普通高等教育“十二五”规划教材

有机化学实验

英汉双语版(第三版)

Experimental Organic Chemistry (Third Edition)

主　编　薛思佳　季　萍　Larry Olson
副主编　赵三虎　任新峰　朱　晔

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北　京

内 容 简 介

本书是科学出版社普通高等教育“十二五”规划教材“有机化学(第二版)立体化教材”的《有机化学实验(英汉双语版，第三版)》部分，与《有机化学(第二版)》和《有机化学学习指导(第二版)》配套出版，方便教学选用。

本书采用英汉两种语言，英文为主，中文相应对照，结合国内外有机化学实验教学的实际编写而成。全书分为6章：有机化学实验的一般知识、有机化学实验的基本操作、有机化合物的制备、综合性实验、文献实验和附录。附录编写了常用实验数据、与化学和有机化学有关的Internet网址及有机化学实验操作考试复习题。

本书可使学生在掌握有机化学实验技能的同时，提高专业英语的阅读能力和水平。可作为高等学校化学、化学(师范)、应用化学和化学工程与工艺专业，以及材料化学、生物技术、制药工程、食品、医药卫生、环境科学和园艺等相关专业的有机化学实验教材；也可以作为有机化学及相关专业硕士研究生的实验参考书；对备考硕士研究生的读者也是一本好的参考书。

图书在版编目(CIP)数据

有机化学实验=Experimental Organic Chemistry: 英汉双语版 / 薛思佳等主编. —3版. —北京：科学出版社，2016.9

普通高等教育“十二五”规划教材

ISBN 978-7-03-049693-5

Ⅰ. ①有… Ⅱ. ①薛… Ⅲ. ①有机化学-化学实验-高等学校-教材-英、汉 Ⅳ. ①O62-33

中国版本图书馆CIP数据核字(2016)第199924号

责任编辑：郭慧玲 / 责任校对：何艳萍

责任印制：赵 博 / 封面设计：迷底书装

科学出版社 出版

北京东黄城根北街16号

邮政编码：100717

http://www.sciencep.com

三河市骏杰印刷有限公司 印刷

科学出版社发行 各地新华书店经销

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2005年2月第 一 版 开本：787×1092 1/16

2007年8月第 二 版 印张：15 3/4

2016年8月第 三 版 2016年8月第七次印刷

字数：380 000

定价：48.00元

(如有印装质量问题，我社负责调换)

第三版前言

本书是科学出版社普通高等教育“十二五”规划教材“有机化学(第二版)立体化教材”的《有机化学实验(英汉双语版，第三版)》部分，与《有机化学(第二版)》和《有机化学学习指导(第二版)》配套出版，方便教学选用。

《有机化学实验(英汉双语版，第二版)》自 2007 年 8 月出版以来，由于其满足了普通高校实施双语教学的需要，因此受到高校教师和学生们的欢迎和肯定。编者在调研的基础上，根据多年来在使用过程中发现的不足和目前高校有机化学实验教学改革的需要，在第二版的基础上对本教材进行了修订。

为适应部分院校有机化学实验教学的实际需要，本书第 3 章增加了 4-硝基苯胺的制备，乙酰二茂铁的制备和生物法合成乙醇 3 个实验;第 4 章增加了从菠菜叶中提取和分离叶色素，从红辣椒中提取、分离和鉴定辣椒红素和 β-胡萝卜素 2 个实验；重新编写了附录中有机化学实验操作考试复习题。为适应网络教学的需要，更新和补充了英文部分 Chapter 1 的 Chemical Literature 以及中文部分附录中与化学有关的 Internet 网址。

本书由薛思佳、季萍、Larry Olson(Arizona State University，USA)担任主编，赵三虎、任新峰、朱晔担任副主编。参加本书编写与修订工作的有：上海师范大学薛思佳，上海工程技术大学任新锋，石家庄学院朱晔、冯海燕、贾会珍、朱云云，忻州师范学院赵三虎、翟保评、孙金鱼等教学一线教师。

由于编者水平有限，书中疏漏和不妥之处在所难免，敬请读者批评指正。欢迎使用，更期盼读者能够喜欢本立体化教材。

编　者

2016 年 5 月于上海

第二版前言

Experimental Organic Chemistry(《有机化学实验》,英汉双语版)自2005年2月出版以来,因为适应了当前普通高校实施双语教学的需要,受到了高校教师和学生们的欢迎与肯定。在广泛调研的基础上,根据两年多来使用中存在的不足和目前高校有机化学实验教学改革的需要,编者代表于2007年3月在上海针对本书的第一版内容进行了研讨,拟定了如下修改意见:

1)更正了第一版中出现的错误。

2)增加连续性和综合性实验内容,精选文献实验内容,适当增加基本操作部分和有机化合物光谱鉴定部分的内容。

3)删除了一些内容较为陈旧,实验药品或产品毒性较大,环境污染较严重的实验。增加了一些新的或有一定操作难度的实验。对同一类型的实验增加了可供选择的内容,使教材更具普适性。

4)对每个实验的中、英文进行适当的调整,力求做到既有利于本书第二版内容的编排,又有利于有机化学实验的教学与实践,又尽量使内容不重复。

5)为增加教学的信息量,附录中增加部分与有机化学有关的INTERNET网址。

本书由薛思佳、季萍、Larry Olson(Arizona State University, USA)主编。参加第二版修订工作的单位和教师为:上海师范大学:薛思佳,林静容,刘国华,肖海波;上海工程技术大学:宋小平,任新锋;石家庄学院:朱云云,贾会珍;忻州师范学院:赵明根,赵三虎;浙江台州师院:蒋华江;盐城师院:王庆东等同志。

第二版教材得到“上海市高校本科教育高地建设项目”的资助。赛默飞世尔科技(上海)有限公司分子光谱部(原美国热电尼高力仪器公司)为本书提供了红外光谱图。在此一并致谢。

限于编者水平,书中错误和不妥之处难以避免,敬请指正。

编　者

2007年6月于上海

第一版前言

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了新的活力。我们在“上海市教委重点教材建设”专项基金的支持下，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》中英文双语教材。

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、核磁共振谱等进行介绍外，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是我们多年来教学研究与实践所形成的较成熟的实验，其中有些实验对以往毒性较大的实验进行了改革，有些更新了实验内容，还有些则完全创新，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。他以多年的教学经验，为本书提出了许多很好的建议，并主审了本书的英文部分，在此向他致以深深的谢意。

第一版由季萍，薛思佳，Larry Olson（Arizona State University, USA）主编。编委会成员：季萍（上海工程技术大学），薛思佳（上海师范大学），张爱东（华中师范大学），宋小平（上海工程技术大学），邹建平（苏州大学），张凤琴（东华大学），潘健民（上海工程技术大学），徐永芬（东华大学）。

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，部分高校的实验教师对本书的完成给予了大力支持，对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

目　录

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Chapter 1 Introduction

Experimental organic chemistry is an integral and basic part of organic chemistry course. With the coming of new techniques, this course is being directed towards the development of small-scale experiment, high-efficient operation 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 computer, and further understand the basic principles of organic chemistry. Students should get into the habit of "preparation (pre-lab)—experiment and record (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 experiment, use it carefully and skillfully; after experiment, clean it up and keep it in order.

(3) In the experiment, keep your experimental area and 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 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 balance, 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 can hurt yourself or other people. Don't speak loudly and 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 experiment utilize mainly glassware, chemicals and electrical appliances, all of which can do harm for the human body and 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 is 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 smooth 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 breath 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 acid, strong base, 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 the 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, 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 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 environmental department for special treatment.

(2) Small amounts of acids, such as hydrochloric, sulfuric, and nitric, or base 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 can 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 Fig. 1.1.

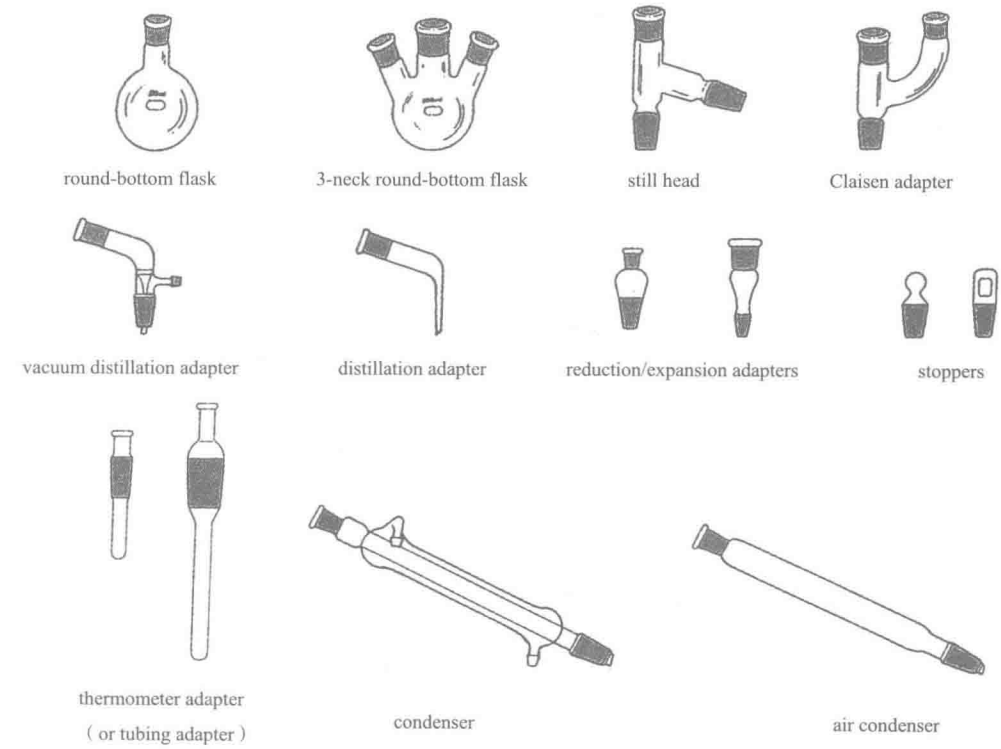

Fig. 1.1 Common Organic Apparatus

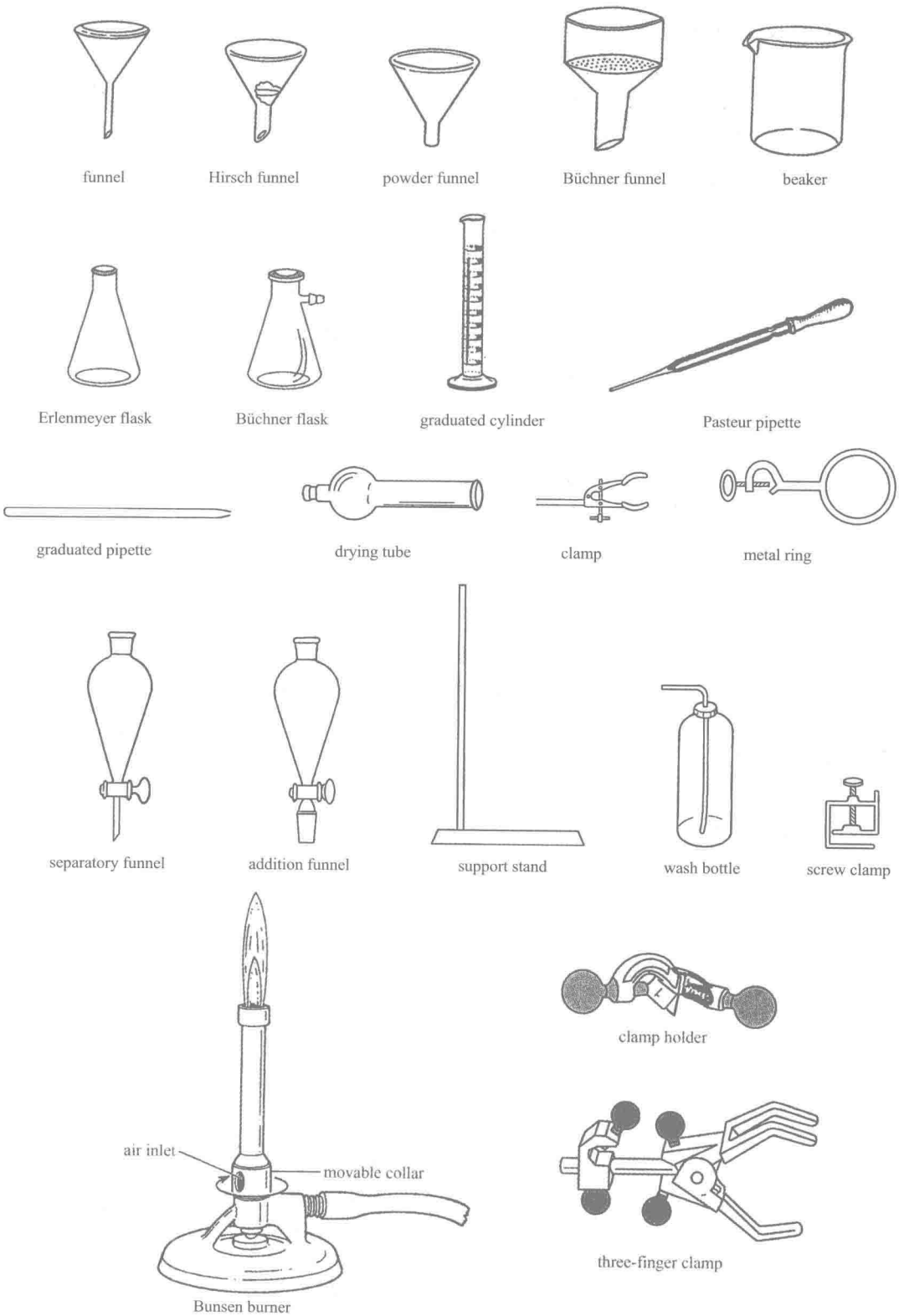

Fig. 1.1 (Continued)

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 away from cracking. The measurement of thermometer doesn't go beyond its graduated range.

1.4.2 Common Apparatus

Common apparatus are shown in Fig.1.2 to Fig. 1.7.

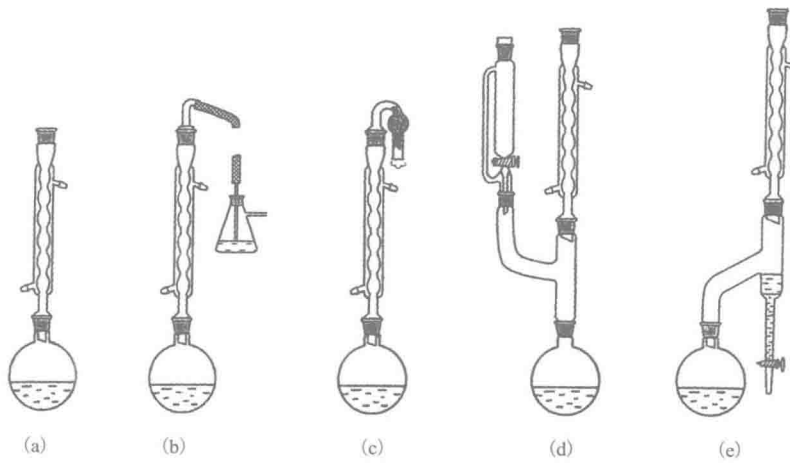

Fig. 1.2 Reflux Apparatus

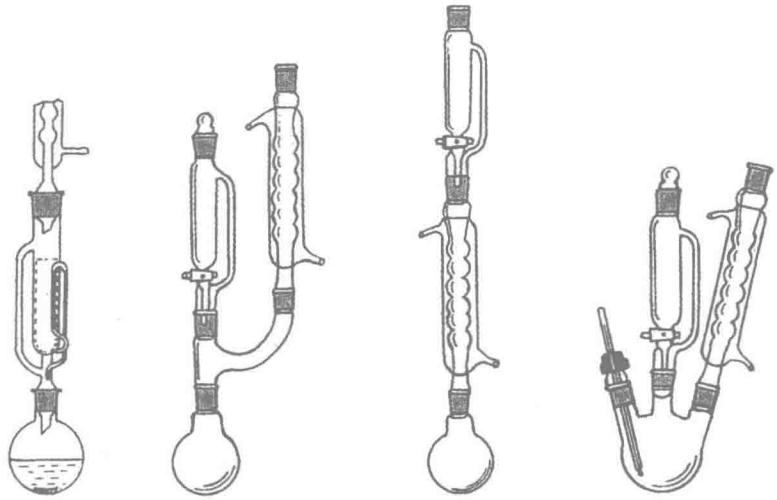

Fig. 1.2 (Continued)

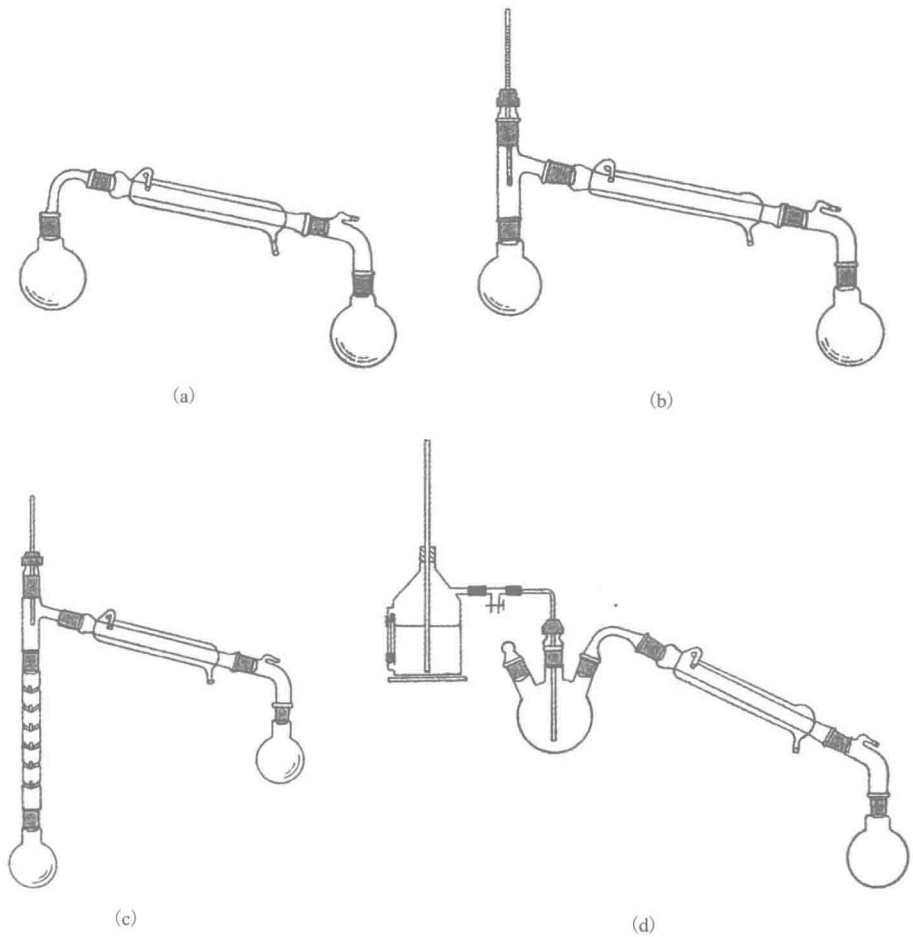

Fig. 1.3 Distillation Apparatus