Chapter 33

Response Surface Methodology

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Introduction

- Response Surface Methodology (RSM) is used to determine how a response is affected by a set of quantitative factors over some specified region.
- This information can be used to optimize the settings of a process to give a maximum or minimum response.
- For a given number of variables, response surface analysis techniques require more trials than the two-level fractional factorial design techniques; hence. the number of variables considered in an experiment may first need to be reduced through either technical considerations or fractional factorial experiments.

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Introduction

- This chapter explains how to apply central composite rotatable and Box-Behnken designs for determining the response surface analysis of variables.
- It discusses extreme vertices and simplex lattice designs along with computer algorithm designs for mixture designs.

33.1 Modeling Equations

- The previous DOE chapters covering 2-level fractional factorial experimentation considered main effects and interaction effects. For these designs the response was assumed to be linear between the levels of the factors.
- The general approach of investigating factor extremes addresses problems expediently with a minimal number of test trials. This form of experimentation is adequate in itself for solving many types of problems, but there are situations in which a response needs to be optimized as a function of the levels of a few input factors. This chapter focuses on such situations.

33.1 Modeling Equations

- The prediction equation for a two-factor linear main-effect model without the consideration of interactions takes the form

$$y = b_0 + b_1 x_1 + b_2 x_2$$

  where y is the response, b_0 is the y-axis intercept, and (b_1, b_2) are the coefficients of the factors. For a balanced experiment design with factor-level considerations for x_1, and x_2, respectively, equal to -1 and +1, the b_1, and b_2 coefficients equate to one-half of the effect and b_0 is the average of all the responses.
- Computer programs can determine these coefficients by such techniques as least squares regression.

33.1 Modeling Equations

- If there is an interaction consideration, the equation model will then take the form

$$y = b_0 + b_1 x_1 + b_2 x_2 + b_{12} x_1 x_2$$

- The number of terms in the equation represents the minimum number of experimental trials needed to determine the model. For example, the equation above has 4 terms; a minimum of 4 trials is needed to calculate the coefficients.
- The 2-level DOE significance tests discussed in previous chapters were to determine which of the coefficient estimates were large enough to have a statistically significant affect on the response (y) when changed from a low (-1) level to a high (+1) level.

33.1 Modeling Equations

- Centerpoints can be added to the 2-level fractional factorial design to determine the validity of the linearity assumption of the model.
- When using a regression program on the coded effects, the fractional factorial levels should take on symmetrical values around zero (i.e., -1 and +1).
- To determine if the linearity assumption is valid, the average response of the centerpoints can be compared to the overall average of the two-level fractional factorial experiment trials.

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33.1 Modeling Equations

- If the first-degree polynomial approximation does not fit the process data, a second-degree polynomial model may adequately describe the curvature of the response surface as a function of the input factors.
- For 2-factor considerations, this model takes the form

$$y = b_0 + b_1x_1 + b_2x_2 + b_{11}{x_1}^2 + b_{22}{x_2}^2 + b_{12}x_1x_2$$

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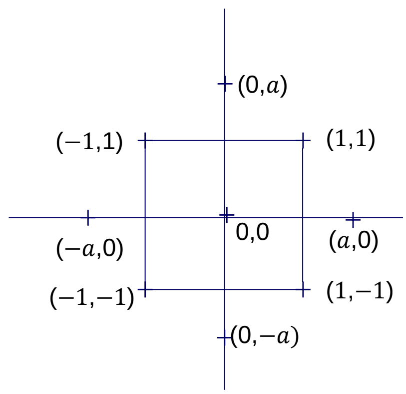
33.2 Central Composite Design

- To determine the additional coefficients of a second-degree polynomial, additional levels of the variables are needed between the end-point levels.
- An efficient test approach to determine the coefficients of a second-degree polynomial is to use a central composite design.



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33.2 Central Composite Design

- An experiment design is said to be rotatable if the variance of the predicted response at some point is a function of only the distance of the point from the center.
- The central composite design is made rotatable when $[a = (F)^{1/4}]$, where F is the number of points used in the factorial part of the design. For two factors $F = 2^2 = 4$; hence, $a = (4)^{1/4} = 1.414$
- A useful property of the central composite design is that the additional axial points can be added to a two-level fractional factorial design as additional trials after the curvature is detected from initial experimental data.

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33.2 Central Composite Design

- With a proper number of center points, the central composite design can be made such that the variance of the response at the origin is equal to the variance of the response at unit distance from the origin (i.e., a uniform precision design).
- This characteristic in the uniform precision design is important because it gives more protection against bias in the regression coefficients (because of the presence of third-degree and higher terms in the true surface) than does the orthogonal design.

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33.2 Central Composite Design

- Table 33.1 shows the parameters needed to achieve a uniform precision design as a function of the number of variables in the experiment.

No. of Variables	No. of Factorial Trials	No. of Axial Trials	No. of Center Trials	a	Total No. of Trials
2	4	4	5	1.4142	13
3	8	6	6	1.6818	20
4	16	8	7	2.0000	31
5	16	10	6	2.0000	32
6	32	12	9	2.3784	53
7	64	14	14	2.8284	92

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33.2 Central Composite Design

- From this table, for example, a design assessing five variables along with all two-factor interactions plus the curvature of all variables would be that shown in Table 33.2.

Fractional Factorial Design

	A	B	C	D	E
1	+1	-1	-1	-1	+1
2	+1	+1	-1	-1	-1
3	+1	+1	+1	-1	+1
4	+1	+1	+1	+1	-1
5	-1	+1	+1	+1	+1
6	+1	-1	+1	+1	+1
7	-1	+1	-1	+1	-1
8	+1	-1	+1	-1	-1
9	+1	+1	-1	+1	+1
10	-1	+1	+1	-1	-1
11	-1	-1	+1	+1	-1
12	+1	-1	-1	+1	-1
13	-1	+1	-1	-1	+1
14	-1	-1	+1	-1	+1
15	-1	-1	-1	+1	+1
16	-1	-1	-1	-1	-1

Axial trials

	A	B	C	D	E
17	-2	0	0	0	0
18	+2	0	0	0	0
19	0	-2	0	0	0
20	0	+2	0	0	0
21	0	0	-2	0	0
22	0	0	+2	0	0
23	0	0	0	-2	0
24	0	0	0	+2	0
25	0	0	0	0	-2
26	0	0	0	0	+2

Center point trials

	A	B	C	D	E
27	0	0	0	0	0
28	0	0	0	0	0
29	0	0	0	0	0
30	0	0	0	0	0
31	0	0	0	0	0
32	0	0	0	0	0

33.2 Central Composite Design

- Data are then analyzed using regression techniques to determine the output response surface as a function of the input variables.

33.2 Central Composite Design

- Comell (1984), Montgomery (1997), and Box et al. (1978) discuss analytical methods to determine maximum points on the response surface using the canonical form of the equation. The coefficients of this equation can be used to describe the shape of the surface (ellipsoid, hyperboloid, etc.).
- An alternative approach is to understand the response surface by using a computer contour plotting program, as illustrated in the next example.
- Determining the particular contour plot may help determine/ change process factors to yield a desirable/improved response output with minimal day-to-day variation.

33.2 Central Composite Design

- Creating a contour representation for the equation derived from an RSM can give direction for a follow-up experiment. For example, if the contour representation does not capture a peak that we are interested in, we could investigate new factor levels, which are at right angles to the contours that appear to give a higher response level. This is called direction of steepest ascent.
- When searching for factor levels that give a lower response, a similar direction of steepest descent approach could be used.

33.3 Example 33.1 Response Surface Design

- A chemical engineer desires to determine the operating conditions that maximize the yield of a process.
- An earlier 2-level factorial experiment of many considerations indicated that reaction time and reaction temperature were the parameters that should be optimized.
- A central composite design was chosen and yielded the responses shown in Table 33.3 (Montgomery I997).

33.3 Example 33.1 Response Surface Design

Natural Variables		Coded Variables		Response		
				Yield	Viscosity	Mole. Weight
u_1	u_2	v_1	v_2	y_1	y_2	y_3
80	170	-1	-1	76.5	62	2940
80	180	-1	1	77.0	60	3470
90	170	1	-1	78.0	66	3680
90	180	1	1	79.5	59	3890
85	175	0	0	79.9	72	3480
85	175	0	0	80.3	69	3200
85	175	0	0	80.0	68	3410
85	175	0	0	79.7	70	3290
85	175	0	0	79.8	71	3500
92.07	175	1.414	0	78.4	68	3360
77.93	175	-1.414	0	75.6	71	3020
85	182.07	0	1.414	78.5	58	3630
85	167.93	0	-1.414	77.0	57	3150

33.3 Example 33.1 Response Surface Design

- A second-degree model can be fitted using the natural levels of the variables (e.g., time = 80) or the coded levels (e.g., time=-1).

33.3 Example 33.1 Response Surface Design

Response Surface Regression: y1 versus v1, v2

The analysis was done using coded units.

Estimated Regression Coefficients for y1

Term	Coef	SE Coef	T	P
Constant	79.9400	0.11909	671.264	0.000
v1	0.9951	0.09415	10.568	0.000
v2	0.5152	0.09415	5.472	0.001
v1*v1	-1.3764	0.10098	-13.630	0.000
v2*v2	-1.0013	0.10098	-9.916	0.000
v1*v2	0.2500	0.13315	1.878	0.103

S = 0.266290 PRESS = 2.35346

R-Sq = 98.27% R-Sq(pred) = 91.81% R-Sq(adj) = 97.04%

Minitab
Stat
DOE
Response Surface
Define/Analyze

33.3 Example 33.1 Response Surface Design

Analysis of Variance for y1

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	28.2467	28.2467	5.64934	79.67	0.000
Linear	2	10.0430	10.0430	5.02148	70.81	0.000
Square	2	17.9537	17.9537	8.97687	126.59	0.000
Interaction	1	0.2500	0.2500	0.25000	3.53	0.103
Residual Error	7	0.4964	0.4964	0.07091		
Lack-of-Fit	3	0.2844	0.2844	0.09479	1.79	0.289
Pure Error	4	0.2120	0.2120	0.05300		
Total	12	28.7431				

Estimated Regression Coefficients for y1 using data in uncoded units

Term	Coef
Constant	79.9400
v1	0.995050
v2	0.515203

Term	Coef
v1*v1	-1.37645
v2*v2	-1.00134
v1*v2	0.250000

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33.3 Example 33.1 Response Surface Design

Response Surface Regression: y1 versus u1, u2

The analysis was done using uncoded units.

Estimated Regression Coefficients for y1

Term	Coef	SE Coef	T	P
Constant	-1430.69	152.851	-9.360	0.000
u1	7.81	1.158	6.744	0.000
u2	13.27	1.485	8.940	0.000
u1*u1	-0.06	0.004	-13.630	0.000
u2*u2	-0.04	0.004	-9.916	0.000
u1*u2	0.01	0.005	1.878	0.103

S = 0.266290 PRESS = 2.35346
R-Sq = 98.27% R-Sq(pred) = 91.81% R-Sq(adj) = 97.04%

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33.3 Example 33.1 Response Surface Design

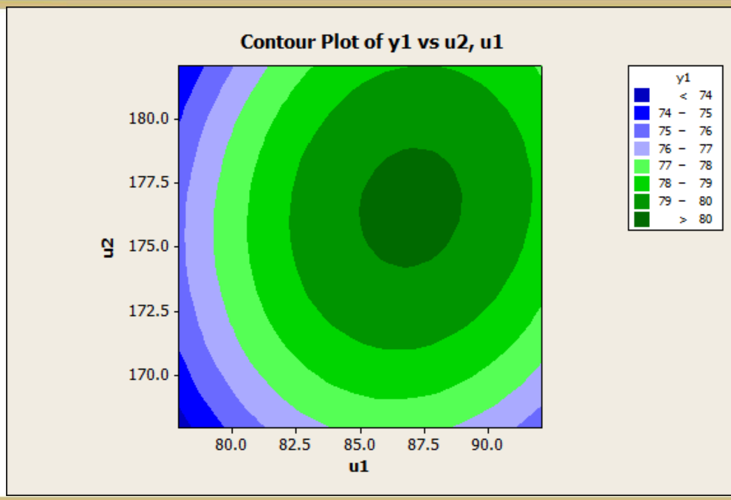
Analysis of Variance for y1

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	28.2467	28.2467	5.64934	79.67	0.000
Linear	2	10.0430	6.8629	3.43147	48.39	0.000
Square	2	17.9537	17.9537	8.97687	126.59	0.000
Interaction	1	0.2500	0.2500	0.25000	3.53	0.103
Residual Error	7	0.4964	0.4964	0.07091		
Lack-of-Fit	3	0.2844	0.2844	0.09479	1.79	0.289
Pure Error	4	0.2120	0.2120	0.05300		
Total	12	28.7431				

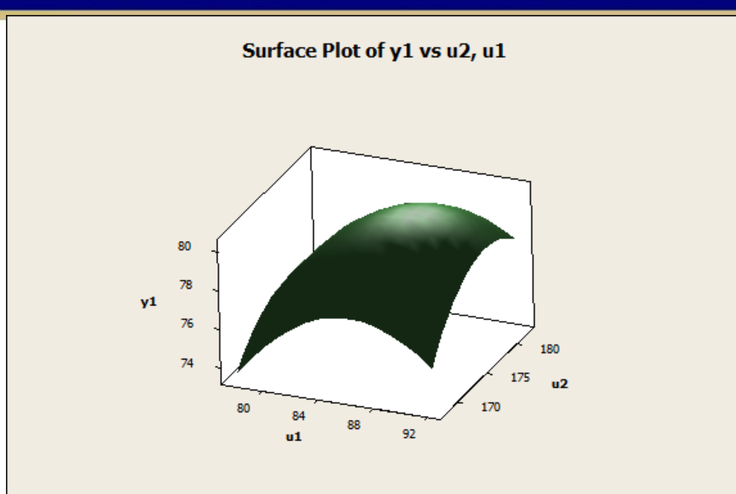
33.3 Example 33.1 Response Surface Design

- From this analysis, the second-degree in terms of the coded levels of the variables is
  $\hat{y} = 79.940 + 0.995v_1 + 0.515v_2 - 1.376{v_1}^2 + 0.250v_1v_2 - 1.001{v_2}^2$
- This equates to an equation for the natural levels of
  $\hat{y} = -1430.69 + 7.81u_1 + 13.27u_2 - 0.06{u_1}^2 + 0.01u_1u_2 - 0.04{u_2}^2$
- The advantage of using the coded levels is that the importance of each term can be easily compared.
- When projections are made from a response surface, it is obviously important that the model fit the initial data satisfactorily.
- Erroneous conclusions can result when there is lack of fit.

33.3 Example 33.1 Response Surface Design



33.3 Example 33.1 Response Surface Design



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33.4 Box-Behnken Designs

- When estimating the first- and second-order terms of a response surface, Box and Behnken (1960) give an alternative to the central composite design approach.
- They present a list of l0 second-order rotatable designs covering 3, 4, 5, 6, 7, 9, 10, 11, 12, and 16 variables.
- However, in general, Box-Behnken designs are not always rotatable nor are they block orthogonal.
- One reason that an experimenter may choose this design over a central composite design is physical test constraints.
- This design requires only three levels of each variable, as opposed to five for the central composite design.

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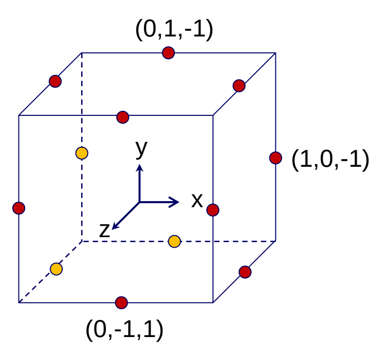
33.4 Box-Behnken Designs

- Figure 33.4 shows the test points for this design approach given three design variables.



x	y	z
1	1	0
1	-1	0
-1	1	0
-1	-1	0
1	0	1
1	0	-1
-1	0	1
-1	0	-1
0	1	-1
0	1	1
0	-1	-1
0	-1	1
0	0	0
0	0	0
0	0	0

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33.5 Mixture Designs

- The experiment designs discussed previously apply to discrete and/or continuous factors, where the levels of each factor are completely independent from the other factors.
- However, consider a chemist who mixes three ingredients together. If the chemist wishes to increase the percentage of one ingredient, the percentage of another ingredient must be adjusted accordingly.
- Mixture experiment designs are used for this situation, where the components (factors/variables) under consideration take levels that are a proportion of the whole.
- Computer-generated designs and analyses are usually better for most realistic mixture problems.

33.5 Mixture Designs

- In the general mixture problem the measured response depends only on the proportions of the components present in the mixture and not on the total amount of the mixture.
- For three components this can be expressed as

$$x_1 + x_2 + x_3 = 1$$

- To illustrate the application of this equation, consider that a mixture consists of three components: A, B, and C. If component A is 20% and B is 50%, C must be 30% to give a total of 100% (i.e., 0.2 + 0.5 + 0.3 = 1).

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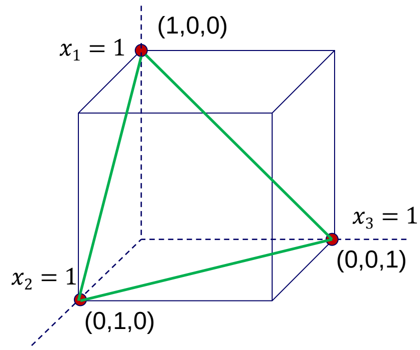
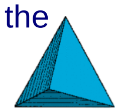
33.5 Mixture Designs

- When three factors are considered in a 2-level full factorial experiment (2^3), the factor space of interest is a cube. However, a three-component mixture experiment is represented by an equilateral triangle.
- The coordinate system for these problems is called a simplex coordinate system.



33.5 Mixture Designs

- A four-component experiment would similarly take on the space of a tetrahedron.



- With three components, coordinates are plotted on equilateral triangular graph paper that has lines parallel to the three sides of the triangle.
- Each vertex of the triangle represents 100% of one of the components in the mixture.
- The lines away from a vertex represent decreasing amounts of the component represented by that vertex.
- The center of the equilateral triangle represents, for example, a mixture with equal proportions (⅓, ⅓, ⅓) from each of the components.

33.5 Mixture Designs

- In a designed mixture experiment, several combinations of components are chosen within the spatial extremes defined by the number of components (e.g., an equilateral triangle for three components).
- In one experiment all possible combinations of the components can be considered as viable candidates for determining an “optimal” response. However, in many situations some combinations of the components are not reasonable or may even cause a dangerous response.
- In this chapter, simplex lattice designs will be used when all combinations of the components are under consideration, while extreme vertices designs will be used when restrictions are placed on the proportions of the components.

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33.6 Simplex Lattice Designs for Exploring the Whole Simplex Region

- The simplex lattice designs (Scheffé 1958) address problems where there are no restrictions on the limits of the percentages of compounds comprising the total 100% composition.
- A simplex lattice design for q components consists of points defined by the coordinates (q, m), where the proportions assumed by each component take $m + 1$ equally spaced values from 0 to 1 and all possible combinations of the components are considered.
- Figure 33.6 illustrates pictorially the spatial test consideration of several lattice design altematives for three and four components.

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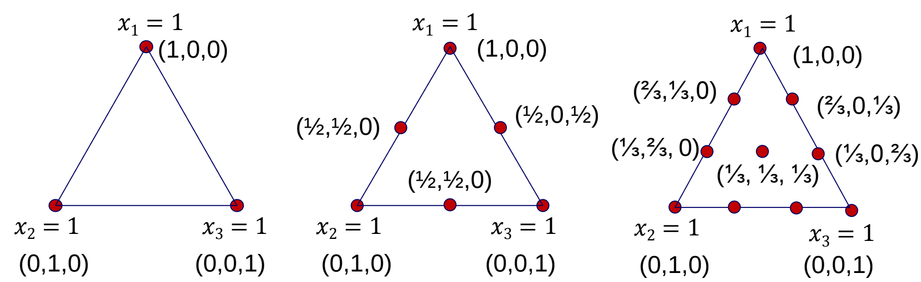
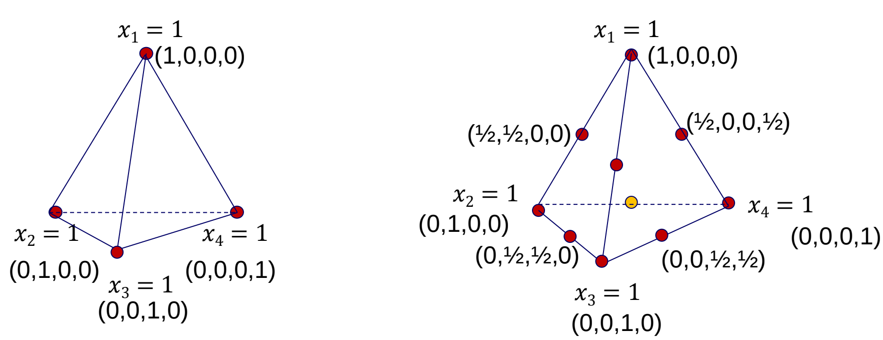
33.6 Simplex Lattice Designs for Exploring the Whole Simplex Region



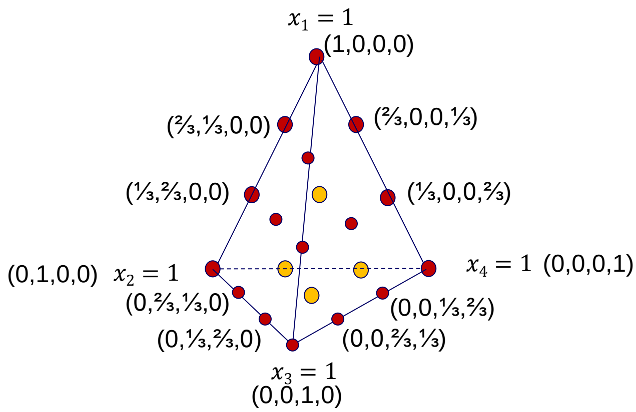
(3, 1) lattice **(3, 2) lattice** **(3, 3) lattice**

33.6 Simplex Lattice Designs for Exploring the Whole Simplex Region



(4, 1) lattice **(4, 2) lattice**

33.6 Simplex Lattice Designs for Exploring the Whole Simplex Region



(4, 3) lattice

33.6 Simplex Lattice Designs for Exploring the Whole Simplex Region

- Cornell (1983) notes that the general form of a regression function that can be fitted easily to data collected at the points of a (q, m) simplex lattice is the canonical form of the polynomial. This form is then modified by applying the restriction that the terms of a polynomial sum to 1.
- The simplified expression for three components yields the first-degree model form

$$y = b_1x_1 + b_2x_2 + b_3x_3$$

- The second-degree model form is

$$y = b_1x_1 + b_2x_2 + b_3x_3 + b_{12}\, x_1x_2 + b_{13}\, x_1x_3 + b_{23}\, x_2x_3$$

- The special cubic polynomial form is

$$y = b_1x_1 + b_2x_2 + b_3x_3 + b_{12}\, x_1x_2 + b_{13}\, x_1x_3 + b_{23}\, x_2x_3 + b_{123}\, x_1x_2x_3$$

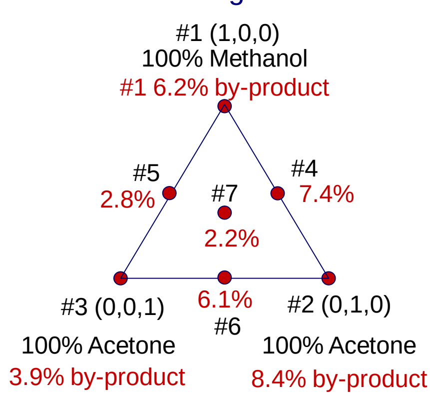
33.7 Example 33.2: Simplex Lattice Mixture Experiment

- Any one combination of three solvents could be most effective in the solvent rinse of a contaminating by-product (Diamond I989).
- A (3, 2) simplex lattice with a center point was chosen for the initial evaluation.
- The design proportions with the by-product responses are shown in Table 33.4.

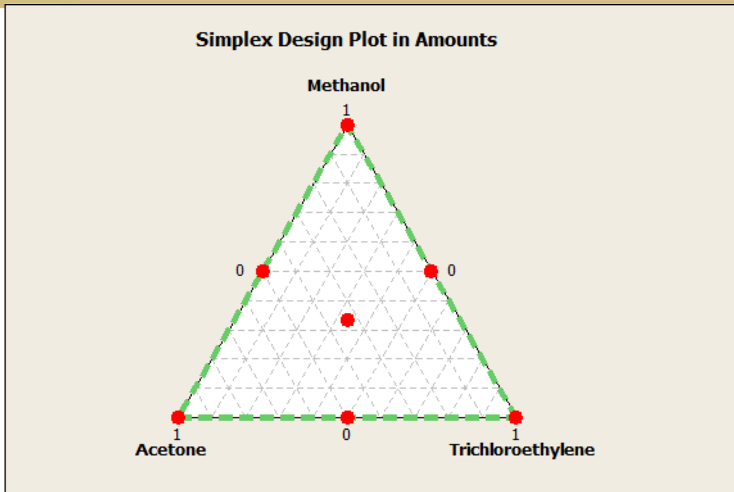
Trial	Methanol	Acetone	Trichloroethylene	By-product(%)
1	1	0	0	6.2
2	0	1	0	8.4
3	0	0	1	3.9
4	½	½	0	7.4
5	½	0	½	2.8
6	0	½	½	6.1
7	⅓	⅓	⅓	2.2

33.7 Example 33.2: Simplex Lattice Mixture Experiment

- A plot of the results is shown in Figure 33.7.



33.7 Example 33.2: Simplex Lattice Mixture Experiment



33.7 Example 33.2: Simplex Lattice Mixture Experiment

- A regression analysis for mixtures could be conducted, but in some cases, including this one, the conclusions are obvious.
- For this example, the best result is the center-point composition; however, there is curvature and a still better response is likely somewhere in the vicinity of this point.
- To reduce the by-product content amount of 2.2%, more experimental trials are needed near this point to determine the process optimum.
- Diamond (1989) chose to consider the following additional trials.
- These points are spatially shown in Figure 33.8, where the lines decrease in magnitude of 0.05 for a variable from an initial proportion value of 1.0 at the apex.

33.7 Example 33.2: Simplex Lattice Mixture Experiment

Regression for Mixtures: By-product(%) versus Methanol, Acetone, ...

Estimated Regression Coefficients for By-product(%) (component proportions)

Term	Coef	SE Coef	T	P	VIF
Methanol	6.40	2.332	*	*	1.599
Acetone	8.60	2.332	*	*	1.599
Trichloroethylene	4.10	2.332	*	*	1.599
Methanol*Acetone	-3.68	10.722	-0.34	0.790	1.569
Methanol*Trichloroethylene	-13.08	10.722	-1.22	0.437	1.569
Acetone*Trichloroethylene	-4.28	10.722	-0.40	0.758	1.569

S = 2.34132 PRESS = 2315.39
R-Sq = 83.53% R-Sq(pred) = 0.00% R-Sq(adj) = 1.20%

33.7 Example 33.2: Simplex Lattice Mixture Experiment

Analysis of Variance for By-product(%) (component proportions)

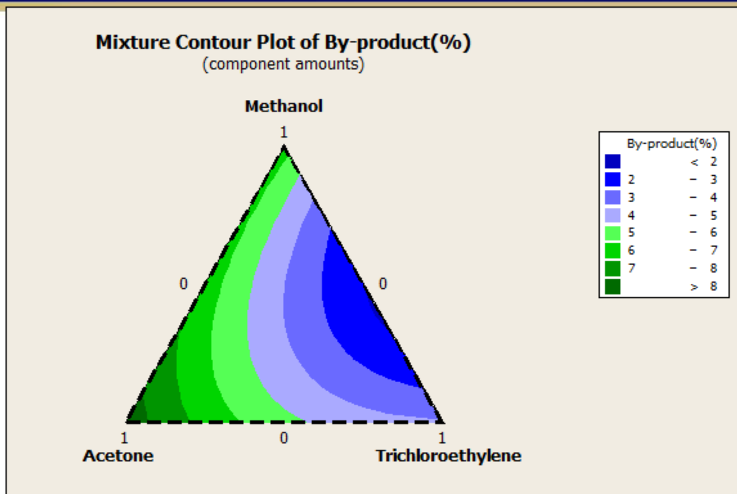
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	27.8068	27.8068	5.56136	1.01	0.634
Linear	2	18.6042	10.1267	5.06335	0.92	0.593
Quadratic	3	9.2026	9.2026	3.06755	0.56	0.726
Residual Error	1	5.4818	5.4818	5.48177		
Total	6	33.2886				

Unusual Observations for By-product(%)

Obs	StdOrder	By-product(%)	Fit	SE Fit	Residual	St Resid
1	1	6.200	6.404	2.332	-0.204	-1.00 X
2	2	8.400	8.604	2.332	-0.204	-1.00 X
3	3	3.900	4.104	2.332	-0.204	-1.00 X

X denotes an observation whose X value gives it large leverage.

33.7 Example 33.2: Simplex Lattice Mixture Experiment



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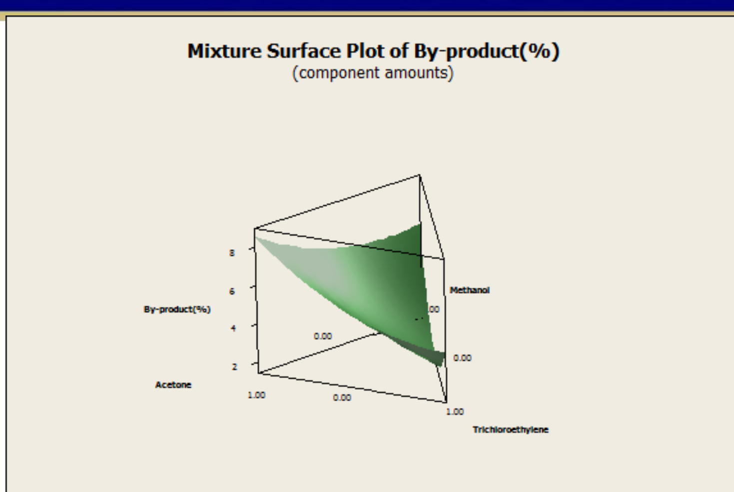
33.7 Example 33.2: Simplex Lattice Mixture Experiment



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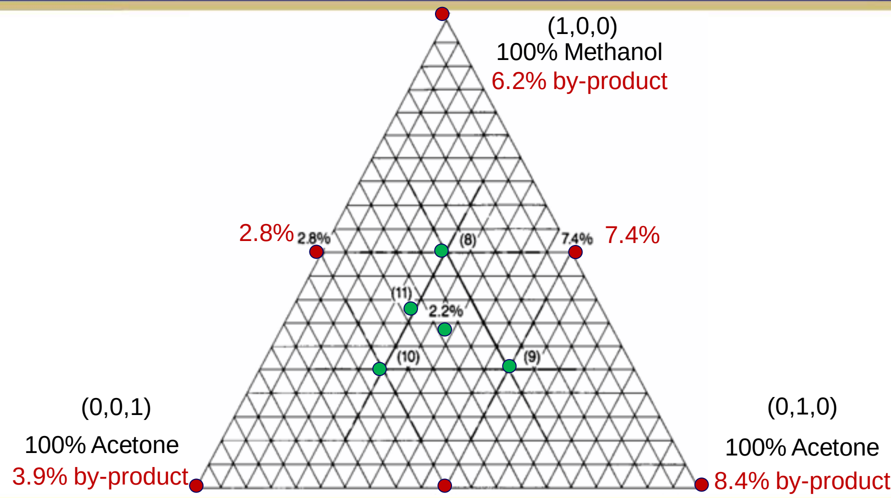
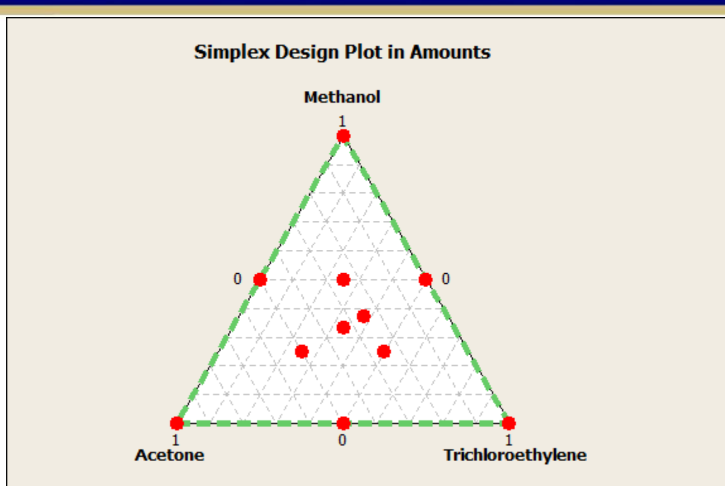
33.7 Example 33.2: Simplex Lattice Mixture Experiment



33.7 Example 33.2: Simplex Lattice Mixture Experiment



33.7 Example 33.2: Simplex Lattice Mixture Experiment

- Table 33.5 illustrates these trials and their rationales.

Exp. Point #	Rationale
8, 9, 10	(3,1) simplex lattice design vertices around the best response with the noted diagonal relationship to the original data points.
11	Because point #5 has the second best result, another data point was added in that direction.
12	Repeat the treatment combination that was the best in the previous experiment and is now the centroid of this follow-up experiment.

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33.7 Example 33.2: Simplex Lattice Mixture Experiment

- The results from these experimental trials are given in Table 33.6.

Trial	Methanol	Acetone	Trichloroethylene	By-product(%)
8	½	¼	¼	3.3
9	¼	½	¼	4.8
10	¼	¼	½	1.4
11	⅜	¼	⅜	1.2
12	⅓	⅓	⅓	2.4

- A plot of the data and an estimate of the response surface is shown in Figure 33.9.

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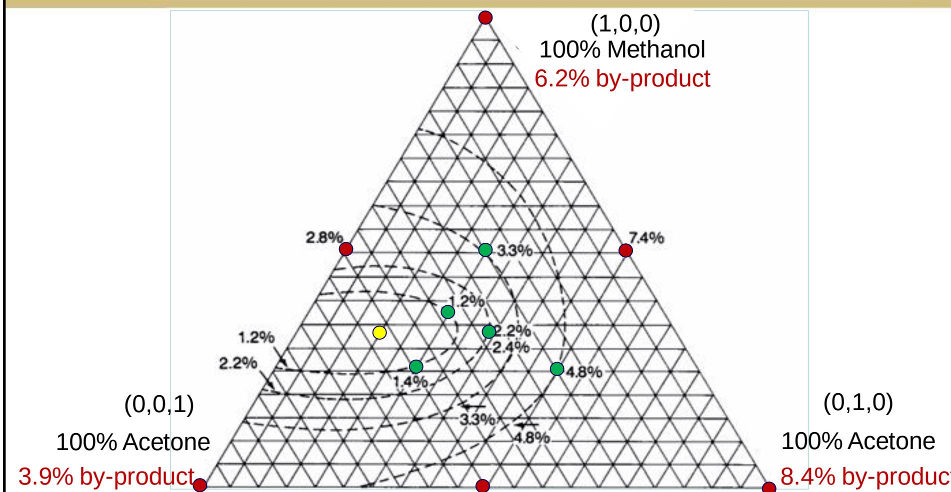
33.7 Example 33.2: Simplex Lattice Mixture Experiment



33.7 Example 33.2: Simplex Lattice Mixture Experiment

Regression for Mixtures: By-product(%) versus Methanol, Acetone, ...

Estimated Regression Coefficients for By-product(%) (component proportions)

Term	Coef	SE Coef	T	P	VIF
Methanol	6.58	1.371	*	*	2.188
Acetone	8.86	1.370	*	*	2.108
Trichloroethylene	4.13	1.371	*	*	2.188
Methanol*Acetone	-5.95	5.868	-1.01	0.350	2.348
Methanol*Trichloroethylene	-17.39	5.743	-3.03	0.023	2.438
Acetone*Trichloroethylene	-7.44	5.868	-1.27	0.252	2.348

S = 1.38681 PRESS = 842.844
R-Sq = 81.62% R-Sq(pred) = 0.00% R-Sq(adj) = 66.30%

33.7 Example 33.2: Simplex Lattice Mixture Experiment

Analysis of Variance for By-product(%) (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	5	51.2431	51.2431	10.24862	5.33	0.033
Linear	2	24.5430	11.3557	5.67785	2.95	0.128
Quadratic	3	26.7001	26.7001	8.90003	4.63	0.053
Residual Error	6	11.5394	11.5394	1.92323		
Lack-of-Fit	5	11.5194	11.5194	2.30388	115.19	0.071
Pure Error	1	0.0200	0.0200	0.02000		
Total	11	62.7825				

Unusual Observations for By-product(%)

Obs	StdOrder	By-product(%)	Fit	SE Fit	Residual	St Resid
2	2	8.400	8.856	1.370	-0.456	-2.13R
5	5	2.800	1.005	1.144	1.795	2.29R
6	6	6.100	4.631	1.179	1.469	2.01R

R denotes an observation with a large standardized residual.

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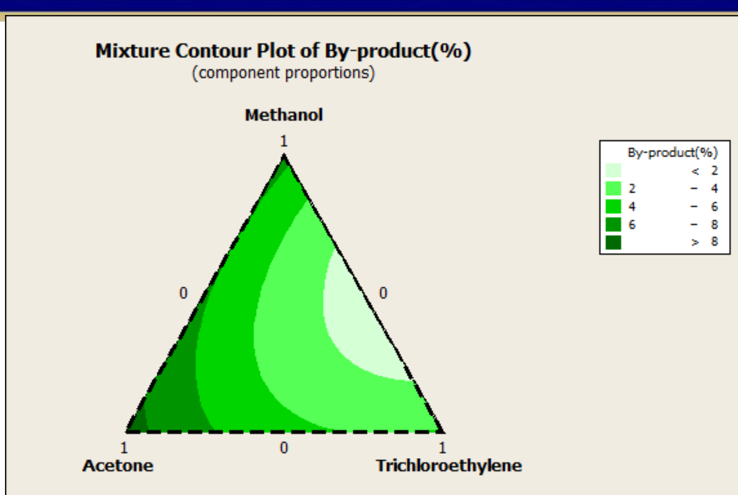
33.7 Example 33.2: Simplex Lattice Mixture Experiment



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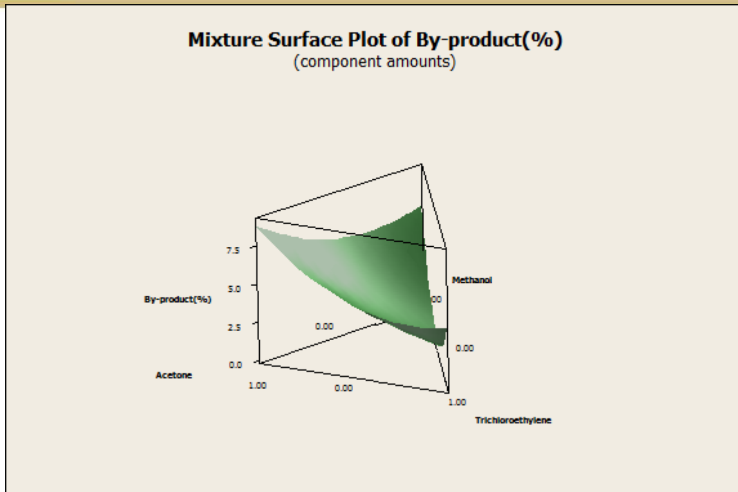
33.7 Example 33.2: Simplex Lattice Mixture Experiment



33.7 Example 33.2: Simplex Lattice Mixture Experiment

- The apparent minimum (shown as the point with no number in Figure 33.9) along with the results from an additional trial setting at

Trial	Methanol	Acetone	Trichloroethylene	By-product(%)
13	0.33	0.15	0.52	0.45

- Additional simplex design trials around this point could yield a yet smaller amount of by-product. However, if the by-product percentage is "low" enough, additional experimental trials might not serve any economic purpose.

33.8 Simplex Lattice Designs for Exploring the Whole Simplex Region

- Consider the situation where a response is a function, not only of a mixture, but also of its process variables (e.g., cooking temperature and cooking time).
- For the situation where there are three components to a mixture and three process variables, the complete simplex-centroid design takes the form shown in Figure 33.10.

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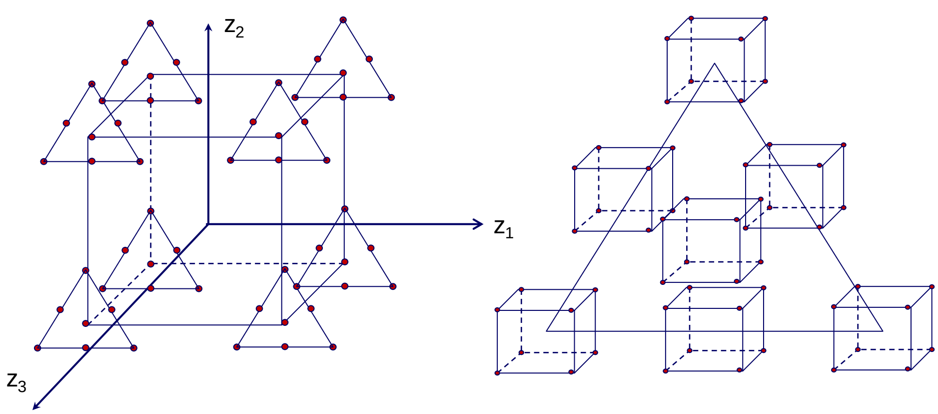
33.8 Simplex Lattice Designs for Exploring the Whole Simplex Region



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33.8 Simplex Lattice Designs for Exploring the Whole Simplex Region

- In general, the number of experimental trial possibilities can get very large when there are many variable considerations.
- Comell and Gorman (1984) discuss fractional factorial design alternatives. Comell (1990) discusses the embedding of mixture experiments inside factorial experiments.
- Algorithm designs, discussed in Section 33.12, can also reduce the number of test trials.

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33.9 Example 33.3: Mixture Experiment with Process Variables

- The data in Table 33.7 are the average of a replicated texture reading in kilogram force required to puncture fish patty surfaces (Comell 1981; Cornell and Gorman 1984: Gorman and Comell 1982) that were prepared under process conditions that had code values of -1 and +1 for
  - z_1: cooking temperature (-1 = 375°F, +1 = 425°F)
  - z_2: cooking time (-1 = 25 min, +1 = 40 min)
  - z_3: deep fat frying time (-1 = 25 sec, +1 = 40 sec)
- The patty was composed of three types of fish that took on composition ratios of 0, 1/3, 1/2, or 1.
- The fish designations are
  - x_1 : mullet
  - x_2: sheepshead
  - x_3: croaker

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33.9 Example 33.3: Mixture Experiment with Process Variables

Coded Process Variables			Mixture Composition (x_1, x_2, x_3)						
z_1	z_2	z_3	(1,0,0)	(0,1,0)	(0,0,1)	(½,½,0)	(½,0,½)	(0,½,½)	(⅓,⅓,⅓)
-1	-1	-1	1.84	0.67	1.51	1.29	1.42	1.16	1.59
1	-1	-1	2.86	1.10	1.60	1.53	1.81	1.50	1.68
-1	1	-1	3.01	1.21	2.32	1.93	2.57	1.83	1.94
1	1	-1	4.13	1.67	2.57	2.26	3.15	2.22	2.60
-1	-1	1	1.65	0.58	1.21	1.18	1.45	1.07	1.41
1	-1	1	2.32	0.97	2.12	1.45	1.93	1.28	1.54
-1	1	1	3.04	1.16	2.00	1.85	2.39	1.60	2.05
1	1	1	4.13	1.30	2.75	2.06	2.82	2.10	2.32

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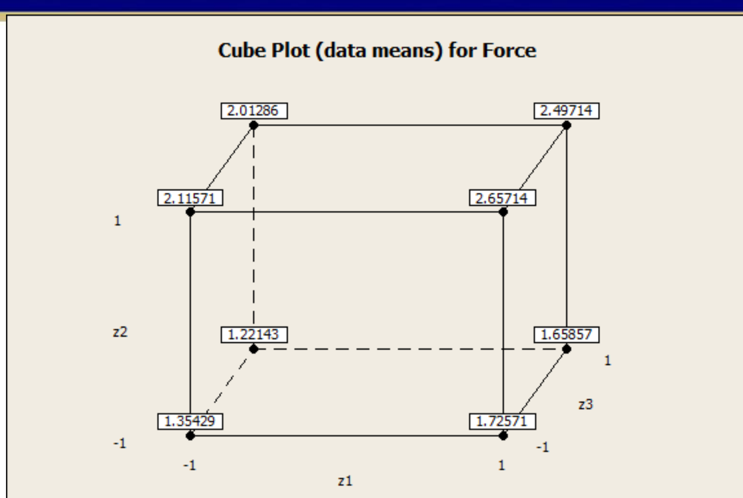
33.9 Example 33.3: Mixture Experiment with Process Variables



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33.9 Example 33.3: Mixture Experiment with Process Variables

Regression for Mixtures: Force versus x1, x2, x3, z1, z2, z3

Estimated Regression Coefficients for Force (component proportions)

Term	Coef	SE Coef	T	P	VIF
x1	2.8645	0.05203	*	*	1.599
x2	1.0745	0.05203	*	*	1.599
x3	2.0020	0.05203	*	*	1.599
x1*x2	-0.9742	0.23914	-4.07	0.000	1.569
x1*x3	-0.8342	0.23914	-3.49	0.001	1.569
x2*x3	0.3558	0.23914	1.49	0.147	1.569
x1*z1	0.4873	0.05203	9.37	0.000	1.599
x2*z1	0.1773	0.05203	3.41	0.002	1.599
x3*z1	0.2498	0.05203	4.80	0.000	1.599
x1*x2*z1	-0.8014	0.23914	-3.35	0.002	1.569
x1*x3*z1	-0.5314	0.23914	-2.22	0.033	1.569
x2*x3*z1	-0.1314	0.23914	-0.55	0.587	1.569

33.9 Example 33.3: Mixture Experiment with Process Variables

Regression for Mixtures: Force versus x1, x2, x3, z1, z2, z3

x1*z2	0.7086	0.05203	13.62	0.000	1.599
x2*z2	0.2561	0.05203	4.92	0.000	1.599
x3*z2	0.4036	0.05203	7.76	0.000	1.599
x1*x2*z2	-0.6614	0.23914	-2.77	0.009	1.569
x1*x3*z2	-0.1214	0.23914	-0.51	0.615	1.569
x2*x3*z2	-0.0064	0.23914	-0.03	0.979	1.569
x1*z3	-0.0878	0.05203	-1.69	0.101	1.599
x2*z3	-0.0803	0.05203	-1.54	0.133	1.599
x3*z3	0.0097	0.05203	0.19	0.853	1.599
x1*x2*z3	0.1055	0.23914	0.44	0.662	1.569
x1*x3*z3	-0.0195	0.23914	-0.08	0.935	1.569
x2*x3*z3	-0.1845	0.23914	-0.77	0.446	1.569

* NOTE * Coefficients are calculated for coded process variables.

S = 0.147710 PRESS = 2.28771

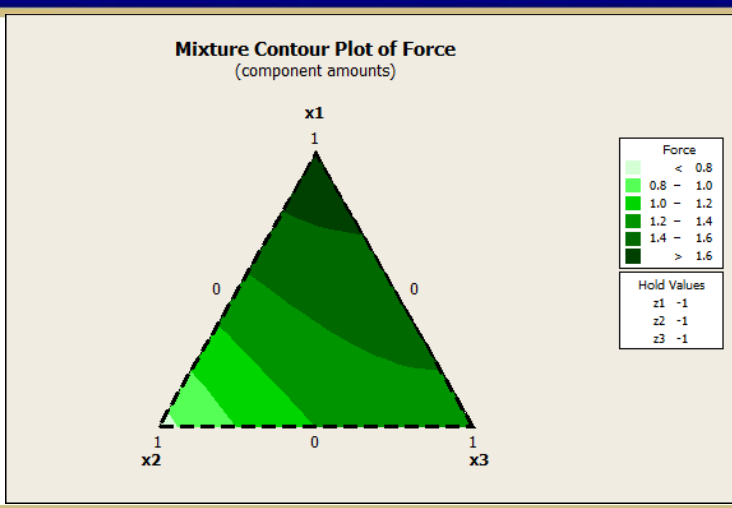
R-Sq = 97.68% R-Sq(pred) = 92.40% R-Sq(adj) = 96.01%

33.9 Example 33.3: Mixture Experiment with Process Variables

Analysis of Variance for Force (component proportions)

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	23	29.4142	29.4142	1.27888	58.62	0.000
Component Only						
Linear	2	14.0361	12.8220	6.41102	293.84	0.000
Quadratic	3	0.6729	0.6729	0.22430	10.28	0.000
Component* z1						
Linear	3	3.3169	2.7025	0.90084	41.29	0.000
Quadratic	3	0.3405	0.3405	0.11349	5.20	0.005
Component* z2						
Linear	3	10.6360	5.9602	1.98673	91.06	0.000
Quadratic	3	0.1703	0.1703	0.05678	2.60	0.069
Component* z3						
Linear	3	0.2234	0.1155	0.03850	1.76	0.174
Quadratic	3	0.0181	0.0181	0.00602	0.28	0.842
Residual Error	32	0.6982	0.6982	0.02182		
Total	55	30.1124				

33.9 Example 33.3: Mixture Experiment with Process Variables



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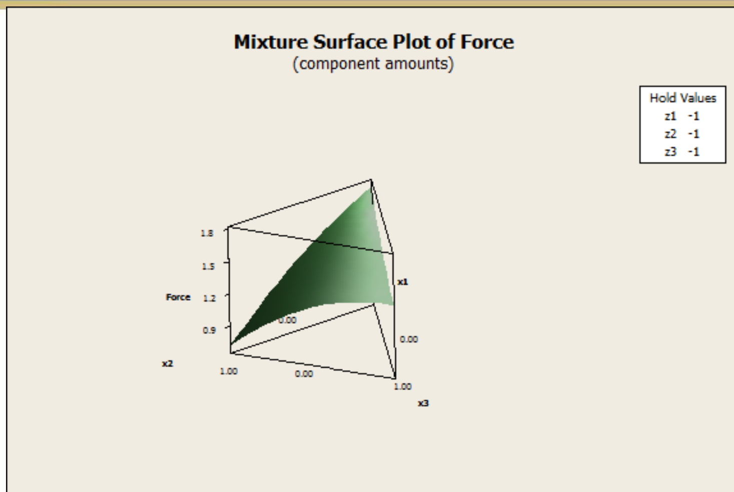
33.9 Example 33.3: Mixture Experiment with Process Variables



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33.9 Example 33.3: Mixture Experiment with Process Variables

- The desired range of fish texture (in the noted scaled units) for customer satisfaction is between 2.0 and 3.5. Other characteristics, not discussed here, were also considered in the actual experiment.
- A computer analysis of these data yielded the coefficient estimates shown in Table 33.8.

	Mean	z_1	z_2	z_3	z_1z_2	z_1z_3	z_2z_3	$z_1z_2z_3$	SE
x_1	2.87	0.49	0.71	-0.09	0.07	-0.05	0.10	0.04	0.05
x_2	1.08	0.18	0.25	-0.08	-0.03	-0.05	-0.03	-0.04	0.05
x_3	2.01	0.25	0.40	0.01	0.00	0.17	-0.05	-0.04	0.05
x_1x_2	-1.14	-0.81	-0.59	0.10	-0.06	0.14	-0.19	-0.09	0.23
x_1x_3	-1.00	-0.54	-0.05	-0.03	-0.06	-0.27	-0.43	-0.12	0.23
x_2x_3	0.20	-0.14	0.07	-0.19	0.23	-0.25	0.12	0.27	0.23
$x_1x_2x_3$	3.18	0.07	-1.41	0.11	1.74	-0.71	1.77	-1.33	1.65

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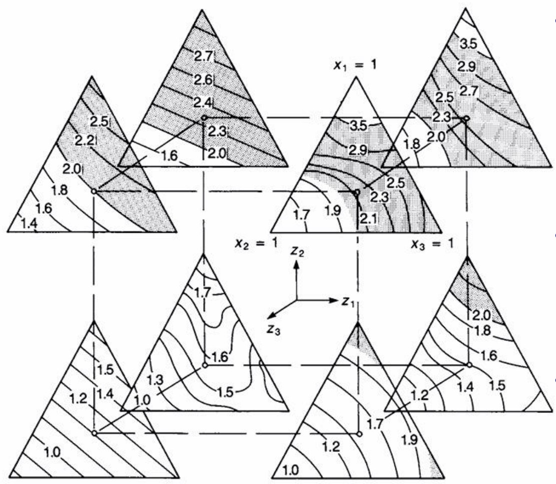
33.9 Example 33.3: Mixture Experiment with Process Variables



- Various x_1, x_2, and x_3 values are substituted to create a contour plot in a simplex coordinate system for each of the eight variable treatments.
- The shaded area in this figure shows when the desirable response range of 2.0 to 3.5 is achieved.
- This figure illustrates that a z_2 = 1 level (i.e., 40 min cooking time) is desirable.

33.9 Example 33.3: Mixture Experiment with Process Variables

- To maximize customer satisfaction, effort should be directed toward achieving the nominal criterion on the average with minimum variability between batches.
- It may be desirable to make the composition of the fish patty so that its sensitivity is minimized relative to deep fat frying time. To address this concern, it appears that a z_1 = -1 level (i.e., 375°F cooking temperature) may be most desirable with a relative high concentration of mullet in the fish patty composition.
- Other considerations to take into consideration when determining the “best” composition and variable levels are economics (e.g.. cost of each type of fish) and other experimental output response surface plots (e.g., taste evaluation).

33.10 Extreme Vertices Mixture Designs

- Extreme vertices designs can take on most of the nice properties of the matrix designs discussed above (Diamond 1989).
- This type of design is explained in the following example.

33.11 Example 33.4: Extreme Vertices Mixture Experiment

- A chemist wishes to develop a floor wax product. The following range of proportions of three ingredients is under consideration along with the noted proportion percentage limitations. The response to this experiment takes on several values: level of shine, scuff resistance, and so forth.
  - Wax: 0-0.25 (i.e., 0%-25%)
  - Resin: 0-0.20 (i.e., 0%—20%)
  - Polymer: 0.70-0.90 (i.e., 70%-90%)
- Again, mixture experiment trial combinations are determined by using a simplex coordinate system. This relationship is noted in Figure 33.12, where the lines leaving a vertex decrease by a magnitude of 0.05 proportion from an initial proportion value of 1.

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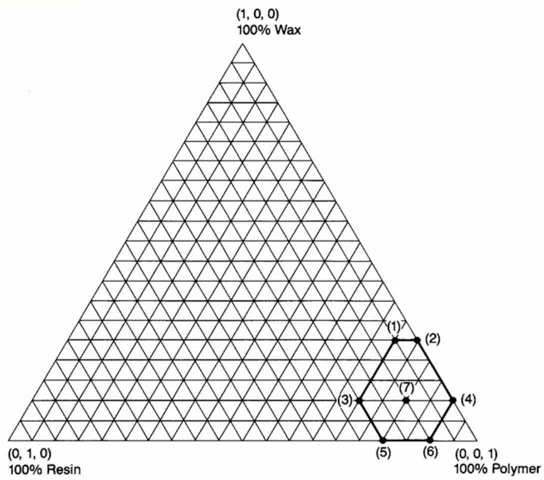
33.11 Example 33.4: Extreme Vertices Mixture Experiment



- The space of interest is noted by the polygon shown in the figure.

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33.11 Example 33.4: Extreme Vertices Mixture Experiment

- The space of interest is noted by the polygon shown in the figure.
- Table 33.9 shows test trials for the vertices along with a center point.

Trail	Wax(x_1)	Resin(x_2)	Polmer(x_3)	Response(y)
1	0.25	0.05	0.70	y_1
2	0.25	0.00	0.75	y_2
3	0.10	0.20	0.70	y_3
4	0.10	0.00	0.90	y_4
5	0.00	0.20	0.80	y_5
6	0.00	0.10	0.90	y_6
7	0.10	0.10	0.80	y_7

- The logic used in Example 33.1 for follow-up experiments can similarly be applied to this problem in an attempt to optimize the process.

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33.14 Additional Response Surface Design Considerations

- When no linear relationship exists between the regressors, they are said to be orthogonal.
- For these situations the following inferences can be made relatively easily:
  - Estimation and/or prediction.
  - Identification of relative effects of regressor variables.
  - Selection of a set of variables for the model.
- However, conclusions from the analysis of response surface designs may be misleading because of dependencies between the regressors.
- When near-linear dependencies exist between the regressors, multicollinearity is said to be prevalent.

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33.14 Additional Response Surface Design Considerations

- Other books (e.g., Montgomery and Peck 1982) discuss diagnostic procedures for this problem (e.g., variance inflation factor) along with other procedures used to better understand the output from regression analyses (e.g., detecting influential observations).
- Additional textbook design alternatives to the central composite and Box-Behnken designs are discussed in Cornell (1984), Montgomery (1997), and Khuri and Comell (1987).
- “Algorithm” designs can also be applied to non-mixture problems, as discussed in B. Wheeler (1989), where, as previously noted, algorithm designs are “optimized” to fit a particular model (e.g., linear or quadratic) with a given set of factor considerations.

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