DISSERTATION

SECOND-ORDER SUB-ARRAY CARTESIAN PRODUCT SPLIT-PLOT DESIGN

Submitted by

Luis A. Cortés-Mestres

Department of Mechanical Engineering

In partial fulfillment of the requirements

For the Degree of Doctor of Philosophy

Colorado State University

Fort Collins, Colorado

Summer 2015

Doctoral Committee:

Advisor: William S. Duff
Co-Advisor: James R. Simpson

Edwin K. P. Chong
Thomas H. Bradley
Shantanu H. Jathar

ABSTRACT

SECOND-ORDER SUB-ARRAY CARTESIAN PRODUCT SPLIT-PLOT DESIGN

Fisher (1926) laid down the fundamental principles of design of experiments: factorization, replication, randomization, and local control of error. In industrial experiments, however, departure from these principles is commonplace. Many industrial experiments involve situations in which complete randomization may not be feasible because the factor level settings are impractical or inconvenient to change, the resources available to complete the experiment in homogenous settings are limited, or both. Restricted randomization due to factor levels that are impractical or inconvenient to change can lead to a split-plot experiment. Restricted randomization due to resource limitation can lead to blocking. Situations that require fitting a second-order model under those conditions lead to a second-order block split-plot experiment. Although response surface methodology has experienced a phenomenal growth since Box and Wilson (1951), the departure from standard methods to tackle second-order block split-plot design remains, for the most part, unexplored. Most graduate textbooks only provide a relatively basic treatise of the subject. Peer-reviewed literature is scarce, has a limited number of examples, and provides guidelines that often are too general. This deficit of information leaves practitioners ill prepared to face the roadblocks illuminated by Simpson, Kowalski, and Landman (2004).

Practical strategies to help practitioners in dealing with the challenges presented by second-order block split-plot design are provided, including an end-to-end, innovative approach for the construction of a new form of effective and efficient response surface design referred to as second-order sub-array Cartesian product split-plot design. This new form of design is an alternative to ineffective split-plot designs that are currently in use by the manufacturing and quality control community. The design is economical, the prediction variance of the regression coefficients is low and stable, and the aliasing between the terms in the model and effects that are not in the model as well as the correlation between similar effects that are not in the model is low. Based on an assessment using well-accepted key design evaluation criterion, it is demonstrated that second-order sub-array Cartesian product split-plot designs perform as well or better than historical designs that have been considered standards up to this point.

ACKNOWLEDGEMENT

I wish to express my sincere gratitude to my advisors, Dr. William S. Duff and Dr. James R. Simpson, and to my doctoral committee, Dr. Edwin K. P. Chong, Dr. Thomas H. Bradley, and Dr. Shantanu H. Jathar for their guidance and expertise. Most of all, I wish to thank Dr. James R. Simpson for his patience, encouragement, motivation, and inspiration through the research. Dr. Simpson is not only a great practitioner and an excellent teacher, but also a good friend. Thank you very much.

Quiero darle gracias a todas las personas que han formado parte de mi vida. Gracias a mis queridos padres Arístides Cortés y Elba Mestres por haberme dado la oportunidad de tener una educación excelente, pero sobre todo, gracias por su ejemplo y su apoyo incondicional. Gracias a mis queridos hijos Javi y Manny, y a mi única hija Mairena, por haber sido mi luz y mi guía. Gracias a mi familia Mejicana Abby, Nikki, y Bre por su apoyo y entendimiento. Los quiero mucho. Que Dios los bendiga.

Ari

TABLE OF CONTENTS

1 Introduction

The quest for understanding is a hallmark in the rise of human civilization. Experimentation is the most fundamental method for acquiring that understanding. It took centuries to formulate the synergistic amalgamation of principles and methods that enable true experimentation—*design of experiments* and *response surface methodology*. Since the development of design of experiments in the 1920's and subsequent expansion of response surface methodology in the 1950's, the use of these methods has expanded remarkably to virtually every industrial, technological, service, and military sector. Both practitioners and scholars undertake tasks of exceptional personal skills and conspire daily to add to the response surface methodology's body of knowledge. Standing on the shoulders of those giants, this research provides a practical application and a foundation for further research on a type of design referred to as *second-order sub-array Cartesian product split-plot design*.

1.1 Research Perspective

Fisher (1926) laid down the fundamental principles of design of experiments: factorization, randomization, replication, and local control of error. Factorization consists of making deliberate, simultaneous changes to the experimental factors to find the individual or mutual effect those factors have on the response variables of interest. Replication is the application of a combination of experimental factors, called treatments, to the experimental units, to obtain a valid estimate of the experimental error. Randomization refers to the random assignment of treatments to experimental units and to the random assignment of experimental runs to treatments. Randomization averages out the effects of undesirable factors present in the experiment, generally enables the assumption that the experimental errors are independent and identically distributed random variables, and leads to an unbiased estimate of variance. Local control of error is an experiment design technique that allows for minimizing the influence of nuisance factors on the response by partitioning the experimental units into homogeneous subsets called blocks. Local control improves the precision of the comparison of factors of interest and reduces or eliminates the component of the variability transmitted from nuisance factors. Sometimes it is not possible to carry out an experiment while adhering simultaneously to all of the principles of design of experiments.

Restrictions in randomization involving heterogeneous experimental settings require some form of local control of error, or error-control design. Blocking is both a technique for controlling error and a form of restricted randomization. Blocking improves the precision of the comparison between factors by arranging the treatments into groups, or blocks, that have similar sources of variability irrelevant to the experiment. The differences in variability between the blocks due to irrelevant sources are identified and removed analytically leaving in the experimental error only the differences within treatments in the same block, which leads to the interpretation that the blocks are homogenous. Similarly, the experimental settings for split-plot experiments are heterogeneous. An experimental setting is homogenous if it is uniform in nature or composition and the transition between state values at any two times depends only on those times. Conversely, a non-uniform experimental setting is heterogeneous. The error-control design is the layout of the treatments in the experiment. The most common error-control designs for industrial applications are the completely randomized design, the randomized complete block design, and the incomplete block design.

Industrial experiments may involve situations in which the complete randomization of experimental runs may not be feasible because of factor level settings that are impractical or inconvenient to change, limitations in the resources available to complete the experiment in homogeneous settings, or both. For example, some experiments involve combinations of materials that are rare-to-find as well as easy-to-find. Other experiments involve the application of some treatments to large experimental units as well as the application of other treatments to smaller experimental units. Similarly, some experiments require the batch processing of experimental units for some factors but not for other factors, or the stream processing of different experimental units requiring the application of the same treatment. Likewise, some experiments involve factors that need to be estimated more precisely than other factors, the comparison of equipment, or factor levels that are hard-to-change and factor levels that are easy-to-change. Practitioners refer to all of these situations collectively as experiments with hard-to-change factors and easy-to-change factors. Restricted randomization due to factor levels that are impractical or inconvenient to change can lead to a split-plot experiment.

Split-plot experiments have their origin in agronomic research. In agronomic experiments, some factors like irrigation method are restricted to large areas of land, called whole-plots. Whole-plots are split into smaller areas of land, called sub-plots, which allow for the individual application of treatments, such as seed variation. Factors associated with whole-plots are called whole-plot factors while factors associated with sub-plots are called sub-plot factors. Because the whole-plots are split into sub-plots, there is more experimental material for the whole-plots than for the sub-plots.

Like in agronomic experiments, in industrial split-plot experiments the hard-to-change factors are typically associated with the whole-plots while the easy-to-change factors are typically associated with the sub-plots. In a simple split-plot experiment, the sub-plot treatments are randomly assigned to the whole-plot treatments. Consequently, a split-plot experiment can be thought of as a superposition, or nesting, of two experiments—one experiment based on the whole-plot treatments and another experiment based on the sub-plot treatments. Thus, whole-plots have one type of experimental units while sub-plots have another type. An experimental unit is the smallest unit of material that is independently treated with a specific combination of factors. Experimental units can consist of one or more observational units. An observational unit is a unit of experimental material where the measurements are taken. Clearly, the experimental units for the whole-plots are larger than for the sub-plots. The sub-plots are the observational units for the whole-plots. Typically, applying treatments to the whole-plots is harder than applying treatments to the sub-plots. In some cases, setting the factor levels for the whole-plots is an imprecise operation; consequently, the whole-plot variance is large compared to the sub-plot variance.

An experiment is subject to unavoidable variations in both the experimental units and the observational units. The variability between and within experimental units form the bases for the experimental error. Because experimental error comes from replicating the experimental units, split-plots have one error term associated with whole-plot treatments and one error term associated with sub-plot treatments—the most essential characteristic of the split-plot experiment. The error terms serve to test the significance of the whole-plot factors, of the whole-plot by sub-plot interactions, and of the sub-plot factors. Clearly, there are more degrees-of-freedom available for estimating the sub-plot variance, σ^2, than for estimating the whole-plot variance, σ_δ^2, which implies better statistical power to detect significant sub-plot effects than whole-plot effects.

Yates (1935) outlined the design of split-plot experiments. Figure 1-1 illustrates a variation of the split-plot experiment for two whole-plot factors and two sub-plot factors at two levels each. The design, referred to as a 2^2x2^2 block split-plot experiment, involves a four-fold randomization schema: (1) the random assignment of blocks to whole-plots; (2) the random assignment of whole-plots to sub-plots; (3) the random assignment of sub-plots to observational units; and (4) the random selection of observational units.

In theory, the design is very simple, although it is more complex than a comparable completely randomized design. First, the randomized whole-plot treatments are arranged in a randomized complete block structure. Then, each whole-plot is divided into sub-plots to which the sub-treatments are randomly assigned and arranged in a randomized complete block structure. Finally, the observational units are randomly assigned to the sub-plot treatments. A randomized complete block error-control design for both the whole-plot treatments and the sub-plot treatments means that each treatment is applied once per block. Thus, the second block is a replicate of the first block.

The design has three residual degrees-of-freedom at the whole-plot level and one residual degree-of-freedom for blocks. If the error-control design for all eight whole-plot treatments was a completely randomized design (without blocks), there would be an extra degree-of-freedom for estimating σ_δ^2. In a split-plot design where the error-control design for the whole-plot treatments is a completely randomized design, the experimental units are the whole-plots. Conversely, in a split-plot design where the error-control design for the whole-plot treatments is a randomized complete block design, the experimental units are the blocks. When executing the split-plot experiment, the sub-plot factors are reset between runs at fixed settings of the whole-plot factors, and the whole-plot factors are reset within and between blocks. Note that the primary reason to carry out a split-plot experiment is to reduce the resetting of the whole-plot factors.

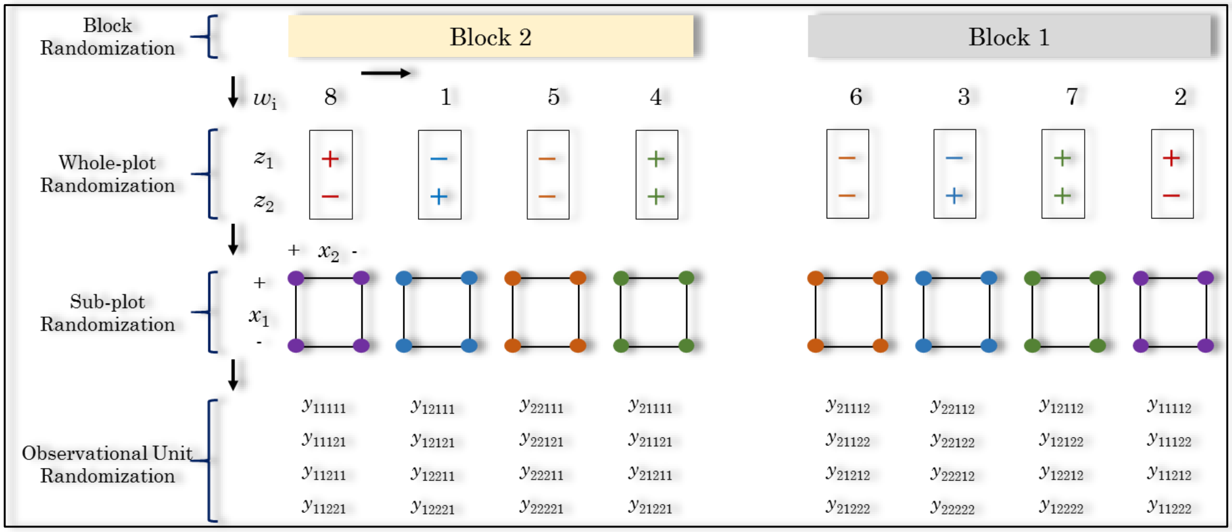


Figure 1-1. Variation of the split-plot experiment

A mixed linear model consistent with the design in Figure 1-1 is:

$$y_{kijmn} = \mu + \eta_k + \alpha_i + \tau_j + (\alpha\tau)_{ij} + \delta_k + \beta_m + \rho_n + (\beta\rho)_{mn} + (\alpha\beta)_{im} + (\alpha\rho)_{in} + (\tau\beta)_{jm} + (\tau\rho)_{jn} + \varepsilon \quad (1\text{-}1)$$

$$k = 1, 2, 3, \ldots, w\,; \quad i = 1, 2, 3, \ldots, a; \quad j = 1, 2, 3, \ldots, b; \quad m = 1, 2, 3, \ldots, c; \quad n = 1, 2, 3, \ldots, d$$

where

$$\delta_k = (\eta\alpha)_{ki} + (\eta\tau)_{kj} + (\eta\alpha\tau)_{kij}$$

$$\varepsilon_{kijmn} = (\eta\beta)_{km} + (\eta\rho)_{kn} + (\eta\beta\rho)_{kmn} + (\eta\alpha\beta)_{kim} + (\eta\alpha\rho)_{kin} + (\eta\tau\beta)_{kjm} + (\eta\tau\rho)_{kjn} + \varepsilon'_{kijmn}$$

$$\delta_k = \mathrm{N}(0, \sigma_\delta^2) \qquad \varepsilon_{kijmn} = \mathrm{N}(0, \sigma^2) \qquad \varepsilon'_{kijmn} = \mathrm{N}(0, \sigma_\epsilon^2)$$

and y_{kijmn} is the observation corresponding to the effects of the i^{th} level of z_1, j^{th} level of z_2, m^{th} level of x_1, n^{th} level of x_2, and the k^{th} block. μ is the grand mean and η corresponds to blocks (or replicates). $\propto_i$ and τ_j are the fixed effects associated with the whole-plot factors, $(\propto\tau)_{ij}$ is the fixed effect due to the two-factor interaction between the whole-plot factors, and δ_k is the pooled whole-plot error component that represents all of the block by whole-plot factors and block by two whole-plot factor interactions. β_m and ρ_n are the fixed effects associated with the sub-plot factors and $(\beta\rho)_{mn}$ is the fixed effect due to the two-factor interaction between the two sub-plot factors. The terms $(\alpha\beta)_{im}$, $(\alpha\rho)_{in}$ $(\tau\beta)_{jm}$, and $(\tau\rho)_{jn}$ are the two-factor interactions between the whole-plot factors and sub-plot factors, and ε_{kijmn} is the pooled sub-plot error component that includes the experimental error ε'_{kijmn}, the interactions between the blocks by sub-plot factors, blocks by two sub-plot factor interactions, and all of the block by whole-plot factor by sub-plot factor interactions. Fixed effect interactions higher than second-order $(\alpha\beta\rho)_{imn}$, $(\tau\beta\rho)_{jmn}$, $(\alpha\tau\beta)_{ijm}$, $(\alpha\tau\rho)_{ijn}$, and $(\alpha\tau\beta\rho)_{ijmn}$ are considered negligible and are not included in the model. All of the effects are considered deviations from the grand mean, there is no correlation between the random effects components and the fixed effects components (strict exogeneity assumption), and the two pooled random error terms are independent and identically distributed. Thus,

$$\sum_{i=1}^{a}\alpha_i = 0 \qquad \sum_{j=1}^{b}\tau_j = 0 \qquad \sum_{m=1}^{c}\beta_m = 0 \qquad \sum_{n=1}^{d}\rho_n = 0$$

$$\sum_{i=1}^{a}(\alpha\tau)_{ij} = \sum_{j=1}^{b}(\alpha\tau)_{ij} = 0 \qquad \sum_{i=1}^{c}(\beta\rho)_{mn} = \sum_{j=1}^{d}(\beta\rho)_{mn} = 0 \qquad \sum_{i=1}^{a}(\alpha\beta)_{im} = \sum_{j=1}^{c}(\alpha\beta)_{im} = 0$$

$$\sum_{i=1}^{a}(\alpha\rho)_{in} = \sum_{j=1}^{d}(\alpha\rho)_{in} = 0 \qquad \sum_{i=1}^{b}(\tau\beta)_{jm} = \sum_{j=1}^{c}(\tau\beta)_{jm} = 0 \qquad \sum_{i=1}^{b}(\tau\rho)_{jn} = \sum_{j=1}^{d}(\tau\rho)_{jn} = 0$$

$$Cov[\delta_k, \alpha_i] = Cov[\delta_k, \tau_j] = Cov[\delta_k, \beta_m] = Cov[\delta_k, \rho_n] = 0$$

$$Cov[\varepsilon, \alpha_i] = Cov[\varepsilon, \tau_j] = Cov[\varepsilon, \beta_m] = Cov[\varepsilon, \rho_n] = 0$$

$$Cov[\delta_k, (\alpha\tau)_{ij}] = Cov[\varepsilon, (\alpha\tau)_{ij}] = 0$$

$$Cov[\delta_k, (\beta\rho)_{mn}] = Cov[\delta_k, (\alpha\beta)_{im}] = Cov[\delta_k, (\alpha\rho)_{in}] = \mathrm{Cov}\left[\delta_k, (\tau\beta)_{jm}\right] = Cov\left[\delta_k, (\tau\rho)_{jn}\right] = 0$$

$$Cov[\varepsilon, (\beta\rho)_{mn}] = Cov[\varepsilon, (\alpha\beta)_{im}] = Cov[\varepsilon, (\alpha\rho)_{in}] = \mathrm{Cov}\left[\varepsilon, (\tau\beta)_{jm}\right] = Cov\left[\varepsilon, (\tau\rho)_{jn}\right] = 0$$

In general, split-plot experiments provide less information on the whole-plot factors relative to a completely randomized experiment—an experiment in which the assignment of treatments to experimental or observational units and the run order are completely randomized—of the same size. However, a gain on information on the sub-plots effects and the sub-plot by whole-plot interactions compensates the loss of information on the whole-plot. Because the estimates involving sub-plot factors are more precise, the sub-plot variance tends to be smaller than the whole-plot variance. The assumption is that these errors are independent and identically distributed normal random variables with constant variance.

Since each whole-plot contains only one whole-plot treatment (i.e. one combination of whole-plot factors), the differences between treatments coincides with the difference between whole-plots. Hence, they are confounded. Thus, there is only a trivial amount of information from between whole-plots comparison while the information within whole-plot comparison is unaffected by the confounding. In experiments with unreplicated whole-plots, only the error term associated with the sub-plots can be estimated and it cannot be used to test the significance of the whole-plot factors. Thus, the reduction of error and the valid estimation of error are key concepts in split-plot experiments.

In many industrial experiments, it is necessary to fit a second-order model to the observations to reveal the true underlying process conditions or product characteristics. Box and Wilson (1951) catalyzed the application of response surface methodology to industrial experiments. Response surface methodology is a sequential strategy of experimentation that incorporates statistical methods to fit a low-order Taylor series approximation to the true underlying mechanism. Because traditional response surface methodology assumes that all factors have the same importance or the same value, which leads to completely randomized experiments, the following second-order Taylor series approximation is commonly used to fit the observations:

$$E(y) = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{j<i}^{k} \sum_{i=2}^{k} \beta_{ji} x_j x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \varepsilon_{ij} \qquad \text{(1-2)}$$

where y represents the response, the $x's$ represents the independent variables or factors to which the observational units are subjected to, the $\beta's$ represents the regression coefficients that are determined empirically, and ε_{ij} represents the random error component that incorporates measurement error, variability from uncontrolled factors, variability in the experimental units, general background noise, etc. The model error is assumed to be an independent and identically distributed $\mathrm{N}(0, \sigma^2)$ random variable and the variance σ^2 is assumed constant throughout the experimental region. For k factors, the number of model terms, including the intercept, is:

$$t = 1 + 2k + \frac{k(k-1)}{2}$$

In a split-plot experiment, the assumption that all of the factors have the same importance or the same value does not hold. To fit a second-order split-plot model to the observations, the Taylor series approximation in Equation (1-2) takes the form:

$$E(y) = \beta_0 + \sum_{i=1}^{p} \beta_i z_i + \sum_{j<i}^{p} \sum_{i=2}^{p} \beta_{ji} z_j z_i + \sum_{i=1}^{p} \rho_{ii} z_i^2 + \sum_{i=1}^{q} \gamma_i x_i + \sum_{j<i}^{q} \sum_{i=2}^{q} \gamma_{ji} x_j x_i + \sum_{j=1}^{p} \sum_{i=1}^{q} \gamma_{ji} z_j x_i + \sum_{i=1}^{q} \theta_{ii} x_i^2 \quad \text{...(1-3)}$$

where now x_q represents the vector for the q sub-plot factors, z_p represents the vector for the p whole-plot factors, and $k = p + q$. The $\beta's$ represent the regression coefficients for the partition of the information matrix that has whole-plot linear terms, $\rho's$ represent the coefficients for the partition that has whole-plot pure quadratic terms, $\gamma's$ represent the coefficients for the partition that contains sub-plot linear terms and whole-plot by sub-plot interaction terms, and $\theta's$ represent the coefficients for the partition that contains sub-plot quadratic terms. For p = 2 and q = 2, Equation (1-3) reduces to:

$$\begin{aligned} E(y) = \beta_0 +& \\ & \beta_1 z_1 + \beta_2 z_2 + \rho_{12} z_1 z_2 + \beta_{11} z_1^2 + \beta_{22} z_2^2 + \\ & \gamma_1 x_1 + \gamma_2 x_2 + \gamma_{12} x_1 x_2 + \gamma_{11} z_1 x_1 + \gamma_{12} z_1 x_2 + \gamma_{21} z_2 x_1 + \gamma_{22} z_2 x_2 + \\ & \theta_{11} x_1^2 + \theta_{22} x_2^2 \end{aligned} \qquad (1\text{-}4)$$

In industrial applications, a set of experiments are typically carried out sequentially, or in blocks, to explore a limited region of interest (region R) that is contained within a larger region of operability (region O). The first block typically consists in running a screening experiment to identify the factors and interactions that have large effects on the responses of interest and to determine the presence of curvature in the response surface. Experiment designs that are suitable for this phase and generally adequate to fit the first-order model include 2^k factorial design with center points and 2^{k-p} fractional factorial design with center points. If the curvature is significant, the first-order model may become inadequate to represent the response surface and a second or higher-order model is needed. Then, in the subsequent blocks, the screening designs are augmented to produce a new design that ultimately produces a response surface model that accurately represents the entire design space and that can be used to determine the combination of factors levels that optimize the responses. Blocking is required to eliminate the effects caused by nuisance factors.

When using Equation (1-3) to model a response surface, practitioners may confront many of the practical issues embedded within the equation that are associated with restricted randomization, design alternatives, non-linear effects, sequential assembly, variance estimation, confounding, experimental and observational units, heterogeneous experimental settings, and enough degrees-of-freedom to estimate all of the model coefficients as well as whole-plot and sub-plot variances. While Myers, Montgomery, and Anderson-Cook (2009) provided design evaluation criteria for split-plot design consistent with the standard Box and Draper (1975), sometimes the selection of a design over various alternatives is not a straightforward process, and the selected design is not adequate for dealing with those issues. Hence, there is a need to adapt or create new approaches to dealing with second-order split-plot design. Simpson, Kowalski, and Landman (2004) illuminated the issues surrounding Equation (1-4) and emphasized the importance of modifying traditional response surface methods to fit specific needs while preserving the desirable properties of the response surface designs.

1.2 Motivation

Simpson, Kowalski, and Landman (2004) studied the effects that four factors had on the aerodynamic performance of a NASCAR Winston Cup Chevrolet Monte Carlo stock car in a wind tunnel. The study considered four significant factors: front height, rear height, yaw angle, and grille configuration. The levels for the front height and rear height factors were hard-to-change during the experiment while the levels for the yaw angle and grille configuration factors were easy-to-change, which lead to a split-plot structure. The design is illustrated in Figure 1-2.

Figure 1-2 is a projection of a four dimensional hyper-space into a two dimensional space. The axes for the whole-plot factors z_1 and z_2 are represented by the abscissa and ordinate. The axes for the sub-plot factors x_1 and x_2 are ovelayed at every design point in z_1 and z_2 with the sub-plot center at the design point. The layout consists of one two-level, replicated whole-plot at each of the four factorial points $(z_1, z_2) = (\pm 1, \pm 1)$ augmented with one whole-plot at the whole-plot center (z_1, z_2) = (0, 0). The replicated whole-plots are shown as concentric squares or concentric circles. The sub-plot structure was similar and consisted of one sub-plot run at each of the four factorial points $(x_1, x_2) = (\pm 1, \pm 1)$ and one whole-plot at the whole-plot center (x_1, x_2) = (0, 0). The center points allowed for testing and isolating curvature at both the whole-plot and sub-plot level. Replication provided degrees-of-freedom for estimating both the whole-plot variance and the sub-plot variance. The model contained terms for all linear effects, all two-factor interactions, and for the confounded sub-plot quadratic terms $\beta(x_1^2 + x_2^2)$ and the confounded whole-plot quadratic terms $\beta(z_1^2 + z_2^2)$.

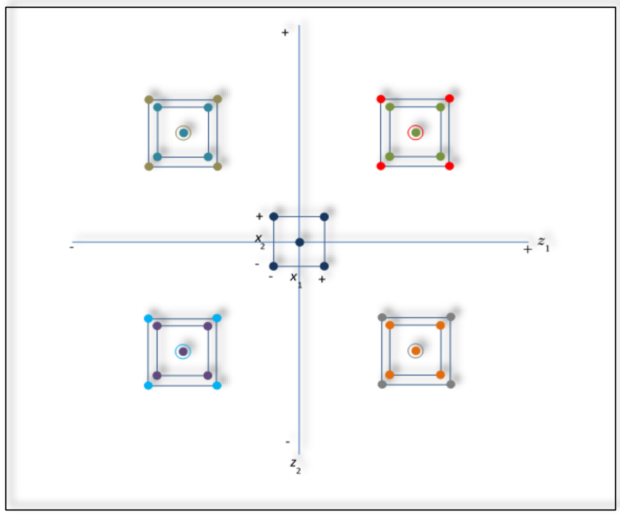


Figure 1-2. Split-plot design by Simpson, Kowalski, and Landman (2004)

Simpson, Kowalski, and Landman (2004) succinctly illuminated the many issues surrounding experimentation under restricted randomization:

- despite of the utility and advantages offered by split-plot experiments, they are frequently overlooked because of their complexity;
- sometimes split-plot experiments are not recognized as such and are inadvertently analyzed as if they were carried out as completely randomized experiments, which leads to inaccurate models;
- the selection of the correct error term for testing the significance of the factors and their interactions is sometimes unclear, especially when the split-plot design produces interactions between the whole-plot factors and the sub-plot factors;
- the whole-plot and sub-plot factor effects could be aliased, which leads to using incorrect error terms for the tests of significance;

- some designs are undesirably large, even for a reasonably small number of factors;
- sometimes the analysis of variance method produces a negative estimate of the interaction component that leads to a sub-plot variance component larger than the whole-plot variance component;
- replicating the whole-plot is necessary for estimating the whole-plot error term, which may increase the cost of the split-plot experiment relative to a completely randomized experiment;
- a typical approach for reducing the cost of split-plot experiments is to place more factors at the whole-plot level, which reduces the power to detect significant effects;

Although the type of situation presented by Simpson, Kowalski, and Landman (2004) was a departure from traditional experiment design, it is commonplace in industrial experiments. Similar situations often require carrying out a sequence of experiments in non-homogeneous settings, where a block factor needs to account for the variability introduced by the non-homogenous settings. Blocking adds complexity to an already complex problem:

- an effective blocking structure can increase the number of whole-plots, and therefore, the cost of the experiment;
- the interaction between blocks by whole-plots increases the experimental error;
- comparisons between blocks have lower precision than comparison within blocks;
- blocking decreases the number of degrees-of-freedom available to estimate effects, which reduces experimental power; and
- blocks increase the complexity of the design, the complexity of the analysis, and the complexity of the interpretation of results;

The relative growth that second-order split-plot design has experienced since Box and Wilson (1951) has been modest relative to the growth that other topics have experienced during the same period. Textbooks such as Montgomery (2004), Myers, Montgomery, and Anderson-Cook (2009), Box and Draper (2007), Cochran and Cox (1992), and Hinkelmann and Kempthorne (1994) provided a treatise of the subject; however, the treatise is not as complete as for other response surface methodology subjects. The peer-reviewed literature for second-order split-plot design, especially with blocking, is scarce, limited in the number of examples, and often provides limited guidelines. This deficit of information leaves practitioners ill prepared to face many of the roadblocks illuminated by Simpson, Kowalski, and Landman (2004). Thus, there is need for alternate approaches in second-order split-plot design, with and without blocking, especially for designs that minimize the amount of replication required to estimate the error terms at the whole-plot level. Similarly, there is a need to improve on the guidance for the selection of good block split-plot designs.

1.3 Research Objectives

The focus of the research was to develop practical strategies to help practitioners overcome some of the challenges presented by second-order split-plot design. Leveraging on the work by Simpson, Kowalski, and Landman (2004), the research aimed at:

- exploring systematic approaches for constructing second-order split-plot designs, with and without blocking;
- identifying or developing suitable second-order split-plot layouts and assessing their performance;
- developing guidance based upon knowledge of better design techniques;
- identifying adequate criterion to further expand the existing second-order split-plot design evaluation criteria; and
- comparing the performance of split-plot designs produced by the research to the performance of standard second-order split-plot designs to build the practitioner's confidence on these designs.

1.4 Application

Fisher (1926) introduced split-plot design. Split-plot design was extremely useful in agronomical experiments where the primary concern was first-order effects. Forms of the split-plot design, like Taguchi's inner and outer array design, found their way into industrial experiments. Taguchi's inner and outer array designs are highly regarded by the quality and manufacturing control community; however, Bisgaard (2000) indicated that they are not widely recognized as split-plot experiments and are incorrectly analyzed as if they were completely randomized designs often resulting in incorrect models. Another disadvantage of Taguchi's inner and outer array designs is the size of the design—they are produced by a full Cartesian product method and often require a number of runs beyond what would be considered practical or economical as shown in chapters 2 and 3.

Second-order split-plot designs began to receive significant attention for use in industrial experiments at the turn of the century. As shown in chapter 2, techniques for generating second-order split-plot experiments, with and without blocking, are complex and very limited. Vining, Kowalski, and Montgomery (2005), Parker, Kowalski, and Vining (2006), and Jones and Nachtsheim (2009) provided attractive designs that have good properties and that allow for estimating all of the model coefficients as well as whole-plot and sub-plot variances. As shown earlier in this chapter, blocking adds significant complexity to the design and analysis of second-order split-plot experiments. Only recently, Wang, Kowalski, and Vining (2009) and Verma *et.al*. (2012) considered designs that incorporate blocking for second-order split-plot experiments.

Letsinger, Myers, and Lentner (1996) investigated the effect of five process variables on a given (proprietary) response variable. The variables included two hard-to-change variables (temperature 1 and pressure 1) and three easy to-change variables (humidity 1, temperature 2, and pressure 2). A second-order model was expected to explain the relationships between the process variables and the response.

Trinca and Gilmour (2001) studied the application of a second-order split-plot design to maximize the yield and purities of two proteins in a protein-extraction process. The practitioners considered five factors: feed position, feed flow rate, gas flow rate, and the concentration of two proteins. The feed position was considered a hard-to-change factor while the other four factors were considered easy-to-change factors.

Vining, Kowalski, and Montgomery (2005) used a second-order split-plot design to study the effect of two-hard-to-change factors and two easy-to-change factors on the strength of ceramic pipe. The two hard-to-change factors were two temperatures in different zones of a furnace and the two easy-to-change factors were the amount of binder in the formulation and the grinding speed.

Myers, Montgomery, and Anderson-Cook (2009) presented an experiment involving four factors that affect the strength of an adhesive used in a medical application. Two of the factors are hard-to-change (whole-plot factors)—cure temperature, percent of resin in the adhesive—while the other two factors are easy-to-change (sub-plot factors)—amount of adhesive and cure time. A second-order model was thought to explain the relationship between the factors and the response.

English, Simpson, Landman, and Parker (2012) characterized the flight performance of a small-scale unmanned aerial vehicle developed for commercial and military operations. The experiment involved a second-order split-plot design with one hard-to-change factor (wing tip height) and two easy-to-change factors (angle-of-attack and yaw angle).

1.5 Original Contribution

An end-to-end, innovative approach for the construction of effective and efficient second-order block split-plot designs is provided by this research in chapters 3 and 4. First, the treatment design of classical second-order designs is partitioned into second-order sub-arrays, which are assigned to the treatments of the whole-plot factors and sub-plot factors. Then, the Cartesian product method is used to form ordered pairs of whole-plot sub-arrays by sub-plot sub-arrays rather than the ordered pairs of full whole-plot arrays by full sub-plot arrays produced by the Taguchi method. Then, the sub-array Cartesian products are concatenated into a new observation design that is referred to as *second-order sub-array Cartesian product split-plot design*. Finally, the sub-array structure is used to provide blocking strategies that permit effective and efficient use of resources.

This new form of second-order split-plot design, produced by this research and discussed in detail in chapter 3, is an alternative to split-plot designs like Taguchi's inner and outer array designs. They exhibit good properties, are practical, easy to construct, easy to evaluate, and in many cases they overcome some of the difficulties presented by other types of designs. The independent nature of the whole-plot and sub-plot sub-arrays facilitates the handling of split-plot designs as a superposition of two different experiments. They allow for estimating the model coefficients for all first-order, two-factor interactions, and pure second-order terms. When the whole-plot and sub-plot sub-arrays are second-order orthogonal blocks, the sub-array product is a second-order orthogonal block design, which is an appealing feature. As shown in chapter 3, the designs are economical, and generally require about one-half of the number of runs required by full Cartesian product designs.

Sub-array Cartesian product split-plot designs are high information-quality designs. The variance of the regression coefficients is low. Similarly, the prediction variance of the regression coefficients is low and stable. The aliasing between the terms in the model and likely effects that are not in the model as well as the correlation between similar effects that are not in the model is low. Based on an assessment using key design evaluation criterion established by Box and Draper (1975) and Myers, Montgomery, and Anderson-Cook (2009), it is demonstrated in chapters 3 and 4 that second-order sub-array Cartesian product split-plot designs perform as well or better than historical designs that have been considered standards up to this point.

1.6 Dissertation Outline

The scope of this research is limited to design, construction, and blocking strategies of conventional second-order split-plot experiments. The research scope addresses experimentation in a region known to contain an optimum or the experimental phase of response surface methodology where an experimental region has been found that contains an optimum. The research is not intended to provide a comprehensive treatise on all types of split-plot experiments. The research effort is restricted to experiments with $2 \leq p \leq 3$ whole-plot factors, $2 \leq q \leq 4$ sub-plot factors, and $k = p + q \leq 7$ total factors, which reflect the range of factors for most industrial split-plot applications.

A subset of the vast body of literature related to second-order split-plot design is discussed in Chapter 2. The seminal work by Fisher (1926), Yates (1935), Box and Wilson (1951), and by Box and Hunter (1957) is highlighted. Fundamental concepts of response surface methodology, blocking, split-plot design, and design evaluation criteria as well as significant contributions in those areas are discussed. Key contributions to second-order split-plot design by Letsinger, Myers, and Lentner (1996), Bisgaard (2000), Vining, Kowalski, and Montgomery (2005), Parker, Kowalski, and Vining (2007a, 2007b), and Jones and Nachtsheim (2009) are reviewed in more detail. Individual attention is placed to recent research in block second-order split-plot design by Wang, Kowalski, and Vining (2009) and by Verma *et. al*. (2012).

An innovative and efficient approach, rooted on traditional response surface methodology and on the adaptation of the Cartesian product method, to construct split-plot designs is provided in Chapter 3. The approach is referred to as *second-order sub-array Cartesian product split-plot design*. The construction of the design is illustrated with sub-arrays derived from central composite, Box-Behnken, and definitive screening designs. The performance of the design is assessed with criterion by Box and Draper (1975) and Myers, Montgomery, and Anderson-Cook (2009)—the pairwise correlation between model terms, the fraction of design space versus the unscaled prediction variance, and the unscaled prediction variance profile. Using the same criterion, the performance of the second-order sub-array Cartesian product split-plot design is reviewed against the performance of standard designs provided by Vining, Kowalski, and Montgomery (2005), Parker, Kowalski, and Vining (2007a), and Verma *et. al*. (2012).

In Chapter 4, a blocking strategy that allows for effective and efficient use of resources in concomitant homogeneous and heterogeneous settings is provided. The performance of the sub-array Cartesian product split-plot block design and the designs provided by Vining, Kowalski, and Montgomery (2005), Parker, Kowalski, and Vining (2007a), and Verma *et. al*. (2012) are assessed with the same criterion used in Chapter 3. A summary is provided in Chapter 5.

2 Literature Review

There is a vast body of literature related to response surface methodology, blocking, restricted randomization, and design evaluation criteria. Myers (1999) provided a review of and outlined the status of response surface methodology. Myers *et al.* (2004) reviewed the developments in response surface methodology from 1989 through 2004, including split-plot experiments, and synthesized the state-of-the-art and areas for research in robust parameter design, response surface designs, multiple responses, generalized linear models, and other topics. The paper presented a brief historical perspective, identified three extensive reviews conducted over the last 50 years, and provided an extensive bibliography. Khuri and Mukhopadhyay (2010) surveyed the development of response surface methodology and provided research directions.

The following sections summarize a subset of the body of literature related to this research. In the next section, the most popular response surface designs are discussed. In Section 2.5, some of the features of good response surface designs, of which the stability of the prediction variance is of outmost importance, are reviewed

Towards the end of the section covering split-plot design, the contributions of key papers, particularly, papers on the construction, design, and blocking of second-order split-plot designs are highlighted. Literature on second-order blocked split-plot designs is rare to find, and only three papers were found: Wang, Kowalski, and Vining (2009), Jensen and Kowalski (2012), and Verma *et. al*. (2012). For detailed information on topics related to this research refer to Montgomery (2004), Myers, Montgomery, and Anderson-Cook (2009), Box and Draper (2007), Cochran and Box (1992), and Hinkelmann and Kempthorne (1994).

2.1 Response Surface Design

Lind (1753) and Pierce and Jastrow (1885) recognized early on the importance of replication, randomization, and local control of error. Lind (1753) studied the systematic effects of citrus on scurvy while controlling the influence of external sources of variability and while replicating the observations. Pierce and Jastrow (1885) demonstrated the principles of randomization, replication, and blocking in an experiment designed to test the notion of a sensory differential threshold using pressure as the stimuli. Equally relevant, Pierce and Jastrow (1885) controlled the factors that influenced the responses, minimized the influence of the nuisance factors, and reset some factors between trials to prevent correlation. Pierce and Jastrow (1885) blocked on one factor and restricted the run order, thus, the experiment resembled a block split-plot experiment.

Fisher (1926) invented design of experiments for agronomic experiments, but through the years, it has found applications in many fields particularly in industrial experiments. Industrial experiments differ from agronomic experiments in two aspects. First, the results of industrial experiments are available almost immediately as oppose to the results of agronomic experiments that can take years. Thus, there is a sense of immediacy in industrial experiments. Second, industrial experiments can be carried out in sequence where the results from each run, or a smaller number of runs, can be used as a "stepping stone" to plan the next experiment. Thus, there is a sense of sequentiality. Box and Wilson (1951) jump-started the development of response surface methodology to take advantage of the immediacy and sequentiality aspects of industrial experimentation. Box (1992) outlined sequential experimentation and the sequential assembly of designs.

Gilmour and Trinca (2006) reinforced the importance of the fundamental principles of experiment design—randomization, replication, and local control of error—and offered advice for situations in which the variability between runs was high. First, the experiment should be design in large stages because the smaller designs are unreliable and changes in the path of steepest ascent could be due to errors. Second, the number of runs in the first stage of experimentation should be large to allow for fitting higher order terms without damaging the estimation of the linear effects model. Third, the experimental region should be large since the errors are larger and variance is more important than the large bias that lead to a decision of using a limited experimental region. Fourth, blocking is very important to isolate unwanted sources of variation. The algorithm by Gilmour and Trinca (2006) is simple: decide on the number of experimental runs, choose treatments with desirable properties for the number of runs available, and arrange the experiment in blocks to preserve the properties of the treatment designs as much as possible.

A common way to express Equation (1-2) in matrix notation is:

$$\boldsymbol{y} = \boldsymbol{X\beta} + \boldsymbol{\varepsilon} \tag{2-1}$$

where $\mathbf{y}$ represents the N x 1 vector of responses, $\mathbf{X}$ represents the N x t model matrix, $\boldsymbol{\beta}$ represents the t x 1 vector of the regression coefficients, and $\boldsymbol{\varepsilon}$ represents the N x 1 vector of random errors. The random error component incorporates measurement error, variability from uncontrolled factors, variability in the experimental units to which the treatments are applied to, general background noise, etc. The error is assumed to be an independent and identically distributed $\mathrm{N}(0,\sigma^2)$ random variable and the variance σ^2 is assumed constant throughout the experimental region. The vector of ordinary least squares estimators of $\boldsymbol{\beta}$ is

$$\widehat{\boldsymbol{\beta}}_{OLS} = (\boldsymbol{X'X})^{-1}\boldsymbol{X'y}$$

and

$$Var(\widehat{\boldsymbol{\beta}}_{OLS}) = \sigma^2(\boldsymbol{X'X})$$

$$\mathbf{M} = \frac{\mathbf{X'X}}{\mathrm{N}}$$

where $\boldsymbol{X'X}$ represents the information matrix, and M represents the moment matrix for a design with N points.

$\widehat{\boldsymbol{\beta}}_{OLS}$ is an unbiased estimator of $\boldsymbol{\beta}$ if the model is correct. Montgomery, Peck, and Vining (2012) established that $\widehat{\boldsymbol{\beta}}_{OLS}$ is the best linear unbiased estimator, which means that $\widehat{\boldsymbol{\beta}}_{OLS}$ has the smallest variance among all unbiased estimators that are linear combinations of the data. Assuming that $\boldsymbol{\varepsilon} = N(0,\sigma^2)$, $\widehat{\boldsymbol{\beta}}_{OLS}$ is also the maximum likelihood estimator of $\boldsymbol{\beta}$. When $E(\varepsilon) = 0$ and $VAR(\boldsymbol{\varepsilon}) = \sigma^2\Sigma$, where Σ is a known n x n matrix, the ordinary least squares estimator of $\hat{\beta}$ is no longer appropriate. The model is transformed to a new set of observations that leads to the generalized least squares estimator, which is:

$$\widehat{\boldsymbol{\beta}}_{GLS} = (\boldsymbol{X'\Sigma^{-1}X})^{-1}\boldsymbol{X'\Sigma^{-1}y}$$

and

$$Var(\widehat{\boldsymbol{\beta}}_{GLS}) = (\boldsymbol{X'\Sigma^{-1}X})^{-1}$$

Ordinary least squares and generalized least squares are two of many regression techniques; however, they serve as example to illustrate the problems associated with using a particular technique. There is not a single best regression method across the board. The best regression method to use is the one that fits the experimental situation. For more details, refer to Montgomery, Peck, and Vining (2012).

The fundamental purpose of a second-order experiment is to model a response surface over the design space to predict changes in the response variables due to changes in inputs variables. Equation (2-1) can be described by the fitted response surface equation:

$$\hat{\boldsymbol{y}}(x) = \beta_0 + \mathbf{x}'\hat{\boldsymbol{\beta}} + \mathbf{x}'\boldsymbol{B}\mathbf{x} + \boldsymbol{\varepsilon} \tag{2-2}$$

where $\mathbf{x}$ represents a point in the design space and $\mathbf{B}$ is an k x 1 matrix whose main diagonal elements represent the pure quadratic terms and the off-diagonal elements represent one-half of the interactions (mixed quadratic) terms. The unscaled prediction variance, which clearly is a measure of the stability of the model predictions, at any point $\mathbf{x}$ extended to the design space is

$$Var[\hat{\boldsymbol{y}}(\mathbf{x})] = \sigma^2\mathbf{x}'(\boldsymbol{X}'\boldsymbol{X})^{-1}\mathbf{x} \tag{2-3}$$

Typically, equation (2-3) is standardized, or scaled, by multiplying by $N/_{\sigma^2}$ to obtain the scaled prediction variance:

$$\frac{N}{\sigma^2}Var[\hat{\boldsymbol{y}}(\mathbf{x})] = N\mathbf{x}'(\boldsymbol{X}'\boldsymbol{X})^{-1}\mathbf{x} \tag{2-4a}$$

which is also expressed as:

$$\frac{Var[\hat{\boldsymbol{y}}(\mathbf{x})]}{\sigma^2} = \mathbf{x}'(\boldsymbol{X}'\boldsymbol{X})^{-1}\mathbf{x} \tag{2-4b}$$

Now, contrast the expressions for the completely randomized model above with the split-plot model. The simplest split-plot model in matrix form is given by:

$$\boldsymbol{y} = \boldsymbol{X\beta} + \boldsymbol{\delta} + \boldsymbol{\varepsilon} \tag{2-5}$$

where $\boldsymbol{y}$ represents the vector of responses, $\mathbf{X}$ represents the model matrix that includes both the whole-plot and the sub-plot terms, $\boldsymbol{\beta}$ represents the vector of regression coefficients, $\boldsymbol{\delta}$ represents the vector of whole-plot error terms, and $\boldsymbol{\varepsilon}$ represents the vector of the sub-plot error terms, and $\delta + \varepsilon \sim N(0, \Sigma)$. Letting σ^2 and σ_δ^2 represent the sub-plot error variance and the whole-plot error variance, and assuming that $\delta_k = N(0, \sigma_\delta^2)$ and $\varepsilon_{ij} = N(0, \sigma^2)$ are independent, the variance–covariance matrix of $\delta + \varepsilon$ is:

$$Var(y) = \boldsymbol{\Sigma} = \sigma^2\mathbf{I} + \sigma_\delta^2\mathbf{J}$$

where $\mathbf{J}$ is a block diagonal matrix of $\mathbf{1}_{\mathbf{bi}}$ x $\mathbf{1}'_{\mathbf{bi\ x\ 1}}$, $\mathbf{I}$ is a block diagonal identity matrix, b_i is the number of sub-plots within the i^{th} whole-plot. In situations where there a whole-plots each with b sub-plots,

$$\boldsymbol{\Sigma} = \begin{bmatrix} \boldsymbol{\Sigma}_1 & 0 & \dots & 0 \\ 0 & \boldsymbol{\Sigma}_2 & \dots & 0 \\ \dots. & \dots & \dots & \dots \\ 0 & 0 & \dots & \boldsymbol{\Sigma}_a \end{bmatrix}$$

where

$$\boldsymbol{\Sigma}_{\mathrm{i}} = \sigma_\delta^2 \mathrm{J}_{\mathrm{bxb}} + \sigma^2 \mathrm{I}_{\mathrm{bxb}} = \begin{pmatrix} \sigma_\delta^2 + \sigma^2 & \sigma_\delta^2 & \cdots & \sigma_\delta^2 \\ \sigma_\delta^2 & \sigma_\delta^2 + \sigma^2 & \cdots & \sigma_\delta^2 \\ \vdots & \ddots & \ddots & \vdots \\ \sigma_\delta^2 & \sigma_\delta^2 & \cdots & \sigma_\delta^2 + \sigma^2 \end{pmatrix} = \sigma^2 \begin{pmatrix} 1+\eta & \eta & \cdots & \eta \\ \eta & 1+\eta & \cdots & \eta \\ \vdots & \ddots & \ddots & \vdots \\ \eta & \eta & \cdots & 1+\eta \end{pmatrix}$$

and $\eta = \frac{\sigma_\delta^2}{\sigma^2}$ is the variance component ratio. The OLS and GLS estimates of $\boldsymbol{\beta}$ and the variance-covariance matrices for each of the estimates are:

$$\widehat{\boldsymbol{\beta}}_{OLS} = (\boldsymbol{X}'\boldsymbol{X})^{-1}\boldsymbol{X}'\boldsymbol{y}$$

$$Var(\widehat{\boldsymbol{\beta}}_{OLS}) = (\boldsymbol{X}'\boldsymbol{X})^{-1}\boldsymbol{X}'\boldsymbol{\Sigma}\mathbf{X}(\boldsymbol{X}'\boldsymbol{X})^{-1}$$

$$\widehat{\boldsymbol{\beta}}_{GLS} = (\boldsymbol{X}'\boldsymbol{\Sigma}^{-1}\boldsymbol{X})^{-1}\boldsymbol{X}'\boldsymbol{\Sigma}^{-1}\boldsymbol{y}$$

$$Var(\widehat{\boldsymbol{\beta}}_{GLS}) = (\boldsymbol{X}'\boldsymbol{\Sigma}^{-1}\boldsymbol{X})^{-1}$$

$$\mathbf{M} = \frac{(\mathbf{X}'\mathbf{\Sigma}^{-1}\mathbf{X})}{\mathrm{N}}$$

The predicted mean response at any point of interest $\mathbf{x}$ in the design space expanded to model form is location is:

$$\hat{y}(\mathbf{x}) = \mathbf{x}'\hat{\boldsymbol{\beta}} \qquad (2\text{-}6)$$

and the prediction variance and the scaled prediction variance at $\mathbf{x}$ is

$$Var[\hat{y}(\mathbf{x})] = \mathbf{x}'(\boldsymbol{X}'\mathbf{\Sigma}^{-1}\boldsymbol{X})^{-1}\mathbf{x} \qquad (2\text{-}7)$$

$$\frac{N}{\sigma_\delta^2+\sigma^2} Var[\hat{\boldsymbol{y}}(\mathbf{x})] = N\mathbf{x}'(\boldsymbol{X}'\mathbf{\Sigma}^{-1}\boldsymbol{X})^{-1}\mathbf{x} \qquad (2\text{-}8)$$

Clearly, the information matrix depends on the ratio of the variance components η. When a split-plot experiment is analyzed as if it was carried out as a completely randomized experiment leads to a larger variance, which makes it difficult for the tests of significance to find significant effects.

The OLS estimates is not the best linear unbiased estimator of $\boldsymbol{\beta}$. The GLS estimate is the best linear unbiased estimator if and only if σ^2 and σ_δ^2 are known and if there is no OLS-GLS equivalence. The estimates are equal if and only if a nonsingular matrix F exists such that $\mathbf{\Sigma}\boldsymbol{X} = \boldsymbol{XF}$. When the OLS and GLS coefficient estimates are equal, the variance-covariance matrix for both coefficient estimates is equal. The equivalence of OLS-GLS estimates in the context of second-order split-plot design is discussed in detail later on.

The most popular response surface designs for fitting second-order models are the central composite design and the Box-Behnken design. Another type is the 3^k general factorial design, which is generally inadequate for many industrial applications because of their size. The central composite design is the workhorse of response surface methodology. Resource constraint often leads to smaller or specialized designs. Figure 1-1 illustrates the central composite, the Box-Behnken, and the 3^k design for perspective.

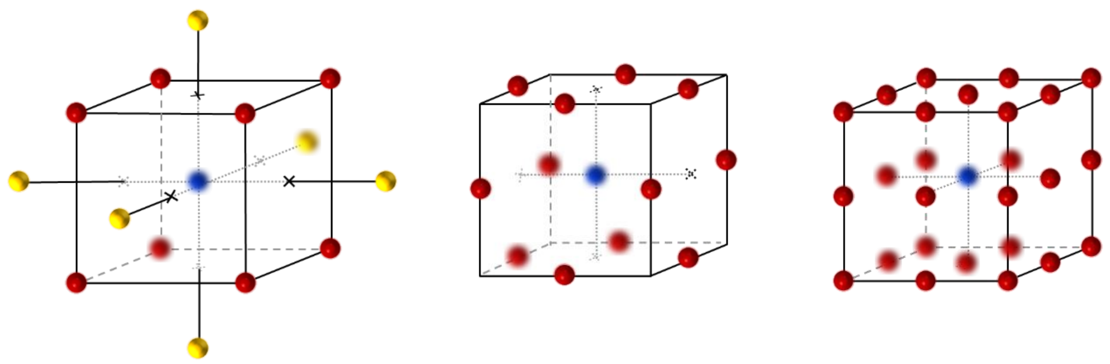


Figure 2-1. Central composite, Box-Behnken, and 3^k designs for k = 3

Central Composite Design

Box and Wilson (1951) introduced the central composite design. Central composite designs are five level designs, which for k factors consist of a combination of 2^k factorial or 2_V^{k-p} fractional factorial designs, center points, and axial points located a distance α from the center of the design. The 2^k or 2_V^{k-p} designs provide for estimating first-order effects, two-factor interactions, and lack of fit if there is replication. The center points provide for estimating pure-error, determining the presence of curvature, and for a more uniform estimation of the prediction variance. The axial points provide for estimating second-order effects. To fit a full second-order model with k factors, central composite designs require $2^k + 2k + k_0$ design points (k_0 is the number of center points) and can estimate $(k + 2)(k + 1)/2$ coefficients. Depending on the number of factors, some central composite designs have circular (k = 2), spherical (k = 3), or hyper-spherical (k > 3) symmetry. Regular central composite designs are orthogonal designs—designs that permit estimating all of the model parameters independently.

The distance α from the center of the design determines the features and properties of the design. The distance α is a function of the number of factors, their levels, and the desired properties of the design. The position of the axial points produces spherical designs (circumscribed and inscribed central composite designs) or cuboidal designs (face-centered central composite designs). Circumscribed central composite designs are rotatable and provide accurate estimates over the design space. A rotatable design is one in which the prediction variance is only a function of the distance from the center of the design. Inscribed central composite designs are rotatable and provide accurate estimates over the central region of the design space. The settings of the axial points at a distance α from the center for each of the factors produce five-level designs for both the circumscribed and inscribed central composite designs. Face-centered central composite designs are cuboidal designs that provide fair to poor estimates over the design space depending on the model. This type of design has the axial points at the center of each face of the cube at a distance $\alpha = \pm 1$, and produce factor settings at three levels. These designs are also produced by augmenting a factorial or resolution V fractional factorial design with axial points.

The addition of the axial points in a central composite design doubles the number of runs relative to a full factorial. Because of this increase, it is common for practitioners to carry out the experiment in blocks. Box and Hunter (1957) derived orthogonal blocking arrangements for several types of design, including an arrangement to block a central composite design in two orthogonal blocks. The arrangement for the central composite design consists of accommodating the 2^k or 2_V^{k-p} and center points in the first block and then accommodating the axial points and additional center points in the second block. Orthogonal blocking implies that the block effects do not affect the ability to estimate the model coefficients independently. Clearly, the location of the axial points and the number of center points have an effect on orthogonal blocking, which means that the block effects can interfere with the estimate of the second-order model coefficients.

The central composite design can be adapted to split-plot experiments. In those cases $k = p + q$, where p represents the number of whole-plot factors and q represents the number of sub-plot factors.

Box-Behnken Design

Box and Behnken (1960) introduced a family of three-level rotatable or near rotatable designs for fitting response surfaces. The construction technique relies on using balanced incomplete block designs and 2^k factorial designs. The design avoids the corners of the design space in favor of edge points located at the mid-level ($x_i = 0$) of the factor levels, which results in poor estimation at the factorial point locations. Thus, Box-Behnken designs are more useful for situations in which there is no interest in predicting at the factorial points of the cube. Like for central composite designs, replicated runs at the center points permit a more uniform estimation of the prediction variance over the design space. Some of the four or less factor designs are more economical than the central composite designs.

Practitioners often associate the Box-Behnken design with cuboidal regions because of its cubic appearance. However, the Box-Behnken design was not meant to be a cuboidal design, it is a spherical design. One can easily appreciate that feature by noting that for $k = 3$ the edge points are a distance of 1.414 from the design center. Because these designs are rotatable or near-rotatable, they require sufficient center points to improve their prediction accuracy.

3^k Design

Three-level factorial designs allow the estimation of curvature. However, there are situations where it is impractical to fit a second-order model with a classical design because it requires too many treatments, it has complex alias structure, or it cannot accommodate constraints associated with block structures, block sizes, or a reduced number of runs. Other situations involve irregular number of treatments, irregular or debarred regions of space, a combination of mixture and process variables, restricted randomization, or non-linear models. Designs that overcome those disadvantages are needed.

Optimal Design

Keifer (1959, 1961) and Keifer and Wolfwitz (1959) laid the foundation for evaluating and comparing designs based on optimal design theory. In this context, designs are optimal if they are “best” with respect to some criterion. The construction of optimal designs relies on an exchange algorithm where, starting from a grid of candidate points and an initial design, the algorithm attempts to improve the selected optimality criterion by exchanging points on the grid but not on the design with points that are on the design. These exchanges guarantee the selection of a nearly optimal design for one criterion. The design construction could be repeated starting from different initial designs until the near to optimal design is selected. The general approach for constructing a design is: (1) select the model to be fitted;

(2) determine the region of operability and constraints; (3) specify the number of runs; (4) specify the optimality criterion for evaluation; and (5) specify a number of design points.

Optimal designs are a good option whenever it is inadequate to use classical designs. While they are optimal according to a single criterion for a specified statistical model, they could be sub-optimal according to another criterion. The designs are model dependent and may require a model that the user may not have. The efficiency of these designs depends on the number of factors, the number of points, and the maximum standard error for prediction over the design space. Typically, the best design for an application is the design with the highest optimality efficiency. The designs have designations corresponding to the letters of the alphabet, such as D-, G-, I-, A-, V-, and E-optimality, to name a few. The most popular are the D-, G-, and I- (or Q-) optimal designs. Below is a list of some of the objectives:

- D-optimal - minimize the generalized variance of the regression coefficients;
- G-optimal - minimize the maximum scaled prediction variance over the design region;
- I-optimal - minimize the average scaled prediction variance over the design region;
- A-optimal - minimize the average variance of the regression coefficients;
- V-optimal - minimize the average prediction variance over a set of specific m points of interest in the design region;
- E-optimal - maximize the minimum eigenvalue of the information matrix

Small or Saturated Designs

There are situations in which scarce resources—funds, time, material, manpower, equipment—makes it impractical to allow the use of standard designs for fitting second-order models, especially when the number of factors k is high. For those situations, small or saturated response surface designs could be attractive. Some of the most popular small or saturated response surface designs and their features are:

- Hoke (1974) introduced saturated, non-orthogonal, second-order designs based on irregular fractions of partially balanced 3^k factorial designs (for $k > 2$). Hoke designs are suitable for cuboidal regions. For a small number of factors ($k < 7$), some of the designs are near-saturated and permit estimating pure error and lack-of-fit. Except for the quadratic terms, the best Hoke designs (for $2 < k < 10$) are comparable to Box and Behnken (1960) designs and Hartley (1959) small composite designs based on the determinant and the trace of the information matrix. Hoke designs have better prediction performance and similar sample size than Hartley small composite designs.

- Doehlert (1970) introduced the uniform shell designs (for $k < 11$) that have an equally spaced distribution of points lying on concentric spherical shells.
- Roquemore (1976) introduced the hybrid designs, a set of saturated or near saturated second-order rotatable or near-rotatable designs for $k = 3$, 4, 5, and 6. Hybrid designs are competitive with central composite designs based on the scaled prediction variance criteria.
- Box and Draper (1974) developed a class of cuboidal, minimum number of points second-order designs to fit the main effects plus interaction model that are optimal for $k = 2$ and 3 but not optimal for $k > 3$.
- Hartley (1959) small composite second-order designs are based on the idea that the cube portion of the composite design can be as low as a Resolution III fraction if the two-factor interactions are not aliased with other two-factor interactions.
- Westlake (1965) found second-order cuboidal designs for $k = 5$, 7, and 9 in 22, 40, and 62 runs expanding the idea from Hartley (1959).
- Rechtschaffner (1967) added a dummy factor, whose main effect and two-factor interactions are assumed to be zero, to saturated fractions of any 2^k and 3^k factorial designs of Resolution V to increases the degrees-of-freedom for estimating pure error and preserving the balanced structure of the design.
- Pesotchinsky (1972) found approximate minimum point D-optimal designs for a small number of factors ($k < 8$).
- Lucas (1974) constructed saturated D-optimum composite designs using a subset of points from the saturated Resolution V of Rechtschaffner (1967).
- Mitchell and Bayne (1978) used an exchange algorithm to find a k-run design that maximizes $|X'X|$ given the number of factors k, a specified model, and a set of candidate points.
- Notz (1982) introduced a class of 3^k (for $k < 7$) minimal point second-order designs with asymptotic D-efficiency of 1 relative to the number of factors k and the number of minimal points q.
- Draper (1985) developed cuboidal, near-saturated designs using the Plackett-Burman designs for the factorial portion of Hartley's small composite designs, which are a

compromise between a saturated small composite design and a central composite design allowing degrees-of-freedom for estimating lack-of-fit.

- Morris (2000) introduced the augmented-pairs designs, a class of three-level designs constructed by combining the levels of every pair of points in a two-level first-order design to form the third level.
- Oehlert and Whitcomb (2005) introduced the Minimum Run Resolution V (MR Res V) designs, which are a class of equireplicated, irregular fractions of 2^k designs constructed using the D-optimality criterion algorithm. These designs provide resolution V designs in fewer runs when regular fraction designs contain significantly more degrees-of-freedom than are needed to estimate the model up to two factor interactions. Judged only on a D-optimality criterion, these designs are more efficient than many other types of designs.
- Haines (2006) provided methods, recent developments, and new techniques for evaluation non-standard designs such as the San Cristobal design, which uses a quadratic response surface to k factors that are restricted to positive or zero.
- Gilmour (2004) introduced subset designs, a class of three-level response surface designs obtained by using subsets of 2^k factorial designs at levels of -1 and 1 for each combination of k factors while holding the other $q - k$ factors at their middle level.

Definitive Screening Design

Jones and Nachtsheim (2011) proposed a class of three-level screening designs for numeric $k > 5$. Definitive screening designs provide estimates of the main effects that are uncorrelated with two-factor interactions and pure quadratic terms. Because they are three-level designs, the quadratic effects are estimable. Definitive screening designs require $2k + 1$ runs. Two-factor interactions are only partially confounded with other two-factor interactions as opposed to Resolution IV screening designs in which two-factor interactions are completely confounded with other two-factor interactions. Pure quadratic effects are not completely confounded with interactions. Jones and Nachtsheim (2011) also provided an algorithm to calculate the pairwise correlation coefficient between two model terms.

2.2 Error-Control Design

Sometimes, practitioners cannot complete an experiment under homogenous settings, and the variability associated with those settings permeates through the response variables and inflates the experimental error. This could be a problem since a precise comparison between and within treatments to detect the effects of the factors of interest requires homogeneous experimental units—a key concept introduced by Fisher (1926). Blocking is a form of local control of error. In a block design, the variability of the experimental units is less than the variability of the experimental units before they were grouped into blocks. A block design is complete if each block contains all of the treatments. Otherwise, they are incomplete. Similarly, a block design is balanced if each block, which represents a level of the block factor, has an equal number of experimental units. Otherwise, they are unbalanced.

Experiment designs have three important components: the treatment design, the observation design, and the error-control design. The treatment design determines the combinations of the factor levels to be studied. The observation design outlines the sequence in which the experimental runs are executed and the level at which the observations are taken; consequently, it informs the relationship between the experimental units and observational units. The error-control design is the layout of the treatments in the experiment. The most common error-control designs for industrial applications are the completely randomized design, the randomized complete block design, and the incomplete block design. In completely randomized designs, the observational units are randomly assigned to treatments, and the experimental units are the observational units. In randomized complete block designs and incomplete block designs, the observational units are randomly assigned to treatments, the treatments are randomly assigned to blocks, and the experimental units are the blocks.

Randomized complete block designs have several advantages: blocking eliminates unwanted sources of variability and decreases the variance, which increases the statistical power; they are useful when the block represents only a single source of variability; and they can accommodate any number of treatments within the blocks. Randomized block designs, however, are not adequate when the blocks are non-homogeneous. An increase in the number of factors increases the risk of increasing the variability in the experimental units.

Khuri (2003) provided a study of response surface models with fixed and random block effects. Khuri (2003) established that blocking increases the prediction variance. However, if the design blocks orthogonally, the blocks do not influence the estimation of the model coefficients and the least squares estimators of the regression variables are the same as without blocking.

Box and Hunter (1957) and Khuri (1992) exposed the conditions for orthogonal blocking in second-order designs. Khuri (1992) generalized those conditions using matrix notation.

Condition (1) $$\sum_{u(l)=1}^{n_l} x_{ui} = 0 \qquad i = 1, 2,.., k,\ l = 1, 2,.., b \tag{2-9}$$

Condition (2) $$\sum_{u(l)=1}^{n_l} x_{ui} x_{uj} = 0 \qquad i \neq j = 1, 2,.., k,\ l = 1, 2,.., b \tag{2-10}$$

Condition (3) $$N \sum_{u(l)=1}^{n_l} x_{ui}^2 = n_b \sum_{u(l)=1}^{N} x_{ui}^2 \qquad i = 1, \ldots, k,\ l = 1, 2, \ldots, b \tag{2-11}$$

The first two conditions imply that the sum of products $x_0, x_1, \ldots, x_k$ is zero for each block, which means that every block (b) in the design must be a first-order orthogonal block design. The third condition is that the contribution to the sum of squares for each factor in each block must be proportional to the block size. Wang, Kowalski, and Vining (2009) extended the second-order orthogonal blocking conditions to second-order split-plot designs.

2.3 Split-Plot Design

Split-plot experiments have their origin in agronomic research during a period called the "complex experiments" period. Fisher (1926) introduced the split-plot design and laid the foundation for the design of experiments. Yates (1933) discussed confounding of main effects and orthogonality in split-plot experiments. Fisher (1925) and Fisher (1935) published what became standards for researchers and practitioners of design and analysis of experiments. Yates (1937) discussed the structure and analysis of split-plot experiments and pointed out their essential characteristics. Plackett and Burman (1946) published some efficient designs to estimate several main effects simultaneously. The design and analysis of split-plot experiments was well understood during this period. The complex experiments period laid the foundation for modern industrial split-plot experiment. Hinkelmann and Kempthorne (1994), Montgomery (2004), Federer and King (2007), and Myers, Montgomery, and Anderson-Cook (2009) discussed industrial split-plot experiments in detail.

Recall that the randomization in a split-plot design leads to a model with two error terms. Bisgaard and de Pinho (2004) showed that the whole-plot factors and their interactions have a larger variance than the sub-plot factors and their interactions. The variance of the whole-plot factors and their interactions has two components—one component coming from the whole-plot and another coming from the sub-plot. The variance of the sub-plot factors and their interactions with other factors has only one component, which is coming from the sub-plot. For any 2^k with N runs, p whole-plot factors, $q = k - p$ sub-plot factors, the variance for the whole-plot factors and their interaction, $\sigma^2_{wp-factor}$, and the variance for the sub-plot factors and any interaction with them, $\sigma^2_{sp-factor}$ are:

$$\sigma^2_{wp-factors} = \frac{4}{N}\left(2^q \sigma^2_\delta + \sigma^2\right) \qquad \sigma^2_{sp-factor} = \frac{4}{N}\sigma^2$$

Inadvertent split-plotting

Wooding (1973), Box (1999), Simpson, Kowalski, and Landman (2004), and Vining (2012) all recognized the role of split-plot experiments in industrial experiments, and warned us that often they are not recognized as such and are incorrectly analyzed as completely randomized designs. Daniel (1959) referred to this as "inadvertent plot splitting". The consequences of inadvertent plot splitting are the mixing of the whole-plot error and the sub-plot error, which inflates the variance of the regression coefficients in the model for the sub-plot factors and mask the effects of the whole-plot factors, which in turn leads to erroneous tests of significance. Based on the specifics of the design, the tests of significance for second-order split-plot designs are tests for purely sub-plot effects, tests for effectively whole-plot effects, and tests for effects somewhere in between.

Hader (1973) compared the distribution of the F-ratio between proper and improper randomization schemas and established the "within" mean squared error (MSE) under proper randomization is an unbiased estimate of σ^2 while under improper randomization the value is biased downwards between 11 – 15%. Likewise, the randomization distribution of the F-ratio is appreciably different between the proper and improper randomizations.

Lucas and Ju (1992) studied completely randomized, completely restricted (two equally sized replicates), and partially restricted (four equally sized replicates) run orders using a central composite design with three whole-plot factors and one sub-plot factor. The runs were equally divided into two blocks at each level of the whole-plot factors. They found that for restricted randomization, the residual standard deviation was much smaller and all regression coefficients except the linear and quadratic coefficients for the whole-plot factors have much smaller standard deviations.

Equally important as the inadvertent split-plotting problem described above, is the failure to reset the between consecutive runs that have the same factor levels in a completely randomized design. Ganju and Lucas (1997 and 1999) illustrated how a situation like this analyzed as if the levels were reset leads to inappropriate tests of significance. Ju and Lucas (2002) demonstrated that split-plot blocking can provide superior sub-plot parameter estimates than completely randomized designs.

Robust parameter design

Taguchi (1987) introduced the Taguchi Method, a philosophy that incorporates principles for making processes insensitive to noise factors and to the variation of input variables. While the objectives behind this philosophy are well structured, the method does not pay attention to randomization and blocking. Although the philosophy behind the Taguchi Method was controversial, it sparked a renewed interest in the 1990's for new approaches to design of experiments. After being neglected for many years, the design and analysis of split-plot experiments for industrial applications became a topic for research.

Box (1999) and Box and Jones (1992 and 2001) pointed out that industrial split-plot designs provide convenient and economical robust designs. Industrial split-plot designs include Taguchi inner and outer array designs, which are often analyzed incorrectly. Split-plot designs can be arranged with either the design effects or the environmental effects applied to the whole-plot or sub-plot. Regardless, all of the design factors by environmental factors interactions are estimated with the sub-plot error. It is extremely importance to discover the effects of the environmental factors on the design factors and the nature of their interactions.

Split-plot fractional factorial design – split-plot confounding

Bisgaard (2000) provided a comprehensive tutorial on fractional factorials in a split-plot structure using the aliasing structure as criteria for selecting a design instead of minimum aberration. Using split-plot confounding to take advantage of using different sub-plot designs was a significant contribution and an important step forward for using fractional factorials split-plots for industrial applications.

Split-plot fractional factorial design - minimum aberration

Minimum aberration designs minimize the number of main effects aliased with low-order interactions. Huang, Chen, and Voelkel (1998) constructed minimum aberration split-plot fractional factorial designs in two ways: (1) construct minimum aberration designs first for for the whole-plot and then for the sub-plot; and (2) find an overall minimum aberration design using an integer programming technique. Bingham and Sitter (1999a) used the minimum aberration criteria to rank some designs created by combining a fractional factorial design at the whole-plot level with a fractional factorial design at the sub-plot level while Bingham and Sitter (1999b) provided theoretical results. Bingham and Sitter (2001) exemplified the effect of restricted randomization on the choice of split-plot design for industrial applications while Loeppky and Sitter (2002) discussed the analysis of those experiments. Minimum aberration is based on two assumptions: (1) higher-order effects are less imortant that lower-order effects; and (2) effects of the same order are equally important. Because minimum aberration designs have a large number of whole-plots with a small number of sub-plots, they are viewed unfavorably for use in industrial applications.

D-optimal split-plot design

Goos and Vandenbroek (2001) developed an algorithm for constructing D-optimal split-plot designs. Goos (2002) addressed some aspects of block and split-plot optimal designs, but did not address second-order block split-plots. Goos and Vandenbroek (2003) constructed D-optimal split-plot designs with specific numbers of whole-plots. Goos (2006) provided an overview of block and split-plot designs and on the evaluation of designs via estimation-based and prediction-based criteria. Goos (2006) illustrated orthogonally blocked D-optimal designs and D-optimal split-plot designs for equivalent estimation. Macharia and Goos (2010) provided D-Optimal and D-efficient equivalent estimation second-order split-plot designs.

Jones and Nachtsheim (2009) proposed a D-optimal split-plot design algorithm that trades replicates at the center points of the whole-plot and sub-plots for sub-plot runs that are at the corners of the design region. Jones and Nachtsheim (2009) provided a comprehensive review of split-plot designs; however, they did not address second-order blocked split-plot designs.

Second-order split-plot design – cross designs

Letsinger, Myers, and Lentner (1996) introduced crossed bi-randomization designs and non-crossed bi-randomization designs. In crossed bi-randomization designs, every combination of the whole-plot factors ($\boldsymbol{z}$) is crossed with every combination of the sub-plot factors ($\boldsymbol{x}$) resulting in identical levels of $\boldsymbol{x}$ in each whole-plot $\boldsymbol{z}$. Conversely, non-crossed bi-randomization designs may contain a different number of sub-plots, or different levels, in each whole-plot. Letsinger, Myers, and Lentner (1996) considered several applications of unreplicated second-order split-plot designs and used ordinary least squares (OLS), generalized least squares (GLS), iterated reweighted least squares (IRLS), and restricted maximum likelihood (REML) to estimate and compare the model regression coefficients. REML outperformed the other estimation techniques and OLS was appropriate only when the whole-plots were balanced. Letsinger, Myers, and Lentner (1996) proved that OLS and GLS are equivalent if the sub-plot had the same experiment designs but did not prove the equivalence with other conditions.

Vining (2012) explained that for the cases reviewed by Letsinger, Myers, and Lentner (1996), REML outperformed the other estimation techniques because the response surface designs were unbalanced. Because the designs were unbalanced, the OLS and generalized least squares estimates were not equivalent; consequently, all of the techniques for estimating the model coefficients are better estimators than OLS. Particularly, GLS is best-unbiased linear estimator if the whole-plot and sub-plot variances are known.

Draper and John (1998) and Trinca and Gilmour (2001) recommended REML as alternative on how to estimate the variance components. REML is applicable to every possible split-plot design and provides good approximations for a good range of variance components; however, the variance component estimates depend on the specified model. Conversely, Bisgaard and Steinberg (1997) and Bisgaard (2000) used the equivalence between OLS-GLS for their first-order and first-order with interactions models. Bisgaard (2000) achieved OLS-GLS equivalence in partial confounding designs even though not all sub-plots had the same experiment design.

Second-order split-plot design - OLS-GLS equivalent estimation

Second-order OLS-GLS equivalent estimation split-plots designs have received significant attention since 2004. Vining, Kowalski, and Montgomery (2005) constructed OLS-GLS equivalent split-plot central composite and Box-Behnken designs, recommended options for obtaining balanced designs, and focused on estimating pure-error. Vining, Kowalski, and Montgomery (2005) derived the necessary conditions to achieve OLS-GLS equivalent estimates for the regression coefficients and proposed two strategies conducive to achieving this condition. One strategy is to arrange each whole-plot with identical sub-plot designs, which typically result in large designs. The other strategy is to use a second-order orthogonal design with an identical number of sub-plot runs, which requires augmenting the design with runs at the center point of the design.

OLS-GLS equivalent estimation designs have some good features. Their construction is independent of a priori knowledge of the variance components. They can be analyzed using GLS algorithms, which are available in most commercial software packages. They are easy to generate. They provide pure-error estimates of the variance components that are independent of the model, which can be used for lack-of-fit tests, but that require an increased number of runs to make possible the estimation. Pure-error estimates are important in the early stages of experimentation. However, many practitioners object to replicating the center points under the precept that center points contribute very little towards building a model. Exact tests can be derived for at least some of the coefficients.

Parker, Kowalski, and Vining (2006) provided a catalog of non-crossed OLS-GLS designs for equivalent estimation. Parker, Kowalski, and Vining (2007a) proposed methods for constructing balanced OLS-GLS equivalent estimation minimum whole-plot designs based on traditional response surface designs. The minimum whole-plot method is intended to reduce the number of whole-plots to the minimum number required to fit a second-order model by redistributing runs that the Vining, Kowalski, and Montgomery (2005) method allocate to overall center points to the whole-plot factorial points. Parker, Kowalski, and Vining (2007a) provided a catalog of balanced and unbalanced central composite designs and Box-Behnken designs while Parker, Kowalski, and Vining (2007b) provided a catalog of Box and Draper, Hoke near saturated, Notz saturated, and hybrid minimum whole-plot designs. Vining and Kowalski (2008) established the appropriate error terms for testing pure sub-plot effects and effective whole-plot effects. Sub-plot residuals are though as individual data values predicted by the sub-plot model and adjusted by the whole-plot mean. Vining (2012) wrapped it all together.

The designs by Vining, Kowalski, and Montgomery (2005) have the most exact tests relative to minimum whole-plot designs and have unrestricted axial values for both whole factors (β) and sub-plot factors (α) axial points. They preserve the OLS-GLS equivalence with model reduction and in designs with whole-plots that only have center point runs. OLS_GLS equivalent designs do not perform well relative to the D-optimality criteria due to the overabundance of runs at the center points of the whole-plots. For minimum whole-plot designs, the OLS-GLS equivalence depends on α and β, and it is not preserved with model reduction or for designs that have whole-plots in which all runs are at the center point.

Second-order block split-plot design

Wang, Kowalski, and Vining (2009) constructed OLS-GLS equivalent central composite block split-plot designs and OLS-GLS equivalent Box-Behnken block split-plot designs from the second-order equivalent estimation designs proposed by Vining, Kowalski, and Montgomery (2005) as well as from the minimum whole-plot designs proposed by Parker, Kowalski, and Vining (2005). While the designs by Wang, Kowalski, and Vining (2009) have many appealing features and properties, the interaction between whole-plot factors is confounded with the block effect in cases when there are only two whole-plot factors. Wang, Kowalski, and Vining (2009) extended the second-order orthogonal blocking conditions to second-order split-plot designs. The conditions are:

$$\sum_{u=1}^{l_1} \frac{z_{ui}}{l_1} = \sum_{u=1}^{l_2} \frac{z_{ui}}{l_2} = \sum_{u=1}^{N} \frac{z_{ui}}{N} \text{ and } \sum_{u=1}^{l_1} \frac{x_{uj}}{l_1} = \sum_{u=1}^{l_2} \frac{x_{uj}}{l_2} = \sum_{u=1}^{N} \frac{x_{uj}}{N} \tag{2-12}$$

$$\sum_{u=1}^{l_1} \frac{z_{ui}x_{uj}}{l_1} = \sum_{u=1}^{l_2} \frac{z_{ui}x_{uj}}{l_2} = \sum_{u=1}^{N} \frac{z_{ui}x_{uj}}{N} \tag{2-13}$$

$$\sum_{u=1}^{l_1} \frac{z_{ui}^2}{l_1} = \sum_{u=1}^{l_2} \frac{z_{ui}^2}{l_2} = \sum_{u=1}^{N} \frac{z_{ui}^2}{N} \text{ and } \sum_{u=1}^{l_1} \frac{x_{ui}^2}{l_1} = \sum_{u=1}^{l_2} \frac{x_{ui}^2}{l_2} = \sum_{u=1}^{N} \frac{z_{ui}^2}{N} \tag{2-14}$$

$i = 1, 2, \ldots, p; j = 1, 2, \ldots, q$

where p represents the number of whole-plot factors, q the number of sub-plot factors, z_{ui} the level of the uth run for the ith whole-plot factor, x_{ui} the level of the uth run for the jth sub-plot factor, l_i the number of sub-plot runs in the ith block, and N the total number of sub-plot runs. For balanced designs, l_1=$l_2 = \cdots = l$ and $l = {}^{N}/_{b}$.

Jensen and Kowalski (2012) used a central composite design to fit a second-order model to the results of a split-plot experiment involving two whole-plot factors and one sub-plot factor in the presence of blocking. Blocking was at the sub-plot level. The design satisfied the conditions for OLS-GLS equivalent estimation. The experiment presented unique challenges for estimating the error terms and for checking the model assumptions. The parameters estimates were obtained using GLS although they could have been obtained using the simpler OLS estimation.

Verma *et.al.* (2012) generated candidate split-plot designs and constructed balanced second-order block split-plot designs using designs by Dey (2009) and unbalanced second-order block split-plot designs using designs by Zhang *et. al.* (2011). Dey (2009) provided 3^k designs considering second-order orthogonal blocking. Zhang *et. al.* (2011) provided small Box-Behnken designs, but did not consider second-order orthogonal blocking. The block size used by Verma *et. al.* (2012) was two. The second-order split-plot designs derived from Dey

(2009) satisfied the second-order orthogonal blocking conditions; but the second-order split-plot designs derived from Zhang *et. al*. (2011) did not. The algorithm to construct a second-order orthogonally blocked design consisted of allocating sub-plots to whole-plots and whole-plots to blocks, sorting on certain factors and replicating whole-plots to achieve block balance, and then adding center points to the whole-plots to obtain a second-order design that blocks orthogonally. While cumbersome, this procedure is consistent with response surface methodology best practices.

Cartesian product split-plot design

Bisgaard (1992) coined the term Cartesian product to describe the scalar product technique that Taguchi (1987) used for crossing the inner factors array and the outer factors array in his product array experiments. The technique is similar the design construction technique proposed by Letsinger, Myers, and Lentner (1996), and to the technique using design arrays and environmental arrays that was introduced by Box and Jones (1992) for robust product design. Throughout this research, the inner factors array is referred to as the sub-plot array and the outer factors array is referred to as the whole-plot array.

Bisgaard (1992) pointed out that Taguchi's inner and outer array experiments are examples of slit-plot designs although they have not been widely recognized as such. It was not until the turn of the millennium, coincident with Bisgaard (2000) and Box and Jones (2001), that split-plot experiments began to gained due recognition as a valuable method for industrial experiments. Bisgaard (2000) and Box and Jones (2001) are re-publications of Bisgaard (1992) and Box and Jones (1992). Figure 2-2 show the relationships between whole-plot and sub-plot arrays, whole-plot and sub-plot factors, observations, and whole-plots.

Whole Plot Array (Z)		Sub-Plot Array (X)									Whole Plot Number
		−	−	+	+	0	0	$-\alpha$	α	0	x_2
z_1	z_2	−	+	−	+	$-\alpha$	α	0	0	0	x_1
−	−	y_{11}	y_{12}	y_{13}	y_{14}	y_{15}	y_{16}	y_{17}	y_{18}	y_{19}	1
+	−	y_{21}	y_{22}	y_{23}	y_{24}	y_{25}	y_{26}	y_{27}	y_{28}	y_{29}	2
−	+	y_{31}	y_{32}	y_{33}	y_{34}	y_{35}	y_{36}	y_{37}	y_{38}	y_{39}	3
+	+	y_{41}	y_{42}	y_{43}	y_{44}	y_{45}	y_{46}	y_{47}	y_{48}	y_{49}	4
$-\beta$	0	y_{51}	y_{52}	y_{53}	y_{54}	y_{55}	y_{56}	y_{57}	y_{58}	y_{59}	5
β	0	y_{61}	y_{62}	y_{63}	y_{64}	y_{65}	y_{66}	y_{67}	y_{68}	y_{69}	6
0	$-\beta$	y_{71}	y_{72}	y_{73}	y_{74}	y_{75}	y_{76}	y_{77}	y_{78}	y_{79}	7
0	β	y_{81}	y_{82}	y_{83}	y_{84}	y_{85}	y_{86}	y_{87}	y_{88}	y_{89}	8
0	0	y_{91}	y_{92}	y_{93}	y_{94}	y_{95}	y_{96}	y_{97}	y_{98}	y_{99}	9

Figure 2-2. Cartesian product of two whole-plot factors and two sub-plot factors

Taguchi's product array experiments are designed to determine the factors that influence a response. The experiments involve crossing 2^{k-p} x 2^{q-r} factorial or fractional factorial arrays, thus, they are limited to first-order effects. Another disadvantage of the Taguchi approach is size of the design, which often requires a number of runs beyond what seems practical or economical. However, the fundamental criticism of Taguchi's product array method is that experiments are carried out as split-plots, but analyzed incorrectly as if they were completely randomized. Bisgaard and Sutherland (2003) showed that Taguchi's famous tile experiment was a split-plot design although it was not recognized as such. They reanalyzed it using a standard split-plot approach.

While incorrectly analyzed, the Taguchi tile experiment was a showcase example for the utility of design of experiments. As discussed by Christenson (2007), Taguchi was an understudy of Dr. Edwards Deming, a strong proponent of the techniques developed by Sir Ronald Fisher. In 1953, Taguchi was working at the INA Seito tile manufacturing company. The INA Seito plant built a new costly kiln for baking the bricks; however, the heating conditions inside the device were not uniform and some of the tile that was placed near the kiln wall broke during baking. The INA Seito plant was at the brink of bankruptcy and replacing the kiln was not an option. Recognizing this issue, Taguchi changed the problem from replacing the kiln to re-formulating the clay. Taguchi identified eight different active ingredients in the clay and with 16 runs he demonstrated that a clay formulation with 5% more lime resulted in a more robust product. That experiment became part of the portfolio of big industrial tests that motivated Japan to embrace this methodology and catapulted the country to become the leader in product development for years to come. The rest is history. Taguchi's inner and outer array experiments are highly regarded by the quality and manufacturing control community. Most likely because of their success, they stifled the intellectual development of the design and analysis of split-plot experiments.

In Chapter 3, the Cartesian product to produce a split-plot design from the perspective of crossing whole-plot sub-arrays by sub-plot sub-arrays rather than from Taguchi's perspective of crossing whole-plot arrays by sub-plot arrays is provided. For fitting a second-order model, it is demonstrated that split-plots built from sub-arrays produce the same quality of information than those produced with arrays but they require substantially less runs.

2.4 Design Evaluation Criteria

The selection of an appropriate experimental design is often affected by factors such as the objective of the experiment, the homogeneity of the experimental units, the resources available to carry out the experiment, the complexity of the model to be fitted, and the capability to estimate internal error. Practitioners can select the most appropriate design by comparing different options over a wide range of characteristics.

General design evaluation criteria

Box and Wilson (1951) identified some characteristics of good response surface designs. Box and Hunter (1957), Box and Draper (1959), Box (1968), and more completely Box and Draper (1975) further refined and expanded those characteristics, which include:

1. distribute the information throughout the experimental region;
2. provide a good fit of the model to the data;
3. detect lack-of-fit;
4. allow transformations;
5. permit the experiment to be carried out in blocks;
6. allow for the sequential assembly of higher-order designs;
7. provide an estimation of internal error;
8. be robust to outliers and the gross violation of normal theory assumptions;

9. require a small number of experimental runs;
10. provide data patterns that allow visual appreciation;
11. ensure simple calculations;
12. be robust to errors in control of factor levels;
13. require a practical number of factor levels;
14. check the homogeneous variance assumption;

Some of the characteristics listed above are inclusive. Box and Hunter (1957) interpreted characteristic number 1 as to satisfy four other characteristics (3, 5, 6, 9). Montgomery (2004) reduced the list by Box and Draper (1975) but pointed out that a good design must provide a good prediction variance profile ($Var[\hat{y}(x)]/\sigma^2$) over the design region.

Clearly, there are tradeoffs in selecting a response surface design. The experimental situation dictates the relative importance of these characteristics. While it is not common to find a design that simultaneously has all of these characteristics, a good design does not need to have them all. Box and Draper (1975) make clear the inherent danger of relying on only a single criterion and recommend choosing a design that balances a number of characteristics. Similarly, Myers *et. al.* (2004) pointed out that the importance of design robustness is underscored by forcing the use of a single criterion. Anderson-Cook, Borror, and Montgomery (2009) discuss the criteria for selecting good designs.

Myers, Montgomery, and Anderson-Cook (2009) adapted the general guidelines to response surface split-plot designs. Notably, the features do not include the ability to conduct an experiment in blocks. Like for a good response surface design, a good split-plot design should balance a number of the characteristics by Myers, Montgomery, and Anderson-Cook (2009), which include:

1. provide a good fit of the model to the data;
2. allow a precise estimation of the model coefficients;
3. provide a good prediction over the experimental region
4. provide an estimation of both whole-plot variance and sub-plot variance;
5. detect lack-of-fit;
6. check the homogeneous variance assumption at the whole-plot and sub-plot levels;
7. consider the cost in setting the whole-plot and sub-plot factors;

8. ensure the simplicity of the design;
9. ensure simple calculations;
10. be robust to errors in control of factor levels;
11. be robust to outliers.

A great tool to compare and evaluate various designs are graphical methods, particularly, the use of the scaled prediction variance. As already discussed, the unscaled prediction variance and the scaled prediction variance for a design of size N are:

$$Var[\hat{\mathbf{y}}(\mathbf{x})] = \sigma^2 \mathbf{x}'(\boldsymbol{X}'\boldsymbol{X})^{-1}\mathbf{x} \quad (2\text{-}15)$$

$$\frac{N}{\sigma^2} Var[\hat{\mathbf{y}}(\mathbf{x})] = N\mathbf{x}'(\boldsymbol{X}'\boldsymbol{X})^{-1}\mathbf{x} \quad (2\text{-}16)$$

Box and Hunter (1957) noted that criteria based only on the variances of the model terms was insufficient for the selection of a second-order design, and concluded that a desirable property was a low prediction variance over the design space. The scaled prediction variance measures the precision of the estimated response over the design space. The estimates are a function of the design, the model, and the location of the prediction. A good design has a reasonably stable prediction variance over the design space. Graphs of the unscaled prediction variance are better tools for evaluating designs than single optimality criteria, especially if the optimal design has an unstable scaled prediction variance.

Zahran, Anderson-Cook, and Myers (2003) proposed using fraction of design space graphs for assessing the prediction capability of response surface designs. The fraction of design space graph plots the range of the scaled prediction variance against the cumulative fraction of the volume of the design space. Park *et.al.* (2005) discussed the prediction variance properties of second-order designs for cuboidal regions. Liang, Anderson-Cook, and Robinson (2006) adapted fraction of design plots to split-plot designs.

$$\frac{N}{\sigma_\delta^2+\sigma^2} Var[\hat{\mathbf{y}}(\mathbf{x})] = N\mathbf{x}'(\boldsymbol{X}'\boldsymbol{\Sigma}^{-1}\boldsymbol{X})^{-1}\mathbf{x} \quad (2\text{-}17)$$

As it will be seen next, minimizing the maximum scaled prediction variance over the design region is consistent with the G-optimality criteria. Similarly, minimizing the average prediction variance over the design region is consistent with I-optimality criteria.

Optimality Criteria

Optimality criterion provides a measure of how good a design is relative to a given objective function for a particular model. The criterion can be classified as information-based, distance-based, or compound. The most popular are D-, G-, Q-criterion.

D-criterion attempts to minimize the variance of the regression coefficients $|(\mathbf{X}'\mathbf{X})^{-1}|$. Since the determinant of $(\mathbf{X}'\mathbf{X})^{-1}$ reflects how well the coefficients are estimated (large implying poor estimation), the objective is to minimize it. The relative D-efficiency of two or more experiment designs is:

$$D_{eff} = \left(\frac{\left|(X_2'X_2)^{-1}\right|}{\left|(X_1'X_1)^{-1}\right|} \right)^{1/p} \qquad \text{or} \qquad D_{eff} = \left(\frac{|M(\xi^*)|}{Max|M(\xi)|} \right)^{1/p}$$

where $\mathbf{X}_1$ and $\mathbf{X}_2$ represent the $\mathbf{X}$ matrices for each design, p represents the number of model parameters, ξ^*represents the relative efficiency of a particular design over all designs ξ. For a split-plot experiment, the D-criterion is:

$$\max_{\mathrm{D}} \left| \frac{(\mathbf{X}'\mathbf{\Sigma}^{-1}\mathbf{X})}{\mathrm{N}} \right|$$

G-criterion attempts to minimize the maximum scaled prediction variance over R:

$$Min\left[Max\ \upsilon(x)\right] = Min\left[Max\ \frac{NVar\left[\hat{y}(x)\right]}{\sigma^2}\right]$$

As previously discussed, the scale prediction variance is an important measure when assessing the prediction performance of a design. For p parameters in a model at a location $\mathbf{x} = [\mathbf{x}_1, \mathbf{x}_2, \ldots, \mathbf{x}_N]$, the G-criterion for a split-plot design is:

$$\max \frac{\mathrm{NVar}\left[\hat{y}(x)\right]}{\sigma_\delta^2 + \sigma^2} \qquad \text{or} \qquad \max \mathrm{N}\mathbf{x}^{\mathbf{d}'}(\mathbf{X}'\Sigma^{-1}\mathbf{X})^{-1}\mathbf{x}^{\mathbf{d}}$$

Q-criterion (also called I- or IV-criterion) attempts to minimize the average scaled prediction variance by dividing v(x) by the volume of R:

$$Min\frac{1}{K}\int_R v(x)dx = Min\ Q(\xi)$$

where $K = \int_R dx$. A design that simultaneously minimizes the variances of all coefficients minimizes the value of the scaled prediction variance, $Q(\xi)$, averaged over the design region. The Q-efficiency for a design ξ is:

$$Q_{eff} = \frac{Min\ [Q(\xi)]}{Q(\xi^*)}$$

For a split-plot experiment, Q-criterion is:

$$\min_Q \frac{\mathrm{N}}{k}\int_R \mathrm{x}'^{\eta}\left[\mathbf{X}'\Sigma^{-1}\mathbf{X}\right]^{-1}\mathrm{x}^{\eta}d\mathrm{x}$$

High D-efficiency is an indication of a good estimation of the model coefficients in terms of generalized variance. High G-efficiency is an indication of good prediction capability in terms of minimizing the maximum scaled prediction variance in the region of interest. High Q-efficiency is an indication of good prediction capability in terms of the minimum average scaled prediction variance in the region of interest.

Cost

The cost of a completely randomized experiment is usually proportional to the overall number of runs because typically, every treatment costs the same amount. This assumption does not hold in a split-plot experiment because a split-plot involves some factors that are more costly to change than others are. Factors that are costly to change are usually assigned to the whole-plot factors, thus, the number of whole-plots drives the cost of the experiment. Because replication to obtain an estimate of the whole-plot variance drives the size of the experiment upwards, practitioners often correlate this increase in overall sample size with an increase in cost. However, the design structure shows that the largest contribution to the number of runs stems from changing the settings of the sub-plot factors. Generally, the cost of changing the settings of the whole-plot factors is significantly greater than the cost of changing the settings of the sub-plot factors. Therefore, it makes sense to judge the cost of a split-plot design by both the number of whole-plots and the number of runs within a whole-plot rather than by the number of total runs alone.

Bisgaard (2000) used cost as part of a multiple criteria to compare the value of the information from the split-plot design against the cost of the runs in a split-plot experiment. Parker, Anderson-Cook, Robinson, and Liang (2007) demonstrated an approach that incorporates a cost function for evaluating the performance of competing second-order split-plot designs, and argued that the number of whole-plots is as important or more than the total number of runs. Additionally, the cost of blocking a split-plot experiment needs to factors in the blocking structure, the number of whole-plots, and the types of whole-plots.

2.5 Summary

Replication, randomization, and local control of error have played an important role in experiments as early as Pierce and Jastrow (1885). Fisher (1926) embedded those principles in the fabric of the design of experiments and introduced the split-plot experiment for agronomic research. Box and Wilson (1951) catalyzed the application of design of experiments to industrial experiments and jumped-started the development of response surface methodology. While response surface methodology has experienced a significant growth since Box and Wilson (1951), the growth of the design and analysis of second-order split-plot experiments, with and without blocking, was not balanced.

The literature research validated the need for improving industrial second-order split-plot design, without and with blocking. There is a vast body of literature related to response surface methodology, blocking, restricted randomization, and design evaluation criteria. Although there is a vast amount of literature on first-order split-plot design, literature on second-order split-plot design, particularly with blocking, is more limited. Only two peer-reviewed papers could be found on the topic. Wang, Kowalski, and Vining (2009) proposed OLS-GLS equivalent estimation, orthogonally blocked central composite split-plot designs. Verma *et. al.* (2012) constructed balanced and unbalanced orthogonally blocked second-order split-plot designs from candidate designs provided by Dey (2009) and Zhang (2011).

Some candidate split-plot designs are too large or inadequate for the region of experimentation. Some of the techniques to generate split-plot designs are ineffective. Thus, there is a tremendous benefit on improving the techniques for generating split-plot designs. Simpson, Kowalski, and Landman (2004) emphasized the importance of adapting traditional response surface methods to fit specific needs while preserving the desirable properties of the response surface designs. These properties include low variance of the regression coefficients, low and stable prediction variance, low correlation (minimum aliasing) between the terms in the model and likely effects that are not in the model, and correlation coefficients between likely effects that are not in the model. The following criterion by Box and Draper (1975) are key in selecting good response surface designs:

- Provide a good fit of the model to the data. Box and Hunter (1957) suggested that this criterion satisfies four other criterion from Box and Draper (1975): detect lack-of-fit, a small number of runs, sequential assembly to higher order, and blocking. The only criterion adapted by Myers, Montgomery, and Anderson-Cook (2009) was the first. The sequential assembly of higher order designs and the ability to carry out the experiment in blocks are also considered two important characteristics for this research. Blocking is discussed in Chapter 4.
- Provide a good prediction over the experimental region. Criterion to be considered include the stability of the prediction variance over the entire design space, the prediction variance at the center of the design space, the maximum prediction variance, the average prediction variance, and the prediction variance at the 90th percentile of the design space.
- Allow a precise estimation of the model coefficients. A measure for this criterion is the pairwise correlation coefficients between model terms.
- Provide an estimation of both the whole-plot variance and the sub-plot variance. The whole-plot variance can be estimated by replicating the whole-plots while the sub-plot variance can be estimated by pooling the sub-plot center runs.

3 Second-Order Sub-Array Cartesian Product Split-Plot Design

A brief summary of the vast body of knowledge related to some aspects of the research was provided in Chapter 2: response surface design, error-control design, split-plot design, and design evaluation criteria. This chapter is dedicated to exploring the systematic construction of second-order split-plot designs using an innovative and effective technique that is consistent with the philosophy of traditional response surface methodology.

An adaptation of the Cartesian product method is used to cross specific arrangements of whole-plot factors and sub-plot factors, called sub-arrays, that are derived from central composite, Box-Behnken, and definitive screening designs to generate a form of split-plot design referred to as *second-order sub-array Cartesian product split-plot design*. Criterion consistent with the design evaluation criteria proposed by Box and Draper (1975) is used to evaluate the performance of the design: the pairwise correlation between model terms, the fraction of design space versus the unscaled prediction variance, and the unscaled prediction variance profile at the center of the design space. Finally, the performance of the design is assessed relative to some of the historical second-order split-plot designs. Among those historical designs are two standards that have served the response surface methodology community well: the OLS-GLS equivalent estimation design and the minimum whole-plot design.

3.1 Design Construction and Evaluation

The general steps for constructing a second-order sub-array Cartesian product split-plot design are:

1. Identify the whole-plot factors (z_1, z_2, …, z_p) and the sub-plot factors (x_1, x_2, …, x_q).
2. Select a second-order design for only the whole-plot factors ($\mathbb{D}_{\boldsymbol{W}}$) and a second-order design for only the sub-plot factors ($\mathbb{D}_{\boldsymbol{S}}$). $\mathbb{D}_{\boldsymbol{W}}$ and $\mathbb{D}_{\boldsymbol{S}}$ do not need to be the same, and should be appropriate for the experiment situation. Thus, $\mathbb{D}_{\boldsymbol{W}}$ and $\mathbb{D}_{S}$ can be any combination of central composite designs, Box-Behnken designs, etc.
3. Assign the whole-plot treatments to the whole-plot array (**W**) and the sub-plot treatments to the sub-plot array (**S**).

4. Partition $\mathbf{W}$ into Γ sub-arrays or blocks and $\mathbf{S}$ into Θ sub-arrays or blocks.
5. Use the Cartesian product method to cross the whole-plot sub-arrays and the sub-plot sub-arrays. The Cartesian product is the set of ordered pairs:

$$\mathbf{W_\Gamma} \times \mathbf{S_\Theta} = \{[(z_1, z_2, \ldots, z_p), (x_1, x_2, \ldots, x_q)] \mid (z_1, z_2, \ldots, z_p) \in \mathbf{W_\Gamma}\ (x_1, x_2, \ldots, x_q) \in \mathbf{S_\Theta}\}$$

6. Concatenate the sub-array Cartesian products into a design matrix.

$$\mathbf{M} = \mathbf{W_i} \times \mathbf{S_j} \mathbin{//} \ldots \mathbin{//} \mathbf{W_\Gamma} \times \mathbf{S_\Theta}\}$$

7. Arrange the block structure to preserve the properties of the treatment design and the observation design.

Steps one through three are considered the Treatment Design, step four is considered the Observation Design, and step five is considered the Error Control Design. Treatment Design and Observation Design are discussed in this chapter. Error-Control Design is discussed in Chapter 4. The construction, evaluation, and assessment of the sub-array second-order Cartesian product split-plot design is illustrated and discussed in terms of the number of sub-array partitions, the size of the sub-arrays, the second-order structure of the array design ($\mathbb{D}_{\boldsymbol{W}}$ and $\mathbb{D}_{\boldsymbol{S}}$), the allocation of treatments to the sub-arrays, and the axial distances α and β. The distance from the center of the sub-plot array to the sub-plot axial points is denoted by $\propto$ and the distance from the center of the whole-plot array to the whole-plot axial points is denote by β. Note that $\propto$ and β do not need to be equal.

One sub-array partition ($\Gamma = \Theta = 1$); $p = q = 2$

To illustrate the construction of a design, consider an experiment with $p = 2$ whole-plot factors and $q = 2$ sub-plot factors. The whole-plot array $\boldsymbol{W_0}$ is the arrangement of the whole-plot factors z_1 and z_2 in a central composite design arrangement ($\mathbb{D}_{\boldsymbol{W}}$). Similarly, the sub-plot array $\boldsymbol{S_0}$ is the arrangement of the sub-plot factors x_1 and x_2 in a central composite design arrangement ($\mathbb{D}_{\boldsymbol{S}}$). The array arrangements are illustrated in Figure 3-1. In terms of nomenclature, a design array is the same as a one-partition sub-array.

$\boldsymbol{W_0}$	
z_1	z_2
-1	-1
1	-1
-1	1
1	1
$-\beta$	0
β	0
0	$-\beta$
0	β
0	0

Whole-plot array

$\boldsymbol{S_0}$	
x_1	x_2
-1	-1
1	-1
-1	1
1	1
$-\alpha$	0
α	0
0	$-\alpha$
0	α
0	0

Sub-plot array

Figure 3-1. Whole-plot and sub-plot sub-arrays from central composite designs ($\Gamma = \Theta = 1$)

The Cartesian product of the whole-plot by sub-plot sub-arrays in Figure 3-1 is given by the product of $\boldsymbol{W_0} = \{(z_1,z_2)\} = \{(-1,-1)\ (1,-1)\ (-1,1)\ (1,1)\ (-\beta,0)\ (\beta,0)\ (0,-\beta)\ (0,\beta)\ (0,0)\}$ by $\boldsymbol{S_0} = \{(x_1,x_2)\} = \{(-1,-1)\ (1,-1)\ (-1,1)\ (1,1)\ (-\alpha,0)\ (\alpha,0)\ (0,-\alpha)\ (0,\alpha)\ (0,0)\}$. The result is the 81 ordered pairs $\{(z_1, z_2),(x_1, x_2)\}$ represented by the 4-fold (z_1, z_2, x_1, x_2) product in Figure 3-2 and Table 3-1. Because there were only one whole-plot sub-array and one sub-plot sub-array, there was no need to partition the sub-array or to concatenate the product of the sub-arrays.

The split-plot design consists of nine unreplicated, balanced whole-plots with nine sub-plot runs. The whole-plot structure consists of one whole-plot at each of the four factorial points $(z_1, z_2) = (\pm 1, \pm 1)$, one whole-plot at each of the four axial points $(z_1, z_2) = (\pm\beta, 0)$ and $(z_1, z_2) = (0, \pm\beta)$, and one whole-plot at the whole-plot center $(z_1, z_2) = (0, 0)$. Each whole-plot contains one sub-plot run at each of the four factorial points $(x_1, x_2) = (\pm 1, \pm 1)$, one at each of the four axial points $(x_1, x_2) = (\pm\alpha, 0)$ and $(x_1, x_2) = (0, \pm\alpha)$, and one at the sub-plot center $(x_1, x_2) = (0, 0)$. Although the design does not provide degress-of-freedom to estimate the whole-plot or sub-plot variances, the estimates can be obtained by augmenting the whole-plot array with one row $(z_1, z_2) = (0, 0)$, which provides a replicate that permits estimating the whole-plot variance. Similarly, estimates of the sub-plot variance can be obtained by augmenting the sub-plot array with one row $(x_1, x_2) = (0, 0)$, which provides a replicate for every sub-plot center point that can be pooled to estimate sub-plot variance. Replicating the whole-plot center point $(z_1, z_2) = (0, 0)$ provides a modest single degree-of-freedom for testing curvature; however, replicating the sub-plot center point $(x_1, x_2) = (0, 0)$ at each of the factorial and axial points of (z_1, z_2) helps isolating the contribution of each quadratic term to the non-linearity of the model. For the resulting design in Table 3-1, $p = 2$, $q = 2$, $k = p + q = 4$, $w = 9$, $s = 9$, and $N = w \times s = 81$.

When $\Gamma = \Theta = 1$, the Cartesian product method produces designs with a randomized complete block error-control design for both the sub-plot treatments and the whole-plot treatments without the need of concatenating the design matrix. These designs are typically large; however, they are practical for situations where a large variation between run to run at the sub-plot level exists and a large sample size is affordable. The whole-plots produced by this method are balanced and have identical sub-plots. A balanced design is one in which all treatment combinations have the same number of observations. Balance minimizes the standard error associated with the regression coefficients.

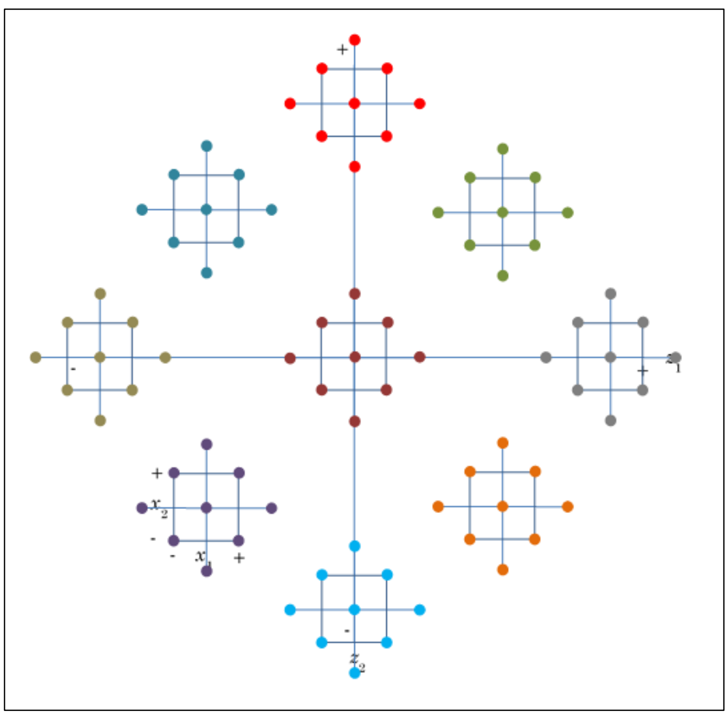


Figure 3-2. Cartesian product split-plot design for ($p = q = 2$; $\Gamma = \Theta = 1$)

Table 3-1. Cartesian product split-plot design ($p = q = 2$; $\Gamma = \Theta = 1$)

w_i	z_1	z_2	x_1	x_2	w_i	z_1	z_2	x_1	x_2	w_i	z_1	z_2	x_1	x_2
	−1	−1	−1	−1		1	1	−1	−1		0	$-\beta$	−1	−1
	−1	−1	1	−1		1	1	1	−1		0	$-\beta$	1	−1
	−1	−1	−1	1		1	1	−1	1		0	$-\beta$	−1	1
	−1	−1	1	1		1	1	1	1		0	$-\beta$	1	1
1	−1	−1	$-\alpha$	0	4	1	1	$-\alpha$	0	7	0	$-\beta$	$-\alpha$	0
	−1	−1	α	0		1	1	α	0		0	$-\beta$	α	0
	−1	−1	0	$-\alpha$		1	1	0	$-\alpha$		0	$-\beta$	0	$-\alpha$
	−1	−1	0	α		1	1	0	α		0	$-\beta$	0	α
	−1	−1	0	0		1	1	0	0		0	$-\beta$	0	0
	1	−1	−1	−1		$-\beta$	0	−1	−1		0	β	−1	−1
	1	−1	1	−1		$-\beta$	0	1	−1		0	β	1	−1
	1	−1	−1	1		$-\beta$	0	−1	1		0	β	−1	1
	1	−1	1	1		$-\beta$	0	1	1		0	β	1	1
2	1	−1	$-\alpha$	0	5	$-\beta$	0	$-\alpha$	0	8	0	β	$-\alpha$	0
	1	−1	α	0		$-\beta$	0	α	0		0	β	α	0
	1	−1	0	$-\alpha$		$-\beta$	0	0	$-\alpha$		0	β	0	$-\alpha$
	1	−1	0	α		$-\beta$	0	0	α		0	β	0	α
	1	−1	0	0		$-\beta$	0	0	0		0	β	0	0
	−1	1	−1	−1		β	0	−1	−1		0	0	−1	−1
	−1	1	1	−1		β	0	1	−1		0	0	1	−1
	−1	1	−1	1		β	0	−1	1		0	0	−1	1
	−1	1	1	1		β	0	1	1		0	0	1	1
3	−1	1	$-\alpha$	0	6	β	0	$-\alpha$	0	9	0	0	$-\alpha$	0
	−1	1	α	0		β	0	α	0		0	0	α	0
	−1	1	0	$-\alpha$		β	0	0	$-\alpha$		0	0	0	$-\alpha$
	−1	1	0	α		β	0	0	α		0	0	0	α
	−1	1	0	0		β	0	0	0		0	0	0	0

Because the whole-plots produced by this method are balanced and have identical sub-plots, the shorthand notation in Table 3-2 is used from this point forward to replace the long form in Table 3-1. In Table 3-2, w_i represents the i^{th} whole-plot, z_{1m} and z_{2n} represent the m^{th} and n^{th} levels of the whole-plot factors z_1 and z_2, and S_0 represents the complete sub-plot array.

Table 3-2. Shorthand for the Cartesian product split-plot design from Table 3-1

w_i	z_1	z_2	S_0
1	-1	-1	S_0
2	1	-1	S_0
3	-1	1	S_0
4	1	1	S_0
5	$-\beta$	0	S_0
6	β	0	S_0
7	0	$-\beta$	S_0
8	0	β	S_0
9	0	0	S_0

where S_0 =

x_1	x_2
-1	-1
1	-1
-1	1
1	1
$-\alpha$	0
α	0
0	$-\alpha$
0	α
0	0

The performance of the design is explored using three well-known key criterion: the pairwise correlation between model terms (Figure 3-3), the fraction of design space versus the unscaled prediction variance (Figure 3-4), and the unscaled prediction variance profile (Figure 3-5). The graphs in Figure 3-3 through 3-5 were constructed using the JMP 11 software by SAS for $\alpha = \beta = 1.414$. Figure 3-3 illustrates the pairwise correlation between model terms. The cells on the graphs represent the pairwise correlation coefficients between model terms as a colored cell with an intensity adjustable between 0 and 1. The correlation coefficient progresses from 0 (in color gray), which indicates that all of the coefficients are independent, to a maximum of 1 (in color burgundy), which indicates perfect correlation. Ideally, it is desirable to have perfect correlation in the diagonal line of cells and no correlation in the off-diagonal cells. Clearly, the graph shows that there is no complete correlation between model terms, whether or not they are determined by the tests of significance to have an influence on the response. The only terms partially correlated are the whole-plot pure quadratics with each other (0.64) and the sub-plot pure quadratics with each other (0.64). This type of correlation is trademark of the central composite design, as it will be shown later in this chapter, although the coefficient of 0.64 for the sub-plot pure quadratics could be a concern for certain industrial applications. All of the model terms for the main effects and the two-factor interactions are clear from correlating with other terms.

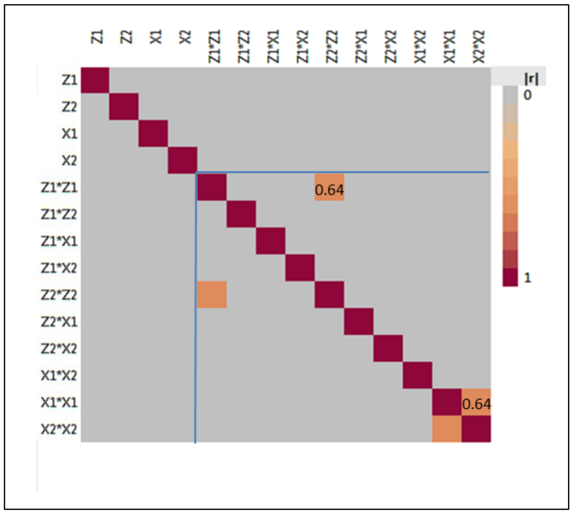


Figure 3-3. Pairwise correlation between model terms

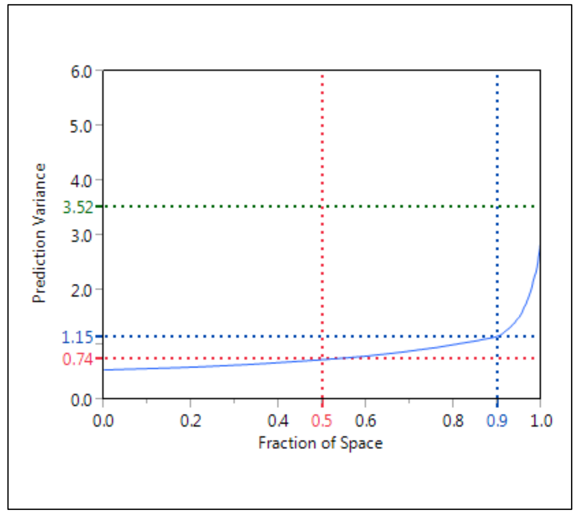


Figure 3-4. Prediction variance vs. fraction of design space

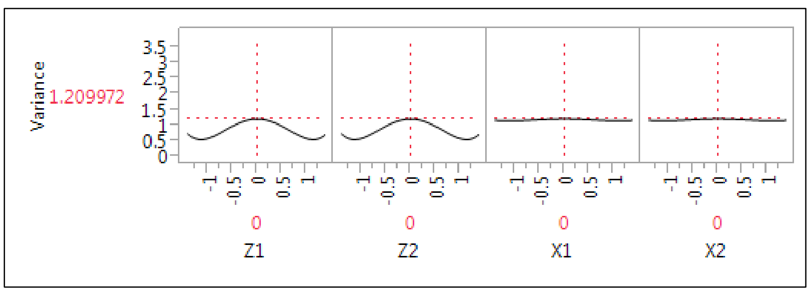


Figure 3-5. Unscaled prediction variance profile.

Figure 3.4 shows the unscaled prediction variance as a function of the fraction of design space. There graph shows that the design has a reasonably stable prediction variance, although not constant, up to about 90% of the fraction of design space (1.15). The maximum prediction variance is 3.52. Figure 3-5 shows that the unscaled prediction variance at the center of the design space (0, 0, 0, 0) is 1.21. This design is adequate; however, there are many situations in which N = 81 is unaffordable, especially in destructive testing. The average prediction variance is 0.86.

Consider now the construction of a design with p = 2 and q = 2 but with both the whole-plot array $\boldsymbol{W}_0$ and the sub-plot array $\boldsymbol{S}_0$ in the Box-Behnken design arrangements ($\mathbb{D}_{\boldsymbol{W}} = \mathbb{D}_{\boldsymbol{S}}$) shown in Figure 3-6. Note that they are like the sub-arrays from the central composite design except that $\alpha = \beta = 1$. This is also the same arrangement obtained for sub-arrays derived from face centered central composite designs and definitive screening designs. This is an interesting feature since it allows the experimenter to think in terms of split-plot factor properties rather than in the particular properties of a design. The performance of the design as a function of α and β is explored later in this chapter in detail.

$\boldsymbol{W}_0$	
z_1	z_2
−1	−1
1	−1
−1	1
1	1
−1	0
1	0
0	−1
0	1
0	0

Whole-plot array

$\boldsymbol{S}_0$	
x_1	x_2
−1	−1
1	−1
−1	1
1	1
−1	0
1	0
0	−1
0	1
0	0

Sub-plot array

Figure 3-6. Whole-plot and sub-plot sub-arrays from Box-Behnken designs ($\Gamma = \Theta = 1$)

Thus, this research is directed towards exploring the constructing of more affordable designs and evaluating their properties. A reduction in size can be obtained by partitioning the **W** and **S** arrays into sub-arrays. Although there are many possible ways to partition the arrays, traditional response surface methodology concepts and best practices are followed to narrow down the choices. One of the best practices followed is the use of center points to get degrees-of-freedom to estimate pure error and to test for curvature.

Two balanced sub-arrays ($\Gamma = \Theta = 2$); $p = q = 2$

Figure 3-7 illustrates the partitioning of both the whole-plot array and the sub-plot array into two sub-arrays each ($\Gamma = \Theta = 2$). Clearly, the sub-array $\mathbf{W}_1$ corresponds to the factorial points of the whole-plot central composite design augmented with one center point and the sub-array **W₂** corresponds to the axial points of the whole-plot central composite design augmented with one center point. The sub-plot sub-arrays $\mathbf{S}_1$ and $\mathbf{S}_2$ can be described in similar terms.

Allocation 1

Whole-plot sub-arrays

$\boldsymbol{W_1}$	
z_1	z_2
-1	-1
1	-1
-1	1
1	1
0	0

$\boldsymbol{W_2}$	
z_1	z_2
$-\beta$	0
β	0
0	$-\beta$
0	β
0	0

Sub-plot sub-arrays

$\boldsymbol{S_1}$	
x_1	x_2
-1	-1
1	-1
-1	1
1	1
0	0

$\boldsymbol{S_2}$	
x_1	x_2
$-\alpha$	0
α	0
0	$-\alpha$
0	α
0	0

Figure 3-7. Whole-plot and sub-plot sub-arrays from central composite designs ($\Gamma = \Theta = 2$)

Crossing the sub-arrays using the Cartesian product method can produce several candidate designs with different characteristics. Because the interested is in estimating the model coefficients for all whole-plot and sub-plot terms, the concatenated design matrix requires for each sub-array to be crossed only once, inedependently of the pairing. Under that rule, second-order sub-array Cartesian product split-plot designs can estimate the model coefficients for the first-order terms, the first-order with interaction terms, and the pure quadratic whole-plot factors, but they do not neccesarily estimate the model coefficients for the pure quadratic sub-plot terms.

Crossing the sub-arrays in Figure 3-7 results in four second-order sub-array Cartesian product split-plot designs, of which only two permit the estimation of all model coefficients. Those two designs are illustrated in Figure 3-8. The other two do not permit estimating the pure sub-plot quadratic terms and have a stronger correlation between model terms.

The designs are identified with the notation D($\Gamma\mathbb{D}_W p$, $\Theta\mathbb{D}_S q$). For $\mathbb{D}_W$ and $\mathbb{D}_S$, the letters B, C, and D are used to indicate that the sub-arrays were partitioned from a Box-

Behnken array, from a central composite array, or from a definitive screening array, although that is not of significance. Also, for simplicity, the sub-arrays are numbered sequentially from this point forward. Table 3-3 shows the sub-array Cartesian product split-plot design D(2C2, 2C2)-1 from Figure 3-8. Table 3-4 illustrates design D(2C2, 2C2)-2 from the same figure. The parameters for the designs are $p = 2$, $q = 2$, $k = p + q = 4$, $w = 10$, $s = 5$, and $N = w \times s = 50$.

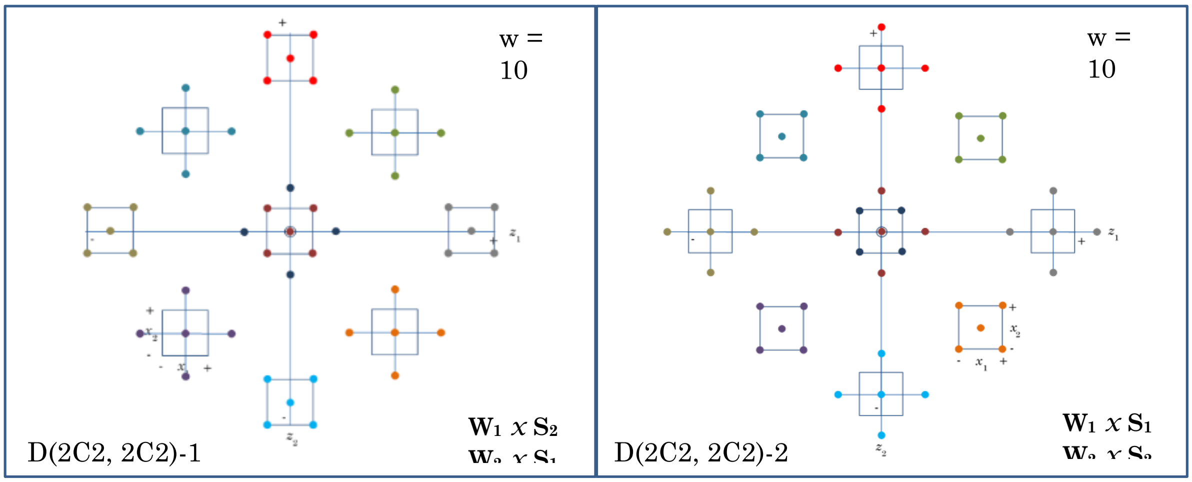


Figure 3-8. Second-order sub-array Cartesian product split-plot designs ($\Gamma = \Theta = 2$)

In the case of designs derived from central composite design arrays, the whole-plots are always balanced and the structure at the sub-plot level is identical. Because the designs are balanced, the variance of the difference between two treatment effects is the same for all pairs of treatments. The whole-plots have the same number of sub-plots. This feature makes easier the task of managing the structure of the whole-plots and sub-plots. Additionally, the analysis of balanced designs is much easier than unbalanced designs.

The whole-plot main effects, whole-plot by whole-plot interactions, and whole-plot pure quadratics effects are associated with the whole-plot effects. The sub-plot main effects, sub-plot by sub-plot interactions, sub-plot by whole-plot interactions, and pure sub-plot quadratic effects are associated with the sub-plot effects. However, it is easy to generate a design that has the sub-plot pure quadratics associated with the whole-plot effects if the experimental situation requires it.

Table 3-3. Second-order sub-array Cartesian product split-plot design D(2C2, 2C2)-1

w_i	z_1	z_2	S
1	−1	−1	S_2
2	1	−1	S_2
3	−1	1	S_2
4	1	1	S_2
5	0	0	S_2
6	$-\beta$	0	S_1
7	β	0	S_1
8	0	$-\beta$	S_1
9	0	β	S_1
10	0	0	S_1

S_1	
x_1	x_2
−1	−1
1	−1
−1	1
1	1
0	0

S_2	
x_1	x_2
$-\alpha$	0
α	0
0	$-\alpha$
0	α
0	0

Table 3-4. Second-order sub-array Cartesian product split-plot design D(2C2, 2C2)-2

w_i	z_1	z_2	S
1	−1	−1	S_1
2	1	−1	S_1
3	−1	1	S_1
4	1	1	S_1
5	0	0	S_1
6	$-\beta$	0	S_2
7	β	0	S_2
8	0	$-\beta$	S_2
9	0	β	S_2
10	0	0	S_2

S_1	
x_1	x_2
−1	−1
1	−1
−1	1
1	1
0	0

S_2	
x_1	x_2
$-\alpha$	0
α	0
0	$-\alpha$
0	α
0	0

Figures 3-9 illustrates the pairwise correlation between model terms as a function of α and β for the design in Table 3-3, D(2C2, 2C2)-1. The design is first-order orthogonal. There is no complete confounding. The only terms partially correlated are the whole-plot pure quadratic terms, the sub-plot pure quadratic terms, and in some cases whole-plot pure quadratic terms with sub-plot plot pure quadratic terms. For a given β and α pair, the pairwise correlation parameters are not identical due to the differences in geometries. The number of cells with pairwise correlation coefficients > 0.0 varied between two and six (out of a possible 91) and had values between 0.17 and 0.48, which is insignificant. As it will be seen in the next section, this type of confounding is typical, to varying degrees, of central composite split-plot designs.

Figure 3-10 illustrates the unscaled prediction variance as a function of the fraction of design space. The prediction variance is stable through the design space, and fluctuated between 0.60 for the design with $\alpha = \beta = 2.0$ and 1.14 for the design with $\alpha = \beta = 1.0$. Figure 3-11 illustrates the prediction variance profile. The unscaled prediction variance at the center of the design space varied between 0.46 to 0.68, which is adequate. Table 3-5 shows the average prediction variance as a function of α and β.

Table 3-5. Average unscaled prediction variance D(2C2, 2C2)-1, Figure 3-8

	$\alpha = 1.0$	$\alpha = 1.414$	$\alpha = 2.0$
$\beta = 2.0$	0.68	0.62	0.60
$\beta = 1.414$	0.86	0.80	0.76
$\beta = 1.0$	1.14	1.05	1.0

Judged solely by the pairwise correlation between model terms (Figure 3-9), the design with $\alpha = \beta = 1.0$ demonstrates better characteristics. However, when judged by the prediction variance vs. the fraction of design space (Figure 3-10), the design with $\alpha = \beta = 2.0$ slightly outperforms the other designs. Furthermore, when using the unscaled prediction variance at the center of the design space (Figure 3-11), the design with $\alpha = 1.0$; $\beta = 2.0$ and the design with $\alpha = 2.0$; $\beta = 1.0$ exhibit better characteristics.

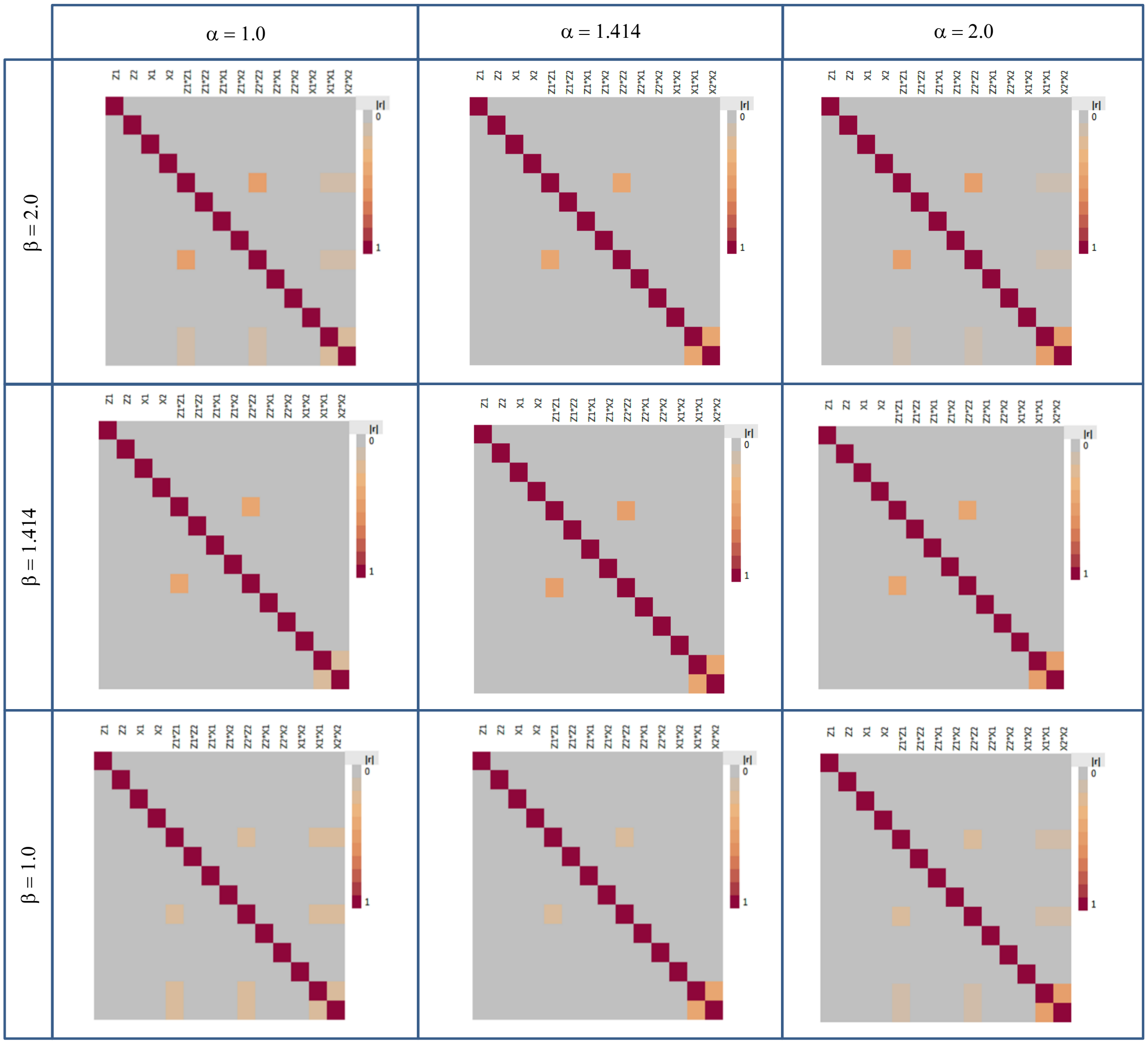


Figure 3-9. Pairwise correlation between model terms as a function of α and β for D(2C2, 2C2)-1, Figure 3-8

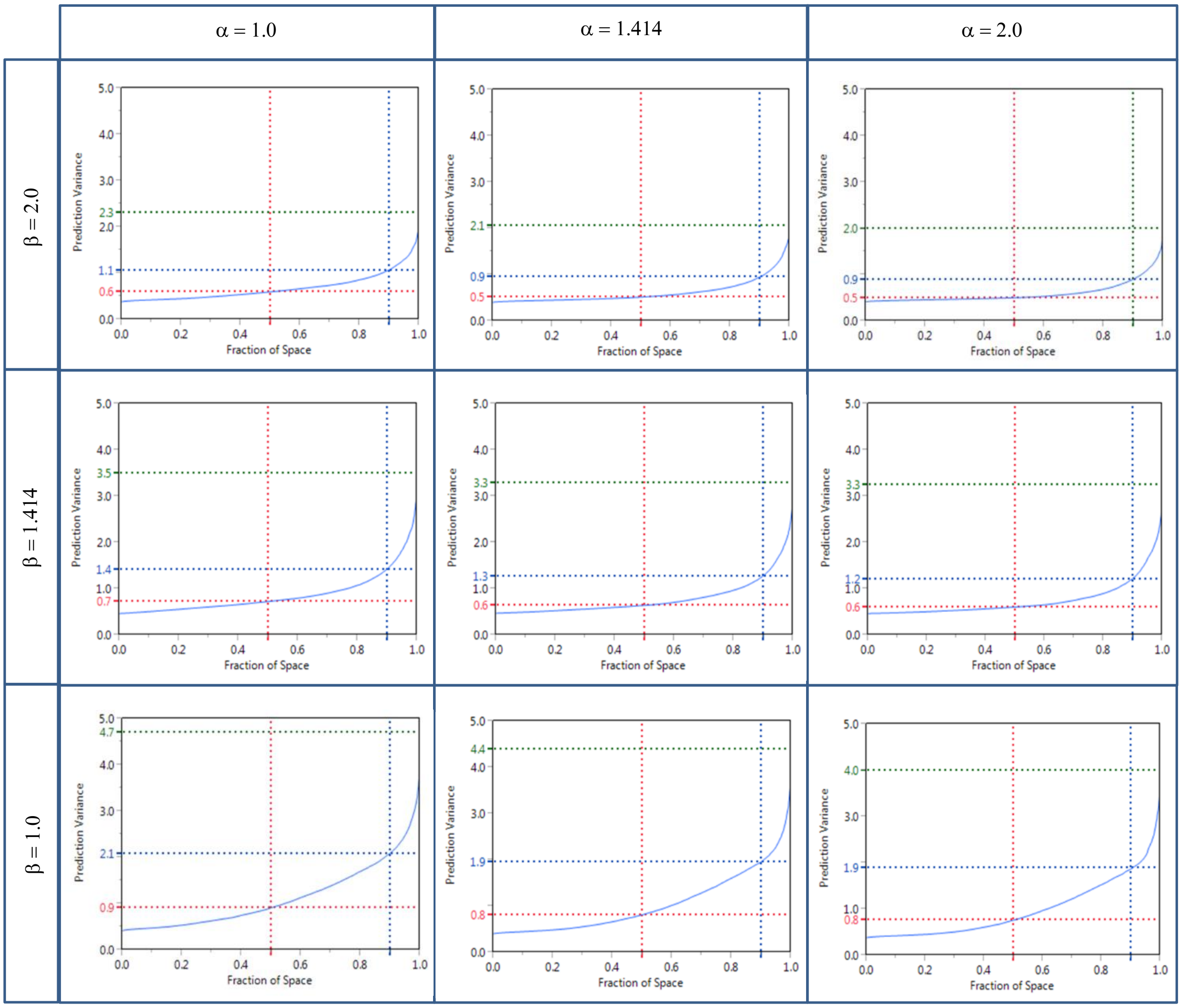


Figure 3-10. Prediction Variance vs. Fraction of Design Space for D(2C2, 2C2)-1, Figure 3-8

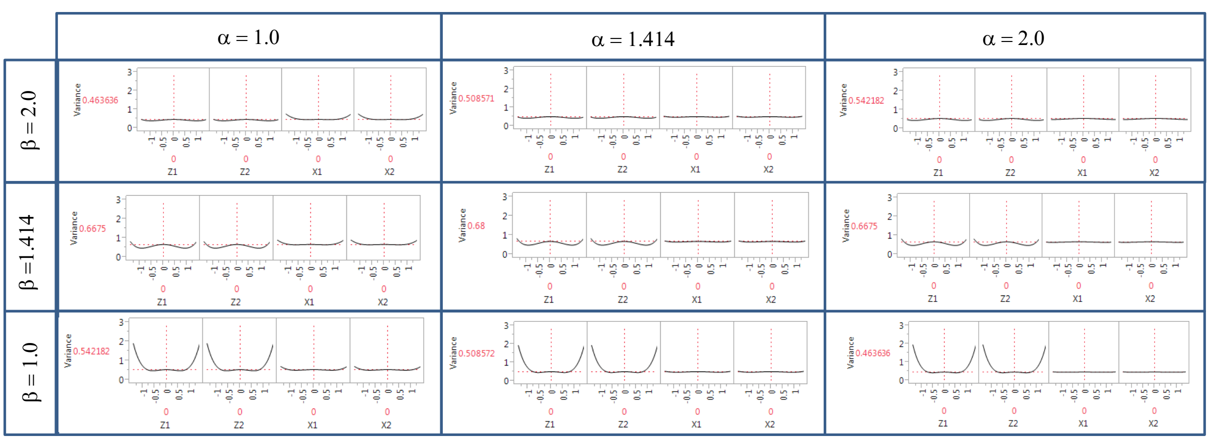


Figure 3-11. Unscaled Prediction Variance Profile for D(2C2, 2C2)-1, Figure 3-8

Earlier, it was mentioned that one of the factors that influence the performance of the designs are the treatment combinations within the sub-arrays. For instance, design D(2C2, 2C2)-1b in Figure 3-13 was created by exchanging some of the treatments between sub-plots sub-arrays $\mathbf{S}_1$ and $\mathbf{S}_2$ in Figure 3-7 to form $\mathbf{S}_3$ and $\mathbf{S}_4$ in Figure 3-12(a), and then taking the product $\mathbf{W}_1 \times \mathbf{S}_3$ // $\mathbf{W}_2 \times \mathbf{S}_4$, where the symbol // denotes the vertical concatenation of the sub-arrays. Similarly, D(2C2, 2C2)-1c was created by exchanging some of the treatments between $\mathbf{W}_1$ and $\mathbf{W}_2$ to form $\mathbf{W}_3$ and $\mathbf{W}_4$ in Figure 3-12(b) and then taking the product $\mathbf{W}_3 \times \mathbf{S}_3$ // $\mathbf{W}_4 \times \mathbf{S}_4$.

While the prediction variance is unaffected by the re-allocation of treatments, the effect of changing the composition of the sub-arrays is more noticeable in the pairwise correlation coefficients. Design D(2C2, 2C2)-1c exhibits partial confounding between sub-plot main effects, between main effects and two-factor interactions, between pure quadratic and first order terms, between pure quadratics and two-factor interactions, and between pure quadratics with other pure quadratics.

Three balanced sub-arrays ($\Gamma = \Theta = 3$); $p = q = 2$

The partitioning into three sub-arrays ($\Gamma = \Theta = 3$) is illustrated in Figure 3-14. The sub-array $\mathbf{W}_5$ corresponds to the factorial points of the whole-plot central composite design, the sub-array $\mathbf{W}_6$ corresponds to the axial points of the whole-plot central composite design, and the sub-array $\mathbf{W}_7$ corresponds to the whole-plot central composite design center point augmented with three additional center points to preserve balance. The sub-array $\mathbf{W}_5$ is useful for estimating main effects, two-factor interactions and testing for curvature. The sub-array $\mathbf{W}_6$ is useful for estimating quadratic effects. The sub-array $\mathbf{W}_7$ is useful for estimating whole-plot pure-error.

Twenty-seven different split-plot designs were produced by applying the design construction technique to the sub-arrays illustrated in Figure 3-14. Of those 27 designs, four allow for estimating the model coefficients for all terms. The layouts for those four designs are illustrated in Figure 3-15 ($\alpha = \beta = 1.414$) along with the layouts for D(3C2, 3C2)-6 and D(3C2, 3C2)-1. The layout for D(3C2, 3C2)-6 did not produce a design while the layout for D(3C2, 3C2)-1 did not allow for estimating the coefficient for x_2^2.

The four layouts that produced designs that allow for estimating all of the model terms have twelve balanced whole-plots with four sub-plot runs per whole-plot. Because **W**$_7$ contains four whole-plot center points, the designs have four replicated overall center points that allow for estimating the whole-plot variance. The performance of the designs is illustrated in Figures 3-16 through 3-18. The interpretation of the pairwise correlation coefficient plots and of the variance prediction graphs is consistent with the interpretation already provided. D(3C2, 3C2)-2, -3, -4, and -5 have identical pairwise correlation coefficients between model terms (six correlated terms with a value of 0.20), identical prediction variance at the center of the design space (0.94), and identical average prediction variance (0.92)

(a) Allocation 2

Whole-plot sub-arrays

W_1	
z_1	z_2
-1	-1
1	-1
-1	1
1	1
0	0

W_2	
z_1	z_2
$-\beta$	0
β	0
0	$-\beta$
0	β
0	0

Sub-plot sub-arrays

S_3	
x_1	x_2
-1	1
1	-1
$-\alpha$	0
0	$-\alpha$
0	0

S_4	
x_1	x_2
-1	-1
1	1
α	0
0	α
0	0

(b) Allocation 3

Whole-plot sub-arrays

W_3	
z_1	z_2
-1	1
1	-1
$-\beta$	0
0	$-\beta$
0	0

W_4	
z_1	z_2
-1	-1
1	1
β	0
0	β
0	0

Sub-plot sub-arrays

S_3	
x_1	x_2
-1	1
1	-1
$-\alpha$	0
0	$-\alpha$
0	0

S_4	
x_1	x_2
-1	-1
1	1
α	0
0	α
0	0

Figure 3-12. Re-allocation of whole-plot and sub-plot sub-arrays treatments ($\Gamma = \Theta = 2$)

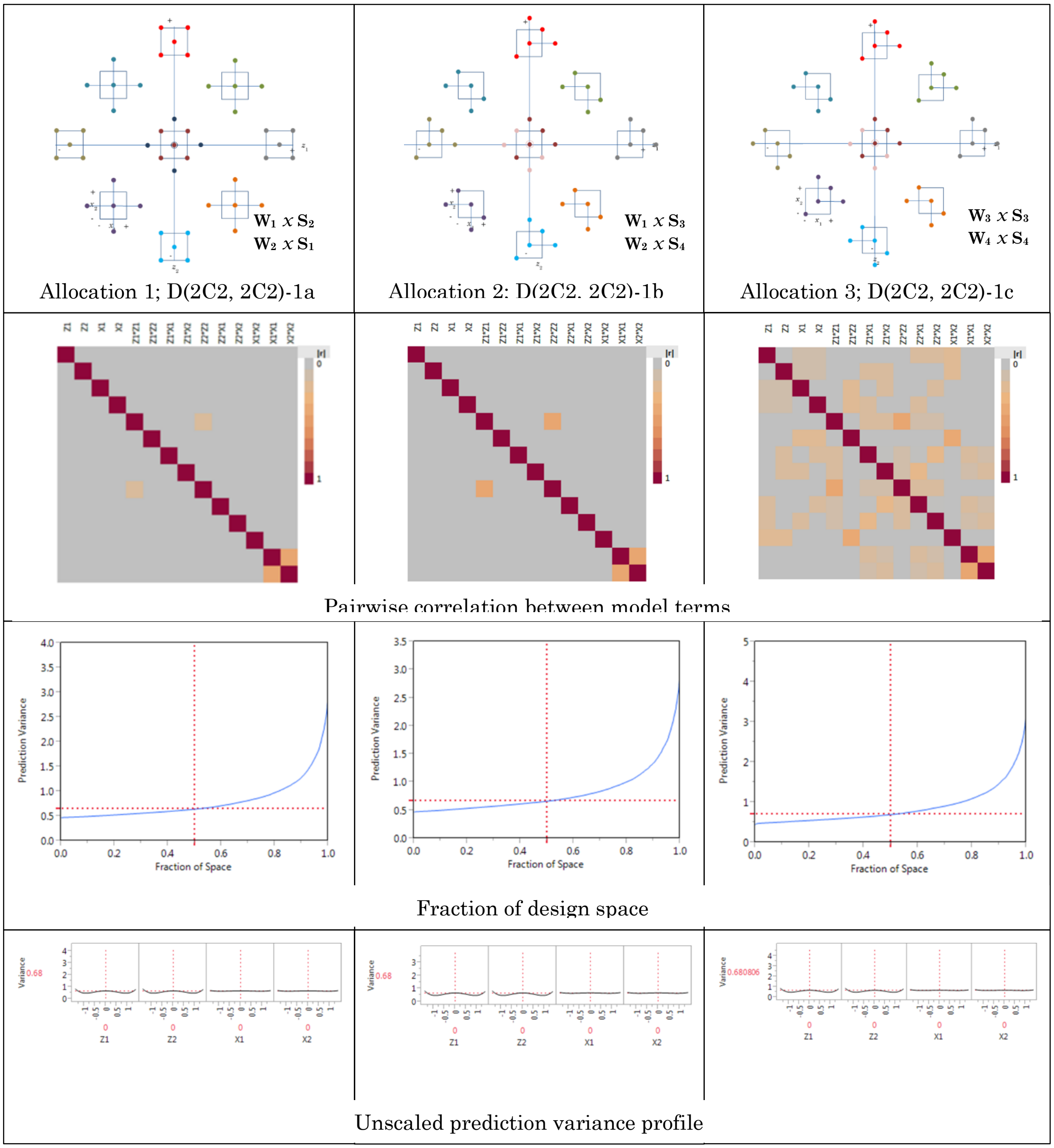


Figure 3-13. Cartesian product split-plot designs with re-allocated factor level treatments ($p = q = 2$; $\Gamma = \Theta = 2$)

W_5	
z_1	z_2
-1	-1
1	-1
-1	1
1	1

W_6	
z_1	z_2
$-\beta$	0
β	0
0	$-\beta$
0	β

W_7	
z_1	z_2
0	0
0	0
0	0
0	0

Whole-plot sub-arrays

S_5	
x_1	x_2
-1	-1
1	-1
-1	1
1	1

S_6	
x_1	x_2
$-\alpha$	0
α	0
0	$-\alpha$
0	α

S_7	
x_1	x_2
0	0
0	0
0	0
0	0

Sub-plot sub-arrays

Figure 3-14. Whole-plot and sub-plot sub-arrays from central composite designs ($\Gamma = \Theta = 3$)

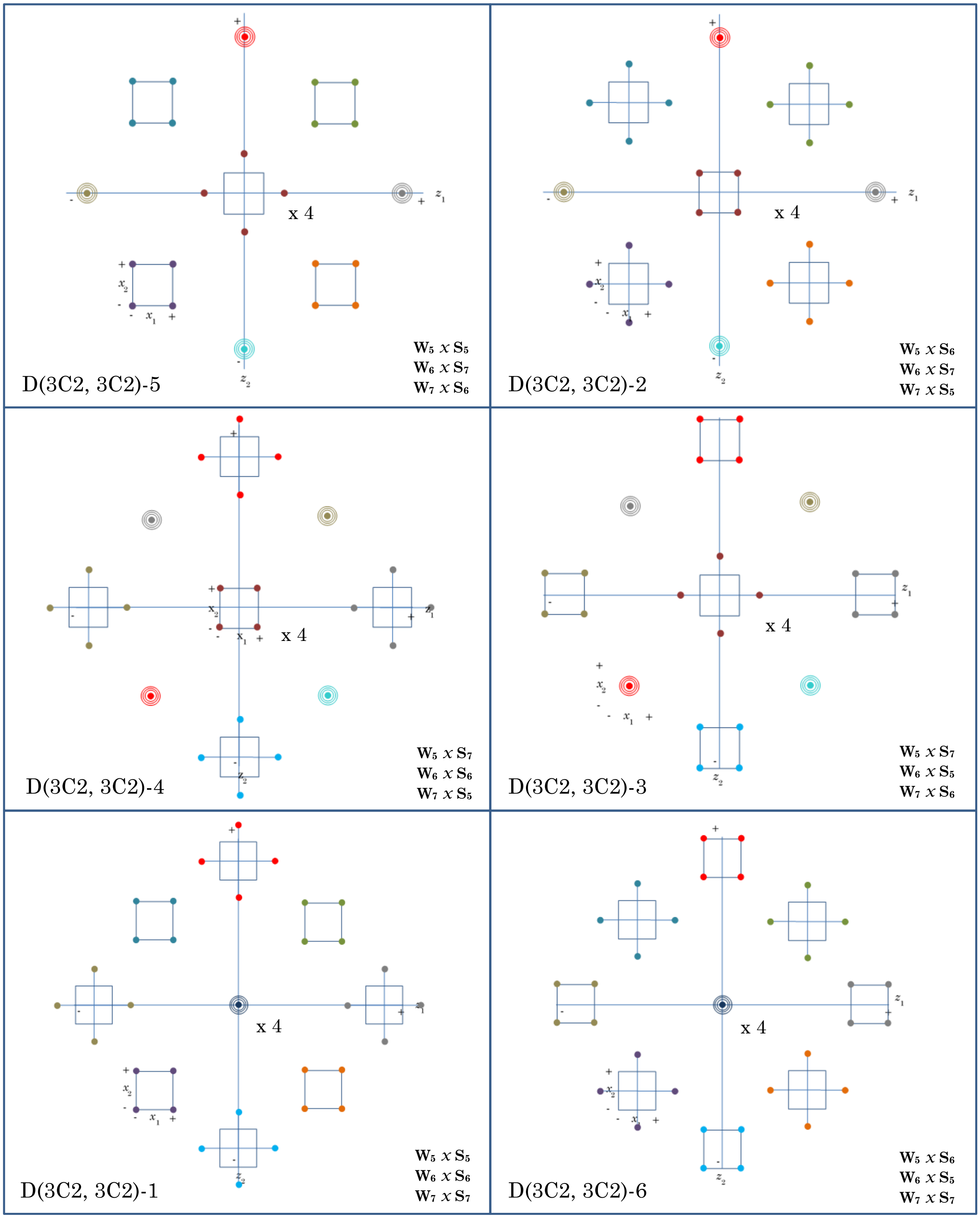


Figure 3-15. Sub-array Cartesian product layouts from balanced sub-arrays ($\Gamma = \Theta = 3$)

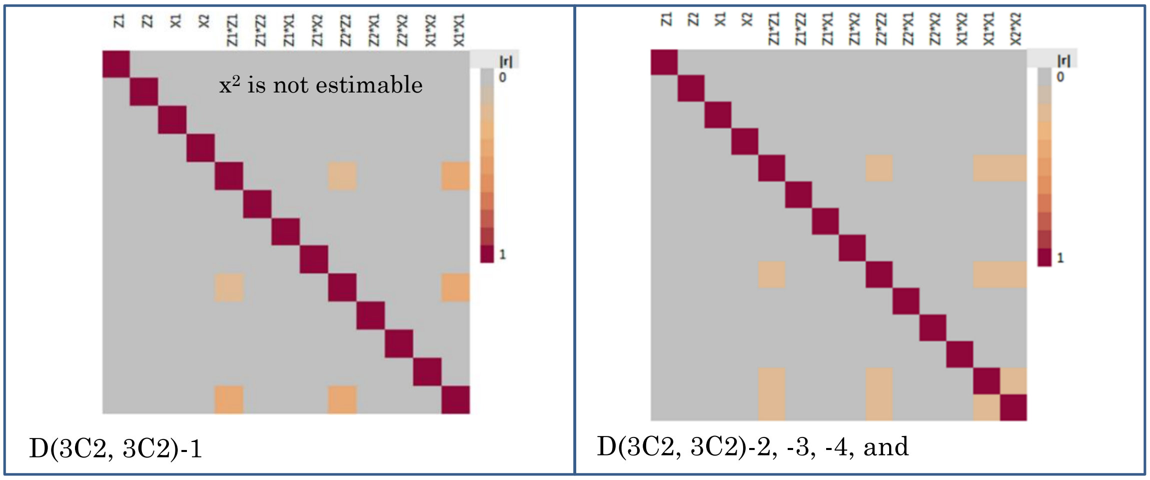


Figure 3-16. Pairwise correlation between model terms for the designs in Figure 3-15

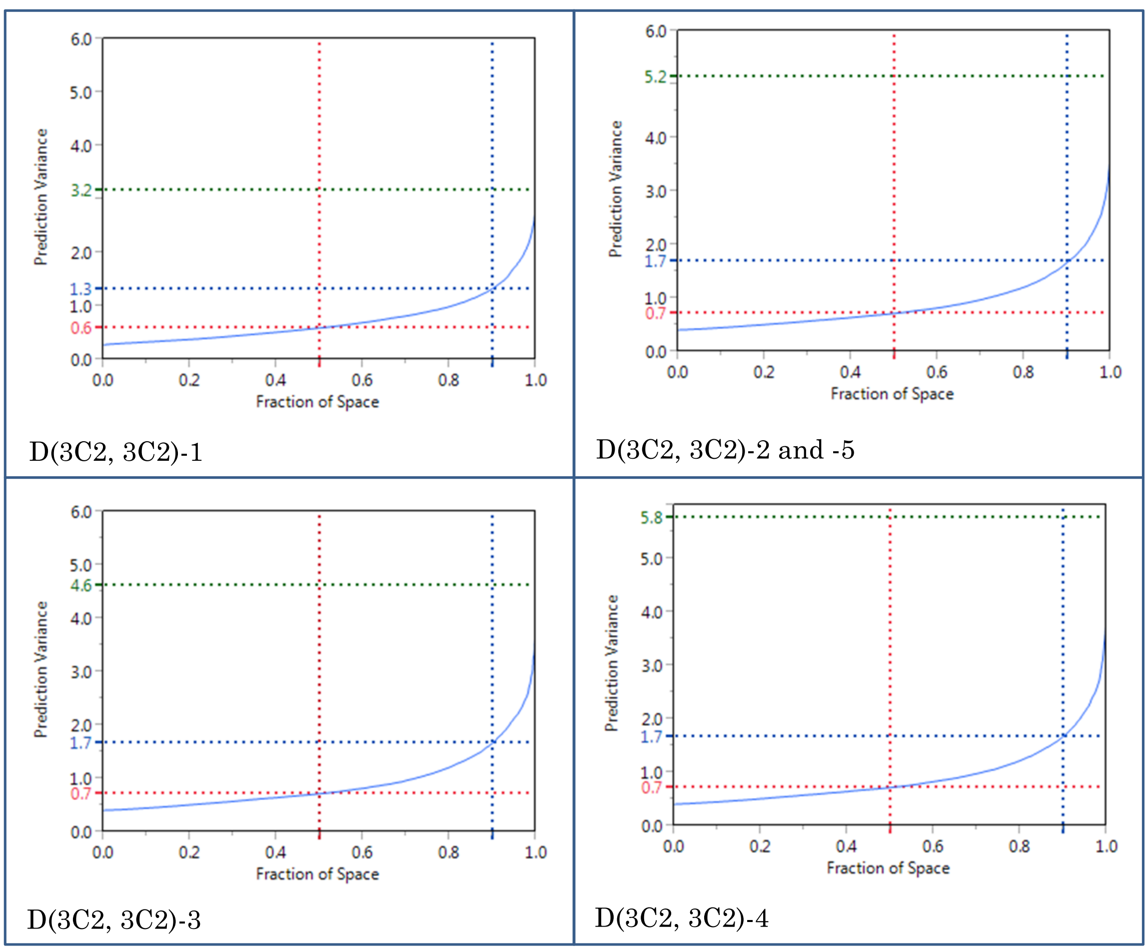


Figure 3-17. Prediction variance vs. fraction of design space for the designs in Figure 3-15

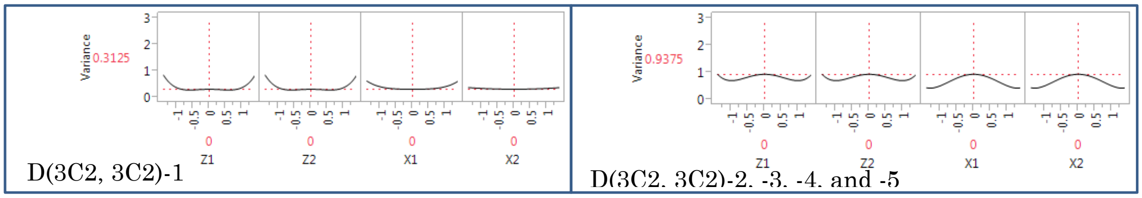


Figure 3-18. Unscaled prediction variance profile for the designs in Figure 3-15

Two balanced sub-arrays ($\Gamma = \Theta = 2$); $p = q \geq 3$

So far, only cases involving $p = q = 2$ have been examined. For cases where $p = q = 3$ or $p = q = 4$, the forms of the sub-arrays, and consequently, the forms of the second-order sub-array Cartesian product split-plot designs, are similar to those for $p = q = 2$ illustrated in Figure 3-2. When both the whole-plot array and the sub-plot array are partitioned into two sub-arrays each, four second-order sub-array Cartesian product split-plot designs are produced, of which, as in the case of $p = q = 2$, only two allow for the estimation of all model coefficients. As k increases, the size of the design increases geometrically. Figure 3-19 illustrates the sub-plot arrays for $p = q = 3$ and $\Gamma = \Theta = 2$, which results in a design with $w = 18$, $s = 9$, and N = 162.

Now, consider exploring the construction of split-plot designs using both orthogonal and non-orthogonal sub-arrays derived from Box-Behnken designs. The corresponding layouts for the whole-plot and sub-plot arrays are illustrated in Figures 3-20 and 3-22.

Figure 3-20 shows the partitioning of both the whole-plot and sub-plot arrays into two sub-arrays each. Note that breaking-up the whole-plot and sub-plot arrays into sub-arrays to form the design matrix sometimes results in designs that do not resemble the features of the design from which the arrays originated. This is also true when other techniques are used to construct split-plot designs, as will be illustrated in Section 3.2. In the case of orthogonal sub-arrays derived from Box-Behnken designs, sub-arrays $\mathbf{W}_1$ and $\mathbf{S}_1$ represent the factorial points of the 2^k design plus one center point although the factorial points of the 2^k design are not present in a Box-Behnken design. Sub-arrays $\mathbf{W}_{11}$ and $\mathbf{S}_{11}$ correspond to the edge points of the Box-Behnken design, which are $\mathbf{W}_2$ and $\mathbf{S}2$ with $\alpha = \beta = 1$.

Whole-plot sub-arrays

W_8			W_9		
z_1	z_2	z_3	z_1	z_2	z_3
−1	−1	−1	−β	0	0
1	−1	−1	β	0	0
−1	1	−1	0	−β	0
1	1	−1	0	β	0
−1	−1	1	0	0	−β
1	−1	1	0	0	β
−1	1	1	0	0	0
1	1	1	0	0	0
0	0	0	0	0	0

Sub-plot sub-arrays

S_8			S_9		
x_1	x_2	x_3	x_1	x_2	x_3
−1	−1	−1	−α	0	0
1	−1	−1	α	0	0
−1	1	−1	0	−α	0
1	1	−1	0	α	0
−1	−1	1	0	0	−α
1	−1	1	0	0	α
−1	1	1	0	0	0
1	1	1	0	0	0
0	0	0	0	0	0

Figure 3-19. Central composite whole-plot and sub-plot arrays divided in two sub-arrays ($p = q = 3$, $\Gamma = \Theta = 2$)

Whole-plot sub-arrays

W_1		W_{11}	
z_1	z_2	z_1	z_2
−1	−1	−1	0
1	−1	1	0
−1	1	0	−1
1	1	0	1
0	0	0	0

Sub-plot sub-arrays

S_1		S_{11}	
x_1	x_2	x_1	x_2
−1	−1	−1	0
1	−1	1	0
−1	1	0	−1
1	1	0	1
0	0	0	0

Figure 3-20. Box-Behnken whole-plot and sub-plot arrays ($\Gamma = \Theta = 2$)

Crossing the sub-arrays in Figure 3-20 results in four second-order sub-array Cartesian product split-plot designs. The parameters for the designs are $p = 2$, $q = 2$,

$k = p + q = 4$, $w = 10$, $s = 5$, and $N = w \times s = 50$. Two of the designs are like D(2C2, 2C2)-1 and D(2C2, 2C2)-2 but with $\alpha = \beta = 1$, which are illustrated in Figure 3-21. The properties of D(2B2, 2B2)-1 can be appreciated in Figures 3-9 through 3-11 ($\alpha = \beta = 1$). The other two designs are not adequate to estimate all of the model terms. One of those designs can not estimate the interaction between the sub-plot factors while the other can only estimate the coefficient for the combined quadratic sub-plot factors.

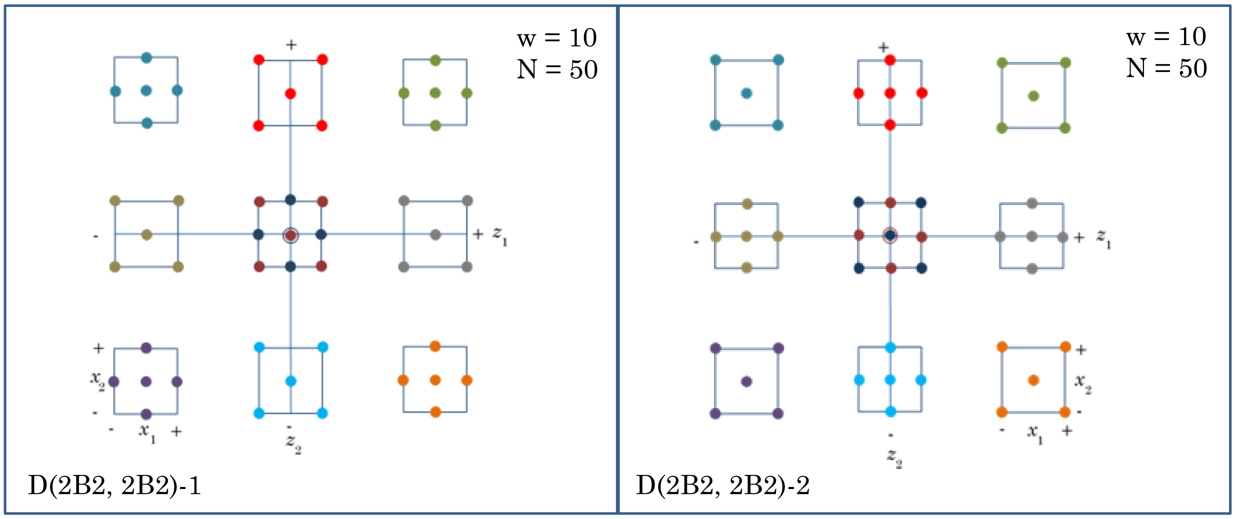


Figure 3-21. Box-Behnken sub-array Cartesian product split-plot designs (Γ = Θ = 2)

Three unbalanced sub-arrays (Γ = Θ = 3); $p = q = 2$

Figure 3-22 illustrates the case where (Γ = Θ = 3) and the replicated points have been removed, which creates non-orthogonal, unbalanced sub-arrays. The standard format for the second-order sub-array Cartesian product split-plot design is illustrated in Table 3-6. The design clearly has some disadvantages. First, there is more correlation between the model terms. Second, the prediction variance is more unstable. Third, because of the geometrical configuration, establishing the relationships for the terms of significance is challenging. Hence, there are no practical benefits in using a design like this one.

W_1	
z_1	z_2
−1	−1
1	−1
−1	1
1	1
0	0

W_2	
z_1	z_2
−1	0
1	0
0	0

W_3	
z_1	z_2
0	−1
0	1
0	0

Non-orthogonal, unbalanced whole-plot sub-arrays

S_1	
x_1	x_2
−1	−1
1	−1
−1	1
1	1
0	0

S_2	
x_1	x_2
−1	0
1	0
0	0

S_3	
x_1	x_2
0	−1
0	−1
0	0

Non-orthogonal, unbalanced sub-plot sub-arrays

Figure 3-22. Non-orthogonal, unbalanced sub-arrays derived from Box-Behnken designs

Table 3-6. Box-Behnken sub-array Cartesian product split-plot design D(2B2, 2B2)-1

w_i	z_1	z_2	x_1	x_2	w_i	z_1	z_2	x_1	x_2	w_i	z_1	z_2	x_1	x_2
	−1	−1	−1	−1		1	1	−1	−1		1	0	−1	−1
	−1	−1	1	−1		1	1	1	−1	7	1	0	1	1
1	−1	−1	−1	1	4	1	1	−1	1		1	0	0	0
	−1	−1	1	1		1	1	1	1		0	−1	0	−1
	−1	−1	0	0		1	1	0	0	8	0	−1	0	1
	1	−1	−1	−1		0	0	−1	−1		0	−1	0	0
	1	−1	1	−1		0	0	1	−1		0	1	0	−1
2	1	−1	−1	1	5	0	0	−1	1	9	0	1	0	1
	1	−1	1	1		0	0	1	1		0	1	0	0
	1	−1	0	0		0	0	0	0					
	−1	1	−1	−1		−1	0	−1	0					
	−1	1	1	−1	6	−1	0	1	0					
3	−1	1	−1	1		−1	0	0	0					
	−1	1	1	1										
	−1	1	0	0										

3.2 Design Performance Assessment

In this section, the performance of the second-order sub-array Cartesian product split-plot design is assessed relative to other historical designs that have provided a significant service to the response surface methodology community. While all of those designs have different structures and were developed with different objectives in mind, they are suitable alternatives for developing practical second-order block split-plot designs. Of course, selecting adequate treatment and observation designs and the proper error-control design needs to be based on the experimental situation. Those designs include the OLS-GLS equivalent estimation designs by Vining, Kowalski, and Montgomery (2005), the minimum whole-plot designs by Parker, Vining, and Kowalski (2007a), and the balanced and unbalanced second-order orthogonally blocked split-plot designs by Verma *et. al.* (2012) derived from Dey (2009) and Zhang (2011).

The assessment is straightforward. In Figures 3-23 through 3-26, the second-order sub-array Cartesian product split-plot designs D(2C2, 2C2)-1 and D(3C2, 3C2)-5 are assessed relative to the central composite designs provided by Vining, Kowalski, and Montgomery (2005) and by Parker, Kowalski, and Vining (2007a). In Figures 3-27 through 3-30, the second-order sub-array Cartesian product split-plot design D(2B2, 2B2)-2 is assessed relative to the Box-Behnken designs provided by Vining, Kowalski, and Montgomery (2005) and by Parker, Kowalski, and Vining (2007a), the D-optimal design provided by Jones and Nachtsheim (2009), and the three-level designs provided by Verma *et. al.* (2012). Clearly, the design performs well relative to those designs when using the pairwise correlation coefficient between model terms and the prediction variance criterion. Summaries for the comparisons between the designs are provided in Tables 3-7 through 3-9. Note that D(2B2, 2B2)-2 is D(2C2, 2C2)-2 but with $\alpha = \beta = 1$.

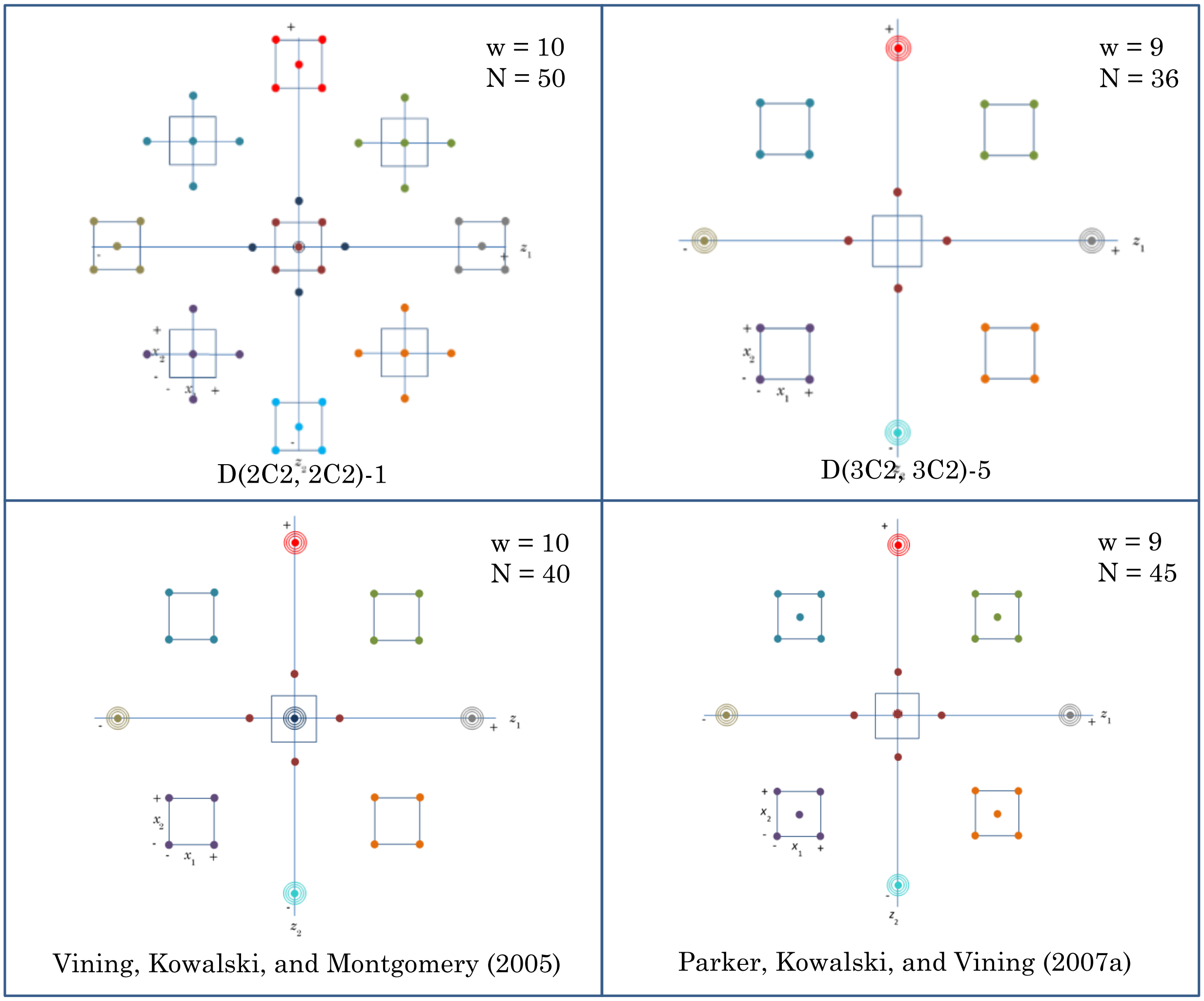


Figure 3-23. Second-order split-plot designs for spherical regions

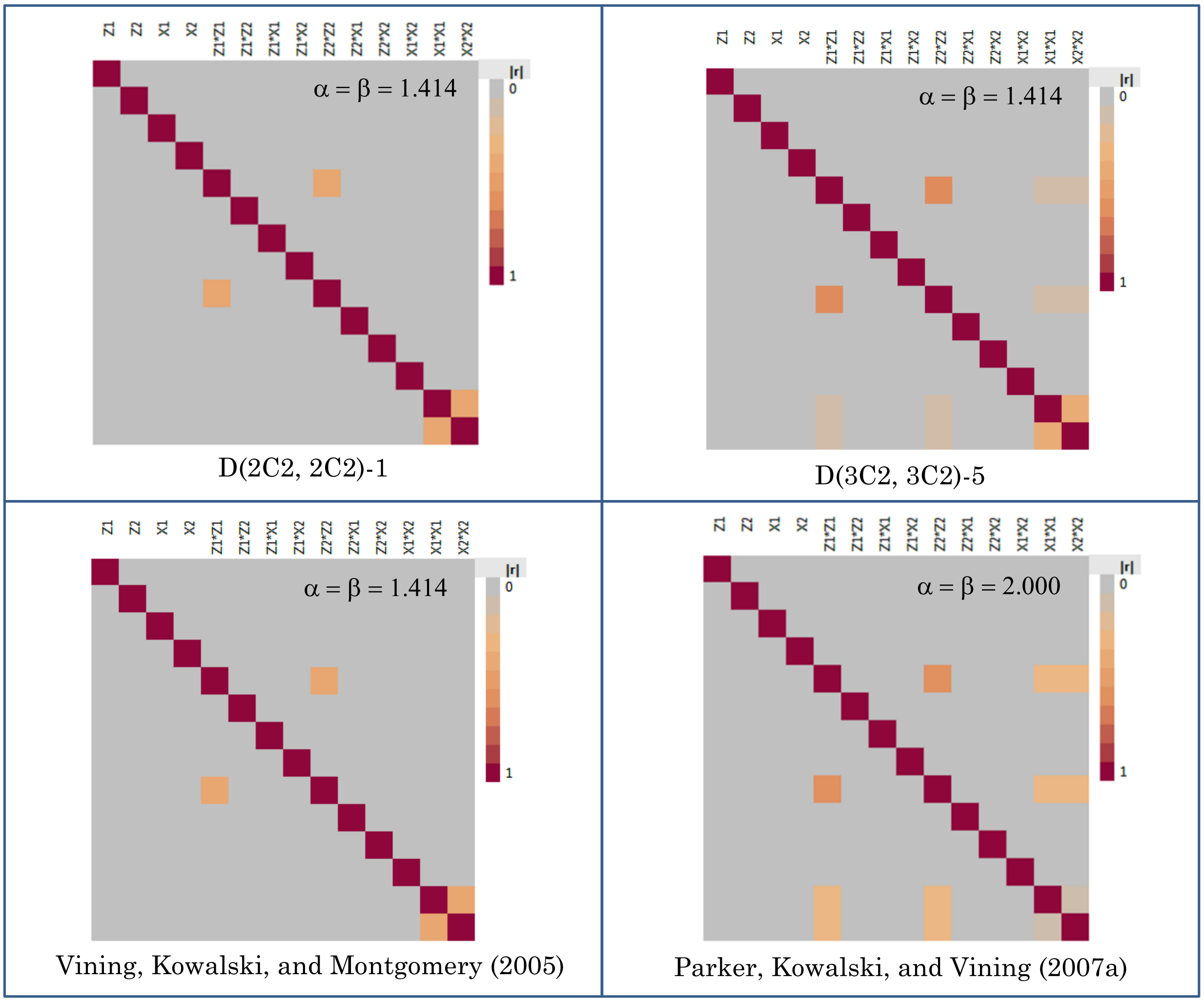


Figure 3-24. Pairwise correlation between model terms for the designs in Figure 3-23

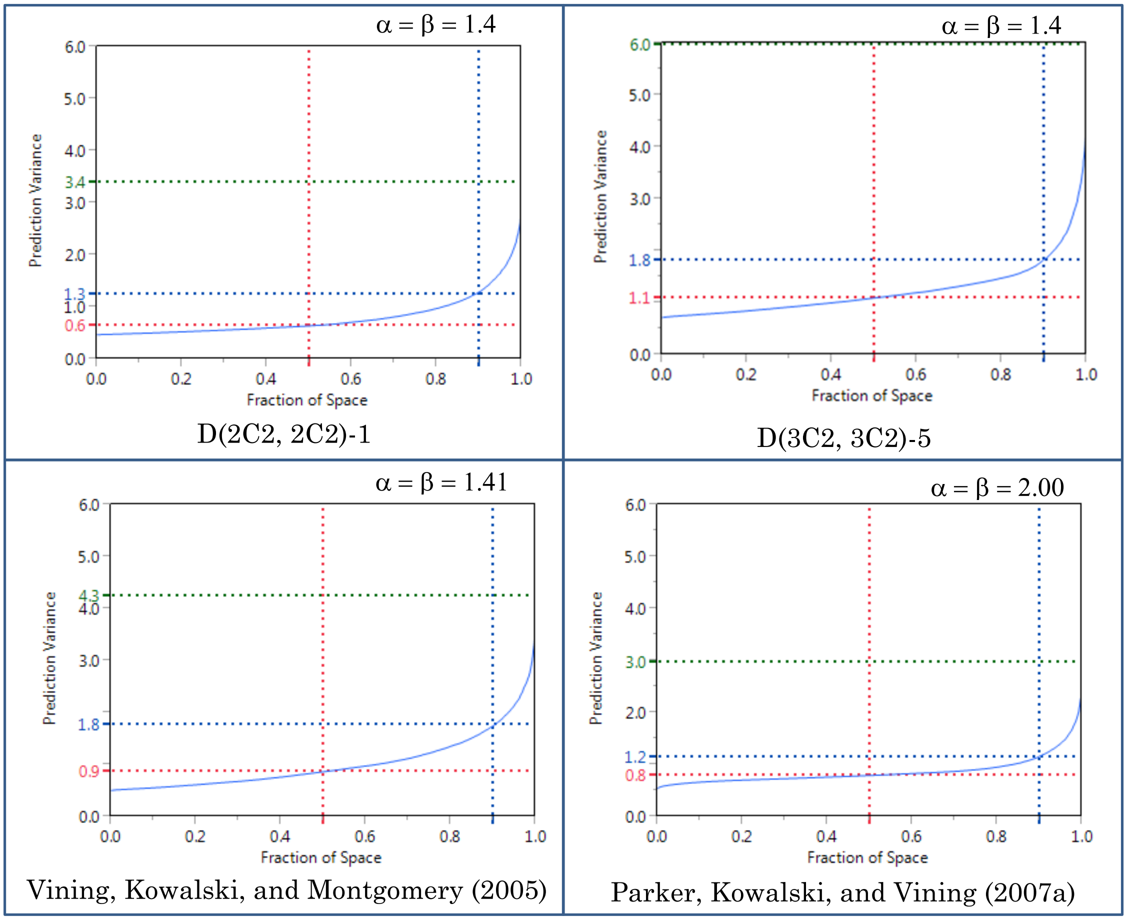


Figure 3-25. Prediction variance vs. fraction of design space for the designs in Figure 3-23

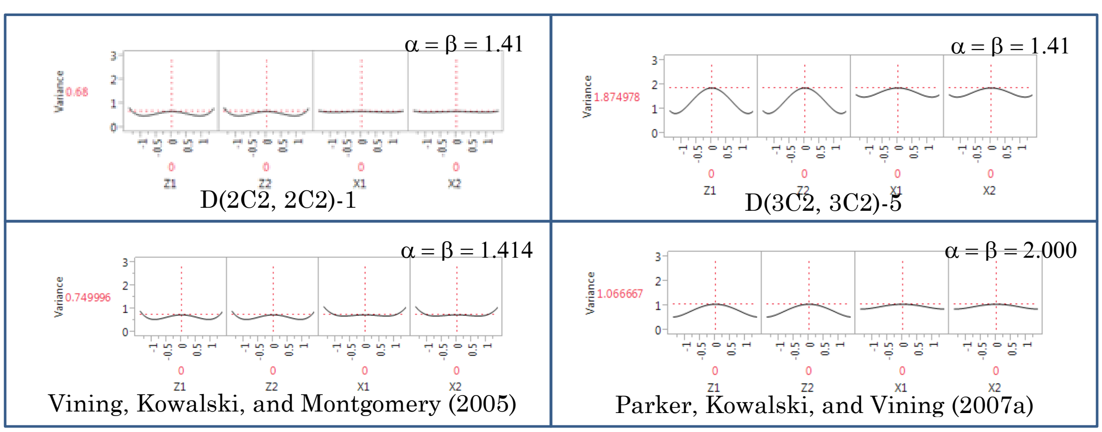


Figure 3-26. Unscaled prediction variance profile for the designs illustrated in Figure 3-23

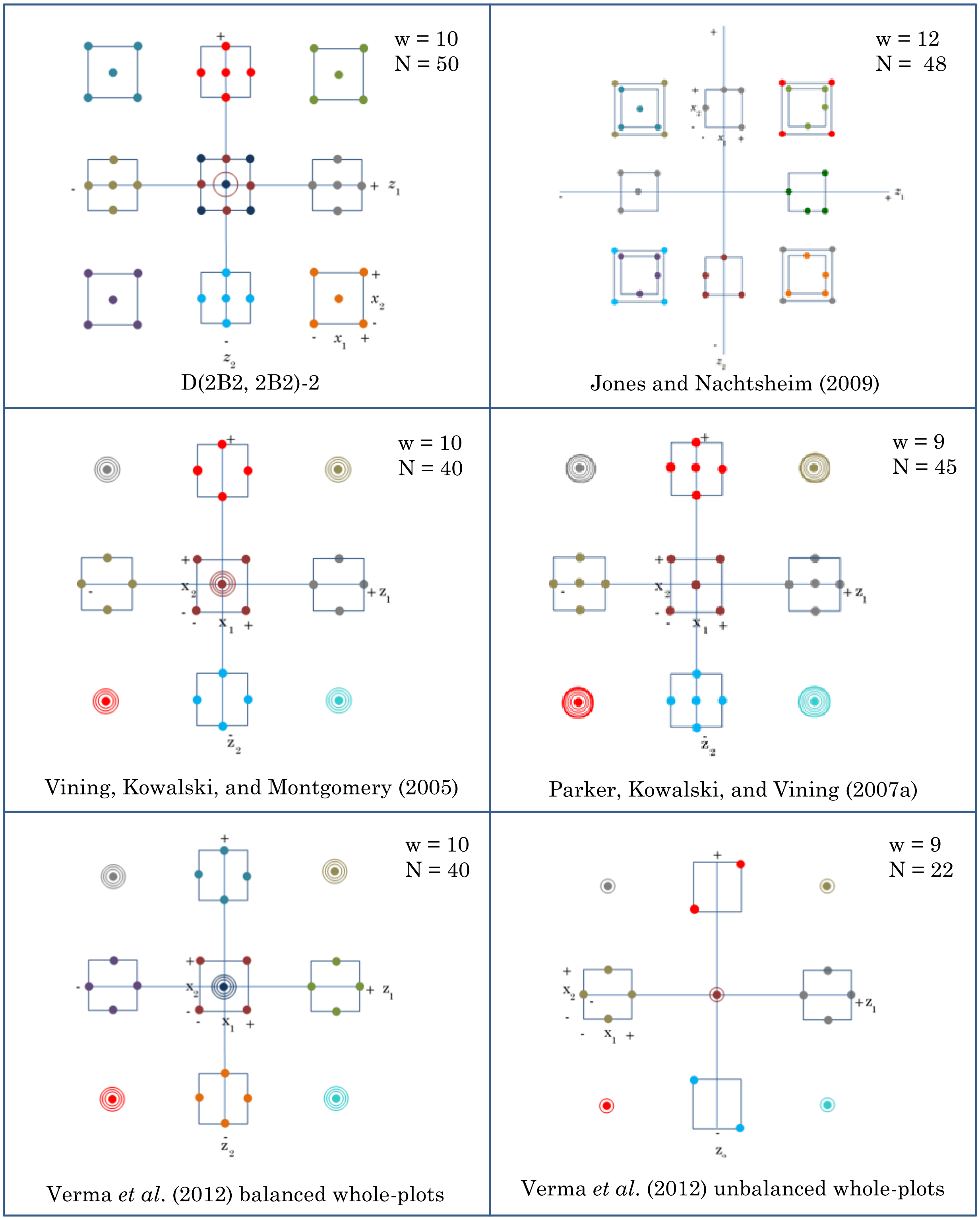


Figure 3-27. Second-order split-plot designs for cuboidal regions

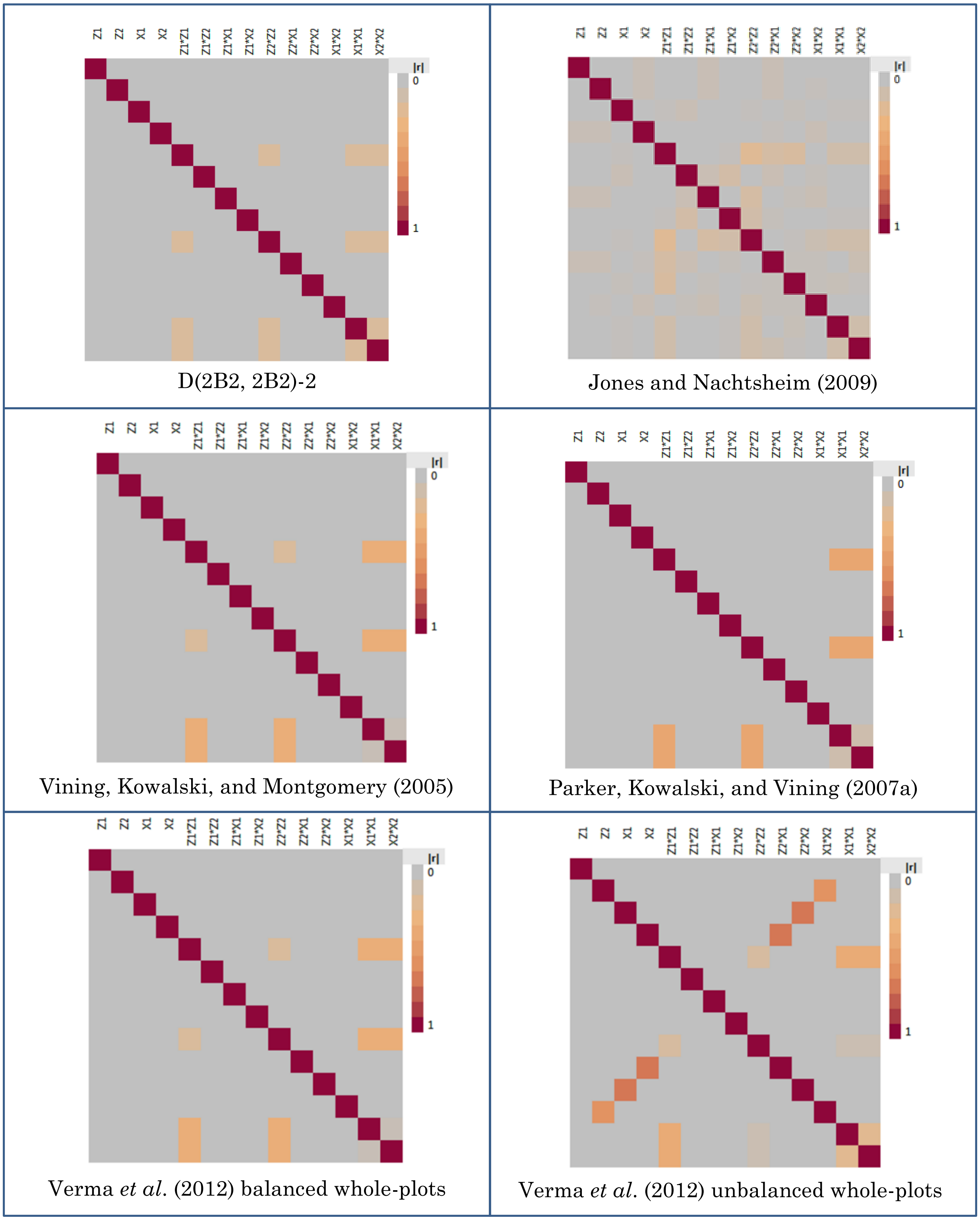


Figure 3-28. Pairwise correlation between model terms for the designs in Figure 3-27

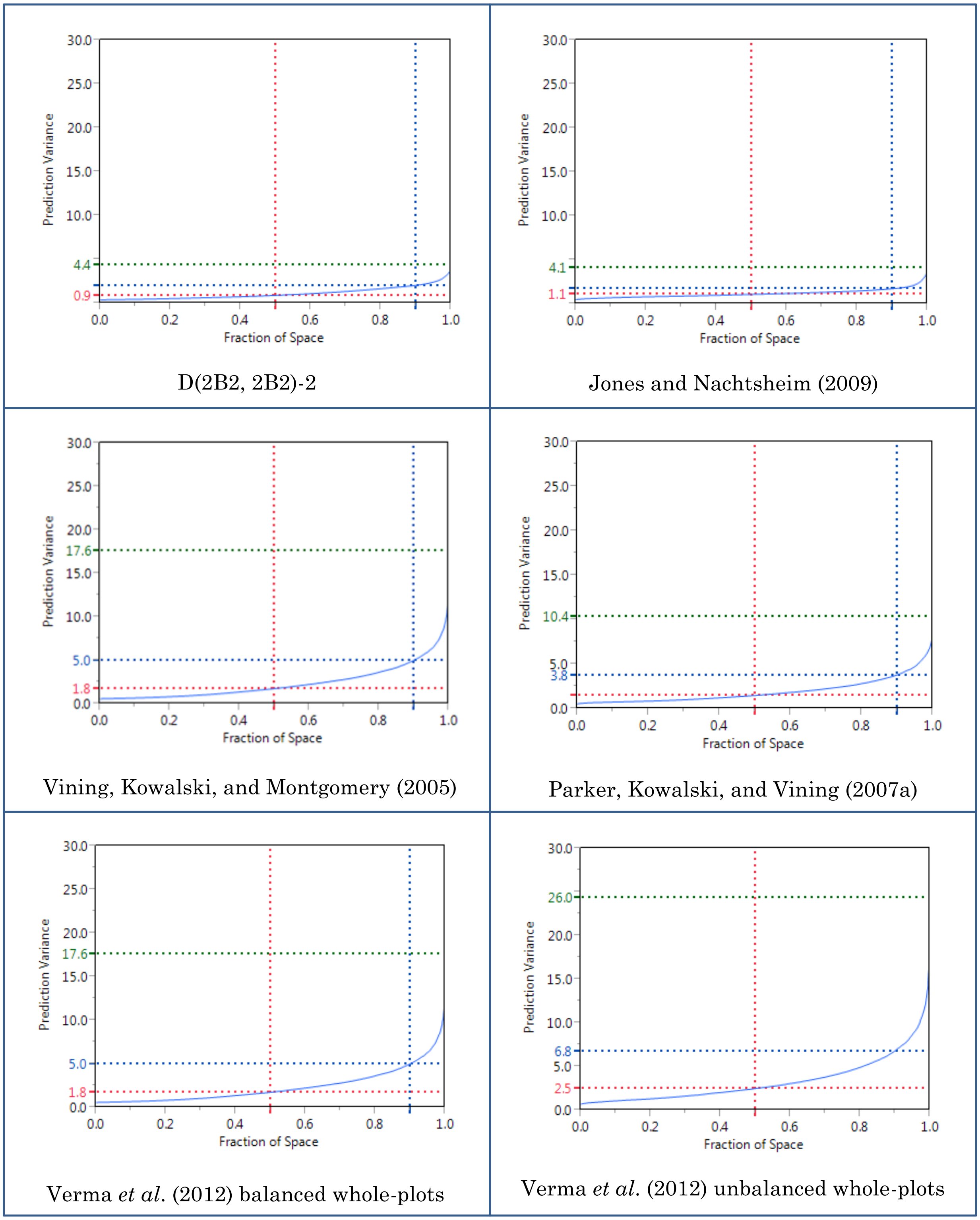


Figure 3-29. Prediction variance vs. fraction of design space for the designs in Figure 3-27

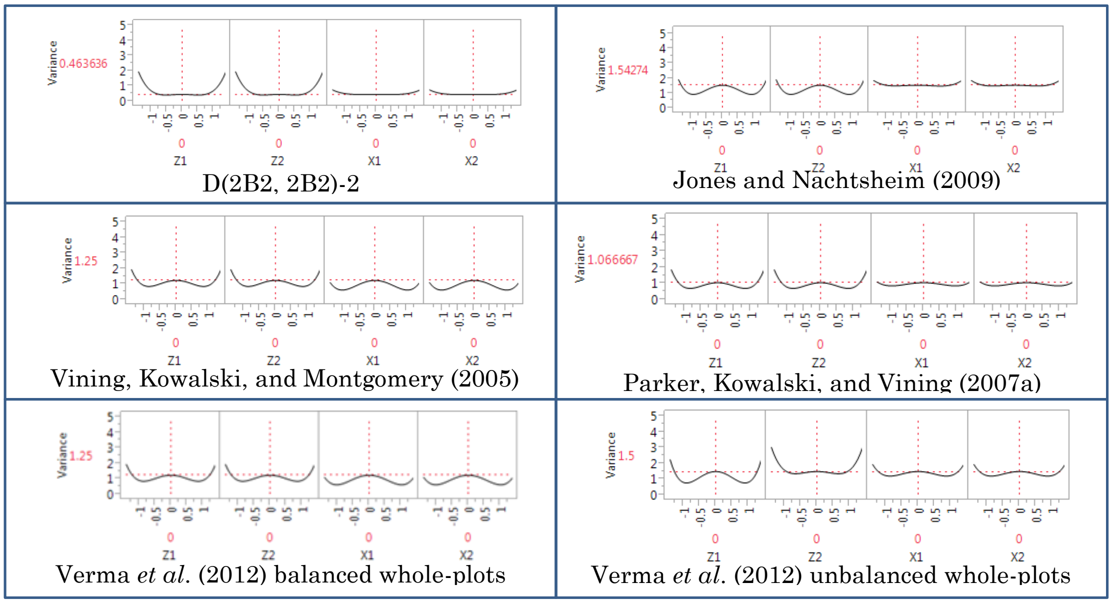


Figure 3-30. Unscaled prediction variance profile for the designs in Figure 3-27

3.3 Summary

To design experiments effectively, practitioners need to have a clear understanding of the capabilities and limitations of the candidate designs they have available. Likewise, they need to have a clear understanding of the information that the design will produce. Second-order sub-array Cartesian product split-plot designs are an alternative that meets those needs.

The independent nature of the whole-plot and sub-plot sub-arrays facilitates the handling of split-plot designs as a real superposition of two different experiments. The designs allow for estimating the model coefficients for all first-order, two-factor interactions, and pure second-order terms. When the whole-plot and sub-plot sub-arrays are second-order orthogonal blocks, the sub-array product is a second-order orthogonal block design, which is an appealing feature. The designs are economical, and generally require about one-half of the number of runs required by full Cartesian product designs. The designs are particularly useful if the shapes of the experimental regions for the whole-plot and sub-plot levels are different.

Generally, second-order sub-array Cartesian product split-plot designs permit estimating the model coefficients for all first-order, two-factor interactions, and pure second-order effects as well as the whole-plot variance and sub-plot variance. Whole-plot sub-arrays and sub-plot sub-arrays are first-order orthogonal, which means that their combinations will be first-order orthogonal. This implies that for a first-order and first-order with interaction model the first two conditions of orthogonal blocking are satisfied as long as the average of every column within a block is zero.

There are some disadvantages to the use of the sub-array Cartesian product split-plot designs. First, the Cartesian product is not a vector operation, which makes it impractical for matrix operations. Second, the designs do not provide for the direct estimation of whole-plot or sub-plot variance. However, the whole-plot and sub-plot sub-arrays can be easily manipulated to produce those estimates by augmenting the sub-plot sub-arrays with $(x_1, x_2) = (0, 0)$ and by replicating at least one of the whole-plots. Clearly, a better estimate of the whole-plot variance is obtained if a whole-plot is replicated 3 to 5 times. In cases that require blocking, the whole-plot variance is confounded with the block variance.

The design performs well, as illustrated in Tables 3-7 through 3-9. The variance of the regression coefficients is low. Similarly, the prediction variance of the regression coefficients is low and stable. The aliasing between the terms in the model and likely effects that are not in the model as well as the correlation between similar effects that are not in the model is low.

The performance of the second-order sub-array Cartesian product split-plot design was assessed relative to the performance of the central composite designs and Box-Behnken designs provided by both Vining, Kowalski, and Montgomery (2005) and Parker, Kowalski, and Vining (2007a), and to the three-level designs provided by Verma *et. al*. (2012). Based on criterion involving prediction variance and pairwise correlation coefficients between model terms, it was demonstrated that second-order sub-array Cartesian product split-plot designs perform as well or better than designs that have been considered standards up to now.

Table 3-7. Performance assessment summary – whole-plot variance estimation

	Vining, Kowalski, and Montgomery (2005) OLS-GLS equiv.	Jones and Natchsheim (2009) D-optimal design	Sub-array Cartesian product split-plot D(2C2, 2C2)-2
Structure			
No. whole-plots	12	12	12
Total runs	48	48	60
Balanced whole-plots	Y	Y	Y
β/α	1.414/1.414	n/a	1.414/1.414
Prediction variance			
@ 50% FDS	0.82	1.09	0.56
@ 90% FDS	1.70	1.73	1.25
@ overall center	0.35	1.54	0.37
Average	0.95	1.18	0.70
Maximum	3.82	4.18	3.00
No. Correlation coefficient |r| (other than main diagonal)			
Clear	85	65	89
0.0 < |r| < 0.50	6	26	2
0.50 < |r| < 1.0	0	0	0

Table 3-8. Performance assessment summary – standard designs

	Vining, Kowalski, and Montgomery (2005) OLS-GLS equiv.	Sub-array Cartesian product split-plot D(2C2, 2C2)-1	Parker, Kowalski, and Vining (2007b) minimum whole-plot	Sub-array Cartesian product split-plot D(3C2, 3C2)-5
Structure				
No. whole-plots	10	10	9	9
Total runs	40	50	45	36
Balanced whole-plots	Y	Y	Y	Y
β/α	1.414/1.414	1.414/1.414	2.0/2.0	2.0/2.0
Prediction variance				
@ 50% FDS	0.90	0.60	0.80	1.10
@ 90% FDS	1.80	1.30	1.30	1.80
@ overall center	0.75	0.68	1.07	1.87
Average	1.03	0.80	0.86	1.25
Maximum	4.30	3.40	3.00	6.00
No. Correlation coefficient |r| (other than main diagonal)				
Clear	89	89	85	85
0.0 < |r| < 0.50	2	2	5	5
0.50 < |r| < 1.0	0	0	1	1

Table 3-9. Performance assessment summary – minimum whole-plot

	Parker, Kowalski, and Vining (2007a) minimum whole-plot	Verma *et. al.* (2012) unbalanced	Sub-array Cartesian product split-plot D(2B2, 2B2)-2
Structure			
No. whole-plots	9	9	9
Total runs	45	22	45
Balanced whole-plots	Y	N	Y
β/α	2.0/2.0	1.0/1.0	1.0/1.0
Prediction variance			
@ 50% FDS	1.5	2.5	1.0
@ 90% FDS	3.8	6.8	2.1
@ overall center	1.1	1.5	0.7
Average	2.0	3.4	1.2
Maximum	11.4	26.0	4.8
No. Correlation coefficient $\|r\|$ (other than main diagonal)			
Clear	86	82	86
$0.0 < \|r\| < 0.50$	5	6	5
$0.50 < \|r\| < 1.0$	0	3	0

4 Second-Order Sub-Array Cartesian Product Split-Plot Block Design

In the previous chapter, second-order sub-array Cartesian product split-plot designs were constructed with sub-arrays derived from central composite designs, Box-Behnken designs, and definitive screening designs. Their treatment design and observation design was examined. Finally, the performance of the designs was assessed relative to some of the historical designs discussed in the literature review that have provided significant service to the response surface methodology community.

In this chapter, the Error-Control Design of second-order sub-array Cartesian product split-plot *block* design is examined and block designs are provided. A goal of this research is to construct second-order block Cartesian product split-plot designs that satisfy as many as the Box and Draper (1975) evaluation criteria as possible, especially designs that allow to estimate the whole-plot and sub-plot variances and that block orthogonally to minimize the influence of the blocks on the estimation of the model parameters. Then, the performance of the designs is assessed relative to the performance of the historical designs used in Chapter 3.

4.1 Design Construction and Evaluation

In this section, the Error-Control Design for balanced and unbalanced orthogonal block second-order sub-array Cartesian product split-plot designs is discussed and their characteristics are evaluated. Recall that the third step in the construction of the second-order sub-array Cartesian product split-plot designs discussed in Chapter 3 was the partition of the whole-plot and sub-plot arrays into sub-arrays or blocks. Those same blocks now play a key role in the overall blocking strategy of the design. The strategy leads to independent error control designs for the whole-plots and sub-plot treatments. Clearly, the number of sub-arrays that can be produced can be exorbitant, so the effort in this research is limited to a few cases. The construction of block split-plot designs using other types of sub-arrays is straightforward.

Table 4-1 provides the block design that results from replicating the sub-arrays illustrated in Figure 3-1 that produce the design in Figure 3-2 and Table 3-1. Before discussing the design in Table 4-1, it should be noted that the design in Table 3-1 is a block design itself with a completely randomized error control design for the whole-plot treatments and a randomized complete block error control design for the sub-plot treatments. In that situation, the whole-plots look like blocks for the sub-plot factors. Clearly, the second-order sub-array Cartesian product split-plot block design in Table 4-1 has a randomized complete block error control design for both the whole-plot treatments and the sub-plot treatments.

While the design has some good attributes, it is quite large and potentially costly when judged by the 16 potential whole-plot factor resets that could be required. The parameters of the design are $b = 2$, $w/b = 9$, $w/s = 9$, and N = 162. The design affords seven degrees-of-freedom for estimating a pooled whole-plot variance estimate. The unscaled prediction variance is stable, and is 0.67 at $(z_1, z_2, x_1, x_2\ )$ = (0, 0, 0, 0). The maximum unscaled prediction variance is 0.67 and the average unscaled prediction variance is 0.43. There is no correlation between block effects and any model terms. This randomized complete block error-control design is appropriate when there are enough homogeneous experimental units in a block such that each treatment can be applied at least once in each block. The shortcoming of this design is that it does not permit the estimation of the whole-plot variance during the first phase of experimentation without sacrificing properties such as second-order orthogonal blocking had a sequential strategy been required, i.e. run Block 1 first and then Block 2. To obtain a whole-plot variance estimation for Block 1, which is needed to determine the significance of the whole-plot factors and their interactions, one whole-plot needed to be added, which would have sacrificed second-order orthogonal blocking. If estimating the whole-plot variance during for Block 1 was a goal, one center point at (z_1, z_2) = (0, 0) can be added to generate an extra degree of freedom that affords the opportunity to estimate the whole-plot variance.

Table 4-1. Balanced complete block sub-array Cartesian product split-plot design ($p = q = 2$), ($\Gamma = \Theta = 1$)

Block 1				**Block 2**			
w_i	z_1	z_2	S	w_i	z_1	z_2	S
1	−1	−1	S	10	−1	−1	S
2	1	−1	S	11	1	−1	S
3	−1	1	S	12	−1	1	S
4	1	1	S	12	1	1	S
5	$-\beta$	0	S	14	$-\beta$	0	S
6	β	0	S	15	β	0	S
7	0	$-\beta$	S	16	0	$-\beta$	S
8	0	β	S	17	0	β	S
9	0	0	S	18	0	0	S

Now, consider design D(2C2, 2C2)-1 illustrated in Table 3-3, although D(2C2, 2C2)-2 could have been selected as well. Two balanced, second-order orthogonal blocks of size $w = 5$ and $s = 10$ are constructed. Second-order orthogonal block designs meet the Box and Hunter (1957) criteria, which Wang, Kowalski, and Vining (2009) expanded to include split-plot designs. One block corresponds to the Cartesian product $\mathbf{W}_1 \times \mathbf{S}_2$ and the other block to the Cartesian product $\mathbf{W}_2 \times \mathbf{S}_1$. The design is illustrated in Table 4-2 and in Figure 4-1.

Now, consider the design in Table 4-3 (refer to Table 3-13) for $p = q = 2$, $\Gamma = \Theta = 3$, and $b = 3$. The blocks correspond to the Cartesian products $\mathbf{W}_1 \times \mathbf{S}_2$, $\mathbf{W}_2 \times \mathbf{S}_3$, and $\mathbf{W}_3 \times \mathbf{S}_1$. Based on the pairwise correlation between model terms and the prediction variances depicted in Figure 4-1, the design in Table 4-2 outperforms the designs in Tables 4-1 and 4-3.

Figure 4-1 provides a comparison between second-order sub-array Cartesian product split-plot block designs for $\Gamma = \Theta = 1$, $\Gamma = \Theta = 2$, and $\Gamma = \Theta = 3$. Note the increase in the prediction variance at (0, 0, 0, 0) as well as the increase on the unscaled prediction variance as a function of fraction of design space. Similarly, the overall number of cells that have pairwise correlation coefficients between model terms greater than zero increases.

Table 4-2. Balanced second-order block sub-array Cartesian product split-plot design ($p = q = 2;\ \Gamma = \Theta = 2; \mathrm{b} = 2$)

Block 1				Block 2			
w_{i}	z_1	z_2	$\mathrm{S_i}$	w_{i}	z_1	z_2	$\mathrm{S_i}$
1	-1	-1	$\mathrm{S_2}$	6	$-\beta$	0	$\mathrm{S_1}$
2	1	-1	$\mathrm{S_2}$	7	β	0	$\mathrm{S_1}$
3	-1	1	$\mathrm{S_2}$	8	0	$-\beta$	$\mathrm{S_1}$
4	1	1	$\mathrm{S_2}$	9	0	β	$\mathrm{S_1}$
5	0	0	$\mathrm{S_2}$	10	0	0	$\mathrm{S_1}$

Table 4-3. Balanced second-order block sub-array Cartesian product split-plot design ($p = q = 2;\ \Gamma = \Theta = 3; \mathrm{b} = 3$)

Block 1				Block 2				Block 3			
w_{i}	z_1	z_2	$\mathrm{S_i}$	w_{i}	z_1	z_2	$\mathrm{S_i}$	w_{i}	z_1	z_2	$\mathrm{S_i}$
1	-1	-1	$\mathrm{S_2}$	5	$-\beta$	0	$\mathrm{S_3}$	9	0	0	$\mathrm{S_1}$
2	1	-1	$\mathrm{S_2}$	6	β	0	$\mathrm{S_3}$	10	0	0	$\mathrm{S_1}$
3	-1	1	$\mathrm{S_2}$	7	0	$-\beta$	$\mathrm{S_3}$	11	0	0	$\mathrm{S_1}$
4	1	1	$\mathrm{S_2}$	8	0	β	$\mathrm{S_3}$	12	0	0	$\mathrm{S_1}$

4.2 Design Performance Assessment

In the previous section, examples of second-order sub-array Cartesian product split-plot block designs for $p = q = 2$, $b = 2$ and $b = 3$ were provided. In this section, the performance of those designs is assessed relative to historical second-order block split-plot designs. Those designs include the OLS-GLS equivalent estimation designs by Vining, Kowalski, and Montgomery (2005) and the minimum whole-plot designs by Parker, Vining, and Kowalski (2007a) blocked by Wang, Kowalski, and Vining (2009), and the balanced and unbalanced second-order block split-plot designs provided by Dey (2009) and Zhang (2011) and blocked by Verma *et. al*. (2012). Figures 4-2 through 4-4 show that second-order sub-array Cartesian product split-plot designs perform as well or better than those designs.

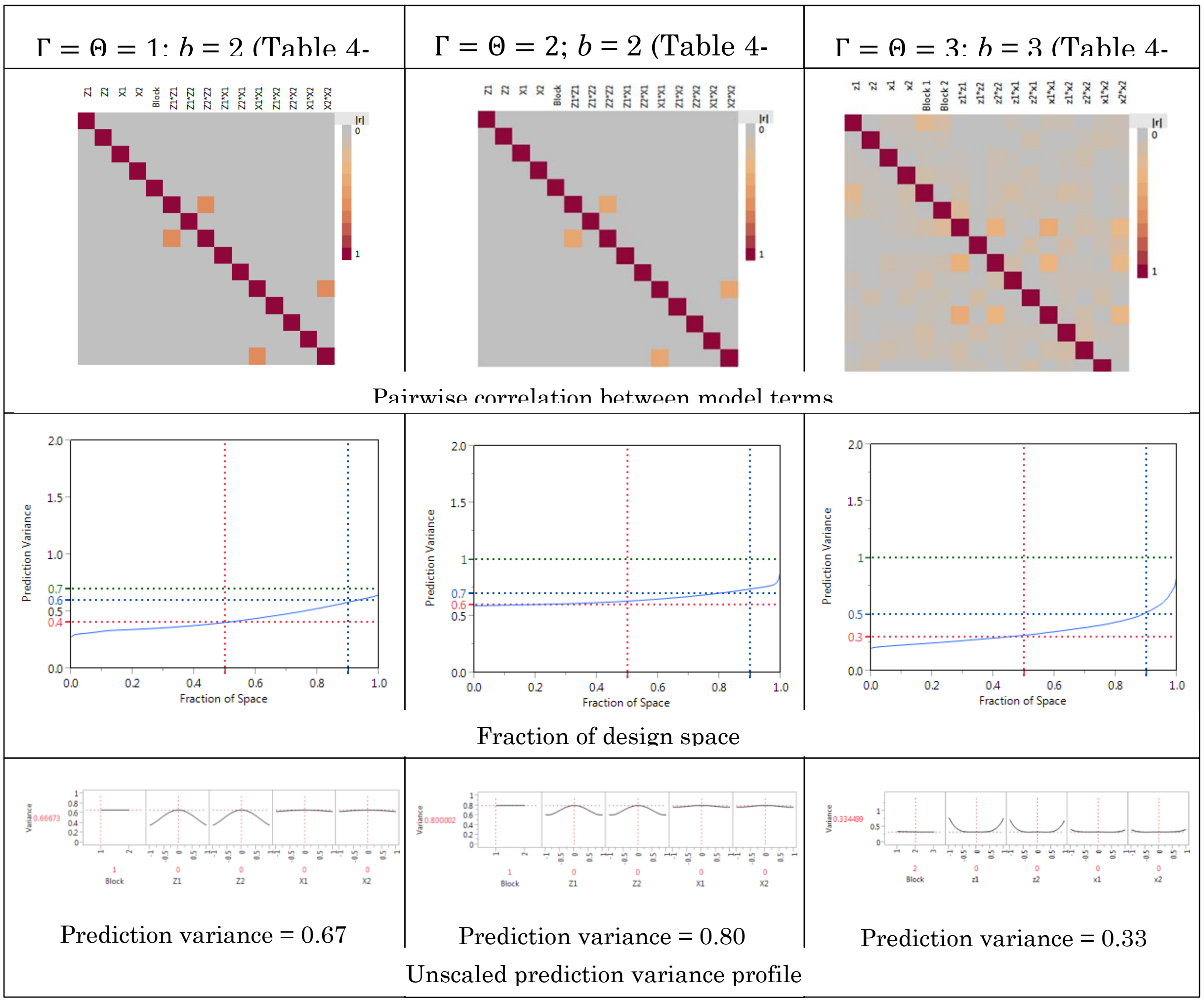


Figure 4-1. Attributes of second-order sub-array Cartesian product split-plot block designs

4.3 Summary

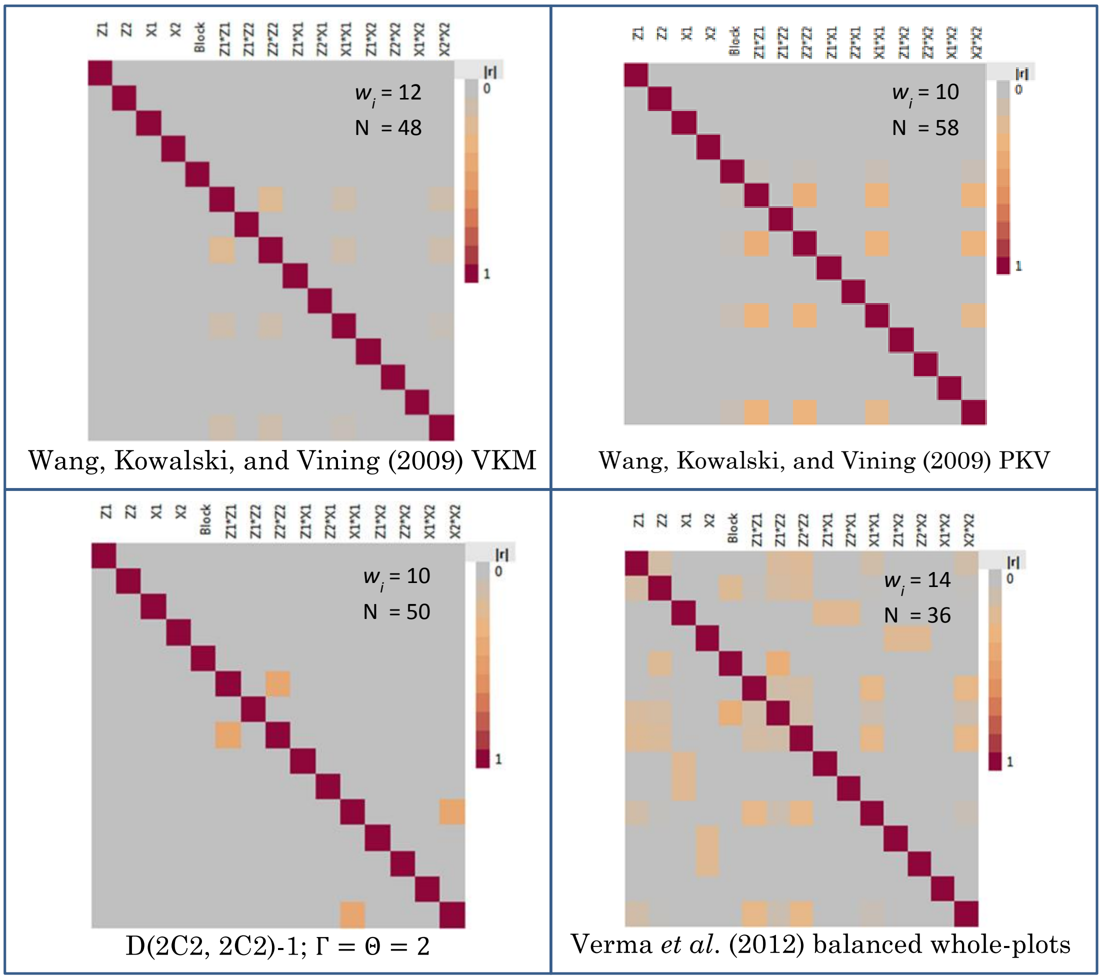
Like the unblocked second-order sub-array Cartesian product split-plot designs, the second-order sub-array Cartesian product split-plot block designs perform well when judged by unscaled prediction variance and pairwise correlation coefficients between model terms criterion as shown in Figure 4-1. The prediction variance of the regression coefficients is low and stable and the aliasing between the terms in the model and likely effects that are not in the model as well as the correlation between similar effects that are not in the model is low.

Table 4-4. Performance assessment summary – blocked designs

	Wang, Kowalski, and Vining (2009) – Vining, Kowalski, and Montgomery (2005)	Sub-array Cartesian product split-plot D(2C2, 2C2)-1	Wang, Kowalski, and Vining (2009) – Parker, Kowalski, and Vining (2007a)	Verma *et. al.* (2012)
Structure				
No. whole-plots	12	10	10	14
Total runs	48	50	58	36
Balanced whole-plots	Y	Y	N	N
β/α	1.414/2.828	1.414/1.414	1.871/2.160	1.0/1.0
Prediction variance				
@ 50% FDS	0.5	0.6	0.7	0.6
@ 90% FDS	0.6	0.7	0.8	1.0
@ overall center	0.51	0.80	0.89	0.74
Average	0.52	0.65	0.71	0.68
Maximum	1.1	1.0	1.0	2.5
No. Correlation coefficient $\|r\|$ (other than main diagonal)				
Clear	85	89	81	72
$0.0 < \|r\| < 0.50$	6	2	10	19
$0.50 < \|r\| < 1.0$	0	0	0	0

The second-order sub-array Cartesian product block split-plot design was assessed relative to the designs provided by Vining, Kowalski, and Montgomery (2005) and by Parker, Kowalski, and Vining (2007a) as blocked by Wang, Kowalski, and Vining (2009) as well as the designs provided by Dey (2009) and by Zhang (2011) as blocked by Verma *et. al.* (2012). Table 4-4 shows that the second-order sub-array Cartesian product block split-plot design outperforms the design provided by Dey (2009) as blocked by Verma *et. al.* (2012) and the design provided by Parker, Kowalski, and Vining (2007a) as blocked by Wang, Kowalski, and Vining (2009) when judged by the prediction variance and the pairwise correlation coefficients between model terms criterion. The design also outperforms the design provided by Zhang (2011) as blocked by Verma *et.al.* (2012). The second-order sub-array Cartesian product block split-plot design also outperform the design provided by Vining, Kowalski, and Montgomery (2005) as blocked by Wang, Kowalski, and Vining (2009) when judged by the pairwise correlation between model terms criterion.

It is proposed to add two criterion to the split-plot evaluation criteria by Myers, Montgomery, and Anderson-Cook (2009): the sequential assembly of higher order split-plot designs and the ability to carry out the split-plot experiment in blocks. Fisher (1926) established blocking as a fundamental technique to control the effects of unwanted sources of variability. Unwanted sources of variability introduced by the experimental situation affect split-plot experiments as they would affect any experiment and need to be controlled.



Wang, Kowalski, and Vining (2009) VKM

Wang, Kowalski, and Vining (2009) PKV

D(2C2, 2C2)-1; Γ = Θ = 2

Verma *et al.* (2012) balanced whole-plots

Figure 4-2. Pairwise correlation between model terms

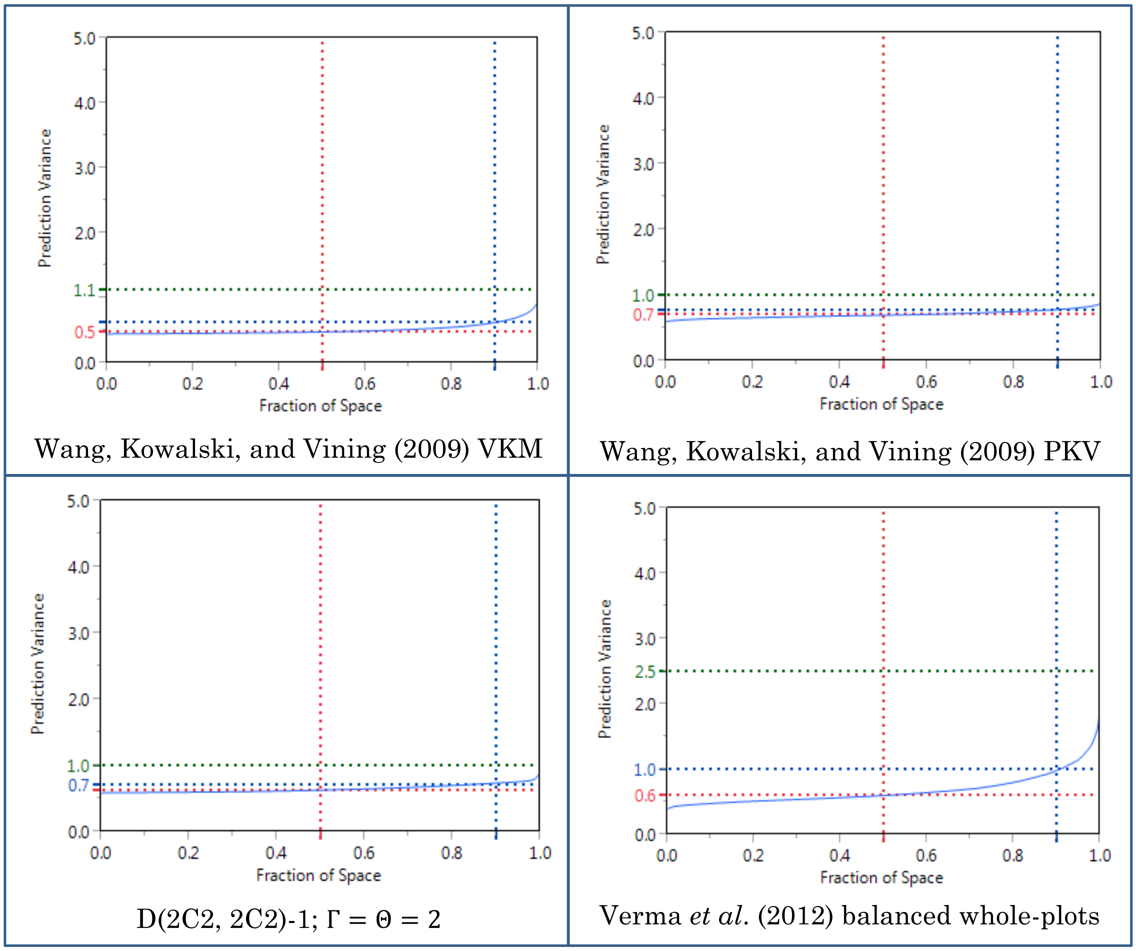


Figure 4-3. Fraction of design space (unscaled prediction variance)

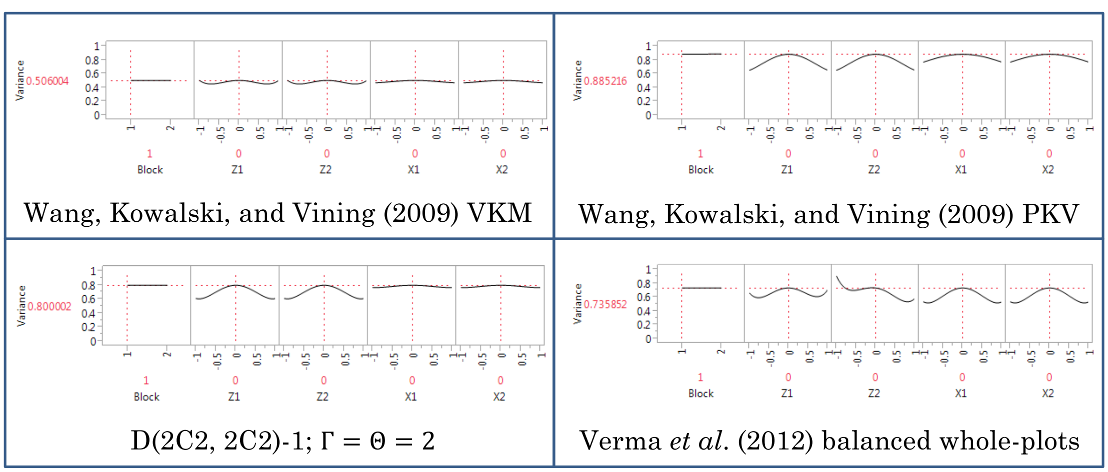


Figure 4-4. Unscaled prediction variance profile

5 Summary and Conclusions

Replication, randomization, and local control of error have played an important role in experiments as early as Pierce and Jastrow (1885). Fisher (1926) embedded those principles in the fabric of the design of experiments and introduced the split-plot experiment for agronomic research. Split-plot design was extremely useful in agronomical experiments where the primary concern was first-order effects. Forms of the split-plot design, like Taguchi's inner and outer array design, found their way into industrial experiments. Taguchi's inner and outer array designs are highly regarded by the quality and manufacturing control community; however, Bisgaard (2000) indicated that they are not widely recognized as split-plot experiments and are incorrectly analyzed as if they were completely randomized designs often resulting in incorrect models.

Box and Wilson (1951) catalyzed the application of design of experiments to industrial experiments and jumped started the development of response surface methodology. While response surface methodology has experienced significant growth since Box and Wilson (1951), the design of second-order split-plot experiments, with and without blocking, started receiving significant attention at the turn of the millennium. Simpson, Kowalski, and Landman (2004) illuminated the issues affecting experimentation under restricted randomization, which were introduced in Chapter 1 to motivate this research.

The need for developing or adapting design techniques to second-order split-plot design, without and with blocking, was validated by the literature review presented in Chapter 2. While the body of literature related to response surface methodology, blocking, restricted randomization, design evaluation criteria, and first-order split-plot design is vast, literature on second-order split-plot design, particularly with blocking, is more limited. Only two peer-reviewed papers addressing the topic were found: Wang, Kowalski, and Vining (2009) proposed OLS-GLS equivalent estimation, rotatable, orthogonally blocked central composite split-plot designs; Verma *et. al.* (2012) constructed balanced and unbalanced orthogonally blocked second-order split-plot designs from historical designs.

There are several techniques to generate split-plot designs. One of those methods is the Cartesian product method, which is ineffective. In Chapter 3, an end-to-end, innovative, and effective approach, rooted on traditional response surface methodology, was used to construct a derivative of the Cartesian product method referred to as *second-order sub-array Cartesian product split-plot design*. Guidance on how to construct balanced and unbalanced designs derived from central composite, Box-Behnken, and definitive screening designs was provided. In Chapter 4, blocking strategies that allow for effective and efficient use of resources in concomitant homogeneous and heterogeneous settings were introduced. In Chapter 4, the addition of two criterion to the split-plot evaluation criteria by Myers, Montgomery, and Anderson-Cook (2009) were proposed: the sequential assembly of higher order split-plot designs and the ability to carry out split-plot experiment in blocks.

In chapters 3 and 4, the features of the designs were demonstrated. The characteristics of the designs were evaluated in view of design evaluation criterion by Box and Draper (1975) and by Myers, Montgomery, and Anderson-Cook (2009), and their performance was assessed relative to other historical designs. To assess the performance of the designs, attention was placed to the stability of the prediction variance over the entire design space, the unscaled prediction variance at the center of the design space, the maximum unscaled prediction variance, the average unscaled prediction variance, and the unscaled prediction variance at the 90^{th} percentile of the design space. The pairwise correlation coefficients between model terms, the ability to estimate both the whole-plot variance and the sub-plot variance, and orthogonal blocking were also considered.

Second-order sub-array Cartesian product split-plot designs have desirable features. The response surface designs for the whole-plot and sub-plot sub-arrays are independent from each other, which really permits treating the split-plot design as a superposition of two different experiments. When the whole-plot and sub-plot sub-arrays are second-order orthogonal, the product is a second-order orthogonal design. The designs typically have, or can be easily augmented to have, a sufficient and economical number of whole-plot and sub-plot degrees-of-freedom that permit estiimating the whole-plot variance and sub-plot variance. The designs could be particularly useful when the shapes of the experimental regions for the whole-plot and sub-plot levels are different.

Some aspects of the sub-array Cartesian product split-plot designs need further research. A mathematical formulation of the design can facilitate the construction and handling of the design. Constructing the designs from small designs such as Hoke (1974) saturated designs, Roquemore (1976) hybrid designs, and Notz (1982) minimal-point designs can provide further insight into the performance of the design. Since the second-order sub-array Cartesian split-plot designs can be constructed with only factorial, axial, and center point sub-arrays, it will be ideal to expand the number of designs based on combinations of those sub-arrays. Albeit the proposed method shows improvement over some state-of-the-art methods, further validation is recommended.

Second-order sub-array Cartesian product split-plot designs are a departure from second-order split-plot state-of-the-art designs. First, the treatment design of classical second-order designs is partitioned into second-order sub-arrays, which are assigned to the treatments of the whole-plot factors and sub-plot factors. Then, the Cartesian product method is used to form ordered pairs of whole-plot sub-arrays by sub-plot sub-arrays rather than the ordered pairs of full whole-plot arrays by full sub-plot arrays produced by the Taguchi method. Then, the sub-array Cartesian products are concatenated into a new observation design. Finally, the sub-array structure is used to provide blocking strategies that permit effective and efficient use of resources.

The new form of second-order split-plot design produced by this research is an efficient and effective alternative to split-plot design methods like the Taguchi's inner and outer array design method. The designs are economical, and generally require about one-half of the number of runs required by full Cartesian product designs. Most of the second-order sub-array Cartesian product split-plot designs permit estimating the model coefficients for all first-order, all two-factor interactions, and all pure second-order terms unlike product array methods where the primary concern is first-order orthogonal effects. The sub-array structure has the desirable feature that the second-order structure of the design typically can be revealed just by inspection of the sub-arrays.

Sub-array Cartesian product split-plot designs are high information-quality designs. The designs perform well. The variance of the regression coefficients is low. Similarly, the prediction variance of the regression coefficients is low and stable. The aliasing between the terms in the model and likely effects that are not in the model as well as the correlation between similar effects that are not in the model is low. Based on an assessment using key design evaluation criterion established by Box and Draper (1975) and Myers, Montgomery, and Anderson-Cook (2009), it was demonstrated that *second-order sub-array Cartesian product split-plot designs* perform as well or better than historical designs that have been considered standards up to now.

The design performs well, as illustrated in Table 5-1 through 5-4. The variance of the regression coefficients is low. Similarly, the prediction variance of the regression coefficients is low and stable. The aliasing between terms in the model and likely effects that are not in the model as well as the correlation between similar effects that are not in the model is low.

The performance of the second-order sub-array Cartesian product split-plot design was assessed relative to the performance of the central composite designs and Box-Behnken designs provided by both Vining, Kowalski, and Montgomery (2005) and Parker, Kowalski, and Vining (2007a), and to the three-level designs provided by Verma *et. al.* (2012). Based on criterion involving prediction variance and pairwise correlation coefficients between model terms, the results in Tables 5-1 through 5-3 demonstrate that second-order sub-array Cartesian product split-plot designs perform as well or better than designs that have been considered standards up to date.

The performance of the second-order sub-array Cartesian product block split-plot designs were assessed relative to the designs provided by Vining, Kowalski, and Montgomery (2005) and Parker, Kowalski, and Vining (2007a) as blocked by Wang, Kowalski, and Vining (2009) as well as the designs provided by Dey (2009) and Zhang (2011) as blocked by Verma *et. al.* (2012). As shown in Table 5-4, second-order sub-array Cartesian product block split-plot design outperform the design provided by Dey (2009) as blocked by Verma *et. al.* (2012) and the design provided by Parker, Kowalski, and Vining (2007a) as blocked by Wang, Kowalski, and Vining (2009) when judged by the prediction variance and the pairwise correlation coefficients between model terms criterion. The second-order sub-array Cartesian product split-plot block design also outperforms the design provided by Zhang (2011) as blocked by Verma *et.al.* (2012). The second-order sub-array Cartesian product block split-plot design also outperform the design provided by Vining, Kowalski, and Montgomery (2005) as blocked by Wang, Kowalski, and Vining (2009) when judged by the pairwise correlation between model terms criterion.

Table 5-1. Performance assessment summary – whole-plot variance estimation

	Vining, Kowalski, and Montgomery (2005) OLS-GLS equiv.	Jones and Natchsheim (2009) D-optimal design	Sub-array Cartesian product split-plot D(2C2, 2C2)-2
Structure			
No. whole-plots	12	12	12
Total runs	48	48	60
Balanced whole-plots	Y	Y	Y
β/α	1.414/1.414	n/a	1.414/1.414
Prediction variance			
@ 50% FDS	0.82	1.09	0.56
@ 90% FDS	1.70	1.73	1.25
@ overall center	0.35	1.54	0.37
Average	0.95	1.18	0.70
Maximum	3.82	4.18	3.00
No. Correlation coefficient |r| (other than main diagonal)			
Clear	85	65	89
0.0 < |r| < 0.50	6	26	2
0.50 < |r| < 1.0	0	0	0

Table 5-2. Performance assessment summary – standard designs

	Vining, Kowalski, and Montgomery (2005) OLS-GLS equiv.	Sub-array Cartesian product split-plot D(2C2, 2C2)-1	Parker, Kowalski, and Vining (2007b) minimum whole-plot	Sub-array Cartesian product split-plot D(3C2, 3C2)-5
Structure				
No. whole-plots	10	10	9	9
Total runs	40	50	45	36
Balanced whole-plots	Y	Y	Y	Y
β/α	1.414/1.414	1.414/1.414	2.0/2.0	2.0/2.0
Prediction variance				
@ 50% FDS	0.90	0.60	0.80	1.10
@ 90% FDS	1.80	1.30	1.30	1.80
@ overall center	0.75	0.68	1.07	1.87
Average	1.03	0.80	0.86	1.25
Maximum	4.30	3.40	3.00	6.00
No. Correlation coefficient |r| (other than main diagonal)				
Clear	89	89	85	85
0.0 < |r| < 0.50	2	2	5	5
0.50 < |r| < 1.0	0	0	1	1

Table 5-3. Performance assessment summary – minimum whole-plot

	Parker, Kowalski, and Vining (2007a) minimum whole-plot	Verma *et. al.* (2012) unbalanced	Sub-array Cartesian product split-plot D(2B2, 2B2)-2
Structure			
No. whole-plots	9	9	9
Total runs	45	22	45
Balanced whole-plots	Y	N	Y
β/α	2.0/2.0	1.0/1.0	1.0/1.0
Prediction variance			
@ 50% FDS	1.5	2.5	1.0
@ 90% FDS	3.8	6.8	2.1
@ overall center	1.1	1.5	0.7
Average	2.0	3.4	1.2
Maximum	11.4	26.0	4.8
No. Correlation coefficient |r| (other than main diagonal)			
Clear	86	82	86
0.0 < |r| < 0.50	5	6	5
0.50 < |r| < 1.0	0	3	0

Table 5-4. Performance assessment summary – blocked designs

	Wang, Kowalski, and Vining (2009) – Vining, Kowalski, and Montgomery (2005)	Sub-array Cartesian product split-plot D(2C2, 2C2)-1	Wang, Kowalski, and Vining (2009) – Parker, Kowalski, and Vining (2007a)	Verma *et. al.* (2012)
Structure				
No. whole-plots	12	10	10	14
Total runs	48	50	58	36
Balanced whole-plots	Y	Y	N	N
β/α	1.414/2.828	1.414/1.414	1.871/2.160	1.0/1.0
Prediction variance				
@ 50% FDS	0.5	0.6	0.7	0.6
@ 90% FDS	0.6	0.7	0.8	1.0
@ overall center	0.51	0.80	0.89	0.74
Average	0.52	0.65	0.71	0.68
Maximum	1.1	1.0	1.0	2.5
No. Correlation coefficient |r| (other than main diagonal)				
Clear	85	89	81	72
0.0 < |r| < 0.50	6	2	10	19
0.50 < |r| < 1.0	0	0	0	0

Bibliography

[1] Anderson-Cook, C. M., Borror, C. M., and Montgomery, D. C. (2009). Response surface design evaluation and comparison (with discussion). *Journal of Statistical Planning and Inference*, **139**, 629-674.

[2] Bingham, D. R., and Sitter, R. R. (1999a). Some theoretical results for fractional factorial split-plot designs. *Annals of Statistics*, **27**, 1240-1255.

[3] Bingham, D. R., and Sitter, R. R. (1999b). Minimum aberration two-level fractional-factorial split-plot designs. *Technometrics*, **41**, 62-70.

[4] Bingham, D. R., and Sitter, R. R. (2001). Design issues in fractional factorials split-plot experiments. *Journal of Quality Technology*, **33**, 2-15.

[5] Bisgaard, S. (1992). The design and analysis of 2^{k-p} x 2^{q-r} inner and outer array experiments. *Center for Quality and Productivity Improvement*, University of Wisconsin, Madison, WI, CQPI Report No. 90.

[6] Bisgaard, S. (2000). The design and analysis of 2^{k-p} x 2^{q-r} split-plot experiments. *Journal of Quality Technology*, **32**, 39-56.

[7] Bisgaard, S., and de Pinho, A. L. S. (2004). The error structure of split-plot experiments. *Quality Engineering*, **16**, 671-675.

[8] Bisgaard, S., and Sutherland, M. (2003). Split-plot experiments: Taguchi's Ina tile experiment reanalyzed. *Quality Engineering*, **16**, 157-164.

[9] Bisgaard, S., and Steinberg, D. M. (1997). The design and analysis of 2^{k-p} x s prototype experiments. *Technometrics*, **39**, 52-62.

[10] Box, G. E. P. (1968). Response surfaces. In D. L. Sills (Ed.) *The International Encyclopedia of the Social Sciences*. New York: MacMillan and Free Press.

[11] Box, G. E. P. (1992). George's Column; Sequential experimentation and sequential assembly of designs. *Quality Engineering*, **5**, 321-330.

[12] Box, G. E. P. (1999). Statistics as a catalyst to learning by scientific method; Part II – Discussion. *Center for Quality and Productivity Improvement*, Report No. 172. University of Wisconsin.

[13] Box, G. E. P., and Behnken, D. W. (1960). Some new three-level designs for the study of quantitative variables. *Technometrics*, **2**, 455-476.

[14] Box, G. E. P., and Draper, N. R. (1959). A basis for the selection odf a response surface design. *Journal of the American Statistical Association*, **72**, 622-654.

[15] Box, G. E. P., and Draper, N. R. (1974). On minimum-point second-order designs. T*echnometrics*, **16**, 613-616.

[16] Box, G. E. P., and Draper, N. R. (1975). Robust designs. *Biometrika,* **62**, 347-352.

[17] Box, G. E. P., and Draper, N. R. (2007). *Response surfaces, mixtures, and ridge analysis.* (2nd ed.). New York: Wiley & Sons.

[18] Box, G. E. P., and Hunter, J. S. (1957). Multi-factor experimental designs for exploring response surfaces. *Annals of Mathematical Statistics*, **28**, 195-241.

[19] Box, G. E. P., and Jones, S. (1992). Split-plot designs for robust product and process experimentation. *Journal of Applied Statistics*, **19**, 3-26.

[20] Box, G. E. P., and Jones, S. (2001). Split-plots for robust product and process experimentation. *Quality Engineering,* **13**, 127-134.

[21] Box, G. E. P., and Wilson, K. G. (1951). On the experimental attainment of optimum conditions (with discussion). *Journal of the Royal Statistical Society B*, **13**, 1-45.

[22] Christensen, E. H., Coombes-Betz, K. M., and Stein, M. S. (2007). *The certified quality process analyst handbook*. Milwaukee: ASQ Quality Press.

[23] Cochran, W. G., and Cox, G, M. (1992). *Experimental Designs*. (2nd ed.). New York: Wiley & Sons.

[24] Daniel, C. (1959). Use of half-normal plots in interpreting factorial two-level experiments. *Technometrics*, *1*, 311-341.

[25] Dey, A. (2009). Orthogonally blocked three-level second-order designs. *Journal of Statistical Planning and Inference*, **139**, 3698-3705.

[26] Doehlert, G. H. (1970) Uniform shell designs. *Journal of the Royal Statistical Society, Series C*, **19**, 231-239.

[27] Draper, N. R. (1985). Small composite designs. *Technometrics*, **27**, 173-180.

[28] Draper, N. R., and John, J. A. (1998). Response surface designs where levels of some factors are difficult to change. *Australian and New Zealand Journal of Statistics*, **40**, 487-495.

[29] English, T. G., Simpson, J. R., Landman, D., and Parker, P. A. (2012). An efficient split-plot approach for modeling non-linear aerodynamic effects. *Quality Engineering*, **24**, 522-530.

[30] Fisher, R. A. (1925). *Statistical methods for research workers*. Edinburgh and London: Oliver and Boyd.

[31] Fisher, R. A. (1926). The arrangement of field experiments. *Journal of the Ministry of Agriculture of Great Britain*, **33**, 503-513.

[32] Fisher, R. A. (1935). *The design of experiments*. Edinburgh and London: Oliver and Boyd.

[33] Federer, W. T., and King, F. (2007). Variations on split-plot and split block experiment designs. New York: Wiley & Sons.

[34] Ganju, J., and Lucas, J. M. (1997). Bias in test statistics when restrictions in randomization are caused by factors. *Communications in Statistics, Theory and Methods*, **26**, 47-63.

[35] Ganju, J., and Lucas, J. M. (1999). Detecting randomization restrictions caused by factors. *Journal of Statistical Planning and Inference*, **81**, 129-140.

[36] Gilmour, S. G. (2004). Irregular four-level response surface designs. *Journal of Applied Statistics*, **31**, 1043-1048.

[37] Gilmour, S. G., and Trinca, L. A. (2006). Response surface experiments on processes with high variation. In *Response surface methodology and related topics,* (A. I. Khuri, ed.), pp. 19-46. Hackensack, NJ: World Scientific Publishing

[38] Goos, P. (2002). *The optimal design of blocked and split-plot experiments*. New York: Springer.

[39] Goos P. (2006). Optimal versus orthogonal and equivalent-estimation design of blocked and split-plot experiments. *Statistica Nerlandica*, **60**, 361-378.

[40] Goos, P., and Vandenbroek, M. (2001). Optimal split-plot designs. *Journal of Quality Technology*, **33**, 436-450.

[41] Goos, P., and Vandebroek, M. (2003). D-optimal split-plot designs with given number and sizes of whole-plots. *Technometrics*, **45**, 235-245.

[42] Hader, R. J. (1973). An improper method of randomization in experimental design. *The American Statistician*, **27**, 82-84.

[43] Haines, L. M. (2006). Evaluating the performance of non-standard designs: The San Cristobal design. In *Response surface methodology and related topics,* (A. I. Khuri, ed.), 251-281. Hackensack, NJ: World Scientific Publishing.

[44] Hartley, H. O. (1959). Smallest composite designs for quadratic response surfaces. *Biometrics*, **15**, 611-624.

[45] Hinkelmann, K., and Kempthorne, O. (1994). *Design and analysis of experiments: Volume 1 – Introduction to experimental design.* New York: Wiley & Sons.

[46] Hoke, A. T. (1974). Economical second-order designs based on irregular fractions of the 3n factorial. *Technometrics*, **16**, 375-384.

[47] Huang, P., Chen, D., and Voelkel, J. (1998). Minimum aberration two-level split-plot designs. *Technometrics*, **40**, 314-326.

[48] Jensen, W. A., and Kowalski, S. M. (2012). Response surfaces, blocking, and split-plots: An industrial experiment case study. *Quality Engineering*, **24**, 531-542.

[49] Jones, B., and Nachtsheim, C. J. (2009). Split-plot designs: What, Why, How. *Journal of Quality Technology*, **41**, 340-361.

[50] Jones, B., and Natchsheim, C. J. (2011). A class of three level designs for definitive screening in the presence of second-order effects. *Journal of Quality Technology*, **43**, 1-15.

[51] Ju, H. L., and Lucas, J. M. (2002). Lk factorial experiments with hard-to-change and easy-to-change factors. *Journal of Quality Technology*, **34**, 411-421.

[52] Kiefer, J. C. (1959). Optimum experimental design (with discussion). *Journal of the Royal Statistical Society B*, **21**, 272-319.

[53] Kiefer, J. C. (1961). Optimum designs in regression problems. II. Annals of Mathematical Statistics, **32**, 298-325.

[54] Keifer, J., and Wolfowitz, J. (1960). The equivalence of two extremum problems. *Canadian Journal of Mathematics*, **12**, 363-366.

[55] Khuri, A. I. (1992). Response surface models with random block effects. *Technometrics*, **34**, 26-37.

[56] Khuri, A. I. (2003). Current modeling and design issues in response surface methodology: GLMs and models with block effects. In R. Khattree and C. R. Rao (Eds.) *Handbook of statistics*, Vol 22, Chapter 6. Amsterdam: Elsevier Science.

[57] Khuri, A. I., and Mukhopadhyay, S. (2010). Response surface methodology. *WIREs Computational Statistics*, **2**, 128-149.

[58] Letsinger, J. D., Myers, R. H., and Lentner, M. (1996). Response surface methods for bi-randomization structures. *Journal of Quality Technology*, **28**, 381-397.

[59] Liang, L., Anderson-Cook, C. M., and Robinson, T. J. (2006). Fraction of design space plots for split-plot designs. *Quality and Reliability Engineering International*, **22**, 275-289.

[60] Lind, J. (1753). *A treatise of scurvy. In three parts. Containing an inquiry into the nature, causes and cure, of that disease. Together with a critical and chronological view of what has been published on the subject.* Edinburgh: Sands, Murray and Cochran.

[61] Loeppky, J. L., and Sitter, R. R. (2002). Analyzing unreplicated blocked or split-plot fractional factorial designs. *Journal of Quality Technology*, **34**, 229-243.

[62] Lucas, J. M. (1974). Optimum composite designs. *Technometrics*, **16**, 561-567.

[63] Lucas, J. M., and Ju, H. L. (1992). Split-plotting and randomization in industrial experiments. *ASQC Quality Congress Transactions*, American Society for Quality Control, Nashville, TN, 374-382.

[64] Macharia, H., and Goos, P. (2010). D-Optimal and D-efficient equivalent estimation second-order split-plot designs. *Journal of Quality Technology*, **42**, 358-372.

[65] Mitchell, T. J., & Bayne, C. K. (1978). D-optimal fractions of three level factorial designs. *Technometrics*, 20, 369-380.

[66] Montgomery, D. C. (2004). *Diseño y análisis de experimentos*. (2da. ed.). Ciudad de Mexico, D.F.: Editorial Limusa, S.A. DE C.V.

[67] Montgomery, D. C., Peck, E. A., Vining, G. G. (2012). *Introduction to linear regression analysis*. (5th ed.). New York: Wiley & Sons.

[68] Morris, M. (2000). A class of three-level experimental designs for response surface modeling. *Technometrics*, **42**, 111-121

[69] Myers, R. H. (1999). *Response surface methodology; current status and future directions*. Journal of Quality Technology, **31**, 30-44.

[70] Myers, R. H., Montgomery, D. C., Vining, G. G., Borror, C. M., and Kowalski, S. M. (2004). Response surface methodology: A retrospective and literature survey. *Journal of Quality Technology*, **36**, 53-77.

[71] Myers, R. H., Montgomery, D. C., and Anderson-Cook, C. M. (2009). *Response surface methodology*. New York: Wiley & Sons.

[72] Notz, W. (1982). Minimal point second-order designs. *Journal of Statistical Planning and Inference*, **6**, 47-58.

[73] Oehlert, G. W., and Whitcomb, P. (2002). Small, efficient, equireplicated resolution V fractions of 2^k designs and their application to central composite designs. *46th Annual Fall Technical Conference,* ASQ and ASA, Valley Forge, PA

[74] Park, Y. -J., Richardson, D. E., Montgomery, D. C., Ozol-Godfrey, A., Borror, C. M., Anderson-Cook, C. M. (2005). Prediction variance properties of second-order designs for cuboidal regions. *Journal of Quality Technology*, **37**, 253-266.

[75] Parker, P. A., Kowalski, S. M., and Vining, G. G. (2006). Classes of split-plot response surface designs for equivalent estimation. *Quality and Reliability Engineering International*, **22**, 291-305.

[76] Parker, P. A., Anderson-Cook, C., Robinson, T. J., and Liang, Li. (2007). Robust split-plot designs. *Quality and Reliability Engineering International*, **24**, 107-121.

[77] Parker, P. A., Kowalski, S. M., and Vining, G. G. (2007a). Construction of balanced equivalent estimation second-order split-plot designs. *Technometrics*, **49**, 56-65.

[78] Parker, P. A., Kowalski, S. M., and Vining, G. G. (2007b). Unbalanced and minimal point equivalent estimation second-order split-plot designs. *Journal of Quality Technology*, **39**, 376-388.

[79] Peirce, C. S., and Jastrow, J. (1885). On small differences in sensation. *Memoirs of the National Academy of Science*, **3**, 73-83.

[80] Pesotchinsky, L. L. (1975). D-optimum and quasi D-optimum second-order designs on a cube. *Biometrika*, 62, 335-340.

[81] Plackett, R. L., and Burman, J. P. (1946). The design of optimum multifactorial experiments. *Biometrika*, **33**, 305-325, 328-332.

[82] Rechtschaffner, R. l. (1967). Saturated fractions of 2^n and 3^n factorial designs. *Technometrics,* **9**, 569-575.

[83] Roquemore, K. G. (1976). Hybrid designs for quadratic response surfaces. *Technometrics*, **4**, 419-423.

[84] Simpson, J. R., Kowalski, S. M., and Landman, D. (2004). Experimentation with randomization restrictions: Targeting practical implementation. *Quality and Reliability Engineering International*, **20**, 481-495.

[85] Taguchi, G. (1987). *System of experimental design, Vols. 1 and 2*, White Plains, NY: Kraus International.

[86] Trench, W. F. (2013). *Introduction to real analysis*. San Antonio, TX; Trinity University.

[87] Trinca, L. A., and Gilmour, S. G. (2001). Multistratum response surface designs. *Technometrics*, **43**, 25-33.

[88] Verma, A., Aggarwal, M. L., Chowdhury, S. R., Singh, P. (2012) *Response surface designs under split-plot structure* (Doctoral dissertation). Retrieved from Shodhganga, Indian ETD Repository (http://hdl.handle.net/10603/28352)

[89] Vining, G. (2012). Split-plot response surface designs. In K. Hinkelmann (Ed.) *Design and analysis of experiments, Vol. 3: Special designs and applications*. Hoboken: Wiley & Sons.

[90] Vining, G. G., Kowalski, S. M., and Montgomery, D. C. (2005). Response surface designs within a split-plot structure. *Journal of Quality Technology*, **37**, 115-129.

[91] Vining, G. G., and Kowalski, S. M. (2008). Exact inference for response surface designs within a split-plot structure. *Journal of Quality Technology*, **40**, 394-406.

[92] Wang, L., Kowalski, S. M., and Vining, G. G. (2009). Orthogonal blocking of response surface split-plot designs. *Journal of Applied Statistics*, **36**, 303-321.

[93] Westlake, W. J. (1965). Composite designs based on irregular fractions of factorials. *Biometrics*, **21**, 324-336.

[94] Wooding, W. M. (1973). The split-plot design. *Journal of Quality Technology*, **5**, 16-33.

[95] Yates, F. (1933). The principles of orthogonality and confounding in replicated experiments. *Journal of Agricultural Science*, **23**, 108-145.

[96] Yates, F. (1935). Complex experiments. *Journal of the Royal Statistical Society,* Supplement 2, 181-247.

[97] Zahran, A., Anderson-Cook, C. M., and Myers, R. H. (2003). Fraction of design space to assess the prediction capability of response surface designs. *Journal of Quality Technology*, **35**, 377-386.

[98] Zhang, T., Yang, J., and Lin, D. K. J. (2011). Small Box-Behken designs. *Statistics and Probability Letters,* **81**, 1027-1033