

Organic Chemistry Experiment

有机化学实验

(英汉双语版)

主 编 柏一慧
副主编 朱钢国 吕 新
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北 京

内 容 简 介

本书根据目前我国高等学校双语教学的需要编写而成。全书共 4 章,包括有机化学实验的一般知识和基本操作、有机化合物制备实验和有机化合物性质实验。其中基本操作实验 10 个,制备实验 30 个,性质实验 7 个。实验还编有注释和思考题,重要的实验配有实验装置图和实验操作流程图。

本书可作为高等学校化学、化工及相关专业本科生的有机化学实验教材,也可作为相关专业学生攻读硕士学位或申请海外高校的实验参考书。

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《有机化学实验》(英汉双语版)

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前 言

随着我国“一带一路”倡议的提出，高校扩大了来华留学教育的招生规模；另外，国内各高校本科生毕业后申请海外留学深造的需要也日益增加，因而相应高校都纷纷推出了全英文授课的教学项目。在此背景下，适合我国高校教学体系的中英文双语教材的出版也提上了日程。目前国内有机化学实验的双语教材不多，难以满足教学需要；而国外的有机化学实验指导书与国内的实验教学内容匹配度不高，因此编者编写了这本双语教材。

本书采用英、汉两种语言，英文为先，中英文并重；参照国内各高校成熟的有机化学实验教材内容，结合浙江师范大学有机化学学科多年来的教学实践，同时参考国外重点高校有机化学实验指导书的英文内容编写而成。本书主要面向化学专业的有机化学实验中英文教学，以及国际化专业的发展需要，使学生掌握有机化学实验所涉及的规则、流程、方法、仪器、工艺、报告等基本内容，培养从事有机化学相关工作的基本能力和素养，并具有适应国际化发展的有机化学专业英语知识背景。

本书共4章，包括：有机化学实验的一般知识、有机化学实验基本操作、有机化合物制备实验、有机化合物性质实验。内容上从基础到应用，从简单到综合；制备实验所涉及有机化合物按国内大多数高校使用的有机化学教材中的编排顺序编写，尽可能使实验教学进度与理论课程进度相匹配，做到理论指导实践，实验促进理论理解。

本书重点在基础、实用、成熟的有机合成实验，尤其注重基本操作，特别强调实验关键步骤的把握及实验安全和环保部分的处理。重要实验项目中增加了详细、明了的实验操作流程图和实验装置图。同时根据浙江师范大学有机化学学科多年的教学积累，对现有教材的实验内容进行了修订，并增加了教学改革的部分内容，提高了实验的成功率和环保性。编者将继续完善本书配套的网络资源，包括电子课件、网站建设、在线答疑，以及视频、动画、实验操作实图等资源的建设。

本书由浙江师范大学有机化学学科组织编写，其中柏一慧担任主编并负责编写第1章和第2章；吕新负责编写第3章实验11~23；张岩负责编写第3章实验24~39；黄鑫负责编写第3章实验40和第4章；朱钢国负责全书内容的审定和实验的设计；浙江师范大学有机化学学科的其他教师均对本书做出直接贡献。本书同时得到了浙江师范大学“十二五”省级实验教学示范中心重点建设项目(软件建设项目)和浙江师范大学化学与生命科学学院教学建设项目的资助，在此一并表示真挚的感谢。

因时间有限，经验不足，书中不足之处在所难免，敬请专家和读者批评指正。

编 者

2019年1月

目 录

前言

Chapter 1	General Knowledge of Organic Chemistry Experiments	1
1.1	Rules for Organic Chemistry Laboratory	1
1.2	Safety Knowledge in the Organic Laboratory	2
1.3	Commonly Used Equipment and Apparatus of Organic Chemistry Experiments	6
1.4	Cleaning and Drying of Glassware	12
1.5	Heating, Cooling and Drying of Reagents in Organic Chemistry Experiments	13
1.6	Basic Requirements for Pre-lab Work, Lab Record and Lab Report	20
Chapter 2	Basic Operations of Organic Chemistry Experiments	25
Exp.1	Determination of Melting Point	25
Exp.2	Simple Distillation and Determination of Boiling Point	29
Exp.3	Recrystallization	32
Exp.4	Extraction	38
Exp.5	Vacuum Distillation	44
Exp.6	Steam Distillation	49
Exp.7	Fractional Distillation	54
Exp.8	Column Chromatography	57
Exp.9	Thin Layer Chromatography	60
Exp.10	Paper Chromatography	64
Chapter 3	Preparation Experiments of Organic Compounds	68
Exp.11	Preparation of Cyclohexene	68
Exp.12	Preparation of 1-Bromobutane	71
Exp.13	Preparation of Bromobenzene	75
Exp.14	Preparation of 2-Methyl-2-Butanol	77
Exp.15	Preparation of Diphenylmethanol	80
Exp.16	Preparation of 1, 1'-Binaphthol (a Reference-based Experiment)	82
Exp.17	Preparation of Di- <i>n</i> -butyl Ether	83
Exp.18	Preparation of Cyclohexanone	86
Exp.19	Preparation of Benzalacetone and Dibenzalacetone	87
Exp.20	Preparation of Benzoin	90
Exp.21	Preparation of Benzoic Acid	92
Exp.22	Preparation of Adipic Acid	94
Exp.23	Preparation of Cinnamic Acid	97
Exp.24	Preparation of Coumarin-3-carboxylic Acid	99

Exp.25	Preparation of Furfuryl Alcohol and Furoic Acid	102
Exp.26	Preparation of Ethyl Acetate	104
Exp.27	Synthesis of Dibutyl Phthalate	107
Exp.28	Synthesis of Ethyl Acetoacetate	109
Exp.29	Preparation of Acetylsalicylic Acid (Aspirin)	110
Exp.30	Preparation of Acetanilide	112
Exp.31	Preparation of <i>p</i> -Acetamidobenzenesulfonyl Chloride	115
Exp.32	Preparation of <i>p</i> -Aminobenzenesulfonamide	118
Exp.33	Green Synthesis of <i>N</i> -bromosuccinimide	120
Exp.34	Synthesis of Methyl Orange	121
Exp.35	The Preparation and Beckmann Rearrangement of Benzophenone Oxime	124
Exp.36	Preparation of Aniline	126
Exp.37	Preparation and Resolution of Racemic α -Phenylethylamine	129
Exp.38	Optical Resolution of <i>Trans</i> -1, 2-diaminocyclohexane	132
Exp.39	Preparation of 2, 3; 5, 6-Di- <i>O</i> -isopropylidene- α -D-mannofuranose	134
Exp.40	Isolation of Caffeine from Tea Leaves	136
Chapter 4	Reactivity Experiments of Organic Compounds	140
Exp.41	The Preparation and Reactivities of Ethylene and Ethyne	140
Exp.42	The Reactivities of Aromatic Compounds	143
Exp.43	The Reactivities of Some Alkyl Halides	145
Exp.44	The Reactivities of Alcohols and Phenols	148
Exp.45	The Reactivities of Aldehydes and Ketones	149
Exp.46	The Reactivities of Carboxylic Acids and Their Derivatives	151
Exp.47	The Reactivities of Amines	152
第 1 章	有机化学实验的一般知识	155
1.1	有机化学实验室规则	155
1.2	有机化学实验室的安全知识	155
1.3	有机化学实验常用仪器和装置	158
1.4	玻璃仪器的洗涤和干燥	163
1.5	有机化学实验中试剂的加热、冷却和干燥	165
1.6	实验预习、实验记录和实验报告的基本要求	169
第 2 章	有机化学实验基本操作	174
实验 1	熔点测定	174
实验 2	简单蒸馏及沸点测定	177
实验 3	重结晶	180
实验 4	萃取	184
实验 5	减压蒸馏	188
实验 6	水蒸气蒸馏	192
实验 7	分馏	196

实验 8 柱色谱	198
实验 9 薄层色谱	201
实验 10 纸色谱	204
第 3 章 有机化合物制备实验	207
实验 11 环己烯的制备	207
实验 12 1-溴丁烷的制备	210
实验 13 溴苯的制备	212
实验 14 2-甲基-2-丁醇的制备	215
实验 15 二苯甲醇的制备	217
实验 16 1,1'-联二萘酚的合成(文献实验)	218
实验 17 正丁醚的制备	219
实验 18 环己酮的制备	221
实验 19 苯叉丙酮和二苯叉丙酮的制备	222
实验 20 安息香的制备	224
实验 21 苯甲酸的制备	226
实验 22 己二酸的制备	228
实验 23 肉桂酸的制备	229
实验 24 香豆素-3-羧酸的制备	232
实验 25 呋喃甲醇和呋喃甲酸的制备	234
实验 26 乙酸乙酯的制备	236
实验 27 邻苯二甲酸二丁酯的制备	238
实验 28 乙酰乙酸乙酯的制备	240
实验 29 乙酰水杨酸(阿司匹林)的制备	241
实验 30 乙酰苯胺的制备	242
实验 31 对乙酰氨基苯磺酰氯的制备	245
实验 32 对氨基苯磺酰胺的制备	247
实验 33 <i>N</i> -溴代丁二酰亚胺的绿色合成	248
实验 34 甲基橙的制备	250
实验 35 二苯甲酮肟的制备及贝克曼重排	252
实验 36 苯胺的制备	253
实验 37 外消旋 α -苯乙胺的制备和拆分	255
实验 38 反-1,2-二氨基环己烷的手性拆分	258
实验 39 2,3;5,6-二- <i>O</i> -异丙叉基- α -D-呋喃甘露糖的制备	259
实验 40 从茶叶中提取咖啡因	261
第 4 章 有机化合物性质实验	264
实验 41 乙烯、乙炔的制备和性质	264
实验 42 芳烃的性质	266
实验 43 卤代烃的性质	268
实验 44 醇和酚的性质	270

实验 45 醛和酮的性质·····	271
实验 46 羧酸及其衍生物的性质·····	272
实验 47 胺的性质·····	273
参考文献·····	276

Chapter 1 General Knowledge of Organic Chemistry Experiments

The laboratory course is crucial for learning organic chemistry. This lab course aims at training and developing students' expertise in scientific method; creating and reinforcing their good work habits in the lab; training their scientific attitude of seeking truth from facts, their abilities for rigorous and detailed observation; guiding students to put forward scientific exploration problems, stimulating students' scientific interest and innovative consciousness.

The basic tasks of this course are:

- (1) To train students to master the basic operating skills of experiments in organic chemistry and enable students to synthesize, isolate, characterize and identify organic compounds properly.
- (2) To guide students to understand the general procedures for conducting a research in organic chemistry; develop students' skills in accessing journal articles, extracting information from them, evaluating data, developing and solving problems, and writing scientific reports correctly.

Organic chemistry laboratories contain certain inherent dangers and hazards. When working in a laboratory, students must learn how to work safely with these hazards in order to prevent injury to themselves and others around them. The following guidelines are here to help. Students should understand and follow these guidelines and act according to the principles behind them to help everybody to be as safe as possible. Students are required to be sure that they are familiar with the safety precautions, hazard warnings and procedures before working in the lab.

1.1 Rules for Organic Chemistry Laboratory

The following rules must be strictly followed by the students to ensure that all the organic chemistry experiments are performed normally, effectively and safely.

- (1) Prepare in advance and make a preview report prior to an experiment. Anyone who gets into the lab without a preview report will not be allowed to start the experiment.
- (2) Know the location and proper use of emergency equipment (water, electricity, safety showers, eye baths, fire extinguishers, and first-aid kits). Follow the instructions during an emergency and notify the instructor immediately.
- (3) Wear a lab coat (and do it up) in all chemistry labs. Wear clothes that provide maximum protection and cover most of the skin. Wear long pants (no shorts, capris, skirts, or dresses). Footwear must completely cover the foot and heel (no sandals, or open-toed footwear).

(4) No food or drink is allowed in the lab.

(5) No noise or disruptive behavior in the lab. Do not leave experiments without permission.

(6) Only the authorized scheduled experiments can be performed in the lab. You are not allowed to alter the procedure of the lab experiments. Carefully follow all instructions. Any changes to the experiments must be made with the approval of the instructor. Conduct the operation seriously, observe the experimental phenomenon carefully, and record the data truthfully during the experiment.

(7) Keep your own work area clean as well as the shared areas. Wastes should not be discarded into the sewer system; they should be stored in appropriate containers in a timely manner and according to the instructions provided in your lab manual. Return all materials to their place after using them.

(8) After an experiment, you should properly clean all the areas/equipment you worked on. Before leaving the laboratory, notify the instructor and ensure that: water and electrical devices at your lab station are turned off; your desktop and work area is thoroughly clean; all equipment in community drawer is complete and well organized; wash your hands, and any other area of skin that has contacted lab equipment or lab benches.

1.2 Safety Knowledge in the Organic Laboratory

Many of the chemicals used in an organic chemistry laboratory are flammable, explosive or toxic, and may cause accidents, such as fire, explosion or poisoning, if not properly handled. In addition, most parts of the equipment used in an organic chemistry lab are commonly made of glass, cuts and scratches may be caused if one is not careful enough. Therefore, students should pay special attention to safety instructions when performing an organic chemistry experiment.

Most laboratory accidents occur when researchers lack understanding and attention to materials and equipment or do not manage them with strict adherence to safety procedure and precautions. However, with careful preparation beforehand, close attention and careful use of chemicals and equipment, accidents can be avoided. Therefore, all students must read and understand the safety instructions before coming to the laboratory, and strictly follow them when conducting an experiment.

1.2.1 Laboratory Safety Rules

(1) Before starting the experiment, the students should carefully check up each item of the working equipment and apparatus to make sure that the equipment is intact and the apparatus is set up properly and fixedly. And permission from the instructor is required to start the experiment.

(2) Experiments must not be left unattended while in progress. The operator should inspect the reaction and check for leaks routinely.

(3) When conducting experiments that are potentially dangerous, the operator should wear necessary safety equipment, such as safety goggles, facemasks or gloves, etc.

(4) Keep ignition sources away from flammable or combustible dangerous substances.

(5) Do not taste chemicals. Do not smoke, eat, or drink in the laboratory. Wash your hands carefully after the experiment.

(6) Know the location, operation and use of the emergency equipment, such as fire extinguishers, sandbox, safety showers, eye baths, and first aid kit, etc.

(7) Reagents used or generated during the experiment should not be discarded arbitrarily. The waste liquid or solid should be disposed into designated containers for unified treatment.

1.2.2 Accident Prevention

1. Fire Prevention

There are many reasons why a fire can start, such as a low-boiling solvent is heated in an open container, or an incorrect heating method is applied, etc. In order to prevent fire hazards in the experiments, the following basic principles should be followed at all times:

(1) **DO NOT** store volatile or flammable chemicals in an open container. When using and handling of flammable liquids, keep away from naked flames, sparks, fire and heat; and maintain adequate ventilation. Water baths can be used to heat low-boiling liquids.

(2) **DO NOT** place volatile or flammable wastes into a waste container or regular trash. When dealing with a large amount of flammable liquid, use bulk waste containers for specific recycling.

(3) If you are caught in a fire, remain calm and take correct measures as soon as possible to control the fire. First, shut off the power immediately and remove the flammable materials. Next, find out the classes of fires and how best to tackle them safely by choosing the most appropriate type of fire protection equipment. Organic laboratories do not use water (extinguishers), fires with chemicals and electrical equipment should be extinguished with other agents. If the fire is small, cover the fire with a damp cloth; when it is large, use a fire extinguisher.

The most common types of extinguishers in laboratories are carbon dioxide extinguishers, dry powder extinguishers and foam extinguishers.

Standard dry powder extinguishers are used commonly in organic laboratories, and special dry powder extinguishers can be used for flammable metals. However they are not recommended for use in enclosed spaces. This is because the powder can be easily inhaled, and also the residue is very difficult to clean up. When using a dry powder extinguisher, pull the pin to discharge the extinguisher; aim at the base of the fire (if you aim at the flames, the extinguishing agent will fly right through); squeeze the top handle to release the pressurized extinguishing agent in the extinguisher; sweep from side to side until the fire is completely out.

Carbon dioxide extinguisher is also a common fire extinguisher in organic labs, which is predominantly used for electrical fire risks and labs with electric equipment.

Foam extinguishers are ideal where cooling a fuel is recommended but not suitable for electrical fires or running fires, because they are water-based.

When using any of these portable extinguishers, it is necessary to aim at the base of the

flames, rather than at the flames themselves.

A small tabletop fire can be put out with some sand in the fire sand bucket.

If your clothes catch fire, remember: stop, drop and roll until the flames have been extinguished. Don't run around, you'll make the flames worse.

2. Explosion Prevention

In order to prevent accidents due to explosions, following things should be noted:

(1) **NEVER** heat a closed system or conduct a reaction in a closed system.

(2) Before starting a distillation or chemical reaction, make certain that the system is vented. Be sure to use glassware designed for vacuum or pressure operations in a vacuum distillation, do not use flat-bottomed or thin-walled glassware. Always check for star cracks in the flasks before beginning a vacuum distillation. **NEVER** distill to dryness.

(3) Ensure to keep the flammable substance away from the source of ignition.

(4) Ethers have to be tested for formation of peroxides before being used. If peroxides are detected, they must be removed with some reagents, such as ferrous sulfate.

(5) Be extremely careful when using explosive materials, such as acetylide of heavy metals, polynitro compounds (picric acid or heavy metal picrates), trinitrobenzene (TNB), etc. **NEVER** strike explosive materials or allow them to be hit. **NEVER** subject explosive materials to excessive impact or friction. Unused explosives and explosive waste **MUST** be removed and disposed of under the supervision.

3. Poison Prevention

(1) **DO NOT** allow laboratory chemicals to come in contact with skin. Appropriate type of gloves, such as chemical resistant gloves, heat resistant gloves, etc., should be worn for handling hazardous materials. Wash your hands before and immediately after working with any laboratory chemicals, even if gloves have been worn. **NEVER** taste any chemical.

(2) When handling fuming acids or volatile irritants, work in a chemical fume hood, wearing appropriate personal protective equipment. Always wash glass labware immediately after use.

(3) If a harmful substance has splashed onto one's skin or mouth, remove any contaminated items and wash the affected area thoroughly with cool water for at least 10 min. If swallowed, try to spit out anything that is remaining in the mouth, wash out mouth with plenty of water, and then drink some fresh water. If necessary, go to the hospital for treatment. If toxic vapors are inhaled, move the victim at once to fresh air and get medical care as soon as possible.

4. Prevention of Burns

A burn occurs when a corrosive, hot or very cold material contacts the skin. All students are required to wear a lab coat at all times. Wear safety goggles and gloves when handling hazardous chemicals to avoid burns. When corrosive chemical contaminates the skin, wipe it off quickly and immediately rinse it with plenty of water and make forward treatments for different burns as

follows.

(1) In case of burns caused by alkalis, use 1% acetic acid solution or 3% boric acid solution to neutralise, and then wash with soap and water.

(2) In case of burns caused by acids, use 3% sodium-hydrogen carbonate (sodium bicarbonate).

(3) In case of burns caused by bromine, use the same procedure as that used for treating an acid burn.

(4) Small burns from touching hot objects should be placed under running cold water for at least 20 min. Major burns need immediate medical attention.

(5) If acid squirts into the eye, wash it by dropping with 2% borax solution, then rinse with physiological saline. In the case of alkali, use 2% boric acid solution, and then rinse with physiological saline, and get medical care as soon as possible.

5. Prevention of Cuts

To avoid cuts, first **NEVER** place glassware under pressure that it is not designed to withstand. Inspect glassware for defects or cracks before use. Do not handle broken glass with bare hands.

In the event of an injury involving a broken glass or sharp instrument, the following steps should be followed. Small cuts should be rinsed thoroughly and examined for traces of foreign materials such as glass. If there is broken glass in the wound, the pieces should be removed carefully first. Bandages are available in the stockroom. Severe cuts and bleeding must be treated by applying direct pressure on the wound to control the rate of bleeding. Seek medical assistance immediately. If the incident involves potential exposure to infectious materials, then immediate medical attention is required.

Each lab should have a first aid kit that is properly stocked for emergency first aid purposes. The first aid kit in the laboratories should contain the following items:

(1) Tweezers, scissors, gauze, cotton wool, bandages, plasters, etc.

(2) Vaseline, band-aid, scald cream, eucalyptus oil, boric acid ointment.

(3) 1% acetic acid or boric acid solution, 1% sodium bicarbonate solution, 2% sodium thiosulfate solution, glycerin, hemostatic powder, medical alcohol, red syrup, gentian violet, and the like.

6. Electrical Safety

Conduct an inspection of laboratory electrical equipment before using it to ensure it is in good condition. Make sure the plug fits snugly into the outlet. Outlets that have loose-fitting plugs can overheat and damage the cord and the plug. **NEVER** force a plug into an outlet if it doesn't fit.

Make sure your hands are dry before plugging or unplugging a power cord. Prevent the human body from contact with electrical conductors. **NEVER** put a metal object in live parts of appliances or outlets.

When the experiment is completed, turn off all power first and unplug the equipment.

1.3 Commonly Used Equipment and Apparatus of Organic Chemistry Experiments

Laboratory glass equipment can be divided into those with ground-glass joints and those without. Standard-taper, ground-glass joint equipment is more convenient and faster to be used. Therefore it is strongly recommended. However, sometimes it is also inevitable to use ordinary glassware.

1.3.1 Standard-taper, Ground-glass Joint Equipment

The standard-taper glassware is manufactured in accordance with international requirements. Pieces of glassware in the same size fit together freely to conveniently set up an apparatus due to the ground glass joints incorporated into each other by an inner (male) joint held above an outer (female) joint. This means it doesn't need to equip plug and drill, also prevent product dirtied by cork or rubber plug. Furthermore, the standard glassware is more favorable to be used in a leak-tight apparatus especially in vacuum distillation because of its tight connections between all parts. What's more, it's safer to use the standard glassware in an experiment involving hazardous or volatile liquids.

The ground glass joints are often labeled in different sizes with different numbers. These numbers represent the outer diameter (OD) in millimeters (mm) at the widest point of the inner joint. The often used standard tapers are 10, 12, 14, 16, 19, 24, 29, 34, and 40 in size.

The ground glass joints can also be labeled with a number, a slash, and another number. The first number represents the OD at the widest point of the inner joint. The second number represents the ground glass length of the joint in millimeters. For example, a 14/20 joint is 14 mm wide at the top (the widest part) and is 20 mm in length. The organic chemistry teaching labs often use 19/22 sized standard taper. Any two joints (male and female) with the same outside diameter can fit into each other, no matter how length the joints are. When connection for two glass apparatuses with different joint size is needed, the connecting adapter (reducing adapter or enlarging adapter) is available to place between to connect them. For example, for enlarging the size of standard-taper glass joints from 14/20, outer to 19/22 inner, the socket size is 14/20, cone size is 19/22.

1. Commonly-used Standard-taper Ground Glassware in Organic Chemistry Experiments

Commonly-used standard-taper ground glassware in organic chemistry Experiments is listed in Fig. 1.1.

2. Tips for Using of Standard-taper Ground Glassware

(1) Before use, all glassware should be checked to ensure that it is free from cracks, flaws or scratches that may cause it to fail in use.

(2) Always keep the ground glass joints of the glassware clean. Wipe sticky dust before connecting the ground joints.

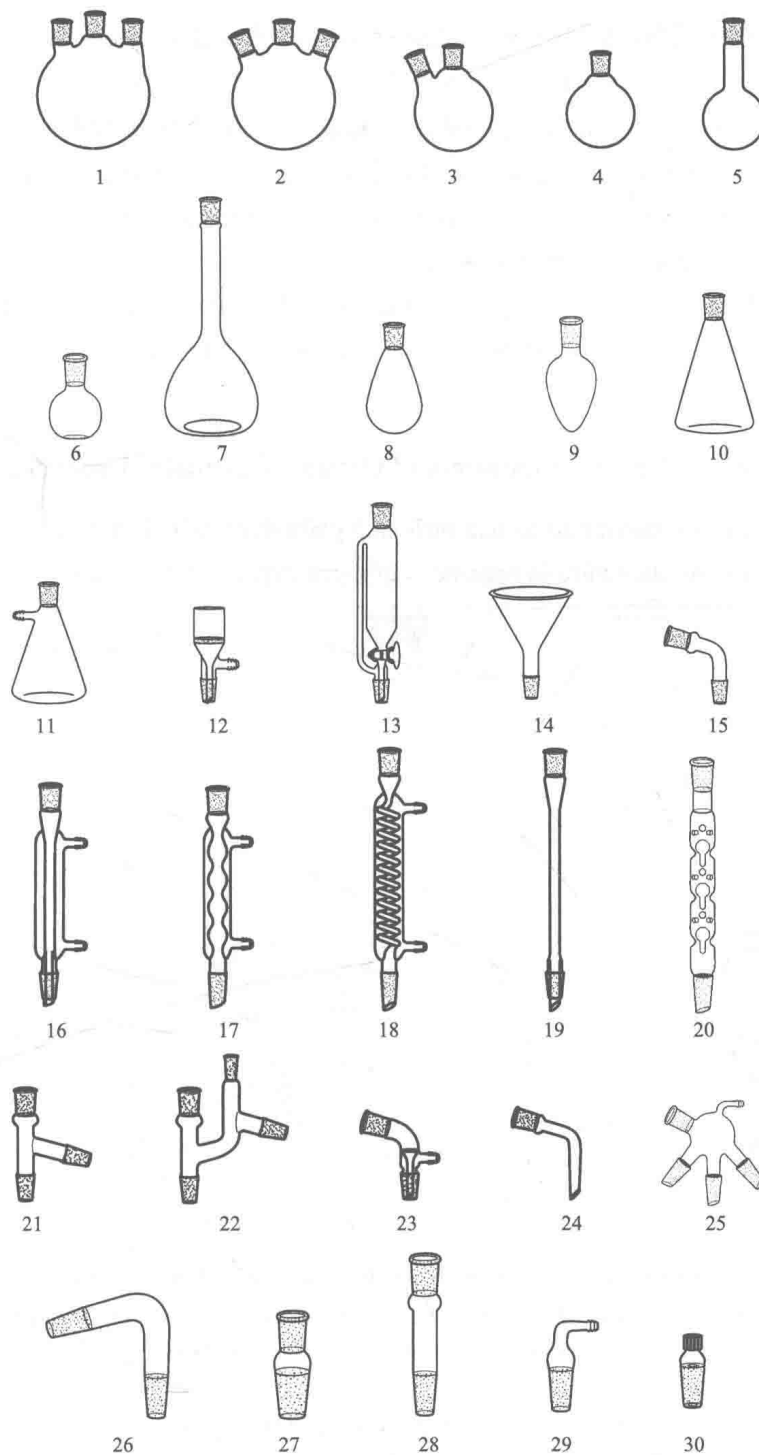


Fig. 1.1 Commonly-used standard-taper ground glassware in organic chemistry experiments

1. three-vertical-neck flask; 2. three-angled-neck flask; 3. two-neck flask; 4. round-bottom flask; 5. long-neck round-bottom flask; 6. flat-bottom flask; 7. long-neck flat-bottom flask; 8. eggplant shaped flask; 9. pear shaped flask; 10. Erlenmeyer flask; 11. suction flask; 12. sand core funnel; 13. constant pressure dropping funnel; 14. funnel; 15. drying tube; 16. straight-shaped condenser; 17. Allihn condenser; 18. graham condenser; 19. air condenser; 20. distilling tube with thorn tube; 21. distillation head; 22. Claisen distillation head; 23. vacuum adapter; 24. distillation adapter; 25. pig receiver; 26. distillation bent; 27. reducing adapter; 28. enlarging adapter; 29. angled cone adapter; 30. thermometer adapter

(3) When assembling, gently screw to connect the socket and the cone. Be careful not to force too much.

(4) There's no need to grease the ground glass joints when being used for general use to avoid potential contamination by the grease. Grease the joints (between the sealing faces) if the reaction involves a strong base to prevent sticking caused by alkali corrosion. For high vacuum applications, vacuum greases should be used prevent leakage.

(5) After finish an experiment, separate apart and clean the socket and the cone rapidly to minimize the risk of the glass joints sticking. Use a piece of paper to gently detach the cone from the socket to prevent sticking.

1.3.2 Commonly-used Ordinary Glassware of Organic Chemistry Experiments

Sometimes, it is also inevitable to use ordinary glassware, which has no ground-glass joints. Commonly-used ordinary glassware in organic chemistry experiments is listed in Fig. 1.2.

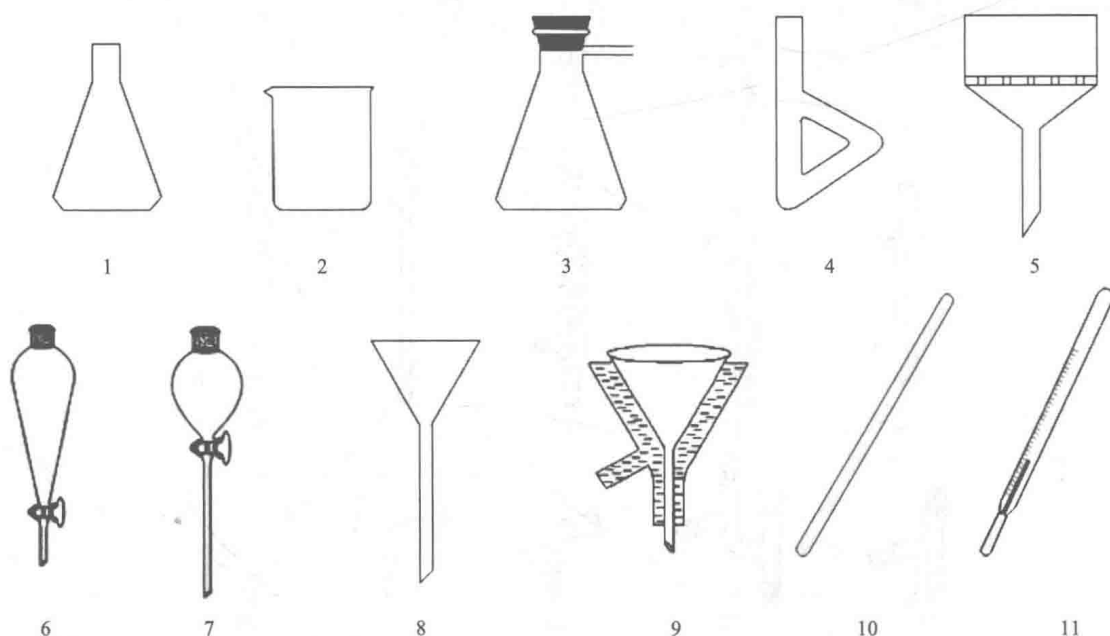


Fig. 1.2 Commonly-used ordinary glassware in organic chemistry experiments

1. Erlenmeyer flask; 2. beaker; 3. suction flask; 4. Thiele tube (b-tube); 5. Büchner funnel; 6. separatory funnel; 7. dropping funnel; 8. funnel; 9. hot water funnel; 10. glass rod; 11. thermometer

The following points should be noted when using ordinary glassware:

(1) No ordinary glassware can be heated directly by fire except a few, such as test tube.

(2) Glassware with thick walls (e.g. suction flask) should **NEVER** be heated over a direct flame. Erlenmeyer flasks are in general not suitable for use under pressure or in a vacuum; wide-mouthed containers such as beakers cannot be used for the storage of flammable solvents.

(3) When storing glass stopcocks and joints, insert a thin strip of paper between joint surfaces to prevent sticking.

(4) **NEVER** use a thermometer to stir, after the thermometer has been used, cool it down