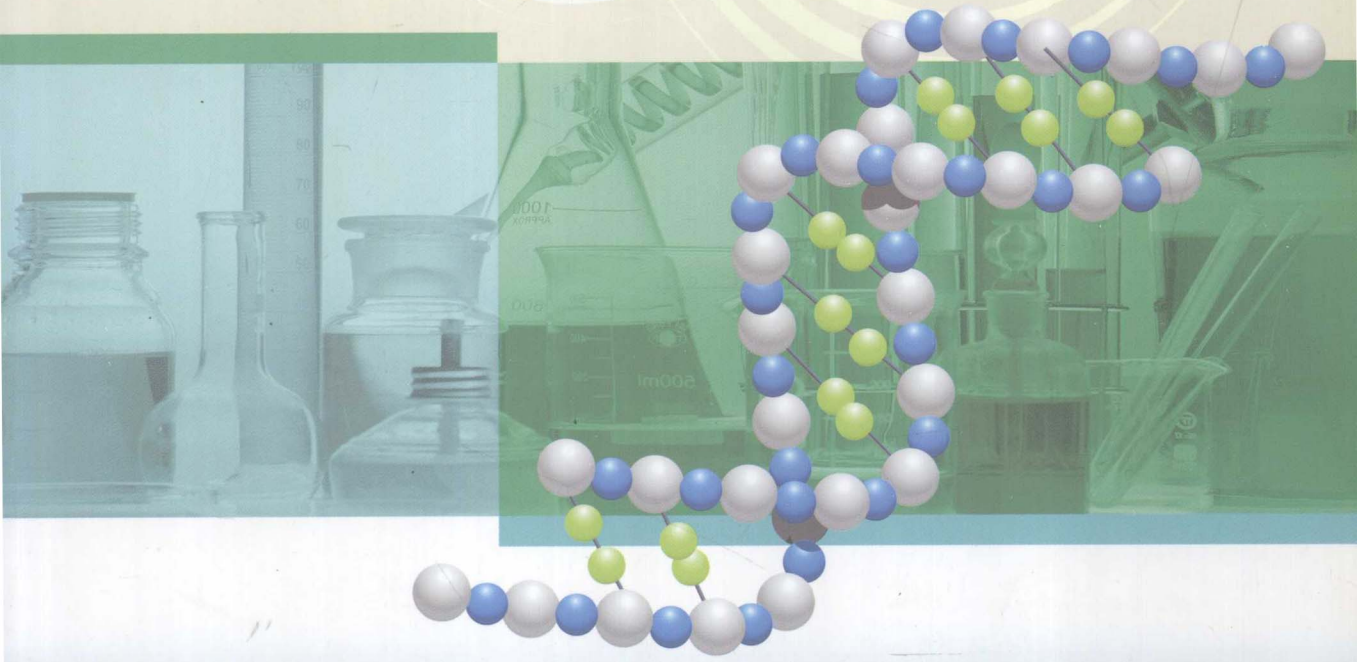
Organic Chemistry Experiments

有机化学实验

Wang Mei Wang Yanhua Gao Zhanxian



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Capsule summary

This book is compiled on the basis of "Organic Chemistry Experiments" (fourth edition, edited by Zhanxian Gao) and with combination of the bilingual teaching experience.

The book encompasses six sections: introduction, basic techniques for organic experiments, basic preparation experiments of organic compounds, comprehensive experiments, self-designing experiments, and investigative experiments. Tables of commonly used data, index, and the Chinese-English professional glossary are attached to the end of the book.

The book was written with emphasis on (1) basic techniques for organic experiments, including some advanced experimental techniques such as microwave reactions and the resolution of racemates; (2) common-scale and miniscale experiments, also taking account of semimicro- and microscale experiments; (3) traditional and representative organic reactions, meanwhile, introducing the concept of "green" synthesis; and (4) training students in basic experimental skills, including developing their ability in experimental design and scientific research as well.

This book can be used as a textbook for the bilingual course of organic chemistry experiments. It can also be used as a teaching reference book for the experiment courses of other related majors.

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Preface

To facilitate the bilingual teaching in the colleges and universities of the whole country, the Ministry of Education put forward a program in 2007 to build 500 state-level model courses of bilingual teaching in five years. The project, "Construction of a model course for bilingual teaching of organic chemistry and experiments" in Dalian University of Technology, is one of the first hundred projects granted by the Ministry of Education for setting up model courses of bilingual teaching. With support from the project of the Ministry of Education, we compiled this textbook of English version for the course of organic chemistry experiments. We translated the main parts of the first and second chapters on comprehensive introduction of organic chemistry experiments and selected 30 representative experiments from Chapters 3 to 6 of the Chinese textbook, "Organic Chemistry Experiments" (fourth edition, edited by Zhanxian Gao, as one of the "Eleventh Five-Year" national planning teaching materials for ordinary higher education),. The book encompasses six sections: introduction, basic techniques for organic experiments, basic preparation experiments of organic compounds, comprehensive experiments, self-designing experiments, and investigative experiments. Tables of commonly used data, index, and the Chinese-English professional glossary are attached to the end of the book.

The book was written with emphasis on (1) basic techniques for organic experiments, including some advanced experimental techniques such as microwave reactions and resolution of racemates; (2) common-scale and miniscale experiments, also taking account of semimicro- and microscale experiments; (3) traditional and representative organic reactions, meanwhile, introducing the concept of "green" synthesis; and (4) training students in basic experimental skills, including developing their ability in experimental design and scientific research as well.

There are 6 Chapters in the book. Chapters 1 to 3 and Section 6. 4 were compiled by Mei Wang and Chapters 4 and 5 as well as Sections 6. 1 to 6. 3 were compiled by Yanhua Wang. Mei Wang checked and revised the whole content of the book. Zhanxian Gao of Dalian University of Technology and Yanmei Li of Qinghua University successively reviewed the manuscript of the book and gave many valuable comments and suggestions; Xiaoan Zhang of Jilin University polished parts of the content; Yi Zhai, Jia Liu, and Chunjiang Fu of Higher Education Press did lots of work for putting out this book. Here we would like to express our thanks to all of them.

As the knowledge of the editors is limited, there might be some improper parts in the book. We sincerely welcome corrections and comments from experts, peers, as well as teachers and students using this book.

Editors

Spring, 2011

Contents

Chapter 1

Introduction

The course of experimental organic chemistry will introduce you to the techniques and procedures used in organic chemistry. You will learn how to handle a variety of chemicals safely and how to manipulate apparatus properly. Along with becoming more skilled in the technical aspects of laboratory work, you should also develop a proper scientific approach to the execution and interpretation of experiments. The "hands-on" experience gained in the laboratory, as you gather and interpret phenomena and data from a variety of reactions, will provide a sense of organic chemistry that is nearly impossible to communicate in classes. Performing reactions in practice will give you a deep impression and better understanding on the theoretical concepts, functional groups, organic reactions and methods you have leant in organic lectures.

1.1 General Rules for Organic Chemistry Laboratory

When entering, working, and leaving the laboratory, you should be clearly aware of and strictly observe the following basic rules to keep good order in laboratories and to ensure laboratory safety.

(1) You should be aware of safety rules and first-aid procedures, and familiar with the layout of the laboratory room. Know the locations of the fire extinguishers, fire blankets, safety showers, gas valves, and electric switches.

(2) Wear laboratory coat when working in the laboratory. Do not wear shorts and scandals in the laboratory. Wear latex gloves when working with concentrated acids, bases, bromine and other particular hazardous chemicals. Never bring food and drink to the laboratory.

(3) Make a theoretical preparation in advance for each experiment by reviewing the basic knowledge related to the experiment, studying the entire experiment procedures, learning the operation rules for the apparatus and equipment you will use and understanding the toxicity and other potential hazards of the chemicals used in experiment. Never begin any experiment until you understand its overall purpose and the reasons for each operation that you are to do.

(4) Strictly observe all the rules and regulations for organic chemistry laboratory. Follow the guidance of your teachers and instructors, and follow the experiment procedures in the textbook. Do not make any modification in experiment without a permission of your instructor. Never work alone in the laboratory and never leave an ongoing experiment unattended.

(5) During experiment, keep the laboratory clean and tidy. Handle the glassware with care and clean it after experiment. Put all glassware and apparatus in good order either on your bench top or in your kit. Keep sinks clean. Do not pour and throw other things into sinks except for neutral and nonhazardous aqueous solution.

(6) After using the chemicals, cover the stopper of the container immediately and put it back to the original place. All reagents prepared should be stuck on a label with name, concentration and date. Always pay attention to economize chemicals, water, electricity and gas.

(7) Read the manuals for instruments and gauges to understand their working principles and operation methods before manipulating them. Operate precision instruments and gauges with care and strictly follow the operation rules. Do not dismantle and remove the instruments without a permission of your instructor.

(8) All solid wastes and organic liquid wastes should be placed in designated containers, respectively.

(9) After experiment, clean the laboratory thoroughly. Before leaving the laboratory, carefully check whether all switches and valves for water, electricity and gas are switched off safely.

1.2 Safe Laboratory Practice and First Aid in Case of an Accident

Chemistry laboratories are potentially dangerous because organic chemistry experiment commonly uses fragile glassware, flammable liquids, toxic chemicals and equipments under vacuum or high pressure. Most organic solvents, such as benzene, alcohols, gasoline, ethers, acetone, and so on, are volatile and highly flammable, and some chemical substances are explosive if handled improperly. Incorrect operations may cause experimental accidents or even disasters. Therefore, you should have a sound knowledge of how to perform experimental work in a safe manner and strictly follow standard safety protocols.

1.2.1 Prevention of Fires and First Aid in Case of a Fire

Occasionally, open flames may be used for heating a reaction mixture or distilling a highboiling point liquid. In such cases, give special precautions for the open flames and strictly follow the guidelines in the "Safety Alert" section.

(1) Always check carefully for cracks, chips or other imperfections in the glassware that you will use, and equip the apparatus with all joints tightly fitted.

(2) Never use a flame to directly heat a flammable liquid. Use a water or steam bath or electrical heat device instead.

(3) Flammable chemicals must be kept and handled in a place away from an open flame.

(4) Do not pour flammable and water-insoluble organic solvents into drains or sinks. They must be recovered after experiment.

(5) Do not store a large amount of flammable liquids in the laboratory.

In case of a fire, quickly turn off the power and gas, and call for help. Remove all flammable liquids and materials from the immediate area, and then extinguish the fire with a fire extinguisher, sand or asbestos cloth. If your clothing is on fire, do not run. Roll on the floor to smother the fire and to keep the flame away from your head. Your neighbors can help to extinguish the flame by using fire blankets, laboratory coats or other items that are immediately available. A laboratory shower, if close by, can be used to extinguish burning clothing.

If burns are minor, apply a burn ointment. In the case of serious burns, do not apply any ointment; seek professional medical treatment at once.

1.2.2 Prevention of Explosions

(1) Some chemicals are explosive if handled incorrectly. For example, peroxides, multi-nitro aromatic compounds and nitrates may explode when heated or knocked. Ethers may contain peroxides, so do not distill them to dryness and it is recommended to remove peroxides from ethers before distillation. Do not dry multi-nitro aromatic compounds in an oven. Mixing alcohol with concentrated nitric acid may cause a fierce explosion.

(2) Wrong assembly of apparatus and incorrect operations can also cause explosion. Do not heat an air-tight system for common distillation or reflux. Always wear safety glasses or goggles in the laboratory.

(3) Immediately turn off the gas when it leaks out, open the windows, and inform your instructor to check and repair it.

1.2.3 First Aid for Cuts and Scalds

Handle glassware with care and be cautious of hot apparatus and solutions to avoid hurt by cuts and scalds. If you are unfortunately injured by broken glass, for minor cuts, squeeze the contaminated blood out immediately, pick out the pieces of broken glass, and rinse the cut thoroughly with distilled water. After this, wipe the cut with tincture of iodine or merbromin, and bind it up with gauze or apply with adhesive bandages. For severe cuts, first bind up the cut with gauze. If the cut bleeds, attempt to stop the bleeding with compress and pressure. Seek the professional medical treatment at once.

In case of scalds, smear some scald ointment on the affected area and coat the injured area with gauze. If the scald is severe, go to the clinic for further treatment.

1.2.4 Prevention of Damages from Hazardous Chemicals and First Aid for Chemical Burns

The variety and potential danger of chemicals used in the organic chemistry laboratory proba-

bly exceed that of any laboratory course you have had. It is imperative to understand the properties of the substances with which you are working, and to take a proper precaution.

(1) Make reactions that will release toxic gases, such as chlorine, bromine, nitrogen oxide and hydrochloride in a hood or in a well-ventilated area, and absorb the toxic gases with a gas trap. Avoid inhaling vapors of organic and inorganic compounds. Work in a hood when handling particularly volatile and noxious chemicals.

(2) Wear latex gloves when handling particularly toxic and corrosive chemicals to avoid contacting with your skin. Do not drop them on the bench and the floor, and never pour or throw them into the drains or sinks. Concentrated acids and bases and bromine are very corrosive and could cause chemical burns, so handle them with great care.

(3) If corrosive chemicals accidentally come in contact with your skin or eyes, wash the affected area immediately with large amounts of running water, and then treat the affected area in the following ways:

For acid-burn, first wash the eyes with 1% sodium bicarbonate solution and wash the skin with saturated sodium bicarbonate solution, and then wash the eyes or skin with water. Finally, apply burn ointment on the affected area.

For base-burn, first wash the eyes with 1% boric acid solution and wash the skin with 1% acetic acid, and then wash the eyes and skin with water. Finally, apply burn ointment on the affected area.

In all instances where eye tissue is contacted with bromine, consult an ophthalmologist as soon as possible after immediately flooding the eyes with water; for bromine-burn of skin, sequentially wash the affected area with petroleum ether and 2% sodium thiosulfate solution, and then smear with glycerol and apply cod liver oil ointment on the affected area.

For treatment of serious burns, see a surgeon after first aid.

1.2.5 Toxicity and Safety Data of Chemicals

Common chemicals are more or less harmful to the health of human beings. Even compounds with pleasant smell are harmful if inhaling too much vapors of the compounds. Some nitrogen-containing and fused ring compounds are very toxic, exhibiting a relatively low lethal dose, and some chemicals are possibly carcinogenic. One of the principles to choose experiments for this textbook is to avoid using particular toxic chemicals in the teaching laboratory. Your actions and those of your labmates, will determine whether you can do experiment in a safe environment.

The increased emphasis on the proper handling of chemicals has led to a number of different types of publications containing key information about the chemical, physical and toxicological properties of the majority of organic and inorganic compounds. The data provided by such references are basically a summary of the information contained in the Material Safety Data Sheets (MSDS) published by the suppliers of chemicals. MSDS data include physical constants,

inflammability and explosibility, reactivity, treatment measures in case of leaking, handling and storage knowledge, health hazards, toxicity data, and so on. From a standpoint of environmental protection, the threshold limit values (TLV) of many chemicals in the area of chemical factories and laboratories have been stipulated by labor and health administrations and the permissible levels of chemicals in the area of laboratories have been set by the occupational safety and health administration (OSHA). The threshold limit values for some commonly used compounds are given below. Before experiment, you should know the properties of the chemicals used in the experiments and refer to MSDS to learn about toxicity and other potential hazards associated with the chemicals.

(1) Toxic solids

compound	TLV① /(mg·m^{-3})	compound	TLV/(mg·m^{-3})
osmium trioxide 三氧化锇	0.002	arsenium 砷化合物	0.5 (based on As)
mercury compounds, especially mercury alkylides 烷基汞	0.01	vanadium pentoxide 五氧化二钒	0.5
thallium salts 铊盐	0.1 (based on Tl)	oxalic acid and oxalate 草酸和草酸盐	1
selenium and its compounds 硒和硒化合物	0.2 (based on Se)	inorganic cyanides 无机氰化物	5 (based on CN^-)

① TLV (threshold limit value): To work in a safe environment, the concentration of the vapor or bug dust of the toxic compound should be kept below the limit value.

(2) Toxic and hazardous gases

compound	TLV/(μg·g^{-1})	compound	TLV/(μg·g^{-1})
fluorine 氟	0.1	hydrofluoride 氟化氢	3
phosgene 光气	0.1	nitrogen dioxide 二氧化氮	5
ozone 臭氧	0.1	nitrosyl chloride 亚硝酰氯	5
diazomethane 重氮甲烷	0.2	cyanogen 氰	10
phosphine 磷化氢	0.3	hydrogen cyanide 氰化氢	10
boron trifluoride 三氟化硼	1	hydrogen sulfide 硫化氢	10
chlorine 氯	1	carbon monoxide 一氧化碳	50

(3) Toxic, hazardous liquids and irritant materials

Contacting with the following compounds for a long time can cause chronic intoxication. The vapors of most of the following compounds are strongly irritant to eyes and respiratory tract.

compound	TLV/($\mu g \cdot g^{-1}$)	compound	TLV/($\mu g \cdot g^{-1}$)
carbonyl nickel compounds 羰基镍	0.001	dimethyl sulfate 硫酸二甲酯	1
methyl isocyanate 异氰酸甲酯	0.02	diethyl sulfate 硫酸二乙酯	1
acraldehyde 丙烯醛	0.1	tetrabromoethane 四溴乙烷	1
bromine 溴	0.1	propenol 烯丙醇	2
3-chloropropane 3-氯丙烷	1	2-butenal 2-丁烯醛	2
chlorophenylmethane 苯氯甲烷	1	hydrofluoric acid 氢氟酸	3
bromophenylmethane 苯溴甲烷	1	tetrachloroethane 四氯乙烷	5
boron trichloride 三氯化硼	1	benzene 苯	10
boron tribromide 三溴化硼	1	bromomethane 溴甲烷	15
2-chloroethanol 2-氯乙醇	1	carbon disulfide 二硫化碳	20

(4) Other toxic materials

some halogen-containing compounds 含卤素化合物

compound	TLV	compound	TLV
bromoform 溴仿	0.5 $\mu g \cdot g^{-1}$	1,2-dibromoethane 1,2-二溴乙烷	20 $\mu g \cdot g^{-1}$
iodomethane 碘甲烷	5 $\mu g \cdot g^{-1}$	1,2-dichloroethane 1,2-二氯乙烷	50 $\mu g \cdot g^{-1}$
carbon tetrachloride 四氯化碳	10 $\mu g \cdot g^{-1}$	bromoethane 溴乙烷	200 $\mu g \cdot g^{-1}$
chloroform 氯仿	10 $\mu g \cdot g^{-1}$	methylene chloride 二氯甲烷	200 $\mu g \cdot g^{-1}$

aromatic and aliphatic amines 芳香和脂肪胺

compound	TLV	compound	TLV
phenylenediamines 苯二胺	0.1 $mg \cdot m^{-3}$	aniline 苯胺	5 $\mu g \cdot g^{-1}$
methoxyanilines 甲氧基苯胺	0.5 $mg \cdot m^{-3}$	methylaniline 甲基苯胺	5 $\mu g \cdot g^{-1}$
nitroanilines 硝基苯胺	1 $\mu g \cdot g^{-1}$	dimethylamine 二甲胺	10 $\mu g \cdot g^{-1}$
N-methylaniline *N*-甲基苯胺	2 $\mu g \cdot g^{-1}$	ethylamine 乙胺	10 $\mu g \cdot g^{-1}$
N;*N*-dimethylaniline *N*,*N*-二甲基苯胺	5 $\mu g \cdot g^{-1}$	triethylamine 三乙胺	25 $\mu g \cdot g^{-1}$

phenols and nitrobenzene derivatives 酚和芳香族硝基化合物

compound	TLV	compound	TLV
picric acid (trinitrophenol) 苦味酸	0.1 $mg \cdot m^{-3}$	nitrobenzene 硝基苯	1 $\mu g \cdot g^{-1}$
dinitrophenols 二硝基苯酚	0.2 $mg \cdot m^{-3}$	phenol 苯酚	5 $\mu g \cdot g^{-1}$
chloronitrophenols 硝基氯苯	1 $mg \cdot m^{-3}$	methylphenols 甲基苯酚	5 $\mu g \cdot g^{-1}$
m-dinitrobenzene 间二硝基苯	1 $mg \cdot m^{-3}$		

(5) Carcinogenic materials

biphenylamines and their derivatives 联苯胺及其衍生物	β-naphthylamine β-萘胺
dimethylaminoazobenzene 二甲氨基偶氮苯	α-naphthylamine α-萘胺
N-methyl-*N*-nitrosylaniline *N*-甲基-*N*-亚硝基苯	*N*-nitrosyldimethylamine *N*-亚硝基二甲胺
N-methyl-*N*-nitrosylurea *N*-甲基-*N*-亚硝基脲	*N*-nitrosylhydropyridine *N*-亚硝基氢化吡啶
bis(chloromethyl) ether 双氯甲基醚	dimethylsulfate 硫酸二甲酯
chloromethyl methyl ether 氯甲基甲醚	iodomethane 碘甲烷
diazomethane 重氮甲烷	β-hydroxypropalactone β-羟基丙酸内酯
benzo [*a*] pyrene 苯并[*a*]芘	dibenzo [*c*,*g*] carbazole 二苯并[*c*,*g*]咔唑
dibenzo [*a*,*h*] anthracene 二苯并[*a*,*h*]蒽	7,12-dimethylbenzo [*a*] anthracene 7,12-二甲基苯并[*a*]蒽
thioacetamide 硫代乙酰胺	thiourea 硫脲
asbestos dust 石棉粉尘	

(6) Toxic materials that have a chronic accumulation effect

Once the following toxic materials are assimilated, they are difficultly discharged from human body, resulting in chronic poisoning.

(a) Benzene.

(b) Lead compounds, especially organic lead compounds.

(c) Mercury and its compounds, especially divalent mercurate and organic mercury compounds.

Proper protecting measures should be adopted when using the afore-listed toxic materials. Avoid inhaling the vapors or the dust from these toxic materials, and do not allow them to come in contact with your skin. Toxic gases and volatile toxic liquids should be handled in a hood or in a well-ventilated area. The surface of mercury should not be exposed to air and it must be covered with water for storage.

1.2.6 Disposal of Chemical Wastes

The proper disposal of chemical wastes is one of the biggest responsibilities that you have in the organic laboratory. The experimental procedures in this textbook have been designed at a scale that should allow you to isolate an amount of product sufficient to see and manipulate, but they also involve the use of minimal quantities of reactants, solvents and drying agents to reduce the amount of chemical wastes. For environmental protection, chemical wastes generated from the organic laboratory must be classified and recovered. Only the nonhazardous, water-soluble, neutral, nonflammable and biologically degradable chemical wastes can be flushed down the drain with excess water. Solid chemical wastes should not be thrown in a trash can.

The recommended procedures that should be followed are described under the heading, "Finishing Touches". The organic laboratory should be equipped with various containers for disposal of hazardous solids, nonhazardous solids, halogenated organic liquids, hydrocarbons and oxygenated organic liquids. The containers must be properly labeled as to what can be put in them. It is very important for safety and environmental reasons that different categories of spent chemicals are segregated from one another. Following is the general rules for disposal of chemical wastes:

(1) All inorganic acids and bases used in this textbook should be neutralized first and diluted with copious water before flushing down the drain.

(2) Different organic liquid wastes should be classified and poured into properly labeled containers, respectively. Waste containers should be stored in a well-ventilated place. Never pour flammable and water-insoluble organic liquids into drains or sinks.

(3) Nonhazardous solid wastes such as alumina, silica gel, drying agents, and so on, should be placed in a designated container after solvents are evaporated in hoods.

(4) Hazardous solid wastes should be disposed of in a specially labeled container. The exact name of the solid wastes should be written on the label.

(5) Broken thermometers are a special problem because they usually contain residue mercury, which is toxic and relatively volatile. Collect all mercury for the broken thermometer and place it in a specially labeled container. The mercury should be covered with water and the container should be tightly closed. If mercury has spilled as a result of the breakage, it should be cleaned up immediately. Consult your instructor about appropriate procedures for doing so.

(6) Chemical wastes that react violently with water such as Grignard reagent, aluminium chloride, acetic anhydride should be decomposed with proper reagents in a hood before disposal.

1.3 Information Sources for Experimental Organic Chemistry

There are varieties of the literature on experimental organic chemistry available in print and online. To be familiar with the literature and handbooks of experimental organic chemistry is important for designing and making organic experiments. This section provides you with a general knowledge about four major categories of the important information sources of organic chemistry, which may be used as valuable lead references to initiate a specific search.

1.3.1 Important Handbooks and Dictionaries

(1) **Beilstein's Handbuch der Organischen Chemie (Beilstein Handbook of Organic Chemistry).** It is perhaps the most complete reference work on organic compounds. It provides full access to the database of over eight million compounds and five million reactions. The records in the Beilstein handbook include molecular structures, chemical and physical properties and constants,

spectral identification, synthetic procedures and relevant references. The printed Beilstein handbook is published in German, but the Beilstein Online database gives information in English. The easier and faster way to get the needed information is to access the CrossFire Beilstein Online database if your library has paid to the online vendor. The CrossFire Beilstein indexes three primary types of data. The substance domain stores structural information with all associated facts and literature references. The reaction domain details the preparation of substances with reaction search queries, scientists can investigate specific reaction pathways. Chemical literature citations, titles, and abstracts, which are hyperlinked to substance and reaction domain entries, are stored within the citation domain.

(2) **Merck Index of Chemicals and Drugs**, 12th ed., Merck and Co., Rahway, NJ. It provides a concise summary of the physical and biological properties as well as uses, toxicity and hazards of more than 10 000 compounds and some literature references. Organization is alphabetical by names, synonyms and trade names. Formula and subject indexes, and a Chemical Abstracts Service registry number index are available for this handbook.

(3) **Dictionary of Organic Compounds**, 6th ed., Buckingham, J., Ed. Chapman and Hall, New York. This dictionary is in nine volumes. Volumes 1～6 contain the data for the compounds, Volume 7 is a name index with cross-references, Volume 8 contains a formula index, and Volume 9 is a Chemical Abstracts Service registry number index.

(4) 有机化学实验常用数据手册,第三版,吕俊民编,大连理工大学出版社,1997. The first part of this handbook contains physical properties of common organic and inorganic compounds, including the Chinese and English names of compounds, their chemical formulas, relative molecular weights, color and crystal shapes, relative densities, melting points, boiling points, refractive indices, solubilities in water, alcohols and ethers, as well as the original sources of all items. The second part contains the thermodynamic data related to the experiment of organic chemistry, and the third part contains the safety data of chemicals. There is a chemical formula index in the end of the handbook.

1.3.2 Important Journals Involving Organic Experiment

Primary research journals are the ultimate source of most of the information on organic chemistry. Some of the important journals and a brief description of their contents are given below.

(1) ***Journal of the Chemical Society, Perkin Transactions***, published by the Royal Society of Chemistry (UK). It publishes articles and a few communications in all areas of organic and bioorganic chemistry.

(2) ***Journal of Organic Chemistry***, published by the American Chemical Society. It publishes articles, communications and notes covering all areas of organic chemistry.

(3) ***Journal of European Organic Chemistry***, published by Join Welly in English. It publishes articles covering all areas of organic chemistry.

(4) ***Journal of American Chemical Education***, published by the division of chemical educa-