11.10 Exercises

1. A mixture experiment was run in three components: x_1, x_2, and x_3.

   (a) If the Scheffé model that best represented the data was $y = 13.5x_1 + 3.2x_2 + 9.8x_3$, then what is the predicted response at the pure blend $x_2 = 1.0$? What is the predicted response at the 50-50 blend of x_1 and x_2?

   (b) If the Scheffé model that best represented the data was $y = 13.5x_1 + 3.2x_2 + 9.8x_3 - 2.9x_1x_2 + 8.7x_1x_3 + 0.9x_2x_3$, and the goal was to increase the response y, then does blending x_1 and x_2 have a synergistic effect or an antagonistic effect? Does blending x_1 and x_3 have a synergistic effect or an antagonistic effect? What is the predicted response at the mixture of equal proportions for all three components?

2. Belloto *et al.* (1985) studied the relation between $y=$ Soluability of phenobarbital and mixture components x_1: ethanol, x_2: propylene gycol and x_3: water.

   (a) Use the `mixexp` package to list the experiments required to fit a linear Scheffé model.

   (b) Use the `mixexp` package to list the experiments required to fit a Scheffé quadratic model.

   (c) Use the `mixexp` package to list the experiments required to fit a special cubic model.

   (d) Use the `mixexp` package to list the experiments required to fit a full cubic model.

3. Cornell (2002) describes an experiment to make a fruit punch composed of three types of juice: x_1: watermellon, x_2: pineapple, and x_3: orange. The general acceptance of the fruit punch was to be determined by a taste panel that would rate them on a 1 to 9 scale where 1=dislike extremely, 5=neither like nor dislike, and 9=like extremely. If the proportions of the three juices are restricted by cost according to the inequalities below

$$0.20 \le x_1 \le 0.80$$
$$0.10 \le x_2 \le 0.40$$
$$0.10 \le x_3 \le 0.50$$

   (a) Graph the restricted experimental region within the simplex.

   (b) Create a design appropriate for fitting the Scheffé linear model.

   (c) Create a design appropriate for fitting the Scheffé quadratic model.

   (d) Is there any value in including an overall centroid or other interior points in the design?

4. In agricultural field tests, two or more herbicides are often mixed together in so-called tank mixes in order to find a mixture that is more effective than individual herbicides in controlling a multitude of pest weeds. In a specific test, various mixtures of x_1: a herbicide formulated to control broad leaf weeds, x_2: a herbicide formulated to control grass seedlings, and $x3$: a general-purpose herbicide were tested. The data from the tests are shown in Table 11.8. The response is the proportion of weeds controlled.

Table 11.8 *Data from Herbicide Tank Mix Experiment*

	Mixture Component			% Weed Control
Run	x_1	x_2	x_3	y
1	1	0	0	73
2	0	1	0	68
3	0	0	1	80
4	$\frac{1}{2}$	$\frac{1}{2}$	0	77
5	$\frac{1}{2}$	0	$\frac{1}{2}$	86
6	0	$\frac{1}{2}$	$\frac{1}{2}$	75
7	$\frac{1}{3}$	$\frac{1}{3}$	$\frac{1}{3}$	92
8	$\frac{1}{3}$	$\frac{1}{3}$	$\frac{1}{3}$	93
9	$\frac{1}{3}$	$\frac{1}{3}$	$\frac{1}{3}$	88

(a) Create the design shown in the table above using the `SLD` function in the `mixexp` package.

(b) Input the data and fit the Scheffé special cubic model.

(c) Make a contour plot of your fitted model and identify a mixture that would result in the highest % weed control.

5. Consider the problem discussed by Anik and Sukumar (1981). They studied the solubility of a butoconazole nitrate imidazole antifungal agent in a mixture of x_1: polyethylene glycol 400, x_2: glycerin, x_3: polysorbate 60, x_4: water, and x_5: poloxamer 407. Constraints on the mixture components are shown below.

$$0.10 \leq x_1 \leq 0.40$$
$$0.10 \leq x_2 \leq 0.40$$
$$0.00 \leq x_3 \leq 0.08$$
$$0.30 \leq x_4 \leq 0.70$$
$$x_5 = 0.10$$

(a) Given the data from Anik and Sukumar's (1981) experiments shown in Table 11.9, determine whether the Scheffé quadratic model in the four mixture components that are not constant can be fit to the data.

(b) Create a design that will allow fitting the quadratic model in x_1 to x_4.

Table 11.9 *Design and Response Data for Solubility Experiments*

Run	x_1	x_2	x_3	x_4	x_5	Solubility, mg/ml
vertices						
1	0.1	0.1	0.0	0.70	0.10	3.0
2	0.1	0.1	0.08	0.62	0.10	7.3
3	0.15	0.4	0.0	0.35	0.10	4.9
4	0.11	0.4	0.08	0.31	0.10	8.4
5	0.4	0.15	0.0	0.35	0.10	8.6
6	0.4	0.11	0.08	0.31	0.10	12.7
centroids						
7	0.1	0.1	0.04	0.66	0.10	5.1
8	0.4	0.13	0.04	0.33	0.10	10.8
9	0.13	0.4	0.04	0.33	0.10	6.6
10	0.216	0.216	0.0	0.468	0.10	4.4
11	0.203	0.203	0.08	0.414	0.10	7.9
12	0.255	0.255	0.08	0.31	0.10	9.4
13	0.275	0.275	0.0	0.35	0.10	5.8
overall centroid						
14	0.21	0.21	0.04	0.44	0.10	5.6

6. Consider the mixture experiment described by Anderson and McLean (1974). They presented a problem where the formula for a flare was obtained by mixing four chemicals x_1: magnesium, x_2: sodium nitrate, x_3: strontium nitrate, and x_4: binder. Constraints on the components were

$$0.40 \leq x_1 \leq 0.60$$
$$0.10 \leq x_2 \leq 0.50$$
$$0.10 \leq x_3 \leq 0.50$$
$$0.03 \leq x_4 \leq 0.08$$

   (a) Given the data from the experiments in Table 11.10, where the response is y=illumination, fit the Scheffé quadratic model to the data.
   (b) Fit the model in Equation (11.9) to the data.
   (c) Which model appears to fit best? Why? What does this imply?

Table 11.10 *Design and Response Data for Flare Experiments*

Run	x_1	x_2	x_3	x_4	y
1	0.4	0.1	0.47	0.03	75
2	0.4	0.1	0.42	0.08	480
3	0.6	0.1	0.27	0.03	195
4	0.6	0.1	0.22	0.08	300
5	0.4	0.47	0.1	0.03	145
6	0.4	0.42	0.1	0.08	230
7	0.6	0.27	0.1	0.03	220
8	0.6	0.22	0.1	0.08	350
9	0.5	0.1	0.345	0.055	220
10	0.5	0.345	0.1	0.055	260
11	0.4	0.2725	0.2725	0.055	190
12	0.6	0.1725	0.1725	0.055	310
13	0.5	0.235	0.235	0.03	260
14	0.5	0.21	0.21	0.08	410
15	0.5	0.2225	0.2225	0.055	425

7. Shumate and Montgomery (1996) developed a TiW plasma etch process for semiconductor manufacturing by studying a mixture of three gases x_1: SR_6, x_2: He, and x_3: N_2. The partial pressures of the three gases were forced to add to a constant value of total pressure to form the mixture constraint. The constraints in micrometers of pressure were

$$100 \leq SF_6 \leq 160$$
$$100 \leq He \leq 160$$
$$SF_6 + He + N_2 = 650$$

Expressing the components as mixture proportions, these constraints are

$$0.153846 \leq x_1 \leq 0.246154$$
$$0.153846 \leq x_2 \leq 0.246154$$

The authors created a design composed of the extreme vertices of the constrained region plus the overall centroid. Six responses were measured for each mixture and the table below shows two of the responses and their specifications.

Response	Name	Specification
y_2	Positive PR etch uniformity	< 2.5%
y_4	Negative PR etch uniformity	< 2.5%

(a) Create the design below using functions in the `mixexp` package and enter data for the two responses that are shown in Table 11.11.

Table 11.11 *Data from Plasma Etch Experiment*

x_1	x_2	x_3	y_2	y_4
0.153846154	0.153846154	0.692307692	0.90	0.93
0.153846154	0.153846154	0.692307692	0.94	0.98
0.246153846	0.246153846	0.507692308	1.82	1.49
0.246153846	0.246153846	0.507692308	1.8	1.64
0.246153846	0.153846154	0.600000000	7.14	7.35
0.246153846	0.153846154	0.600000000	7.10	7.27
0.153846154	0.246153846	0.600000000	2.62	2.16
0.153846154	0.246153846	0.600000000	2.73	1.88
0.153846154	0.200000000	0.646153846	3.01	2.70
0.200000000	0.153846154	0.646153846	4.50	4.25
0.246153846	0.200000000	0.553846154	4.79	4.50
0.200000000	0.246153846	0.553846154	2.88	2.36
0.200000000	0.200000000	0.600000000	1.22	1.54

(b) Fit the Scheffé quadratic model for both responses.

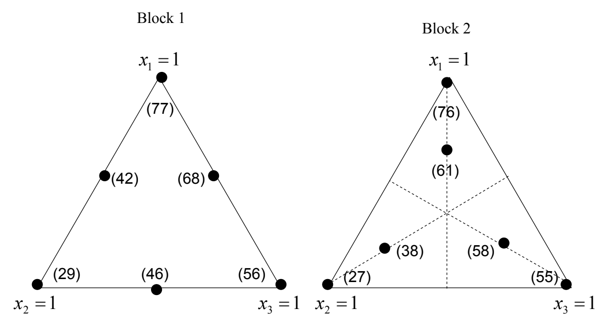
(c) Use the `ModelPlot` function in the `daewr` package to plot the contour at the specification boundary and contours at the specification boundary ± 0.5 for both responses in the pseudo component space.

(d) By examining your contour plots, find a region in the constrained space where the fitted models predict that both response specifications can be met.

8. If all three components in a mixture design have the same lower and upper

constraints (L=0.20, U=0.50), use Table 11.5 to create an orthogonally blocked mixture design, for fitting the Scheffé quadratic model, with two blocks of four mixtures in each block.

9. The numbers in parentheses in the figure below are the responses for the design shown in Figure 11.19.



 (a) Fit the Scheffé quadratic model to the data in the figure.
 (b) Verify that the design blocks orthogonally by checking to see that the type II and type III sum of squares for the quadratic cross product terms are the same, using the `Anova` function in the `car` package.

10. Table 11.12 shows the results of Chau and Kelly's (1993) mixture process variable experiments (MPV) with printable coating material, which were described in Section 11.7.

 (a) Enter the data for the design and response
 (b) Fit a simplified model of the form of Equation (11.31) with only one process variable z (..e., leave out product terms involving $z_l z_m$).
 (c) Does the process variable thickness affect the opacity?
 (d) Since the process variable can only be set to the low or high level, can you tell from the fitted equation what level of the process variable will result in the maximum opacity?
 (e) Simplify the equation you found in (b) by by fixing z=+1, and modifying the coefficients of x_1, x_2 and x_3. Use constrained optimization like the example on 486, to find the maximum opacity and the mixture that is predicted by the fitted model to result in that maximum.

Table 11.12 *Mixture Experiments with Printable Coating Material*

Run	x_1	x_2	x_3	z	Opacity
1	0.13	0.53	0.34	−1	0.698
2	0.13	0.53	0.34	−1	0.711
3	0.13	0.53	0.34	1	0.912
4	0.13	0.53	0.34	1	0.930
5	0.13	0.60	0.27	−1	0.700
6	0.13	0.67	0.20	−1	0.710
7	0.13	0.67	0.20	−1	0.680
8	0.13	0.67	0.20	1	0.908
9	0.13	0.67	0.20	1	0.901
10	0.29	0.37	0.34	−1	0.772
11	0.29	0.51	0.20	−1	0.772
12	0.45	0.21	0.34	−1	0.823
13	0.45	0.21	0.34	−1	0.798
14	0.45	0.21	0.34	1	0.992
15	0.45	0.28	0.27	−1	0.818
16	0.45	0.35	0.20	−1	0.802
17	0.45	0.35	0.20	1	0.976
18	0.45	0.35	0.20	1	0.940

11. Cornell (1988) presented the original example of mixture experiment for producing vinyl for automobile seat covers. The three mixture components were plasticizers x_1 through x_3, and the process variables were z_1 (rate of extrusion) and z_2 (temperature of drying). The response y is a scaled thickness value. The data is shown in Table 11.13, and is available in the data set `vinyl` in the `daewr` package.

    (a) Given that this is a cross between an extreme vertices design (plus the overall centroid appropriate for fitting a Scheffé linear model in the mixture components) and a 2^2 factorial in the process variables, what is the appropriate model for this data?

    (b) Recall the data from `dawer` and fit the model by least squares using the R `lm` function.

    (c) In this experiment, the runs were not made in a completely random order, but rather randomized within whole plots and then run as a split-plot experiment. Refit the model using the `lmer` in the R package `lme4` including a random term for whole plots like the example shown in Section 11.8. Is there any difference between the parameter estimates and the significance tests you get in (b) and (c)? What does this imply?

Table 11.13 *Data for Mixture Experiments for Producing Vinyl*

				z_1:	1	−1	−1	1
				z_2:	−1	1	−1	1
Whole Plot	x_1	x_2	x_3					
1	0.85	0	0.15		8	12	7	12
2	0.72	0	0.28		6	9	7	10
3	0.6	0	0.15		10	13	9	14
4	0.47	0.25	0.28		4	6	5	6
5	0.66	0.125	0.215		11	15	9	13
6	0.85	0	0.15		7	10	8	11
7	0.72	0	0.28		5	8	6	9
8	0.6	0	0.15		11	12	10	12
9	0.47	0.25	0.28		5	3	4	5
10	0.66	0.125	0.215		10	11	7	9