Exercises for Chapter 12

12.1 You have a situation with three mixture components (X_1, X_2, and X_3) and no constraints.

a) List the experiments or mixtures that would be required to fit a special cubic model in the three components X_1, X_2, and X_3.

b) How would this list change if the components were subject to the following constraints:

$$0.20 \le X_1 \le 1.0$$
$$0.25 \le X_2 \le 1.0$$
$$0.15 \le X_3 \le 1.0$$

c) List the experiments required to fit the linear model in five components X_1, X_2, X_3, X_4, and X_5.

12.2 Soo, Sander, and Kess[1] Investigated the texture of shrimp patties made by blending proportions of isolated soy protein (X_1), sodium chloride (X_2), sodium tripolyphosphate (X_3), and Alaskan shrimp (X_4). The proportions of the ingredients were bounded by the constraints:

$$0.05 \le X_1 \le 0.10$$
$$0.01 \le X_2 \le 0.03$$
$$0.001 \le X_3 \le 0.005$$
$$0.85 \le X_4 \le 1.0$$

The experimental design and resulting data are shown below:

1 Soo, H.M., Sander, E.H., and Kess, D.W. (1978) "Definition of a Prediction Model for Determination of the Effect of Processing and Compositional Parameters on the Textural Characteristics of Fabricated Shrimp", *Journal of Food Science*, 43 pp. 1165-1171.

Run	ISP X_1	NaCl X_2	STP X_3	Shrimp X_4	Texture Y
1	0.10	0.03	0.005	0.865	9.88
2	0.10	0.03	0.001	0.869	9.35
3	0.10	0.01	0.005	0.885	9.85
4	0.10	0.01	0.001	0.889	9.65
5	0.05	0.03	0.005	0.915	9.35
6	0.05	0.03	0.001	0.919	7.90
7	0.05	0.01	0.005	0.935	7.65
8	0.05	0.01	0.001	0.939	7.85
9	0.10	0.02	0.003	0.877	9.75
10	0.075	0.03	0.003	0.892	8.03
11	0.075	0.02	0.005	0.900	8.03
12	0.075	0.02	0.001	0.904	8.60
13	0.075	0.01	0.003	0.912	8.05
14	0.05	0.02	0.003	0.927	7.65
15	0.075	0.02	0.003	0.902	8.18
16	0.10	0.03	0.005	0.865	9.60
17	0.10	0.01	0.005	0.885	9.55
18	0.05	0.03	0.001	0.919	7.72
19	0.05	0.01	0.001	0.939	7.63
20	0.075	0.02	0.003	0.902	8.48

a) Fit the Scheffé quadratic model to the data using regression

b) Using the coding and scaling: $X_1' = (X_1 - 0.075)/0.025$, $X_2' = (X_2 - 0.02)/0.01$, and $X_3' = (X_3 - 0.003)/0.002$, fit the slack variable model to the coded and scaled proportions.

c) Make effect plots for the 4 components and explain in words the effects of the components upon the temperature.

12.3 A mixture experiment in three components X_1, X_2, and X_3 was run.

a) If the fitted slack variable model is:

$Y = 12.0 + 3.1X_1 + 4.4X_2 + 0.7X^2{}_1 + 0.3X^2{}_2 + 1.4X_1X_2$,

then write the model in the Scheffé form.

b) What is the predicted response from the model given in a) at the 100% X_1 mixture.

c) Which components are synergistic (i.e., a mixture of the two results in a higher response than the pure blends).

12.4 Beloto, Jr. et al.[2] Studied the relation between Y= Solubility of phenobarbital and mixture components X_1 = ethanol, X_2 = propylene gycol and X_3 = water.

a) List the experiments required to fit a linear model

b) List the experiments needed to fit a quadratic model

c) list the experiments needed to fit a special cubic model

[2] Belloto Jr., R.J., Dean, A.M, Moustafa, M.A. , Molokhia, A.M., gouda, M. W., and Sokoloski, T.D. (1985), "Statistical Techniques Applied to Solubility Predictions and Pharmaceutical Formulations: An Approach to Problem Solving using Mixture Response Surface Methodology" *International Journal of Pharmaceutics* 23, pp. 195-207.

12.5 Narcy and Renaud[3] Studied mixtures of three components to see their effects on the viscosity and cold water clear point of a light-duty liquid detergent. Below is a list of some of their experiments and results.

#	Water X_1	Alcohol X_2	Urea X_3	Y_1 Viscosity	Y_2 Clear Point
1	1	0	0	362.5	35.0
2	0	1	0	78.0	11.3
3	0	0	1	1630.0	23.5
4	0.5	0.5	0	165	12.8
5	0.5	0	0.5	537.5	6.0
6	0	0.5	0.5	202.5	5.0
22	0.33	0.33	0.33	265.0	5.0

a) Fit the Scheffé linear model to the viscosity data.

b) Fit the Scheffé linear model to the clear point data.

c) Fit the Scheffé quadratic model to the viscosity data.

d) Fit the Scheffé quadratic model to the clear point data.

e) If Experiment 22 were replicated resulting in a viscosity of 285.1, calculate the pure error sum of squares and the lack of fit F-test for the quadratic model. Would you consider the quadratic model to be adequate?

f) Using the additional data value for viscosity given in e) calculate the coefficients in the special cubic model for viscosity.

[3] Narcy, J.P. and Renaud, J. (1972) "Use of Simplex Experimental Designs in Detergent Formulation", *Journal of the American Oil Chemists= Society* 49, pp. 598-608.

12.6 Use the data in Table 12.6 to answer the following questions.

a) Using the formulas in Table B.6-2, fit a quadratic model to the pseudo components of the data.

b) Convert the equation you found in Part a) relating the response to pseudo components (X_i')

into an equation relating Y to the actual components (X_i) using the transformation,

$X_i = (1 - \sum_{i=1}^{k} L_i) X_i' + L_i$.

12.7 Given the following experimental conditions and data:

#	X1	X2	X3	Y (replicates)
1	0.8	0.2	0.0	4, 6
2	0.3	0.7	0.0	8, 11
3	0.3	0.2	0.5	13, 9
4	0.55	0.45	0.0	15, 19
5	0.3	0.45	0.25	20, 23
6	0.55	0.2	0.25	18, 17
7	0.466	0.366	0.166	19, 22, 24, 17

a) Convert the components to pseudo components (X_i') and calculate the coefficients of the quadratic Scheffé model using the formulas shown in Table B.6-2

b) Using the transformation $X_i = (1 - \sum_{i=1}^{k} L_i) X_i' + L_i$ convert the equation you found in Part a) relating the response to pseudo components (X_i') into an equation relating Y to the actual components (X_i) .

c) Set up an X-matrix or use a regression computer program, and calculate the coefficients for the model you found in Part b) directly from the data in the table.

d) Using a computer program, or by repeated calculation of predicted values, sketch the contours of your fitted surface and find the combination of X_1, X_2, and X_3 that maximizes Y .

12.8 Anik and Sukumar[4] modeled the soluability of an antifungal agent in terms of the proportions of X_1 = polysorbate 600, X_2 = polyethylene glycol 400, X_3 =glycerin, and X_4 = water. If the constraints on the proportions are given as follows:

$$0.0 \leq X_1 \leq 0.08$$
$$0.1 \leq X_2 \leq 0.40$$
$$0.1 \leq X_3 \leq 0.40$$
$$0.2 \leq X_4 \leq 0.80$$

a) Use the algorithm of Snee and Marquardt described in Section 12.3.3.2 to find the extreme verticies for this constrained region.

b) List the experiments that you would use to fit a linear model

c) Compute the face centroids.

d) List the experiments you would run to fit a quadratic model.

[4] Anik, S. T. and Sukumar, L. (1981) "Extreme Vertexes Design in formulation Development: Solubility of Butoconazole Nitrate in a Multi-component System", *Journal of Pharmaceutical Sciences* 70, pp. 897-900.

12.9 Given the 13 motor octane values determined in the mixture screening design in Table 12.16

Run	Motor Octane
1	78
2	86
3	98
4	94
5	86
6	92.7
7	74
8	90
9	77
10	79
11	92
12	93
13	88

a) Use the regression procedure to determine the coefficients in a linear Sheffé Model.

b) Make an effect plot of the six components and determine if any can be dropped from the study.

12.10 Make an effect plots using the quadratic equations fit to the data from Problem 12.5 and explain in words the effects of the three components.

12.11 Given that six components of a mixture have the following constraints:

$$0.1 \le X_1 \le 0.2$$
$$0.1 \le X_2 \le 0.4$$
$$0.1 \le X_3 \le 0.5$$
$$0.0 \le X_4 \le 0.5$$
$$0.0 \le X_5 \le 0.6$$
$$0.0 \le X_6 \le 0.8$$

a) Set up a 12 run screening design

b) Set up a 16 run screening design

12.12 *Bohl[5] studied the strength characteristics of a plastic compound as a function of X_1 = virgin resin plus two additives X_2 = glass fiber, and X_3 = glass micro-spheres. The data from his experiment is shown in the table below. Two responses are shown (tensile strength and cost in cents per inch).*

	virgin resin	glass fiber	micro-spheres	tensile strength	material cost
run	X_1	X_2	X_3	Y_1	Y_2
1	1.0	0.00	0.0	100	100
2	0.9	0.00	0.10	177	100
3	0.9	0.10	0.0	86	94.1
4	0.86333	0.06666	0.06666	139	96.0
5	0.86333	0.06666	0.06666	137	96.0
6	0.8	0.0	0.2	217	100
7	0.8	0.10	0.10	148	94.1
8	0.8	0.20	0.0	79	88.1

[5] Bohl, A. H., "A Formulation Tool," *Chemtech*, May 1988, pp. 284 - 289.

a) Fit the quadratic Scheffé model for each response.
b) Perform a Lack-of-Fit test for the quadratic model of tensile strength.
c) Make an effect plot for each of the two responses
d) Overlay contour plots of the two responses (or use a nonlinear optimization program like the Solver in Excel) to predict the mixture proportions that would result in a tensile strength of at least 160 with a minimum cost.

12.13 Refer to Section 12.6.3 for the following instructions.

a) Include terms for $X_1X_2Z_1$. $X_1X_2Z_2$, and $X_1X_2Z_1Z_2$ to the X matrix, and fit the expanded model via regression.

b) Calculate the t-statistic for each term.

c) Calculate the F-statistic for the expanded model.