

CHE 302
CHEMICAL ENGINEERING LABORATORY I

by

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4th Edition

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PREFACE TO THE FOURTH EDITION

Welcome to the first course that ties everything together in the Chemical Engineering curriculum. You have at this point taken about five core chemical engineering courses and are enrolled in two others. After building a rigorous theoretical background in these courses, the Che302 lab is quite different not only because it is based on hands-on application of theoretical principles, but also because this is the first time you observe how real life can often diverge from a simplified theoretical approach, and how the nonidealities must be taken into account by the engineer. The groundwork for chemical engineering design, which is the ultimate goal of the engineer, is thus laid.

This “future engineer”, you, who is trained extensively to consider several parameters at once, will also need to explain your findings or observations in meetings or in written reports. These meetings, reports, or articles are often prepared by a group of coworkers and often have a deadline. The Che302 class, with the weekly reports and the final presentation, provides ample opportunity to hone your communication and time management skills.

Dear students, you will most likely spend a considerable amount of time for your first report. Application of theoretical principles for a given question (technical skills) while you pay attention to format (communication skills) has a learning curve. But, by the third report, I promise you that report writing for this class or for other classes will be easy as 1-2-3 for you!

There are a few people I would like to thank. First and foremost, our tireless and meticulous lab coordinator, Melek Selcen Başar. She is the locomotive in this class, keeping me, the teaching assistants and the student groups working with Swiss watch accuracy. We are very lucky to work with Esmâ Toprak, our laboratory supervisor, who keeps our laboratories in top shape. I thank her for her efforts throughout the years. I would also like to thank our chemist, Belgin Balkan, for maintaining our laboratory equipment and chemicals.

This year we overhauled the lab booklet to bring it up to date and to include more and detailed information particularly on theoretical background and safety. All teaching assistants spent their winter break on revising sections of the booklet. Utku Deniz and Coşar Doğa Demirhan helped me in the final edit to streamline the individual sections. I would also like to thank all teaching assistants for this huge effort. The author list for this booklet is in fact quite long and I would like to thank all past and present teaching assistants and faculty members who have contributed to the booklet and to this course.

Assoc. Prof. Elif Özkırmılı Ölmez

PREFACE TO THE THIRD EDITION

Over the past three years, several modifications have been made on the second edition. Ten new experiments have been added to the previous ones by the efforts of our research assistants. These new experimental set-ups are listed in Appendix A. Chapter 2, which is on laboratory safety, has been extensively revised and expanded with the help of our Laboratory Supervisor.

It has been a pleasure for me to guide the operation of the ChE 302 course for the last three years. Having previously served as a teaching assistant at the Chemical Engineering Department for five years, I should acknowledge that the main contributors to this course are the teaching assistants (15-20 TA's every semester). Therefore, I would like to express special thanks to Mehmet Mete Altıntaş (Laboratory Organizer) in the name of all the research assistants, who have helped me a lot so far. Some names that come to mind are Burak Alakent, Selen Aydoğan, Bahar Bilgen, Nebile Demir, Delal Dink, Mehmet Gökben, Sezen Gürdağ, Başak Işın, Defne Kayrak, Şafak Kırca, Neşe Kurt, Mehmet Mercangöz, Levent Obut, Orhan Oruç, Banu Özkan, Sinem Özyurt, Berna Sarıyar, Başak Saygılı, Yeliz Turgut, Kerem Uğuz, Korkut Uygun, Pınar Pir Yunt. Last but not least, I would like express my deepest appreciation to Esmâ Toprak (Laboratory Supervisor), Belgin Balkan (Chemist), and Bilgi Dedeoğlu (Laboratory Technician) for their continual efforts through the years. I am sure that our efforts will continue in the future semesters to improve our existing laboratory facilities and provide an up-to-date education to our students.

Assoc. Prof. Pemra Doruker

PREFACE TO THE SECOND EDITION

Today, the discipline of Chemical Engineering is still continuing to contribute in numerous ways to worldwide industrial progress. Significant strides have been made in process and equipment design methods. Similarly, there have been noticeable changes in the equipment and existing experimental procedures in our laboratory. With the arrival of new equipments and new parts, more experiments have been developed and the existing ones are revised. Thus, present laboratory manual represents an attempt to bridge the previous edition with the current and future trends and needs.

A number of contributions have been made since publication of the first edition. Each of the fourteen chapters has been revised and updated. Ten new experiments have been added to widen the scope on the applications of the basic fundamentals of Chemical Engineering as well as to incorporate the new coming equipments on the subjects of extraction, control and heat transfer. The list of the new experiments is given in Appendix A.

In preparing this second edition we intended to extend those features that have facilitated self-study. The theory parts were rewritten in detail giving a presentation of organized concepts and useful relationships. An introductory reference source is given at the end of each experimental section. A list of keywords is also provided as a further assistance in the literature survey. Figures were redrawn by computer. The details of each apparatus on the figures, the written specifications and the introductory explanation of each apparatus were extended. Finally, the report objectives, i.e. the discussion topics, were revised. The present booklet is the result of three years of teaching of ChE 302 and ChE 401- Chemical Engineering Laboratory I and II, respectively, at the Boğaziçi University. I am vastly indebted to many people who assisted in the preparation of this second edition. Nilgün Akın, Erhan Aksoylu, Mehmet Mete Altıntaş, Nihat Baysal, Gülnur Birol, Cantürk Boyacı, Banu Boz, Petek Çakar, Mete Demircan, Pemra Doruker, Saybil Ercan, Elif Hamurcu, Ravza Karaselçuk, Özlem Keskin, Alp Er Konukman, Orli Navaro, Seza Orçun, Banu Özkan, Leyla Özkan, Pınar Özkan, Berna Sarıyar, Can Şenol, Ebru Toksoy, Derya Uztürk, Candan Yıldır, Serhan Yemez. To all of these graduate assistants and others who have contributed, I want to express my sincere gratitudes. I particularly acknowledge Mehmet Mete Altıntaş, Can Şenol and Derya Uztürk who did all the computer work, i.e., typing, formatting and drawing, and thus made the appearance of this second booklet possible. I regret it is impossible to give individual thanks to the innumerable students at their 3rd and 4th year of Chemical Engineering Department who have contributed in their way to this new edition. Students should know that their contributions are greatly appreciated.

Assoc. Prof. Kutlu Ş. Ülgen

PREFACE TO THE FIRST EDITION

This booklet has been prepared for the use of our students taking the first Chemical Engineering Laboratory course, presently ChE 302, in our curriculum. It is an edited and expanded compilation of various individual leaflets we have used over the past many years. The first chapter covers the basic information about the course and a suggested course outline. Chapter Two is on laboratory safety; students, please do not overlook it! Good and correct safety habits will stop many serious injuries and even losses of lives both in labs and plants. Starting with the third chapter the experiments are grouped on the basis of fundamental subjects with a short general introduction for each chapter and some general references concerning the subject matter. On each subject some basic introductory information is given on the fundamentals of the experiments in the chapter. Furthermore, some more specific information as well as the minimum requirements for the experiment reports, which the students are asked to prepare, are also given for each experiment. We hope that this booklet would serve our students' needs better and thus make our lab course more orderly, productive and efficient.

Since the founding of our Department in 1958 by the late Prof. Turgut NOYAN, efforts with limited funds and personnel have been made for the establishment of a basic and functional Chemical Engineering Laboratory. The first version of such a laboratory was operational in the very early 1960's. A concentrated attempt to improve and renovate the laboratory was made in the early 1970's. At that time new and additional experimental kits and equipment were acquired. However, this time laboratory floor space turned out to be the major bottle neck and many of the newly acquired equipment couldn't be set up. We moved into our present laboratories in early 1980's and thus we were able to set up all the experimental equipment, even those which were still in crates. We now have a fully functional lab which we continuously try to improve by acquiring, developing and/or constructing additional equipment for new and improved experiments.

Even a long list won't suffice to mention the names of all those members of our Faculty and Staff ("Staff" definitely includes all those Graduate Assistants who really did and still do most of the work in the lab and in the courses associated with it!) who gave many days, weeks, months and years of their time and lots of physical and mental effort in trying to set up the labs and make them function safely and economically over the years. I am surely hopeful that we will show the same performance in the years to come. Lately I notice that some of our students who have been using the lab are second generation budding chemical engineers. They use basically the same lab as their parent(s) have but with newer and more sophisticated equipment. On the other hand, some of us

who were students or graduate assistants in the lab are now members of the Departmental Faculty. Here I would like to take the opportunity to thank all those who have contributed to the construction and safe operation of the lab over the past many years. Thanks also are due to those contributions, monetary or in kind, from our alumni and some concerned industrial companies. Quite frequently these contributions are of vital importance. I sincerely hope and believe that we will all work together, in the future as in the past, to have a better lab with even more exciting and varied experiments and safe operation.

Prof. Öner HORTAÇSU

1. CHEMICAL ENGINEERING LABORATORY

The design and contents of chemical engineering laboratories may vary considerably depending upon the institutions in which the laboratories are established. These institutions may be universities, or at times government or private research institutions. However, even such variances may occur at the more detailed levels, in general all chemical engineering laboratories may be considered to be similar since they all contain various equipment set-ups designed to experiment with chemicals in various forms. These experiments may be designed for the production or development of new or known chemicals with known or new processes which may still be in their development phase. Furthermore almost all chemical engineering laboratories have facilities to develop, test or demonstrate some or many of the accepted unit operations of chemical engineering. Another common feature of chemical engineering laboratories may be the testing and characterization of the thermal, physical and chemical properties of chemicals. Dynamics and control of unit operations and complete processes may be yet another facet of some chemical engineering laboratories. Thus even though all chemical engineering laboratories may have few or many features in common it is almost impossible to talk of a standard chemical engineering laboratory.

In the curriculum of Department of Chemical Engineering of Boğaziçi University there are two laboratory courses which are presently numbered as ChE 302 and ChE 401, i.e., Chemical Engineering Laboratory I and II respectively, which are offered to the students in the sixth and seventh semesters of the eight semester undergraduate curriculum. Both courses carry a three semester credit load with one lecture hour per week and one afternoon (nominally four hours) of laboratory work. These two courses for most part are conducted in the same laboratory spaces and sometimes similar equipment are used in both. However, the aims and the emphasis of these courses are very different. ChE 302, i.e., Chemical Engineering Laboratory I, is made up of specific and closely defined experiments assigned to students with the intention of making them familiar with the actual applications of the fundamental concepts covered in the lecture courses. On the other hand ChE 401, i.e., Chemical Engineering Laboratory II, consists of fewer but more comprehensive and open ended small project type assignments and has ChE 302 as a pre-requisite. The intention of this course is to make the students get used to working semi-independently in the laboratory in a project oriented atmosphere as they develop new experimental set-ups and/or take comprehensive sets of data on existing experimental set-ups and analyzing their findings on the basis of the fundamental concepts involved. The presentation and defense of the findings in these project oriented experiments constitute an essential and integral part of ChE 401. This booklet is aimed

towards the ChE 302 Laboratory with the aim of making the course easier to deal with for the students. No book is intended for ChE 401, since it cannot be standardized in any way in its present form.

1.1. Aims of the Chemical Engineering Laboratory Course

Chemical Engineering Laboratory I, i.e., ChE 302, aims to familiarize the students with some of the basic concepts of chemical engineering and concentrates mostly on fluid mechanics, heat transfer, reaction engineering, process control and modeling, and mass transfer experiments. Another major aim of the course is to familiarize the students with the concepts of accuracy and precision as well as the fundamentals of the application of statistics in engineering experimentation as it is related to model building, model parameter estimation and experimental design. This part of the course is introduced mostly in the one hour lecture period held each week.

The course is not intended to be parallel to one or more theoretical courses present in the Chemical Engineering curriculum. It is felt that to do an experiment before one is formally introduced to the theory relevant to the experiment has as many advantages as the reverse procedure. To have done the experiment prior to a formal introduction to theory will definitely help a faster and firmer understanding of the theory when it is introduced. On the other hand, if the formal introduction to theory has preceded the experimentation it should help the experimenter to grasp and perform the experiment easier and also help reinforce the available theoretical background. All students taking ChE 302 will have a chance to do at least a few experiments in to which they have not yet been formally introduced as well as doing some other experiments about which they already have been introduced to the theoretical fundamentals concerning the experiment.

In ChE 302 laboratory students are asked to work in groups of three and perform nominally seven experiments during the semester. Because the number of student groups since about 1986 has been exceeding ten, it is no longer possible to give all groups the same set of experiments as it was previously done. Different sets of seven experiments assigned to each group are prepared to include a balanced set of small and large scale experiments as well as to include experiments concerning the more frequently used unit operations in chemical engineering practice.

1.2. The Organization of the Course

The information given below is a summary of the past many years' practices and it is quite possible that in the future the course will be organized roughly in a similar fashion. However, the actual organization of the course is the responsibility of the faculty member who is assigned to the course for that specific semester. It is totally up to the specific faculty member responsible for the course to alter any parts or all of the course organization based on various factors and conditions which he/she may consider to be sufficient for such a change.

As already noted above in ChE 302 students work in groups of three normally left to the wish of the students. At times one or two groups of two students may be permitted if the number of students taking the course warrants such a structure. The group members may have to work long hours together for the preparation of reports. Thus students should be careful in the organization of the group to ensure a maximum of harmonious working atmosphere.

The set of experiments and the schedule, i.e., the dates at which these experiments should be performed, is posted within the first few days of the semester to the attention of all students taking ChE 302. Chemical engineering graduate assistants are appointed as the supervisors for each experiment. These persons are fully responsible for the experiment and the students are directly responsible to them. Each group is expected to submit a report for each experiment performed at the report due date and time to their Supervisor (Graduate Assistant) responsible for the experiment. The reports are due in one full week, i.e., the next laboratory period.

All three members of a group should be present in the laboratory at the scheduled time to be able to do the experiment assigned to their group for that week. All of the group members will be tested individually on their knowledge of the fundamentals relevant to the specific experiment for the week prior to the start of the experiment. A group may start their experiment only after they have received the formal O.K. from the Supervisor (Graduate Assistant) responsible for their specific experiment. The group members have to fully adhere to the instructions given by the Experiment Supervisors fully and promptly.

Starting with Chapter Three some introductory information has been given for your convenience for each experiment. These short introductions are intended to serve as preliminary guides for the experiment. They have not been prepared to serve as detailed guides to the relevant theory and to give the detailed calculation methods expected in the report you have to submit for the experiment.

It is up to the experimental group to search to relevant literature on the subject and to decide on the details of the report. The guidelines in this book constitute the less than the bare minimum of the information required in the report which has to be submitted on the experimental findings and their evaluation. So the students are expected to add considerable amounts of fundamental information and discussions of the experimental findings, i.e., results, to the report.

A list of basic literature sources you should refer to in preparing your reports as well as in getting prepared for the experiments is given at the end of each section. The students are urged to use all additional sources you feel are pertinent to your work. However, please be extremely careful to cite all reference sources used in your report using one of the few accepted referencing methods.

In your experiments you should take care in producing precise and accurate data. You should also make sure that your report includes a discussion about the precision and accuracy of your results in terms of accepted statistical methods of data and model evaluations.

The course also has a lecture period each week. Concepts of accuracy and precision, experimental design, statistical evaluation of data, basics of mathematical models and model parameter estimation techniques (curve fitting and regression) are some of the topics covered in this part of the course.

1.3. Evaluation of Student Performance in the Course

Student performance on the material covered in the lecture part of the course, such as error analysis, is evaluated independently. However, the students are asked to apply these techniques in their experiment reports as much as possible. On the other hand, students taking the Chemical Engineering Laboratory I, presently ChE 302, are evaluated and graded on the following points for their performance in the laboratory.

- Evaluation before and after each experiment (on individual basis)
- Laboratory Performance (on individual basis)
- Report Evaluation (on group basis)
- Midterm and Final Exams (on individual basis)
- Presentation (on individual and group basis)

For a group to start an experiment on its due date, it is required that they have made some preparations. Thus, each member of the group is given a written quiz by the Graduate Assistant responsible for the experiment. The evaluation of this quiz is done on the spot and the group members are allowed to go on with the experiment, if they have shown satisfactory performance.

Laboratory performance of individual group members is done by the graduate assistant responsible for the experiment depending on the individual contributions of each group member during the course of the experiment.

The laboratory reports are handed in according to an accepted format by the students one full week after the experiment to the appropriate graduate assistant, i.e., the person who supervised them on that specific experiment a week ago. The report is evaluated by the graduate assistant and the course instructor according to accepted standards in the course.

Two exams are given to evaluate the performance of students on an individual basis. These exams cover the specific experiments performed by the groups and the lectures given during the semester. Students will be asked to present one of their reports to the interested members of the Departmental Faculty and Staff as well as to their classmates. Finally, the course grade is given on the basis of a weighted calculation of the above individual points.