GETTING THE MOST OUT OF A LABORATORY COURSE

AZIZ M. ABU-KHALAF
King Saud University • Riyadh 11421, Saudi Arabia

The laboratory is the proper place for students to apply the theories and principles of chemical processes and unit operations that they have learned in the classroom. It can also be used to simulate industrial practices where the ability to operate plants, to perform original designs, and to modify existing processes is required. Operating a chemical plant requires that the chemical engineer be proficient in problem solving and troubleshooting, and over the years considerable efforts have been exerted to strengthen the links between industry and academia in order to attain that proficiency. Examples include laboratory experiments, practice schools,[1] research projects, summer internships in industry,[2] co-op projects,[3] and others.

This paper will review the current goals of a laboratory course and present the author's experiences in using laboratory time to cover several important topics related to both industry and academia. The subjects that will be discussed are safety procedures, startup and shutdown, troubleshooting, calibration with statistical applications, maintenance, and mathematical modeling and simulation. Different available experiments will be used to achieve these goals.

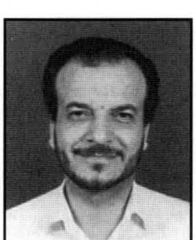
LABORATORY OBJECTIVES

Traditionally, a laboratory course emphasizes, through practical sessions, the understanding and application of theories and principles taught in the classroom and presented in textbooks. During the course, the students become familiar with the available process equipment, with instrumental analysis, and with various measurement techniques. They also practice report writing, deal with experimental errors, and learn to recognize the discrepancy between theory and practice.

A new look[4] with new objectives in the chemical engineering laboratory has been suggested, with some of the following objectives:

- *Using the laboratory to develop engineering awareness.*[5] Engineering awareness is developed through several applications. Students gain practical experience, acquire skills, and get an idea of the technical difficulties encountered in the design and construction of processing units.
- *Introducing statistical concepts in the experiments.*[6] Statistical ideas are incorporated into existing experiments, which are modified through the analysis of data, to introduce certain statistical concepts.
- *Mathematical modeling and simulation.*[7-10] A step-by-step method is followed to develop steady-state and dynamical models representing experimental systems. Students are asked to perform analytical and numerical solutions, using available simulation packages.
- *Troubleshooting.*[11] Troubleshooting experiments are described to develop the student's ability to diagnose and correct unacceptable process performance.
- *Simulation of industrial work.*[12] The procedures used in a typical unit operations lab course are modified to simulate industrial practice. Applied problems and instructions are included.
- *Performing economic evaluations.*[13] Estimations of capital and operating costs are performed on typical experiments. Scale-up and economic optimization of an existing plant are considered.

While some of these goals pertain to industrial practice, others reinforce mathematical, statistical, economical, and process concepts. New and/or modified experiments are developed as necessary to meet the goals, and other objectives can be included, such as familiarizing the students with safety regulations, maintenance, and calibration. It is not difficult to achieve all of these goals. The following sections describe our method for meeting the goals within the time



Aziz M. Abu-Khalaf *has educational interests that include developing new objectives and improving the performance of laboratories at the Chemical Engineering Department at King Saud University. Research interests include controlled release systems and corrosion. He can be reached by e-mail at AMKHALAF@KSU.EDU.SA.*

allotted to a typical lab course.

> *This paper . . . review[s] the current goals of a laboratory course and present[s] the author's experiences in using laboratory time to cover . . . topics related to both industry and academia. The subjects . . . are safety procedures, startup and shutdown, troubleshooting, calibration with statistical applications, maintenance, and mathematical modeling and simulation.*

SAFETY PROCEDURES

Students usually underestimate the importance of safety measures in the lab and occasionally argue about strict safety regulations, particularly when they feel the running experiment is safe. But experiments are not the only source of danger in the lab. For example, danger could come from a pipe carrying hot water or steam to other parts of the lab. To counter this, we emphasize the following items in such a way that they become part of the student's daily practice, both in and out of the lab:

- We familiarize the students with hazard symbols, terms, and abbreviations. Symbols include those on personal protective equipment, dangerous materials, and workplace labels. Terms such as threshold limit value (TLV), hazard rating (HR), lower and upper flammability limits (LFL and UFL), etc., are covered, and the most common abbreviations, such as ACGIH (American Conference of Governmental Industrial Hygienists) and MSDS (Material Safety Data Sheet) are defined. The concepts of fire triangle and tetrahedron are fully explained.
- We teach the students to always be ready for an emergency. We point out the location of emergency outlets, fire extinguishers, in-line showers, and emergency eyewash stations.
- We ask that students wear a lab coat (or apron) during each lab session, and that other personal protective items be used when necessary. For example, the students should wear, as needed, safety eyewear (splash goggles), a face shield, and gloves during distillation and reaction experiments, etc. We ask them to put on dust masks during a solid-handling experiment and anti-noise ear muffs during noisy runs such as a cooling tower tutor.

We also show the students a video tape on safety or refer them to related books.[14,15] We give them specific assignments on safety topics and discuss the topics later in a seminar session. In particular, we ask them to compare the safety equipment that is available in the lab with the recommendations in a standard reference. The students can also be asked to prepare data sheets on the specific materials and hazard symbols that will be involved in the experiment they plan to do (references 14 and 16 are very useful in this regard).

Finally, a sheet of safety procedures specific to a certain lab is distributed to the students. It contains general procedures (disposal, proper use of service equipment, gas cylinders, etc.), evacuation instructions in case of fire or chemical spillage, etc. It should also include symbols and abbreviations of equipment and pipelines in the lab.

The lab instructor might also include other topics such as hazard-assessment techniques. The most common is the hazard and operability study (HAZOP), which is a systematic technique for identifying all plant or equipment hazards and operability problems.[17]

STARTUP AND SHUTDOWN

In industry, startup (putting the process and its related equipment into proper operation) and shutdown (when the operation is completely stopped) are related to integrated equipment and interdependent processes. The equipment is often outside the plant and susceptible to weather effects, whereas the lab usually contains small-scale individual equipment with relatively constant ambient conditions.

Startup and shutdown procedures depend on the process, the materials, and the type of equipment being used. The following is a list of common rules used in industry—based on my own experience as a co-op student in a refinery and as a production engineer in other plants. The rules should be emphasized during the lab session.

- Never introduce a cold stream suddenly into hot equipment (such as a heat exchanger). Similarly, a hot stream should not be suddenly introduced into cold equipment.
- Introduce the cold stream first, and stop the hot stream first.
- Any change in operating conditions should be done gradually and should not exceed the listed operating conditions of the specific equipment.
- Make sure that the equipment is gradually, completely, and safely drained and purged.

An effective teaching method is to listen to the students' suggestions and ideas about startup and shutdown and then to discuss with them the significance and implications of the general rules above. This helps them to realize that a sudden change in temperature might cause a thermal or a mechanical stress and result in a bad effect on the equipment. For example, high pressure surges can destroy pressure gauges or other measuring devices or dislocate trays from a distillation column. They will also learn that rapid venting could result in a cooling effect that might freeze the remaining liquid, and that complete drainage and venting is necessary with flammable materials to avoid the formation of explosive mixtures.

Table 1 is a general guide of the steps that should be taken for startup and shutdown. Since startup and shutdown are transient in nature, they can be modeled mathematically and their behavior can be theoretically predicted.

TROUBLESHOOTING

Troubleshooting is the ability to characterize or diagnose a problem and to present corrective action to solve it. This ability is essential for people involved with the operation of chemical plants—in particular shift engineers. Abnormal operation can be harmful to operators, to equipment, and to product quality. Corrective action might be constrained by the time and safety of both the employees and the equipment, so a systematic approach should be followed in troubleshooting. This requires a good theoretical background in the process, experience, and engineering sense. A systematic approach requires the presence of enough and reliable (relative to operators and instruments) data and information about the situation, recognizing the problem, choosing the corrective action from the different alternatives, and later feedback. Knowledge-based systems[18] are currently used to improve plant operations by using automated diagnosis.

Trouble in industry can be caused by[19] such things as misoperation, false alarms, equipment or chemical failure, inadequate equipment design, and process failure. Lab experiments can be effectively used to familiarize students with troubleshooting procedures by designing special experiments for this purpose[11] or by using available experiments with problems that occur either naturally or that are created intentionally.

At the conclusion of the course, students should be asked to summarize their experience in the form of a list that other students can benefit from, revise, and extend if possible. In this way, they learn to keep records of all failure and breakdowns as is done in industry. There should be a thorough discussion of the cause and effect of the problem and how to solve it in light of the systematic approach mentioned above. Students should perform the required modifications when necessary. Table 2 gives some examples of typical problems, their cause, and possible corrective actions.

MAINTENANCE

Maintenance is all important. It keeps plants running, prevents troubles, and identifies the cause of inadequate performance.[19] The availability of maintenance software can help an operation to run smoothly.[20] When students locate a trouble source, they should participate in the repair and maintenance process. For example, they can dismantle (with the help of technicians) a malfunctioning pump for general checkup or repair; they can clean a dirty reactor or a packed bed; they can check valves for leakage; and when it is necessary to modify a design, they can do it themselves.

It is important to familiarize students with the important and related terminology of maintenance. This includes planned maintenance, corrective maintenance, routine maintenance, servicing maintenance, running maintenance, and shutdown maintenance. Other suggested projects include

- Cleaning equipment such as heat exchangers, cooling towers, coolers in distillation columns, etc. This requires understanding of fouling and scales and the methods of cleaning. The severity of deposits can determine the method to be used: in running maintenance, chemical treatment is applied without the need to dismantle the equipment; for heavier deposits, fluids under high pressure and/or temperature (such as steam) are used. When these methods fail, the equipment must be completely dismantled to remove the adhering deposits. This is known as shutdown maintenance.
- Water treatment. This can be accomplished by preventing the formation of deposits by using antifoulants, preventing the reactions that cause deposits by using inhibitors, and using dispersants to prevent coagulation of suspended solids.
- Noisy equipment. Fans and other equipment such as mixers should be frequently inspected since friction is a possible source of ignition.

TABLE 1
Steps for Startup and Shutdown

- ► Discuss with the students the objectives of the experiment, the measurement techniques and instruments, and the materials and the form of energy to be used. Relate the last with the safety section regarding precautions and handling.
- ► Ask the students to familiarize themselves with the system, to locate and check all the relevant equipment and valves (all types, including relief valves), and draw a simple but detailed flowchart, with full identification of all relevant parts. On a separate sheet, list all input and output valves and designate them as "open" or "closed" for startup.
- ► With the help of the above list, check which valves to open and which to keep closed. Refer to the guidelines given before, particularly if heating/cooling is involved. Make sure that the measuring equipment is ready. While other steps might be specific, when dealing with a reacting system you generally introduce both reactants simultaneously, or add one and then the other, or fill the tank with one reactant and then add the other stream.
- ► Follow up the experiment, take the measurements, and keep watching the input and output streams (through flowmeters), the heat source, and the workability of other equipment.
- ► Prepare yourself for shutdown. Again, refer to the valve list generated at the beginning of the preparation and to the general guide list.

- <u>Replacing steel pipes</u> in old equipment with plastic pipes.

CALIBRATION WITH STATISTICAL APPLICATIONS

The objective of calibration is to prepare calibration curves and to apply useful statistical techniques. Examples include constructing calibration curves of conductivity against concentration and recording liquid flow rate with a rotameter. In the case of conductivity, standard solutions are prepared and their conductivity measured by a suitable conductivity meter. The concentration range should cover the actual expected measurements. With a rotameter, an adjustable flow from a suitable pump is passed through the meter and its reading is recorded; then the effluent is measured at certain time intervals. A smooth curve can thus be generated.

Statistical analysis can reveal the presence of errors in the measurements and the propagation of these errors later in the calculation of an unknown sample concentration.[21] I recommend that the students perform the following calculations:

- Use linear regression to get the best fit. Calculate the slope and intercept of the calibration line and the percentage fit, which is 100 (correlation coefficient)^2.
- Calculate the residual standard deviation, which is equal to the square root of residual sum of squares divided by residual degrees of freedom.
- Decide whether there is a fixed bias or a relative bias, or both or neither. Fixed bias results in an intercept that is not equal to zero, and a relative bias results in a slope that is not equal to unity.
- Quantify the precision of the prediction of the true concentration of unknown samples.
- Several calibration lines can be collected from different groups, and a mean value can be calculated. A t-test can be used by each group to compare its value with this mean. Finally, a repeatability test can be used to check the precision of the test method. Groups will test similar unknown samples and quantify the repeatability as r=t 2s, where r is the repeatability measure, s is the standard deviation of the readings, and t is the t-test at a certain confidence limit.

Statistical analysis could also be applied to the experiments themselves, *e.g.,* determining the order of the reaction and the reaction rate constant from a batch reactor run. These values are then used in the continuous reactor calculations. It is appropriate to analyze propagation of errors throughout these calculations and to do some other significant tests on the value of the reaction rate constant.

Other suggested[5] calibration projects are a mercury-in-glass thermometer, a thermistor, and type-T and -K thermocouples.

MATHEMATICAL MODELING AND SIMULATION

Mathematical modeling is the process of describing and approximating actual physical systems using mathematical tools. A real process is mathematically abstracted for purposes of understanding and predicting its behavior. Reducing the experimental effort required to design or optimize the process is another motivation for developing a mathematical model. The model can be checked against experimental data and then reconsidered in order to be more effective and more useful in achieving the required objectives.

The first step in model formulation[22,23] usually involves drawing a picture of the system under investigation and selecting the important dependent (responding) and independent (changing) variables, along with the parameters that

TABLE 2
Typical Problems, Causes, Remedies

Experiment	*Problem*	*Possible Cause (Remedy)*
Cooling tower	Wet-bulb temperature equal to or greater than dry-bulb temperature of outlet air	• False reading (Check measuring devices) • Wick is not wetted (Check water and wick) • Air is blocked (Check air flow)
	Temperature gradient of water at the bottom section is very small	• False reading (Check measuring devices) • Lack of water (Check water flow) • Air is blocked (Check air flow)
Distillation	Conductivity readings from different trays are not consistent	• False readings (Select the correct range at the calibrated temperature) • Incorrect sampling (Cover drawn samples with aluminum foil, cool them to calibration temperature using a water bath, and make sure probe is well immersed in the sample.)
Reactor	Fluctuated flow, with air bubbles in flow lines	• Direct pumping within short distance (Use head tanks. If bubbles are still present, increase the tank height.) • Low level in feed/head tanks (Increase the level of solution in the tanks.) • Back pressure (Check for any resistance to flow.)

are expected to be important (physical constants, physical size, and shape). The second step is bringing together all applicable physical and chemical information, conservation laws, and rate expressions. The third step requires setting down of finite or differential volume elements, followed by writing the conservation laws. Then an appropriate mathematical solution method is sought with the proper choice of the boundary value of the dependent variables, which finally relates dependent variables to one or more independent variables.

Mathematical modeling seems to be a difficult subject to many students as well as to people working in industry. Dealing with differential equations in industry is sometimes simply avoided and the steady-state simulators are "tricked" into doing the work.[24] But, modeling can be made simple and interesting when doing lab experiments, particularly when a team effort is practiced. In industry the team might include specialists such as chemists and statisticians.[25] In the lab, students should perform the tasks with the help of the instructor.

I use a CSTR setup[7-9] to study startup and shutdown processes. It can be versatile. Students can study second-order or first-order reactions under isothermal or nonisothermal conditions; they can model the dynamics and steady-state behavior of the system; they might consider different approaches to startup and then model them. Other experiments are used to build simple models. For example, in a cross-flow heat exchanger, a model is built to describe the transient heat-transfer process between a heated element of copper and air.

The developed models could be solved analytically and/or numerically; using both approaches is preferable. I encourage the students to use available packages in order to emphasize principles rather than programming. For example, they write a main program and call available subroutines from IMSL, or use MATLAB. Sometimes I ask them to use spreadsheets, which are preferred by industrial people and can be used in a solid-handling experiment to simulate the breakage of a known sample of a solid material.[26] I ask the students to solve problems related to the subject of the experiment from their textbooks in an attempt to form a link between lectures and lab work. For example, I ask them to solve problems on transient balances related to crushing and grinding, and to build the model.[27]

Data acquisition software can serve as a convenient tool for quickly developing computer simulations of chemical engineering unit operations for use in classroom demonstrations.[28] These packages can be used to create virtual unit operations. Simulation is used to avoid some of the disadvantages associated with certain experiments.[29] This is useful, for example, when a great amount of time is needed to perform the experiments or when a complex phenomenon such as ion exchange or adsorption is considered.

DISCUSSION AND CONCLUSIONS

The objective of a lab course is no longer a matter of data collection and the preparation of a full and lengthy report. Many useful things can be extracted from a lab session when the new objectives are properly invested. The list might look lengthy, but this is not a problem as long as the objectives are achieved within the time limit without affecting the scheduled experiments. Safety procedures require one session and should be stressed in every other session. Maintenance and troubleshooting are performed using available experiments. Startup and shutdown are related to every experimental run. Mathematical modeling and simulation should be done in connection with the specified experiment.

Several important points can be drawn from this discussion:

- Industrial work can be effectively simulated in the unit operations lab without affecting the academic approach. This is achieved by stressing subjects such as startup, safety, troubleshooting, report writing, statistical analysis of errors, and modeling.
- The ability to solve problems and troubleshoot is developed by following systematic procedures of safety and of startup and shutdown, and by allowing the students to tackle practical problems and search for corrective solutions.
- Available experiments can be used to achieve the required goals, and when necessary new experiments can be introduced or existing ones can be modified. Students can participate in all of these activities.
- Students realize that simple subjects look difficult when they are not understood or practiced. This is obvious in practicing startup and shutdown procedures. They should be taught to think of applications in order to understand and memorize more easily.
- A worker who understands hazard and safety precautions improves work practice and becomes aware of protection and handling procedures.
- The team effort should not be ignored. It can be practiced by considering troubleshooting, modeling, and analysis.
- Subjects are interrelated. For example, mathematical modeling (in particular, the dynamic type) can help troubleshooting that is usually transient in nature.
- The typical unit operations lab is a fruitful area where many applied subjects can be practiced effectively.

The instructor should make sure that the students understand and grasp the above topics. This could be achieved by discussions during the lab sessions, by oral and written exams, and by asking the students to write short reports as part of a final exam. An experience like the one we have presented here will, hopefully, give the instructor courage to teach such a course without hesitation.

ACKNOWLEDGMENT

Thanks to the editors and to the reviewers of this paper for their helpful remarks.

REFERENCES

1. Johnston, B.S., T.A. Meadowcroft, A.J. Franz, and T.A. Hatton, "The MIT Practice School," *Chem. Eng. Ed.,* **28**(1), 38 (1994)
2. Riggs, J.B., and R.R. Rhinehart, "Introducing Graduate Students to the Industrial Perspective," *Chem. Eng. Ed.,* **31**(2), 188 (1997)
3. Chung, J., "Co-op Student Contribution to Chemical Process Development at DuPont Merck," *Chem. Eng. Ed.,* **31**(1), 68 (1997)
4. Douglas, J.M., "ChE Lab: A New Look," *Chem. Eng. Ed.,* **9**(1), 8 (1975)
5. England, R., and R. Field, "Using the Laboratory to Develop Engineering Awareness," *Chem. Eng. Ed.,* **23**(3), 144 (1989)
6. Burke, A., A. Phatak, B. Hudgins, and P. Reilly, "Introducing Statistical Concepts in the Undergraduate Laboratory," *Chem. Eng. Ed.,* **27**(2), 130 (1993)
7. Abu-Khalaf, A.M., "Mathematical Modeling of an Experimental Reaction System," *Chem. Eng. Ed.,* **28**(1), 48 (1994)
8. Abu-Khalaf, A.M., "Dynamic and Steady-State Behavior of a CSTR," *Chem. Eng. Ed.,* **30**(2), 132 (1996)
9. Abu-Khalaf, A.M., "Start-Up of a Non-Isothermal CSTR: Mathematical Modeling," *Chem. Eng. Ed.,* **31**(4), 250 (1997)
10. Kofke, D., M.R. Grosso, S. Gollapudi, and C. Lund, "CESL in the Chemical Engineering Simulation Laboratory," *Chem. Eng. Ed.,* **30**(2), 114 (1996)
11. Myers, K.J., "Troubleshooting in the Unit Operations Laboratory," *Chem. Eng. Ed.,* **28**(2), 120 (1994)
12. Marrero, T.R., and W.J. Burkett, "Introducing Industrial Practice in the Unit Operations Lab," *Chem. Eng. Ed.,* **29**(2), 128 (1995)
13. Langrish, T.A., and W. Davies, "Putting Commercial Relevance into the Unit Operations Laboratory," *Chem. Eng. Ed.,* **28**(1), 40 (1995)
14. Lewis, R., *Rapid Guide to Hazardous Chemicals in the Workplace,* 3rd ed., Van Nostrand Reinhold (1994)
15. Stricoff, R., and D. Walters, *Laboratory Health and Safety Handbook,* Wiley (1990)
16. Weiss, G. (editor), *Hazardous Chemicals Data Book,* Noyes Data Corporation (1980)
17. Peters, M.S., and K.D. Timmerhaus, *Plant Design and Economics for Chemical Engineers,* 4th ed., McGraw Hill (1990)
18. Ramanathan, P., S. Kannan, and J.F. Davis, "Use Knowledge Based Systems Programming Toolkits to Improve Troubleshooting," *Chem. Eng. Prog.,* **89**(6), 75 (1993)
19. Gans, M., D. Kohan, and B. Palmer, "Systemize Troubleshooting Techniques," *Chem. Eng. Prog.,* **87**(4), 25 (1991)
20. Kmetz, C.J., "An In-Plant Container-Labeling Program," *Chem. Eng. Prog.,* **86**(3), 33 (1990)
21. Caulcutt, R., and R. Boddy, *Statistics for Analytical Chemists,* Chapman and Hall (1983)
22. Rice, R.G., and D.D. Do, *Applied Mathematics and Modeling for Chemical Engineers,* John Wiley (1995)
23. Barton G.W., "Model Development and Validation: An Iterative Approach," *Chem. Eng. Ed.,* **26**(2), 72 (1992)
24. Horwitz, B.A., "Avoid Nausea when Solving Dynamic Problems," *Chem. Eng. Prog.,* **92**(3), 44 (1996)
25. Semple, T.C., and J.H. Worstell, "Rethink Your Model for Improving Processes," *Chem. Eng. Prog.,* **92**(3), 56 (1996)
26. McCabe, W.L., J.C. Smith, and P. Harriott, *Unit Operations of Chemical Engineering,* 4th ed., McGraw-Hill (1985)
27. Felder, R.M., and R.W. Rousseau, *Elementary Principles of Chemical Processes,* 2nd ed., John Wiley (1986)
28. Davis, R.A. "Create Virtual Unit Operations with Your Data Acquisition Software," *Chem. Eng. Ed.,* **29**(4), 270 (1995)
29. Anklam, M.R., R.K. Prudhomme, and B.A. Finlayson, "Ion Exchange Chromatography Laboratory: Experimentation and Numerical Modeling," *Chem. Eng. Ed.,* **31**(1), 26 (1997) ❐

ChE book review

Mathematical Methods in Chemical Engineering

by A. Varma and M. Morbidelli

Oxford University Press, New York, NY; 690 pgs; $80 (1997)

Reviewed by
E. James Davis
University of Washington

This text follows the Minnesota tradition of applied mathematics in chemical engineering established by Professors Amundson and Aris in that the treatment of the numerous topics is rigorous and vigorous. There is an attempt to complement the mathematical fundamentals with examples arising in chemical engineering, but the balance between theory and application is not uniform throughout the book. The authors naturally lean toward examples from their own research and experience.

The nine chapters cover a wide range of subject matter starting with matrix theory and proceeding to a particularly long chapter dealing with first-order linear ordinary differential equations and stability theory. With respect to Chapter 1, the authors acknowledge their debt to Professor Amundson; that chapter summarizes Amundson's book *Mathematical Methods in Chemical Engineering: Matrices and Their Application.* The first two chapters account for almost one-third of the 690-page book, but Chapter 2 addresses subjects such as Liapunov's direct method and the Hopf bifurcation theorem not covered in typical texts on advanced engineering mathematics. The 135 pages of Chapter 2 include interesting applications such as the analysis of the Belousov-Zhabotinskii oscillatory reaction.

Chapters 3 and 4 are more conventional in their coverage of linear ordinary differential equations and special functions, respectively. A clear presentation of the Green's function for solving nonhomogeneous equations is a plus. The applications included in these chapters are rather lean, and the presentation of orthogonal polynomials such as the Chebyshev and Laguerre polynomials and other special functions is left to problems at the end of Chapter 4 without relevant applications.

The classification of partial differential equations in Chapter 5

Continued on page 219