

# PUTTING COMMERCIAL RELEVANCE INTO THE UNIT OPERATIONS LABORATORY

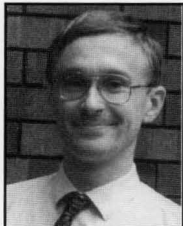
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Imagine that you are a junior engineer in a minerals processing company. Your manager calls you in one day and the following conversation ensues:

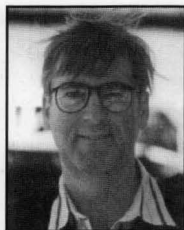
*We have a new project to recover a valuable ore product. Your part is the filtration of a slurry. We want to separate 50 tons per week of dry alumina. It will be worth \$1,000 per dry ton. Find out the most profitable way to do it. You will have to do some experiments to get the rates of filtration. We only have a small test rig, so you will have to scale the whole thing up. I have forgotten all of the theory, so you figure it out and give me a report in two weeks' time that I can take to the next Board Meeting and tell them how much we have to spend. It'll have to be good because I don't want to make a fool of myself. Off you go.*

So begins a typical student pep talk for the unit operations laboratory where we have attempted to introduce the flavor of a real commercial enterprise. Until recently, all of our laboratory experiments addressed only the "engineering science" aspects of the work. Typically, students were asked to get data from a rig, to do correlations, and to compare



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these with literature values and theories. With little perceived relevance and no significant goal for their work, they would struggle to find something relevant to say. Frequently the implied conclusion to their investigations was a grumpy "so what!"

Of the thirteen experiments available, we have now introduced a measure of commercial relevance into two of them: Filtration and Leaching. The first is a realistic problem of process scale-up, and the second is an economic optimization of an existing plant.

## FILTRATION

Students are asked to design a full-scale filtration process for a hypothetical mineral company, Total Recovery and Marketing Proprietary Limited (TRAMPL). Because they have access only to a laboratory-scale filter for detailed work, the problem is one of scale-up. Using constant pressure operation on a laboratory scale filter, students must produce a set of filtrate volume-versus-time data from which they calculate two fundamental design parameters: 1) the specific filter cake resistance, and 2) the filter medium resistance. These parameters can then be applied to determine the operation of a full-scale plant.

A major variable is the addition of a "filter aid." Addition of filter aid (diatomaceous earth) should reduce the cake resistance and consequently the filtration cycle times. The resultant reduction in capital and labor costs should be offset by greater running costs to pay for the filter aid. Students find the economic optimum of their scaled-up process.

Certain design constraints are provided:

- The scale of the desired operation (how many tons per week of alumina are to be recovered)
- The nature and composition of the slurry which will be separated
- The capital costs of installing filtration equipment
- The running costs in terms of labor and overheads
- The costs of added reagents such as filter aid which may prove useful in lowering costs by speeding up production rates

The provision of these economic constraints is additional to any physical constraints due to the size and layout of the laboratory equipment.

Students write a report with the aim of conveying to TRAMPL managers enough written information to convince them that the experimental work and the conclusions drawn can be relied upon. We emphasize that management should be able to make a confident decision, based on the report, that spending capital and employing staff to carry out the process specified will bring a good return on investment for the company. In the briefing sheets which accompany the laboratory experiment, considerations in deciding the content of critical sections of the report, such as the Introduction, the Apparatus and Method sections, the Results and Discussion sections, and especially the Summary, are outlined. This additional guidance is important, since reports to management must be succinct as well as relevant.

In designing their experimental protocol, students are asked to anticipate all the independent parameters that make an impact on the outcome, such as the filter area of the laboratory scale rig and the filter area and frame volume of the full-scale rig and filter-aid dosage. We emphasize that students should design the experimental work considering dosages of filter aid which are relevant both economically and technically. This can be done by assuming some simple profitability constraints and showing that the possible range of filter aid dosage is between zero and a certain upper limit. At the upper limit, the cost of the filter aid begins to make the process uneconomic. Information and the problem statement given to the students are shown in Table 1.

The equipment consists of a laboratory plate-and-frame filter press (see Figure 1) that can be operated at constant pressure but not at constant

**TABLE 1**  
**Technical and Economic Information for the Filtration Experiment**

**Technical Design Parameters**

Required rate of dry alumina production: 50 tons per week  
Slurry concentration: 5% (weight/weight in water)

**Fixed Costs**

- ▶ Cost of 24-frame filter press (second-hand) ----- \$10,000/filter press
- ▶ Installation (piping and solids handling plant) ----- \$40,000/filter press
- ▶ Slurry feed pump(s) ----- Students specify flowrate and get capital cost by asking local suppliers
- ▶ Overheads (independent of number of filter presses) ----- \$200,000/year

**Variable Costs**

- ▶ Filter aid ----- \$700/ton
- ▶ Running costs ----- \$100/24-hour day per filter press
- ▶ Labor (all-inclusive costs to TRAMPL)
  - for day shift, 7:30 am to 3:30 pm ---- \$24/hour per laborer
  - for night shifts and weekends ----- \$36/hour per laborer

**Financial workup**

- ▶ Estimated project life ----- 5 years
- ▶ Value of solids recovered ----- \$1,000/dry ton
  - Assume 10% interest rate and 10% rate of inflation
  - Assume that equipment is totally written off after 5 years

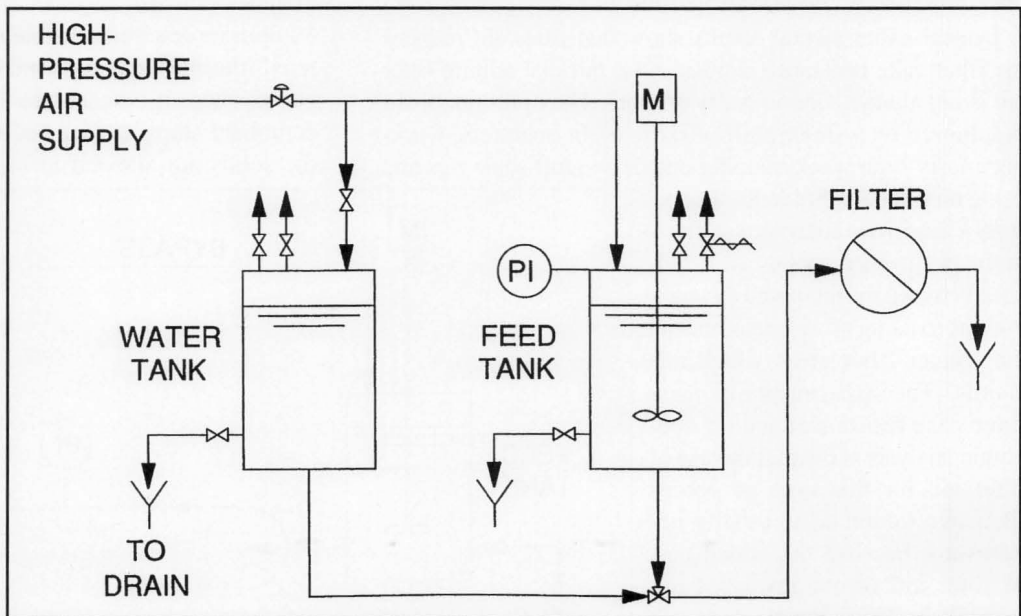


Figure 1. Schematic diagram of laboratory test filter plant.

flowrate. Students carry out most of the experimental trials with this equipment. There is limited access to a full-size plate-and-frame filter press (see Figure 2) which can be operated at constant flowrate for testing, although the full-scale filter is to be designed for constant pressure using a centrifugal slurry pump to deliver the feed. This situation simulates the case where a company laboratory is modestly furnished with small-scale equipment and there is limited access to a working large-scale filter which is available, say, at a neighboring company. It is possible to do a few quick but not elaborate measurements on the large-scale gear such as total frame volume and the maintenance time, *e.g.*, the time taken to dismantle the press, remove the solids, and return the press to service.

We direct students to Volume 2 of Coulson, Richardson, Backhurst, and Harker<sup>[1]</sup> for filtration theory, and to Sinnott<sup>[2]</sup> for the discounted cash flow analysis which is required for the economic analysis. Students are encouraged to decide for themselves which theories need to be used and to describe them sufficiently well so that the reader can follow the arguments presented. Typically, they choose to perform four runs at filter-aid dosages of 0, 1%, 2%, and 3% of the dry solids concentration. For a uniformly formed cake of constant specific resistance, a plot of  $t/V$  versus  $V$  (where  $t$  is cumulative time and  $V$  is cumulative filtration volume) produces a straight line. The slope of this line is proportional to the specific cake resistance and the intercept is proportional to the medium resistance. The expression for specific cake resistance contains the term  $1/A^2$  (where  $A$  is the filter area), and the expression for medium resistance contains the term  $1/A$ . Finding the working resistances for the full-scale filter is then easily done by factoring in the appropriate ratio of areas of the small-to-large scale filters.

Typical experimental results show that filter aid reduces the filter cake resistance significantly, but that adding filter aid is not always economically optimal. The optimum profit is achieved by a single shift of six to eight operators, working a forty-hour week, running one or two full-scale rigs and using no filter aid. Students observe from a sensitivity analysis that maximum profit corresponds to a balance between the increased throughput due to using more operators and the greater labor costs which this entails. The experimentally measured cake resistances and the economic analysis show that the use of filter aid for this type of porous filtercake (alumina) is usually unnecessary because the added cost of filter aid is not rewarded adequately by lower capital and labor costs.

Conceptual mistakes can lead to absurd results. The full-scale filter press is designed to hold twenty-four frames for normal operation. For experimental scale-up purposes, we have it set up with only two frames. Students may forget that the installed working area of this filter is twelve times the filter area of two frames. If the rest of their analysis is correct, they will discover that not one or two filter presses but twelve or twenty-four are required!

To complete the exercise successfully, the technical and economic analysis must be reported succinctly in a self-contained "Summary." Managers need to be able to read a summary and get an immediate idea of what the report is about and of what use it is to their company. A frequent oversight is to omit the aim and purpose of the laboratory exercise and launch immediately into the numerical results. Students know that the academics who mark their work are totally familiar with the experiments, and some of them lose sight of the need to write for a readership which may be unfamiliar with those experiments. Although it is frequently only one-half of a page of writing, we emphasize that the summary is the most difficult part of any report to write well and it deserves a significant effort.

## LEACHING

Students are presented with a simulated commercial problem in the minerals-processing industry involving optimization. There is a virtually unlimited supply of low-grade ore containing a valuable solute, and a three-stage countercurrent leaching plant of fixed maximum capacity with which to extract this solute. The design and performance of this model plant have been previously described.<sup>[3]</sup> Feed consists of a slurry of inert material, usually 20% by wt. of PVC granules in water, together with a dye (fluorescein) at 50 to 80 ppm representing the valuable solute. This slurry is metered into the first-stage mixer via a positive-displacement pump where it contacts the overflow from stage two. The combined slurry is then fed to the first-stage settler where the solids are allowed to settle, leaving as underflow and

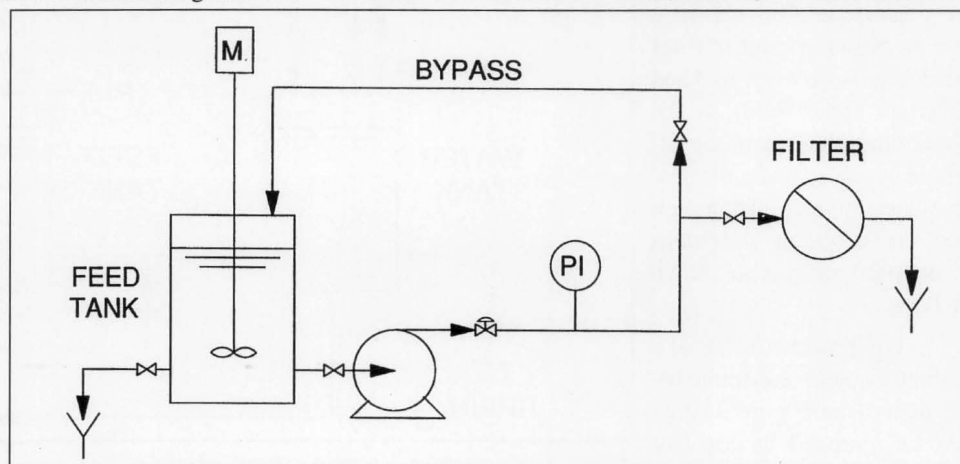


Figure 2. Schematic diagram of full-scale filter press plant.

producing an overflow which is the concentrated solution product stream. The underflow from stage one is the feed to the stage-two mixer, which contacts the stage-three overflow, etc. Make-up water is fed into the third-stage mixer and the well-washed inert solids leave as underflow from the third-stage settler. Dye concentrations are determined spectrophotometrically and flowrates are determined by volumetric apparatus and stopwatch.

As in nearly all separation technology (of which this is just one example) there is a compromise between "recovery" and "quality" of the valuable product. At low make-up water flowrates the product solution is obtained at relatively high concentration, but its flowrate is small and total recovery of the chemical is relatively low. As make-up water flowrate increases, the product solution concentration falls—but the total recovery increases asymptotically to 100%.

To make this laboratory experience more like the real thing, economic criteria are given to the students describing

- The value of the dry solid chemical
- The cost of processing the product solution (by evaporation, for example)
- The cost of power, maintenance, and labor
- The cost of treating unrecovered chemical, since it may be an undesirable burden on the environment if it is dumped as landfill, for instance.

The equations for income and costs have been arbitrarily defined. Overall profit is simply income minus costs. Thus

$$I = Q(10 - 100/C_p)$$

where

$$I = \text{income (\$/hour)}$$

$Q$  = dye production rate (gram/hour)

$C_p$  = dye concentration in product stream (ppm)

This equation gives a steadily falling income, even becoming negative, as the product stream becomes more dilute. This reflects the greater costs of subsequent processing. There are penalty costs for disposal or treatment of unrecovered dye and a flat-rate running cost which applies regardless of dye recovery. These costs are represented by

$$C = 2 + 2(C_u)(V_u)$$

where

$C$  = Total running costs (\$/hour)

$C_u$  = concentration of dye in underflow (ppm)

$V_u$  = solution flowrate in underflow (m<sup>3</sup>/hour)

The constant \$2/hour is the flat-rate running cost and the coefficient 2 is the treatment cost at \$2/gram of dye.

The object of the laboratory exercise is to operate the leaching process in such a way as to maximize the profit. Students must identify the key parameter to be varied (most importantly, the water flowrate and the underflow solids concentration) and the range of operation to find the maximum profit most efficiently. Students need to set a number of run conditions, operate the laboratory plant to achieve steady state, and perform mass balances to show that the data can be relied upon before determining peak profitability.

In order to estimate the optimal experimental range, we ask students to write a mathematical model of the simpler case of a perfectly mixed single-stage plant which is conveniently done using a spreadsheet calculation. In this case, the dye-containing slurry is mixed with water and the mixture is allowed to separate into an underflow (UF) fraction containing all of the solids together with some solution and an overflow (OF) fraction containing dye solution only. Table 2 shows a typical result with make-up water flowrate varying between 0 and 50 liters/hour. As can be seen, the addition of water steadily reduces the dye concentration such that the dye lost in the UF decreases. Although the dye concentration in the OF also decreases, the total recovery of dye increases. As an internal control of the calculation, a very large value of make-up water, *e.g.*, 1000 liters/hour, shows that recovery approaches 100% as expected. Using our arbitrary formulae for incomes and costs it can be seen that

- Income is at a peak at 20 liters/hour of make-up water flow
- Costs steadily decrease

**TABLE 2**  
**Economics of Single-Stage Leaching Plant**

Make-up Water Flowrate (liters/hour)	Dye Conc'n. (ppm)	Dye in UF (g/h)	Dye in OF (g/h)	Recovery (%)	Income (\$/hour)	Costs (\$/hour)	Profit (\$/hour)
0	60.00	0.785	0.94	55	7.85	3.57	4.28
5	51.12	0.669	1.06	61	8.52	3.34	5.18
10	44.54	0.583	1.14	66	8.88	3.17	5.71
15	39.45	0.516	1.21	70	9.04	3.03	6.01
20	35.41	0.464	1.26	73	9.07	2.93	6.15
25	32.12	0.420	1.31	76	9.00	2.84	6.16
30	29.39	0.385	1.34	78	8.86	2.77	6.09
35	27.08	0.355	1.37	79	8.66	2.71	5.95
40	25.12	0.329	1.40	81	8.42	2.66	5.76
45	23.41	0.307	1.42	82	8.14	2.61	5.53
50	21.93	0.287	1.44	83	7.84	2.57	5.26
1000	1.68	0.022	1.71	99	-84.51	2.04	-86.55

- Profit is at a peak at 25 liters/hour of water flow

It now becomes interesting to compare the profits generated by the three-stage experimental rig to those generated by the single-stage theoretical prediction. The commercial justification of using a more complex and expensive three-stage rig is that it should create a more concentrated product dye solution and result in less dye lost in the underflow. We should expect therefore that the three-stage rig should return a greater profit and use less make-up water to do so, compared to the single-stage prediction. Results from laboratory experiments (see Figure 3) show that the three-stage rig returned a maximum profit of 14.1 \$/hour at a water flowrate of 23 liters/hour. The single-stage prediction returned corresponding values of 6.2 \$/hour and 25 liters/hour.

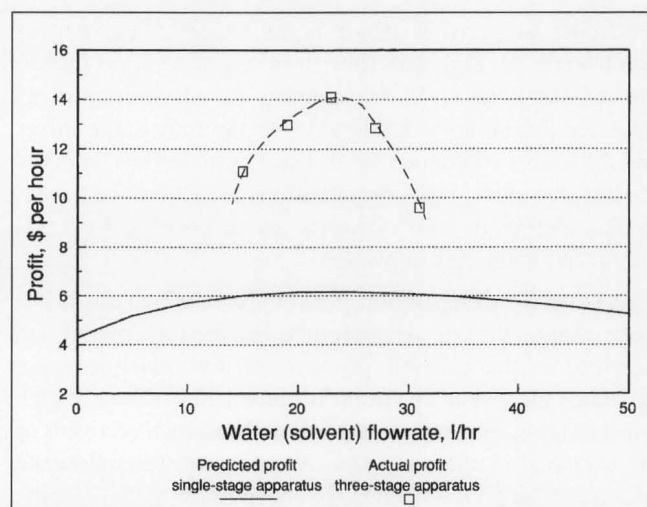
The overall mass balance around the process indicates the reliability of the measurements as well as the level of understanding students have for steady state operation. With careful operation and analytical measurement, discrepancies in the mass balances for both dye and solids should be less than 10%. Discrepancies of this magnitude are within the 95% confidence limits of the uncertainties in the experimental measurement of concentration (the largest contribution to error) and flowrate. From these mass balances students are encouraged to argue a case that their data indicate steady state operation with the flow of all streams accounted for, before they discuss the effects of experimental variables such as feed or water flowrate.

Students work in groups of two, taking turns to act as Group Leader. We assess students on their ability to design a set of experiments to achieve peak profitability most efficiently and convincingly. Emphasis is placed on the ability of the Group Leader to organize the work to be done on the day in question, and on the ability of both students to communicate the critical operation parameters in a concise form in their reports which are due two weeks after the date of the experiment.

The Unit Operations Laboratory is a core element of the undergraduate curriculum at the University of Sydney. At this stage of their studies, the third year of a four-year curriculum, they have had initial exposure to economic theory, but are not expected to perform elaborate economic analysis. Despite this, the introduction of economic relevance into the Unit Operations Laboratory has been accepted and even welcomed by the students.

## DISCUSSION

Putting a commercial flavor into the Unit Operations Laboratory has increased the perceived relevance to students as well as enhancing the engineering-science aspects of the work. By making the laboratory results "do something," we find that all students, whether proficient or of average ability, must understand what the relevant equations actually



**Figure 3.** Predicted profit from a single-stage mathematical model and actual profit from the three-stage plant.

mean and not just how to substitute blindly into them. With this emphasis there is a greater point in interpreting results critically. In particular, the analysis of experimental error, which has been hitherto a vague concept at best, is now given tangible importance as dollar figures depend on the outcome. Students perceive that their decisions in the design of the process may lead to unrealistic operating costs and that these decisions will be challenged by management. In this case they are encouraged to take the initiative and to seek better alternatives.

We use the commercial aspects of the laboratory to promote a more responsible attitude toward experimental work and report writing. In the past we have been aware of the students' tendency not to analyze data critically. Lines of best fit might be drawn through a random scatter of data points, and data which do not fit some pet theory might be conveniently ignored. Values of profit, equipment sizes, transfer coefficients, etc., might be reported with four or five significant figures when the primary data have two.

Just as "real life" managers might react, we are especially critical of reports which disown responsibility for students' work, with statements such as "...there were leaks in the rig which did not allow precise analysis of results..." or "...owing to lack of agreement between theory and experiment, additional work will be required..." or "...there were many errors in this work, the major one being human error and to a lesser extent experimental error...". We tell students that a professionally written report presents a case to the reader that the results are reliable within certain confidence limits. Including a list of perceived sources of error, as shown in the last example, with no explanation and no reassurance that the errors do not contradict the conclusions, spells instant self-disqualification. Our simulated management teams are not interested in excuses. This message is getting through;

students have increasingly accepted the challenge of being responsible for their work and they now feel more productive and stimulated. As a result, the addition of an economic flavor in the Unit Operations Laboratory has been well received by students who have appreciated its relevance to education leading to a commercial career.

## CONCLUSIONS

By providing a balance between an innovative commercial aspect and the traditional engineering-science aspect of the Unit Operations Laboratory, we have introduced elements of "real-life" into laboratory work. Solving problems which have the flavor of industry makes laboratory work more challenging and interesting and, we think, more relevant for the students.

In the future we intend to extend the approach beyond the two experiments discussed here and involve the majority of the Unit Operations Laboratories. Most experiments can be augmented readily, according to the same principles used with our leaching and filtration experiments, by 1) defining profitability equations for an existing plant or process and seeking an economic optimum, or 2) defining a scale-up problem and using laboratory data to predict the economics of a full-scale plant and process.

## REFERENCES

1. Coulson, J.M., J.F. Richardson, J.R. Backhurst, and J.H. Harker, *Chemical Engineering. Volume 2: Unit Operations*, 3rd ed., Chapter 9 (Filtration) and Chapter 10 (Leaching); Pergamon Press, Oxford, England (1978)
2. Sinnott, R.K., *Chemical Engineering. Volume 6: An Introduction to Chemical Engineering Design*, Chapter 6 (Costing and Project Evaluation), Pergamon Press, Oxford, England (1983)
3. Davies, W.A., "A Three-Stage Counter Current Leaching Rig for the Senior Laboratory," *Chem. Eng. Ed.*, **23**(2), 96 (1989) □

## Heterogeneous Reaction Rate Data

*Continued from page 25.*

Figure 3 shows that  $p_{\text{SO}_3}$  is a linear function of  $p_{\text{SO}_2}$ , implying that these variables were actually not changed independently during the experiments. Thus, there is no way to separate the information in the reaction rate data related to these two variables. Plotting  $p_{\text{O}_2}$  versus  $p_{\text{SO}_3}$  gives similar results, indicating that there is also a linear dependency between these two variables.

## CONCLUSIONS

Initial results have shown that the reaction rate data of Table 1 can be represented well by the rate expression of Eq. (1). It is very tempting to jump to the conclusion that the experimental data verifies the mechanism postulated in Eq. (1), but using numerical and statistical analysis of the data,

we have proven that such a conclusion is completely groundless because

1. The data itself is not experimental, but extrapolated, the accuracy of which is impossible to assess.
2. Because of the large value of the equilibrium coefficient and limited accuracy of the reaction rate values, no effect of the reversibility can be detected in the reaction rate values. As a result, assuming irreversible reaction yields a more accurate correlation over that obtained with the reversible model.
3. The partial pressures of  $\text{SO}_3$ ,  $\text{SO}_2$ , and  $\text{O}_2$  were not varied independently during the experiments; there is linear dependency between the partial pressures of  $\text{SO}_3$ ,  $\text{SO}_2$ , and  $\text{O}_2$ . As a result, it is impossible to discriminate between the effects of  $p_{\text{SO}_3}$ ,  $p_{\text{SO}_2}$ , and  $p_{\text{O}_2}$  on the reaction rate.
4. The equation

$$r = 1.62 \times 10^{-5} p_{\text{SO}_3}^{(-2.27)}$$

has been found to best represent the data in Table 1, but because of the limitations of the data that were mentioned earlier and the empirical nature of the power-law rate expression, there is absolutely no certainty that this rate expression is applicable for other combinations of partial pressures.

We have used one particular example to demonstrate several potential pitfalls in correlation of experimental data. It can be expected that in most practical cases not all these pitfalls will show up; but these four points can serve as guidelines in assessing the quality of the data, the accuracy of the correlation, and the adequacy of a model to represent the data.

In conclusion, it is appropriate to quote Churchill, who noted that "...if the observed behavior of the process requires the use of a more complex model than the data justifies, resolution lies in the laboratory rather than in further analysis."

## REFERENCES

1. Churchill, S.W., *The Interpretation and Use of Rate Data: The Rate Concept*, revised printing, Hemisphere Publishing Co., Washington, DC (1979)
2. Smith, J.M., *Chemical Engineering Kinetics*, 3rd ed., McGraw-Hill, New York, NY (1981)
3. Olson, R.W., R.W. Schuler, and J.M. Smith, "Catalytic Oxidation of Sulfur Dioxide: Effect of Diffusion," *Chem. Eng. Prog.*, **46**(12), 614 (1950)
4. Shacham, M., and M.B. Cutlip, *POLYMATH 3.0 Users' Manual*, CACHE Corporation, Austin, TX (1993)
5. Brauner, N., and M. Shacham, "Model Discrimination and Error Analysis in Heterogeneous Rate Data Regression for Irreversible Reactions," submitted for publication (1993) □