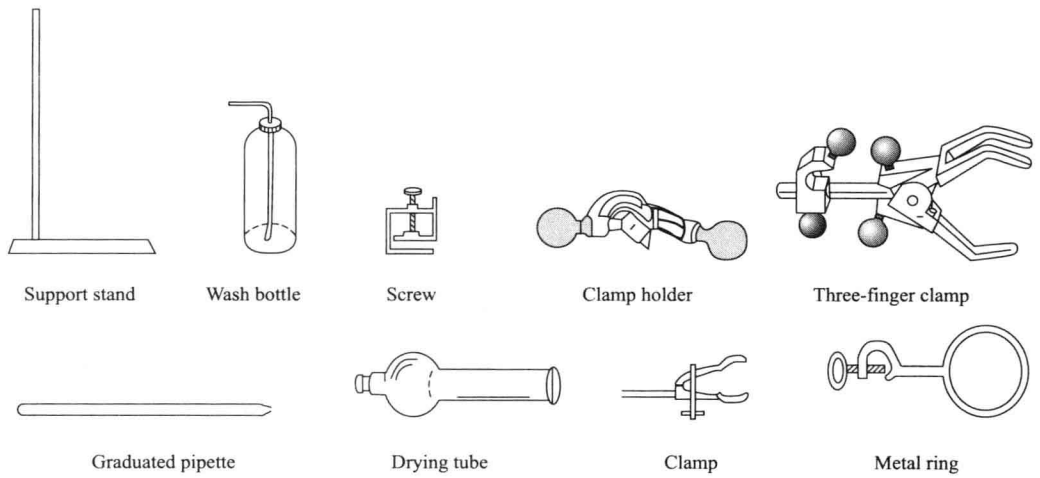


Figure 1.4 Lab Equipment (Ⅳ)

Various funnel is used for different filtration; Addition funnel is used for adding liquids; Separatory funnel is used for extraction and reaction.

Drying tube is used for drying gases. Support stand, clamp holder and clamp are all used for fixing.

1.2.2 Common Apparatus

All kinds of common apparatus are shown in Figure 1.5～Figure 1.11.

The rotary evaporator is used for the removal of volatile solvent from solution, leaving behind the non-volatile component (see Figure 1.10).

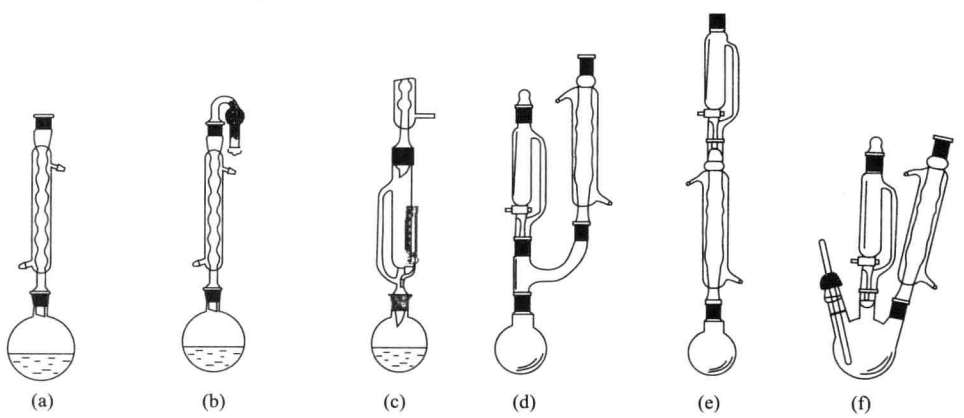


Figure 1.5 Reflux Apparatus

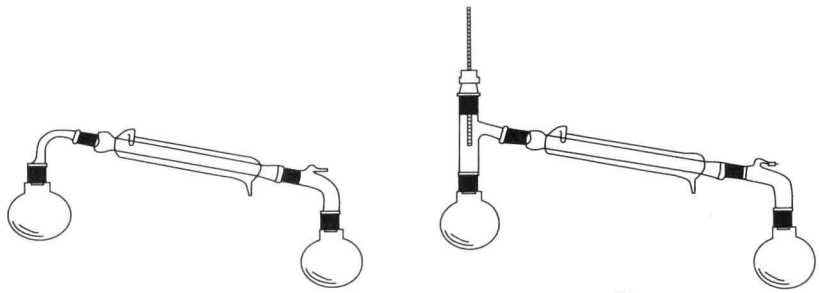


Figure 1.6 Distillation Apparatus

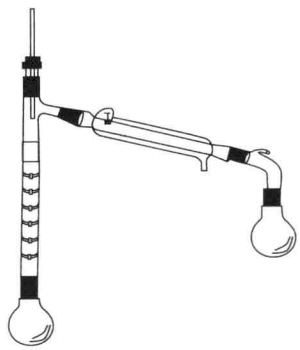


Figure 1.7 Fractional Distillation Apparatus

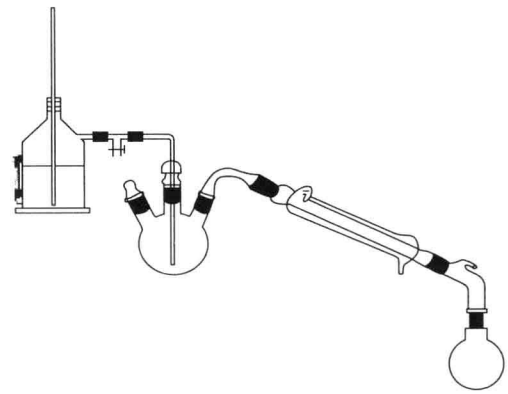


Figure 1.8 Steam Distillation Apparatus

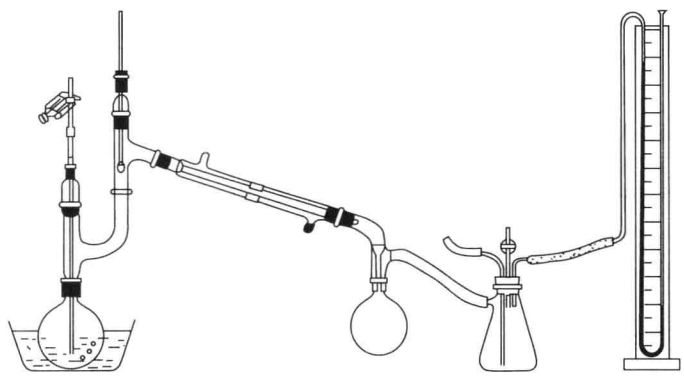


Figure 1.9 Vacuum Distillation Apparatus

1.2.3 Notes of using glassware

① All should be used carefully, avoiding impact or breakage.

② Don't heat directly except the beaker, flask and tube.

③ Flask and flat-bottom flask cannot withstand reduced pressure and should not be used in such systems.

④ After cleaning up glassware containing a stopper, a small piece of paper must be put between the stopper and ground joint to avoid adhesion.

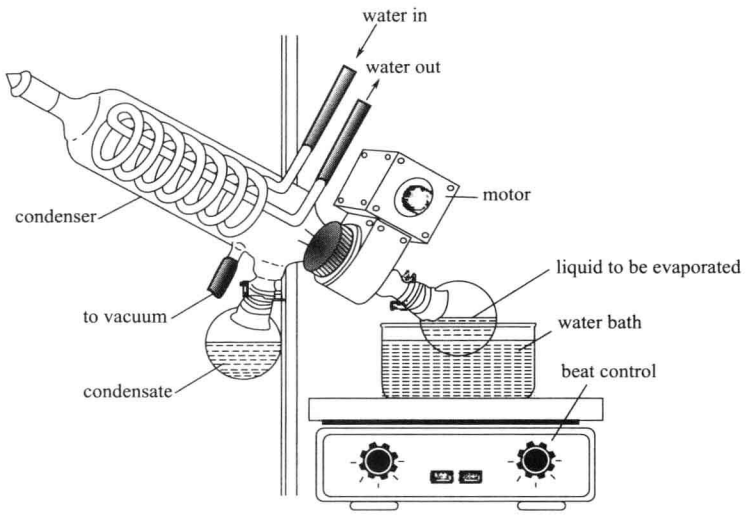


Figure 1.10 A Rotary Evaporator with Condenser and Receiving Flask

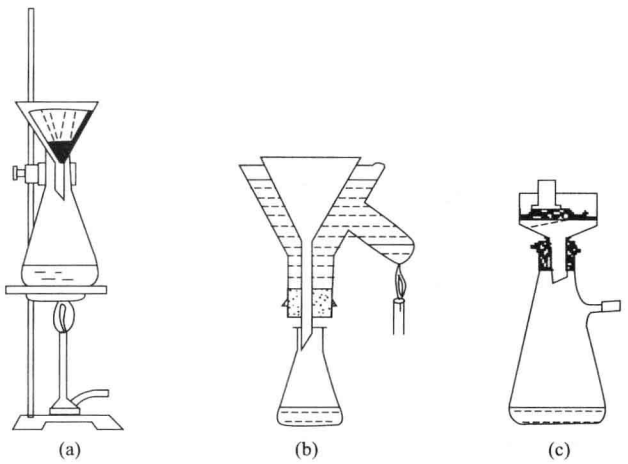


Figure 1.11 Heat Filtration and Vacuum Filtration

⑤ The glass of a mercury bulb is thin and ease-to-break, thus should be used with care. Never use it as a stirring rod. After use, cool it down, and rinse it afterwards to keep it from cracking. The measurement of the thermometer doesn't go beyond its graduated range.

1.2.4 Notes of assembling apparatus

① All glassware and accessories must be clean and fitted properly.

② When assembling the apparatus follow the principle of "bottom-to-top, left-to-right", step by step.

③ When disassembling, observe the rule of "right-to-left, up-to-down", one by one.

④ A reaction apparatus under the ordinary pressure must have an opening to the atmosphere to avoid development of a dangerously high pressure within the system when heat is applied.

⑤ All experimental apparatus must be tight, right, tidy and safe. All ground-glass joints should be connected snugly.

1.2.5 Cleaning Glassware

Always wash your glassware at the end of the experiment with water and either detergent or a mild scouring powder using an appropriate brush to remove most organic chemicals adhered to the glass walls. The inside and outside of all pieces of apparatus should be scrubbed and rinsed thoroughly with water afterwards. The final rinse can also be done with distilled or deionized water as required. Sometimes an ultrasonic oscillators might be useful for cleaning.

Never use chemical reagents or organic solvents thoughtlessly to rinse glassware. This may produce waste, and create a hazardous situation, resulting in additional pollution to our environment.

1.2.6 Drying Glassware

The common methods of drying glassware are as follows:

(1) Air dry In order to let water stream down, the glassware can be left upside down on a drying rack to dry.

(2) Oven dry The glassware can be dried quickly by placing it in an oven. For complete drying, glass should be left in an oven at 110～120℃ for several hours. Besides this, an air flow drier or hair drier also can be used.

(3) Organic solvent dry When wet glassware must be dried quickly for immediate use, it may be rinsed with small amounts of organic solvent such as 95% ethanol or acetone, which must be drained into an assigned bottle after use. Use a hair drier to evaporate the solvent afterwards.

Experiment: Practice on Simple Glass Working

Cutting and bending a glass tube and rod as well as preparing a capillary are all the primary operations in organic experiment.

(1) Cutting a Glass Tube and Rod

① Place the glass tube (or rod) flat on the table. Hold the tube firmly and create a scratch on the glass wall surface by drawing a tiny sand wheel or three corner file perpendicularly across it a couple of times (Do not saw back and forth. A short, single sharp scratch is more likely to produce a clean even edge. At the same time, keep the tube dry).

② Hold the tube in both hands, one on each side of the scratch. Keep your thumbs as close as possible to the scratch away from your body. Apply gentle pressure with your thumbs behind the scratch and push with the fingers in the direction of its length. The glass tube should break cleanly at the scratch (Keep them away from your eyes when broken).

③ Light the burner and get a hot blue flame. Pass one end of tube or rod through the

flame a few times, rotating the tube until the rough edges become smooth[1].

(2) Bending Glass Tube

① Light the burner and open the air hole. This gives a hot blue flame. Using both hands, rotate the dry tube in a declining position back and forth through the top of the flame.

② Keep rotating the tube until it glows red and has become soft. Then take the tube out of the flame and bend to the desired angle.

③ Don't twist the tube while heating and bending in order to ensure that the two ends of the bent tube remain in the same plane.

④ It is necessary to get a smooth bend without any kinks or constrictions. Check whether the bend is at the required angle (e. g. 90°) and whether the whole tube is in one plane[2] after the tube is bent. Then put it on the asbestos mat to cool (Don't put it on the table directly).

(3) Procedure

① Take several used tubes to practise the basic operation of cutting, heating, rotating and bending, to improve your glassworking skills.

② Preparation of a stirring rod: take a glass rod of 5mm diameter and 20cm length. Heat the two ends of the tube respectively in a hot blue flame until each end of it becomes round shaped.

③ Bending the tube: take two tubes of 5mm diameter, and bend them into 90°and 70° tubes respectively[3].

Notes

[1] Do not heat too much and do not stop rotating. Place the hot glass on an asbestos pad to cool. When the tube is cool, fire polish the other cut end. Again allow to cool. Be careful of the rough edges of the tube which sometimes may cut your hands

[2] After bending the tube, anneal it in a small flame and then put it on asbestos pad directly

[3] The length of the glass tube is changeable as required.

1.3 General Lab Safety

Generally speaking, organic experiments utilize mainly glassware, chemicals and electrical appliances, all of which can do harm to the human body and the environment if used improperly. Chemicals are hazardous because of their flammable, explosive, volatile, corrosive and toxic properties. Also, there is the possibility of experimental accidents to glass equipment and electrical appliances if operated incorrectly. Therefore, organic lab is potentially one of the most dangerous locations for students.

1.3.1 Fire-proof

The experimental operation must be normalized and the apparatus must be assembled correctly. Flammable, explosive and volatile chemicals mustn't be discarded randomly and

must be recovered specifically after the experiment. They should be kept away from an open flame. In case of a fire, first of all, cut power and the gas off, move the flammable and explosive reagents away, and then put the fire out in a proper way using a fire extinguisher, asbestos cloth, covering with sand, or rushing water and so on.

1.3.2 Explosion-proof

The apparatus should be assembled correctly. The whole system should not be made tight in the process of normal distillation and reflux. Distillation to dryness is also a dangerous practice because of the possible presence of peroxides or other explosive materials in the dry residue in the flask. The glassware and apparatus should be checked first to determine whether it can withstand the system pressure before vacuum distillation. If you don't add any boiling chips when starting distillation, stop heating immediately and re-add them after cooling. Keep the cooling water moving smoothly during distillation.

A fierce explosion or combustion can be produced when some organic compounds come into contact with oxidizers. Beware of their handling and storage.

1.3.3 Poisoning-proof

There are different ratings of toxicity among most organic reagents. The experiment with an irritant or toxic gas discharged must be always carried out in a hood or in a well-ventilated circumstance, or using a gas trap.

The manipulation of toxic or corrosive chemicals should follow the designated procedures strictly. Don't touch or come into contact directly with them. Keep them away from your mouth or cuts or abrasions of the skin, and never pour them into the sewer.

If you have some poisoning symptoms such as dizziness, headache, or other symptoms during the experiment you should leave the laboratory area and move to an area where you can breath fresh air and rest. In case of the poisoning is severe or symptoms persist, you should receive medical treatment.

1.3.4 Prevent Chemical Burns

Chemicals such as strong acids, strong bases, bromine, etc, should be used with great care in order to avoid contact with your skin which could cause chemical burns. In case of such an accident, wash the affected area immediately with copious amounts of running water, and then further treatments as follows.

Acid-injury: use 1% $NaHCO_3$ solution for the eye-wash and 5% $NaHCO_3$ solution for skin-wash.

Base-injury: use 1% boric acid for the eye-wash and 1%～2% acetic acid for skin-wash.

Bromine-injury: wash immediately with alcohol, and smear with glycerol or coat with a scald ointment.

If the situation is severe, go to hospital after first aid.

1.3.5 Cuts and Scalds

An accident involving cut or scald occurs in the use of glassware or manipulation of glassware if operated improperly. In case of such an accident, deal with it by the following methods.

Cuts: Cuts from broken glass are a constant potential hazard during experiments. The cut should be rinsed thoroughly with running water or hydrogen peroxide for a while to ensure that all tiny pieces of glass are removed. After this, wipe the cut with merbromin and bind up with gauze; if the cut is severe, first bind up with gauze and then send the patient to the hospital.

Scalds: Smear some scald ointment on the affected area if the situation is just a bit superficial; coat with scald ointment and go to the hospital for further treatment if the situation is severe.

1.4 Disposal of Lab Waste

Experimental operations always generate different kinds of solid or liquid waste. Waste disposal has been one of the major environmental problems of modem society. Special measures should be taken to observe national regulations and local organic lab rules of waste disposal. The handling of such wastes in the lab can be done in the following way:

① All waste generated in the lab can be classified into solid or liquid waste, and hazardous or nonhazardous waste, and disposed of properly. Some hard-to-handle hazardous waste should be delivered to the environmental department for special treatment.

② Small amounts of acids such as hydrochloric, sulfuric, and nitric, or bases such as sodium or potassium hydroxide, should be neutralized first and diluted with large amounts of water before flushing down the drain.

③ Organic solvents should be poured into properly labeled waste containers and stored in a well-ventilated place.

④ Nonhazardous solid waste such as paper, broken glass, corks, alumina, silica gel, magnesium sulfate, calcium chloride, and so on, should not be blended with other hazardous waste, and can probably go into the ordinary dustbin. Hazardous solid waste should be disposed of in a labeled container. The exact name of the contents should be written on the label.

⑤ Chemicals that react violently with water should be decomposed in a suitable way in a hood before disposal.

⑥ Some carcinogens and substances suspected of causing cancer must handled with great care, avoiding contact with your body.

1.5 Common Organic Solvents

(1) Ethyl Ether

Additional name(s): Ethoxyethane; ether; diethyl ether; ethyl oxide; diethyl ox-

ide. Molecular formula: $C_4H_{10}O$. Molecular weight: 74.12. Elemental analysis: C 64.82%, H 13.60%, O 21.59%. Line Formula: $C_2H_5OC_2H_5$. Properties: Mobile, very volatile, highly flammable liquid; Explosive! Characteristic, sweetish, pungent odor; Burning taste. Tends to form explosive peroxide under the influence of air and light, esp. when evaporation to dryness is attempted. Peroxides may be removed from ether by shaking with 5% aq ferrous sulfate solution. Addition of naphthols, polyphenols, aromatic amines, and aminophenols has been proposed for the stabilization of ethyl ether. d_4^{20} 0.7134; m. p. −116.3℃ (stable crystals); b. p. 34.5℃; Flash point, closed cup: −49℃. Ether is slightly sol in water and water is slightly sol in ether. Miscible with lower aliphatic alcohols, benzene, chloroform, other fat solvents, many oils. Caution: Keep away from fire; Potential symptoms of overexposure are dizziness; drowsiness; headache, excitedness and narcosis; nausea, vomiting; irritation of eyes, upper respiratory system and skin.

(2) Ethyl Alcohol

Additional name(s): Ethanol; absolute alcohol; anhydrous alcohol; dehydrated alcohol; ethyl hydrate; alcohol. Molecular formula: C_2H_6O. Molecular weight: 46.07. Elemental analysis: C 52.14%, H 13.13%, O 34.73%. Line Formula: CH_3CH_2OH. Properties: Clear, colorless, very mobile, flammable liquid; pleasant odor; burning taste; absorbs water rapidly from air. d_4^{20} 0.789; b. p. 78.5℃; m. p. −114.1℃; Flash point, closed cup: 13℃. n_D^{20} 1.361. Miscible with water and with many organic liquids. Caution: Keep tightly closed, cool, and away from flame!

(3) Benzene

Molecular formula: C_6H_6. Molecular weight: 78.11. Elemental analysis: C 92.26%, H 7.74%. Properties: Clear, colorless, volatile, highly flammable liquid; characteristic odor. d_4^{15} 0.8787; b. p. 80.1℃; m. p. 5.5℃; n_D^{20} 1.5011; Flash point, closed cup: −11℃. Slightly sol in water. miscible with alcohol, chloroform, ether, acetone, glacial acetic acid, carbon disulfide, oils. Caution: Keep in well-closed containers in a cool place and away from fire. Potential symptoms of overexposure by inhalation or ingestion are dizziness, headache, vomiting, visual disturbances, staggering gait, hilarity, fatigue, CNS depression, and loss of consciousness, respiratory arrest. Chronic exposure has been associated with bone marrow depression and leukemia. Direct contact may cause irritation of eyes, nose, respiratory system and skin; dermititis may develop due to defatting action. Aspiration into the lung may lead to chemical pneumonitis.

(4) Toluene

Addition name(s): Methylbenzene; phenylmethane. Molecular formula: C_7H_8. Molecular weight: 92.14. Elemental analysis: C 91.25%, H 8.75%. Properties: Flammable, refractive liq; benzene-like odor. d_4^{20} 0.866; m. p. −95℃; b. p. 110.6℃; n_D^{20} 1.4967; Flash point, closed cup: 4.4℃, Very slightly sol in water; miscible with alcohol, chloroform, ether, acetone, glacial acetic acid, carbon disulfide. Caution: Readily absorbed by inhalation ingestion and somewhat by skin contact. Direct contact may cause severe dermatitis due to drying and defatting action. May present lung aspiration hazard if ingested. Potential symptoms of acute overexposure by inhalation may include local initiation; CNS excitation and depression. Low concentrations may result in transitory

mild upper respiratory tract initiation, mild eye irritation, lacrimation, metallic taste, slight nausea, hilarity, lassitude, drowsiness and impaired balance. High concentrations may cause paresthesia, vision disturbances, dizziness, nausea, headache, narcosis and collapse; death from respiratory failure or sudden ventricular fibrillation. Chronic overexposure by inhalation has been associated with hepatotoxicity and nephrotoxicity. Syndromes following chronic inhalation involve severe muscle weakness, cardiac arrhythmias, gastrointestinal and neuropsychiatric complaints.

(5) Acetone

Additional name(s): 2-Propanone; dimethylketone; beta-keto propane; pyroacetic. Molecular fomula: C_3H_6O. Molecular weight: 58.08. Elemental analysis: C 62.04%, H 10.41%, O 27.55%. Line Formula: CH_3COCH_3. Properties: Volatile, highly flammable liquid; characteristic odor; pungent, sweetish taste. d_{25}^{25} 0.788; b. p. 56.5℃; m. p. −94℃; n_D^{20} 1.3591. Flash point, closed cup: −18℃. Miscible with water, alcohol, dimethylformamide, chloroform, ether, most oils. Caution: Keep away from fire! Keep away from plastic eyeglass frames, jewelry, pens and pencils, rayon stockings and other rayon garments.

(6) Methanol

Additional name(s): Methyl alcohol; carbinol. Molecular formula: CH_4O. Molecular weight: 32.04. Elemental analysis: C 37.48%, H 12.58%, O 49.93%. Line Formula: CH_3OH. Properties: Flammable, poisonous, mobile liq. Slight alcoholic odor when pure; crude material may have a repulsive, pungent odor. Burns with anon-luminous, bluish flame. d_4^{20} 0.7915; m. p. −97.81℃. b. p. 64.7℃; n_D^{20} 1.3292; Flash point, closed cup: 12℃. Miscible with water, ethanol, ether, benzene, ketones and most other organic solvents. Caution: Poisoning may occur from ingestion, inhalation or percutaneous absorption. Acute Effects: Headache, fatigue, nausea, visual impairment or complete blindness (may be permanent), acidosis, convulsions, mydriasis, circulatory collapse, respiratory failure, death.

(7) Ethyl Acetate

Additional name(s): Acetic acid ethyl ester; acetic ether. Molecular formula: $C_4H_8O_2$. Molecular weight: 88.11. Elemental analysis: C 54.53%, H 9.15% O 36.32%. Line Formula: $CH_3COOC_2H_5$. Properties: Clear, volatile, flammable liq; characteristic fruity odor; pleasant taste when diluted. Slowly dec by moisture, then acquires an acid reaction. Absorbs water (up to 3.3% w/w). d_4^{20} 0.902; d_{25}^{25} 0.898; b. p. 77℃; m. p. −83℃; Flash point 7.2℃ (open cup). Explosive limits (% vol in air): 2.2 to 11.5. n_D^{20} 1.3719. 1mL dissolves in 10mL water at 25℃; Miscible with alc, acetone, chloroform, ether. Caution: Keep tightly closed in a cool place and away from fire. Potential symptoms of overexposure are irritation of eyes, nose and throat, narcosis, dermatitis.

(8) Chloroform

Additional name(s): Trichloromethane. Molecular formula: $CHCl_3$. Molecular weight: 119.38. Elemental analysis: C 10.06%, H 0.845% Cl 89.09%. Properties: Highly refractive, nonflammable, heavy, very volatile, sweet-tasting liquid; characteristic odor. d_{20}^{20}