

英汉双语教材

上海市教育委员会高校重点教材建设项目

有机化学 实验

Experimental Organic Chemistry

季萍 薛思佳 主编
Larry Olson

33



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Experimental Organic Chemistry

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(英汉双语教材)

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北 京

内 容 简 介

本书依据有机化学实验双语教学的实践经验,采用英汉两种语言,结合国内外有机化学实验的教学实际情况编写而成。全书分四部分:有机化学实验的一般知识;有机化学实验的基本原理及操作;有机化合物的制备,文献实验,常用数据附录及思考题。

本书可使学生在掌握有机化学实验技能的同时,提高英语阅读能力和水平,适合化学及相关专业学生和教师使用及参考,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

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近年来,有机化学实验课程在教学内容、教学方法、教学手段上有了很大的变化。实验技术的更新、双语教学的实施不断地为这门传统的基础实验课程注入新的活力。我们在“上海市教委重点教材建设”专项基金和“上海市教委 21 世纪第二期课程建设”项目的支持下,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,参考了许多国内外出版的同类教材,并向国内外同行请教,注重有机实验的小量化、绿色化,以英文为主,中文相对应,编写完成了这本《有机化学实验》英汉双语教材。

本书对基本操作和实验方法用英文作了较为详细而精炼的描述。为了加强基本实验技能的训练,使学生加深对实验原理和实验操作的理解,本书在有关章节中均附有详细的注释、思考题、参考文献,以便于教学或学习。除了对薄层色谱、柱色谱、纸色谱、气相色谱、液相色谱、红外光谱、核磁共振谱等介绍外,绝大多数的有机合成实验中都附有红外光谱图。本书中涉及的合成实验皆是编者多年来教学研究与实践所形成的较成熟的实验,其中有些实验毒性较大,我们对其进行了改革;有些实验更新了内容,还有些则为完全创新(如:微波萃取等)。为拓宽教学内容,激发学习兴趣,本书还引入了具有微型化、特色化的文献实验。本书在写作过程中得到了美国 Arizona State University 的 Larry Olson 教授的大力帮助。他以丰富的教学经验和先进的教学理念,为本书提出了许多很好的建议,并编写与主审了本书的英文部分,在此向他致以深深的谢意。

本书得到“上海市教委重点教材建设基金”的资助,美国热电尼高力仪器公司为本书提供了所有的红外谱图,德国布鲁克公司为本书核磁共振氢谱的撰写提供了有关资料,部分高校的实验教师对本书的完成给予了大力支持,在此一并表示真诚的感谢。

由于作者水平有限,不当之处在所难免,敬请读者批评赐教。

编 者

2004 年 12 月

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Part 1 Introduction

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, data-processing with the computer, and further understand the basic principles of organic chemistry. Students should get into the habit of “preparation (pre-lab)—experiment and recording (in-lab)—summary (post-lab)”.

1.1 General Rules for the Organic Chemistry Lab

In order to ensure all experiments go smoothly and laboratory safety is observed, all 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 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.

1.2.1 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.

1.2.2 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.

1.2.3 Poisoning-proof

There are different ratings of toxicity among most organic reagents. The experiment with an irritative 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 breathe fresh air and rest. In case of the poisoning is severe or symptoms persist, you should receive medical treatment.

1.2.4 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.

1.2.5 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.

1.3 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 modern 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.

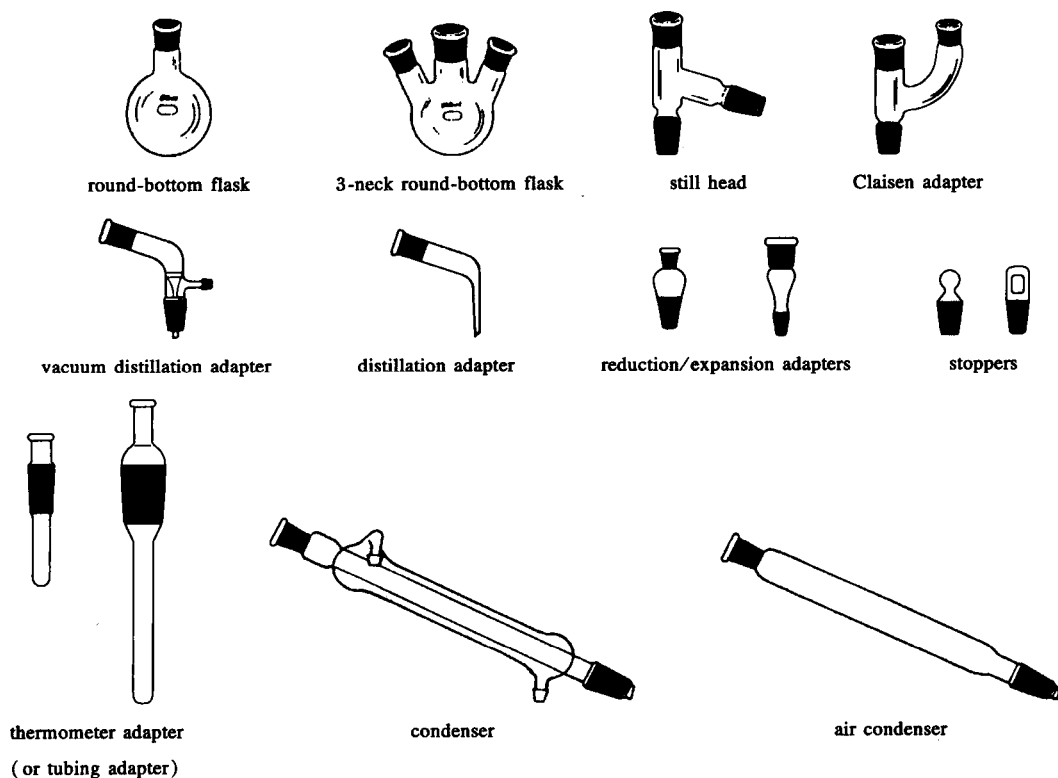
5. Chemicals that react violently with water should be decomposed in a suitable way in a hood before disposal.

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

1.4 Common Lab Equipment and Apparatus

1.4.1 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.



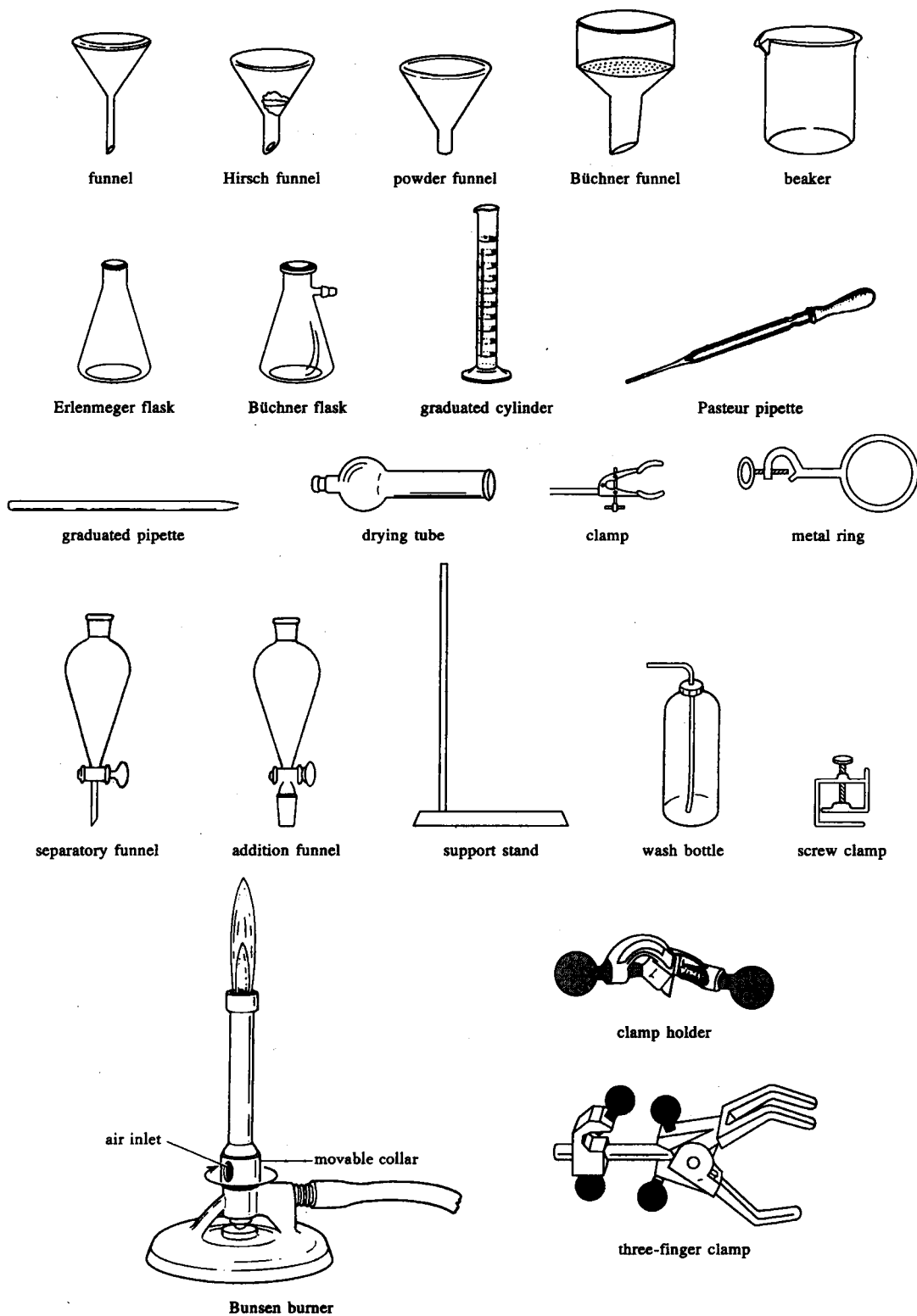


Figure 1.1 Common Organic Apparatus

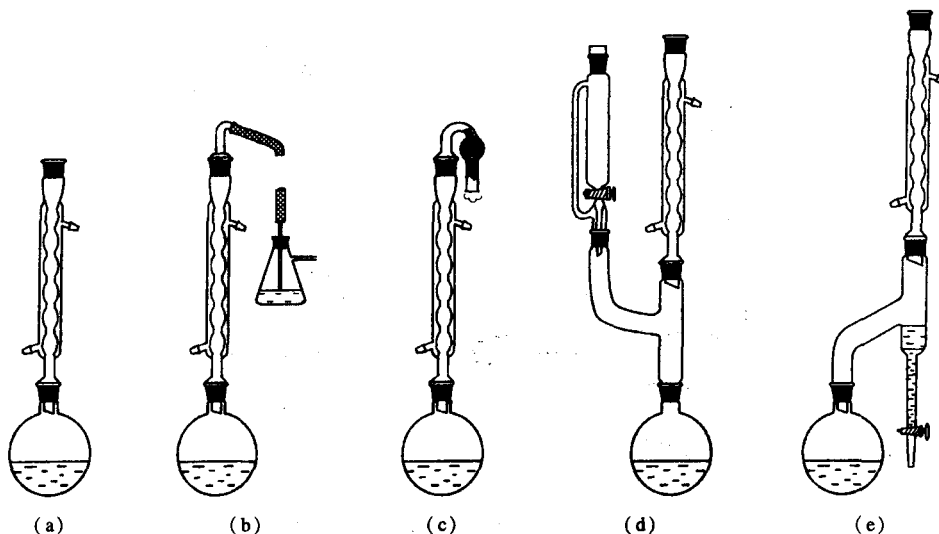
Note 1

Round-bottom flask for distillation, reflux;
 Three-neck flask for more complicated reaction set-ups (two-neck flasks are also available);
 Erlenmeyer flasks for titration, crystallization, preparation;
 Beakers for heating, mixing;
 Addition funnel for adding liquids;
 Separatory funnel for extraction and reaction work-up;
 Condenser for distillation;
 Air condenser for distillation with high boiling liquids;
 Drying tube for drying gases;
 Still head for distillation;
 Various adapters for distillation, vacuum distillation;
 Suction flask (filter flask) for collecting the filtrate.

Note 2

1. All should be used carefully, avoiding impact or breakage.
2. Don't heat directly except the beaker, flask and tube.
3. Erlenmeyer 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.
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.

1.4.2 Common Apparatus



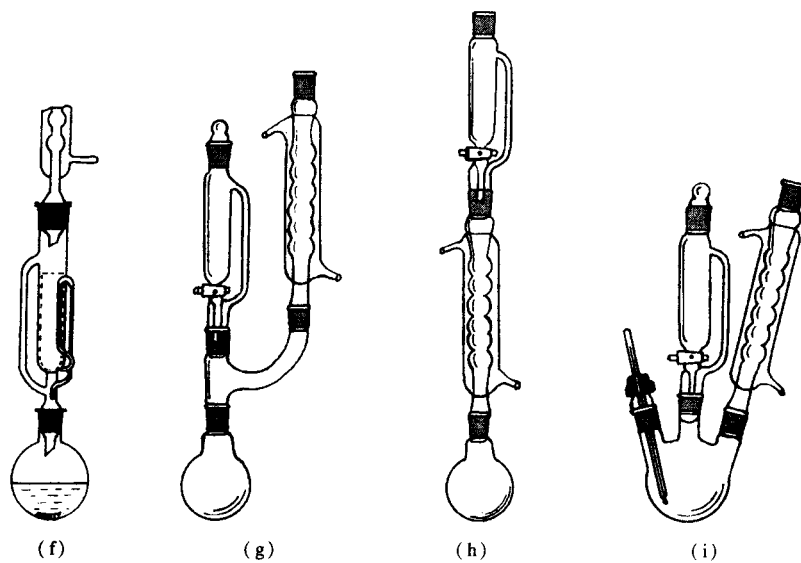
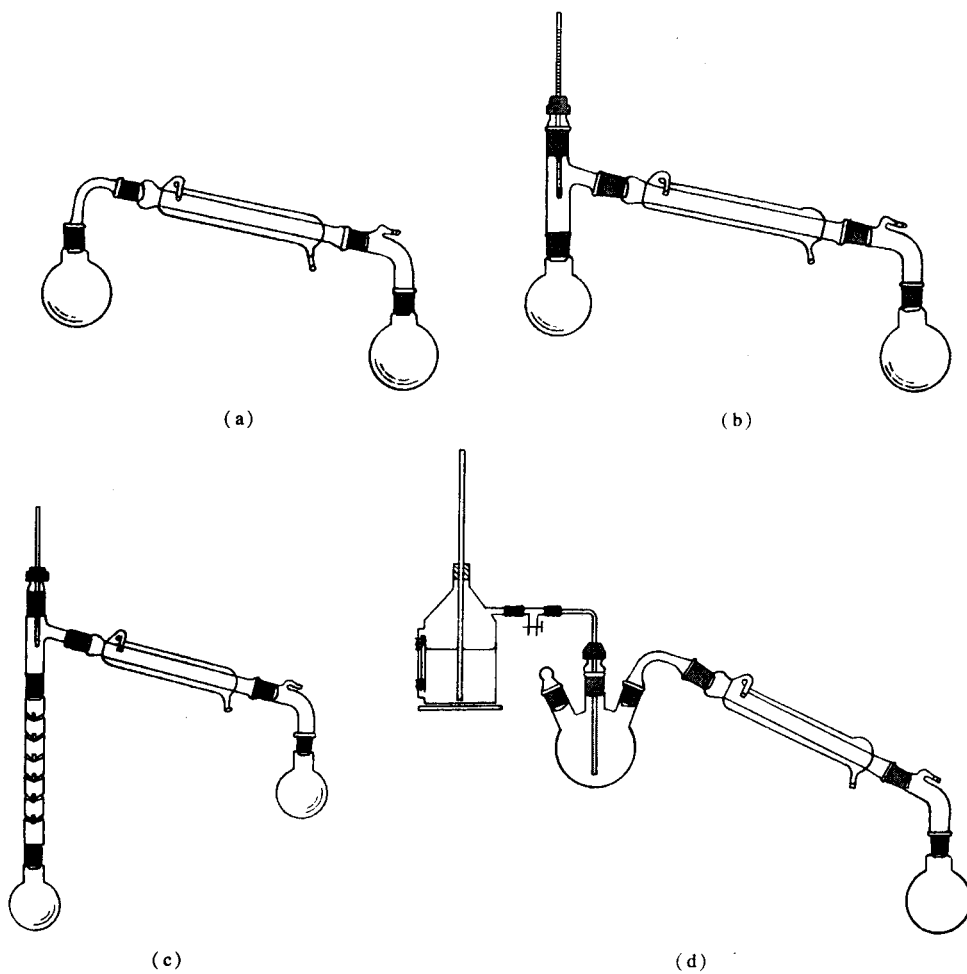


Figure 1.2 Reflux Apparatus



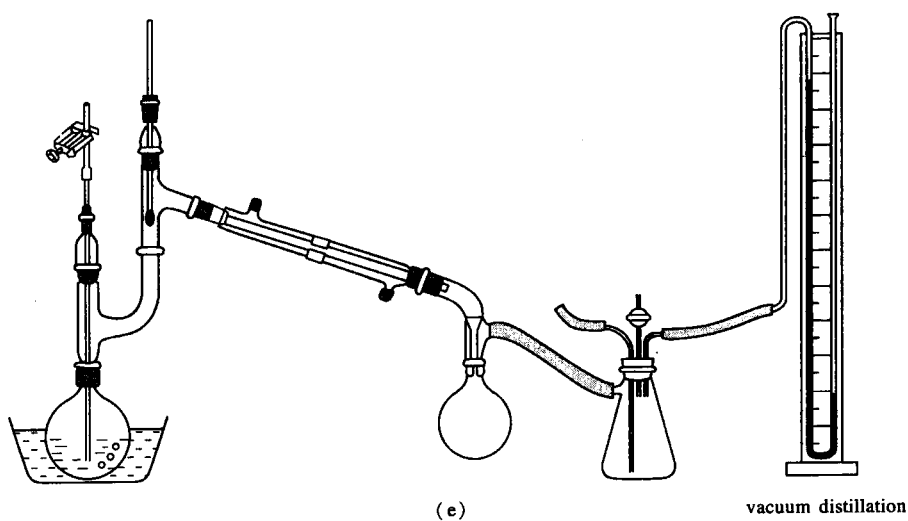


Figure 1.3 Distillation Apparatus

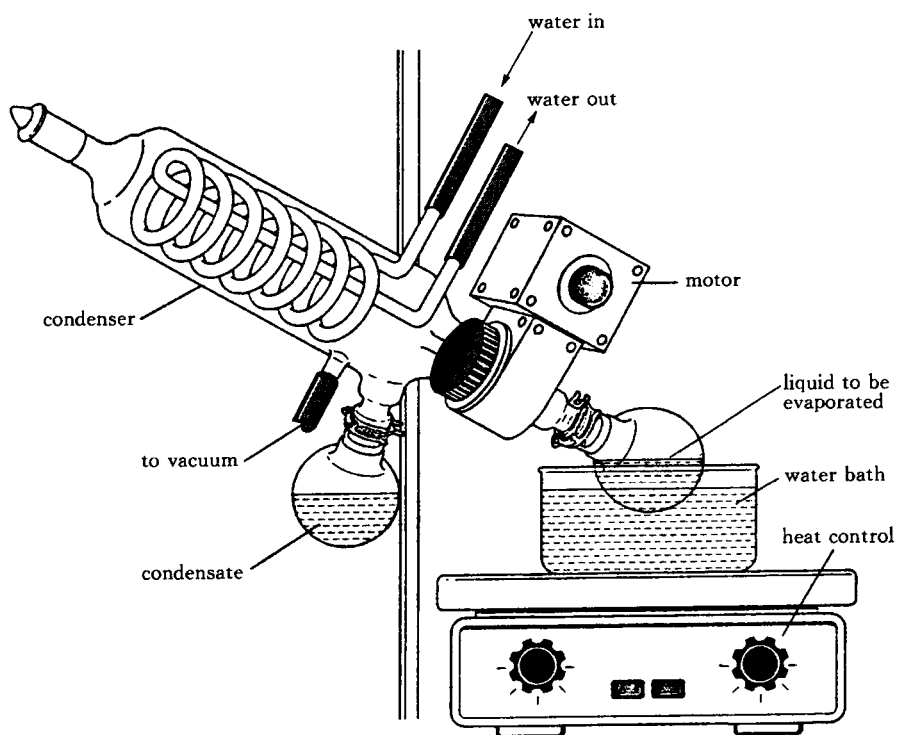


Figure 1.4 A Rotatory Evaporator with Condenser and Receiving Flask