ChE laboratory

THE LARGE LABORATORY COURSE

Organize It to Parallel Industrial Process Development

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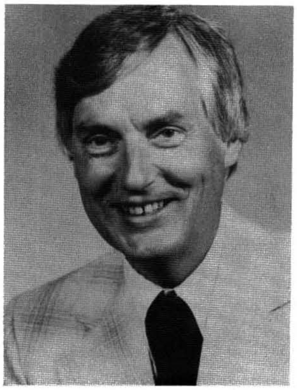
OUR FINAL CHEMICAL engineering laboratory course essentially doubled because of both a record enrollment of seniors and a simultaneous curriculum change that offered this course only once a year. Faced with 180 seniors in a single chemical engineering laboratory course, we realized that something had to be done to relieve the overwhelming strain that this large enrollment exerted on our manpower and facilities. Smaller classes of a previous decade permitted informal instruction, and the learning experience was tutorial. A large enrollment requires an alternative approach which is necessarily more structured. Therefore, we needed a structure that would allow efficient and timely communication among students and faculty. Our course size and organization contrast with that of Rochefort *et al.* [1], who developed a process laboratory for a class size under forty.



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Since the laboratory would run all day, all week, we thought, "Why not organize the course to operate as an industrial process development department?" Just as industry subdivides process development along product lines, we grouped the laboratory projects into divisions of commonality managed by a faculty member and supervised by a teaching assistant. Although this reorganization did not reduce the faculty's workload, it did make the teaching more efficient.

Pedagogically a laboratory course should culminate with students preparing excellent technical reports. However, such a goal is often thwarted because most student effort occurs at the end—the all-nighter! To prevent this last-minute rush, we patterned our guidance after that which we experienced in industry where management is continually updated through scheduled meetings and written memos.

The key meeting that we stress is the preparation conference. At this meeting, several groups in a common project area orally present their proposed work. The faculty member reviews the proposals and offers suggestions to improve or redirect the work. Since many of the ideas discussed at the conference are of mutual benefit to all the participants, we find these multiple-group sessions are an effective way to reach a large but manageable number of students. Other written and oral follow-ups, such as sample calculations and an oral progress report, are scheduled when criticism is most useful—not saved for grading at the end.

COURSE ORGANIZATION

In the course we reorganized, students must complete conceptual process designs based on laboratory data. These designs range from a single piece of equip-

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ment to an entire plant. The course is divided into three project cycles, each lasting four and a half weeks or nine three-hour laboratory periods. During a cycle, a design project from one of the three areas (Table 1) is completed.

In the past a faculty member typically handled one or two laboratory divisions with all twelve of the projects in Table 1. This situation led to little improvement in the projects. Twelve divisions were needed to accommodate the 180 students. We decided to follow an industrial-type line organization (Figure 1) and to restructure the course along project areas. Thus, each area was headed by a faculty member (manager) and a graduate teaching assistant (supervisor). Assistants supervised laboratory divisions where all projects were conducted, but their grading assignment was in a limited project area. By concentrating each individual's effort to fewer projects, we could

- **improve our guidance of the students**
- **increase the possibility of improving our laboratory projects**

To further simulate an industrial assignment in process engineering, we give the group members specific job titles; these job responsibilities rotate through the three cycles

TABLE 1
Laboratory Design Project List

PROJECT	TITLE
Reaction/Reactors	
R1	**Continuous Stirred-Tank Reactor**
R2	**Continuous Esterification in a Tubular Reactor**
R3	**Gas-Phase Dehydrogenation**
R4	**Catalytic Cracking**
Staged Separations	
S1	**Extraction in an Agitated Staged Column**
S2	**Continuous Fractionation in a Bubble-Cap Column**
S3	**Continuous Fractionation in a Sieve-Tray Column**
S4	**Batch Fractionation in a Packed Column**
Transfer Operations	
T1	**Gas Absorption in a Packed Bed**
T2	**Water Cooling in a Servel Tower**
T3	**Air-Water Contact in a Fluidized Bed**
T4	**Continuous Drying**

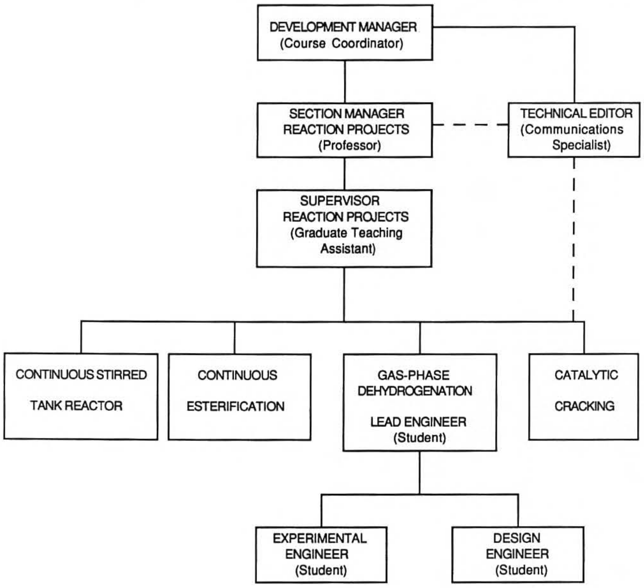

FIGURE 1. Organization chart for Reaction/Reactor projects.

- **The lead engineer is responsible for the execution of the project**
- **The experimental engineer is responsible for experimental aspect of the project**
- **The design engineer is responsible for design aspect of the project**

ACTIVITIES IN A PROJECT CYCLE

In this section, we will outline the activities of the students as they develop their process designs. Table 2 is typical of a project outline the lead engineer should develop when planning the project's key events. With such an outline, the group has an idea of what must be accomplished and when. We will refer to the six written and oral assignments (A1-A6) that we require as we develop what activities occur during a project cycle.

Design Problem

An open-ended design is presented to the students during the first period of the project cycle. We have selected the reaction project R3, Gas-Phase Dehydrogenation, to serve as an example throughout this article. Part of its statement is

> **Acetone can be produced by gas-phase dehydrogena-**

tion of isopropanol catalyzed by 0.5% platinum on silica-gel support at temperatures near 200°C. The reaction is

$$(CH_3)_2CHOH \rightarrow (CH_3)_2C{=}O + H_2 \qquad (1)$$

When the temperature is high or the residence time is long, a dehydration reaction also takes place and produces propylene as follows:

$$(CH_3)_2CHOH \rightarrow CH_2{=}CHCH_3 + H_2O \qquad (2)$$

The product desired is 99% acetone; the Sales Division will estimate the market later. If there were no complications from the competing reaction, this could conceivably be obtained in a single pass through the tubular reactor. However, by-product formation may not permit this, and it may be necessary to use a lower conversion followed by a purification (probably by distillation) with the isopropanol recycled. Obtain the dependence of conversion and fractional yields on processing factors; the design could be optimized using this information.

Preparation Conference

The idea of a planning discussion is not a new one; however, we feel the emphasis that we place on this conference is. In the past an informal conference would take place before the students knew what the equipment could do. The professor would question the students to determine if they understood how to operate the equipment and what engineering principles the equipment demonstrated. Often times the students' answers were vague; the meeting would degenerate and end with the professor telling the students, "Go back and research the problem further." The end result was an unproductive exchange of ideas.

In a well-run industrial organization, sound planning is a must. Planning requires sufficient preparation, and ideas that are forwarded must be well-conceived. At the planning meetings, the engineers are generally those who present the ideas while management sits in review. These formal meetings are much more productive than an informal gathering where the manager, rather than the engineer, forces the issue.

For these reasons, we moved the conference from the first to the third period and instituted a formal planning meeting, which we felt would be a springboard for the project. Much spadework must be done before the preparation conference, for example, to identify the operating range of the equipment, calibrate analytical instruments and make a few scouting runs to iron out experimental difficulties. The formal presentation forces the students to gather their thoughts into a more coherent package. Students who are well-prepared will spend less time in subsequent laboratory periods deciding what they need to do next. The required text of Holman [2] and a guidance document, which details each engineer's role, help them to prepare for their conference.

These conferences last about thirty minutes and include a five-minute presentation from each group member. The lead engineer chairs the preparation conference. Each engineer also develops a one-page outline with attachments that can be reviewed during the talk. Here the objectives, approach, and division of effort are established. We challenge the students to create an industrial situation, or "scenario," as a basis for their proposed work. This scenario establishes an engineering need and thus better defines the project.

For the dehydrogenation example, project objectives are first defined from the chemical reactions. Acetone production involves parallel reactions and requires that the kinetics of each be determined as a function of temperature. From published kinetics on other catalysts, the leader selects a temperature that favors acetone production. A conversion is chosen and the reactor and distillation columns are sized. Vapor-liquid equilibrium data can be obtained from literature to design the separation units. By obtaining rough estimates for the kinetics, the group then has a feel for the order of magnitude of the rate constant. This is an extremely important exercise to go through because few experimenters enter the laboratory without some idea of what they expect to find.

The experimental engineer must calibrate a gas chromatograph with known compositions of simulated product mixtures. Reaction scouting experiments are essential because they enable the engineers to sharpen their experimental technique and provide information needed for the experimental design. The time required to conduct a single run is needed to define the maximum number of runs that can be performed during the project. The range of important factors can also be obtained, preferably before the preparation conference. With the above information, the experimental engineer can design an experiment from which the kinetics can be modeled. Typically a first-order kinetic model with an Arrhenius temperature dependence is tested for adequacy. Space-time and temperature are the factors, and conversion is the response. If the first-order model is not adequate, then other kinetic models, such as higher orders, must be evaluated. The experimental engineer's design must have enough levels of each factor and enough replicates so a suitable kinetic model can be built.

The design engineer presents a flow sheet of the conceptual process based on limited data or estimates. Flows and compositions are projected, and the need for the experimental data is established. Thus the design engineer learns how to design the various equipment items in parallel with experimentation. For example, the distillation columns to separate the reaction products can be designed for the number of stages required to attain a desired product and recycle purities. Energy balances for reactors, reboilers, and condensers are made. When production rates are obtained, final flowsheet values are ratios of the preliminary ones.

We have found that jointly conducting all four conferences in a common area is beneficial. From the presentations and discussions the students learn much about the related projects of other groups. Since they will never conduct these other projects, they broaden their technical knowledge as well as learn how to present and discuss a project. Furthermore, their interest is focused on similar concepts that can be valuable for their own project.

The lead engineer prepares minutes of the conference and discusses the disposition of unresolved items. Such items often include redirection of the project, correction of erroneous concepts and equations, and collection of needed literature data. As in an efficient industrial planning meeting, much is accomplished in a limited time through a well-prepared conference and the written minutes.

Sample Calculations

With transfers, promotions and project changes so frequent in industry, supervisors are often unaware of the project details. This lack of detailed knowledge is simulated in education by changes in teaching assignments. Therefore the engineer must regularly report details of the project to inform the supervisor as well as clarify his own understanding.

Two additional checkpoints of this type that appear in Table 2 are the sample experimental and design calculations; they are given to the supervisor at periods 5 and 6. These calculations explain the details to the group supervisor and require him to update his understanding of the work. Furthermore, the supervisor has an opportunity to question these details and explanations before extensive calculations are completed. This review of the sample calculations helps the teaching assistants play a more active role in the laboratory.

Oral Progress and Final Design Reports

The students receive a document that describes the content of each section of the final design report. In many other project courses final results are presented in an oral report. At this time the instructor should call attention to errors, lest the student audience believe the results presented are correct. The instructor must be negative in pointing out errors, discrepancies and inadequacies; unfortunately these errors are never corrected because the final report is already completed.

With large enrollments, faculty-student contact must be efficient and effective. By presenting oral progress reports one week before the final written report is due, improvements can be suggested and carried out by the group. Typical action includes correcting misconceptions, gathering needed additional data, better modeling of the process steps, and improving the design. As an example, one group presented a dehydrogenation reactor designed for 11% conversion because this was the highest they obtained in the laboratory. Their data were from a laboratory reactor and should have been used only to model the kinetics. The selected conversion was ridiculously low for a plant design. Through our questions and comments we were able to lead them to a reasonable design report.

Our approach parallels industrial practice where revisions resulting from meetings are commonplace, and final reports are almost always issued *after* the information is disseminated at a meeting of the concerned parties. Industry cannot afford the time delay of waiting for final report preparation before making business decisions.

TABLE 2
Outline of a Project Flow Sheet for Dehydrogenation

PERIOD	ASSIGNMENT	ENGINEERING ACTIVITIES
1,2		• establish analytical methods • conduct scouting experiments • design experiments • conceive process
3	A1 Preparation Conference	• establish kinetics • collect non-experimental data needed for design
4	A2 Minutes of Conference	
5	A3 Sample Experimental Calculations	• replicate data
		• test models
6	A4 Sample Design Calculations	• specify process design
7,8	A5 Oral Progress Report	• analyze experimental error • assemble final tables and figures • complete design
9	A6 Final Report	• integrate and proofread final report

MANAGER-ENGINEER CONTACT

Scheduled meetings already discussed are the preparation conference and oral progress report. Two additional places for contact are the laboratory and consultation sessions. The manager should personally interact with the engineers in the laboratory, but cannot continuously supervise them since the laboratory operates all day, every day. Still, morale of the engineers and supervisors is boosted by the manager's interest in the project. In the laboratory, the manager assesses the project status and progress, and sometimes rolls up his sleeves to fix or operate the equipment. He questions the engineers and offers advice to help the group stay "on-track." The group that is on top of their project has an opportunity to review progress by telling the manager about their accomplishments and future work. A group that is not well-prepared can lapse into details of technical problems that they should solve themselves. Of course, they are so informed.

For example, students raise questions about how to determine feed composition in the dehydrogenation. They know how to obtain the relative amounts of isopropanol, acetone, and water in the product stream from the GC calibration. However, the amount of isopropanol in the feed is needed to determine conversion and yields. In the laboratory reactor isopropanol is fed to the dehydrogenation reactor by saturating a noncondensable carrier gas in a bubbler. A typical student question is, "How do we measure the feed composition?" Our response is, "You can't! What can you do?" They should assume that the carrier gas is saturated with isopropanol because nitrogen/isopropanol mixtures are not available for calibration.

The consultation sessions regularly bring the groups together on a project-basis. At the beginning of a project cycle, information already discussed under "Preparation Conference" is amplified and applied to the specific projects. After the conference, these sessions are used to

- **exchange technical information that helps solve experimental difficulties**
- **consider alternative designs that promote the divergent thinking portion of the problem solving process**
- **elicit further information about the project that helps the convergence to a solution.**

EXAMPLES OF TECHNICAL IMPROVEMENTS DURING THE COURSE

Examples of improvements from both the experimental and the design parts of the project are presented here. These improvements are products of how we reorganized this course. On the dehydrogenation project we observed erratic behavior of the catalyst that was difficult to trace to the temperature or chemical exposure history. Switching from nitrogen carrier gas to nitrogen containing 10% hydrogen helped to maintain the catalyst's activity, and thus more consistent conversions were obtained. Gas sampling of feed and product streams at high flow rates was also a problem. A low-volume, three-way valve was installed which cured the sampling problem. These improvements were conceived and executed because the concerned faculty member specialized in the project, and he carefully observed and listened to many student groups.

Student engineers often decide the design needs a "safety factor" and therefore increase the equipment size by, say, 50%. We point out that such overdesign can kill a project through excessive capital costs and that a more objective method is needed. Our method uses the experimental uncertainty in the key design parameter. The design engineer must consider this uncertainty to assure that the plant will operate at the designed capacity. For design purposes a conservative limit on the parameter is selected instead of its best estimate. For example, if the kinetic constant of a first-order reaction, k, is the key parameter in designing the reactor, the lower limit of the confidence interval on k can be used to size this reactor. A somewhat larger reactor will be designed to provide a safety factor for the uncertainty in k. By intercepting a loose and inappropriate use of a "safety factor" at the oral progress report we offer the engineers an opportunity to strengthen their final report.

EVALUATION OF STUDENTS

Grades are based on the elements described in this section; we attempt to parallel the responsibility and rewards that a manager assigns to his engineers on a process development project. Each engineer is graded on the preparation conference, laboratory performance, the written report and the oral report. The responsibilities for the preparation conference have been described; performance involves efficiently executing these tasks during the laboratory.

The written report is evaluated for both technical content and communication skill. The technical grade is assessed from sections of the engineer's primary responsibility. Also, each member shares responsibility with others in the group. In industry, responsibility and rewards are closely related, and this concept underlies our decision to have the lead engineer graded on all sections of the report. The experimental and design engineers aid in coordinating the report so each receive an added *one-fourth* of the technical grade of the leader's and other engineer's sections. A separate communications instructor assigns a writing grade for content, organization, development, style, and mechanics of the sections written by each engineer.

The oral report is graded for skill shown in communicating the scientific and engineering concepts with an intelligent but uninformed technical audience. The grade is based on the choice of subject matter, organization, balance, and the content of displays. Two separate evaluations are made; one is by the technical supervisor and the other by the communications instructor for the presentation in each respect.

COURSE EVALUATION AND CONCLUSIONS

Students evaluated aspects of the course on a five-choice scale from "strongly agree" to "strongly disagree"; written comments were also solicited. Since the students have limited industrial experience we did not attempt to have them evaluate how well our approach parallels industry. That could be the subject of a questionnaire after they have had working experience.

They overwhelmingly concluded that the projects reinforced and extended the concepts of prerequisite courses in general. Specifically, the junior chemical engineering laboratory course was cited in this category. But also, they wanted better coordination between the prerequisite courses and the laboratory projects. The reaction projects, which required full plant designs, were rated as challenging and practical; however, the students thought more time should be allotted to complete the difficult projects, or else the scope of such projects should be reduced. The open-ended problem statment was credited with improving their engineering problem-solving skills. Further, the group seminar approach to the reaction project preparation conference was judged to increase their knowledge of the reaction area.

Most students expressed a need for meeting with the manager between the preparation conference and the oral progress report. They also strongly favored a class period to transmit initial information about each project. Further, they thought manager visits to the laboratory were essential, but supervisors were not considered helpful in the execution of the projects. They thought a Laboratory Center with computer terminals, where calculations and report drafts could be done, would have increased their efficiency. Finally, equipment was judged to operate poorly. (So what's new?)

In an unusual comment, one student suggested that the communications portion be considered a separate course so that it could be listed on their resumes; it might help in job search!

ACKNOWLEDGEMENTS

Laboratory projects have been developed over many years, and we recognize the contributions of the faculty members involved. Many of these projects were devised by John M. Woods. In addition to the authors, in the two years preceding this organization, A. H. Emery, N. H. L. Wang, and D. P. Kessler contributed while they served as managers and Frank S. Oreovicz sharpened the students' communication skills. E. I. Franses taught the prerequisite laboratory course and contributed ideas through that course. We also acknowledge R. P. Andres, school head, for supporting this concept of teaching the senior chemical laboratory course for 180 students.

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ALARM SYSTEM DESIGN

Continued from page 25.

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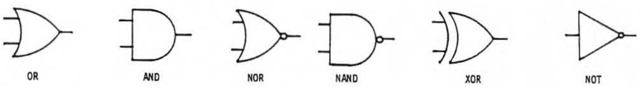
APPENDIX

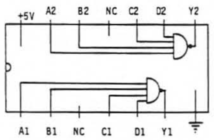
TRUTH TABLES FOR VARIOUS TWO-INPUT GATES

INPUTS		OUTPUTS				
A	B	AND	OR	NAND	NOR	XOR
0	0	0	0	1	1	0
0	1	0	1	1	0	1
1	0	0	1	1	0	1
1	1	1	1	0	0	0

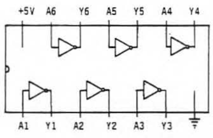
INPUT	OUTPUT
A	NOT
0	1
1	0

SYMBOLS FOR VARIOUS TWO-INPUT GATES





7420 Dual 4-Input NAND gate $Y = \bar{A}.\bar{B}.\bar{C}.\bar{D}$



7404 Hex Inverter $Y = \bar{A}$