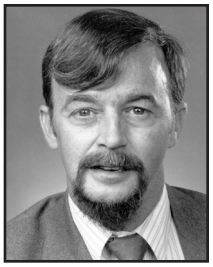
A MOVEABLE FEAST—
A Progressive Approach to the Unit Operations Laboratory

Wm. Curtis Conner Jr., Karl D. Hammond,* and Robert L. Laurence
University of Massachusetts • Amherst, MA 01003

Most undergraduate laboratory courses require groups of students to reproduce specific experiments repeatedly over the course of a term or year. While such repetition might ensure that all students achieve a minimal level of understanding or proficiency, teachers are hard pressed to ensure that each group performs its own experiments, turns in its own work, and does not "borrow" from prior groups. All experiments can be only moderately complex, since students are not allowed to communicate with others and learn from their experience. Students are then faced with the prospect of translating the skills they learn in these isolated experiments into the effective research practices they will be required to use for the rest of their careers.

We have developed a completely different approach to teaching the undergraduate laboratory course that solves the problems associated with repetition by doing away with repetition entirely. We treat each experimental assignment as a charge to study a specific problem that the students themselves propose based on the experience gained by previous groups running the same experimental apparata, the concepts the students have learned in their classes, and journal articles and/or books they have found. We challenge each new group of students to build on the results of prior groups and to achieve different, increasingly sophisticated goals.

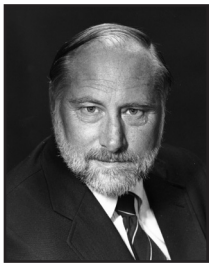
This format requires that experimental stations be sufficiently complex that they enable successive groups to tackle progressively more and more sophisticated experiments throughout the year. Each group is involved in experiments at most experimental stations by the end of the year, and each experimental station carries with it a different level of



William Curtis Conner Jr. *completed his B.S. in chemical engineering in 1968 and his Ph.D. in catalytic chemistry in 1973, both at the Johns Hopkins University. After four years at the Allied Chemical Corporation, he joined the faculty of the University of Massachusetts Amherst in chemical engineering in 1979. Most recently, he has studied the influence of microwaves on catalysis, sorption, and porous catalyst synthesis.*

Karl Hammond *studied chemical engineering at Caltech, where he earned a B.S. in 2003. Between 2004 and 2009, he earned his Ph.D. in chemical engineering at the University of Massachusetts Amherst. He was a lecturer at UMass Amherst in chemical engineering for the fall of 2009, teaching the undergraduate laboratory course. He is currently a post-doctoral researcher at the University of Tennessee, Knoxville, where he studies the materials science associated with magnetic confinement fusion.*

Robert Laurence *is a professor of chemical engineering (emeritus) at the University of Massachusetts Amherst, where he joined the faculty in 1968. He obtained his Sc.B. in chemical engineering practice from MIT in 1957, continued on to the University of Rhode Island for a master's degree (1960), and earned his Ph.D. from Northwestern University in 1965. He has spent his career studying polymerization kinetics and reactor engineering and modeling polymerization processes; he is a co-author of the textbook* Polymerization Process Modeling *(Wiley-VCH, 1995). Professor Laurence is the namesake of the Robert L. Laurence Award of the New England Rugby Football Union.*



* *Present address: Department of Nuclear Engineering, University of Tennessee, Knoxville, TN, 37996.*

prior research—the first station is essentially a "new project," while the last station represents the combined effort of months of research by colleagues (which they are then to continue). Further modifications to the laboratory procedure (manuals and working knowledge) are implemented by the students throughout the term of the laboratory and from year to year to enable knowledge and skills to be passed along to subsequent groups.

This laboratory approach was developed over the course of several years, and was catalyzed by a $250,000 grant from Analog Devices in 1987. This grant enabled us to integrate process control computers and online monitoring and control instruments throughout the laboratory.[1] The laboratory has developed into one of our capstone courses, wherein all aspects of the curricula are revisited and reviewed. Our faculty supports expending the effort to continue to develop this course. This course is valued at four credits due to the oral and written reports involved.

STRUCTURE OF THE COURSE

In contrast to conventional laboratory courses—such as those our students experience in introductory chemistry and physics—in which students are discouraged from using results from prior groups, we *require* students to share their results with groups that will be using the same experimental station to perform a modified experiment. The students are asked to give a proposal to ensure their planned experiment will be feasible and will supplement the knowledge gained by previous groups. This is accomplished through oral presentations given to the entire class. Students are encouraged and expected to ask questions and listen to the instructors' questions as well. We also ask students to share comments with the presenters (funneled through the teaching assistants to ensure anonymity) so as to better their presentations for the next round. At the end of each cycle, the students give a final oral report to the class (and again are required to ask questions and give peer review), followed by a written report describing what they have learned. This report is intended to be similar to a technical report or journal article.

At the beginning of the year, students are asked to divide themselves into groups of three to four students each. Larger groups are inadvisable—it becomes easy for one or more students to "coast" in a large group, learning little while getting by on their group members' hard work. Each group is asked to do three experiments per semester (for groups of three) or four per semester (for groups of four), and each of the members of the group serves as *group leader* for one experiment. This individual is required to coordinate the overall operation of the group, give the oral reports, coordinate peer reviews of other group leaders' presentations, and be the final authority on the content of the final written report.

Each group is in the laboratory for five four-hour periods (the fifth is a make-up period) per experiment and is involved in the oral proposal/report periods. This reflects a total of about 24 hours over six afternoons per experiment, with three or four experiments per semester. Groups rotate between the 10 to 12 experimental stations described below. There are 40 potential laboratory periods per semester, and the laboratory hours thus fill half of these with days not in the laboratory. These days are used for reports, data analysis, report writing, and some classroom instruction. Classroom instruction includes scientific literature searching, safety, hazardous-waste handling, and supplemental topics specific to certain experiments.

The rest of this section outlines a typical sequence of events for one cycle of the laboratory, during which each group does experiments at one station.

PRELIMINARY PROPOSALS

About two to four days before the students enter the laboratory, they give an oral presentation to the entire class (instructors and students) that details what they plan to do with their time in the lab. For the first experiment, this proposal is largely based on the initial assignment given to them by the instructors (similar to what might be done in a traditional laboratory course). In subsequent cycles and during the second semester, the groups propose the experiments they will do after consultation with the literature, prior groups and their reports/presentations, and the instructors. Each group's group leader—because the leadership position rotates with each experiment, each student does it twice throughout the year—presents an oral presentation to the class and instructors. This is typically 15-20 minutes plus 15-20 minutes of discussion and includes:

1. *Introduction to the experiment and its applications outside the laboratory*
2. *Background/theory (including results from prior groups and literature)*
3. *Experimental equipment and procedure*
4. *Data collection and analyses to be performed, especially those methods that differ from those employed by previous groups*
5. *Unusual safety and/or practical precautions and procedures*

CONDUCTING EXPERIMENTS

The students are allowed five four-hour periods in the laboratory in which to conduct their experiments, with the fifth period specifically designated as an "extra" day in which to check results and/or compensate for equipment glitches in previous periods. The students are required to analyze the results during this time period and are expected to show graphs and preliminary analyses when they come into the laboratory for the third period. This minimizes the number of incidents in which students gather four or five laboratory periods' worth of data, stay up all night analyzing them, and

attempt to write up their results only to realize that they did not collect enough data to complete their analyses and/or need to repeat some experiment or another.

Students are supervised in the laboratory by at least one teaching assistant, who is primarily responsible for answering questions and ensuring that the students observe proper safety practices and label hazardous waste and storage containers properly. The teaching assistants are asked to "check out" each group at the end of the laboratory by looking at the experimental station and the students' lab notebooks and checking each for consistency with safety and data integrity policies.

FINAL REPORTS

Final oral reports are presented two to four school days after the last laboratory session during each cycle. The group leaders present 15–20 minute presentations focusing on results and analyses, especially those results that differed from or were unexpected based on the results of previous groups' studies. Each group is also charged with suggesting experiments for the next group on the same experimental station. The students and instructors ask questions during and after these reports, which help each group refine the analyses and better understand the results. These suggestions are usually incorporated into the final written report, due a week or so later. Peer and self-evaluations (all oral presentations are videotaped for self-analyses) are submitted electronically to the instructors. The peer reviews are compiled by one of the teaching assistants and sent back to the presenters anonymously.

About a week after the final presentations (during which the proposals for the next experiment and possibly the first day of the next experimental cycle are occurring), the students hand in their final report for that experiment. This report incorporates discussion of results obtained by the group writing the report, from the group(s) that ran the experiment in past lab cycles, and from the scientific literature and/or their textbooks. The structure of this report is typical of a technical report. The entire process gives our students their first real experience with scientific writing, from developing a proposal to searching for prior literature on the topic to structuring and communicating their results to their colleagues. We employ Turnitin® to scan all reports for plagiarism from prior reports and other sources. Plagiarism is punished severely, in accordance with university policy.

EXPERIMENTAL STATIONS

We currently have 12 experimental stations in our laboratory, up to eight of which are used at any given time. This is an ever-changing list, as we modify several of the experiments each year and add one new experiment every two years on the average. Less productive experiments—those that are difficult to perform and/or do not serve as effectively as teaching aids—are typically phased out to make way for more "current" experiments. For example, four years ago we developed a biodiesel experiment, which produces diesel-range fuel from vegetable oil that can be used in a 6 kW diesel generator by students in mechanical engineering. Nearly all of our experiments have interfaces with computer controls, which allow us to encode flexible, changeable interfaces with which to run any number of experiments.[1] A description of each experiment currently used in our laboratory is given in the rest of this section.

1. Methanation in a Fixed-Bed Catalytic Reactor

Syngas ($CO + H_2$) is reacted over a Ni/Al_2O_3 catalyst to produce methane and water. We have two reactors, one approximating a CSTR and the other a PFR. An in-line infrared CO detector is employed to quantify the conversion. Aspects that can be studied include the general kinetics (Langmuir–Hinshelwood kinetics), including activation energies; the regions for diffusion control; and catalyst deactivation (and regeneration). Using the attached computer, one can manipulate the compositions via mass flow controllers and the reaction temperatures in each reactor via a PID controller. The catalyst can be changed such that later groups can make their own catalysts for study if desired. Students are encouraged to make use of a BET system in another laboratory to estimate surface areas and eventually estimate catalyst turnover frequencies.

2. Polymerization Kinetics

Alkyl methacrylates (methyl- and others) are polymerized using AIBN or benzoyl peroxide as initiators in this batch polymerization studied in a dilatometer. The polymerization reaction can be run in several ways: bulk (no solvent), solution/slurry, or emulsion in a selection of solvents. An oil bath is employed to control the temperature. Differences in the kinetics are studied; specifically, the apparent activation energies will differ under different conditions. In most cases, the differences will be reflected in the nature of the polymer product that is produced. The molecular weight (M_N, M_W) and its distribution (PDI) are measured with gel permeation chromatography (GPC) in our polymer science and engineering department. These experiments are typical of the development of new polymer products, wherein a range of reaction variables is studied to produce polymers for specific applications based on the polymer properties (molecular weight and its distribution).

3. Binary Distillation

Methanol is separated from water using a 13-foot bubble-plate column with 13 trays. Reflux, steam, product, and feed flows can be manipulated while each tray's temperature, the reboiler height, and the steam pressure are measured online by a computer. A gas chromatograph (GC) is employed to analyze gas or liquid compositions drawn from each tray. The primary control variables are the steam flow rate into the reboiler and the reflux flow rate. The dependent variables are the bottoms and distillate compositions. There are logical

combinations of control and manipulated variables: steam-bottoms and reflux-distillate. It is crucial that the students present a proper energy analysis of this system. The dynamics of this semi-pilot sized system are somewhat slow and the optimum protocol for changes in the system parameters can be investigated. A diagram of the computer interface for this experiment is shown in Figure 1.

4. Production of Biodiesel From Vegetable Oil

Vegetable oils are converted to diesel fuel replacements (fatty acid alkyl esters) by transesterification with methanol or ethanol. The reaction is base-catalyzed, and students have the ability to choose between several catalysts, including NaOH, $NaOCH_3$, KOH, $KOCH_3$, $Ca(OH)_2$, $Ca(OCH_3)_2$, and SrO. The students have the opportunity to study the catalytic kinetics of the process, which, in excess methanol, is roughly an $A \rightarrow B \rightarrow C \rightarrow D$ irreversible reaction (where A, B, C, and D are the triglyceride, diglyceride, monoglyceride, and fatty acid alkyl ester, respectively). The products are analyzed by gas chromatography, if appropriate, or high-pressure liquid chromatography. The pH is measured for waste oil feeds to ascertain any free fatty acids present and the presence of water is quantified in each phase before and after reaction.

5. Characterization & Control of a Heat Exchanger

This station consists of two sequential shell-and-tube heat exchangers: a steam-water heat exchanger followed by a water-water heat exchanger. The water-water heat exchanger can be operated in co-current or counter-current configurations. Temperature is measured at 15 positions throughout the water streams, inside the steam chamber, and on the steam pipe walls. These temperatures are monitored by a computer, which also allows the students to control the water and steam flows. The focus of the first few experimental cycles is typically on the mechanics of heat exchange in the various configurations: measuring heat transfer coefficients, co-current vs. counter-current, verifying correlations for the Nusselt number vs. Reynolds number, and so on.

The focus of later experiments is typically on control. There are several options available to control the heat exchanger, and students are asked to choose one. These options include control of any of several temperatures (of the process or cooling water at various points), employing any of several inputs (steam, cooling, and process water), and using various control strategies (open-loop modeling, tuning relations, step/impulse responses, and frequency response). At first, simple P, PI, PD, and PID control can be employed. Students are encouraged to use simulation methods, especially with MATLAB® and Simulink,® to interpret their results (with and without feedback) as early as possible. This station is also able to study frequency response techniques, including gain/phase margin, Bode diagrams, and Nyquist diagrams—a part of control theory that is often overlooked in chemical engineering courses.

6. Membrane Separation by Permeation

The permeation station, similar to those described by Davis and Sandall,[2,3] allows students to study the enrichment of oxygen from binary and ternary gas mixtures flowing in series or in parallel into two polymeric membranes (Permea, Inc.; St. Louis, MO). Groups investigate the separation of oxygen from nitrogen, argon, helium, or carbon dioxide as binary mixtures. In later experiments, students can choose to work with mixtures of oxygen and carbon dioxide in ternary mixtures, such as $N_2/O_2/CO_2$ and $He/O_2/CO_2$, often with different mole fractions. We have an online infrared detector in addition to oxygen sensors to allow the students to detect carbon dioxide as well as oxygen. Groups propose to compare the enrichment of oxygen as a function of flow rate/back pressure from different feeds and to compare these to prior separations (employing different feed compositions and/or different column configurations).

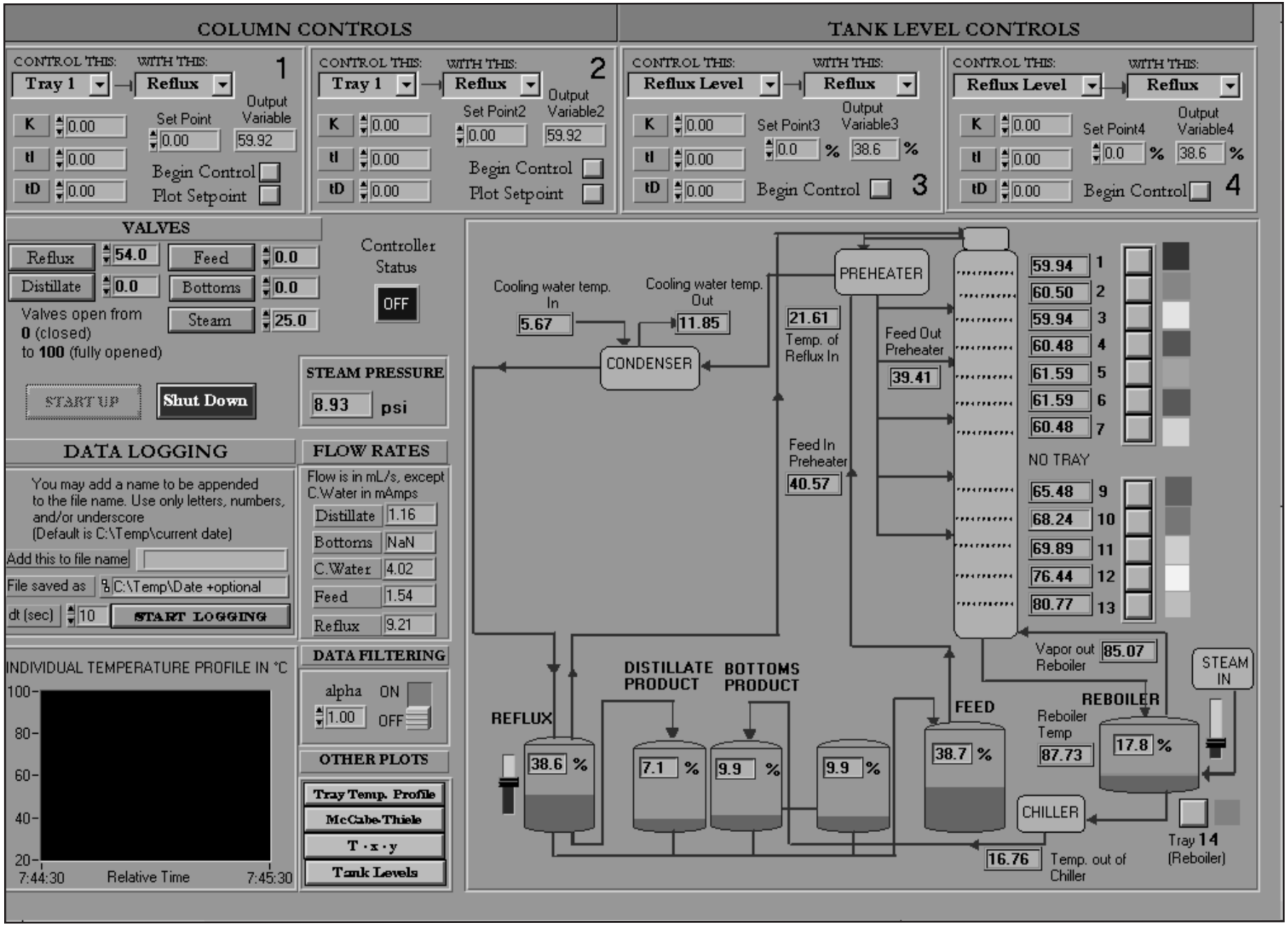
7. Ion Exchange

The ion exchange station employs a variety of ion-exchange resins for the removal of cupric sulfate (or optionally other metals) from an aqueous solution. A 4-inch-long column, 1/2 inch in diameter, is employed with an in-line UV/visible spectrometer as a detector. A controllable liquid pump is employed along with valves to bypass the column and change the feeds. We have several resins of differing capacities and mesh sizes available, all strong acid cation resins produced by Dow. Each new group chooses a resin to compare to the prior results and to show and explain the differences. Equilibrium adsorption isotherms are measured in batch experiments prior to the online measurements in the column by computer. Column regeneration and the dynamics of the processes involved can also be studied. Pressure and flow measurements across the column enable the students to analyze flow in porous media. Modeling the column as a series of CSTR's and/or PFR's is encouraged.

8. pH Control

This experiment involves the control of a liquid-phase stirred tank reactor to control the pH of acid-base streams. The basic setup is based on the work of Henson.[4] As in all control situations, the initial approach is to understand and analyze the dynamics followed by the design and implementation of a control scheme. A buffer stream can also be introduced into the reactor as a disturbance as well as a way to moderate the pH. Students have the potential to control several reactor

Figure 1 (facing page). *Computer interface for our distillation experiment. The upper portion of the interface allows students to adjust the gains to simple PID controllers, while the rest of the diagram reports all online measurements. This type of interface allows the students to perform a wide variety of experiments on the column.*


COLUMN CONTROLS
CONTROL THIS: Tray 1
WITH THIS: Reflux
1
K 0.00
tI 0.00
tD 0.00
Set Point 0.00
Output Variable 59.92
Begin Control
Plot Setpoint
CONTROL THIS: Tray 1
WITH THIS: Reflux
2
K 0.00
tI 0.00
tD 0.00
Set Point2 0.00
Output Variable2 59.92
Begin Control
Plot Setpoint
TANK LEVEL CONTROLS
CONTROL THIS: Reflux Level
WITH THIS: Reflux
K 0.00
tI 0.00
tD 0.00
Set Point3 0.0 %
Output Variable3 38.6 %
Begin Control
3
CONTROL THIS: Reflux Level
WITH THIS: Reflux
K 0.00
tI 0.00
tD 0.00
Set Point4 0.0 %
Output Variable4 38.6 %
Begin Control
4
VALVES
Reflux 54.0
Distillate 0.0
Feed 0.0
Bottoms 0.0
Steam 25.0
Valves open from 0 (closed) to 100 (fully opened)
Controller Status
OFF
START UP
Shut Down
STEAM PRESSURE
8.93 psi
DATA LOGGING
You may add a name to be appended to the file name. Use only letters, numbers, and/or underscore
(Default is C:\Temp\current date)
Add this to file name
File saved as C:\Temp\Date +optional
dt (sec) 10
START LOGGING
FLOW RATES
Flow is in mL/s, except C.Water in mAmps
Distillate 1.16
Bottoms NaN
C.Water 4.02
Feed 1.54
Reflux 9.21
INDIVIDUAL TEMPERATURE PROFILE IN °C
100
80
60
40
20
7:44:30
Relative Time
7:45:30
DATA FILTERING
alpha 1.00
ON
OFF
OTHER PLOTS
Tray Temp. Profile
McCabe-Thiele
T - x - y
Tank Levels
Cooling water temp. In 5.67
Cooling water temp. Out 11.85
CONDENSER
PREHEATER
21.61 Temp. of Reflux In
Feed Out Preheater 39.41
Feed In Preheater 40.57
59.94 1
60.50 2
59.94 3
60.48 4
61.59 5
61.59 6
60.48 7
NO TRAY
65.48 9
68.24 10
69.89 11
76.44 12
80.77 13
Vapor out Reboiler 85.07
STEAM IN
REBOILER
Reboiler Temp 87.73
17.8 %
DISTILLATE PRODUCT
BOTTOMS PRODUCT
REFLUX
FEED
38.6 %
7.1 %
9.9 %
9.9 %
38.7 %
CHILLER
16.76 Temp. out of Chiller
Tray 14 (Reboiler)

parameters, including acid, base, and buffer flow rates (in and out); reactor volume (liquid depth); and reactor stirring. There are any of several control schemes that can be implemented from simple P/PI/PID to MIMO. Groups choose a scheme, collect appropriate data on dynamics, implement the scheme, and evaluate the resulting controlled dynamics. The nonlinearity of pH is a challenge. Groups can build on the work of previous groups to implement more and more sophisticated controllers in later cycles. Frequency response analysis can be utilized in these experiments as well, if desired.

9. Polymer Extrusion

The purpose of polymer extrusion is to produce a continuous strand of polymer of uniform dimension from pellets. Several dies of varying dimensions, including round and ribbon dies, are available. The conditions throughout the extruder control the nature of the product. This experiment should employ experimental design techniques to optimize the production process and its influence on dimension and uniformity for different die sizes. We have several choices of polymers available, and each group is charged with characterizing the product in terms of dimension and uniformity. Die swelling can be a complicating factor for certain polymers under specific conditions, and the students are asked to estimate the extent of die swelling for their polymer. We have access to differential scanning calorimetry to characterize the polymer crystallinity, which will depend on process conditions. By employing polymers of different colors, students may be able to estimate the uniformity of the mixing during the extrusion process and examine the effects of mixing on material properties. A photograph of our extruder in operation is included as Figure 2.

10. Polymer Injection Molding

This station makes use of a polymer injection molding apparatus to produce a part (plastic dog bone or spiral) with certain properties. Dog bones can be tested with an Instron mechanical testing instrument to compute engineering stress/strain curves either in stretch to failure or three-point bending. Normally, the goal is to produce the strongest part, but it could also be to produce a part that breaks within a given range of stresses. There are several variables that can be manipulated in this process that change the strength, uniformity, and/or mechanical performance of the final part. These are ideally suited to experimental design techniques, which allow the students to learn as much as possible while wasting as little time and material as possible. The spiral mold allows students to study heat transfer and viscosity as a function of injection pressure by observing how far the polymer makes it into the mold. We have several polymers to choose from for injection molding and extrusion, including polycarbonate (Lexan®), acrylonitrilebutadiene-styrene (Cycolac®), polypropylene-poly(phenylene oxide) (Noryl®), and high-density polyethylenes of different molecular weights and polydispersity indices.

11. Fermentation

This experiment studies fermentation of sugars—either synthetic sugar/water solutions or juices—using a 1.25 L BioFlo III stirred tank fermenter (New Brunswick Scientific; Edison, NJ). This allows students to study a living catalyst (yeast) and measure the kinetics of the yeast's production of ethanol. Two varieties of yeast are available, each of which produces a different rate of sugar metabolism. The setup allows students to change reactor volume, stirring rate, and sugar source. Students are expected to test and understand the changing kinetics of yeast growth and ethanol production to determine the optimum mixing, feed (including O_2), and temperature settings that maximize alcohol production and/or yeast growth. The groups can also measure the total CO_2 production due to yeast growth and alcohol production by performing a total carbon balance for the process.

12. Protein Separation

This experiment uses chromatography to separate active biological enzymes (acid phosphotase, AcP) from wheat germ or non-pathogenic E. coli bacteria. Students extract AcP using osmotic lysis, pass the extracted material through an ion exchange chromatographic column and collect fractions, measure the enzyme concentration of each fraction, measure the total protein content of each fraction, and use this information to improve the process and make suggestions for the design of an industrial-scale system. Enzyme concentrations are determined by measuring the rate of the enzyme-catalyzed

Figure 2. *Students extruding Lexan® (polycarbonate) with a ribbon die while following the solidification on the conveyor belt using an infrared camera.*

conversion of p-nitrophenyl phosphate into p-nitrophenol by assuming Michaelis–Menten kinetics with excess substrate. We have recently acquired a UV-Vis spectrometer to follow the separations on-line with a flow cell.

EXAMPLE PROJECT PROGRESSIONS

There are many possible sequences of projects that can be accomplished at each experimental station over the course of the year. We give three examples here, based on students' work in one particular semester.

Methanation

The first two groups attempted to extract the apparent reaction order using the CSTR. The third group found their results to be inconsistent, so they proposed to study the same thing (apparent reaction order) using the PFR instead. The fourth group, similarly, found their results to be questionable, so they proposed experiments to measure (a) the order of the reaction, (b) the activation energy (by studying different temperatures), and (c) the effect of residence time on conversion (and whether it was consistent with the orders found by previous groups). Another group proposed to do experiments to estimate the Thiele modulus and therefore the extent of diffusion vs. reaction control, as well as the turnover frequency.

This sequence is typical of a first semester: students think they understand kinetics and reactor engineering, proceed to take measurements of, say, the rate, plot it against inlet concentration over a relatively narrow range of conversions, and assume they have a graph that tells them the reaction order. The instructors point out the error of their ways, and they either attempt to fix it in later experiments or explain the problem in the final report so that subsequent groups can do the experiment over again (with their own modifications) to obtain the results they think are appropriate and correct. The resulting reaction orders and activation energies can be compared to the numerous literature studies of methanation over similar nickel catalysts. Langmuir–Hinshelwood rate expressions are employed to rationalize the observed orders.

Heat Exchange

The first groups proposed to measure the heat transfer coefficients in the steam-water section and the water-water section, respectively. The next group proposed to corroborate the first groups' values through Wilson plots and other correlation-based methods. Subsequent groups attempted to characterize the response of the process water temperature to a change in process water flow rate, finding a first order plus time delay model to be more or less adequate. Another group chose to characterize the response of process water temperature to changes in cooling water flow rate, finding that a second order model that included inverse response was necessary; another group varied steam pressure. Later groups attempted to find the effective operating range of the models they had developed, the range of PID gains that could be used, and the frequency response (closed and open-loop Bode diagrams and resulting analysis) of both their models and the real system. All later groups explored the differences—particularly the non-linearities and actuator saturations that exist—between their model and the real thing.

The heat exchanger, when used in this manner, is a particularly effective teaching tool: students learn that they are not quite as comfortable as they thought they were with concepts such as overall heat transfer coefficients and effectiveness factors, and they learn volumes about the practicalities of control that are only possible to hint at in a classroom setting.

Polymer Extrusion

The first group to study extrusion was given its choice of polymer among the five polymers and several batches per polymer available, selecting a particular batch of Lexan polycarbonate. They observed correlations between temperature and pressure, torque and flow rate, and torque and temperature at a given screw speed. They also found (perhaps unwittingly) that drawing the polymer during cooling changes the tensile strength. Subsequent groups studied each of these phenomena in more depth. One group attempted to maximize tensile strength in high-density polyethylene (HDPE); another studied the effect of screw speed vs. ultimate tensile strength in HDPE; another studied the effects of mechanically mixing different colors and lots of Lexan polycarbonate; the next studied the effect of nozzle temperature and cooling rate on the extent of crystallinity in HDPE; another studied the effects of nozzle temperature, screw speed, and composition for mixing two batches of Noryl PPX [poly(phenylene ether) mixed with polypropylene], also studying the effect of drawing rate on physical properties. The last group studied die swell using a circular die using Lexan polycarbonate and Noryl PPX. The extrusion and injection molding experiments are, for many students, the first exposure to things like stress and strain, polymer rheology, and concepts like tensile strength.

OUTCOMES

Our unit operations laboratory has developed into a capstone course in our curriculum. It draws upon and incorporates material from all prior courses in the department, including material balances, thermodynamics, fluid mechanics, heat and mass transfer, separations, kinetics and reactor engineering, design, and control. Material from many classes taught outside the department (*e.g.*, spectroscopy, chemistry, rheology) is often utilized as well. Many experiments make use of concepts from several courses, as detailed in Table 1 (next page). In addition, this format accentuates

- *Group work, including inter-group interactions.*
- *Leadership skills*
- *Oral communications, including self- and peer review*
- *Scientific writing*
- *Real-time data acquisition*

TABLE 1
Outcomes From Specific Courses Used in the Course Of Each Experiment

	Mass/Energy Balances	Thermodynamics	Fluid Mechanics	Heat Transfer	Mass Transfer	Kinetics	Reactor Engineering	Separations	Control	Design	Bioengineering	Polymers/Rheology	Materials Science	Design of Experiments
Methanation	X	X		*	*	X	X		*	*			*	*
Polymerization	*	X	*	*	*	X	*	X		*		X	X	*
Distillation	X	X		X	X			X	*	X				*
Biodiesel	*	X		*	*	X	X	X		*	X			*
Heat Exchange	X	X	X	X					*	X				*
Membrane Separation	X	*			X			X		*		*	*	*
Ion Exchange	X	X			X	*	X	X		*			*	
pH Control	X	X					X		X	*				
Extrusion		X	X	X	*							X	X	X
Injection Molding		X	X	*	*							X	X	X
Fermentation					X	X	X	X	*	*	X			*
Protein Separation	X	X			X	X		X		*	X			*

Experiments that require a given subject matter for all work on that station are denoted "X"; those that can use that subject matter (depending on the experiment) are denoted by an asterisk (*).

- *Statistical analysis and experimental design*
- *Searching for and understanding prior studies—including primary literature*
- *Project proposals*

This format prepares students not only for laboratory work, but teaches them to function as engineers and scientists and gives them valuable training across all facets of the discipline.

An ongoing website (<http://www.ecs.umass.edu/che/che401-2/>) is maintained throughout the semester with equipment manuals as well as ongoing lab manuals that are updated as the students learn how best to run each experiment. Safety is a prime concern, and the students receive training before they enter the laboratory and are immediately sent out if violations (such as lack of safety glasses or improper footwear) are observed. Students are asked to prepare a "SAFE" form before each new experiment and inform the class of any unusual hazards at the time of their proposal.

Student feedback from this course is generally quite positive, even though the workload is significant. More often than not, the students remark, "Now I know how that actually works!" or "It worked so much better in class…." or "Could we modify the experiment next time to look for…?" The open format allows the last question to be answered "yes" more often than not, which gives the students an additional feeling of empowerment over the work they do.

CONCLUSIONS

This approach to the senior laboratory course has been integrated into our curriculum for more than 15 years now, and we are very pleased with the outcome. Students learn as much or more about some topics—particularly catalytic and polymerization kinetics—than they ever could in their regular courses. It also empowers students: *they* develop the assignments, *they* determine which analyses are appropriate, *they* determine the level of work that is necessary to obtain the results they need, and *they* determine what shreds of knowledge and experience are important enough to warrant being discussed in both their own reports and in the lab manuals. The writing, oral communication, and planning skills they learn as part of the proposal/final report structure are indispensable, giving our students a head start on the skills necessary to create the project/grant proposals, journal articles, technical reports, and other scientific communications they will be asked to write during the remainder of their careers.

ACKNOWLEDGMENTS

The authors thank Sue Roberts, Mike Henson, and Neil Forbes for their help with the fermentation, pH control, and protein separation experiments as well as numerous students and teaching assistants over the years for writing lab manuals, designing computer interfaces, and testing new experiments. We owe special thanks to Gary Czupkiewicz, Paul Garbin, Joe Smith, and Tom Spooner for providing invaluable techni-

cal support and other assistance. The department is greatly indebted to Analog Devices for donating, some 25 years ago, start-up money (so to speak) that allowed us to implement computer-controlled measuring apparatus on most experiments in the laboratory. We are also grateful to GE Plastics (now SABIC Innovative Plastics) and DOW (formerly Union Carbide) for donating many of the polymers we use in the extrusion and injection molding experiments. We are also indebted to HiTech Trader and Gillette for the donation of many pieces of equipment for our laboratory.

REFERENCES

1. Conner, W.C., "Incorporation of Process Control Computers in the Undergraduate Laboratory: A Case Study," *Chem. Eng. Ed.*, **24**(2), 106 (1990)
2. Davis, R.A., and O.C. Sandall, "A Membrane Gas Separation Experiment for the Undergraduate Laboratory," *Chem. Eng. Ed.*, **25**(1), 10 (1991)
3. Davis, R.A., and O.C. Sandall, "A Simple Analysis for Gas Separation Membrane Experiments," *Chem. Eng. Ed.*, **37**(1), 74, (2003)
4. Henson, M.A., *Feedback Linearization Strategies for Nonlinear Process Control*, Ph.D. Dissertation, University of California, Santa Barbara (1992) ❐