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Teaching Statistical Experimental Design Using a Laboratory Experiment

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Abstract

An experimental project introduces the concepts of statistical experimental design to undergraduates in a laboratory setting. A safe, inexpensive and easily operable experiment uses a gas chromatograph to give quantitative results and to allow students to concentrate on applying statistical skills without being impeded by complex equipment or experimental methods. One of the unique aspects of the experiment is a trade-off between the two most significant variables, forcing students to compromise in the selection of optimum conditions. Such compromises are typical in many real-world industrial situations. The experiment has been used for several years in the undergraduate chemical engineering laboratories at the University of North Dakota. **Keywords: Statistics, Experimental Design, Laboratory.**

I. Introduction

Undergraduates often have little exposure to statistical experimental design even though ABET is increasing the emphasis on including more statistical design/quality control in the undergraduate curriculum.[1] A recent series of articles[2-8] aimed at the practicing engineer and scientist, have dealt with the application of statistics to achieve quality control and process improvement. This emphasis on the application of statistics and quality improvement in industry has had an impact on the statistical skills needed by undergraduate engineers. In response to suggestions from our Alumni Advisory Board, we have wanted to increase the statistical and experimental design skills of our graduates for several years. To achieve this goal, the department introduced a semester-long fourth-year technical elective course in "Engineering Statistics," increased the emphasis of statistics in the four-semester-long undergraduate laboratory sequence, and developed a "workshop" course taken by all students in conjunction with the third laboratory course.

Our departmental philosophy is that the laboratory sequence is where the students have the opportunity to develop and practice some of the skills or "tools" that they will need as practicing engineers; these include observation, analysis, computation, writing, public speaking, statistical analysis and experimental design.

Experimental design involves using statistical methods in the planning of experiments so that statistically valid results can be obtained in an efficient manner. Unfortunately, many engineers and scientists still spend their energies on experimental programs that produce much data but little information. Part of this failure stems from the study-only-one-factor-at-a-time syndrome, where the effect of one factor is determined while all other variables are held constant.[9] This approach may ultimately lead to an understanding of the effect of the various factors, but does not allow the discovery of any interactions and usually requires an exhaustive number of experiments. A significant motivation for using experimental design is that it reduces the number of required experiments to determine the effects of the variables of interest. However, experimental design's greatest value lies in forcing the experimenter to use more forethought in the scheduling of runs and statistical analysis injects greater rigor into the interpretation of the results. This reduces the chances of an experimental program concluding with uninterpretable or meaningless results.

The undergraduate laboratory is an excellent place for a student to learn the true relationship between theory and practice. It has been recognized that experimentation is a critical and distinguishing element of the engineering profession.[10] Students entering into an undergraduate laboratory have spent most of their scientific life on the abstract side of measurements and think in terms of pure theoretical relationships between variables, not of the relationship of measured variables contaminated by experimental error.[11] They tend to think there are only two options if the results do not agree with the theory: 1) there is something wrong with the equipment or its value in elucidating the theory, or 2) the real and theoretical worlds cannot be bridged, so theory is of little value in the real world. It is our goal to design undergraduate experiments in such a way that the true relationship between theory and practice can be appreciated by the students and the presence of error can be accounted for in a rational manner. At the same time, the experiment should demonstrate some physical or chemical relationship. Above all, we believe that each experiment should be a learning experience in experimentation[11] so that students gain some skills and knowledge which will help them in future experiments.

We developed an experiment to introduce students to the concepts of experimental design and to help students apply and practice statistical skills learned in the classroom. For the experiment, we needed a process that had at least two (preferably three) variables on which several experiments could be run within a short time. McCluskey and Harris[12] described a simple and safe experiment that used a 2^3 factorial design to determine the "best" cup of coffee as a means of teaching experimental design. The primary drawback to

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their experiment is the difficulty in quantifying the results. We needed a safe, simple and inexpensive experiment which yielded clear quantitative results. We developed a gas chromatography (GC) experiment to fulfill these requirements.

Gas chromatography is an analytical technique used to obtain quantitative and qualitative measurements of a liquid or gas mixture. Because of its convenience, sensitivity, versatility and speed, the GC is a popular analytical device used in most industrial and university laboratories. This article describes the GC experiment developed to introduce students to the experimental design of experiments. This experiment has been implemented and refined during the last five years and successfully meets the desired objectives of being safe and simple while yielding quantitative results. A more complete description of gas chromatography is given elsewhere.[13]

Other equipment could be used to develop experiments that reinforce the concepts of experimental design. The main requirement is that there are two to three variables that effect the final outcome. It is also valuable to have two responses (outcomes) that are important and must both be optimized (compromised). Any production situation should work that balances product quality against production rate. For example in a mechanical engineering laboratory, a lathe experiment could be used which optimizes surface roughness (< acceptable) and production rate (maximum) versus the three adjustable parameters of lathe speed, feed and depth of cut.

The ChE undergraduate curriculum at UND includes a four-semester-long laboratory sequence that begins the second semester of the second year and continues through to the first semester of the fourth year. Their experimental tasks become more complex and the analysis more extensive as the students progress through the laboratory sequence. During the third laboratory course, the students concurrently enroll in a workshop on statistics and learn about the basic application of statistics to data analysis.[14] During the course they learn about the statistical design of experiments including factorial, fractional factorial, and response surface designs. The students are expected to utilize the statistical concepts they are learning in the course in the laboratory.

II. THEORY

In the statistics course discussed above, the students learn efficient experimental strategies (also called designs) for three situations: screening, crude optimization, and detailed optimization. Regardless of the situation, the basic goal is the same—to obtain a mathematical model to describe the system under study. However, the complexity of the mathematical model changes to reflect the amount of information desired. The mathematical models used by our students are given below for three independent variables or factors, X_1, X_2, and X_3.

Screening:

$$f = b_0 + b_1X_1 + b_2X_2 + b_3X_3 \quad (1)$$

Crude Optimization:

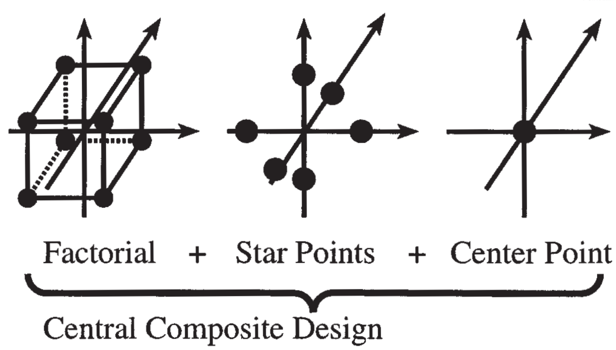
$$f = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 \quad (2)$$

Refined Optimization:

$$f = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{23}X_2X_3 + b_{11}X_1^2 + b_{22}X_2^2 + b_{33}X_3^2 \quad (3)$$

Although these models are just simple polynomials in the X variables (factors), they have been found to be quite adequate for the majority of practical problems. Once the experimental situation is recognized, the students must take enough data to estimate the coefficients in the model and be able to assess the statistical significance of each term. The final model (with only statistically significant effects) can then be used to draw whatever conclusions are warranted about the system under investigation.

The cornerstone of an experimental plan is the 2-level factorial design. It is used for estimating the coefficients in Equation 2. The mathematical model includes not only main (linear) terms but cross-products which allow for interaction of the factors. As a practical matter, center points are usually added to check on the adequacy of the model and to give a pure estimate of experimental error. For screening, which is used to sift out the important factors from a large number of potentially significant ones, a fraction of the 2^k factorial design is run to estimate the coefficients in Equation 1. This simple linear model is all that can be afforded at this stage of experimentation. Finally, if a detailed optimization is required, a response surface design is run to fit a full quadratic polynomial (Equation 3). The design most often used is a Central Composite design (as shown in Figure 1 for three factors) which adds axial points to a 2^k factorial design (plus center points). The other common response surface design was developed by Box and Behnken[15] and is shown in Figure 2. It cannot be built up from a



Block	Run	X1	X2	X3	
1	1	-1	-1	-1	
2	2	1	-1	-1	
2	3	-1	1	-1	Factorial Points
1	4	1	1	-1	
2	5	-1	-1	1	
1	6	1	-1	1	
1	7	-1	1	1	
2	8	1	1	1	
3	9	$-\alpha$	0	0	
3	10	α	0	0	Star Points
3	11	0	$-\alpha$	0	
3	12	0	α	0	
3	13	0	0	$-\alpha$	
3	14	0	0	α	
All	15-20	0	0	0	Center Points

Figure 1. Central Composite Design for 3 factors. Design commonly used to obtain a detailed response surface for optimization. Can be built up from fractional factorial and 2^k factorial design.

factorial design (a negative aspect), but it has fewer runs and is, therefore, a good design when the extra precision of the Central Composite is not needed. It also has only three (equally spaced) levels of each factor, which is often advantageous. For the experiment discussed in this paper, the Box-Behnken design is recommended to our students.

Once the data are collected using the experimental design, the data are analyzed rigorously. A four-step procedure is summarized in Table 1 which we require our students to use to come to the "best" model for their data. By "best" we mean the simplest model that adequately describes the response surface. We encourage the use of spreadsheets for this analysis since the use of statistical packages often makes the analysis too "canned" and inhibits learning of all the steps involved.

III. Task Assignment

Most of the experiments in the two third-year laboratory courses are based on a textbook.[16] For other experiments the students receive a manual which has a three-to-four page description of the additional experiments. The description gives general underlying principles and desired results of the experiment without necessarily being a "cook book." Before the students can initiate an experiment they must pass a prelaboratory quiz. This assures that they are familiar with the procedures and purposes of the experimental investigation. For the GC statistical design experiment, they have to set up their experimental design and schedule of experiments before entering the laboratory to collect data.

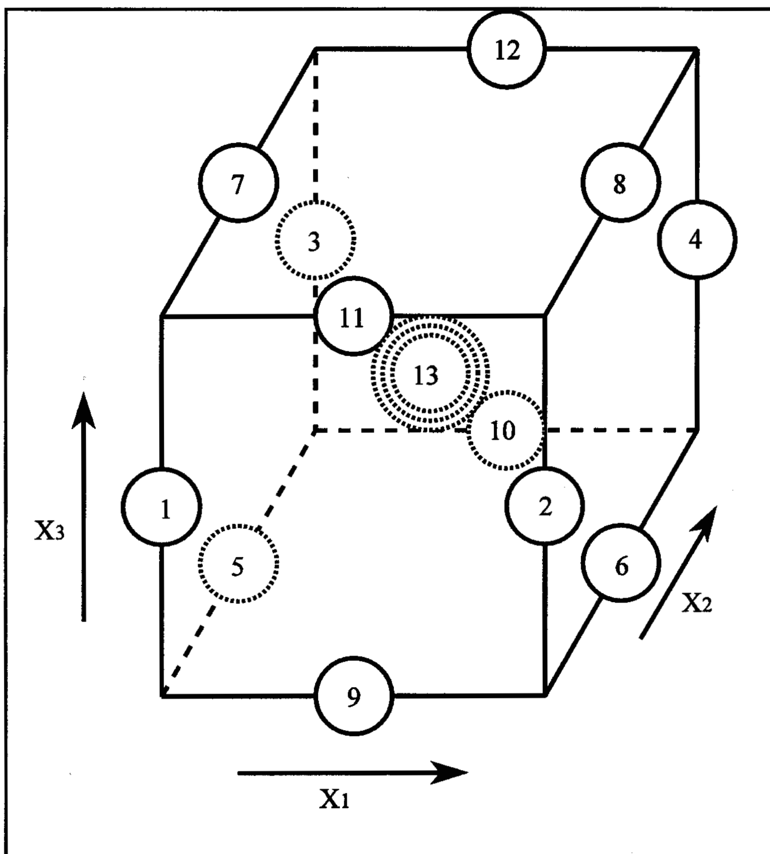
A. Project Description

The students are asked to optimize the operating conditions of a GC so that the analysis of a two-component liquid mixture (ethanol/methanol) can be performed in the minimum amount of time with the desired level of accuracy (resolution). A problem that occasionally occurs in GC analyses is that of peak resolution. Due to similarities of the physical properties of some substances, their GC peaks will overlap. This often leads to inaccurate or unusable results. Typical procedures to improve peak resolution include using a smaller sample size, operating the GC at a lower temperature, or using a lower carrier gas flow rate. The last two procedures also lead to longer times for analysis since the sample will take a longer time to elute from the GC column.

The output of the GC is processed with an integrator that gives the results of elution time, area under the peak (A), and the area-to-height ratio (*A/H*) for each compound eluting from the GC. The *A/H* is essentially the width of the peak at half of its height and corresponds to the time that the bulk of the component is actually eluting from the GC. (If you assume that the peak is approximated by a triangle, then the area is $(B \times H)/2$, and *A/H* is the half width of the peak). One method to quantify the resolution of peaks is by calculating the following:

Y_1 = (Time between peak midpoints)/$(A/H_{peak\,1} + A/H_{peak\,2})$ (4)

Higher values of Y_1 indicate better resolution of the peaks. To



Run	X_1	X_2	X_3
1	-1	-1	0
2	1	-1	0
3	-1	1	0
4	1	1	0
5	-1	0	-1
6	1	0	-1
7	-1	0	1
8	1	0	1
9	0	-1	-1
10	0	1	-1
11	0	-1	1
12	0	1	1
13	0	0	0
14	0	0	0
15	0	0	0

Figure 2. Box-Behnken[15] design for 3 factors. Advantages of only three, equally spaced levels for factors and only requires 15 runs to fit full quadratic response surface model.

1. FIT FULL QUADRATIC MODEL

Fit a full quadratic equation using least squares regression.

2. CHECK DATA FOR OUTLIERS

a. Calculate the difference between the model and the data points (residuals).

b. Construct a Normal plot (or better, a Half-Normal plot) of the residuals(17).

c. If the residuals look OK (fall on a straight line), proceed to Step 3.

d. If any residuals are "too big," throw out the worst data point and go back to Step 1.

3. TRIM MODEL DOWN TO THE SMALLEST ADEQUATE FUNCTION

a. Check each independent variable to see if it can be deleted. This is done by fitting the model with and without the variable under scrutiny. If m terms are dropped when the variable is deleted, you must see if the Sum of Squares (of residuals) increased by significantly more than $m \times s^2$, where s is the estimated standard deviation of the random errors in the data. Note: s^2 is estimated from the fit of the bigger model (with v degrees of freedom) and significance is judged by comparing the ratio of $(SS_{\text{Increase}}/m)/s^2$ to an F-distribution with m and v degrees of freedom.

b. Delete any <u>high order terms</u> (for the important variables) that are not significant. This should be done one term at a time, starting with the term that has the smallest t-value. The t-statistic for each coefficient is calculated by dividing the coefficient by its standard deviation. If the t-statistic is less than the critical value from tables for 95% confidence (approximately 2), then the term is not significantly different from zero and can be dropped. Note: It may happen that a linear term is not significant even though an interaction or a quadratic term with the same variable is significant. In this case (not very common), it is recommended that you <u>do not</u> drop the linear term.

4. CHECK MODEL ADEQUACY

a. Check Lack-of-Fit to ensure that a quadratic model is adequate(17). This is done by breaking the residual sum of squares, SS_R, and its associated degrees of freedom, v_R, into two pieces:

i. Sum of Squares for Pure Error (from replicates), SS_{PE},

ii. Sum of Squares due to Lack of Fit, SS_{LoF}, by difference ($SS_{LoF} = SS_R - SS_{PE}$) Note: $v_{LoF} = v_R - v_{PE}$.

The Lack-of-Fit variance, s_{LoF}^2, can then be calculated ($s_{LoF}^2 = SS_{LoF} / v_{LoF}$) and checked to see if it is significantly bigger than $s^2 = s_{PE}^2 = SS_{PE} / v_{PE}$. The significance is determined by comparison to an F-distribution with v_{LoF} and v_{PE} degrees of freedom.

b. Check residuals for trends. This is done by plotting the residuals against any variable that makes sense (e.g. run order, X_1, etc.). If there are any trends, it is an indication that something should be added to the model.

Table 1. Summary of four-step procedure for determination of the best response surface model.

ensure good resolution with no distortion of the peaks due to overlap, Y_1 should have a value of at least 1.5. Optimal operating conditions will also include the shortest operation time which gives an adequate value of Y_1. The second function that the students optimize (minimize) is the time for the last peak to pass through the GC, or

$$Y_2 = \text{(Time for second peak)} \quad (5)$$

The students are told to use a Box-Behnken[15] statistical design which gives all the information to fit the response surface of three variables (factors) using only 15 experiments (which includes three replicates at the average conditions) The three operating variables are the sample size, the GC oven temperature, and the carrier gas flow rate. They are given the limits on the operating conditions (maximum and minimum sample, operating temperature and carrier gas flow rate) and told to design a series of experiments which will enable them to determine the optimum operating conditions (minimize Y_2 for conditions where Y_1 is at least 1.5).

B. Experimental Work

Using the Box-Behnken approach, the students can plan their experiments by operating each variable at three different levels. They typically operate at two convenient limits of the operating range of the variable and at a midpoint level. Table 2 shows some typical operating conditions for the three variables, and Table 3 shows the required runs for the experimental design. Each variable (factor) is evaluated at three equally spaced levels; these are shown in coded form in which the largest value of a variable is one and the lowest is negative one. The variables are coded using

$$X_i = (\text{Factor Value}_i - \text{Center})/(\text{High Value} - \text{Center}) \quad (6)$$

There are several statistical reasons why the variables should be coded, the major one being to minimize interdependency of the coefficients in the quadratic equation. It also puts all factors on the same scale, so that the most important coefficient has the largest absolute value.

To avoid the risk of any mechanical or operator biases clouding the conclusions, the various runs should be performed in random order. However, the time required to vary the oven temperature prohibits total randomization in the four-hour-long laboratory. We suggest that the students group *some* (not all) of the runs at a given temperature. The "Run Order" column in Table 3 demonstrates a typical ordering of the runs that allows some randomness and yet keeps down the number of times that the oven temperature is changed. Also, the run order is set up so that the three runs at replicate conditions are dispersed throughout the series of experiments.

Students collect data following the run order. After the operating conditions are set and the GC stabilized, the students inject their sample, and the integrator gives them the quantitative results to calculate Y_1 and Y_2. Depending on conditions, a run will take between 2- and 15-minutes for both components to elute from the GC, with most runs being on the order of 3- to 5- minutes. Typically, about five minutes are required for the GC to stabilize after adjusting carrier gas flow rate and about 20 minutes for it to stabilize after changing the temperature. All 15 runs can usually be completed within three to four hours. Sometimes the students will split the runs between two four-hour laboratory periods.

C. Sample Results

The final two columns in Table 3 are typical results obtained by a student. As mentioned above, the ensuing data analysis is most often done on a spreadsheet. Multiple regression analyses are completed for both Y_1 and Y_2 to fit the general quadratic model (Equation 3) with the coded independent variables (Step 1 in Table 1). The students then check for outliers (Step 2 in Table 1) by constructing a Half-Normal plot[14,17] of the residuals (Figure 3). Any residual that falls far off the line of the Half-Normal plot indicates that it does not fit the quadratic model very well and should not be used in the calculation of the coefficients. Had any outliers been detected, they would have been removed (one at a time) and the regression repeated. Sometimes additional data points are collected to replace the outliers.

Next the best response surface model is found by eliminating any nonsignificant terms (Step 3 in Table 1). The multiple regression package of the spreadsheet gives all of the coefficients of the quadratic model with their corresponding standard errors. From these the t-statistic can be determined to test whether any of the coefficients in the quadratic model are nonsignificant. The left-hand columns of Table 4 give the regression analysis for the Y_1 values given in Table 3. As shown, for this set of data, the coefficient b_{23} for the interaction effect between variables X_2 and X_3 is not shown to be significantly different than zero (its t-statistic is less than two). The data are then regressed again with a model that eliminates the X_2X_3 interaction, and the results are given in the right-hand columns of Table 4. In this case, all of the remaining coefficients are shown to be statistically significant, and the response surface model is completed.

Finally, the statistical analysis is completed by checking the adequacy of the model (Step 4 in Table 1) by comparing the Lack-of-Fit variance, s_{LoF}^2, to the Pure-Error variance s_{PE}^2. Also, the residuals are plotted against run order, X_1, X_2, and X_3 to see if any trends appear. The response surface model is then plotted as contour plots using a graphical software package. In this case, since X_1 (sample size) had the least effect (as noted by the smallest coefficients), plots are made of the responses versus X_2 and X_3. Several plots of the re-

Variable	Level -1	Level 0	Level +1
Sample size (μliter)	0.2	0.6	1.0
Gas flow (cm^3/min)	15	50	85
GC Oven Temp (K)	400	435	470

Table 2. Variables and typical operating levels.

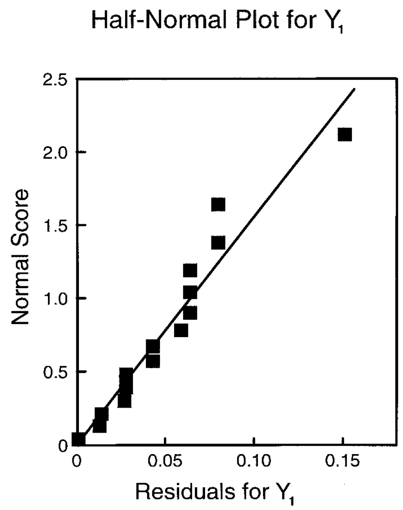


Figure 3. Half-Normal plot of residuals for typical student data for Y_1. Used as initial stages of statistical analysis to eliminate poor data (outliers).

sponse surface can be generated for both Y_1 and Y_2. Figures 4 and 5 show two such plots generated for the data in Table 3 with the response surface models developed by the student. By overlaying the Y_1 and Y_2 contour plots (Figure 6), the point can be found where Y_2 is minimized subject to the constraint that Y_1 is 1.5 or greater. For these data, the optimum gas flow rate, X_2, is 68 cm^3/min, and the oven temperature, X_3, is 440 K. It should be noticed from the contour plots that this experiment demonstrates vividly the compromises that must often be made in the real world between various objectives. In this case, a compromise must be made between speed and accuracy (resolution). Resolutions of more than 3 could be obtained, but the price is doubling or tripling of the analysis time.

IV. Discussion

A motivating factor behind the development of this experiment was the feedback we received from our Alumni Advisory Board and the prevalence of articles[2-8] on the need for more skills in statistical design of experiments and statistical analysis of data for

Run	Coded Sample Size	Coded Gas Flow	Coded Temp	Run* Order	Resolution Y_1	Run Time Y_2
1	-1	-1	0	8	2.81	5.95
2	+1	-1	0	9	1.91	5.53
3	-1	+1	0	7	1.49	1.98
4	+1	+1	0	10	1.25	1.89
5	-1	0	-1	3	3.11	5.82
6	+1	0	-1	4	2.30	5.34
7	-1	0	+1	14	1.04	1.63
8	+1	0	+1	13	0.865	1.51
9	0	-1	-1	2	3.09	11.6
10	0	+1	-1	5	2.25	3.96
11	0	-1	+1	11	1.44	3.83
12	0	+1	+1	12	0.771	1.15
13	0	0	0	1	1.59	2.67
14	0	0	0	6	1.60	2.70
15	0	0	0	15	1.61	2.68

* Randomized run order to minimize mechanical or operator bias. See text for discussion in Section III.B.

Table 3. Box-Behnken experimental design for three factors with a typical run order and experimental responses.

quality improvement. One of our responses has been to implement a course taken concurrently with this laboratory course that teaches the students some of the principles of experimental design and analysis. The course was placed in the middle of the laboratory sequence so that the students could take advantage of the information. At this level, the students have grappled with trying to interpret experimental data from the previous laboratory courses and have experienced some of the pitfalls of applying linear regression to unplanned data.[9] The students then have two laboratories to practice and hone their newly-learned statistical skills.

This experiment, which was developed to reinforce the concepts of experimental design and statistical analysis, has been successful for various reasons. First, the experimental portion of the project is relatively simple and safe. With adequate planning, the complete set of runs can be finished within four hours. Sometimes the students will split the runs between laboratory days, but more often, they will complete a second pass through their experimental design to have more replicate data. With the use of an integrator, the students can concentrate on the experimental design and statistical analysis without being burdened with interpreting the chromatogram. In experiments in previous laboratory courses, the students are introduced to the operation and theory of gas chromatography. Another advantage of the statistical-experimental-design-using-a-GC project is that the response signal can be easily quantified. The integrator gives *A/H* ratios and retention times for each peak, and the students can easily transfer these results to a computer for processing. The multiple regression analysis and the determination of the best response surface model by the elimination of all nonsignificant coefficients can be completed entirely using a statistical software package. Typically the students perform the multiple linear regression using a spreadsheet since they are more familiar with it. We encourage this since it reinforces the methodology for only a bit more effort. Finally, another reason for the success of this experiment is that the effects of the variables are not easily predicted which is often the case in real-world industrial optimization problems.[18] As was the case in the example shown, the student's results will often predict curvature in the response surface and the optimal operation conditions are not on one of the boundaries or limits of operating conditions. The task of finding conditions that are a good compromise for two responses is a very realistic situation.

First Regression				Final Regression			
Constant		1.596		Constant		1.598	
Std Error of Y Est.		0.10016		Std Error of Y Est.		0.09802	
R Squared		0.99375		R Squared		0.99282	
No. of Observations		15		No. of Observations		15	
Degrees of Freedom		5		Degrees of Freedom		6	
Coefficients				Coefficients			
Coef.	Value	Std. Err.	t	Coef.	Value	Std. Err.	t
b_1	-0.2644	0.03541	7.466	b_1	-0.2644	0.03465	7.629
b_2	-0.4372	0.03541	12.35	b_2	-0.4373	0.03465	12.62
b_3	-0.8296	0.03541	23.43	b_3	-0.8296	0.03465	23.94
b_{12}	0.1618	0.05008	3.230	b_{12}	0.1618	0.04901	3.300
b_{13}	0.1580	0.05008	3.155	b_{13}	0.1580	0.04901	3.224
b_{23}	0.04325	0.05008	0.8636*	b_{23}			
b_{11}	0.1040	0.05213	1.995	b_{11}	0.1040	0.05101	2.039
b_{22}	0.1628	0.05213	3.122	b_{22}	0.1628	0.05101	3.191
b_{33}	0.1265	0.05213	2.427	b_{33}	0.1265	0.05101	2.480

*** Small *t*-value indicates that this coefficient is not significantly different from zero and the interaction term is eliminated from the response surface model.**

Table 4. Typical regression analysis to determine response surface model (Analysis for Y_1, resolution of peaks.)

This experiment, like most good research projects, has several facets worthy of additional exploration. Some students have used different experimental design models (factorial, central composite) with similar results. Of course a simple factorial experimental design can only be used to develop a linear empirical model. Other variables such as the ratio of the two components in the mixture can also be examined. Many GCs are set up with multiple columns, so GC column length could also be a variable.

The student response to the experiment has generally been excellent. Most come away from it with an appreciation for the usefulness of experimental design to minimize the number of experiments that need to be run while maximizing the predictive information. All students obtain at least a basic level of skill in statistical analysis of data and gain some sense of how to deal with experimental error in their analyses. We have noted since introducing the experiment that many students will apply experimental design concepts in subsequent courses, in laboratories, and in individual research projects. Students who have worked in industry, either in a summer job or a more extensive cooperative education internship, have found many uses for the skills developed. Feedback from the recent graduates and employers has also been very positive. Most important, the feedback from our Alumni Council which represents a broad range of industry has been overwhelmingly supportive.

V. CONCLUSIONS

A simple and safe experiment with easily quantified results has been developed using gas chromatography to demonstrate the concepts of experimental design and statistical analysis of experi-

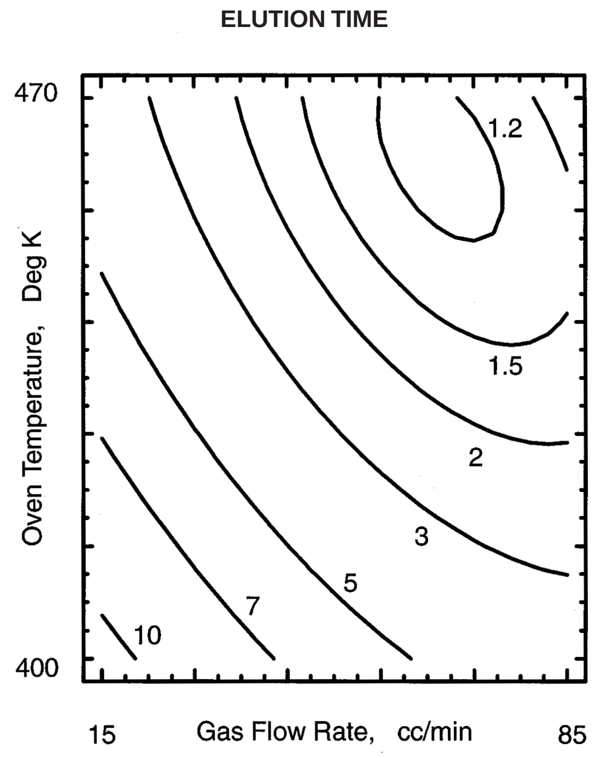


Figure 5. Contour plot of Y_2 (Analysis Time) as a function of X_2 (Oven Temperature) and X_3 (Gas Flow Rate) at the average value of X_1=1 (Sample Size). Plot shows operating conditions that minimizes the total analysis time.

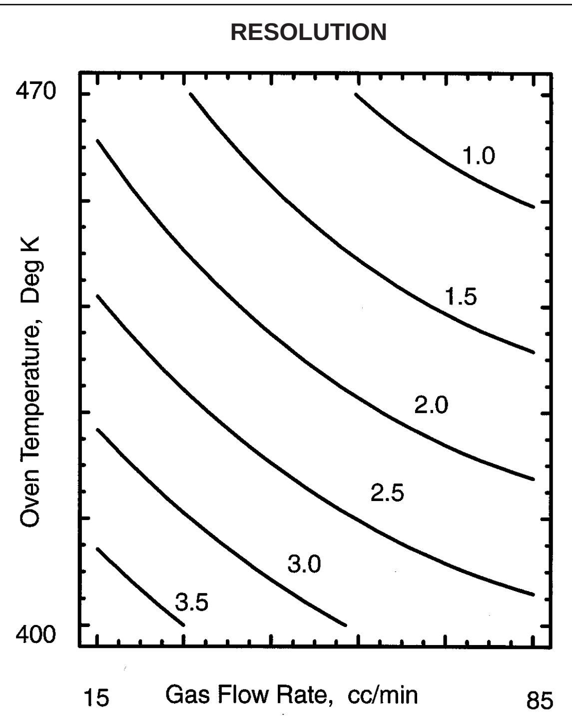


Figure 4. Contour plot of Y_1 (Resolution of Peaks) as a function of X_2 (Oven Temperature) and X_3 (Gas Flow Rate) at the average value of X_1=1 (Sample Size). Plot shows regions on response surface map where acceptable resolution ($Y_1 \geq 1.5$) are obtainable.

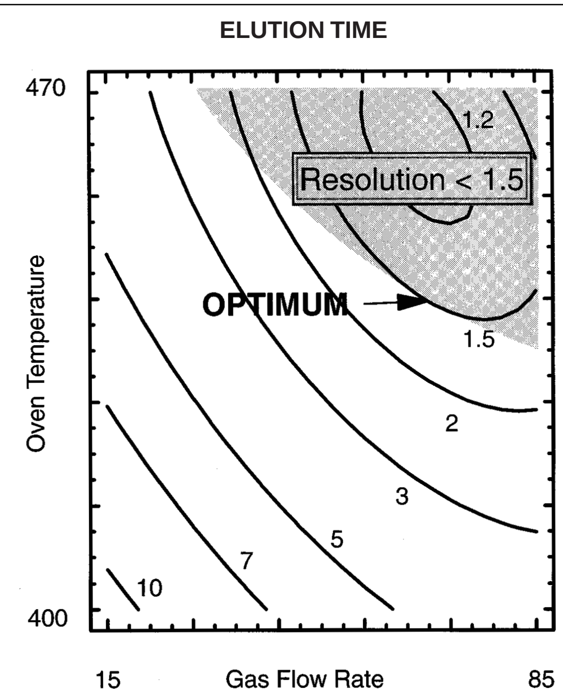


Figure 6. Contour plot of Y_2 (Analysis Time) as a function of X_2 (Oven Temperature) and X_3 (Gas Flow Rate) at the average value of X_1=1 (Sample Size) with the resolution constraint (Y_1) overlaid. This plot shows the need to compromise needs to be made between accuracy (resolution) and speed of analysis.

ments. The experiment has been designed and implemented so that the students can concentrate on developing and refining their statistical skills without getting bogged down with esoteric aspects of the project.

The experiment has been implemented and refined over the last five years and the response has been enthusiastic from the currently enrolled students and from the recent graduates. In summary, our experience with this experiment has been quite positive. Operation of the experiment goes well, and most of the students report that they developed an appreciation for the usefulness of statistical experimental design.

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