PERSONALIZED, INTERACTIVE, TAKE-HOME EXAMINATIONS

For Students Studying Experimental Design

WILLIAM A. JACOBY
University of Missouri • Columbia, MO 65211

In this day and age, many chemical engineers seek jobs traditionally filled by engineers from other disciplines, and the chemical engineering curriculum, particularly electives, can help enhance their prospects in that respect.[1] One crosscutting skill set that facilitates this trend is expertise in statistical methods.[2] Employers particularly value knowledge of the techniques of experimental design and quality control.[3,4]

The University of Missouri-Columbia's Department of Chemical Engineering offers a three-semester-hour course called "Experimental Design and Statistical Quality Control for Chemical Engineers." It is the most popular undergraduate elective, perhaps because it can be taken in lieu of a required course in probability and statistics offered in the College of Arts and Sciences. Graduate students, who must complete an additional semester project, also take the course.

The examinations described in this article are personalized and interactive in the sense that the students are allotted a prescribed number of experiments. Using a sequential approach in which some fraction of the experimental budget is expended in the first submission, each student submits a carefully formatted table of experimental conditions (factor-levels for each of the variables under consideration). The instructor uses a computer model that includes a random error term as a virtual laboratory to efficiently generate a unique data set for each submission. After interpreting the data from the first set of experiments, the student submits additional experiments and receives additional sets of unique data until his or her experimental budget is expended. The appropriate set of experimental designs must be combined with accurate calculations and insightful analysis to arrive at "the truth,"

William A. (Bill) Jacoby *received his PhD from the University of Colorado in 1993. He worked as a research engineer at the National Renewable Energy Laboratory until 1997 when he joined the faculty at the University of Missouri-Columbia. His research interests include photocatalysis, thermal catalysis, and biotechnology.*

TABLE 1
List of Topics in "Experimental Design and Statistical Quality Control for Chemical Engineers"

1. Normal distribution and the central limit theorem
2. Statistical quality control: creating, maintaining, and interpreting SQC charts
3. Statistical quality control: rational subgroups and interpretation
4. Significance testing
5. Z distribution
6. t distribution
7. Statistical dependence
8. Random sampling
9. Randomization
10. Blocking
11. Confidence intervals
12. Inferences about variances
13. Error propagation
14. Comparing more than two treatments
15. Empirical and theoretical models
16. Analysis of variance
17. Multiple comparisons
18. Randomized blocks with replication
19. Designs with more than one blocking variable
20. Balanced incomplete blocked designs
21. Full factorial designs
22. Interpreting the results of full factorial experiments
23. Determining significance of effects in factorial experiments
24. Applications of statistical quality control
25. Partial factorial designs
26. Design resolution
27. Confounding patterns
28. Sequential design of experiments; additional runs
29. Analysis of Residuals
30. Parsimony in empirical models
31. Linear regression
32. Nonlinear regression

TABLE 2
Problem Statement

You have accepted a job at Cavitron, a small start-up company. Cavitron is attempting to commercialize a turn-key, skid-mounted "pump-and-treat" system for use in oxidizing the organic and chlorinated organic compounds in aqueous mixtures.

Hydrodynamically induced cavitation is the operating principle for the treatment device, which is referred to as a "jet reactor." When polluted water is pumped at high pressure and high velocity through an appropriately designed nozzle and around an appropriately designed obstruction, microscopic bubbles form and implode in the fluid. Local temperatures reaching 800°C and local pressures in excess of 5,000 psi accompany the formation and implosion of the bubbles. Organic vapors predominate (relative to water vapor) in the bubbles. In the presence of dissolved oxygen and other oxidative species, as well as a miscible fluid catalyst (with appropriate vapor pressure), each bubble is a microreactor in which some fraction of the organic vapor is oxidized.

Your first project for Cavitron is to set up and operate a skid-mounted system for treating the leachate from a hazardous waste landfill. You will draw polluted water from the containment pond, treat it, and pump it back into the pond. Since each waste stream is different, the operating conditions for this application must be optimized. The response to be optimized is single-pass conversion (treatment efficiency). Table 3 lists seven standard process variables routinely evaluated at each installation. Factor level settings that experience has shown are in the proper experimental spaces are also provided. Your first task involves determining the effect of these seven "standard" process variables on treatment efficiency.

The Research and Development Department would also like you to evaluate four experimental modifications to the jet reactor. Field data is essential to verify laboratory results. At some point during your experimental campaign, you are to install the experimental modifications and proceed with testing. Table 3 also lists these experimental modifications (variables) and their factor level values. Your second task involves evaluating the effect of these experimental variables of treatment efficiency.

Your tasks are tabulated more specifically below.

Task 1a: Determine the sign and magnitude of the significant main effects and interactions of the standard process variables on the treatment efficiency of the unit.
Task 1b: Formulate an empirical model and evaluate its validity.
Task 1c: Recommend operating settings for these seven variables.

Task 2a: Determine the sign and magnitude of the significant main effects and interactions of the experimental modifications on the treatment efficiency of the unit.
Task 2b: Appropriately modify your empirical model from Task 1b and evaluate its validity.
Task 2c: Make recommendations about whether these four modifications should be adopted in future production units.

Time and budget constraints will allow you to perform 24 experiments. These may be submitted in whatever increments you choose over the next five days. Submit your sets of experimental conditions electronically and you will receive your data via return e-mail.

TABLE 3
Standard Variables and Experimental Variables/Modifications and Factor Levels

Standard Variables and Factor Levels

Symbol	Description	- Level	+ Level
P	Pressure in the nozzle	2000 psi	3000 psi
L	Length of the pretreatment capillary	10 m	20 m
T	Temperature of the pretreatment capillary	25°C	70°C
C	Concentration of the catalyst	0.05 M	0.10 M
A	Angle of the obstruction	0°	5°
D	Diameter of the obstruction	5 cm	8 cm
X	Distance between nozzle and obstruction	0.5 mm	0.75 mm

Experimental Variables and Factor Levels

Symbol	Description	- Level	+ Level
S	Supersaturated oxygen	Off	On
K	Catalyst type	Standard	Experimental
O	Ozone generator	Off	On
N	Nozzle design	Standard	Experimental

an accurate estimate of the parameters of the model used to generate the data.

COURSE STRUCTURE

The latest rendition of the course (spring semester, 2002) met for 50-minute sessions on Mondays, Wednesdays, and Fridays for fifteen weeks. Table 1 lists the topics discussed. They were selected to provide a practical statistical toolbox to chemical engineers in research, process engineering, and manufacturing.

The availability of computational tools, principally a personal computer and associated software, has allowed an increase in the complexity of calculations presented in chemical engineering classes, as well as in the homework assignments. In this class, most lectures (as well as all examples and homework solutions) were performed using the Excel™ spreadsheet program. These spreadsheets were made available, at the appropriate time, to the students via e-mail. This allowed the use of a relatively old but well-written and classic text that does not explicitly employ computer techniques or software.[5] Fortunately, on Mondays and Wednesdays the course met in a computer lab where each student had access to a computer. The use of a computer lab during class, however, is not required in the administration of this type of examination.

DESCRIPTION OF THE EXAMINATION

It is difficult to give a comprehensive examination in a computationally intensive course when there are constrictions of class duration and/or access to computers in the classroom. Most chemical engineering examinations are completed during a single class period without the aid of computers. The availability of a computer lab does not circumvent the time constraint. The challenge for the instructor under these circumstances is to write an exam that promotes learning, discriminates among the students, and is consistent with the course content and homework.

Take-home examinations are an attractive option, but raise another problem: academic dishonesty. Although the percentage of students who collaborate improperly on take-home examinations is small, there is an opportunity for a minority to gain an unfair advantage. A take-home exam in which each student has a unique data set generated from a model including a random-error term eliminates the opportunity for one student to copy another's work. The use of several different models to generate the students' data sets provides a further obstacle to dishonest collaboration, but must be accounted for during record-keeping and grading.

Table 2 is the problem statement from a personalized, interactive, take-home examination based on this concept. Prior to the class in which it was presented, an electronic version was e-mailed to each of the students as a worksheet in an Excel spreadsheet. This spreadsheet also included a worksheet containing Table 3, which includes the standard variables and the experimental variables/modifications as well as their factor-level settings. Also included was an abbreviated version of Table 4 (no factor levels, data, etc.), which was formatted for submission of experiments.

An individual student has a budget of 24 experiments. For a particular experiment, the model shown as Eq. (1) generates a data point:

$$y = I + \frac{P}{2}X_P + \frac{T}{2}X_T + \frac{D}{2}X_D + \frac{S}{2}X_S + \frac{O}{2}X_O + \frac{S \times O}{2}X_{S \times O} + \varepsilon \quad (1)$$

where y is the response, the single-pass conversion (%), and I is the overall average response (I = 15%). The X-variables (X_P, X_T, X_D, X_S, X_O) have a value of -1 for the experiments in which the indexed variable is set at the minus level and +1 for the experiments in which the indexed variable is set at the plus level. X_{SxO} is the factor level of the interaction between the S variable and the O variable, and its value is the sign of their product. The magnitudes of the main effects and interactions used in the model to generate the data are shown in Table 5. The student chooses the values of all 11 variables for each experiment. The variables that are not included in the model used to generate the data set (L, C, A, X, K, N) are inert.

Equation 1 is an empirical model used to interpret data from factorial experiments. Theoretical models can also be appended with error terms to generate unique data sets for take-home examinations in core subjects such as thermodynamics and transport phenomena. More empirical curricula (*e.g.*, kinetics) are even more amenable to the technique.

The student submits a total of 24 experiments via e-mail over a period of five days. Most students submitted three sets of eight experiments each. It took about two minutes to open an e-mail, open the experimental design, insert the student's input into the model to generate a data set, save the data set, attach it to a return e-mail, and send. For example, if a class had 20 students, they would request 60 data sets, requiring the instructor to spend two hours generating data. The data generation process could be easily automated. The time required to write and grade this exam is similar to a conventional exam.

Based on the individualized data sets, the student must determine which of these variables has a significant effect on

TABLE 5
Summary of Results

Main Effect/ Interaction	*Model Parameter*	*Estimate from Data*	*Error*	*Recommended Setting*
P	2.0	2.1	5%	+
L	—	—	—	-
C	—	—	—	-
T	-1.5	-1.8	-23%	-
A	—	—	—	-
D	-1.5	-1.7	-16%	-
X	—	—	—	-
S	3.0	2.9	4%	+
K	—	—	—	-
O	2.0	2.5	27%	+
SxO	3.0	3.0	0%	NA

TABLE 4
Summary of Experimental Campaign

	Standard Process Variables							Exp. Variables						
Exp. #	**P**	**L**	**C**	**T = PxL**	**A = PxC**	**D = LxC**	**X = PxLxC**	**S**	**K**	**O**	**N**	**Single Pass Conversion (%)**	**Empirical Model Prediction (%)**	**Residual (%)**
1	-1	-1	-1	1	1	1	-1	-1	-1	-1	-1	11.8	11.1	0.7
2	1	-1	-1	-1	-1	1	1	-1	-1	-1	-1	14.8	15.0	-0.2
3	-1	1	-1	-1	1	-1	1	-1	-1	-1	-1	13.9	14.6	-0.7
4	1	1	-1	1	-1	-1	-1	-1	-1	-1	-1	15.5	14.9	0.6
5	-1	-1	1	1	-1	-1	1	-1	-1	-1	-1	13.2	12.8	0.4
6	1	-1	1	-1	1	-1	-1	-1	-1	-1	-1	16.4	16.7	-0.4
7	-1	1	1	-1	-1	1	-1	-1	-1	-1	-1	12.6	12.9	-0.4
8	1	1	1	1	1	1	1	-1	-1	-1	-1	13.4	13.2	0.2

Variable	**Main Effect (%)**	**Abbreviated Confounding Pattern**
P	**2.2**	**P + LxT + CxA + DxX**
L	-0.2	L + PxT + CxD + AxX
C	-0.1	C + PxA + LxD + TxX
T	**-1.0**	**T + PxL + AxD + LxX**
A	-0.1	A + PxC + TxA + PxX
D	**-1.6**	**D + LxC + TxA + PxX**
X	-0.2	X + CxT + LxA + PxD
Average =	**13.9**	

Exp. #	**P = SxK**	**L**	**C**	**T = SxO**	**A**	**D = KxO**	**X**	**S**	**K**	**O**	**N = SxKxO**	**Single Pass Conversion (%)**	**Empirical Model Prediction (%)**	**Residual (%)**
9	1	-1	-1	1	-1	1	-1	-1	-1	-1	-1	13.4	13.4	0.0
10	-1	-1	-1	-1	-1	1	-1	1	-1	-1	1	12.6	13.0	-0.4
11	-1	-1	-1	1	-1	-1	-1	-1	1	-1	1	12.9	13.0	-0.1
12	1	-1	-1	-1	-1	-1	-1	1	1	-1	-1	16.2	16.8	-0.6
13	1	-1	-1	-1	-1	-1	-1	-1	-1	1	1	16.4	16.4	0.0
14	-1	-1	-1	1	-1	-1	-1	1	-1	1	-1	18.7	18.4	0.3
15	-1	-1	-1	-1	-1	1	-1	-1	1	1	-1	12.2	12.6	-0.4
16	1	-1	-1	1	-1	1	-1	1	1	1	1	18.5	18.8	-0.3

Variable	**Main Effect (%)**	**Abbreviated Confounding Pattern**
P	2.0	**P + SxK + TxD + OxN**
T	1.5	**T + PxD + SxO + KxN**
D	-1.9	**D + PxT + SxN + KxO**
S	2.8	**S + PxK + TxO + DxN**
K	-0.3	K + PxS + TxN + DxO
O	2.7	**O + PxN + TxS + DxK**
N	-0.1	N + PxO + TxK + DxS
Average =	**15.1**	

Exp. #	**P**	**L**	**C**	**T**	**A**	**D**	**X**	**S**	**K**	**O**	**N**	**Single Pass Conversion (%)**	**Empirical Model Prediction (%)**	**Residual (%)**
17	1	-1	-1	-1	-1	1	-1	-1	-1	-1	-1	15.4	15.2	0.2
18	1	-1	-1	1	-1	1	-1	-1	-1	-1	-1	13.3	13.4	-0.2
19	1	-1	-1	-1	-1	1	-1	1	-1	-1	-1	15.3	15.1	0.2
20	1	-1	-1	1	-1	1	-1	1	-1	-1	-1	13.3	13.3	-0.1
21	1	-1	-1	-1	-1	1	-1	-1	-1	1	-1	14.5	14.7	-0.2
22	1	-1	-1	1	-1	1	-1	-1	-1	1	-1	12.8	12.9	-0.1
23	1	-1	-1	-1	-1	1	-1	1	-1	1	-1	20.4	20.6	-0.2
24	1	-1	-1	1	-1	1	-1	1	-1	1	-1	18.9	18.8	0.1

Variable or Interaction	**Main Effect (%)**
T	**-1.8**
S	**3.0**
O	**2.4**
TxS	0.1
TxO	0.2
SxO	**3.0**
TxSxcO	0.0
Average =	**15.5**

the single-pass conversion and which are inert. The sign and magnitude of the significant main effects must also be determined. Further, any significant interactions among the standard variables must be identified and their signs and magnitudes estimated. The student must also formulate an empirical model and evaluate its validity and recommend operating settings for these seven variables. Finally, each student must similarly assess the effects and interactions of four additional experimental variables of interest to the Research and Development Department.

Performing a full factorial experiment with eleven variables would require 2,048 experiments. As the experimental budget is about 1% of this amount, the use of highly fractionated partial factorial designs is required.

SOLUTION TO THE EXAMINATION

The first step in one of many effective solution strategies is to design and perform a 2^{7-4}_{III} partial factorial experiment focusing on the standard process variables. This is a resolution III "main effects" design because it estimates the main effects subject to a confounding pattern including two-way interactions. Aspects and advantages of this type of design are discussed in the course textbook.[5]

The first eight experiments shown in Table 4 prescribe this design. Pressure in the nozzle (P), length of the pretreatment capillary (L), and concentration of catalyst (C) are taken as the "live" variables. Their factor levels are assigned in standard order, as they would be for a 2^3 full factorial experiment.

The four remaining variables in the standard process variable set are temperature in the pretreatment capillary (T), angle of obstruction (A), diameter of obstruction (D), and distance between the nozzle and the obstruction (X). The levels of these variables are set according to the four combinations of interactions possible among the three live variables (*i.e.*, T=PxL, A=PxC, D=LxC, and X=PxLxT). Since all of the possible interactions among the three live variables were used as aliases for the additional variables, the design is referred to as fully saturated. The experimental variables/modifications are held at the minus (standard or unmodified) level for the first set of experiments.

Eight experiments were performed—therefore, eight parameters (the average and the seven main effects) can be estimated from the data. Each main effect is subject to confounding by fifteen other interactions. An abbreviated confounding pattern, including only the confounding two-way interactions, is also shown in Table 4. The data in the column headed "Single-Pass Conversion (%)" were generated using the model shown in Eq. (1).

Quantitative methods of determining significant effects are discussed in the course text[4] and will not be covered here. Examination of Table 4 reveals that the first eight experiments correctly indicate that P, T, and D may be important variables, while the remaining standard process variables (L, C, A, and X) may be relatively inert.

Personalized, interactive, take-home examinations are not subject to the constraints of class duration and availability of computers . . . they can be more complex and thorough.

After evaluating the first set of experiments, the principle of sequential design of experiments must be practiced in the second design. This solution strategy involves another set of eight experiments, shown as experiments 9 through 16 in Table 4. In this design, the intent is to begin investigation of the experimental variables/modifications, while confirming and improving the estimates of the three standard variables judged to be significant. The experimental modifications/variables supersaturated oxygen (S), catalyst type (K), and ozone generator (O) are the live variables in a second 2^{7-4}_{III} partial factorial experimental design. The alias for the final experimental variable/modification, nozzle design (N), is the three-way interaction among the live variables (N=SxKxO). This design is also fully saturated in that the remaining three possible interactions among the live variables are used as aliases for the three variables judged to be significant during the first set of experiments (P=SxK, T=SxO, D=KxO). Table 4 also includes the data for these experiments, the abbreviated confounding pattern, and the parameter estimates based on the data.

The parameter estimates show that the experimental variables/modifications S and O may be significant, while K and N may be inert. Further, the estimates of the standard variable parameters P and D are confirmed. These estimates are subject to entirely different confounding patterns, lending credence to the assumption that it is these main effects and not their confounding two-way interactions that are significant.

The temperature variable, T, however, is a different matter. While both the first and second sets of experiments resulted in estimates of similar magnitude, the sign changed. This suggests the presence of a significant interaction. Careful examination of the abbreviated confounding patterns for both the first and second sets of experiments reveals that an interaction between S and O is the most likely candidate, as both are significant variables whose interaction has not been previously aliased to an inert variable. Therefore, the final eight experiments in the experimental budget are expended performing a 2^3 full factorial experiment using variable T, S, and O. This design has the advantage that all interactions are explicitly estimated. As shown in Table 4, this design provides an unambiguous estimate of the T effect, confirms and refines the estimate of the S and O effects, and reveals an important two-way interaction oxygen supersaturation and ozone generation (SxO).

Table 5 summarizes the information gleaned from the experimental campaign and compares it to the actual parameters of the model used to generate the data. Three of the standard process variables were found to be significant, while the other four were determined to be inert. Two of the four experimental variables/modifications were determined to be significant, while the other two were inert. For all eleven variables, these determinations were correct (in agreement with the model used to generate the data). Further, the signs of all the effect and the interaction were also correct and the magnitudes were accurate between +/-30%. A column of recommended settings is also included in Table 5. For the inert variables, decisions about the settings are based on what might be expected to be easiest and cheapest.

Two empirical models can be developed from the data. The first

$$y = 13.9 + \frac{2.1}{2} X_P + \frac{-1.8}{2} X_T + \frac{-1.7}{2} X_D \tag{2}$$

predicts the single-pass efficiency of the jet reactor in its standard configuration (unmodified, all experimental variables/modifications at the minus level) as a function of the three significant standard variables. This model was used to generate the predicted values of the single-pass efficiency for the first eight experiments in Table 4.

The second model

$$y=15.3+\frac{2.1}{2}X_P+\frac{-1.8T}{2}X_T+\frac{-1.7}{2}X_D+\frac{2.9}{2}X_S+\frac{2.5}{2}X_O+\frac{3}{2}X_{S\times O} \tag{3}$$

predicts the performance of the jet reactor in its experimental configuration. It has a higher average and includes the S and O effects as well as their interaction. This model was used to generate the predicted values of the single-pass efficiency for the final sixteen experiments in Table 4.

Variables for both models are defined as in Eq. (1). Equation 3 is the experimental estimation of "the truth," as described by Eq. (1). Analysis of the residuals, tabulated in Table 4, was undertaken according to standard procedures and confirms the validity of the models.[5]

STUDENT FEEDBACK

An interactive learning environment was established and persisted throughout the week of the exam. This excitement was felt by both the students and the instructor. Table 6 shows the results of a feedback survey administered to the class. There were 19 respondents. The results document that a personalized, interactive, take-home examination is not only a good learning tool, but is also popular with the students. Three estimates of the central tendency are included to aid in interpretation.

TABLE 6
Summary of Anonymous Feedback Survey
(1 - Strongly Agree; 2 - Agree; 3 - Neutral; 4 - Disagree; 5 - Strongly Disagree)

	Avg.	Median	Mode
I understand the partial factorial experimental designs better as a result of this exam.	1.7	2	2
I understand the sequential nature of experimental design better as a result of this exam.	1.6	2	2
I like the individualized data concept.	2.0	2	2
I liked this exam.	2.2	2	2
I like take-home exams.	1.6	2	1
I lerned a lot while working on this exam.	2.1	2	2
This exam was a superior learning experience relative to the other exams for this class.	2.4	2	2
This exam was a superior learning experience relative to exams in other engineering courses.	2.6	3	3
I spent more time on this exam relative to the other exams for this class.	2.1	2	2
I spent more time on this exam relative to exams in other engineering courses.	2.6	3	2
This exam really sucked.	4.0	4	4

CONCLUSIONS

Personalized, interactive, take-home examinations are not subject to the constraints of class duration and availability of computers. Therefore, they can be more complex and thorough. Because a unique data set is generated for each student, the opportunities for dishonest collaborations are reduced. The use of several models to generate the students' data sets is a further barrier to cheating. Taking advantage of ubiquitous e-mail connectivity and the speed and storage capacity of modern personal computers, data generation and dispersal is expeditious.

The interactive aspects of the examination and the prescribed experimental budget allow a hands-on exploration of the concept of sequential design of experiments. Student feedback regarding the exam was favorable.

This type of examination can be adapted for use in other chemical engineering courses. In the future, elimination of the instructor interface during data generation will streamline the process.

REFERENCES

1. Cussler, E.L., "Do Changes in the Chemical Industry Imply Changes in Curriculum?" *Chem. Eng. Ed.,* **33**(1) (1999)
2. Fahidy, T.Z., "An Undergraduate Course in Applied Probability and Statistics," *Chem. Eng. Ed.,* **36**(2) (2002)
3. Moen, R.D., T.W. Nolan, and L.P. Provost, *Quality Improvement Through Planned Experimentation,* 2nd ed.,McGraw Hill, New York, NY (1999)
4. Montgomery, D.C., *Introduction to Statistical Quality Control,* 2nd ed., John Wiley & Sons, New York, NY (1991)
5. Box, G.E.P., W.G. Hunter, and J.S. Hunter, *Statistics for Experimenters,* John Wiley & Sons, New York NY (1973) ❐