CHEMICAL PROCESS SYSTEMS: A SECOND COURSE IN CHEMICAL ENGINEERING

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IN 1976 THE UNDERGRADUATE chemical engineering curriculum at N. C. State was revised. The changes involved mostly non-chemical-engineering course offerings, but a consequence of the revision was that a block of four chemical engineering credits became available in the second term of the sophomore year.

In designing a course to fill this block, our primary goal was to provide an experimental background to complement and reinforce the calculation-oriented material presented in the stoichiometry course. At the same time, each of us had his pet nomination for the "What this curriculum needs more of is . . . ." sweepstakes; popular entries included statistics, process instrumentation, physical property estimation, computer applications, technical report writing, and the chapter on transient balances that the stoichiometry course never gets to. We therefore set out to fill as many of these voids as we could with the new course, without allowing the course to degenerate into a grab bag of apparently unrelated topics.

The result of this effort is CHE 225—Chemical Process Systems. The course consists of three lecture hours and one two-hour laboratory session per week. It follows the stoichiometry course in the curriculum and precedes the unit operations and thermodynamics sequences. The lecture topics covered in the course are listed in Table 1, and the experiments performed are given in Table 2.

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TABLE 1
Lecture Topics

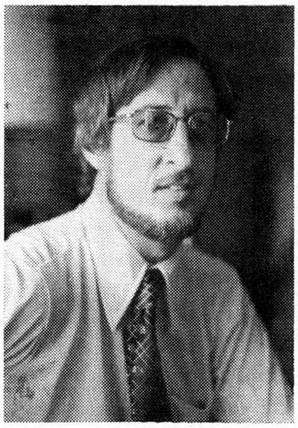
1. SURVEY OF PROCESS INSTRUMENTATION AND MEASUREMENT METHODS. Temperature, pressure, and flow-rate sensors. Review of DC instrumentation—galvanometers, ammeters, voltmeters, null-point potentiometers, Wheatstone bridge. DP cells and control valves. (1 week)
2. STATISTICAL DATA ANALYSIS. Probability distributions. True and sample means and standard deviations. The z and t distributions. Precision of measured and calculated quantities. Propagation of error. Calculation of confidence limits. Linear regression. (6 weeks)
3. PHYSICAL PROPERTY MEASUREMENT AND ESTIMATION METHODS. Descriptive material centered on the subject matter of Experiments 6-9 (Table 2). (2 weeks)
4. MATERIAL AND ENERGY BALANCES ON UNSTEADY-STATE SYSTEMS. Setting up and solving differential balances on simple lumped-parameter systems, including batch and continuous-stirred-tank reactors. (3 weeks)
5. INTRODUCTORY SYSTEM DYNAMICS AND MODELING. First-order processes and instruments; determination of static sensitivity and time constant. Qualitative behavior of second-order devices. Qualitive introduction to control. (2 weeks)

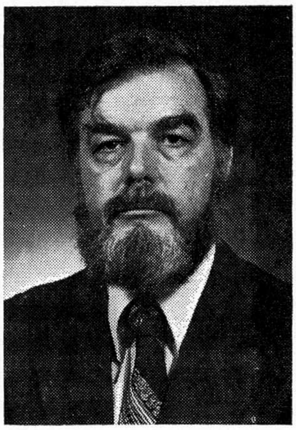
The paragraphs that follow summarize the principal features of the course.

LECTURES

AS TABLE 1 INDICATES, the lecture material is divided into several blocks. The subject areas are process variable measurement methods, statistical data analysis, physical property measurement and estimation, transient material and energy balances, and introductory system dynamics. The relationships among these topics that give the course coherence are conveyed primarily through the homework, including the laboratory data analysis.

Several sets of homework problems assigned during the term illustrate the lecture material and the calculations associated with the experiments.





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In addition, four computer problems are assigned that incorporate several of the techniques introduced in this course and in the stoichiometry course. Problems given recently include trial-and-error determination of a multicomponent vapor dew point, calculation of an adiabatic flame temperature using Newton's rule, forward integration of a transient material balance using Simpson's rule, and solution of material balance equations for a multistage separation process.

LABORATORY

THE LABORATORY IS organized into four blocks of experiments (Table 2). The students work in groups of three or four, with each group performing one experiment per week. The groups rotate through all the experiments in a block before moving on to the next block, so that in a given laboratory session as many as four different experiments may be going on concurrently.

The experiments are all relatively simple; our emphasis is on the analysis and interpretation of data, and on technical report writing. The handout for an experiment consists of a set of instructions on what is to be done and what is to be calculated, several discussion questions concerning the subject of the experiment, and in some cases a supplementary set of notes giving background information not provided in the lectures. Group reports, containing Procedure, Results, and Conclusions sections with appendices for detailed calculations and raw data, are due at the following laboratory period. Quality of writing and organization are given as much weight as technical content in grading the reports.

The course generally provides the students with their initial exposure to technical writing, as their first reports make transparently clear. By the end of the semester, however, most squads catch on to the way the game is played: their reports have clearly defined beginnings, middles, and ends; figures are labeled; results are displayed prominently in the Results section, rather than being buried somewhere in the Procedure or Conclusions: detailed calculations are placed in appendices; and most prose is a reasonable approximation of standard English.

A good illustration of the approach taken in the laboratory is provided by Experiment 6 (Table 2). The students carrying out this experiment are presented with a Cottrell pump apparatus, including a vacuum pump, water condenser, and mercury manometer, and are instructed in its use by a teaching assistant. They first measure the boiling point of carbon tetrachloride at several pressures, including atmospheric pressure; they then replace

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the 0-110°C thermometer used in this part of the experiment with a Beckmann thermometer, remeasure the boiling point of pure carbon tetrachloride at atmospheric pressure, and finally measure the boiling points of three aliquots of a solution of an unknown solute in carbon tetrachloride. (They are given the mass ratio of solute to solvent.)

The required analysis, which they have a week to complete, involves the following calculations:

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1. Verify the validity of the Clausius-Clapeyron equation by plotting the vapor pressure data in a suitable manner.
2. Fit a line to a subset of the data close to atmosphere pressure by linear regression.
3. Estimate the heat of vaporization of carbon tetrachloride at 1 atm from the regression coefficient, and calculate a 95% confidence interval for the estimate.
4. Compare the estimated value with tabulated values from Felder and Rousseau [1] (the stoichiometry text) and Perry's Handbook [2], and with values estimated from Trouton's rule and Chen's equation [1].
5. Use the boiling point elevation data to estimate the molecular weight of the unknown solute, and calculate a 95% confidence interval for the estimate.

The function of the Cottrell pump and the assumptions underlying the Clausius-Clapeyron equation and the colligative solution property formulas should all have been discussed in the lectures prior to the experiment, and in addition the pertinent material is summarized in handouts made available in the laboratory. The required statistical methodology is covered in lectures well before the experiment is performed.

The experiments in Block III of Table 2 may overlap in part with those performed in the physical chemistry laboratory at some schools. This is not a problem at N.C. State, since we have replaced the traditional physical chemistry course (on the grounds of excessive overlap with our stoichiometry and thermodynamics courses) with a special topics course in physical chemistry. Departments having a separate physical chemistry laboratory course might wish to modify the experiment selection to eliminate duplication.

INFORMATION SOURCES

Perhaps not surprisingly, no single reference has been found suitable for the course as it is currently constituted. We have used Holman [3] and Graham [4], supplemented by readings in the Chemical Engineers' Handbook [2], for material on instrumentation and statistics, and we use Felder and Rousseau [1] for transient material and energy balances and some of the assigned computer problems. Most lecture material on statistics, physical property measurement, and system dynamics is summarized in class handouts, supplemented by suggested readings in Salzberg *et al.* [5] and Reid *et al.* [6] for physical property determination and Spiegel [7] for statistics.

SUMMARY OF COURSE OBJECTIVES

The primary objective of the course is to introduce the experimental side of chemical process technology. The lectures and laboratories provide an understanding of how the variables and physical properties that underlie all process calculations are measured, and how the measured values are analyzed statistically and converted into forms useful for process calculations. The students learn extensions of the steady-state analysis presented in the stoichiometry course, and they are introduced to elementary notions of process and instrument dynamics. They are also exposed to a variety of computer applications, including off-line process data analysis, process simulation, and on-line data logging and control. Finally, they are sent into the unit operations laboratory sequence with a good introductory background in process instrumentation, data analysis, and technical report writing.

COURSE EVALUATION

The students respond positively to the laboratory portion of the course; the only common requests are for more independence and more background on the hybrid simulation and digital control computer experiments. They, and we, are reasonably satisfied with the mix of topics covered in lectures, although several would prefer less statistics and more systems analysis. Several have commented that the lecture material on balances and the computer problems enabled them to pull together much of the material in the stoichiometry course that they had not fully understood the first time around. It will be interesting to ask the same students, a year or two later, how the course affected their perceptions of the rest of the chemical engineering curriculum. □

REFERENCES

1. R. M. Felder and R. W. Rousseau, *Elementary Principles of Chemical Processes*, New York, John Willey & Sons (1978).

TABLE 2
Experiments

BLOCK I. PROCESS VARIABLE MEASUREMENT

1. TEMPERATURE MEASUREMENT. Make Cu-constantan thermocouples using both carbon-arc and mercury-arc weld methods. Calibrate both at the steam point. Compare the mean emf with a tabulated value. Use the statistician's t-test to see whether the responses of the two thermocouples are significantly different.
2. PRESSURE MEASUREMENT. Calibrate a bourdon gauge against manometers and a deadweight tester. Estimate the uncertainty associated with the determination of an absolute pressure using the bourdon gauge and a laboratory barometer. Investigate hysteresis effects in the bourdon gauge.
3. FLOW-RATE MEASUREMENT. Calibrate a water rotameter and an orifice meter. Fit a line to the rotameter calibration curve by linear regression. Estimate the discharge coefficient of the orifice meter.
4. DC MEASUREMENTS. Use a Wheatstone bridge to determine the internal resistance of a micro-ammeter. Compare the precision of this measurement with that of an indirect measurement using a battery and a board-mounted resistor. Measure the resistance of a platinum resistance thermometer at two temperatures, and calculate the temperature coefficient of resistance.

BLOCK II. DIGITAL DATA MONITORING

5. Use a digital process-control computer to monitor, average, and print out (a) thermocouple readings, (b) the transient response of a DP cell level indicator in a tank being drained, and (c) the "noisy" flow of liquid in a pipeline governed by a pneumatic control valve at several valve pressure settings.

BLOCK III. PHYSICAL PROPERTY DETERMINATION

6. VAPOR PRESSURES AND BOILING POINT ELEVATION. Use a Cottrell pump apparatus to determine the boiling point of carbon tetrachloride at six or seven pressures. Use a Beckman thermometer to determine the boiling point elevation caused by the presence of a weighed amount of an unknown solute. Use a semilog plot and the Clausius-Clapeyron equation to calculate the heat of vaporization of carbon tetrachloride, and compare the results with tabulated and estimated values; then calculate the unknown solute molecular weight and determine a 95% confidence interval for the estimate.
7. GAS CHROMATOGRAPHY. Calibrate a gas chromatograph to analyze liquid mixtures of methanol and isopropanol. Study the effects of carrier gas flow rate and sample volume on retention time and resolution.
8. DENSITOMETRY. Using a Westphal balance, measure the densities of distilled water at two temperatures, of pure isopropanol, and of three water-isopropanol mixtures. Compare the pure component densities with tabulated values, estimate the coefficient of thermal expansion of water and calculate a confidence interval for the estimate, and statistically test the hypothesis that the density of a 50% water-isopropanol mixture is significantly different from the value calculated assuming volume additivity.
9. ELECTROLYTIC CONDUCTION. Measure the specific and equivalent conductances of aqueous solutions of potassium chloride and acetic acid. Use the latter results to estimate the dissociation equilibrium constant of acetic acid.

BLOCK IV. PROCESS DYNAMICS

10. STIRRED-TANK DYNAMICS. Feed cold water into a well-stirred heated tank initially containing hot water, and withdraw water at the same rate, monitoring the effluent temperature. Calculate the heating rate from the final temperature, and analyze the transient response to show that the system functions as a first-order process. Determine the time constant, and from it the throughput rate.
11. HYBRID SIMULATION OF A FIRST-ORDER PROCESS. Study the performance of a first-order process simulated on a hybrid computer. Verify the exponential character of the response to step forcing, and examine the dependence of the response on the magnitude of the time constant. Observe how the response changes if proportional and integral control elements are added.
12. REACTION KINETICS. Carry out the saponification of ethyl acetate with sodium hydroxide in a batch reactor at 30°C, beginning with the reactants in stoichiometric proportion and following the progress of the reaction with a pH meter. Confirm an assumed second-order rate law by the method of integration, using linear regression to estimate the rate constant, and determine a confidence interval for the estimate.

2. R. H. Perry and C. H. Chilton, Eds., *Chemical Engineers' Handbook*, 5th Edition, New York, McGraw-Hill (1973).
3. J. P. Holman, *Experimental Methods for Engineers*, 2nd Edition, New York, McGraw-Hill (1971).
4. A. R. Graham, *An Introduction to Engineering Measurements*, Englewood Cliffs, Prentice-Hall (1975).
5. H. W. Salzberg, J. I. Morrow and S. R. Cohen, *Laboratory Course in Physical Chemistry*, New York, Academic Press (1966).
6. R. C. Reid, J. M. Prausnitz and T. K. Sherwood, *The Properties of Gases and Liquids*, 3rd Edition, New York, McGraw-Hill (1977).
7. M. R. Spiegel, *Statistics*, Schaum's Outline Series, New York, McGraw-Hill (1961).