# A REALISTIC EXPERIMENTAL DESIGN AND STATISTICAL ANALYSIS PROJECT

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The teaching of statistics can be one of the most challenging topics in the engineering curriculum. Students often find the subject matter abstract and the plethora of equations used in analysis rather confusing. For these reasons, an applied approach that emphasizes and reinforces how concepts presented in the statistics course can be used in the practice of engineering has been proposed.[1] An example is the use of the senior laboratory course to reinforce the concepts presented in the engineering statistics course.[2] A stronger emphasis on case studies and realistic problems of direct interest to engineering students is also suggested to help motivate and create a more positive attitude toward statistics[3] and engineering education in general.[4]

The statistical analysis project described in this article began as a reactor simulation for a senior design course project. It was later integrated into the professional development course, and, after a curriculum revision, the Applied Statistics course over the last five years. The novel aspects of this project are that the students are given a budget with which to perform their experimental study, and the experimental results are made available to the students one day after an experiment is requested. Although a process simulation is generating the experimental results, the intent is to mimic a realistic experimental study where results are not available immedi-

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ately and there is an economic limit imposed on the amount of information that can be obtained.

The pedagogical advantage of this approach is it requires students to efficiently plan and adjust their experimental data collection. A similar experimental design philosophy for a gas chromatography experiment is described in Reference 5. It also incorporates student data into the analysis exercise. The integration of data sets collected by students into the teaching of statistics as part of class projects and exercises has been widely advocated. The benefits of this integration are the incorporation of problem-based learning into the statistics course,[6] and the recognition that experimental data sets represent observations from a larger population distribution, which may yield different "answers" from a statistical analysis.[7] An important goal of any engineering statistics presentation is the appreciation that a single measurement does not represent the "true" value.[8]

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The approach in this article also avoids the "video game" syndrome that can occur in process simulation exercises. Although simulation modules can be very useful teaching and learning aids in chemical engineering education, they can also impart an exhaustive iteration approach to problem solving and a lack of appreciation for the true time-scale of real engineering processes. The addition of a cost and delay of simulation results in this project is intended to address this issue.

## EXPERIMENTAL ANALYSIS PROJECT OVERVIEW

In this project, the students determine the kinetic rate constants of both the forward and reverse reaction for the hydrolysis of ethylene to form ethanol.

$$C_2H_2 + H_2O \rightleftharpoons C_2H_5OH$$

The hydrolysis is a vapor phase reaction that is catalyzed by phosphoric acid supported on porous solid catalyst pellets. The reaction rate for the hydrolysis can be expressed as

$$R(A) = k_f P_E P_W - k_r P_A \qquad (1)$$

in which R(A) is the rate of formation of ethanol (gmol/lit–min), $k_f$ is the forward reaction rate constant (gmol/lit–min–bar$^2$), $k_r$ is the reverse reaction rate constant (gmol/lit–min–bar), and $P_E$, $P_W$, $P_A$ are the partial pressures (bar) of ethylene, water, and alcohol.

The students are told that they have a packed-bed tubular reactor available to carry out hydrolysis reaction experiments. They must specify the molar flow rates of the feed components, the outlet reactor pressure, and the average reactor temperature for each experiment. The molar feed rates of the reactants (steam and ethylene) and an inert gas (methane) may be varied by adjusting the corresponding flow controllers. Methane is supplied to the reactor in order to dilute the reacting species and prevent a runaway reaction. The average reactor temperature and reactor outlet pressure can also be varied by adjusting the

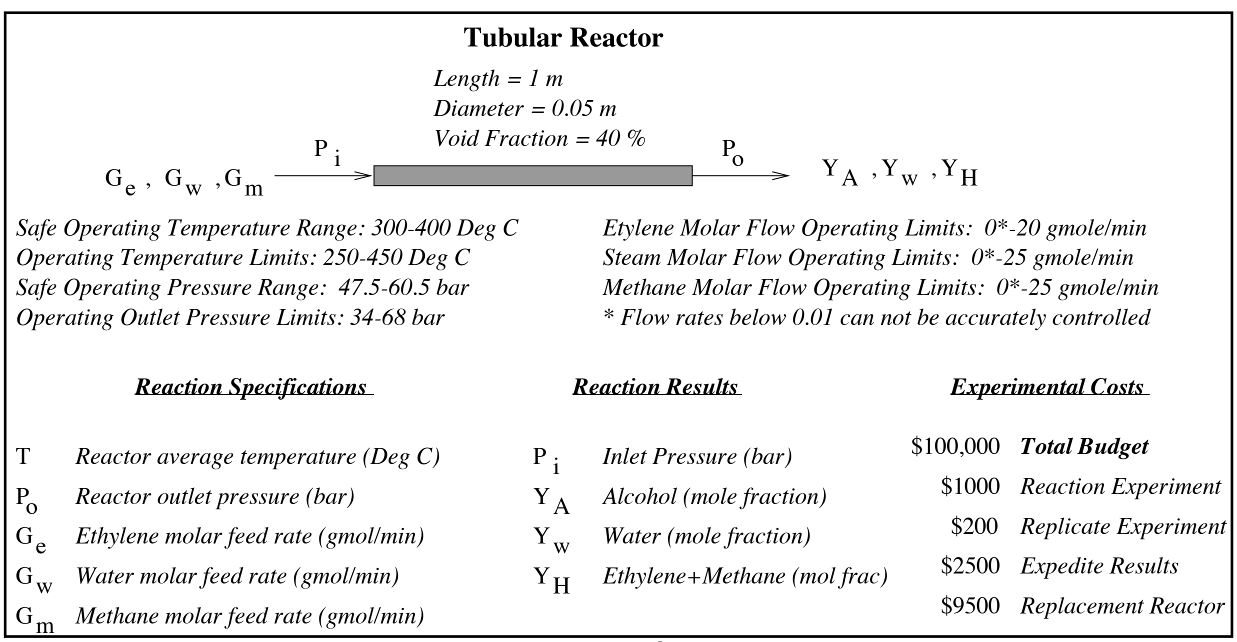

***Figure 1.*** *Experimental reactor system*

respective controllers. The reactor outlet gas stream is sampled and analyzed for alcohol fraction and hydrocarbon fraction (ethylene plus methane). Since water cannot be analyzed, it is determined by difference.

The students are given a feasible reactor temperature range of 300 °C to 400 °C and inlet pressure range of 45 to 65 bar. Under these conditions, the reactor can be safely operated. There is a potential, however, for the reactor to detonate due to an exothermic, runaway reaction at higher temperatures or pressures. The students are informed that temperatures beyond 400 °C and inlet pressures beyond 70 bar are dangerous and can very likely result in detonation of the reactor. Operation of the reactor with methane in the feed at the higher temperature and pressure range is also recommended. The students must therefore first determine safe operating conditions from initial experimental trials as discussed in the sequel.

The project is carried out in two- or three-person groups. Each student group is given a $100,000 budget to carry out the experiments necessary to determine the reaction-rate constants. Each experiment costs $1,000 for the initial run at a given set of operating conditions and $200 for each replicate run at the same conditions. The results from each experiment are made available the day after they are requested. An additional $2,500 cost is incurred in order to receive the results on the same day for each expedited experiment and replicate requested. Experiments can no longer be carried out when there are insufficient funds to cover the cost. If the chosen operating conditions cause the reactor to detonate, the students are charged $9,500 for a replacement. The intent of this aspect of the project is to illustrate that, as in an actual experimental study, there are consequences to poor experimental design choices. A schematic of the reaction system is presented in Figure 1.

## EXPERIMENTAL STUDY

The students are asked to determine the Arrhenius equation parameters, activation energy, and pre-exponential factor for the forward and reverse rate constants. They are also asked to verify that the rate constants follow the Arrhenius equation

$$k = k_o \exp\left(-E_a / RT\right) \tag{2}$$

over the feasible reactor temperature range where $k_o$ is the pre-exponential factor, $E_a$ is the activation energy, and T is absolute temperature. Both $k_o$ and $E_a$ can be determined by obtaining each rate constant at two or more temperatures and using the logarithmic transformation of Eq. (2)

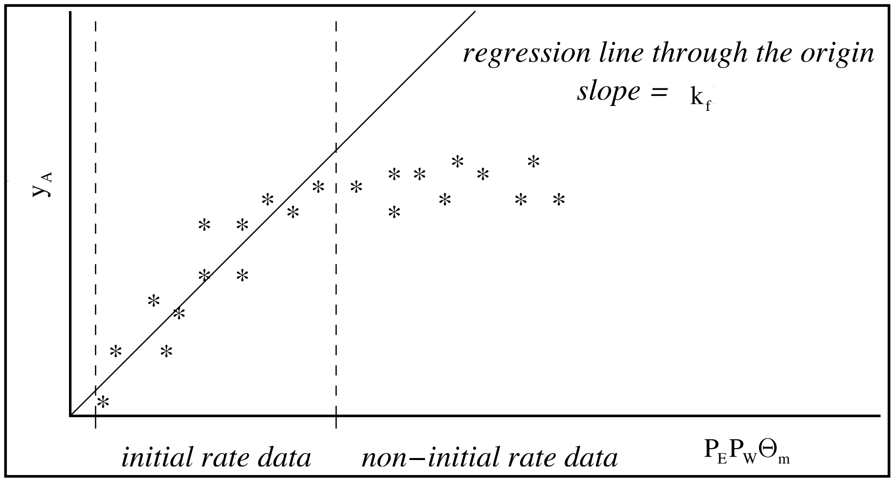


***Figure 2.*** *Example initial rate data regression.*

$$\ln k = \ln k_o - \frac{E_a}{R}\frac{1}{T} \tag{3}$$

where ln $k_o$ is the y-intercept and $-E_a/R$ is the slope of a linear regression of ln k as a function of 1/T. In order to determine the forward and reverse rate constants students must carry out two different types of experiments.

### Initial Rate Experiments

The initial rate method of measuring reaction rate constants is used to determine the forward reaction rate constant $k_f$. This technique makes the following assumptions: 1) there is so little product formed that the reverse reaction is negligible; and, 2) the conversion of the reactants is small enough that their concentrations may be taken as constant. Using these initial rate method assumptions with an ideal tubular reactor results in the following relationship for the outlet alcohol mole fraction

$$y_A = k_f P_E P_W \Theta_m \tag{4}$$

where $y_A$ is the mole fraction of alcohol in the exit gas, $k_f$ is the forward reaction rate constant, $P_E$ and $P_W$ are the partial pressures of the reactants, and $\Theta_m$ is the molar space time defined as

$$\Theta_m = V / F \tag{5}$$

in which V is the void volume of the reactor and F is the molar feed rate of gas entering the reactor.

Determination of the forward rate constant can be accomplished by noting that $y_A$ is directly proportional to the product $P_E P_W \Theta_m$ in Eq. (4) where the proportionality constant is $k_f$. A plot of $y_A$ vs. $P_E P_W \Theta_m$ should be a straight line through the origin with slope $k_f$. When $\Theta_m$ increases beyond the value where the initial rate method assumptions are valid, $y_A < k_f P_E P_W \Theta_m$ because the reverse reaction will begin to become significant. Therefore, one would expect the data to begin to deviate from a straight line when the initial rate method assumptions are no longer valid, as illustrated in Figure 2.

A value for the forward rate constant can be determined from the slope of a linear regression on the initial rate experimental data through the origin. The confidence interval on the rate constant is obtained from the confidence interval on the slope of the regression line.

### Equilibrium Experiments

If the reactor is operated at low enough feed rates, the reaction will reach equilibrium at the reactor outlet. The equilibrium constant for the reaction can then be determined from these experiments:

$$K_P = \frac{P_A}{P_e P_w} = \frac{y_A}{y_E y_W P_o} = \frac{k_f}{k_r} \qquad (6)$$

where $P_o$ is the reactor outlet pressure. The reverse reaction rate constant can be determined once the forward rate constant and the equilibrium constant are known from Eq. (6).

Determination of the equilibrium constant can be accomplished by noting that $y_A$ is directly proportional to the product $y_E\, y_W\, P_o$ in Eq. (6), where the proportionality constant is $K_P$. A plot of $y_A$ vs. $y_E\, y_W\, P_o$ should be a straight line with slope $K_P$. When $\Theta_m$ is below the value required for the reaction to reach equilibrium, $y_A < K_P\, y_E\, y_W\, P_o$. Therefore, one would expect the data to deviate from a straight line when the reaction is not at equilibrium, as illustrated in Figure 3.

The equilibrium constant can be determined from the slope of a linear regression on the equilibrium experimental data through the origin. The confidence interval on the equilibrium constant is obtained from the confidence interval on the slope of the regression line. The reverse rate constant is calculated from the ratio of the forward rate constant to the equilibrium constant at a given temperature.

## EXPERIMENTAL PROCEDURE

The students are instructed to select at least three temperatures to study. At each temperature, they are encouraged to perform exploratory experiments to determine the feed rate range that will give measurable initial rates and the feed rate range that results in equilibrium. Based on this information, a series of initial rate experiments to determine the forward rate constant and equilibrium experiments to determine the equilibrium constant should be conducted at different feed rates and compositions.

In order to carry out initial rate experiments, the reactor must be operated with high feed rates that result in low outlet alcohol concentration and low consumption of reactants. Although short residence times are necessary for the assumptions made by the initial rate method in Eq. (4) to be valid, the high flow rates will also result in high inlet reactor pressures due to pressure drop across the catalyst packed in the tube. Therefore, students are encouraged to initially obtain an estimate of the pressure drop at high flow rates. Class discussion is used to suggest that this analysis may be safely carried out by operating the reactor without one of the reactants. The low feed rates necessary for the equilibrium assumption in Eq. (6) to be valid can be obtained without similar issues.

Class discussion is also used to point out possible sources of variability in the reaction system study such as error in laboratory analysis and experimental operating conditions. Measuring instruments are often imprecise and/or inaccurate, operating conditions cannot be set precisely as desired, and factors that cannot be observed or controlled can affect the behavior of any system under study. Therefore, any attempt to duplicate or repeat a single set of experimental conditions will usually produce different results. Sometimes the magnitude of this variation is small enough that useful conclusions can be drawn from a single experiment. At other conditions, however, an experiment must be repeated a number of times to be confident that the average value is an acceptable representation of the actual value.

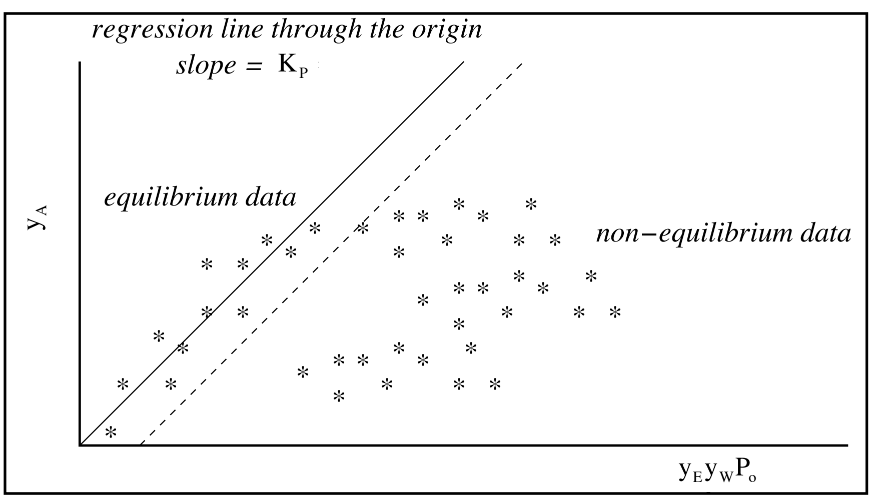

***Figure 3.*** *Example equilibrium data regression.*

## EXPERIMENTAL DATA

The students obtain experimental data by e-mailing the desired reaction conditions for each experiment using a specified procedure outlined in the project description handout. The costs of the experiments are deducted from the student group's budget as they are performed. The results are made available by e-mail to each group member the morning of the following day for normal experiments and by that afternoon for expedited experiments. The results include a summary of the experimental costs and the remaining budget.

A separate e-mail account using the class number as the e-mail address is created each year for this project. Scripts were developed to extract the operating conditions from the e-mail message, pass this information into the simulation and run it, create a report containing the experimental results and budget information, and then e-mail this report back to the student group. The original intent was to automatically perform each of these tasks without the intervention of the instructor. This approach, however, was quickly abandoned. The ability of undergraduate students to continuously find incorrect permutations to the required e-mail format resulted in increasing complexity to the data extraction script. Keeping in touch with each group's progress and the experiments they requested was also valuable. For these reasons, the project is administered by manual execution of the scripts. The administration task typically takes no more than 10 to 15 minutes each morning. As the report deadline approaches, the time commitment does increase slightly as a larger fraction of student groups request experiments on a given day.

## PROCESS SIMULATION

The reactor simulation is performed using the Octave computational environment running under the Debian linux operating system. Octave is a freely available mathematical computation package with similar capability to MATLAB. We note, however, that the Octave program files generated to support this project will not run in MATLAB. Additional information on Octave may be found at the Web site <www.octave.org>.

The reactor is simulated using an isothermal, steady-state, ideal plug flow reactor model. The forward and reverse rate constant activation energy and pre-exponential factor values are modified by the instructor each year. Literature values for these parameters are not used in order to prevent the more industrious student from obtaining the answer and reverse engineering their analysis. The values are also changed each year in order to prevent the less industrious student from getting values out of a prior-year project report. We note that these values are a function of the catalyst system used in the reactor and would be expected to change with different catalysts.

Normally distributed random variation is added to the specified values for reactor operating conditions. A standard deviation of $7.5 \times 10^{-3}$ mol/min is used for the variation added to each of the requested molar flow rate values and $5 \times 10^{-4}$ bar is the standard deviation used for the variation added to the requested outlet pressure. There is no variation added to the requested average reactor temperature and the simulation assumes a constant temperature at this value. The pressure drop across the reactor is determined from the expression

$$P_i = P_o + \alpha u^{\beta} \quad (7)$$

where $P_i$ is the inlet pressure (bar), $P_o$ is the specified outlet pressure (bar), u is the inlet gas velocity (m/min), $\alpha = 1.25 \times 10^{-4}$ and $\beta = 1.25$. These values provide a reasonable pressure drop range for the molar flow rate limits. Slight changes in these values have been made between years. Normally distributed random variation with a standard deviation on the order of $2 \times 10^{-3}$ is added to the ethanol mole fraction. The standard deviation of the variation in the hydrocarbon mole fraction is typically half that of the ethanol variation. Slight changes in these values have been made between years. The water mole fraction is determined by different checks made to ensure that reported values are positive and consistent.

Determination of reactor detonation is made by comparing the requested reactor average temperature and computed inlet pressure to a table of values. Temperatures below 375 °C or inlet pressures below 70 bar cannot result in detonation. Temperatures above 400 °C require inlet pressures above 69 bar for detonation, temperatures above 390 °C require inlet pressures above 72.5 bar, and so forth. These limits are chosen to make detonation rather difficult unless one is either very careless or intentionally wants to detonate the reactor. There have been few unintentional reactor detonations in our experience with this project. There have been a number of groups, however, who intentionally try to detonate the reactor with their last experiment. Although this practice is not within the scope of presenting a realistic experience to the students, it is not actively discouraged because it does provide a source of amusement and a final goal for some group members.

***The students are informed that temperatures beyond 400 °C and inlet pressures beyond 70 bar are dangerous and can very likely result in detonation of the reactor. . . . The students must therefore first determine safe operating conditions from initial experimental trials . . . ..***

## STATISTICAL ANALYSIS

For each temperature selected, the students are instructed to plot the experimental outlet alcohol mole fraction as a function of $P_E P_W \Theta_m$ and $y_E y_W P_o$ to determine which data points represent initial rate conditions and which data points represent equilibrium conditions. Deviation from the lines shown in Figures 2 and 3 by a given data point can be caused by experimental variation and/or violation of the assumptions made in the corresponding derivation. Although replicate experimental runs can help quantify the experimental variability, they do not provide the information necessary to exactly determine the point at which the initial rate and equilibrium assumptions are violated. This determination requires some judgment by the students.

A linear regression analysis on the selected initial rate and equilibrium data points is performed using a least squares linear fit through the origin at each temperature studied. The

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forward rate constant and equilibrium constant are determined from the slope of the respective lines. A 95% confidence interval on each of these values is determined from the standard error of the slope. These calculations are typically performed by the students using the EXCEL regression data analysis tool. The formulas may also be found in a number of introductory statistics texts. An extensive summary of statistics texts can be found in Reference 8 and is not replicated here.

A value for the reverse rate constant can be obtained from rearranging Eq. (6) to yield $k_r = k_f / K_P$ . The determination of a confidence interval, however, is more problematic. The reverse rate constant is the ratio of two independent t-distributed random variables. The result is a Cauchy distributed random variable with an undefined variance.[9] The unbounded variance arises from the fact that there is a finite probability that the equilibrium constant can be within an arbitrarily small neighborhood of zero. Further discussion of this aspect of the project is presented in the section on discussion topics.

A linear regression analysis based on Eq. (3) can be performed on both the forward and reverse rate constants to determine the activation energy and the log of the pre-exponential factor. This linear regression is also typically performed by students using the EXCEL regression data analysis tool. The students are asked to explain any rate constant values that they believe are inconsistent with the others and excluded from the regression. The activation energy can be determined from the slope using the relationship $E_a = -mR$, where m is the linear regression slope, and a 95% confidence interval can be determined from the confidence interval of the slope by scaling with the gas constant. The pre-exponential factor can be determined from the exponential of the intercept.

The students are asked to determine an estimate of the error variance for the laboratory ethanol analysis from the variance of residuals for each initial rate constant and equilibrium constant linear regression. The result is two error-variance estimates for each temperature studied. They are asked to discuss any differences between the estimated variances and whether the error in the alcohol analysis depends on the amount present in the sample. A 95% confidence interval on the analysis error is determined from the standard error computed from a pooled variance.

## REPORTING REQUIREMENTS

Students report their results in a short group memo to the instructor. The memo must contain a description of how the group arrived at their results, and enough detail for someone to replicate their results. An appendix to the memo should contain all of the data that was obtained. Plots of all the initial rate and equilibrium data with the regression line and an indication of which points were used in the regression must be included for each temperature selected. An Arrhenius plot for the forward and reverse rate constants with the regression line and an indication of any rate constant values that were not used in the regression must also be included.

Each group is scheduled for a 10-minute appointment with the instructor where only the instructor and the group members are present. The students turn in the memo, present their results, and answer any questions about their experimental plan and statistical analysis. The intent of this oral presentation is to provide an opportunity for the students to experience a technical interaction with a supervisor that many will encounter early in their careers as practicing engineers.

## DISCUSSION TOPICS

The project described in this article brings up a number of topics for discussion concerning the application of the statistical analysis techniques presented in the Applied Statistics course. The first topic typically brought up in discussion is the method used to determine valid initial rate and equilibrium experimental data. Although many student groups use the "eyeball" method to perform this determination, a more rigorous approach is to perform the regression with and without a given data point and look at the effect on the slope, confidence interval, and correlation coefficient. For points that are questionable, replicate experimental data should be used to help determine whether the deviation is due to experimental error alone.

A second topic for discussion is the basis for the linear regressions used in this project. The students are reminded that the regression equations given in their statistics text, and carried out by EXCEL, assume that there is no error in the independent variable. This assumption is clearly violated in the rate and equilibrium constant regressions due to error in the outlet composition measurements and the Arrhenius expression regression due to error in the average reactor temperature. Although an estimate of the magnitude of independent variable error can be obtained from the ethanol analysis error variance, a formal treatment of linear regression in this case is outside of the scope of the one-semester Applied Statistics course. It is anticipated that student groups would acknowledge that the regression assumption was violated. Very few student groups, however, realize this point without being prompted during the group oral presentation or class discussion. A very valuable contribution from this aspect of the project is to reinforce to the students that they must consider the basis and limitations of a statistics formula before they start performing any calculations.

Some student groups attempt to determine a reverse rate constant confidence interval by dividing the maximum error of the forward and equilibrium constants. A less suspect approach adopted by many student groups is to determine the confidence interval by approximating the variance from the forward rate and equilibrium constant variances as follows

$$s_{k_r}^2 = \left|\frac{\partial k_r}{\partial k_f}\right|^2 s_{k_f}^2 + \left|\frac{\partial k_r}{\partial K_P}\right|^2 s_{K_P}^2 = \frac{1}{K_P^2} s_{k_f}^2 + \frac{k_r^2}{K_P^4} s_{K_P}^2 \qquad (8)$$

where the partial derivatives are obtained from the rearrangement of Eq. (6), and $s^2_{kf}$, and $s^2_{KP}$ are obtained from the standard error of the slope from corresponding linear regressions. This variance is used to compute the standard error and a confidence interval is obtained from a t-distribution. A confidence interval for the pre-exponential factor is obtained by most student groups from the exponential of the 95% confidence interval of the intercept. Some groups determine the variance of the pre-exponential factor from that of the intercept from

$$s_{k_o}^2 = \left|\frac{\partial \ln(k_o)}{\partial k_o}\right|^2 s_{\ln(k_o)}^2 = \frac{1}{k_0^2} s_{\ln(k_o)}^2 \qquad (9)$$

and then compute the standard error and confidence interval using this variance and a t-distribution. These approaches are not correct. Confidence intervals on the reverse rate constant and pre-exponential factor cannot be determined because the parameter variance is undetermined. This aspect of the project attempts to reinforce the concept presented early in the statistics course that nonlinear transformations of normal or t-distributed random variables no longer retain their original distribution. Although it is fair to criticize the practice of asking for values that cannot be computed by the students, they may very well find themselves in this position later in their careers and should have some experience in realizing this point.

A further area of discussion on this topic is how one could obtain a confidence interval for the reverse rate constant and whether there is a more accurate method to determine its value. The students are prompted to consider a revision of the experimental plan that involves performing initial rate experiments using ethanol as the feed. In this case, the reverse rate constant can be determined directly from a single set of experiments in the same manner as the forward rate constant.

## STUDENT PERFORMANCE

The student groups are given about five weeks toward the end of the semester to complete this project. They are reminded in class during this period that it takes time to obtain data and they should not wait for the last minute to begin collecting data. Most student groups have successfully determined forward and reverse rate constants for at least three temperatures and have obtained reasonable values for the activation energy and pre-exponential factor. Very few groups have been unable to determine these values. The most typical reasons are the group started their data collection too late in the semester to obtain enough data and/or they were very inefficient in their experimental plan and expended their budget. Grading of the project in these cases is based on their pattern of experimental data requests. Groups that started early and appeared to have a plan but didn't quite get enough good data are treated in a much more forgiving manner than groups that waited for the last minute to request all of their data with little or no planning.

Groups have been formed both by students' own selection and by assignment of the instructor. There have been fewer cases of incomplete or poorly executed projects with the assigned groups, in our experience. Groups are instructed not to discuss any aspect of the assignment with anyone outside of their group, including the exchange of experimental data. Although it is difficult to enforce complete compliance with this policy, analysis of requested experiments has not revealed any obvious signs of copying experimental designs between groups or the use of data that was not requested by a group. We note that no two groups have ever obtained the same values for the Arrhenius parameters or used exactly the same number of experiments in a given semester. We have not performed this analysis between different semesters.

## CONCLUSIONS

The experimental design and statistical analysis project documented in this article has been developed to provide a realistic experience to students. Based on comments contained in course surveys, students have found the project to be interesting and worthwhile. A number of students have made positive comments on the realistic nature of the experience. Although not incorporated into the scope of this project, additional studies—such as an analysis of variance to determine the sources of variability in the experimental data—can be included within the framework discussed in this article. This project has also provided valuable documentation of the students' ability to design, conduct, analyze, and interpret experiments for Criterion 3b of the current ABET criteria.[10]

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