

## CUTTING EXPERIMENTAL DESIGNS INTO BLOCKS

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### Summary

Most experimental material in agriculture and industry is heterogeneous in nature and therefore its statistical analysis benefits from blocking. Many experiments are restricted in time or space, and again blocking is useful. This paper adopts the idea of orthogonal blocking of Box & Hunter (1957) and applies it to optimal blocking designs. This approach is then compared with the determinant-based approach described in the literature for constructing block designs.

*Key words:* A-optimality; D-optimality; fractional factorial designs; interchange algorithm; mixture designs; optimality; orthogonal arrays; orthogonal blocking; response surface designs.

### 1. Introduction

Of Fisher's three principles of design of experiments, i.e. randomization, replication and blocking, blocking is the most difficult because it places special constraints on experimental designs. It is well known that proper blocking reduces experimental error. Reduced error makes an experiment more sensitive in detecting significance of effects, so less experimentation may be necessary. The catalogues and determinant-based algorithms produced by Atkinson & Donev (1992 Chapter 15) and Cook & Nachtshiem (1989) for constructing block designs have helped scientists and engineers to find suitable blocked designs. However, there are situations in which this is not the case. Consider the following three examples.

1. A study is developing antioxidants from naturally occurring substances in milk. An experiment is conducted to test the antioxidant activity of heated casein–sugar mixtures as a function of initial pH, casein concentration and sugar concentration. For ease of setting factor levels, pH is set at three levels (6.8, 7.8 and 8.8), casein is set at three levels (5, 7.5 and 10%) and sugar is set at three levels (0, 2.5 and 5%). There are three batches of milk from three factories which can accommodate 9–10 runs each. The three-factor central-composite design (CCD) in three blocks of sizes (6, 6, 8) of Box & Hunter (1957 Table 4) is a possibility. However, the scientist objects to the unequal block sizes and asks, if possible, that all combinations of the three factors be included. Thus one needs to block a  $3^3$  factorial. One of the blocking algorithms from the papers mentioned above could be employed, except that there is no guarantee that all runs of the  $3^3$  factorial would appear.
2. Cook & Nachtshiem (1989) discuss an experiment to study the main effects and two-factor interactions of four process variables on the texture of a finished food product with 18 runs in three days (blocks). The blocks of their design solution obtained by selecting 18 runs

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from a candidate set of  $3 \times 2^4$  runs by the  $D$ -optimality criterion are:

	b	d	c	ab	ad	abcd
(1)	ac	abc	abd	acd	bcd	
	a	ac	bc	bd	cd	abcd

It can be seen that factor A is partially confounded with the second block because four runs are made at high level of A but only two runs are made at low level of A. Similarly, factor C is partially confounded with each of the three blocks. The food scientist is more interested in the main effects than two-factor interactions and wants an alternative design in which the main effects are orthogonal to blocks. As the determinant-based algorithms cannot generate the design with this constraint, we may wonder whether such a design exists.

3. Draper *et al.* (1993) describe a bread-making experiment at Spillers Milling Ltd, UK. Their solution for this experiment is a Latin square-based four-component mixture design in four blocks of nine runs each (Draper *et al.*, 1993 Table 2). This design is  $D$ -optimal and orthogonally blocked. However, among the 36 blends suggested, only 13 are distinct and 12 of the 13 are only binary blends plus a four-component blend (see comment at the bottom of Draper *et al.*, 1993 p. 270). No three-component blends are suggested. Despite the design's high  $|X^T X|$  value, the power of the test for lack of fit of the second-degree would be improved by the presence of some three-component blends and therefore we should insist on their inclusion in this experiment. Can a new design with three-component blends added be orthogonally blocked?

In this paper, we apply the approach of orthogonal blocking of the response surface designs (RSDs) of Box & Hunter (1957) to optimal blocking of other types of designs such as fractional factorials and mixture designs. We discuss solutions for the aforementioned examples given by this approach. Whenever possible, we compare these solutions with those constructed by the determinant-based approach in terms of  $A$ - and  $D$ -optimality and a measure of orthogonality of a blocked design which is defined in the next section.

### 2. A general approach to blocking

Let  $(z_{i1}, \dots, z_{ib}, x_{i1}, \dots, x_{im}, \dots, x_{i(p-b)})$  denote the  $i$ th row of the extended design matrix  $X$  for  $n$  runs in  $b$  blocks of sizes  $n_1, n_2, \dots, n_b$  involving  $b$  block variables and  $p - b$   $x$ -variables ( $m$  main-effect variables and  $p - b - m$  derived variables). The derived variables include  $\binom{m}{2}$  two-factor interaction terms and  $m$  squared terms for the second-order response surface model (example 1),  $\binom{m}{2}$  two-factor interaction terms for a factorial model with interactions (example 2), and a mixture model (example 3). This paper assumes all models have the usual independent and identically distributed  $N(0, \sigma^2)$  error terms.

The block variable  $z_{iw}$  ( $w = 1, \dots, b$ ) is a dummy variable taking value 1 if the run  $i$  belongs to block  $w$  and 0 otherwise. If possible, the runs should be allocated to blocks such that each non-block variable is orthogonal to the block variables.

Partition  $X$  as  $[Z \ X]$ . Then  $X^T X = \begin{bmatrix} Z^T Z & Z^T X \\ X^T Z & X^T X \end{bmatrix}$ , and  $Z^T X$  can be written as

$$\begin{bmatrix} s_{11} & s_{12} & \cdots & s_{1(p-b)} \\ s_{21} & s_{22} & \cdots & s_{2(p-b)} \\ \vdots & \vdots & & \vdots \\ s_{b1} & s_{b2} & \cdots & s_{b(p-b)} \end{bmatrix}, \tag{1}$$

where  $s_{wj}$  is the contribution of block  $w$  to  $\sum_{i=1}^n x_{ij}$ , i.e.  $\sum_{w=1}^b s_{wj} = \sum_{i=1}^n x_{ij}$ . The condition for a non-block variable  $j$  to be orthogonal to the block variables is  $s_{wj}^* = s_{wj} - (n_w/n) s_{.j} = 0$ ; where  $s_{.j} = \sum_{w=1}^b s_{wj}$  (see Box & Hunter, 1957 Equation 84). Basically, this condition says that the contribution of block  $w$  to  $\sum_{i=1}^n x_{ij}$  must be proportional to the block size  $n_w$ . A design is said to be orthogonally blocked when each non-block variable is orthogonal to the block variables. For an orthogonally blocked design, the inclusion of blocks does not affect the estimated regression coefficients of the  $x$ -variables and so the only effect of blocking is to reduce the magnitude of the experimental error (Box & Hunter, 1957 p. 228).

To use the idea of orthogonal blocking of Box & Hunter (1957), first construct a suitable unblocked design and then allocate the runs of this design to blocks such that  $f = \sum s_{wj}^{*2}$  is minimized. A blocking algorithm called CUT that implements this approach is outlined in the Appendix. The next paragraph shows that minimizing  $f$  also results in designs that are good with respect to the  $D$ - and  $A$ -optimality criteria.

Let  $X_c = XT = [Z \ X]T = [Z \ X_c]$  where  $X_c$  is the centred  $X$  matrix and

$$T = \begin{bmatrix} I_b & -U \\ \mathbf{0} & I_{p-b} \end{bmatrix}$$

with  $U$  the  $b \times (p-b)$  matrix with columns equal to the mean of the corresponding column of  $X$ . Let  $M = X_c^T X_c$ . Without loss of generality, assume that  $M$  is of full rank. Let  $\lambda_1, \dots, \lambda_p$  be the eigenvalues of  $M$ . Since  $\text{trace}(M) = \sum \lambda_i = \text{constant}$ , and  $\text{trace}(M^2) = \sum \lambda_i^2$ , minimizing  $f = \sum s_{wj}^{*2}$  (i.e. minimizing the sum of squares of the elements of  $Z^T X_c$ ) which is equivalent to minimizing  $\text{trace}(M^2)$  is the same as making the  $\lambda_i$  as equal as possible with  $\sum \lambda_i = \text{constant}$ . The proposed criterion is an approximation of the  $A$ -optimality criterion which requires the minimization of  $\sum \lambda_i^{-1}$  ( $= \text{trace}(M^{-1})$ ), or the  $D$ -optimality criterion which requires the maximization of  $\prod \lambda_i$  ( $= |M|$ ) (see Kiefer, 1959). In a sense, it is closely allied to the  $(M, S)$ -optimality criterion introduced by Eccleston & Hedayat (1974) in the incomplete block designs settings.

**Remarks**

1. Partition  $(X^T X)^{-1}$  as

$$(X^T X)^{-1} = \begin{bmatrix} C_{11} & C_{12} \\ C_{21} & C_{22} \end{bmatrix}$$

where  $C_{11}$  is the covariance matrix of the estimated regression coefficients of the  $b$  block variables and  $C_{22} = (X^T X - X^T Z(Z^T Z)^{-1} Z^T X)^{-1}$  is the covariance matrix of the  $p - b$   $x$ -variables (assuming  $\sigma^2 = 1$ ). Let  $M^{-1}$  be accordingly partitioned as

$$M^{-1} = \begin{bmatrix} C_{11}^* & C_{12}^* \\ C_{21}^* & C_{22}^* \end{bmatrix}$$

where  $C_{22}^* = (X_c^T X_c - X_c^T Z(Z^T Z)^{-1} Z^T X_c)^{-1}$ . It can be shown that  $C_{22}^* = C_{22}$ . Therefore, minimizing  $\text{trace}(M^{-1})$  results in minimizing  $\text{trace}(C_{22})$ , i.e. the sum of the variances of the estimated regression coefficients of the  $p - b$  non-block variables.

2. Since  $|M| = |X_c^T X_c| = |(XT)^T(XT)| = |X^T X| = |Z^T Z| |C_{22}^{-1}|$  and since for fixed block sizes  $|Z^T Z|$  is a constant, maximizing  $|M|$  is the same as minimizing the determinant of the covariance matrix of the estimated regression coefficients of the  $p - b$   $x$ -variables.

TABLE 1  
Experimental designs for Example 1

(A)						(B)											
Block 1		Block 2		Block 3		Block 1		Block 2		Block 3							
-1	-1	1	-1	-1	0	-1	-1	-1	0	0	1	-1	0	0	0	1	0
-1	0	1	-1	0	-1	-1	0	0	-1	1	1	1	1	-1	-1	-1	1
-1	1	-1	-1	1	0	-1	1	1	1	-1	-1	0	0	-1	0	0	-1
0	-1	-1	0	-1	1	0	-1	0	-1	-1	-1	-1	-1	-1	-1	-1	0
0	0	-1	0	0	0	0	0	1	-1	-1	1	1	-1	1	1	-1	-1
0	1	0	0	1	1	0	1	-1	1	1	-1	0	1	0	-1	1	-1
1	-1	0	1	-1	-1	1	-1	1	0	-1	-1	0	-1	1	1	0	1
1	0	0	1	0	1	1	0	-1	-1	1	-1	1	0	-1	1	-1	-1
1	1	1	1	1	-1	1	1	0	1	1	1	-1	1	1	1	1	1

3. When there is no blocking (i.e.  $Z = \mathbf{1}_n$ ) or when the design is orthogonally blocked (i.e.  $Z^T X_c = \mathbf{0}_{b \times (p-b)}$ ),  $C_{22}$  becomes  $(X_c^T X_c)^{-1}$ . We therefore define the following measure of orthogonality called the block factor of a blocked design:

$$BF = \left( \frac{|X^T X| / |Z^T Z|}{|X_c^T X_c|} \right)^{1/(p-b)}.$$

The maximum value of  $BF$  is 1 which occurs when there is no blocking or when the design is orthogonally blocked.

### 3. Discussion

We now discuss the solutions obtained by the CUT algorithm for the examples given in the Introduction.

#### 3.1. Example 1

In Table 1, (A) shows a three-factor second order RSD for the casein–sugar mixture example. This orthogonally blocked design was obtained by dividing the  $3^3$  factorial into three blocks. Blocks 1 and 3 of (A) each have three corner points, three edge centres and three face centres. Block 2 has a centre point, two corner points and six edge centres. Let  $D = |M|$  and  $T = \text{trace}(C_{22})$ . Design (A) has  $BF = 1$ ,  $D = 1.587 \times 10^{12}$  and  $T = 0.9167$ .

Design (B) is a non-orthogonal blocked design with only 19 distinct points, constructed by implementation of the Cook & Nachtsheim (1989) algorithm (see Miller & Nguyen, 1994). This design has  $BF = 0.994$ ,  $D = 2.729 \times 10^{12}$  and  $T = 1.007$ .

The variances of the terms  $x_1, x_2, x_3, x_1x_2, x_1x_3, x_2x_3, x_1^2, x_2^2, x_3^2$  of (A) are 0.056, 0.056, 0.056, 0.083, 0.083, 0.083, 0.167, 0.167 and 0.167 and for (B) they are 0.0501, 0.049, 0.049, 0.060, 0.060, 0.060, 0.223, 0.231 and 0.223. Unlike (B), the covariances of mentioned terms of (A) are all 0.

#### 3.2. Example 2

To construct a design for the second example, augment a  $2^4$  factorial with a fold-over pair of treatments, say (1) and abcd. The partition of the 18 points into three blocks is:

ab	ac	bc	ad	bd	cd
(1)	(1)	abc	abd	bcd	abcd
a	b	c	d	abcd	abcd.

TABLE 2  
*Designs for Example 3*

(A)				(B)			
Block 1				Block 1			
0.00	0.00	0.25	0.75	0.00	0.05	0.70	0.25
0.00	0.75	0.00	0.25	0.00	0.25	0.70	0.05
0.25	0.00	0.00	0.75	0.00	0.70	0.05	0.25
0.25	0.00	0.75	0.00	0.05	0.00	0.70	0.25
0.75	0.00	0.00	0.25	0.05	0.25	0.00	0.70
0.75	0.25	0.00	0.00	0.05	0.70	0.25	0.00
0.00	0.25	0.50	0.25	0.25	0.00	0.05	0.70
0.00	0.50	0.25	0.25	0.25	0.05	0.00	0.70
0.25	0.00	0.50	0.25	0.25	0.70	0.00	0.05
0.25	0.25	0.50	0.00	0.70	0.00	0.25	0.05
0.25	0.50	0.00	0.25	0.70	0.05	0.25	0.00
0.25	0.50	0.25	0.00	0.70	0.25	0.05	0.00
Block 2				Block 2			
0.00	0.00	0.75	0.25	0.00	0.05	0.25	0.70
0.00	0.25	0.00	0.75	0.00	0.25	0.05	0.70
0.00	0.25	0.75	0.00	0.00	0.70	0.25	0.05
0.00	0.75	0.25	0.00	0.05	0.00	0.25	0.70
0.25	0.75	0.00	0.00	0.05	0.25	0.70	0.00
0.75	0.00	0.25	0.00	0.05	0.70	0.00	0.25
0.00	0.25	0.25	0.50	0.25	0.00	0.70	0.05
0.25	0.00	0.25	0.50	0.25	0.05	0.70	0.00
0.25	0.25	0.00	0.50	0.25	0.70	0.05	0.00
0.50	0.00	0.25	0.25	0.70	0.00	0.05	0.25
0.50	0.25	0.00	0.25	0.70	0.05	0.00	0.25
0.50	0.25	0.25	0.00	0.70	0.25	0.00	0.05

Note that (1) appears twice in block 2 and abcd appears twice in block 3. All main effects of this design are orthogonal to blocks (in each block, each factor has three runs at high level and three runs at low level). The  $Z^T X$  matrix of this design is as follows:

A	B	C	D	AB	AC	AD	BC	BD	CD
0	0	0	0	2	2	2	2	2	2
0	0	0	0	2	2	2	2	2	2
0	0	0	0	2	2	2	2	2	2

This design has  $BF = 0.950$ ,  $D = 3.562 \times 10^{14}$ ,  $T = 0.604$  while the corresponding Cook & Nachtsheim (1989) design has  $BF = 0.959$ ,  $D = 3.942 \times 10^{14}$  and  $T = 0.605$ .

**3.3. Example 3**

The designs given in Table 2 are two alternative orthogonally blocked four-component mixture designs constructed by CUT. Each design is obtained by dividing 24 distinct blends into two blocks. Design (A) has 12 distinct binary blends (0.00, 0.00, 0.25, 0.75), (0.00, 0.00, 0.75, 0.25), etc., and 12 three-component blends (0.00, 0.25, 0.25, 0.50), (0.00, 0.25, 0.50, 0.25), etc. Design (B) has 24 three-component blends (0.00, 0.05, 0.25, 0.70), (0.00, 0.05, 0.70, 0.25), etc.

**Remarks**

1. Design (B) is not isomorphic to the Latin square-based design in Draper *et al.* (1993 Table 6) obtained by equating  $(a, b, c, d)$  to (0.00, 0.05, 0.25, 0.70) because the latter has only 16 distinct blends.

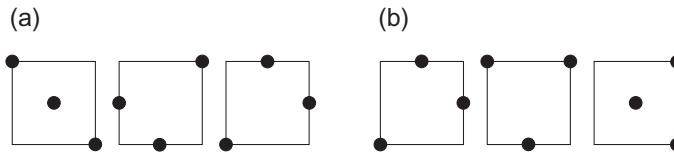


Figure 1.

2. The determinant-based approach does not offer any solution for this example as  $D = 0$  in this case.
3. It is conventional to add a centroid blend to each block. In our four-component example, the centroid blend is  $(0.25, 0.25, 0.25, 0.25)$ . To analyse these blocked mixture designs, reduce the number of block terms by one (see Cornell, 1990; or Draper *et al.*, 1993).
4. Designs with four blocks can be obtained by duplicating (A) (or B)).

A referee has pointed out that, in the first two examples, designs constructed by the determinant-based approach increased the values of both  $D$  and  $T$  (these designs are more  $D$ -optimal but less  $A$ -optimal than CUT designs). Minimizing  $f = \sum s_{w_j}^2$  does not automatically maximize  $D$  (or  $BF$ ) at the cost of maximizing  $T$ . Although this observation cannot be generalized, it is true in several other examples and here is one of them.

Atkinson & Donev (1992 Figure 13.1) show a two-factor second order RSD in three blocks each of size three with  $BF = 0.914$ ,  $D = 8064$  and  $T = 2.035$  (see Figure 1(b)). Figure 1(a) is a graphical display of the partition of the  $3^2$  factorial into three blocks by CUT.

The CUT design has  $BF = 0.871$ ,  $D = 7776$  and  $T = 1.833$ . The terms  $x_1$ ,  $x_2$ ,  $x_1x_2$ ,  $x_1^2$ ,  $x_2^2$  of CUT design are orthogonal to one another and terms  $x_1$ ,  $x_2$ ,  $x_1^2$ ,  $x_2^2$  of CUT design are orthogonal to blocks. The corresponding design of Atkinson & Donev (1992 Figure 13.1) has none of these desirable features. Note that for this design, if block effects are not real and are excluded from the model, i.e. there is only one block instead of three blocks, the  $3^2$  factorial has  $D = 5184$  and  $T = 1.583$  while the points in the design of Atkinson & Donev (1992 Figure 13.1) have  $D = 4224$  and  $T = 1.951$ .

#### 4. Concluding remarks

This paper uses the idea of orthogonal blocking of Box & Hunter (1957) to develop a blocking algorithm called CUT. Examples have illustrated the performance of the CUT algorithm and shown that this algorithm is a good supplement to existing blocking algorithms. Another way of testing CUT's performance is to shuffle all the runs of any of the orthogonally blocked designs in the literature and use CUT to rearrange these runs into appropriate blocks so that the orthogonal blocking condition is obtained. The CUT algorithm has been successfully tried with all blocked CCDs of Box & Hunter (1957), blocked Box–Behnken designs of Box & Behnken (1960), blocked mixture designs in Cornell (1990 Section 8.1) and Draper *et al.* (1993), blocked fractional factorials in Bisgaard (1994 Table 1a and 1b) without the help of the blocking generators used in these tables. An additional use of CUT is to construct new orthogonal arrays (OAs) from existing OAs. For example,  $L_{36}(6^3 \cdot 2)$  and  $L_{36}(6^3 \cdot 3)$  can be constructed by dividing an  $L_{36}(6^3)$  into two and three blocks respectively. Nguyen (1996b) discusses the use of CUT to construct near-orthogonal arrays.

Note that the orthogonally blocked Box–Behnken design for four factors is only available in three blocks. In the following, CUT is used to divide the unblocked Box–Behnken design

for four factors in 26 runs into two orthogonal blocks. The first block is shown below. The second block is obtained by switching the signs of entries in columns 1–4.

(1)	(2)	(3)	(4)
-1	-1	0	0
-1	1	0	0
0	0	1	1
0	0	-1	1
-1	0	0	-1
1	0	0	-1
0	-1	-1	0
0	-1	1	0
1	0	1	0
1	0	-1	0
0	1	0	1
0	1	0	-1
0	0	0	0

As expected, execution time for the CUT algorithm is shorter than for any other determinant-based algorithms. The construction of most of the designs in this paper is almost instantaneous on a PC.

The CUT program is a module of the Gendex toolkit; and the URL of this toolkit is <http://designcomputing.hypermart.net/gendex>.

**Appendix: the CUT blocking algorithm**

Before discussing the CUT algorithm which implements the blocking approach in Section 2, we present some matrix results. Without loss of generality, let  $\mathbf{x}_i^T$  and  $\mathbf{x}_u^T$  be two row vectors of  $\mathbf{X}$  which correspond to two runs, one in block 1 and the other in block 2. The effect on  $\mathbf{Z}^T \mathbf{X}$  obtained by the swap of block assignments of these two runs (i.e. the run that was in block 1 is now put in block 2 and vice versa) is the same as adding the matrix  $\Delta$  to  $\mathbf{Z}^T \mathbf{X}$ , where

$$\Delta = \begin{bmatrix} \mathbf{x}_u^T - \mathbf{x}_i^T \\ \mathbf{x}_i^T - \mathbf{x}_u^T \\ \mathbf{0}_{(b-2) \times (p-b)} \end{bmatrix}, \tag{2}$$

in which  $\mathbf{0}_{(b-2) \times (p-b)}$  denotes a  $(b - 2) \times (p - b)$  matrix of 0s. The CUT algorithm based on the above matrix result is as follows:

1. Allocate randomly the  $n$  runs of the chosen unblocked design to  $b$  blocks. Form (1) and calculate  $s_{wj}^* = s_{wj} - n_w/n s_{.j}$  ( $w = 1, \dots, b; j = 1, \dots, p - b$ ) and  $f = \sum s_{wj}^{*2}$ .
2. Repeat searching for a pair of runs belonging to two different blocks such that the swap of block assignments of these two runs results in the biggest reduction in  $f$ . If the search is successful, swap block assignments of these two runs and update  $f$  and (1) using (2). This process is repeated until  $f = 0$  or  $f$  cannot be reduced further.
3. Calculate  $|M|$  and  $\text{trace}(C_{22})$  and  $BF$ .

Each try consists of steps 1–3. Several tries are made for each design and the one with the smallest  $f$  is chosen. The algorithm stops when the number of tries is exhausted or when  $f = 0$  (i.e.  $BF = 1$ ).

### Remarks

1. To calculate the change in  $f$  or to update  $f$  in step 2, note that if  $s_{wj}$  is increased by an amount  $\delta$  then  $s_{wj}^{*2}$  is increased by an amount  $\delta^2 + 2\delta s_{wj}^*$ .
2. The second example is a common situation in which the orthogonality between the input variables and the block variables is considered more important than the orthogonality between the derived variables and the block variables. In this situation, CUT uses two objective functions  $g$  and  $f$ . If  $X$  is partitioned as  $X_1$  and  $X_2$  where  $X_1$  is an  $n \times m$  matrix and  $X_2$  is an  $n \times (p - b - m)$  matrix, then  $g$  is the sum of squares of the elements of  $Z^T X_1$ . A design is selected if it has a smaller  $g$  than the previous design or the same  $g$  but smaller  $f$  (the sum of squares of the elements of  $Z^T X$ ).
3. A similarity exists between the CUT algorithm and the NOA algorithm of Nguyen (1996a) for constructing supersaturated designs. These algorithms and the BIB algorithm of Nguyen (1994) for constructing incomplete block designs, are examples of an interchange algorithm.

### References

- ATKINSON, A.C. & DONEV, A.N. (1992). *Optimum Experimental Designs*. Oxford: Oxford University Press.
- BISGAARD, S. (1994). Blocking generators for small  $2^{k-p}$  designs. *J. Quality Technology* **26**, 288–296.
- BOX, G.E.P. & BEHNKEN, D.W. (1960). Some new three-level designs for the study of qualitative variables. *Technometrics* **2**, 455–475.
- BOX, G.E.P. & HUNTER, J.S. (1957). Multifactor experimental designs for exploring response surfaces. *Ann. Math. Statist.* **8**, 195–241.
- COOK, R.D. & NACHTSHEIM, C.J. (1989). Computer-aided blocking of factorial and response surface designs. *Technometrics* **31**, 339–346.
- CORNELL, J.A. (1990). *Experiments with Mixtures, Designs, Models, and the Analysis of Mixtures Data*, 2nd edn. New York: John Wiley & Sons, Inc.
- DRAPER, N.R., PRESCOTT, P., LEWIS, S.M., DEAN, A.M., JOHN, P.W.M. & TUCK, M.G. (1993). Mixture designs for four components in orthogonal blocks. *Technometrics* **35**, 268–276.
- ECCLESTON, J.A. & HEDAYAT, A. (1974). On the theory of connected designs: characterization and optimality. *Ann. Statist.* **2**, 1238–1255.
- KIEFER, J. (1959). Optimum experimental designs. *J. R. Stat. Soc. Ser. B Stat. Methodol.* **21**, 272–319.
- MILLER, A.J. & NGUYEN, N-K. (1994). A Fedorov exchange algorithm for  $D$ -optimal designs. *Appl. Statist.* **43**, 669–678.
- NGUYEN, N-K. (1994). Construction of optimal incomplete block designs by computer. *Technometrics* **36**, 300–307.
- NGUYEN, N-K. (1996a). An algorithmic approach to constructing supersaturated designs. *Technometrics* **38**, 69–73.
- NGUYEN, N-K. (1996b). A note on constructing near-orthogonal arrays with economic run size. *Technometrics* **38**, 279–283.