Constructing Small Response Surface Designs with Orthogonal Quadratic Effects using Cyclic Generators

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Abstract

The central composite designs (CCDs; Box & Wilson, 1951) for fitting the second-order response surface require a large number of 2-level runs at the first stage, especially when the number of factors is large. The small composite designs (SCDs; Draper & Lin, 1990; Nguyen & Lin, 2011) were developed for fitting the same model using a much less number of 2-level runs at the first stage. The 2-level runs at the first stage of CCDs and SCDs are fairly arbitrary. This paper introduced an algorithm which can augment any standard 2-level first-order design with additional 3-level

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runs to form a second-order design. These augmented runs are made up of circulant matrices. All designs produced by this algorithm have the orthogonal quadratic effect property. The CCDs and SCDs are special cases this algorithm.

Keywords: Augmented-pair designs; Composite designs; Circulant matrices; Orthogonal quadratic effects; Plackett-Burman design.

1 Introduction

Consider a screening experiment in a pharmaceutical process (extrusion-spheronnization) in which the formulation contains a drug substance, a plastic diluent and a binder (Lewis et al. 1999). The experimenters wish to perform an optimization process and the response of interest is the percentage mass yield of pellets having a particle size between 900-1,100μm. The seven process variables (factors) in this extrusion-spheronnization are: (**1**) % amount of binder (0.5-1%); (**2**) amount of water (40-50%); (**3**) granulation time (1-2 min); (**4**) spheronization load (1-4 kg); (**5**) spheronization speed (700-1,100 rpm); (**6**) extruder rate (15-60 rpm) and (**7**) spheronization time (2-5 min). Figure 1 shows the 8-run Plackett-Burman design (PB Design; Plackett & Burman, 1946) used for this screening experiment.

The estimates of the coefficients using the first-order model are: $b_0 = 62.0\%$ (constant term), $b_1 = 5.0\%$, $b_2 = 3.3\%$, $b_3 = 0.8\%$, $b_4 = 1.3\%$, $b_5 = -4.1\%$, $b_6 = -0.2\%$ and $b_7 = -6.0\%$. Let us assume that, after studying the magnitude of these estimates of the coefficients, the experimenters think that the factors (1), (2), (5) and (7) deserve further

(1)	(2)	(3)	(4)	(5)	(6)	(7)
-1	-1	-1	1	-1	1	1
1	-1	-1	-1	1	-1	1
1	1	-1	-1	-1	1	-1
-1	1	1	-1	-1	-1	1
1	-1	1	1	-1	-1	-1
-1	1	-1	1	1	-1	-1
-1	-1	1	-1	1	1	-1
1	1	1	1	1	1	1

Figure 1: The 8-run PB design for the extrusion-spheronnization study.

studies and decide to augment the four columns corresponding to these four factors with additional runs, so that the second-order model can be fitted with the combined data from both stages. Augmenting these four columns with eight axial runs will result in an SCD. Augmenting them with $\binom{8}{2}$ $(= 28)$ runs using the approach described in Morris (2000) will result in an augmented-pair design (APD) in 36 $(= 8 + 28)$ runs. Is there a different method to augment these columns?

This paper discusses an algorithm which can be used to augment any standard 2-level first order design, a PB design or a fraction 2^{k-p} of any resolution with 3-level runs. The augmented runs are made up of circulant matrices. As CCDs and SCDs are special cases of the designs constructed this way, we call our designs generalized SCDs or GSCDs. Like CCDs and SCDs, GSCDs are second-order designs with the orthogonal quadratic effect (OQE) property. This property is possessed by several popular second-order designs such as CCDs, SCDs, APDs and Box-Behnkens designs or BBDs (Box & Behnkens, 1960). Designs with the OQE property have the quadratic effects being orthogonal to all main and interaction effects. This is an important property as the quadratic effects, which could not be estimated in the first stage, can be estimated with the maximum precision in the second stage (Nguyen & Lin, 2011; Nguyen & Pham, 2016). The information matrix of a design for m factors with the OQE property and its inverse will have the form

$$\begin{pmatrix} \mathbf{A} & \mathbf{0}' \\ \mathbf{0} & \mathbf{B} \end{pmatrix}, \tag{1}$$

and

$$\begin{pmatrix} \mathbf{A}^{-1} & \mathbf{0}' \\ \mathbf{0} & \mathbf{B}^{-1} \end{pmatrix}, \tag{2}$$

respectively where $\mathbf{A}$ and $\mathbf{A}^{-1}$ are square matrices of size $m+1$ and $\mathbf{B}$ and $\mathbf{B}^{-1}$ are square matrices of size $m + \binom{m}{2}$. In the next paragraph we will explain the conditions of the OQE property in the context of GSCDs.

2 Structure of the information matrix of GSCDs

Consider the design matrix of a GSCD of the form

$$\mathbf{D} = (\mathbf{D}_0' \ \mathbf{D}_1', \ldots, \mathbf{D}_r')' \tag{3}$$

where $\mathbf{D}_0$ is a standard 2-level design or some columns of a PB design of order $n_0 \times m$ and $\mathbf{D}_1, \ldots, \mathbf{D}_r$ are circulant matrices of order $m \times m$. Then the size of $\mathbf{D}$ is $(n_0 + rm) \times m$.

Let $\mathbf{X}_{n\times p}$ denote the expanded design matrix for the second-order model, where $p = 1+2m+\binom{m}{2}$ is the number of parameters. The uth row of $\mathbf{X}$ is $(1, d_{u1}^2, \ldots, d_{um}^2, d_{u1}, \ldots, d_{um}, d_{u1}d_{u2}, \ldots, d_{u(m-1)um})$. Nguyen & Lin (2011) showed that the following conditions imply the OQE property:

$$\Sigma d_i d_j = 0 \ (i < j,\ i,\ j = 1, \ldots, m) \tag{4}$$

$$\Sigma d_i^2 d_j = 0 \ (i < j,\ i,\ j = 1, \ldots, m) \tag{5}$$

$$\Sigma d_i^2 d_j d_k = 0 \ (i \neq j,\ i \neq k,\ j < k,\ i,\ j,\ k = 1, \ldots, m) \tag{6}$$

where the summations are taken over the n design points. The circulant matrix $\mathbf{D}_q$ $(q = 1, \ldots, r)$ generated by the row vector $(d_{q1},\ d_{q2},\ \ldots,\ d_{qm})$ of length m will be of the form:

$$\begin{pmatrix} d_{q1} & d_{q2} & \cdots & d_{qm} \\ d_{qm} & d_{q1} & \cdots & d_{q(m-1)} \\ \ddots & \ddots & \ddots & \ddots \\ d_{q2} & d_{q3} & \cdots & d_{q1} \end{pmatrix}. \tag{7}$$

The m runs of the circulant matrix $\mathbf{D}_q$ will contribute $\Sigma_q d_i d_j$ $(i < j,\ i,\ j = 1, \ldots, m)$, $\Sigma_q d_i^2 d_j$ $(i < j,\ i,\ j = 1, \ldots, m)$ and $\Sigma_q d_i^2 d_j d_k$ $(i \neq j,\ i \neq k,\ j < k,\ i,\ j,\ k = 1, \ldots, m)$ to the summations in (4), (5) and (6) respectively. Note that $\mathbf{D}_0$ contributes zero to these summations. Due to the cyclic nature of the circulant matrices, it can easily be seen that $\Sigma_q d_1 d_2 = \Sigma_q d_2 d_3 = \Sigma_q d_3 d_4$, etc., $\Sigma_q d_1^2 d_2 = \Sigma_q d_2^2 d_3 = \Sigma_q d_3^2 d_4$, etc. and $\Sigma_q d_1^2 d_2 d_3 = \Sigma_q d_2^2 d_3 d_4$, etc.

Therefore, it is only necessary to compute $\Sigma_q d_1 d_2,\ \Sigma_q d_1 d_3,\ \Sigma_q d_1 d_4$, etc.$\Sigma_q d_1^2 d_2,\ \Sigma_q d_1^2 d_3,\ \Sigma_q d_1^2 d_4$,etc., $\Sigma_q d_1^2 d_2 d_3,\ \Sigma_q d_1^2 d_2 d_4$ and $\Sigma_q d_1^2 d_3 d_4$, etc. Thus for a GCSD of the form in (3), the conditions which imply the OQE property become:

$$\Sigma d_1 d_j = 0 \ (j = 2, \ldots, m) \tag{8}$$

$$\Sigma d_1^2 d_j = 0 \ (j = 2, \ldots, m) \tag{9}$$

$$\Sigma d_1^2 d_j d_k = 0 \ (j < k, j,\ k = 2, \ldots, m) \tag{10}$$

where the summations are take over the n design points. We will utilize this result in the next section.

3 The circulant augment algorithm

To construct a GSCD for m factors we choose a base matrix $\mathbf{D}_0$ of size $n_0 \times m$ and augment it with r circulant matrices each of size $m \times m$ such that $n_0 + rm > p$. The circulant augment (CA) algorithm requires the following steps:

1. Pick m columns randomly from a standard 2-level fractional design or a PB design of n_0 runs to form $\mathbf{D}_0$.

2. Initialize a matrix $\mathbf{d}$ of size $r \times m$ by setting the first x elements of $\mathbf{d}$ to 1, the next x elements to -1 and the remaining to 0. Randomize the elements of $\mathbf{d}$. Calculate $\mathbf{J}_q$ $(q = 1, \ldots, r)$ from each row of $\mathbf{d}$ and $\mathbf{J} = \Sigma_{q=1}^{r} \mathbf{J}_q$. Calculate f, the sum of squares of the elements of $\mathbf{J}$.

3. Search for a pair of entries in $\mathbf{d}$ such that the position swap of these two entries results in the biggest reduction in f. If the search is successful, update f and $\mathbf{d}$. Repeat this step until $f = 0$ or f cannot be reduced further.

Remarks

1. The three steps of our algorithm make up a try. Among all tries with $f = 0$ and the minimum of $r_{\max}$, the maximum of the correlation coefficients among the last $2m + \binom{m}{2}$ columns of the model matrix, we select the one with the highest $|\mathbf{X}'\mathbf{X}|$.

2. The fact that $\mathbf{d}$ has the same value of ±1's will ensure that the resulting design is balanced, i.e. its factors have the same number of ±1's.

3. Step 1 is not required if $\mathbf{D}_0$ consists of the significant factors of a screening design in the first stage.

4. The value for x in step 2 is set by trial and error. For $r = 2$, $x = 1$. For $r = 4$, $x = 4$ when $m = 3$ and $x = 6$ when $m =$ 4-7.

Following is an example of calculating vector $\mathbf{J}$ from a matrix $\mathbf{d}$ with four generating vectors: $\mathbf{d}_1$=(1, 1, -1, 0), $\mathbf{d}_2$=(1, 0, 1, -1), $\mathbf{d}_3$=(-1, -1, -1, 0) and $\mathbf{d}_4$=(1, 1, 0, -1). The readers can verify that the corresponding vectors $\mathbf{J}_q$ $(q = 1, \ldots, r)$ are: $\mathbf{J}_1$=(0, -2, 0, 0, 0, 2, -1, -1, 1), $\mathbf{J}_2$ = (-2, 2, -2, 0, 2, 0, -1, 1, -1), $\mathbf{J}_3$ =(2, 2, 2, -2, -2, -2, 1, 1, 1) and $\mathbf{J}_4$=(0, -2, 0, 2, 0, 0, 1, -1, -1) and $\mathbf{J} = \Sigma_{q=1}^{4}\mathbf{J}_q = \mathbf{0}$ where $\mathbf{0}$ is a null vector.

4 Discussion

Table 1 displays the goodness statistics of 63 GSCDs constructed by the CA algorithm in Section 3 for $m = 3, \ldots, 7$ and $n_0 = 8,\ 12,\ 16,\ 20,\ 24,\ 28$ and 32. There are 28 GSCDs with $r = 2,\ x = 1$ and 35 GSCDs with $r = 4,\ x = 4$ for $m = 3$ and $x = 6$ for $m > 3$. These goodness statistics are the d-value, $r_{\max},\ v_Q,\ v_M$ and v_I. d-value is the second-order d-efficiency of the design, which is calculated as

$$|\mathbf{X}'\mathbf{X}|^{1/p}/n \tag{11}$$

where $\mathbf{X}$ and $p\ (= 1+2m+\binom{m}{2})$ are the expanded design matrix and the number parameters for the second-order model respectively. This d-value, known as "information per point", is a popular measure of goodness of a design (Draper & Lin, 1990, Nguyen & Lin, 2011). $r_{\max}$ has already been defined in the previous section. $v_Q,\ v_M$ and v_I are the maximum variances of the m quadratic effects, of the m main effects and of the $\binom{m}{2}$ interactions respectively. Let us use denote a GSCD with $r = 2$ by GSCD(2)and a GSCD with $r = 4$ by GSCD(4). In Table 1, five GSCD(2)'s are also SCDs and four GSCD(4)'s are also CCDs.

It can be seen in Table 1 that the SCDs are inferior to the corresponding GSCD(4)'s for the same values of (m, n_0) with respect to all goodness statistics. Users of SCDs should be aware that the $r_{\max},\ v_M$ and v_I values of SCDs are very high. In Table 1, the $r_{\max}$ values of SCDs range from 0.607 to 0.943. Also, the v_M and v_I values of SCDs are about 5 to 10 times more than those of the corresponding GSCD(4)'s. It is interesting to note that the d-values of CCDs are very close to the corresponding values of GSCD(4)'s. The $r_{\max}$, v_Q, v_M and v_I values of CCDs, however, are still much larger than the ones of GSCD(4)'s. Note that GSCD(4)'s have $2m$ extra runs.

There are a number of (m, n_0) combinations when both CCDs and SCSs are not available, such as (5, 8), (6, 8), (6, 12), (7, 8),(7, 12), (7, 16) and (7, 20). For these combinations, solutions can be found with the GSCD and APD approaches. As the number of runs of APDs for $n_0 = 8,\ 12,\ 16,\ 20$, etc. are 36, 78, 136 and 210 respectively, only APDs for $n_0 = 8$ seem popular. The next paragraphs compares our GSCDs and the APDs for $(m, n_0) = (4,\ 8)$ and $(5,\ 12)$. It is interesting to note that our GSCD(4) for $(m, n_0) = (7,\ 8)$ and the corresponding APD are identical if the design in the first stage is a PB design for seven factors.

Let us return to the example in the Introduction and compare three different candidate augmented parts for the four chosen columns (1), (2), (5) and (7) in Figure 1. They are: (A) the eight axial runs; (B) the 16 runs generated by four cyclic generators (1, 1, -1, 0), (1, 0, 1, -1), (-1, -1, -1, 0) and (1, 1, 0, -1); and (C) the 28 runs generated by the APD approach. The 8-run 2-level design in Figure 1 together with the runs in (A) will result in a 16-run SCD; with the runs in (B) will result in a 24-run GSCD(4); and with runs in (C) will result in a 36-run APD.

Let us use the vector (d-value, $r_{\max}$, v_Q, v_M, v_I) to summarize the goodness statistics of each candidate design. For the 16-run SCD this vector is (0.308, 0.894, 0.403, 0.500, 0.625). For the 24-run GSCD(4) it is (0.446, 0.224, 0.375, 0.060, 0.070). For the 36-run APD it is

(A)

1	0	0	0
-1	0	0	0
0	1	0	0
0	-1	0	0
0	0	1	0
0	0	-1	0
0	0	0	1
0	0	0	-1

(B)

1	1	-1	0
0	1	1	-1
-1	0	1	1
1	-1	0	1
1	0	1	-1
-1	1	0	1
1	-1	1	0
0	1	-1	1
-1	-1	-1	0
0	-1	-1	-1
-1	0	-1	-1
-1	-1	0	-1
1	1	0	-1
-1	1	1	0
0	-1	1	1
1	0	-1	1

(C)

0	1	0	-1
0	0	1	0
1	0	1	-1
0	1	1	0
1	0	0	0
1	1	0	0
0	0	0	-1
-1	0	0	0
0	0	0	-1
-1	1	0	0
0	0	-1	0
0	1	-1	0
-1	0	-1	-1
0	-1	1	0
-1	0	1	1
0	-1	0	1
0	0	0	1
-1	-1	0	0
0	0	1	0
1	-1	0	0
1	0	0	0
0	-1	0	-1
0	0	0	1
0	1	0	1
-1	0	0	0
1	0	-1	1
0	-1	-1	0
0	0	-1	0

Figure 2: Candidate augmented parts for the extrusion-spheronnization study.

(0.373, 0.258, 0.115, 0.054, 0.089). It can be seen that this SCD have a big price to pay in terms of the goodness statistics.

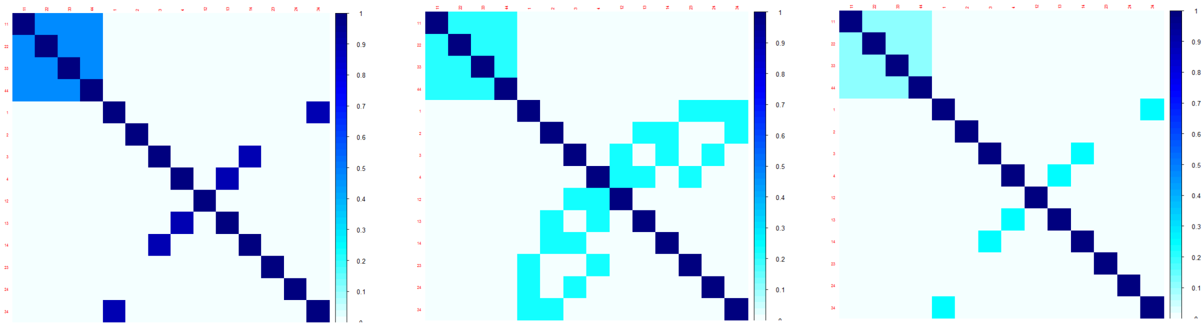
Let us have another example in which our GSCD could be used. Bermejo-Barrera et al. (2001) conducted an experiment to optimize the ultrasonic bath-induced acid leaching for the determination of trace elements in seafood products by atomic absorption spectrometry. The seven variables (factors) are (**1**) Nitrid acid concentration (M), (**2**) Hydrochloric acid concentration (M), (**3**) Hydrogen peroxide concentration (M), (**4**) Acid solvent volume (mL), (**5**) Ultrasonic water-bath temperature (^{o}C), (**6**) Ultrasound exposure time (min) and (**7**) Mussel particle size (μm). Seven columns of a 12-run PB design were used to select the most significant variables that affect to the acid leaching process (Figure 3), while CCDs were used to find the optimum values for the variables involved in acid leaching.

(1)	(2)	(3)	(4)	(5)	(6)	(7)
1	-1	1	-1	-1	-1	1
1	1	-1	1	-1	-1	-1
-1	1	1	-1	1	-1	-1
1	-1	1	1	-1	1	-1
1	1	-1	1	1	-1	1
1	1	1	-1	1	1	-1
-1	1	1	1	-1	1	1
-1	-1	1	1	1	-1	1
-1	-1	-1	1	1	1	-1
1	-1	-1	-1	1	1	1
-1	1	-1	-1	-1	1	1
-1	-1	-1	-1	-1	-1	-1

Figure 3: The 12-run PB design for the study of Bermejo-Barrera et al. (2001).

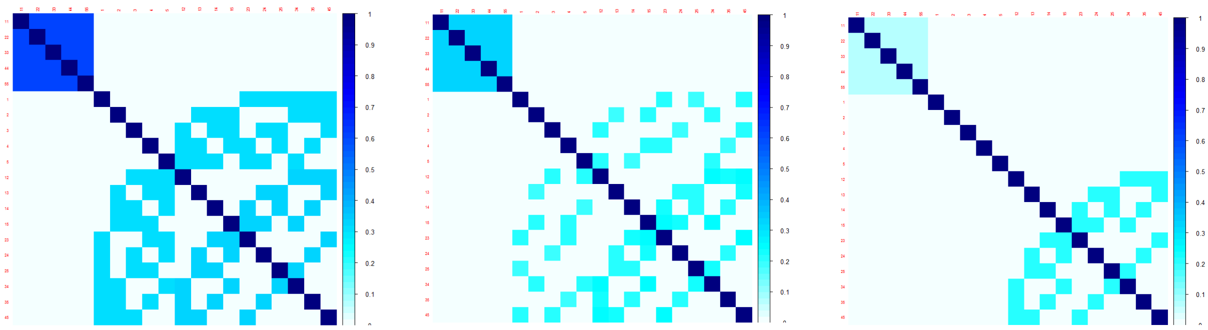
This experiment was also discussed in Mee (2011) p. 206. In this paper, we assume that the experimenters found the significant variables were 1-4 and 7.

Let us compare three types of candidate augmented parts: (A) 10 axial runs; (B) 20 additional runs generated by four cyclic generator (-1, 1, 0, -1, 0), (0, 1, 0, -1, -1), (0, 1, 1, 0, 1) and (-1, 0, -1, 0, 1); and (C) 66 (= $\binom{12}{2}$) runs obtained by the APD approach. The 12-run 2-level design in (A) together with the runs in (A) will result in a 22-run SCD, with the runs in (B) will result in a 32-run GSCD(4) and with the runs in (C) will result in a 78-runs APD. The vector (d-value, $r_{\max}$, v_Q, v_M, v_I) of the 22-run SCD is (0.259, 0.607, 0.411, 0.417, 0.536), of the 32-run GSCD(4) is (0.408, 0.333, 0.333, 0.051, 0.089) and of the 78-run APD is (0.341, 0.208, 0.052, 0.024, 0.048).



SCD GSCD APD

Figure 4: CCPs of a 16-run SCD, a 24-run GSCD and a 36-run APD for four factors.



SCD GSCD APD

Figure 5: CCPs of a 22-run SCD, a 32-run GSCD and a 78-run APD for five factors.

To visualize the aliasing pattern among the columns of the model matrix of each candidate design in the previous paragraph, we make use of the correlation cell plots (CCPs). These plots, proposed by Jones & Nachtsheim (2011), display the magnitudes of the correlations between quadratic effects, main effects and 2-factor interactions of each designs. The color of each cell in these plots goes from white (no correlation) to dark (correlation of 1 or close to 1). The CCPs of the candidate designs in the first example are in Figure 4 and the ones in the second example in Figure 5. All six CCPs in Figures 4 and 5 show that the quadratic effects are orthogonal to the main effects and 2-factor interactions. They also show that the main effects are orthogonal to one another. It can be seen that the magnitude of correlation is very high among the quadratic effects of the SCD.

In summary, this paper introduces a new class of second-order designs with orthogonal quadratic effects using cyclic generators or GSCDs. It describes an algorithm to construct GSCDs and compare them with more popular designs such as SCDs, CCDs and APDs. The advantage of GSCD over SCD, CCD and APD is its flexibility: the 2-level factorial part n_0 could have different sizes and the circulant augmented part could also have different sizes. As the percentages of the 0-level for each factors of BBDs with the recommended number of factor are too high (for BBDs for 3-7 factors these percentages are 47, 56, 65, 56 and 61% respectively) and very often, the extreme setting ± 1's are the settings in which the experimenters are interested and not the neutral setting which is zero, our GSCDs could also be considered good alternatives to BBDs.

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Table 1: Comparison of the goodness statistics of GSCDs for $r = 2$ and $r = 4$

		$r=2$						$r=4$					
m	n_0	n	d_2	r_{max}	v_Q	v_M	v_I	n	d_2	r_{max}	v_Q	v_M	v_I
3	8	14†‡	0.463	0.300	0.406	0.100	0.125	20	0.452	0.250	0.375	0.063	0.083
	12	18	0.455	0.357	0.396	0.079	0.092	24	0.405	0.224	0.333	0.053	0.066
	16	22	0.464	0.389	0.391	0.056	0.063	28	0.455	0.167	0.312	0.042	0.050
	20	26	0.451	0.409	0.388	0.047	0.052	32	0.447	0.154	0.300	0.037	0.043
	24	30	0.447	0.423	0.385	0.038	0.042	36	0.445	0.125	0.292	0.031	0.036
	28	34	0.435	0.433	0.384	0.034	0.036	40	0.436	0.118	0.286	0.028	0.032
	32	38	0.429	0.441	0.383	0.029	0.031	44	0.432	0.100	0.281	0.025	0.028
4	8	16†	0.308	0.894	0.403	0.500	0.625	24	0.446	0.224	0.375	0.060	0.070
	12	20	0.395	0.524	0.398	0.106	0.127	28	0.468	0.200	0.333	0.042	0.052
	16	24‡	0.457	0.556	0.396	0.056	0.063	32	0.462	0.154	0.313	0.039	0.044
	20	28	0.440	0.576	0.394	0.052	0.057	36	0.467	0.143	0.300	0.031	0.036
	24	32	0.447	0.59	0.394	0.043	0.046	40	0.460	0.118	0.292	0.029	0.032
	28	36	0.445	0.600	0.393	0.035	0.038	44	0.459	0.111	0.286	0.025	0.028
	32	40	0.449	0.608	0.392	0.029	0.031	48	0.452	0.095	0.281	0.023	0.025
5	8	-	-	-	-	-	-	28	0.372	0.667	0.344	0.067	0.190
	12	22†	0.259	0.607	0.411	0.417	0.536	32	0.409	0.333	0.333	0.048	0.083
	16	26‡	0.440	0.639	0.41	0.056	0.063	36	0.444	0.357	0.328	0.039	0.052
	20	30	0.406	0.659	0.409	0.061	0.068	40	0.446	0.375	0.325	0.034	0.048
	24	34	0.430	0.673	0.409	0.056	0.061	44	0.455	0.389	0.323	0.029	0.040
	28	38	0.436	0.683	0.408	0.038	0.041	48	0.458	0.400	0.321	0.026	0.034
	32	42	0.456	0.691	0.408	0.029	0.031	52	0.466	0.409	0.320	0.023	0.028
6	8	-	-	-	-	-	-	32	0.303	0.667	0.171	0.109	0.264
	12	-	-	-	-	-	-	36	0.348	0.500	0.167	0.071	0.173
	16	28†	0.263	0.943	0.423	0.500	0.562	40	0.399	0.676	0.164	0.081	0.109
	20	32	0.309	0.709	0.422	0.417	0.482	44	0.423	0.542	0.163	0.042	0.063
	24	36	0.368	0.723	0.422	0.117	0.109	48	0.445	0.556	0.162	0.039	0.054
	28	40	0.392	0.733	0.421	0.074	0.069	52	0.459	0.567	0.161	0.032	0.039
	32	44‡	0.456	0.741	0.421	0.029	0.031	56	0.491	0.576	0.161	0.023	0.028
7	8	-	-	-	-	-	-	36	0.269	0.667	0.117	0.055	0.180
	12	-	-	-	-	-	-	40	0.277	0.250	0.115	0.092	0.182
	16	-	-	-	-	-	-	44	0.319	0.800	0.113	0.094	0.193
	20	-	-	-	-	-	-	48	0.356	0.500	0.113	0.057	0.119
	24	38†	0.253	0.756	0.432	0.47	0.725	52	0.386	0.286	0.112	0.046	0.089
	28	42	0.318	0.767	0.432	0.293	0.284	56	0.409	0.375	0.112	0.044	0.075
	32	46	0.358	0.970	0.431	0.500	0.531	60	0.461	0.704	0.111	0.047	0.056

†SCDs, ‡CCDs.