AN INTRODUCTORY ChE LABORATORY INCORPORATING EC 2000 CRITERIA

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In developing a laboratory course sequence for chemical engineering undergraduates, it is necessary to define overall course objectives as well as objectives for individual experiments. This would correspond to defining the overall course objectives and the objectives of each lecture for a traditional lecture-based course. In the past four years in this journal alone, over twenty articles[1-23] have appeared describing new and innovative individual experiments. But objectives of the course as a whole and how they are to be defined have received less attention.[24-29]

The new ABET EC 2000[30] explicitly requires that engineering departments develop in their students "the ability to design and conduct experiments as well as analyze and interpret data." Additionally, these same students must be able to "function on multidisciplinary teams," and "communicate effectively." It is incumbent on the department to document that the students have these abilities. A logical place to explicitly incorporate the development of these skills into an undergraduate curriculum is within the laboratory sequence. Here, we can not only develop the statistical experimentation and communication skills, but we can also document the progress of students in these critical areas. In addition, we can use a continuous feedback loop to revise and improve the experiments as we receive input from our alumni, advisory boards, and recruiters concerning the effectiveness and suitability of the courses for the employability of our students.

With consensus from our department's Industrial Advisory Board, we undertook a comprehensive review of our entire laboratory sequence almost two years ago. This review identified that our students needed to improve their understanding of the abstract concepts of experimental design and data analysis and be given more opportunities to practice these skills in the laboratory. Therefore, we developed a four-course sequence: one lecture course (which was new to the curriculum) and three laboratory courses (which were in the curriculum but were extensively modified) of increasing complexity, that integrated experimentation with statistical concepts and engineering science and design. These courses are summarized below:

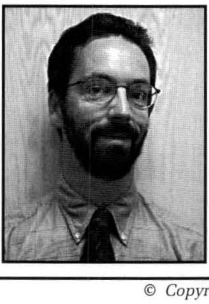
Chemical Engineering Data Analysis *A 3-credit, second-semester Sophomore course covering the theoretical aspects of experimental design and data analysis.*

Process Instrumentation Laboratory *A 2-credit, first-semester Junior laboratory introducing the students to measurement techniques, statistical analysis of engineering data, report writing, and oral presentations in small teams.*

Transport Operations Laboratory *A 2-credit, second-semester Junior laboratory in thermodynamics and heat, mass, and momentum transport where teams of students measure transport coefficients using statistically designed experiments and report their results both in writing and orally.*

Unit Operations Laboratory *A 2-credit, first-semester Senior laboratory where small teams of students characterize the performance of several unit operations and use their results in solving design problems. Written and oral reports are required.*

In this paper, the Process Instrumentation Laboratory, which was completely redesigned with new experiments, data analysis, and reporting requirements, is described in detail. By carefully selecting and designing the experiments and the organization of the course, it was possible to have the stu-



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dents meet several course objectives, including

- Conducting engineering experiments using varied experimental designs
- Analyzing experimental data using several statistical techniques
- Using different measurement methods
- Exposing the students to a variety of engineering phenomena
- Developing the student's written and oral presentation skills

COURSE ORGANIZATION

The course met for 3 hours twice each week for 16 weeks. The first four weeks were spent in 1-hour lectures reviewing statistical design of experiments and data analysis. Also included in this introductory section were lectures on laboratory safety, right-to-know training, laboratory notebook keeping, report preparation, and oral presentations.

The final twelve weeks covered the actual experimentation, analysis, reporting, and presentation phase of the course. This phase was divided into three blocks of four weeks. For each block, the students were divided into teams of 3-4 students, and each team conducted three experiments. At the conclusion of the first two experiments, the students submitted individual memorandum reports (a 2-3 page report suitable for submission to a technical manager, plus 5-10 pages of attachments documenting the procedure and data analysis and answering questions specific to the experiment). One member of each team also gave a five-minute oral presentation. At the conclusion of the third experiment in each block, the team submitted a formal report and the students who had not done an oral report did individual poster presentations of their results. New teams were formed at the beginning of each block with the same procedure for experimentation and reporting. Thus, each student submitted six memorandum and three formal reports and conducted two oral and one poster presentation during the semester.

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Several additional aspects of the course organization are worth mentioning:

- Two days prior to each experiment (typically after the oral reports), the students were given an hour in the laboratory to review the experimental set-up.
- Each experiment had to be conducted in the allotted time (3 hours); any group not finished within that time received a zero grade for the experiment.
- Reports were graded for both technical content and for composition, grammar, readability, and conciseness. The faculty instructor was responsible for the technical content while a professional technical writer evaluated writing-related issues.
- Instant feedback was provided for the oral reports by both the class and the instructor. The presenter's group members were required to identify at least one thing about the presentation they thought was excellent and one that needed improvement. The instructor and the other students provided additional comments to the presenter as soon as the presentation was finished. This allowed all students to hear positive comments as well as areas for improvement on 9-12 presentations in a single afternoon.
- During the poster sessions, faculty and visiting industrial scientists and engineers were invited to review the posters and quiz the presenters about their work. Feedback was given immediately to the students concerning their presentation as well as their poster design.

STUDENT BACKGROUND

The students enrolled in this course typically had completed

- Engineering Data Analysis and Experimental Design
- Mass and Energy Balances
- Transport Operations I: Fluid Flow
- Chemical Engineering Thermodynamics I: Engineering Thermodynamics
- Differential Equations
- Freshman Chemistry
- Freshman Composition

In addition, the students had completed, as part of Freshman chemistry courses, the equivalent of a 2-credit general chemistry laboratory course, so they had not yet covered the engineering science background for many of the experiments. Thus, in the descriptions of the experiments and the data analysis, it was necessary to either provide the missing information (*i.e.,* the theoretical description) or to provide the appropriate references.

LABORATORY EXPERIMENTS

The experiments for the laboratory were selected and designed with the following objectives:

- Each experiment had to be completed in the allotted time (3 hours)
- Each experiment had to produce a sufficient number of data points (depending on the design, this required between 8 and 30 points per experiment) to allow statistical analysis to justify conclusions drawn by the students.
- Each experimental design (which included fractional factorials, Graeco-Latin squares, blocking, nested, and mixtures designs) should be used at least twice during the semester.
- The experimental conditions had to be easily changed so no two groups performed exactly the same experiment.
- Different fields of engineering science were to be explored.

- Some of the experiments had to explore topics that had not been covered extensively by their prior classroom experience as preparation for lifelong learning.
- For some of the experiments, the students were required to rely only on the statistical analysis of the data to develop their conclusions because of a lack of an engineering science description of the phenomena. But when a suitable engineering science description was available, the students were required to statistically validate the mathematical expressions using their data.
- When applicable, students were required to use ASTM standards.

In addition, there were the following constraints:

- Minimal use of hazardous or dangerous chemicals.
- Minimal cost of the individual experiments and, where possible, use of existing facilities and instrumentation.

A short description of each of the nine experiments can be found in Table 1. The balance of design and topics provided good coverage of the topics and designs within the constraints of the experiments (see Table 2).

REACTIONS AND COMMENTS FROM STUDENTS

The most common reaction from students was that the most difficult portion of the course was analysis of their data. At the beginning of the semester, most of the students viewed data analysis as a cookbook task that could be done with little thought. Throughout the semester, the students repeatedly asked what the answer should be and how they should get it. Only toward the end of the semester did most students begin to realize that data analysis was a process of discovery and that their data would have to lead them to the answer. Of course, the consensus was that this was much more difficult and time consuming than they had planned and that waiting until the last moment to conduct the analysis ensured that they would not finish in time.

TABLE 1
Descriptions of Experiments

Specific gravity of aqueous solutions • The specific gravity of mixtures of water, salt, and sugar were measured using a hydrometer. Since the maximum solubility of both solids was about 5% by weight, the simplex-centroid mixtures design was constrained to $0.95 \le x_{water} \le 1.00$, where x_{water} is the mass fraction of water in the solution. The data anlaysis required the students to develop a statistically significant polynomial expression for the specific gravity and plot contours of constant specific gravity on triangular graph paper. By changing the solutes, the experimental factor space can be altered, which changes the data analysis.

Heat transfer from fins • The effects of four factors on the convective heat transfer coefficient from fins were determined by measuring the end-face temperature on 16 different fins of various geometry and materials, as dictated by a 4x4 Graeco-Latin square design. A nonlinear least-squares analysis allowed the students to determine the best-fit convective heat transfer coefficients for the top and side of the fins. The end temperatures calculated with these coefficients were compared to those measured to determine if any of the factors affected the difference between the measured and calculated temperatures, *i.e,* the students were required to statistically validate the underlying engineering science. To alter this experiment, we have changed the bath temperature and could use a fan to change the convective heat transfer coefficient.

Efflux time from a baffled tank • Various baffle configurations were added to a gravity-drained tank to study their effect on drain time. Length and diameter of the exit pipe were also varied as dictated by a 3x3 Graeco-Latin square design. A simple ANOVA was used to determine the factors that significantly affected the efflux time. By changing the variable assignment in the design, a different experiment results.

Absorption by activated carbon • Blue food coloring was absorbed from aqueous solutions of various strengths by a commercial activated carbon. Factors examined in the 2^{5-1} fraction factorial design included amount of solution, concentration of food coloring in solution, the contact time, the ratio of carbon to solution, and the mixing speed. ANOVA was used to determine the significant factors. Many factors can be changed in this experiment, *e.g.*, type of carbon or colorant, temperature of the solution, etc., to create different experiments.

Acid neutralization • A three-component, constrained simplex-centroid mixtures design was used to select the compositions for ten solutions of vinegar and two commercial antacids. Solution pH was measured using a digital pH meter. ANOVA and linear least squares to determine a statistically significant polynomial fit of the data were used, and then contours of constant pH were plotted on triangular graph paper. By changing the brand of antacids, this experiment can be changed.

Frictional losses in pipes • The Fanning friction factor was calculated for laminar and turbulent flow in PVC and copper pipes of various diameters based on pressure drop measured using an inclined manometer. Due to time considerations, a balanced incomplete blocking design was used to select the factor space combinations to be tested. Linear regression allowed the students to determine if the Hagen-Poiseuille law was valid.

Rotameter calibration • A blocking design, using the operator as the blocking factor and rotameter reading as the independent factor, was used to determine the experimental space to create a calibration curve for a salt-water solution in a rotameter. ANOVA was used to identify the significant factors and linear regression was used to develop a calibration curve and a 95% confidence interval for the predicted values. This experiment was changed by altering the density of the fluid used in the rotameter.

Efficiency of a parallel-plate exchanger • A 2^{4-1} fractional factorial was used to evaluate the efficiency of a simple parallel-plate heat exchanger (custom designed and manufactured for this course) using the inlet temperatures and flow rates as the independent factors. The students had to calculate the overall resistance to energy transfer for both the cold and hot sides, determine if they were affected by any of the factors, and decide whether or not the two coefficients were statistically different. By changing the number of plates in the exchanger and the thickness of the plates, the experiment could be altered.

Viscosity of aqueous solutions • The effect of a proprietary food thickener on the apparent viscosity of aqueous solutions as a function of shear rate and thickener concentration was measured using a rotating spindle viscometer. A two-level nested design was used to determine the factor space combinations to be tested, and ANOVA was used to analyze the data. Changing the concentration and type of thickener changed the experiment from group to group.

The second most common reaction was that the experiments were relatively simple to conduct and that they could easily be accomplished in the allotted time. Having both the in-lab preview and oral presentations by other students greatly facilitated this efficiency. But there were some problems with completing the experimental design (*i.e.*, completely specifying all the trials, including replicates, that would be done) prior to beginning the experiments. In several cases this meant that the students failed to conduct a sufficient number of experiments to conduct a satisfactory analysis of their data.

The students also appreciated the fact that the experiments were always ready to run. Thanks to the help of an outstanding teaching assistant and staff engineer, the experiments were turned on and warmed up before the students arrived in the lab; the students did not have to wait for water baths to heat or for instrumentation to warm up before they were ready to begin. The teaching assistant and staff engineer were available to answer questions during the lab and to help solve equipment problems that arose (which happened about once every other week). The students truly appreciated the willingness to help and approachability of both individuals.

Little comment was made by students regarding the use of a technical editor to assist in grading the written reports. The editor commented on the marked improvement of the writing as the semester progressed, however. Having to write six memorandum reports and three formal reports gave the students ample opportunity to improve—the average writing grade increased by nearly 5 points (out of a possible 20) over the course of the semester. The students also made little comment about the oral and poster presentations. Again, grades significantly improved during the semester—the average grade on the initial oral reports was ten points (on a 50-point scale) lower than the average grade on the final oral reports.

TABLE 2
Summary of Experiments, Experimental Designs, and Engineering Topics Covered in the First Laboratory Course

Experimental Design	Engineering Topic: *Heat Transfer*	*Mass Transfer*	*Momentum Transfer*	*Chemical Reaction*	*Physical Properties*
Graeco-Latin Square	Heat Transfer From Fins		Efflux Time From a Baffled Tank		
Fractional Factorial	Parallel Plate Heat Exchanger	Adsorption by Activated Carbon			
Constrained Mixtures				pH of Aqueous Solutions	Specific Gravity
Blocking			Frictional Losses in Pipes		
Nested					Viscosity of Aqueous Solutions

The most frustrating aspect of the course for many students was the different backgrounds in statistics of the students. In addition to the statistics course offered in the department, other courses were accepted as satisfying the course prerequisite. Most of the other courses did not have the same emphasis on data analysis as the departmental course and instead focused on probability and combinatorial theory. Students who had taken the departmental course often found themselves teaching the other students how to conduct the data analysis and interpret their data. Although this was probably a great learning experience for the students, they resented the time it required for what to them was no return.

LESSONS LEARNED AND RECOMMENDATIONS

In general, the laboratory worked extremely well considering it was the first time the course was offered in this manner. From the instructional side, the following lessons were learned (or, in some instances, relearned):

- To compliment the experiments, the initial phase of the course needed to focus more on how to develop a design so that the proper factor space combinations and replicates would be tested. In the lab manual, the experimental design was specified, but the details were left for the student to determine, which they did not always complete prior to the experiments.
- To improve the written and oral communication skills, more time needed to be devoted to reviewing the structure and organization of technical communication during the initial phase of the course. In conjunction with this, reviewing document design aspects would also be warranted.
- To assist students who had taken a non-departmental experimental statistics course, grouping them together and reviewing the design and analysis techniques weekly assisted in reducing both the intra- and inter-group variability.
- To provide sufficient time for data analysis, the laboratories should be conducted on Thursdays, with the reports due on the following Tuesday. Initially, the labs were done on Tuesdays, with reports due Thursday—leaving insufficient time to conduct the analysis.
- To enhance the quality of the formal reports, students need to cover a broader scope of material than the experiments for the memorandum reports. For example, the mixtures experiments could involve a fourth component or the evaluation of the heat exchanger could include the effects of the

number of plates.

- To ensure that the students can complete the experiments in time, a hands-on teaching assistant is absolutely necessary.
- To reduce student frustration at having to work for an extended period of time with an under-achieving lab partner, groups need to be reformed randomly and frequently. Having each student work in three groups over the semester seemed to avoid intra-group problems.

In addition to addressing the lessons above, the following recommendations are also suggested:

- To cover more chemical engineering science (in particular, chemical reactions and kinetics), a greater breadth of experiments is needed.
- To further improve the writing skills, it would have helped if report writing would have included revising some of the reports until all structure, organization, and grammatical problems were corrected.

ACKNOWLEDGMENTS

Without the energy and expertise of Jim Anthony and James Autry, this laboratory would not have been as successful as it was. Jim Anthony, the departmental engineer, was responsible for building and assembling the experiments. James Autry, my teaching assistant, was responsible for making sure that the experiments were operating every week and answering questions about experimental procedures and laboratory safety. I am also thankful for the comments made by one of the reviewers about the value of exploring topics not yet covered in courses.

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