高等学校双语推荐教材

# **Basic Experiments of Chemical Engineering**

(化工基础实验)

天津大学化工学院 韩 优 范江洋 付 雁 肖晓明 编译

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### 内容提要

本书内容分为两部分。第一部分介绍化工原理实验涉及的理论知识,涵盖了科学安排实验和定量评价实验结果的方法,以及传统和现代的实验方法、测试技术与技巧,具体包括实验误差的分析与估算、实验数据处理、正交试验设计方法和化工实验参数测量技术;第二部分主要介绍化工单元操作实验,包括流体流动综合实验、传热综合实验、正交过滤实验、多相搅拌实验、洞道干燥实验、精馏实验、吸收实验和萃取实验,每个实验包含了实验目的、实验原理、操作步骤、注意事项、实验流程图和实验报告要求等,同时还介绍了化工实验室安全规定和相关的安全知识。

本书注重化工基础实验中的共性问题,拓宽基础,有较强的通用性, 既可作为高等学校化工、化学、环境工程、食品科学与工程、材料等专业的国际化教材,又可供科研和实验工作者参考。

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# 前言

21 世纪是全球化的时代,全球化时代要求高等教育培养国际化人才,而构建国际化课程体系是高校培养国际化创新型人才的基础和核心。化工基础实验作为化工、化学、材料、环境等专业的重要基础课程,其国际化水平直接影响专业课程的国际化程度。教材是一切教学的基础,为了更好地开展化工基础实验国际化教学,我们编著了这本化工基础实验英文教材。本书强调对学生进行实验研究全过程的多种能力和素质的培养与训练,力求通过实验培养学生掌握综合运用理论知识、解决实际问题和正确表达实验结果的方法,开拓学生的实验思路,增强创新意识,并将实验技能的传授和语言的训练有机融为一体,推进我国国际化教育的进程。

本书内容分为两部分:第一部分介绍化工基础实验涉及的理论知识,涵盖了科学安排实验和定量评价实验结果的方法,以及传统和现代的实验方法、测试技术与技巧。具体包括实验误差的分析与估算、实验数据处理、正交试验设计方法和化工实验参数测量技术。第二部分主要介绍化工单元操作实验,包括流体流动综合实验、传热综合实验、正交过滤实验、多相搅拌实验、洞道干燥实验、精馏实验、吸收实验和萃取实验等8个实验。每个实验包含了实验目的、实验原理、操作步骤、注意事项、实验流程图、实验报告要求等,同时还包括了化工实验室安全规定和相关的安全知识。本书注重化工基础实验中的共性问题,拓宽基础,有较强的通用性,既可作为相关高等学校的国际化教材,又可供科研和实验工作者参考。

本书由韩优主编, 范江洋、付雁、肖晓明参与编写。在编著的过程中,参考了国内外相 关的教材,也得到了天津大学化工学院化工基础实验中心所有老师的大力支持,在此一并表 示衷心的感谢!

鉴于编著者学识有限,书中难免有不妥之处,衷心希望读者给予指教,帮助本书日臻完善。

韩优 2017年3月于天津大学北洋园

# **Preface**

The 21st century is the age of globalization, which requires that the higher education should cultivate international talents. For colleges and universities, building an international curriculum system is the foundation and core of cultivating international innovative talents. Basic Experiments of Chemical Engineering is an important foundation course for the major of chemical engineering, chemistry, material, environmental engineering and so on. The degree of its internationalization directly affects the internationalization of the professional courses. The textbook is the foundation of the teaching. In order to carry out the international teaching of basic chemical engineering experiments preferably, we have compiled the English textbook of *Basic Experiments of Chemical Engineering*. The textbook emphasizes on training students' abilities and qualities during the process of experimental research. It strives to cultivate the students to master the comprehensive use of theoretical knowledge, to solve the practical problems and to express the experimental results correctly. Besides, the students' experimental thoughts and their consciousness of innovation can be developed through the Basic Experiments of Chemical Engineering. The textbook combines the teaching of experimental skills and the training of language skills, which contributes to the promotion of international education in our country.

This textbook includes two parts. The first part is the introduction of theoretical knowledge involved in the basic chemical engineering experiments, including the scientific methods of arrangement experiments, the quantitative evaluation of the experimental results, the traditional and modern experimental methods as well as the testing techniques and skills. The Chapter 1 is the estimation and analysis of experimental errors, the Chapter 2 is the experimental data processing, the Chapter 3 is the orthogonal experimental design method and the Chapter 4 is the measurement of common physical parameters in chemical industry. The second part is the introduction of experiments on eight unit operations of chemical engineering, including the fluid flow experiment, heat transfer experiment, orthogonal filtration experiment, multi-phase agitation experiment, tunnel drying experiment, distillation experiment, absorption experiment and extraction experiment. Each experiment contains experimental objective, theory, operations, precautions, flow chart, report requirements and so on. The second part also includes the chemical laboratory safety precautions and the relevant safety knowledge. The textbook focuses on the common problems during the chemical engineering experiments and has the strong versatility. The textbook can be used as the international teaching material in related colleges and universities. It also can be an English reference book for the scientific researchers and the experimental engineers.

This book was edited by You Han, Jiangyang Fan, Yan Fu and Xiaoming Xiao. During the compiling process, we have referred many domestic and foreign textbooks, and received the

support from all the faculties in the Experiment Center of Basic Chemical Engineering of TianJin University. We would like to express our heartful thanks to you all.

Due to the limitation of the contributors' knowledge, there may be something inadequate in the book. We sincerely hope the readers can give us any advice or comment to improve the

You Han

March 2017, in Tianjin University Peiyang Park

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# Introduction

# 1. Main features of Basic experiments of chemical engineering

Basic experiments of chemical engineering is a highly practical basic course on engineering and technology, in which the basic principle of science and the experimental methods of engineering are utilized to solve practical problems in chemical engineering and its related fields. Its purpose is to solve multi–factor, multi–variable, comprehensive and industry–related problems. Therefore, it has significant reality and particularity.

- (1) Basic experiments of chemical engineering closely interrelates with classroom teaching, practice, chemical engineering design, etc., forming an organic whole. In this course, students should observe experimental phenomena, such as flooding, fluidization, etc.; measure basic parameters, such as temperature, pressure, flow, etc.; find out important laws of chemical engineering processes, such as fluid flow in the pipe or through the particle bed, etc.; measure the performance of chemical engineering equipments, such as the performance of a centrifugal pump, the plate efficiency of a distillation column, the number of mass—transfer unit of an absorber and so on. Therefore, Basic experiments of chemical engineering is an important pathway to consolidate theoretical knowledge on transfer and unit operation of chemical engineering, as well as to master new knowledge and skills.
- (2) Basic experiments of chemical engineering focuses on practical engineering, and differs from experimental teaching of other basis courses including physics, inorganic chemistry, organic chemistry, analytical chemistry, physical chemistry, etc. The object of this course is to solve practical problems of chemical engineering involving multiple variables and different methodologies. Therefore, it is not appropriate to indiscriminately use common methodologies from physical or chemical experiments in the experiment of chemical engineering. It is important to experience engineering processes and grasp universal methods on solving engineering problems. Besides, apparatus used in this course is quite different from other basic experiments. They are similar or identical to practical apparatus in the actual chemical engineering process. Every experiment is equivalent to a basic process in the chemical engineering production. The conclusions obtained from these experiments have guiding significance on designing apparatus of unit operation and determining operation parameters of chemical processes.
- (3) Owing to the complexity of chemical engineering process, it is difficult to find out an appropriate theory to explain the influence of engineering factors. Although theoretically qualitative description can be made, it is hard to give quantitative analysis on many chemical engineering processes. To obtain or determine some critical design or operational parameters, it is essential to carry out experiments rather than theoretical calculations. For some students or technicians who initially contact unit operation, it is also necessary to strengthen their

understanding on chemical engineering processes or apparatus through doing experiments.

Therefore, it is beneficial for undergraduate students majored in chemical engineering to study *Basic experiments of chemical engineering*. It is an important way not only to help them in theoretical courses, but also in studying experimental methods, technical skills and knowledge, to improve their abilities to solve practical engineering problems in their future career.

# 2. Requirements of pre-lab

Because of the particularity of *Basic experiments of chemical engineering*, we require students to prepare experiments carefully to make sure to be aware of apparatus, flow chart, control points, security of unit operation. The concrete requirements are as follows.

- (1) Read guide books, theoretical books and related reference books carefully. Be aware of experimental purpose, task and requirement. Analyze theoretical basis according to the experimental task. Understand flow chart and apparatus design. Find out data to be measured and develop preliminary experimental procedures.
- (2) Get familiar with experimental apparatus and procedures. Find out control points. Understand apparatus, meters, setting up procedures and control methods. Keep in mind the key points of operation as well as attentions.
- (3) To obtain an effective relationship of different parameters, determine the data range and interval preliminarily, and estimate changing rules of experimental data.
- (4) Write pre-lab report including experimental purpose, task, principle, flow chart, procedure and attention. Design original data table, and write down name, representative symbol and unit of each physical quantity to be measured.

# 3. Experimental attentions

Experimental operation is the core of experiment teaching since it is the only way for students to understand the apparatus of unit operation, achieve the optimization of process, analyze unusual experiment phenomena and take effective measures.

- (1) Before experiments, check whether all the devices and meters are intact; whether all the motors, fans, and pumps work well; whether all the valves are on the correct position. Setting up experiment is not allowed without careful checking.
- (2) During experiments, pay close attention to the meters, and regulate them in time. Make sure that experiments run at presetting conditions. Don not record data immediately after changing experimental conditions. It usually takes some time to reach the stable status of a chemical engineering process. Moreover, all the meters possess intrinsic hysteresis phenomena to some extent. Therefore, it is important to record data under stable conditions.
- (3) Avoid operating or recording without observing experimental phenomena. Experimental phenomena are greatly associated with intrinsic mechanism of many chemical engineering processes, e. g., the relationship between two-phase contact and plate efficiency. Therefore, good habits of observation are necessary characteristics of an engineer.
- (4) When unusual phenomena occur during the experiments, or experimental data have

significant errors, describe them in your laboratory report. Group members should discuss the related reasons with teachers. Find out and solve problems in time, or give reasonable analysis and explanation of experimental phenomena in your report.

- (5) Record the experimental data on the original data table prepared before experiment. Guarantee that the data are clear, reliable, intact, and can reflect the accuracy of meters used in the experiments. Normally data should be recorded at the next digit of the minimum scale of a meter. Check data carefully to avoid accidental mistakes. Physical quantity should be accompanied with name, symbol and unit.
- (6) Students can not stop the experiments without the checking of original data by teachers. Switch off apparatus, gas, water, electricity, etc., according to the operation procedures. Clean up the laboratory before leaving.

# 4. Basic contents of a laboratory report

Laboratory report is a primary estimation on experimental object and work. It is also a comprehensive and systematical summary, which is an essential part of an experiment. Writing laboratory report is a process of processing measured data, analyzing observed phenomena, finding out objective rules and internal relations. Therefore, writing laboratory report is a basic training for undergraduate students majored in science or engineering.

The contents of a laboratory report are as follows.

- (1) Title. Experiment title should be simple, clear, accurate and understandable. Try to use words as little as possible to reflect your experiment contents, e. g., measurement of fluid flow friction.
- (2) Purpose. Give concise explanations on the reasons for doing this experiment, and list all the problems to be solved.
- (3) Principle. Concisely explain the experimental principles, including main concepts, important laws and formula. It needs to be accurate and sufficient.
- (4) Flow chart. Draw a schematic figure of the equipment used in the experiment, including the labels of devices, meters, valves and measuring points. Write down the figure legends and the corresponding names of all the labels below.
- (5) Procedures and security points. Practical procedures can be divided into several steps, normally based on changing one parameter to another. The explanations of different operation steps should be simple and clear.

Special security points should be included in the report to attract your attention during the experiment. Pay attention to some operations which are liable to cause danger or damage of equipments.

(6) Data recording. Experimental data are direct values read from instruments or meters. The significant digit is determined by the accuracy of the measuring meter used in the experiment. Read data correctly and record them accurately. Record experimental data in the original data table, or put them in the appendix of the lab report when the data are lengthy.

- (7) Data sorting table and graphic illustration. This is one of the most important parts in the laboratory report. Sort out and process experimental data in the form of table or graph. Data processing should obey the rules of significant digit. Normally intermediate and final calculation results are shown in a data processing table. This table should be designed elaborately to easily display the changing rules and the parameter correlations. Sometimes in order to illustrate interrelations intuitively, graphic methods are usually adopted. The requirements of tabulation and graphic method are systematically described in Chapter 2. Data can not be modified without repeating the experiment. Furthermore, it is forbidden to fabricate experimental data.
- (8) Example of data processing. To show how to obtain the results shown in the table or graph, take one set of your raw data for example, and list each step of the calculation process.
- (9) Result analysis and discussion. This part is very important because it is a concrete manifestation of one's theoretical level. Meanwhile, it is also general analysis of experimental methods and results. Discussion is only related to your experimental content, which includes: (I) Analyze and explain experimental results using appropriate theories, demonstrating the inevitability of experimental laws. (II) Analyze and discuss unusual phenomena in the experiment. (III) Analyze the degree and possible sources of experimental errors, and how to improve the measuring accuracy. (IV) Demonstrate the practical value and the significance of your experimental results in the actual chemical engineering production. (V) Propose further research directions or suggestions for the experimental methods and apparatus.
- (10) Conclusion. Conclusion is a final judgement on the basis of your experimental results. It relies on both practice and theory.

# Chapter 1

# **Estimation and Analysis of Experimental Errors**

Due to imperfect experimental methods, apparatus, surrounding environments, measuring meters, as well as human observation, there usually exist inevitable deviations between experimental data and true values. Numerically this is demonstrated in error, which is inevitable and universal. In order to reduce or eliminate errors, it is necessary to study and measure experimental errors. Through estimation and analysis of experimental errors, we can understand the sources and the effects of errors, and determine main factors leading to experimental errors. Moreover, in the experimental preparation, we can properly organize experimental process, rationally use apparatus and measuring methods, reduce or eliminate sources of errors, finally improve experimental quality.

# 1.1 Experimental Errors

# 1.1.1 Direct and Indirect Errors

Measurements are divided into direct measurements and indirect measurements, depending on different measuring methods. A direct measurement value is directly read from instruments or meters. For example, length by ruler, time by stopwatch, temperature by thermometer, pressure by pressure gauge, etc. An indirect measurement value is obtained through processing direct values by certain functions. For example, in the determination of the volume V of a cylinder, we firstly measure the height H and the diameter D, and then we calculate the volume V through the formula  $V=\pi D^2H/4$ . In this case, the volume V is an indirect measurement value. In chemical engineering principle experiment, most of the measurement values are indirect.

# 1.1.2 True Value of Experimental Data

True value is an objective value of a certain physical quantity. Due to imperfect apparatus, methods, environments, person and procedures, experimental errors are inevitable in the measurements. Therefore, a true value is an ideal value and is not able to be measured. In the analysis of measurement errors, normally we use the following values instead of true values.

## 1.1.2.1 Theoretical true value

This kind of true value is able to be theoretically confirmed. For example, interior planar angles of a triangle sum to 180°; metrological values decided by General Conference of Weight and Measures, such as thermodynamic temperature 0~273.15 K; some values expressed by theoretical formula, etc.

### 1.1.2.2 Relative true value

In some measurements, we often use the relative true value measured by high accuracy class instruments to substitute the true value. For example, compared with common flowmeters, the flow measured by a high class turbine flowmeter can be accepted as a true value.

## 1.1.2.3 Average value

The average value is an average result of a physical quantity calculated by a number of measurements, which is used to substitute the true value. If the number of measurements is infinite, the average value should be very close to the true value. Actually the number of measurements is limited, therefore, the average value is approximately close to the true value.

# 1.1.3 Definition and Representation of Error

### 1.1.3.1 Definition of error

The error is a deviation between the measured value (direct or indirect value) and the true value (objective value). It can be expressed as follows.

Error=Measured value-True value

Obviously, the magnitude of error reflects how the measured value deviates from the true value.

## 1.1.3.2 Representation of error

(a) Absolute error and relative error

The absolute error is the deviation of the measured value x and the true value A:

$$D(x) = |x - A| \tag{1-1}$$

In the engineering calculation, the true value A is usually substituted by the average value  $\bar{x}$  or the relative true value, then Eq.(1–1) is changed to be:

$$D(x) = |x - \overline{x}| \tag{1-2}$$

Although the absolute error is important, it is not sufficient to explain the accuracy of measurement. In other words, it is not able to totally reflect the accuracy of measurement. Also, sometimes the same absolute error may result in completely different results. For example, in weight measurement, the quality of measurement can not be judged from the maximum absolute error. If the object weights dozens of kilograms, the maximum absolute error of 1 g shows high accuracy. If the object weights only a few grams, this measurement doesn't make any sense.

Obviously, in order to determine the accuracy of measurement, it is necessary to compare the absolute error with the measured value. The relative error is the ratio of the absolute error and the measured value:

$$E_r(x) = \frac{D(x)}{|A|} \tag{1-3}$$

Using the average value  $\bar{x}$  to substitute the true value A:

$$E_{\rm r}(x) \approx \frac{D(x)}{|\overline{x}|} = \frac{|x - \overline{x}|}{|\overline{x}|}$$
 (1-4)

The measured value:

$$x = \overline{x}[1 \pm E_r(x)] \tag{1-5}$$

Pay attention that the absolute error is a dimensional value, while the relative error is a dimensionless proper fraction. In chemical engineering experiment, the relative error is usually expressed as % or ‰.

- (b) Arithmetic mean deviation  $\delta$  and standard deviation  $\sigma$
- (i) Arithmetic mean deviation for measuring *n* times:

$$\delta = \frac{\sum_{i=1}^{n} |x_i - \overline{x}|}{n} \tag{1-6}$$

Don't forget the absolute value in the above function. Otherwise, the algebraic sum of  $(x_i - \overline{x})$  will be zero.

(ii) Standard deviation for measuring n times:

$$\sigma = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n-1}}$$
 (1-7)

(iii) Relationship and difference between arithmetic mean deviation and standard deviation:

For n-times measurements, the worse repeatability is, the greater degree of dispersion is, the greater random error is, and the higher  $\delta$  and  $\sigma$  are. Therefore,  $\delta$  and  $\sigma$  are used to measure the repeatability, the dispersion degree and the random error. The arithmetic mean deviation is not able to show fluctuations of each measurement. The arithmetic mean deviation of the measurements with wide dispersion is possible to be equal to that of the measurements with narrow dispersion. In contrast, standard deviation is sensitive to fluctuations and demonstrates dispersions better.

Example 1-1 Two groups of measured results are listed below (unit of cm):

Group A: 4.3 4.4 4.2 4.1 4.0

Group B: 3.9 4.2 4.2 4.5 4.2

Try to solve the arithmetic mean deviation and the standard deviation.

Answer: The average values:

$$\overline{x}_{A} = \frac{4.3 + 4.4 + 4.2 + 4.1 + 4.0}{5} = 4.2$$

$$\overline{x}_{B} = \frac{3.9 + 4.2 + 4.2 + 4.5 + 4.2}{5} = 4.2$$

The arithmetic mean deviation:

$$\delta_{A} = \frac{0.1 + 0.2 + 0.0 + 0.1 + 0.2}{5} = 0.12$$

$$\delta_{\rm B} = \frac{0.3 + 0.0 + 0.0 + 0.3 + 0.0}{5} = 0.12$$

The standard deviation:

$$\sigma_{A} = \sqrt{\frac{0.1^{2} + 0.2^{2} + 0.1^{2} + 0.2^{2}}{5 - 1}} \approx 0.16$$

$$\sigma_{B} = \sqrt{\frac{0.3^{2} + 0.3^{2}}{5 - 1}} \approx 0.21$$

From the above example, although group A and B have the same arithmetic mean deviation, their dispersion degree are obviously different. Standard deviation can reflect the dispersion degree of measured data. The more accurate experiment is, the smaller standard deviation is. Therefore, we usually employ standard deviation to estimate the magnitude of random errors.

(iv) Relationship between the standard deviation and the absolute error

The absolute error of the average value  $\bar{x}$  for measuring n times:

$$D(\overline{x}) = \frac{\sigma}{\sqrt{n}} \tag{1-8}$$

The relative error of the average value  $\bar{x}$ :

$$E_{r}(\overline{x}) = \frac{D(\overline{x})}{|\overline{x}|} \tag{1-9}$$

From the above function, the smaller standard deviation  $\sigma$  and the more times measurements are, the smaller absolute error of the average value  $D(\bar{x})$  is. Therefore, it is an effective method to reduce random errors by increasing measurement times n.

### 1.1.4 Classification of Errors

Errors are classified as systematic errors, random errors and faulted errors, according to their properties and sources.

### 1.1.4.1 Systematic errors

Systematic errors are caused by some fixed factors. For multiple measurements under the same conditions, both the magnitude and the plus-minus of errors remain constant, or change with certain rules. Therefore, some systematic errors show linear, non-linear, or periodic changes with time, and some of them are not time-dependent.

Sources of systematic errors: (I) Measuring instruments (design drawback, non-standard manufacture parts, improper installation, non-calibration, etc.); (II) Environmental factors (surrounding temperature, humidity and pressure); (III) Measuring methods (approximation method or approximation function); (IV) Habit or bias from an operator.

In summary, systematic error has fixed bias and defined rule. Generally it can be corrected or eliminated by special measurements or functions.

### 1.1.4.2 Random errors

Random errors are caused by some factors which are not easy to be controlled. For multiple measurements under the same conditions, both the magnitude and the plus-minus of these errors are uncertain. Random errors do not have fixed magnitude and bias. They usually obey statistical laws,