An Algorithm for Generating Response Surface Split-Plot Designs

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Abstract

In an industrial experiment, the presence of Hard-to-Change factors may force the experimenter to use a split-plot type experimental structure. The corresponding Response Surface model then is a linear mixed model. Different design methodologies and estimation techniques are available in the literature, for constructing a Response Surface split-plot design and for estimating the variance components of the model. We discuss the need for developing a new design construction methodology and present an algorithm for generating a D-optimal Response Surface split-plot design such that it has pre-specified numbers of degrees of freedom for estimating the variance components using the randomization based approach. An advantage of this approach is that it gives pure-error estimates of the variance components.

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Chapter 1

Introduction

Split-plot designs were originally developed for use in agricultural studies. These designs were basically the modified forms of randomized block designs for use in a situation where it was not possible to perform all the field operations on small plots. When conducting a field experiment, it is quite possible, for practical convenience or because of technical reasons, that one or more factors require a large area for application whereas some other factors can be applied and tested on a relatively smaller field. This type of experimental structure leads to split-plot designs. The large experimental unit is called a whole-plot and a factor applied to this experimental unit is called a whole-plot factor. The small experimental units are called sub-plots and the factors applied to these experimental units are called sub-plot factors. An early history of split-plot designs with their application can be seen in Yates (1935)[29].

Suppose that, (Jones & Nachtsheim, 2009[19]), we are interested in testing the effect of different irrigation systems and fertilizers on a crop yield. We have two different irrigation systems and two different types of fertilizer to test. Due to technical reasons, different irrigation systems cannot be applied to an area smaller than a field but different types of fertilizer can effectively be applied to a smaller area of the field. Further suppose that we have four fields to conduct the experiment. In this situation, different levels of the irrigation system are applied to the fields with complete randomization. One way to perform the complete randomization is, as described by Bailey (2008, p.147)[3], to write down a design on a paper where fields are numbered from 1 to 4 and each level of irrigation system is applied to an equal number of

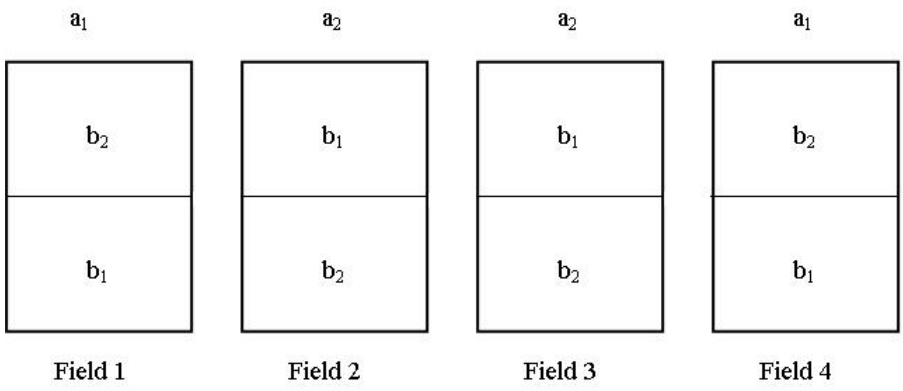

Figure 1.1: Experimental layout for field experiment

fields, that is 2 in this case. For example, one of the irrigation systems, say a_1, is applied to the fields numbered 1 and 2, while the other, say a_2, is applied to the fields numbered 3 and 4. Then we get a random permutation of the numeric sequence by using a pack of cards or by generating random numbers by a computer or calculator. Suppose that the random permutation is $1, 4, 2, 3$, then a_1 will be applied on field 1 and 4 and a_2 will be applied on field 2 and 3. Similarly, different types of fertilizer are randomly applied to the plots within each field and an independent randomization is performed for every field (see fig. 1.1). Here, each field is a whole-plot and irrigation system is the whole-plot factor whereas plots within each field are sub-plots and fertilizer is the sub-plot factor and the whole experimental structure is a split-plot design. More recent details on split-plot designs and several variations of such designs can be found in Montgomery (2008)[24]

The split-plot type experimental structure is common in industrial experiments also. As noted by Cochran & Cox (1957)[9], one set of factors may require a large amount of experimental material while another set of factors might be tested on a smaller amount of material. For illustration consider an example given by Bingham & Sitter (2001)[5]. A company wants to test the effects of some factors on its final wood product. The manufacturing process of the product consists of two stages. In the first stage, one set of factors is combined with pieces of wood to form a large batch of wood, this batch is then divided into four sub-batches and each sub-batch is then processed

with another set of factors to produce the final product (sheet). Here, each batch forms a whole-plot and the factors applied to produce a batch are the whole-plot factors whereas each sub-batch forms a sub-plot and the factors applied to these sub-batches are the sub-plot factors. Another situation that leads to the use of a split-plot design is when there exist one or more factors that require more time, cost or other resources for changing level settings than other factors. These factors are called the Hard-to-Change (HTC) factors and the other factors are called the Easy-to-Change (ETC) factors. In this situation, it is desired to change the level settings of the HTC factors as infrequently as possible. This goal can be achieved by randomly applying the HTC factors to larger experimental units and the ETC factors to small experimental units. Here, a large experimental unit can be a physically large experimental unit or it can be a longer period of time, such as a full day. This situation is further explained with examples in section 1.3.

Many industrial experiments are conducted with an objective to determine the relation between the response and different level settings of factors of interest, also called the input variables. It is believed that this relation can be approximated by a linear regression model. The methodology to approximate the true relation between a response and a set of input variables through a linear regression model is called Response Surface (RS) Methodology. RS methodology is a collection of statistical designs and techniques useful for product optimization within a region of interest and it has now become an important part of industrial experiments.

Because, in a split-plot design, we have two different types of experimental units, large and small, and randomization is performed independently at two different levels, whole-plot level and sub-plot level, so we have two different sources of error. First, the whole-plot factors are randomly applied to the whole-plots that generate whole-plot error and then sub-plot factors are randomly applied to the sub-plots that generate the sub-plot error. So, the RS model for a split-plot design is a linear mixed model that contains two error terms, one for the whole-plot error and the other for the sub-plot error. These terms are assumed to be random and uncorrelated having zero mean and constant, but usually unknown, variances which are usually referred to as the variance components of the model. These variance components are involved in estimating the fixed effects of the model and their standard errors. So, a careful estimation of the variance components is required for obtain-

ing reliable results. The estimation of these variance components has been of much interest in recent years. Different techniques are available in the literature for estimating the variance components including Residual Maximum Likelihood (REML), a randomization based approach and a Bayesian approach.

Another issue, when estimating the variance components, is the numbers of degrees of freedom available for estimation. It is important to have appropriate numbers of degrees of freedom for obtaining good estimates of the variance components because the estimates obtained by using only a few numbers of degrees of freedom are not generally considered very reliable. Some authors recommend the use of at least five degrees of freedom for obtaining good estimates of the error variance (Cox, 1958 p.167 [10]). Numbers of degrees of freedom used to estimate the variance components also play an important role in testing the significance of different effects and/or drawing inferences about the model parameters. Higher numbers of degrees of freedom help obtaining more precise estimates than those obtained by using lower numbers of degrees of freedom.

Several design construction methodologies have been developed for constructing RS split-plot designs advocating different techniques for estimating the variance components. But, as we will see later, hardly any of these design construction methodologies generates efficient designs with the freedom of choosing the numbers of degrees of freedom for estimating variance components. In this thesis we will address this problem by proposing an algorithm that would generate a D-optimal RS split-plot design with pre-specified numbers of degrees of freedom for estimating the variance components using randomization based approach. This approach would give us unbiased pure error estimates of the variance components. For a RS model, the variation among the observed data due to experimental error can be partitioned into pure error and lack-of-fit components. The pure error component is based on the true replication of treatments (different combinations of factor levels). The true replication of a treatment demands that if that treatment is applied to an experimental unit, the whole experimental process (changing of factor levels) must be redone when the same treatment is applied to another experimental unit. The randomization based approach suggests using a full treatment model, by ignoring any treatment structure in a split-plot design. So, we will be thinking of a split-plot design as a general incomplete block

design considering whole-plots as blocks and sub-plots as experimental units within blocks. We will assume that treatments having no particular structures are randomly allocated to experimental units within blocks each of the same size. There are some standard results in the literature for estimating the variance components for a general incomplete block design from a treatment model with the related degrees of freedom. We will use and modify these results to establish the theoretical basis for our algorithm.

In this chapter, we give an introduction to split-plot designs. Sections 1.1 and 1.2 contain some material on classical split-plot designs and their analysis with a focus on agricultural experiments. In section 1.3, we discuss the use of split-plot designs in industrial experiments.

1.1 Classical Split-plot Designs

Split-plot designs, like many other designs, were initially used in field experiments. These designs can be used in the following two situations (Cox 1958, §7.4 [10]).

1. When the number of treatment combinations exceeds the number of homogeneous experimental units arranged in blocks.

2. When it is not possible to perform all experimental operations on smaller experimental units.

When dealing with the first situation, one option could be to hold the levels of one or more factors constant for a block so that the number of treatment combinations reduces to a reasonable size. For illustration suppose that we want to investigate the effect of 3 factors, A, B, and C, each with two levels, on a crop yield. So, the number of treatment combinations is 8. Further suppose that we have four fields for performing the experiment but each field is divided into 4 plots. Since, the number of treatment combinations exceeds the number of units in a block a Randomized Complete Block Design cannot be used. So, to deal with this situation one option could be to hold the level of factor A constant for each block, thus the number of treatment combinations involving factors B and C will reduce to 4. Here each field will form a whole-plot and A is the whole-plot factor. Whereas each plot will form a sub-plot and factors B and C are sub-plot factors. But this approach should

only be adopted if a classificatory factor, such as factor A in our example, is included in the experiment only to get information about its interaction with other factors and its main effects are of little interest.

The second situation is described in the example where we are interested in investigating the effect of different irrigation systems and fertilizers on a crop yield. There, the irrigation system could not be applied on smaller experimental units (plots) so the problem was addressed by applying each level of the irrigation system to a larger experimental unit (field) and each level of fertilizer to a smaller experimental unit (plot). Here the larger experimental unit forms a whole-plot and the smaller experimental unit forms a sub-plot.

Split-plot designs can also be useful, and advantageous, in the situation when there is a need to include an extra factor in the experiment. If an experiment is planned to be executed such that one or more factors are applied to the large experimental units then another factor that can be effectively applied on relatively smaller experimental units can be included in the experiment with little extra cost. Thus useful additional information can be gained without much extra cost.

1.2 Design and Analysis

We go back to our example where we are interested in testing the effect of 2 different irrigation systems and 2 different types of fertilizer on a crop yield. Let us denote the irrigation system as factor A and the fertilizer as factor B. Two different levels of factor A are denoted by a_1 and a_2 and two different levels of factor B are denoted by b_1 and b_2. We assume that we have 4 different fields to conduct the experiment. First, the levels of factor A are applied to the whole-plots (fields) with complete randomization, as stated earlier, and then different levels of factor B are applied, again with complete randomization, to sub-plots (plots within each field). A possible experimental layout is then described in Fig. 1.1. Note that each whole-plot has equal (single) replication of both sub-plot factor levels, thus all the whole-plots are of the same size of 2. There is also an equal replication of whole-plot factor levels.

For a split-plot design assuming one HTC factor (A) with a levels and one

ETC factor (B) with b levels we write, following Hinkelmann & Kempthorne (2008, p.538) [17], our model as

$$y_{ijk} = \mu + \alpha_i + \gamma_{ij} + \eta_k + (\alpha\eta)_{ik} + \epsilon_{ijk} \tag{1.1}$$

where $i = 1, 2, ..., a$, $j = 1, 2, ..., r$, $k = 1, 2, ..., b$, y_{ijk} is the response at the j^{th} replicate of the i^{th} level of factor A and the k^{th} level of factor B, μ is the overall mean, α_i is the fixed effect of the i^{th} level of factor A, γ_{ij} is the error term for the j^{th} replicate of the i^{th} level of factor A, η_k is the fixed effect of the k^{th} level of factor B, $(\alpha\eta)_{ik}$ is the fixed interaction effect of the i^{th} level of factor A and the k^{th} level of factor B, and ϵ_{ijk} is the error term for the j^{th} replicate of the combination of i^{th} level of factor A and the k^{th} level of factor B.
Under complete randomization, we assume that $E(\gamma_{ij}) = 0$, $E(\epsilon_{ijk}) = 0$, $cov(\gamma_{ik}, \epsilon_{ijk}) = 0$,

$$cov(\epsilon_{ijk}, \epsilon_{i'j'k'}) = \begin{cases} \sigma^2_\epsilon & \text{if } ijk = i'j'k' \\ 0 & \text{otherwise,} \end{cases}$$

and

$$cov(\gamma_{ij}, \gamma_{i'j'}) = \begin{cases} \sigma^2_\gamma & \text{if } ij = i'j' \\ 0 & \text{otherwise.} \end{cases}$$

Note that there are two error terms in the model, one for the whole-plot error and the other for the sub-plot error. This is due to the fact that we are using two different types of experimental units and the randomization is being performed independently at two different levels.

If the objective of the analysis is to check the significance of the effects of whole-plot and sub-plot factors then this objective can be achieved by using the ANOVA table. The ANOVA table for a split-plot design under model (1.1), taken from Hinkelmann & Kempthorne (2008, p.536)[17], is given in table 1.1.

The first part of the table contains the whole-plot analysis where the whole-plot factor (factor A) is compared with the whole-plot error (Error 1). And the second part of the ANOVA table shows the sub-plot analysis where sub-plot factor (factor B) and whole-plot and sub-plot factor interactions (AB) are compared with the sub-plot error (Error 2). By looking at the

Table 1.1: ANOVA table for a split-plot design

Source	DF	SS	E(MS)
Factor A	$a-1$	$rb\sum_i (\bar{y}_{i..} - \bar{y}_{...})^2$	$\sigma_\epsilon^2 + b\sigma_\gamma^2 + rb\frac{\sum \alpha_i^2}{a-1}$
Error 1	$a(r-1)$	$b\sum_{i,j} (\bar{y}_{ij.} - \bar{y}_{i..})^2$	$\sigma_\epsilon^2 + b\sigma_\gamma^2$
Factor B	$b-1$	$ra\sum_k (\bar{y}_{..k} - \bar{y}_{...})^2$	$\sigma_\epsilon^2 + ra\frac{\sum \eta_k^2}{b-1}$
AB	$(a-1)(b-1)$	$\sum_{i,j,k} (y_{ijk} - \bar{y}_{ij.} - \bar{y}_{i.k} + \bar{y}_{i..})^2$	$\sigma_\epsilon^2 + r\frac{\sum (\alpha\eta)_{ik}^2}{(a-1)(b-1)}$
Error 2	$a(r-1)(b-1)$	$\sum_{i,j,k} (y_{ijk} - \bar{y}_{ij.} - \bar{y}_{i.k} + \bar{y}_{i..})^2$	σ_ϵ^2
Total	$rab-1$	$\sum_{i,j,k} (y_{ijk} - \bar{y}_{...})^2$	

ANOVA table, we can see that the numbers of degrees of freedom (DF) for the sub-plot error are greater than those for the whole-plot error. This means that the main effects of the whole-plot factor are less precisely estimated as compared to the main effects of the sub-plot factor and sub-plot and whole-plot factor interactions.

1.3 Industrial Split-plot Designs

As stated earlier, there are at least two situations that can lead to the use of a split-plot design while conducting an industrial experiment. The first situation is when one set of factors is applied to a large amount of experimental material and another set of factors is applied to a relatively small amount of experimental material. The second situation is when one or more factors, involved in the experiment, are HTC and some other factors are ETC. In this situation it is not desirable to change the level settings of the HTC factors frequently. So the HTC factors are applied to larger experimental units and the ETC factors are applied to smaller experimental units. In the first situation, the large amount of experimental material is physically divided into several portions of a smaller amount of material and another set of factors is then applied to these smaller amounts of experimental material. But in the second situation, the large experimental unit is used for multiple experimental runs under different settings of ETC factors but it is not necessarily divided physically into several sub-units. The concept of the whole-plot is nearly the

same in both situations but there is a slight variation in the sub-plot concept.

We illustrate these two situations by giving some examples from the literature. The first two examples illustrate the situation where one set of factors was applied to a large amount of experimental material, that large experimental unit was then divided into several small units and a second set of factors was then applied to those small experimental units. The last two examples describe the situation where factors were classified as HTC and ETC. The HTC factors were applied to a large experimental unit and that large experimental unit was then used for multiple experimental runs with different settings of the ETC factors.

Example 1 (Box & Jones, 1992) *Consider the cake baking example given by Box & Jones (1992)[7]. The manufacturer of a cake mix wants to find out the the best recipe for his product according to a given criterion. The product is sold in a box at market and recommended levels of time and temperature to bake the cake in an oven are given on the box. It is known that the temperature indicator in different ovens might not be* 100% *accurate. Also, people do not always strictly follow the time recommendations for cake baking and hence frequently undercook or overcook the cake. So, the manufacturer is interested in finding a cake mix recipe that is robust to some minor changes in recommended levels for time (S_1) and temperature (S_2) given on the box. There are* 3 *factors involved in the product recipe: flour (W_1), shortening (W_2) and egg powder (W_3). So, in total there are* 5 *factors,* 3 *recipe factors (W_1, W_2, W_3) and* 2 *environmental factors (S_1, S_2) and each factor will be used on two levels, say, high and low. The authors give three different design strategies for conducting the experiment. All of those strategies lead to the use of a split-plot type design but we mention here only one of those. First,* 8 *large batches of cake mix are made using* 8 *different settings of the recipe factors, then each of these large batches is divided into* 4 *sub-batches and these* 4 *sub-batches (for every large batch) are then processed under* 4 *different settings of environmental factors.*

In this design strategy, 3 *factors, flour (W_1), shortening (W_2) and egg powder (W_3), are applied to a large amount of experimental material (large batches of cake mix) and the other* 2 *factors are applied to a smaller amount of experimental material (sub-batches). So, this experimental set-up is a split-plot design where large batches of cake mix are whole-plots and factors*

applied to these experimental units are whole-plot factors while sub-batches are sub-plots and the factors applied to these sub-plots are sub-plot factors.

Example 2 (Bingham et al, 2004) *Consider another example from the food industry given by Bingham et al (2004) [4]. An experiment was conducted in a cheese-making factory. The objective of the experiment was to study some quality characteristics of cheese production. The cheese production process consists of two stages. At the first stage, the milk is processed into a batch of curds and at the second stage these curds are processed into the cheese. Experts identified* 9 *different factors that can affect the quality characteristics of the cheese production under study. Two of the factors, say* W_1 *and* W_2*, are thought to affect the production at the first stage while the remaining factors, say* S_1*,* S_2*,* S_3*,* S_4*,* S_5*,* S_6 *and* S_7*, affect the production at the second stage. It was recommended to run the experiment at two stages. At the first stage, a large amount of milk was processed in a tank under different settings of factor* W_1 *and* W_2*. Then the processed milk from a single tank was divided into several batches of curds and these batches were then processed into cheese under different settings of the remaining* 7 *factors. This was a case of a split-plot design where factors* W_1 *and* W_2 *were applied to a large amount of experimental material so these are the whole-plot factors while the remaining* 7 *factors were applied to a smaller amount of experimental material so these* 7 *factors are the sub-plot factors.*

Example 3 (Gilmour et al, 2000) *Gilmour et al (2000) [12] reported the freeze-dried coffee experiment. The objective of the experiment was to study the effect of five factors on preserving the volatile compounds when freeze drying the coffee. The five factors were: pressure* (W_1)*, solids content* (S_1)*, slab thickness* (S_2)*, temperature* (S_3) *and freezing rate* (S_4)*. Each factor was to be tested at* 3 *different levels. It was known that the process would be fairly variable between experimental runs but there would be more day to day variation. Available resources could allow a maximum of* 30 *experimental runs for the experiment and it was decided to run the experiment over* 6 *days and with* 5 *runs per day. However, it was not possible to execute all* 30 *experimental runs under complete randomization of all factor levels within the given time frame because the factor pressure* (W_1) *needed to be changed manually and it took a long time when changing from one level to another. In other words, pressure was a HTC factor. The experimenter wanted to fix a level of the pressure factor for a full day and then randomly run the*

five different level settings of ETC factors. So, the design chosen was a split-plot design where levels of the HTC factors were applied to days (large experimental units) and levels of ETC factors were applied to the runs (small experimental units) within each day. Hence, each day forms a whole-plot and pressure is a whole-plot factor while each run forms a sub-plot and the remaining 4 *factors are sub-plot factors.*

Example 4 (Jones & Goos, 2007) *Jones & Goos (2007)[18] report an experiment on Polypropylene. The experiment was run by four Belgian companies to investigate the effect of several factors on the adhesive properties of Polypropylene. The problem under study was the gas plasma treatment applied to the Polypropylene surface for glues and coatings to adhere well. The experimenters were mainly interested in finding economical plasma treatments that cause good adhesion properties of Polypropylene. Four factors related to the plasma treatment, namely gas flow rate* (S_1)*, the power* (S_2)*, the reaction time* (S_3) *and the type of gas* (S_4)*, were selected by the engineers. In addition to the factors that are related to the plasma treatment, seven additives in Polypropylene were also included in the experiment because the engineers strongly believed that some of those additives had an effect on adhesive properties of Polypropylene. Those additives were: ethylene propylene diene monomer (EPDM) rubber* (W_1)*, ethylene copolymer content of the rubber* (W_2)*, talc* (W_3)*, mica* (W_4)*, lubricant* (W_5)*, UV stabilizer* (W_6) *and ethylene vinyl acetate* (W_7)*. One of the four companies was responsible for producing batches of Polypropylene and these batches would then be further processed in the other three companies. For obtaining the required results, the experiment needed* 100 *runs. However, it was not possible for the company to produce* 100 *different large batches of Polypropylene to conduct* 100 *independent experimental runs because it was a labour intensive job. So, it was agreed that the company would produce* 20 *large batches of Polypropylene and these large batches would be processed under* 100 *different gas plasma treatments.*

The experiment consisted of two stages. At the first stage, large batches of polypropylene were produced with additives and each of these large batches was then used for multiple runs under different settings of gas plasma treatment. Since the 7 *additives were applied to large experimental units (batches of Polypropylene) and every large experimental unit was used for multiple runs, those factors were HTC factors and factors involved in runs were ETC factors. This experimental set-up exhibits a split-plot structure where each large*

batch of Polypropylene forms a whole-plot and the 7 *additives are whole-plot factors whereas each run on a large batch forms a sub-plot and the* 4 *factors involved at the run level are sub-plot factors.*

In the remaining part of this thesis, we will discuss RS split-plot designs and different methodologies and techniques already available in the literature for constructing RS split-plot designs. We also present a new design methodology for constructing D-optimal RS split-plot designs. In the next chapter, we will focus on the design and analysis of RS split-plot type experiments after introducing the RS methodology and RS model for split-plot designs. Then we will review different approaches available in the literature for estimating the variance components of a RS model, with a focus on the randomization based approach. In chapter 3, we review some of the design construction methodologies available in the literature for generating RS split-plot designs and discuss why there is a need for a new design methodology. In chapter 4, we present a new design methodology, and give two different computer search algorithms, for constructing a RS split-plot design. Then we use our algorithm to generate designs for some of the design problems already discussed in the literature and discuss the designs. In chapter 5, we give concluding remarks.

Chapter 2

Response Surface Split-Plot Designs

In this chapter, we focus on the use of split-plot designs in RS experiments. In section 1, we give a brief introduction to RS methodology, RS models and parameter estimation. Section 2 describes the situations for RS experiments that lead to the use of split-plot designs. We give the RS model for split-plot designs and briefly state the method for estimating the fixed effects of the model. In section 3, we review some of the techniques available in the literature for estimating the variance components of the RS model for a split-plot design. The analysis of a split-plot design as an incomplete block design and the estimation of the variance components is described in section 4.

2.1 Response Surface Methodology

In many scientific investigations, the engineers and scientists study a system or process where their interest lies in determining the relationship between the output and the input of that process or system. The output is often called the response and the input is usually called input variables or factors that can affect the response. Suppose that the true relation between the response and the input factors can be described as

$$y = g\left(X_1, X_2, ..., X_k\right) + \epsilon, \tag{2.1}$$

where y is the response, the X's are the factors of interest, usually measured on a continuous scale, g is a function of these factors and ϵ is a statistical

error term that represents other sources of error not captured by the function g. The function g is usually unknown and might be very complicated. So, the research problem is to approximate this true but unknown function through a linear regression model. Since, the relationship between the response and input factors can be shown graphically as a surface lying over the range of the input factors, it is also called a response surface study. A RS study is usually carried out for two reasons.

1. To approximate the true relationship, between the response and the input factors, through a regression model.

2. To find out the settings for input factors for which the response is optimal (maximum or minimum.)

RS methodology is a strategy that can effectively be used to achieve these objectives.

Response surface methodology is based on the idea that the true relation between a response and the levels of one or more factors can be approximated by a linear polynomial model through a Taylor series expansion of the true function. The idea was originally developed by Box & Wilson (1951)[8] and now has become an important part of industrial experiments. The RS methodology is a collection of statistical designs and optimizing techniques that can be effectively used to explore and optimize a product or process, within a region of interest, when the factors involved in the experiment are quantitative in nature. Sometimes it is convenient to transform the original factors in, usually dimensionless, coded forms. The true unknown function (2.1) then can be written as

$$y = g\left(x_1, x_2, ..., x_k\right) + \epsilon, \tag{2.2}$$

where $x_1, x_2, ..., x_k$ are coded variables. The experiment is run under different and pre-planned settings of factor levels and the response is measured for each level setting, also called an experimental run. This information (measured response) is then used to build an empirical model and this fitted model is further explored, through a series of experiments, for attaining the setting of factor levels where the response is optimum.

Most experimental designs used in the RS methodology are based on the assumption of complete randomization. This assumes that the levels of the

input factors are applied to the experimental units with complete randomization as described in chapter 1. The assumption of complete randomization also demands the re-setting of all factor levels for each and every experimental run even if the successive runs have the same settings for one or more factors. For example, suppose that we are randomizing the two levels of a factor, say oven temperature, along with some other factor levels. The two levels are $40°C$ (t_1) and $60°C$ (t_2). The randomized sequence for application is ($t_1t_2t_1t_1t_2t_2$....). Then, although the 3^{rd} and the 4^{th} experimental runs have the same setting for the oven temperature, still we need to re-adjust the temperature to the t_1 level for the 4^{th} experimental run otherwise the corresponding error terms will not be uncorrelated which is an important assumption in RS model fitting. Hence, under the assumption of complete randomization the true relation is approximated by a linear polynomial model and perhaps the most often used approximation is a second order model.

For example, suppose that we are studying a process with two input factors (in coded form) and one response. We wish to determine the relationship between the input factors and the response. Different level settings of input variables and corresponding responses are given in table 2.1. Further suppose

Table 2.1: Data set for the fictitious example

x_1	x_2	y
-1	-1	46.5
-1	1	45.5
1	-1	57.5
1	1	50.5
0	0	50.0
0	0	50.0
1.414	0	55.7
-1.414	0	44.4
0	1.414	47.2
0	-1.414	52.8

that it is known, from previous experience, that the true relation can be well approximated by a second order polynomial model. Then a second order polynomial model, for our example, with two input factors can be written as

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 + \epsilon, \tag{2.3}$$

where the β's are model parameters and need to be estimated. Similarly, for f input factors, the equation (2.3) can be generalized as

$$y = \beta_0 + \sum_{i=1}^{f} \beta_i x_i + \sum_{i=1}^{f} \beta_{ii} x_i^2 + \sum_{i=1}^{f-1} \sum_{j=i+1}^{f} \beta_{ij} x_i x_j + \epsilon. \tag{2.4}$$

For computational ease, it is often convenient to write the model in matrix form. For our example, we can write the model (2.3) as

$$\mathbf{y} = \mathbf{X}\boldsymbol{\beta} + \boldsymbol{\epsilon} \tag{2.5}$$

where the $\mathbf{y}$ is a vector of responses (column 3 of table 2.1), $\boldsymbol{\epsilon}$ is a vector of errors, $\boldsymbol{\beta} = [\beta_0, \beta_1, \beta_2, \beta_{11}, \beta_{22}, \beta_{12}]'$ and $\mathbf{X} = [\mathbf{1}_n \quad \mathbf{x}_1 \quad \mathbf{x}_2 \quad \mathbf{x}_1^2 \quad \mathbf{x}_2^2 \quad \mathbf{x}_1\mathbf{x}_2]$, where $\mathbf{1}_n$ is a column vector of 1's of dimension n, n is the total number of experimental runs and $\mathbf{x}_1$ and $\mathbf{x}_2$ are the first and second columns of table 2.1 respectively, $\mathbf{x}_1^2$ is the vector whose l^{th} element is the square of the l^{th} elements of $\mathbf{x}_1$, $\mathbf{x}_2^2$ is the vector whose l^{th} element is the square of the l^{th} elements of $\mathbf{x}_2$ and $\mathbf{x}_1\mathbf{x}_2$ is the vector whose l^{th} elements is the product of the l^{th} elements of $\mathbf{x}_1$ and $\mathbf{x}_2$. $\mathbf{X}$ is also called the model matrix and is always known. Then the general second order polynomial model given by equation (2.4) can also be expressed in matrix form as (2.5)where $\mathbf{y}$ is an $(n \times 1)$ vector of responses, $\mathbf{X}$ is a $(n \times p)$ known design matrix, $\boldsymbol{\beta}$ is a $(p \times 1)$ vector of unknown model parameters and $\boldsymbol{\epsilon}$ is an $(n \times 1)$ vector of errors. Under complete randomization, it is typically assumed that $\boldsymbol{\epsilon} \sim N(\mathbf{0}, \sigma^2\mathbf{I}_n)$, where $\mathbf{I}_n$ is an identity matrix of order n and $\sigma^2 \geq 0$.

The model parameters are then estimated using the ordinary least squares method. If we write $\hat{\boldsymbol{\beta}}_{OLS}$ as the OLS estimator of $\boldsymbol{\beta}$ then, if $\mathbf{X}$ has a full column rank,

$$\hat{\boldsymbol{\beta}}_{OLS} = (\mathbf{X}'\mathbf{X})^{-1}\mathbf{X}'\mathbf{y}, \tag{2.6}$$

and

$$\mathbf{V}(\hat{\boldsymbol{\beta}}_{OLS}) = (\mathbf{X}'\mathbf{X})^{-\mathbf{1}}\sigma^2, \tag{2.7}$$

where $\mathbf{V}$ stands for the variance-covariance matrix. More details about RS Methodology can be found in Box & Draper (2007)[6] and Myers et al (2009)[25].

2.2 RS Model for a Split-plot Design

For a split-plot design, with w_p whole plots, the RS model can be written as

$$\mathbf{y} = \mathbf{X}\boldsymbol{\beta} + \mathbf{Z}\boldsymbol{\gamma} + \boldsymbol{\epsilon}, \tag{2.8}$$

where $\mathbf{Z}$ is a $n \times w_p$ block incidence matrix of the form

$$\mathbf{Z} = \begin{pmatrix} \mathbf{1}_k & \mathbf{0} & \cdots & \mathbf{0} \\ \mathbf{0} & \mathbf{1}_k & \cdots & \mathbf{0} \\ \vdots & \vdots & \ddots & \vdots \\ \mathbf{0} & \mathbf{0} & \cdots & \mathbf{1}_k \end{pmatrix},$$

with (i, j) element 1 if the i^{th} experimental run is in the j^{th} whole-plot and 0 otherwise, k is the whole-plot size, $\boldsymbol{\gamma}$ is a $(w_p \times 1)$ vector of whole-plot errors and $\boldsymbol{\epsilon}$ is an $(n \times 1)$ vector of sub-plot errors. We assume $\boldsymbol{\epsilon} \sim N(0, \sigma^2_\epsilon \mathbf{I}_n)$, $\boldsymbol{\gamma} \sim N(0, \sigma^2_\gamma \mathbf{I}_{w_p})$, $cov(\epsilon_i, \gamma_j) = 0$ for $i = 1, 2, ..., n$ and $j = 1, 2, ..., w_p$ and the remaining terms have same meanings as in (2.5). The covariance matrix $\boldsymbol{\Sigma}$ of the response vector $\mathbf{y}$ is of the form

$$\mathbf{V}(\mathbf{y}) = \boldsymbol{\Sigma} = \sigma^2_\gamma \mathbf{Z}\mathbf{Z}' + \sigma^2_\epsilon \mathbf{I}_n. \tag{2.9}$$

The error variances (σ^2_ϵ, and σ^2_γ) are also called the variance components of the model.

For a completely randomized design the model parameters are estimated using OLS but it is no longer an appropriate estimation method for analysing a split-plot design. Instead we use the generalized least squares method for estimating $\boldsymbol{\beta}$ which is

$$\hat{\boldsymbol{\beta}}_{GLS} = (\mathbf{X}'\boldsymbol{\Sigma}^{-1}\mathbf{X})^{-1}\mathbf{X}'\boldsymbol{\Sigma}^{-1}\mathbf{y}, \tag{2.10}$$

and the variance-covariance matrix is given as

$$\mathbf{V}(\hat{\boldsymbol{\beta}}_{GLS}) = (\mathbf{X}'\boldsymbol{\Sigma}^{-1}\mathbf{X})^{-1}. \tag{2.11}$$

Usually the variance components (σ_γ^2 and σ_ϵ^2) are not known and therefore cannot be used directly in (2.10) and (2.11). So these variance components are first estimated and then these estimates are used in expressions (2.10) and (2.11). In that case

$$\hat{\boldsymbol{\beta}}_{FGLS} = (\mathbf{X}'\hat{\boldsymbol{\Sigma}}^{-1}\mathbf{X})^{-1}\mathbf{X}'\hat{\boldsymbol{\Sigma}}^{-1}\mathbf{y}. \tag{2.12}$$

For more details on RS methods for split-plot designs see Letsinger et al (1996)[20].

2.3 Estimation Techniques

The variance components are used in estimating the model parameters of RS models and also in drawing inferences about these parameters. So the estimates of the variance components should be as close to the true values as possible. Different techniques are available for estimating these variance components, we mention some of these.

Letsinger et al (1996)[20] suggested the use of residual maximum likelihood (REML) for estimating the variance components of a RS model for a split-plot design. After that, the use of REML for estimating the variance components has become widely accepted. But different authors have pointed out several issues related to this method for estimating the variance components. For example Gilmour & Goos (2009)[11] discussed the Freeze-Dried Coffee experiment that was originally reported by Gilmour et al (2000)[12] and was also described in Example 3 of section 1.3. The original design suggested by the authors is given in table 2.2. Gilmour & Goos (2009)[11] showed that for this design and data, the REML estimate of the whole-plot error variance turns out to be 0. The authors analysed the data given in table 2.2 using several computer based packages for statistical analysis and reported that all the packages estimated the whole-plot error variance to be 0. They also discussed the implication of different methods for determining the degrees of freedom for estimating the whole-plot variance and reported that different packages used different degrees of freedom for testing the parameter effects.

While explaining, the authors said that for a non-orthogonal split-plot design whole-plot totals contain some information about the sub-plot model

Table 2.2: The design given by Gilmour et al (2000) for the Freeze-Dried Coffee experiment. Here **y** represents the response vector.

whole plot	W_1	S_1	S_2	S_3	S_4	**y**	whole plot	W_1	S_1	S_2	S_3	S_4	**y**
1	1	0	0	0	1	66.0	4	1	0	0	-1	0	66.9
1	1	0	0	1	0	66.1	4	1	1	0	0	0	79.2
1	1	-1	0	0	0	57.8	4	1	0	0	0	-1	65.2
1	1	0	0	0	0	66.0	4	1	0	-1	0	0	73.8
1	1	0	1	0	0	51.9	4	1	0	0	0	0	67.9
2	0	0	0	0	0	70.9	5	-1	0	0	0	0	69.2
2	0	-1	1	-1	1	56.8	5	-1	1	1	-1	1	85.4
2	0	1	1	1	-1	62.4	5	-1	1	-1	1	-1	74.3
2	0	1	-1	-1	-1	83.6	5	-1	-1	1	1	-1	50.4
2	0	-1	-1	1	1	65.2	5	-1	-1	-1	-1	1	60.3
3	-1	0	0	0	0	71.4	6	0	1	-1	1	1	89.2
3	-1	1	1	1	1	97.9	6	0	0	0	0	0	68.5
3	-1	-1	1	-1	-1	54.9	6	0	1	1	-1	-1	75.6
3	-1	-1	-1	1	-1	61.7	6	0	-1	1	1	1	56.5
3	-1	1	-1	-1	1	80.4	6	0	-1	-1	-1	-1	68.4

parameters. This information uses some of the degrees of freedom at the whole-plot level and even sometimes, especially if there are only a few whole-plots, leaves 0 degrees of freedom for estimating the whole-plot error variance although some packages show that there are still some degrees of freedom available. The authors concluded that since REML is based on asymptotic results, it might not work well for small experiments. In this situation, the authors suggested the use of a Bayesian approach where some prior beliefs are made about the variance components. If the data contains enough information, the Bayesian approach might give similar results to REML and prior beliefs might be overruled. But if the data do not contain enough information, the resulting estimates of the variance components would heavily depend on the prior beliefs. However, one advantage of this approach is that there would not be any problem for determining the number of the related degrees of freedom.

2.4 Randomization Based Approach

Another method already available in the literature, for estimating the variance components, is called a randomization based approach. In this method, we ignore the treatment structure in a split-plot design, i.e. we ignore that some factors are HTC and the other factors are ETC, only to obtain the estimates of the variance components, and assume that the whole-plots formed by the HTC factors are blocks and sub-plots formed by the ETC factors are experimental units arranged in blocks. Then, in this situation, split-plot designs are simply incomplete block designs where treatments are applied to the experimental units arranged in blocks. The corresponding model is then the full treatment model and the ANOVA table gives the estimates of the variance components with related numbers of degrees of freedom. Suppose that we want to apply t treatments to w_p blocks each with k experimental units, where $t > k$ and treatment i is replicated r_i times, $(i = 1, 2, ..., t)$, such that $\sum_{i=1}^{t} r_i = n$. Then, following Hinkelmann & Kempthorne (2005, p.329)[16], we can write our model for a general incomplete block design (IBD) as

$$y_{ijl} = \mu + \tau_i + \gamma_j + \epsilon_{ijl}, \tag{2.13}$$

for $(i = 1, 2, ..., t)$, $(j = 1, 2, ..., w_p)$ and $(l = 0, 1, 2, ..., n_{ij})$. Where y_{ijl} is the response at the l^{th} experimental unit that receives the treatment i in the

j^{th} block, μ is the overall mean, τ_i is the effect of the i^{th} treatment, γ_j is the random effect of the j^{th} block, ϵ_{ijl} is the error associated with y_{ijl} and n_{ij} denotes the number of times treatment i appears in block j, $E(\epsilon_{ijl}) = 0$, $E(\gamma_j) = 0$, $cov(\epsilon_{ijl}, \gamma_j) = 0$,

$$cov\left(\epsilon_{ijl}, \epsilon_{i'j'l'}\right) = \begin{cases} \sigma^2_\epsilon & \text{if } ijl = i'j'l'; \\ 0 & \text{otherwise,} \end{cases}$$

and

$$cov\left(\gamma_j, \gamma_{j'}\right) = \begin{cases} \sigma^2_\gamma & \text{if } j = j'; \\ 0 & \text{otherwise.} \end{cases}$$

We write model (2.13) in matrix form as

$$\mathbf{y} = \mu\mathbf{1}_n + \mathbf{X}_\tau\boldsymbol{\tau} + \mathbf{Z}\boldsymbol{\gamma} + \boldsymbol{\epsilon}, \tag{2.14}$$

where $\mathbf{y}$ is an $(n \times 1)$ vector of the responses, $\mathbf{1}_n$ is an $(n \times 1)$ vector of 1's, $\mathbf{X}_\tau$ is an $(n \times t)$ treatment incidence matrix with i^{th} column having r_i unity elements and $n - r_i$ zero elements such that $\mathbf{x}_i'\mathbf{x}_i = r_i$ and $\mathbf{x}_i'\mathbf{x}_j = 0$ where $\mathbf{x}_i$ and $\mathbf{x}_j$ is the i^{th} and j^{th} column of $\mathbf{X}_\tau$ and $i \neq j$, $\boldsymbol{\tau}$ is a $(t \times 1)$ vector of treatment effects, $\mathbf{Z}$ is an $(n \times w_p)$ block incidence matrix as described in section 2.2, $\boldsymbol{\gamma}$ is a $(w_p \times 1)$ vector of random block effects, and $\boldsymbol{\epsilon}$ is an $(n \times 1)$ vector of errors. Under complete randomization, we assume that $E\left(\boldsymbol{\epsilon}\right) = \mathbf{0}$, $\mathbf{V}\left(\boldsymbol{\epsilon}\right) = \sigma^2_\epsilon\mathbf{I}_n$, $E\left(\boldsymbol{\gamma}\right) = \mathbf{0}$, $\mathbf{V}\left(\boldsymbol{\gamma}\right) = \sigma^2_\gamma\mathbf{I}_{w_p}$. Then the model given by (2.14) is a full treatment model and the variance components of this model can be estimated by, following Hinklemann & Kempthorne (2005)[16], Yates' procedure. This method suggest the fitting of the full treatment model assuming fixed block effects then the block error variance can be estimated using extra sums of squares for blocks given the treatments. Since the estimates of the variance components taken from the full treatment model depend on the true replication of treatments, these are also called the pure error estimates of the variance components. These estimates of the variance components can then be plugged in (2.12) to estimate the fixed effects of the RS model. Furthermore these pure error estimates can also be used to check the lack-of-fit of a RS model. In the next section we explain how the pure error estimates of the variance components can be obtained by using a full treatment model for a split-plot design.

The analysis of an IBD is performed at two stages. At the first stage, the analysis is performed using the standard ordinary least squares method

Table 2.3: ANOVA Table for Model (2.14)

Source of Variation	Degrees of freedom	Sums of Squares
$\mathbf{X}_\tau \vert \mathbf{1}_n$	$t-1$	$\mathbf{T}'\mathbf{R}^{-1}\mathbf{T} - \frac{G^2}{n}$
$\mathbf{Z} \vert \mathbf{1}_n\mathbf{X}_\tau$	$rank(\mathbb{C}_2) = d$	$\hat{\boldsymbol{\gamma}}'\mathbb{Q}_2$
Residual	$n-t-d$	by difference
total	$n-1$	$\mathbf{y}'\mathbf{y}$-$\frac{G^2}{n}$

for estimating the fixed effects of treatments and blocks. This is called the Intra-Block analysis. At the second stage, the block effects are assumed to be random and analysis is performed mainly on the basis of block totals rather than of single observations. This is called the Inter-Block analysis. Here we describe these two analyses, as given by Hinklemann & Kempthorne (2005, Ch.1)[16].

2.4.1 The Intra-Block Analysis

The model for a general incomplete block design is given by (2.14). If we write $\mathbf{W} = (\mathbf{1_n} \quad \mathbf{X}_\tau \quad \mathbf{Z})$, and $\boldsymbol{\Theta}' = (\mu \quad \boldsymbol{\tau}' \quad \boldsymbol{\gamma}')$, then

$$\mathbf{y} = \mathbf{W}\boldsymbol{\Theta} + \boldsymbol{\epsilon}. \tag{2.15}$$

The normal equations for model 2.15 can be written as

$$(\mathbf{W}'\mathbf{W})\,\boldsymbol{\Theta} = \mathbf{W}'\mathbf{y}, \tag{2.16}$$

These equations are then solved to obtain the estimates for treatment effects and block effects. Then the ANOVA table based on the model (2.14) is given in table 2.3, where

$$\begin{aligned} \mathbb{C}_2 &= \mathbf{K} - \mathbf{N}'\mathbf{R}^{-1}\mathbf{N}, \\ \mathbb{Q}_2 &= \mathbf{B} - \mathbf{N}'\mathbf{R}^{-1}\mathbf{T}, \\ \mathbb{C}_2\hat{\boldsymbol{\gamma}} &= \mathbb{Q}_2, \end{aligned} \tag{2.17}$$

and

$$\hat{\boldsymbol{\gamma}} = \mathbb{C}_2^{-}\mathbb{Q}_2.$$

Where $\mathbf{N}$ is a design incidence matrix with element i, j equal to the number of times treatment i is replicated in block j, $\mathbf{T} = \mathbf{X}_\tau{}'\mathbf{y} = [T_1 T_2 ... T_t]'$

and T_i represents i^{th} treatment total, $\mathbf{R} = diag[r_1 \quad r_2 \quad ... \quad r_t]$, $\mathbf{B} = \mathbf{Z}'\mathbf{y} = [B_1 B_2 ... B_{w_p}]'$ and B_j is the j^{th} block total, $\mathbf{K} = k\mathbf{I}$, $\mathbb{C}_2^-$ is a generalized inverse for $\mathbb{C}_2$, $G = \mathbf{y}'\mathbf{1}$ and d in the ANOVA table 2.3 is defined as rank($\mathbb{C}_2$).

The sum of squares for the blocks given in the ANOVA table 2.3 is used to estimate the block error variance with the related degrees of freedom given as rank($\mathbb{C}_2$).

2.4.2 The Inter-Block Analysis

The Inter-Block analysis of IBD's is based on the argument that the block totals also provide some information about the treatment comparisons. For the Inter-Block analysis, consider the model (2.14)

$$\mathbf{y} = \mu\mathbf{1}_n + \mathbf{X}_\tau\boldsymbol{\tau} + \mathbf{Z}\boldsymbol{\gamma} + \boldsymbol{\epsilon}.$$

Now the $\boldsymbol{\gamma}$ is assumed to be a vector of random effects such that $E(\boldsymbol{\gamma}) = \mathbf{0}$, $\mathbf{V}(\boldsymbol{\gamma}) = \sigma_\gamma^2\mathbf{I}$ and $\boldsymbol{\gamma}$ and $\boldsymbol{\epsilon}$ are uncorrelated. Since the Inter-Block analysis deals with the block totals, pre-multiplying equation (2.14) by $\mathbf{Z}'$ gives

$$\mathbf{Z}'\mathbf{y} = k\mu\mathbf{1}_{w_p} + \mathbf{N}'\boldsymbol{\tau} + k\mathbf{I}\boldsymbol{\gamma} + \mathbf{Z}'\boldsymbol{\epsilon}. \tag{2.18}$$

That gives

$$E\left(\mathbf{Z}'\mathbf{y}\right) = k\mu\mathbf{1}_{w_p} + \mathbf{N}'\boldsymbol{\tau}, \tag{2.19}$$

$$\begin{aligned} \mathbf{V}\left(\mathbf{Z}'\mathbf{y}\right) &= k^2\sigma_\gamma^2\mathbf{I} + k\sigma_\epsilon^2\mathbf{I} && (2.20)\\ &= k\sigma_\epsilon^2\left(\mathbf{I} + k\frac{\sigma_\gamma^2}{\sigma_\epsilon^2}\mathbf{I}\right) && (2.21)\\ &= \mathbf{L}\sigma_\epsilon^2, && (2.22) \end{aligned}$$

where $\mathbf{L} = diag\left(k(1 + k\frac{\sigma_\gamma^2}{\sigma_\epsilon^2})\right) = diag(l)$. Then we use

$$\mathbf{z} = \mathbf{L}^{-1/2}\mathbf{Z}'\mathbf{y}, \tag{2.23}$$

as our response vector because

$$\mathbf{V}(\mathbf{z}) = \sigma_\epsilon^2\mathbf{I}, \tag{2.24}$$

that satisfies the condition for using the ordinary least squares method. Now from equation (2.19) we get

$$E(\mathbf{z}) \quad = \mathbf{L}^{-1/2}\left(k\mu\mathbf{1}_k + \mathbf{N}'\boldsymbol{\tau}\right) \quad = \mathbf{L}^{-1/2}\begin{pmatrix}\mathbf{k} & \mathbf{N}'\end{pmatrix}\begin{bmatrix}\mu \\ \boldsymbol{\tau}\end{bmatrix}, \tag{2.25}$$

where $\mathbf{k} = k\mathbf{1}_n$. Then the normal equations are

$$\begin{bmatrix}\mathbf{k}' \\ \mathbf{N}\end{bmatrix}\mathbf{L}^{-1}\begin{pmatrix}\mathbf{k} & \mathbf{N}'\end{pmatrix}\begin{bmatrix}\tilde{\mu} \\ \tilde{\boldsymbol{\tau}}\end{bmatrix} = \begin{bmatrix}\mathbf{k}' \\ \mathbf{N}\end{bmatrix}\mathbf{L}^{-1}\mathbf{Z}'\mathbf{y}. \tag{2.26}$$

Hinkelmann & Kempthorne (2005)[16] noted that, for $w_p < t$, which is a normal case for RS split-plot designs, the rank of the coefficient matrix would be less than t so not all treatment contrasts would be estimable using block totals. The authors suggested that the Intra and the Inter block information can be combined additively into a single analysis to get the best information about the treatment effects. This analysis is called the Combined analysis.

2.4.3 The Combined Analysis

For the Combined analysis, we will use the same model as given by (2.14) and with same assumptions i.e. we assume random block effects as we assume for split-plot designs. Then

$$E(\mathbf{y}) = \mu\mathbf{1}_n + \mathbf{X}_\tau\boldsymbol{\tau}, \tag{2.27}$$

and

$$\boldsymbol{\Sigma} = \mathbf{V}(\mathbf{y}) \quad = \mathbf{Z}\mathbf{Z}'\sigma_\gamma^2 + \sigma_\epsilon^2\mathbf{I} \quad = \text{diag}(\mathbf{V}_1, \mathbf{V}_2, ..., \mathbf{V}_w)\sigma_\epsilon^2, \tag{2.28}$$

where $\mathbf{V}_i = \sigma^2\left(\mathbf{I}_k + \dfrac{\sigma_\gamma^2}{\sigma_\epsilon^2}\mathbf{1}_k\mathbf{1}_k'\right)$. If we write $\tilde{\mathbf{W}} = \begin{pmatrix}\mathbf{1}_n & \mathbf{X}_\tau\end{pmatrix}$, and $\tilde{\boldsymbol{\Theta}}' = \begin{pmatrix}\mu & \boldsymbol{\tau}'\end{pmatrix}$, then the normal equations can be written, using the generalized least squares methods, as

$$\left(\tilde{\mathbf{W}}'\boldsymbol{\Sigma}^{-1}\tilde{\mathbf{W}}\right)\hat{\tilde{\boldsymbol{\Theta}}} = \tilde{\mathbf{W}}'\boldsymbol{\Sigma}^{-1}\mathbf{y}, \tag{2.29}$$

where $\boldsymbol{\Sigma}^{-1} = \text{diag}\ \left(\mathbf{V}_1^{-1}, \mathbf{V}_2^{-1}, ..., \mathbf{V}_w^{-1}\right)$ and $\mathbf{V}_i^{-1} = \sigma_\epsilon^{-2}\left(\mathbf{I}_k - \dfrac{\sigma_\gamma^2}{\sigma_\epsilon^2 + k\sigma_\gamma^2}\mathbf{1}_k\mathbf{1}_k'\right) =$ $\sigma_\epsilon^{-2}\left(\mathbf{I}_k - \mathbf{c}\mathbf{1}_k\mathbf{1}_k'\right)$, and $\mathbf{c} = \dfrac{\sigma_\gamma^2}{\sigma_\epsilon^2 + k\sigma_\gamma^2}$. Then the authors showed that the coefficient matrix for equation (2.29) does not have a full rank and the easiest

Table 2.4: ANOVA Table for Model 2.14

Source	E(SS)
$\mathbf{Z}\|\mathbf{1}_n\mathbf{X}_\tau$	$d\sigma_\epsilon^2 + \left(n - \sum_{ij} \frac{n_{ij}^2}{r_i}\right)\sigma_\gamma^2$
Residual	$(n-t-d)\sigma_\epsilon^2$

way to solve the normal equation is to put $\mu = 0$. So, the normal equations reduce to

$$\left(\mathbf{X}\prime_\tau \mathbf{\Sigma}^{-1}\mathbf{X}_\tau\right)\hat{\hat{\tau}} = \mathbf{X}'_\tau\mathbf{\Sigma}^{-1}\mathbf{y}. \tag{2.30}$$

Then the equation (2.30) can be also be written as

$$\left[\mathbf{R} - \mathbf{N}\mathbf{K}^{-1}\mathbf{N}' + \mathbf{N}\mathbf{L}^{-1}\mathbf{N}'\right]\hat{\hat{\tau}} = \mathbf{T} - \mathbf{N}\mathbf{K}^{-1}\mathbf{B} + \mathbf{N}\mathbf{L}^{-1}\mathbf{B}, \tag{2.31}$$

which is a additive combination of Intra and Inter Block normal equations.

2.4.4 Yates' Procedure

The variance components (σ_γ^2) and (σ_ϵ^2) in equation (2.30) are usually unknown and therefore need to be estimated. These estimated values of the variance components are then used to estimate the treatment effects. One method for estimating the variance components is called Yates Procedure. For estimating the variance components using Yates Procedure, we will fit the model (2.14) without assuming that block effects are random. We will compute the sums of squares for treatments given the mean and for the blocks given the mean and the treatments. Then we will find the expected values of the mean squares assuming that the block effects are random. Thus σ_γ^2 is estimated by the expected mean square for blocks given the mean and treatments and σ_ϵ^2 is estimated by the expected residual mean square. Expected sums of squares for the model (2.14) assuming random block effects (taken from Hinkelmann & Kempthorne (2005)[16])are given in table 2.4.

From the table we get

$$\mathrm{E}\left(\text{MS Residual}\right) = \sigma_\epsilon^2. \tag{2.32}$$

So

$$\hat{\sigma}_\epsilon^2 = \text{MS Residual}. \tag{2.33}$$

$$E\left(\text{MS}\left[\mathbf{Z}|\mathbf{1}_n\mathbf{X}_\tau\right]\right) = \sigma^2_\epsilon + \frac{(n - \sum_{ij}\frac{n^2_{ij}}{r_i})}{d}\sigma^2_\gamma$$
$$= \text{E (MS Residual)} + \frac{(n - \sum_{ij}\frac{n^2_{ij}}{r_i})}{d}\sigma^2_\gamma.$$

or

$$\hat{\sigma}^2_\gamma = \frac{d}{\left(n - \sum_{ij}\frac{n^2_{ij}}{r_i}\right)}\left[\text{MS}\left(\mathbf{Z}|\mathbf{1}_n\mathbf{X}_\tau\right) - \hat{\sigma^2_\epsilon}\right]. \tag{2.34}$$

Thus (2.33) and (2.34) respectively give unbiased estimators of σ^2_ϵ and σ^2_γ.

If we ignore the treatment structure in a split-plot design and consider it as a general incomplete block design where whole-plots serve a blocks then the corresponding treatment model and model assumptions will be the same as given for the combined analysis of a general incomplete block design. Hence, using the Yates' procedure we can estimate the variance components from the treatment model and then these estimates can be used in the response surface model.

We know, from table 2.3, that the numbers of degrees of freedom related to the sub-plot and the whole-plot error variances are $n - t - d$ and d respectively, where $d = \text{rank}(\mathbb{C}_2)$. So the matrix $\mathbb{C}_2$ plays an important role for determining the numbers of degrees of freedom for estimating the pure error variance components. The rank of the matrix $\mathbb{C}_2$ can be determined by checking if the design is connected or not. For a connected design $\text{rank}(\mathbb{C}_2) = w_p - 1$ and for a disconnected design $\text{rank}(\mathbb{C}_2) < w_p - 1$. We will use the concept of a connected design to prove the theoretical results for our new design methodology given in chapter 4. A connected design can be defined as: two treatments are said to be connected if we can move from one to another through a chain consisting alternately of treatments and blocks such that every treatment is contained in the adjacent block. A design is said to be connected if all the treatments are connected, otherwise it is called disconnected. We know, From equation (2.17), that the matrix $\mathbb{C}_2$ depends on the design incidence matrix $\mathbf{N}$. So, whether a design is connected or not can also be checked by the design incidence matrix $\mathbf{N}$. If two non-zero elements of matrix $\mathbf{N}$ can be connected through vertical and horizontal lines such that the vertices are at non-zero elements then the two treatments are

connected. The design is connected if a given treatment is connected to all other treatments. All the information given in this paragraph can be seen in Hinkelmann & Kempthorne (2005, p.14-16)[16].

The information matrix, $\mathbb{C}_2$, and the concept of a connected design plays an important role in our algorithm, given in chapter 4. We use the concept of a connected design in section 4.4 to prove the theoretical results for the new design methodology. In our algorithms, given in sections 4.5 and 4.6, we construct matrix $\mathbb{C}_2$ and calculate its rank to determine the available numbers of degrees of freedom for estimating the pure error variance components.

Chapter 3

Design Construction Methodologies

In this chapter we will look at some of the methodologies already available in the literature for constructing RS split-plot designs. We will briefly explain these methods giving some examples. In section 1, we discuss equivalent estimation designs. Section 2 describes the methodology for generating D-optimal RS split-plot designs. The methodology for constructing D-efficient equivalent estimation designs is described in section 3. In section 4, we describe a stratum-by-stratum construction method with an example.

3.1 Equivalent Estimation Designs

As we have described earlier, the analysis of a split-plot design using OLS can be misleading, instead it is recommended to use GLS estimates of model parameters. However, Letsinger et al (1996)[20] proved that for first order split-plot designs OLS estimates can be equivalent to GLS estimates if every whole-plot receives the same combinations of the sub-plot factor levels. Later Vining et al (2005)[28] and Parker et al (2007a) [26] presented design methodologies for constructing second order split-plot designs that allow the equivalence of OLS and GLS estimates.

3.1.1 General Condition for Equivalence of OLS and GLS

A necessary and sufficient condition for the equivalence of OLS and GLS estimates (McElroy, 1967 [23]) is that there exists a nonsingular matrix $\mathbf{F}$ such that

$$\mathbf{XF} = \mathbf{\Sigma X}, \tag{3.1}$$

where $\mathbf{X}$ and $\mathbf{\Sigma}$ are the same as defined earlier. Parker et al (2007) developed a general condition, valid only for the split-plot designs, for the equivalence of OLS and GLS estimates. Here, we show how they derived that condition. Putting equation (2.9) in (3.1) and pre-multiplying by $(\mathbf{X'X})^{-1}\mathbf{X'}$ we get

$$\mathbf{F} = \mathbf{Q}\sigma_\gamma^2 + \mathbf{I}\sigma_\epsilon^2, \tag{3.2}$$

where

$$\mathbf{Q} = (\mathbf{X'X})^{-1}\mathbf{X'ZZ'X}. \tag{3.3}$$

Putting (3.2) in (3.1) we get

$$\mathbf{XQ} = \mathbf{ZZ'X}, \tag{3.4}$$

Since (3.4) depends only on the model matrix $\mathbf{X}$ and the block incidence matrix $\mathbf{Z}$, it is possible to numerically check the equivalence of OLS and GLS when generating a split-plot design using a computer based search algorithm. By writing the model matrix $\mathbf{X}$ as,

$$\mathbf{X} = \begin{pmatrix} \mathbf{1}_k & \mathbf{H}_{D1} & \mathbf{H}_{Q1} & \mathbf{E}_{D1} & \mathbf{E}_{Q1} \\ \mathbf{1}_k & \mathbf{H}_{D2} & \mathbf{H}_{Q2} & \mathbf{E}_{D2} & \mathbf{E}_{Q2} \\ \vdots & \vdots & \vdots & \vdots & \vdots \\ \mathbf{1}_k & \mathbf{H}_{Dw_p} & \mathbf{H}_{Qw_p} & \mathbf{E}_{Dw_p} & \mathbf{E}_{Qw_P} \end{pmatrix},$$

where $\mathbf{1}_k$ is a unit vector of order k and represents the intercept term, $\mathbf{H}_{Di}$ contains all the columns that represent the main effects and two factor interactions of the HTC factors in the design matrix, $\mathbf{H}_{Qi}$ contains the columns that denote pure quadratic effects of the HTC factors, $\mathbf{E}_{Di}$ contains the columns that denote main effects and two factor interactions of the ETC factors and ETC and the HTC factors interactions and $\mathbf{E}_{Qi}$ contains the columns that denote the pure quadratic effects of the ETC factors, in whole-plot i. We assume w HTC and s ETC factors.

By considering a choice for the design matrix $\mathbf{X}$ for which

$$\mathbf{Q} = \begin{pmatrix} k & \mathbf{0}' & \mathbf{0}' & \mathbf{0}' & \mathbf{v}_0' \\ \mathbf{0} & k\mathbf{I}_D & \mathbf{0} & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & k\mathbf{I}_Q & \mathbf{0} & \mathbf{V}_w \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & \mathbf{0} & \mathbf{V}_s \end{pmatrix},$$

and by putting matrix $\mathbf{X}$, $\mathbf{Q}$ and $\mathbf{Z}$ in equation (3.4), the authors reduced the equivalence condition to,

$$\mathbf{1}\mathbf{1}'\mathbf{E}_{D_i} = \mathbf{0}, \tag{3.5}$$

and

$$\mathbf{1}\mathbf{1}'\mathbf{E}_{Q_i} = \mathbf{1}_n\mathbf{v}_0' + \mathbf{H}_{Q_i}\mathbf{V}_w + \mathbf{E}_{Q_i}\mathbf{V}_s, \tag{3.6}$$

for the design matrix $\mathbf{X}$. Here $\mathbf{I}_D$ and $\mathbf{I}_Q$ are identity matrices of order $(w \times w)$ and $\left(\left[w + \frac{w(w-1)}{2}\right] \times \left[w + \frac{w(w-1)}{2}\right]\right)$ respectively, $\mathbf{V}_s$ is a $(s \times s)$ matrix, $\mathbf{v}_0'$ is a vector of length s and $\mathbf{V}_w$ is a $(w \times s)$ matrix. The equation (3.5) and (3.6) are sufficient conditions for the equivalence estimation. The first condition, expressed by (3.5), requires an orthogonal split-plot design within each whole-plot so that that the column sums of the main effects and two factor interactions in the split-plot design should be 0 within each whole-plot. The second condition belongs to the pure quadratic effects of the split-plot design and it is relatively difficult to satisfy. Here we discuss two different techniques to construct equivalent estimation designs.

3.1.2 VKM Methodology

Vining et al (2005)[28] presented a design methodology that allows the modification of standard second order RS designs (e.g. Central Composite Designs (CCD's) and Box-Behnken Designs) to accommodate split-plot type experiments. They also developed the conditions for these designs that, if satisfied, allow the equivalence of OLS and GLS estimates of model parameters. Such designs are now called the equivalent estimation Designs. Following Parker et al (2007b)[26], we will call their construction method the VKM method. Here we briefly describe the VKM design methodology and conditions for the equivalence estimation.

To modify a CCD into a split-plot design, a completely randomized CCD is arranged in such a way that the levels of the HTC factors remain constant for consecutive runs forming whole-plots of the experiment while the levels of the ETC factors can be changed run to run forming the sub-plots within each whole-plot. Experimental runs can be added or subtracted within each whole-plot to achieve a constant whole-plot size. Then the experimental design is further modified to achieve the property of equivalent estimation. To construct equivalent estimation split-plot designs, they consider a choice for the design matrix $\mathbf{X}$ for which $\mathbf{v}_0' = \mathbf{0}$ and $\mathbf{V}_w = \mathbf{0}$ in $\mathbf{Q}$. Then the condition for the equivalence given by (3.6) becomes

$$\mathbf{1}\mathbf{1}'\mathbf{E}_{Q_i} = \mathbf{E}_{Q_i}\mathbf{V}_s \tag{3.7}$$

For a CCD, the authors defined $\mathbf{V}_s = \dfrac{k}{s}\mathbf{1}_s\mathbf{1}_s'$, when s is a multiple of 2, and showed that the equivalence condition given by (3.7) can be satisfied if the whole-plot size is constant and all the sub-plot axial runs are in a single whole-plot. So, based on (3.5) and (3.7), they stated three conditions to obtain the equivalence of OLS and GLS estimates;

1. Every whole-plot has the same number of sub-plots.
2. The column sums of the ETC factors' main effects and two factor interactions are 0 in each whole-plot.
3. The axial runs of the ETC factors are run in a single whole-plot.

The authors further suggested the augmentation of a few whole-plots, with all factors at the central level, to the design for obtaining pure error estimates of the variance components. An equivalent estimation design, with one HTC factor and two ETC factors, constructed by this approach, is given in table 3.1.

These designs have some interesting properties. One property of these designs is obviously the equivalence of OLS and GLS estimates of the model parameters. This equivalence is independent of the model and holds for sub-models as well. For example, an equivalent estimation design with 4 factors, the equivalence property will hold even if the model if fitted by using only 2 or 3 factors only. Since this strategy allows the equivalence of OLS and GLS estimates, it can be particularly useful for practitioners who do not have

Table 3.1: An equivalent estimation design for 1 HTC and 2 ETC factors constructed by the VKM methodology (Parker et al, 2007). Here α and α_2 are the axial distances for the ETC and the HTC factors respectively.

Whole plot	W_1	S_1	S_2	Whole plot	W_1	S_1	S_2
1	-1	-1	-1	4	α_2	0	0
1	-1	1	-1	4	α_2	0	0
1	-1	-1	1	4	α_2	0	0
1	-1	1	1	4	α_2	0	0
2	1	-1	-1	5	0	$-\alpha$	0
2	1	1	-1	5	0	α	0
2	1	-1	1	5	0	0	$-\alpha$
2	1	1	1	5	0	0	α
3	$-\alpha_2$	0	0	6	0	0	0
3	$-\alpha_2$	0	0	6	0	0	0
3	$-\alpha_2$	0	0	6	0	0	0
3	$-\alpha_2$	0	0	6	0	0	0

access to modern statistical packages that can provide GLS estimates of the model parameters using REML. But these designs are inefficient as pointed out by Goos (2006)[13]. For example, if we look at the third and fourth whole-plot of the design given in table 3.1, we can see that only a single treatment is being replicated in every experimental run in each whole-plot. This might not be the best use of the available resources. This becomes even clearer if the number of ETC factors is not a multiple of 2. Table 3.2 represents an equivalent Estimation design with one HTC and three ETC factors. The axial points of the experiment are run in whole-plots 3 to 7 and the last whole-plot contains the centre point runs only. That means, only 9 of the treatments are used for the last 48 experimental runs. It seems reasonable to point out here that although these designs allow the estimation of model parameters without estimating the variance components, to draw inferences about the model parameters one has to estimate the variance components because the variance-covariance matrix for the parameter estimates depends on $\mathbf{\Sigma}$.

Another property of this technique is that it gives pure-error estimates

Table 3.2: An equivalent estimation design for 1 HTC and 3 ETC factors constructed by VKM methodology. (Parker et al, 2007). Here α and α_2 is the axial distance for the ETC and the HTC factors respectively.

Whole plot	W_1	S_1	S_2	S_3	Whole plot	W_1	S_1	S_2	S_3
1	-1	1	-1	1	5	0	-α	0	0
1	-1	-1	1	1	5	0	α	0	0
1	-1	-1	-1	-1	5	0	-α	0	0
1	-1	1	1	-1	5	0	α	0	0
1	-1	-1	-1	1	5	0	-α	0	0
1	-1	1	1	1	5	0	α	0	0
1	-1	1	-1	-1	5	0	-α	0	0
1	-1	-1	1	-1	5	0	α	0	0
2	1	1	-1	1	6	0	0	-α	0
2	1	-1	1	1	6	0	0	α	0
2	1	-1	-1	-1	6	0	0	-α	0
2	1	1	1	-1	6	0	0	α	0
2	1	-1	-1	1	6	0	0	-α	0
2	1	1	1	1	6	0	0	α	0
2	1	1	-1	-1	6	0	0	-α	0
2	1	-1	1	-1	6	0	0	α	0
3	-α_2	0	0	0	7	0	0	0	-α
3	-α_2	0	0	0	7	0	0	0	α
3	-α_2	0	0	0	7	0	0	0	-α
3	-α_2	0	0	0	7	0	0	0	α
3	-α_2	0	0	0	7	0	0	0	-α
3	-α_2	0	0	0	7	0	0	0	α
3	-α_2	0	0	0	7	0	0	0	-α
3	-α_2	0	0	0	7	0	0	0	α
4	α_2	0	0	0	8	0	0	0	0
4	α_2	0	0	0	8	0	0	0	0
4	α_2	0	0	0	8	0	0	0	0
4	α_2	0	0	0	8	0	0	0	0
4	α_2	0	0	0	8	0	0	0	0
4	α_2	0	0	0	8	0	0	0	0
4	α_2	0	0	0	8	0	0	0	0
4	α_2	0	0	0	8	0	0	0	0

of the variance components. These estimates are obtained by adding some replicated whole-plots, with all factors at central level, to the original design. As the pure error estimates are based on several replications of only a single treatment, it might not be the best option to use. This technique gives $w_r - 1$ degrees of freedom for estimating the whole-plot error variance where w_r denotes the number of times a whole-plot with all central points is replicated. So, for every single degree of freedom, one would need to add k centre points (all factors at 0 level) in the experiment. For example, consider the design given in table 3.2. As we have discussed in the first chapter, some authors recommend to use at least 5 degrees of freedom for estimating the error variance. So, if we want to get 5 degrees of freedom for estimating the whole-plot variance, we would need to add 5 more whole-plots, each containing 8 centre points only, to the experiment. This would add 40 extra runs into the experiment given in table 3.2 and thus almost half of the experiment would be based on the replication of centre point runs only. Furthermore, although this technique gives freedom to choose the number of degrees of freedom for estimating the whole-plot error variance, it does not give a pre-specified number of degrees of freedom for estimating the sub-plot variance.

3.1.3 Minimum Whole-plot (MWP) Method

Parker et al (2007a)[26] presented another approach for constructing equivalent estimation split-plot designs. That technique is called the Minimum Whole-plot (MWP) method. This technique is also developed to modify a CCD to accommodate a split-plot type structure with a minimum number of whole-plots. For these designs, the number of whole-plots is equal to the number of combinations of the HTC factor levels in a completely randomized CCD. Again, a completely randomized CCD is arranged in such a way that the levels for the HTC factors remain constant for several consecutive runs while the levels of the ETC factors may change from run to run. This technique forms the whole-plots and sub-plots of the experiment. Experimental runs within each whole-plot can be added or subtracted for a constant whole-plot size. And the equivalence of OLS and GLS estimates is then achieved by further modification.

To achieve the equivalence property, the authors considered a choice for matrix $\mathbf{Q}$ as given before but with $\mathbf{V}_s = \mathbf{0}$. Then the equivalence condition

given by (3.6) becomes

$$\mathbf{1}\mathbf{1}'\mathbf{E}_{Q_i} = \mathbf{1}_n\mathbf{v}_0' + \mathbf{H}_{Q_i}\mathbf{V}_w. \tag{3.8}$$

The authors stated that in this case the equivalence condition will depend on the values for α and α_2, where α and α_2 denote the distance from the centre for the ETC factors and for the HTC factors respectively. This technique allows the inclusion of centre runs of the ETC factors within each whole-plot without damaging the property of equivalence estimation. An equivalent estimation design constructed by using this approach is given in table 3.3.

Table 3.3: An equivalent estimation design for 1 HTC and 2 ETC factors constructed by the MWP methodology. (Parker et al, 2007). Here α and α_2 are the axial distances for the ETC and the HTC factors respectively.

Whole plot	W_1	S_1	S_2	Whole plot	W_1	S_1	S_2
1	-1	-1	-1	4	α_2	0	0
1	-1	1	-1	4	α_2	0	0
1	-1	-1	1	4	α_2	0	0
1	-1	1	1	4	α_2	0	0
1	-1	0	0	4	α_2	0	0
2	1	-1	-1	5	0	$-\alpha$	0
2	1	1	-1	5	0	α	0
2	1	-1	1	5	0	0	$-\alpha$
2	1	1	1	5	0	0	α
2	1	0	0	5	0	0	0
3	$-\alpha_2$	0	0				
3	$-\alpha_2$	0	0				
3	$-\alpha_2$	0	0				
3	$-\alpha_2$	0	0				
3	$-\alpha_2$	0	0				

These designs are more or less similar to the designs described in the previous section except that these designs include centre points for the ETC factors within each whole-plot and need relatively fewer whole-plots mainly because this approach does not allow the inclusion of a whole-plot with all

factors at the centre level. This can be confirmed by comparing the two designs given in tables 3.1 and 3.3. Although, the MWP design has one fewer whole-plot than the VKM design, because it does not have a whole-plot with all factors at 0 level, it has one more experimental run as compared to those in the VKM design. So, as far as, the optimal use of the resources is concerned, this approach might not be the best option available. Also, unlike the VKM methodology, the equivalence property is model dependent and does not work if pure quadratic effects are removed from the model. Another difference is the estimation of the variance components. Since this approach is based on the minimum number of whole-plots concept, it rarely allows treatment replication between whole-plots. That means there will hardly be any degrees of freedom available for obtaining the pure error estimates for the whole-plot error variance. Although, it allows the inclusion of sub-plot centre runs within each whole-plot that can generate degrees of freedom for estimating the sub-plot pure error variance.

3.2 D-Optimal Designs

One approach for constructing a RS split-plot design is to use computer based search algorithms for generating a D-optimal design. Such designs are constructed by searching (within a region of interest) for the values of factor levels for which the determinant of the variance matrix of the parameter estimates is minimum. For a completely randomized design (CRD), the D-optimality criterion minimizes the determinant of the variance matrix and it is equivalent to maximizing $|\mathbf{X}'\mathbf{X}|$. The matrix $\mathbf{X}'\mathbf{X}$ for a CRD is also called the information matrix. For our thesis, we will use the D-optimality criterion for maximizing the determinant of the information matrix. If we represent a CRD by ξ_n, then its information matrix denoted by $M(\xi_n)$ can be expressed as

$$M(\xi_n) = \mathbf{X}'\mathbf{X}. \tag{3.9}$$

A D-optimal design is one for which $|M(\xi)|$ is maximum. The relative D-efficiency of a CRD, $\xi_{n,1}$ with respect to another CRD, $\xi_{n,2}$, can be computed as

$$D_{R.E} = \left[\frac{|M(\xi_{n,1})|}{|M(\xi_{n,2})|}\right]^{1/p}, \tag{3.10}$$

where p is the number of the model parameters.

For RS split-plot designs, the model parameters are estimated using GLS and the GLS estimator and its variance matrix are given in equation (2.10) and (2.11) respectively. So, a D-optimal RS split-plot design is for which $|\mathbf{X}'\boldsymbol{\Sigma}^{-1}\mathbf{X}|$ is maximum.

3.2.1 Constructing D-Optimal Designs

There are mainly two types of algorithm available in the literature for constructing a D-optimal design. The first type of algorithm is called sequential algorithms. As the name suggests, these algorithms construct a D-optimal design by either sequentially adding the candidate points to a starting design or by sequentially deleting the design points from a starting design. These procedures are also called forward and backward procedures. In a forward procedure, a starting design is generated randomly with n_0 experimental runs where $n_0 < n$. Then a D-optimal design with n experimental runs is obtained by sequentially selecting a design point from the candidate set of points that contributes the most in maximizing the determinant of the information matrix (a design point with the largest prediction variance) and then adding it into the starting design. However, for a single starting design, this approach may not necessarily end with a D-optimal design. Therefore, this forward procedure is repeated for several starting designs and the best design is then chosen for executing the experiment. The second procedure, also known as the backward procedure, starts with a random starting design with n_c experimental runs, where $n_c > n$ and a D-optimal design with n experimental runs is obtained by sequentially deleting the design points that contribute the least in maximizing the D-criterion value (the design points with the least prediction variance) from the starting design. Again this procedure is adopted for several starting designs and the best design with the highest optimality criterion value is selected.

A second type of algorithm is called the exchange algorithm. These algorithms are mainly used to improve a starting design with n experimental runs. A design obtained from using a forward or backward procedure can further be improved by using an exchange algorithm. Unlike forward or backward procedures, exchange algorithms construct a D-optimal design by making exchanges between the candidate set of points and a starting design with n experimental runs. The exchange algorithms can further be classified into point exchange algorithms and coordinate exchange algorithms.

Point exchange algorithms improve a starting design by exchanging the design points between the candidate set of points and the starting design. To illustrate, consider that we have a starting design with n experimental runs and we want to improve that design in order to get a D-optimal design. An exchange algorithm will exchange the first design point with the first candidate point and calculate the D-criterion value. Then the first design point will be exchanged with the second candidate point and the D-criterion value would be calculated. This procedure will continue until the algorithm exchanges the first design point with the last candidate point and calculate the D-criterion value. Similarly each and every design point is exchanged with all candidate points one by one and the best exchange that maximizes the D-criterion value is saved. If an increase in the D-criterion value is recorded then this exchange procedure is performed again until there is no further increase in the D-criterion value. Because the procedure allows the replication of the design points so the selection is made with replacement i.e. a design point selected in the design still remains available in the set of candidate points and can be selected again at some point. A D-optimal design is found by improving several starting designs. A second approach for an exchange algorithm, also known as the KL exchange algorithm, does not consider all possible exchanges between design points and candidate points. Instead only a set of K design points with smallest prediction variances are exchanged with a set of L candidate points with largest prediction variances.

Coordinate exchange algorithms are used to improve a random starting design by exchanging the coordinates (levels of a single factor) of the design points with all possible or available levels of that factor one by one. Again, for illustration, just consider that we have a starting design generated randomly with n experimental runs. Then the first coordinate of the first design point is replaced one by one with all the available levels of that factor, the D-criterion value is calculated and if there is any increase in the criterion value then this exchange is saved. Then replace the second coordinate of the first design point, calculate the D-criterion value and if there is any increase in the criterion value save that exchange. Similarly keep exchanging the coordinates of all the design points until the last coordinate of the last design point is exchanged. If at least one improvement is recorded during this exchange process, start the process again by exchanging the first coordinate of the first design point. This exchange process continues until there is no further increase in the D-criterion value. More details about sequential and exchange

algorithms can be found in Atkinson et al (2007, Ch.12)[2].

3.2.2 Exchange Algorithm for Generating D-optimal RS Split-plot Designs

Several papers have been published in recent years advocating the use of the D-optimal designs and presenting exchange algorithms for generating D-optimal RS split-plot designs. For a split-plot design the HTC factor levels need to be constant for several experimental runs while the ETC factor levels may change from run to run. This creates a difference when developing a search algorithm for generating a RS split-plot design although the basic technique is the same as described in the last section. Another issue is that for a RS split-plot design the D-optimality criterion depends on the model matrix $\mathbf{X}$ and the variance-covariance matrix $\mathbf{\Sigma}$. This requires that the experimenter must have determined the RS model to be fitted and should also have some knowledge about the values for the variance components. These values for the variance components can be obtained if some experiments were executed previously for the same research problem or by personal experience or judgement of the experimenter. As a result, it is quite possible to obtain different optimal designs for different values of the variance components.

Goos & Vandebroek (2001)[14] proposed a point exchange algorithm for generating a RS split-plot design with n experimental runs without imposing any restriction on the numbers of whole-plots or a fixed whole-plot size although the authors claimed that such conditions can easily be included in their algorithm. They proposed a search algorithm which takes into account the presence of the variance-covariance matrix $\mathbf{\Sigma}$ in the optimality criterion but it does not enforce the constant HTC factor levels for some consecutive runs. The authors showed that, for a split-plot design, the information matrix $\mathbf{M}$ can be written as

$$\mathbf{M} = \mathbf{X}'\mathbf{\Sigma}^{-1}\mathbf{X} = \frac{1}{\sigma_\epsilon^2}\left(\sum_{i=1}^{w_p}\sum_{j=1}^{k_i} f(w_i, s_{ij})f'(w_i, s_{ij}) - \sum_{i=1}^{w_p}\frac{\eta}{1+k_i\eta}(\mathbf{X}_i'\mathbf{1}_{k_i})(\mathbf{X}_i'\mathbf{1}_{k_i})'\right), \tag{3.11}$$

where k_i is the i^{th} whole-plot size, $\eta = \sigma_\gamma^2/\sigma_\epsilon^2$ is the ratio of the variance components, $\mathbf{X}_i$ is the model matrix that belongs to the whole-plot i, and $f'(w_i, s_{ij})$ represents the factor level settings at the j^{th} sub-plot of the i^{th}

whole-plot and the HTC factor level settings are expressed by w_i whereas the ETC factor level settings are expressed by s_{ij}. The expression in (3.11) makes it easier to update the information matrix after each exchange of the design points. Because this algorithm does not have any restriction on the number of whole-plots and sub-plots within each whole-plot, the generated design does not necessarily look like a split-plot design with the levels of the HTC factors constant for some consecutive runs. But different treatments having the same combinations of the HTC factor levels can then be grouped together to form the whole-plots of the experiment.

Later Goos & Vandebroek (2003)[15] gave another point exchange algorithm for generating a RS split-plot design with n experimental runs with given numbers of whole-plots and sub-plots within each whole-plot. The first stage of their algorithm is to generate a starting design. The starting design is generated by first randomly assigning the different settings, at least as many as the number of the pure whole-plot model parameters, of the HTC factors to the whole-plots. Then a starting design is generated by using a forward procedure i.e. first a random number of design points are selected at random from the set of candidate points and each of these design points is allocated to the whole-plots with the corresponding whole-plot factor settings and then the candidate points with the largest prediction variances are sequentially added to complete the design. The improvement is made using three different techniques. First by exchanging the design points with the candidate points that have the same whole-plot factor settings. Then design points are exchanged between whole-plots having same settings of the whole-plot factors. And finally improvement is made by exchanging the whole-plot factor settings between whole-plots. If an improvement is recorded, these exchange processes are repeated until there is no further improvement in the D-criterion value. A design generated by this approach is given in table 3.4.

Jones & Goos (2007)[18] presented a coordinate exchange algorithm for generating a RS split-plot design. An advantage of their approach is that it does not require a pre-specified set of candidate points instead it generates a starting design by randomly choosing the required levels for the HTC and the ETC factors. Since our algorithm is based on this approach, we will discuss this algorithm in more detail in the next chapter. Arnouts & Goos (2010)[1] gave mathematical formula for fast updating the determinant of the information matrix for coordinate exchange algorithm for generating split-

Table 3.4: A D-optimal design for 1 HTC and 4 ETC factors given by Goos & Vandebroek (2003). Here W represents the HTC factor and S_i denotes the i^{th} ETC factor.

Whole plot	W_1	S_1	S_2	S_3	S_4	Whole plot	W_1	S_1	S_2	S_3	S_4
1	-1	1	-1	-1	1	12	0	0	-1	1	1
1	-1	1	1	1	-1	12	0	1	1	1	-1
2	-1	-1	0	-1	-1	13	1	-1	-1	1	-1
2	-1	1	1	-1	1	13	1	1	1	-1	-1
3	-1	-1	1	1	1	14	1	-1	0	0	1
3	-1	1	1	-1	-1	14	1	1	1	1	1
4	-1	-1	-1	1	1	15	1	-1	-1	-1	-1
4	-1	1	-1	-1	-1	15	1	0	1	1	-1
5	-1	-1	-1	1	-1	16	1	-1	-1	1	1
5	-1	0	1	0	0	16	1	1	1	-1	1
6	-1	-1	1	-1	1	17	1	0	-1	-1	1
6	-1	1	-1	1	0	17	1	1	-1	1	0
7	-1	-1	-1	-1	1	18	1	-1	1	-1	-1
7	-1	-1	1	1	-1	18	1	1	0	0	1
8	-1	-1	1	-1	-1	19	1	-1	0	0	-1
8	-1	1	-1	1	-1	19	1	1	-1	-1	1
9	-1	-1	-1	0	0	20	1	-1	1	1	1
9	-1	1	0	1	1	20	1	1	-1	1	-1
10	0	0	0	-1	0	21	1	-1	1	-1	1
10	0	1	-1	0	1	21	1	1	-1	-1	-1
11	0	0	-1	0	-1						
11	0	1	0	-1	0						

plot designs. But none of the above mentioned algorithms for generating the D-optimal RS split-plot designs allows one to pre-specify the numbers of degrees of freedom for estimating the pure error variance components.

3.3 D-Optimal Equivalent Estimation Designs

The idea of D-optimal equivalent estimation design was developed by Parker et al (2007a)[26]. Since the equivalence condition given by equation 3.4 depends only on the model matrix and block incidence matrix, it is possible to numerically check if the condition is satisfied for a design. This makes it possible to use a computer based search algorithm for constructing such designs. Parker et al (2007a)[26] used the point exchange algorithm for generating D-optimal split-plot design proposed by Goos & Vandebroek (2003)[15] to generate D-optimal equivalent estimation designs including the condition for the equivalence in the algorithm. Later Macharia & Goos (2010)[21] presented a coordinate exchange algorithm for generating a RS split-plot design. Their algorithm is basically based on the Jones & Goos (2007)[18] algorithm but they add the equivalent estimation condition, described in (3.4), in their algorithm. So, with every coordinate exchange, their algorithm computes the D-optimality criterion value and checks if the equivalent condition is satisfied. If there is an increase in the criterion value the algorithm stores the new design as the optimal design and if the equivalence condition is also satisfied that this design is also stored as an equivalent estimation design. As a result, the algorithm gives two designs as output; one as the D-optimal design and the other as the D-efficient equivalent estimation design. The authors showed that for many design problems a D-optimal design is also an equivalent estimation design. A D-efficient equivalent estimation design given by Macharia & Goos (2010)[22] is given in table 3.5.

This approach joins two popular approaches for constructing RS split-plot designs such that the generated design is D-efficient and equivalent estimation. Although this approach gives equivalent estimation designs with optimal use of the available resources, to estimate the variance components and to draw inferences about the model parameters it rarely allows any degrees of freedom for obtaining the pure error estimates of the variance components.

Table 3.5: A D-efficient equivalent estimation design for 2 HTC and 1 ETC factor given by Macharia & Goos (2010)[22].

Whole plot	W_1	W_2	S_1
1	-1	-1	-1
1	-1	-1	1
2	-1	1	-1
2	-1	1	1
3	-1	0	0
3	-1	0	1
4	0	1	-1
4	0	1	0
5	1	-1	-1
5	1	-1	1
6	1	-1	-1
6	1	-1	1
7	1	1	-1
7	1	1	1

3.4 Stratum-by-Stratum Construction Method

Trinca & Gilmour (2001)[27] presented a methodology for constructing RS designs in the presence of HTC factors. Their methodology can be used to construct RS designs for more than two strata. In fact, their approach can be used for constructing RS designs with any number of strata. Such designs are called multi-stratum RS designs of which the split-plot design is a special case with two strata. The approach is based on choosing a treatment design at each stratum for the factors involved in that stratum and then transforming the treatment design into a block design where the units of the higher stratum serve as blocks. Then this block design is combined with the treatment design in the higher stratum to obtain the final design.

3.4.1 Design Construction Methodology

Let us suppose that the factors are classified into groups according to the level of difficulty for changing their level settings (for example, from the Hardest-to-Change to the Easiest-to-Change). Then we say the unit structure is $U_1/U_2/.../U_m$, where the Hardest-to-Change factors are applied in the highest stratum U_1 and the Easiest-to-Change factors are applied in the lowest stratum U_m. Let f_i denote the numbers of factors applied at stratum U_i. Then the design construction methodology can be described as:

1. Starting from the highest stratum, if $f_i > 0$ choose a treatment design for f_i factors applied at U_i $(i = 1, 2, ..., m)$. The treatment design can be a regular design e.g. a factorial design, central composite design, etc. This would help in obtaining good estimates of the coefficients of the factors applied in stratum U_i.

2. Arrange the treatment design for U_i $(i \geq 2)$ obtained in 1, in blocks, where units for the higher stratum U_{i-1} serve as blocks, and obtain a good block design. A computer search is usually required to obtain a good block design under a pre-specified design criterion. Initially the treatment design is randomly arranged in blocks and then this initial design is improved, in terms of the given design criterion, by interchanging the treatments between different blocks. The interchange of treatments between blocks continues until there is no improvement in the design. This improvement process is repeated for several initial designs and the best available design is chosen.

3. Combine the block design for stratum U_i, obtained in 2, with the treatment design for the factors applied in stratum U_{i-1}. Again, a computer search is used to obtain a good combined design that ensures the good estimation of the coefficients of the interaction terms for the factors applied in stratum U_i and U_{i-1}. The combined design is obtained by initially assigning the blocks of the block design, obtained in 2, to different factor level combinations of the treatment design for U_{i-1} and then improving the initial design. The improvement is made under the given design criterion and by exchanging the blocks between different factor level combinations of the treatment design and then re-ordering the blocks between the same factor level combinations of the treatment design. The improvement process is repeated for several starting designs and the best available design is chosen.

4. Step 3 is repeated for $U_{i-2}, U_{i-3}, ..., U_m$.

This approach is open for using any suitable design criterion and gives a design that is optimal (under the design criterion) within strata. But, like D-optimal designs, it rarely allows the estimation of pure-error estimates of the variance components and, unlike equivalent estimation designs, it does not allow the equivalence of GLS and OLS estimates of the model parameters.

3.4.2 An Example

Trinca & Gilmour (2001)[27] give a step by step example for constructing a multi-stratum RS design with 3 strata using a stratum-by-stratum construction algorithm. An experiment involving four factors is to be run in five blocks. Each block has three main units and each main unit has three subunits. Two of the factors (W_1, W_2) are to be applied in the second stratum (to the main units) and remaining two factors (S_1, S_2) are to be applied in the last stratum (to the subunits). So and $f_1 = 0, f_2 = 2, f_3 = 2$ and each factor is at three levels.

We discuss the same example but we consider that we need to construct a split-plot design (a design with 2 strata) where two factors (W_1, W_2) are defined as HTC and will be applied to the whole-plots (main units) and the remaining two factors (S_1, S_2) are defined as ETC and will be applied to the sub-plots (subunits) within each whole-plot. So, we ignore the highest

stratum (blocks) in the original example. Then the step-by-step construction of the design is described below.

1. The first step is to choose two treatment designs, one for the factors applied to the whole-plot level and the other for the factors applied to the sub-plot level. At the whole-plot level, we have two factors applied to the 15 units. The design chosen was a 3 level CCD with the factorial points replicated twice and 3 centre point runs. At the sub-plot level we have two factors applied to the 45 units. The design chosen was the full 3^2 factorial design replicated five times.

2. Once we have chosen the treatment design for both strata, the next step is to arrange the treatment design, chosen at the sub-plot level, in 15 blocks each of size 3. These 15 blocks are defined by the units to which the HTC factors are applied. At this stage we would need a design criterion and a computer based search algorithm. A good block design, found by the authors under the weighted mean efficiency criterion, is given in table 3.6.

3. After constructing a good block design at the sub-plot level, the next step is to combine the treatment design chosen for W_1, W_2 with the block design obtained in step 2. For this, it is necessary to take into account the good estimation of the interactions between W_1, W_2 and S_1, S_2 without affecting the estimation of the other terms of S_1 and S_2 obtained in the block design in step 2. The best combination can be found by interchanging the 15 blocks between 15 different treatment combinations of the factors W_1, W_2. Again, at this stage, we would need a design criterion and a computer algorithm to search for the best combination of the two designs. The best combination, found by the authors under the weighted A-efficiency criterion, is given in table 3.7.

Table 3.6: The design given by Trinca & Gilmour (2001) at Step 2.

Block	S_1	S_2	Block	S_1	S_2
1	-1	0	9	-1	0
1	0	-1	9	0	-1
1	1	1	9	1	1
2	-1	-1	10	-1	0
2	0	0	10	0	1
2	1	1	10	1	-1
3	-1	-1	11	-1	1
3	0	1	11	0	0
3	1	0	11	1	-1
4	-1	-1	12	-1	1
4	0	1	12	0	0
4	1	0	12	1	-1
5	-1	0	13	-1	-1
5	0	-1	13	0	0
5	1	1	13	1	1
6	-1	-1	14	-1	1
6	0	1	14	0	0
6	1	0	14	1	-1
7	-1	0	15	-1	1
7	0	1	15	0	-1
7	1	-1	15	1	0
8	-1	1			
8	0	-1			
8	1	0			

Table 3.7: Final Design given by Trinca & Gilmour (2001) and constructed by using a stratum-by-stratum construction method.

Whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	-1	-1	-1	9	0	-1	-1	0
1	-1	-1	0	0	9	0	-1	0	1
1	-1	-1	1	1	9	0	-1	1	-1
2	-1	-1	-1	1	10	0	0	-1	0
2	-1	-1	0	0	10	0	0	0	-1
2	-1	-1	1	-1	10	1	0	1	1
3	-1	0	-1	-1	11	1	1	-1	1
3	-1	0	0	1	11	1	1	0	0
3	-1	0	1	0	11	1	1	1	-1
4	-1	1	-1	1	12	1	1	-1	-1
4	-1	1	0	-1	12	1	1	0	0
4	-1	1	1	0	12	1	1	1	1
5	-1	1	-1	0	13	1	-1	-1	1
5	-1	1	0	-1	13	1	-1	0	-1
5	-1	1	1	1	13	1	-1	1	0
6	0	0	-1	1	14	1	0	-1	-1
6	0	0	0	0	14	1	0	0	1
6	0	0	1	-1	14	1	0	1	0
7	0	0	-1	-1	15	1	-1	-1	0
7	0	0	0	1	15	1	-1	0	-1
7	0	0	1	0	15	1	-1	1	1
8	0	1	-1	0					
8	0	1	0	1					
8	0	1	1	-1					

Chapter 4

New Design Methodology

In this chapter we propose a new methodology for constructing RS split-plot designs. In section 1, we specify the research problem we will be working on in this chapter. In section 2, we mention the work that motivated our study. Section 3 contains the new design methodology for constructing second order D-optimal RS split-plot designs. Section 4 contains some theoretical results that support our design methodology. In sections 5 and 6 we give two computer algorithms for generating second order RS split-plot designs. Section 7 is the last section that contains some computational results.

4.1 The Research Problem

As we have seen in the previous chapters, there are several techniques available for constructing RS split-plot designs and estimating the variance components. Some design methodologies construct very efficient designs but they rarely allow the estimation of pure error variance components with pre-specified numbers of degrees of freedom. Instead these techniques recommend REML for estimating the variance components. We have already discussed that REML might not work well for the non-orthogonal split-plot designs with only a few whole-plots, because in this case the REML estimate of the whole-plot error variance often turns out to be 0. Another design methodology (equivalent estimation designs) allows the pure error estimates of variance components but this methodology does not generate efficient designs. Our aim is to develop a new design methodology that allows pure error estimates of the variance components, making it possible to test the lack of

fit of the RS model, and also generates efficient designs making optimal use of the available resources. So, we state our research problem as

to develop a methodology for constructing a D-optimal RS split-plot design such that the generated design has the pre-specified number of degrees of freedom for obtaining the pure error estimates of the variance component in each stratum.

Our objective is to develop a general methodology that can be used to generate a split-plot design for fitting a response surface model of any (first, second or higher) order . So, as compared to the equivalent estimation design methodology, that works only for the second order RS split-plot designs, our design methodology would be more general.

4.2 Motivation

Jones & Goos (2007)[18] gave a coordinate exchange algorithm for generating D-optimal split-plot designs. This two stage algorithm first generates a starting design by randomly generating all the required factor levels and then it improves this starting design by the coordinate exchange procedure. Fig 4.1 illustrates how the algorithm works. This motivated our work to develop an algorithm that generates a D-optimal design with some restriction on the numbers of degrees of freedom for estimating the pure error variance components. Here we briefly describe their algorithm.

4.2.1 The Input Information

The algorithm assumes that the experimenter has the following information: number of HTC and ETC factors, the number of whole-plots, the whole-plot size, number of levels for each factor, a set of candidate values for factor levels, e.g. $-1, 0, 1$, prior point estimate of the ratio of the variance components and the model to be fitted.

4.2.2 Generating a Starting Design

The first stage of the algorithm is to generate a random starting design. A starting design is generated by randomly generating the required levels for

all the HTC and the ETC factors. Starting from the first whole-plot, a level for the first HTC factor is generated randomly and that is assigned to the first k rows of the first column of the design matrix. Similarly a level for the second, third and, so on, for the w^{th} HTC factor is randomly generated and applied to the first k rows of the corresponding column of the design matrix. Hence the levels for all the HTC factors are randomly generated and applied to the first whole-plot. The same technique is then used to generate the levels of the HTC factor for all the remaining whole-plots. Once all the required levels for the HTC factors are generated then the algorithm generates the levels for the ETC factors. Starting from the first experimental run, a level for the first ETC factor is randomly generated, then for the second ETC factor and so on until a level for the last ETC factor is generated. Hence, all the required levels for the ETC factors in the first experimental run are generated. Using the same procedure required levels of all the ETC factors are randomly generated run to run and thus a random starting design is generated. The starting design follows the split-plot structure where levels of the HTC factors remain constant within a whole-plot whereas the levels of the ETC factors may change run by run. The algorithm places the levels of HTC factors in the first w columns of the design matrix and the levels of the ETC factors in the last s columns of the design matrix. Then the D-criterion value for this starting design is computed.

4.2.3 Improving the Design

The next stage is to improve the starting design and search for the best possible design, in terms of the D-optimality criterion. The improvement is made by exchanging each factor level with all the values listed in the corresponding set of candidate values for those factor levels one by one. That is, starting from the first whole plot, the level of the first HTC factor is replaced by, say, -1,0,1 one by one, the D-criterion value is calculated with each exchange and if there is any increase in the criterion value then this exchange is saved otherwise no change is made. Similarly, the algorithm continues exchanging the coordinates of all the design points until the last coordinate of the last design point is exchanged and saved if there is an increase in the criterion value. If at least one improvement is recorded during this exchange process, the process of exchanging the coordinates starts again from the first coordinate of the first design point. This exchange process continues until there is no further increase in the D-criterion value. The

process of improving a starting design is executed for several starting designs.

4.3 New Design Methodology

In this section, we present a new approach for constructing RS split-plot designs. This technique allows the construction of a RS split-plot design that is D-optimal and also it reserves the pre-specified numbers of degrees of freedom for obtaining the pure error estimates of the variance components. The design methodology consists of the same two stages of generating a starting design and then to improve the starting design. For generating a starting design we would need all the information given in section 4.2.1. So, we assume that the factors have been classified into ETC and HTC factors. The number and different candidate values of factor levels, the number of whole-plots and a fixed whole-plot size and the RS model to be fitted have also been decided. Some additional information that would be required in this situation is the pre-specified numbers of degrees of freedom for estimating the variance components.

A starting design can be generated randomly, say for example, using Jones & Goos algorithm. However, a starting design generated completely randomly would not always guarantee the pre-specified numbers of degrees of freedom for estimating the variance components. Consequently, it may not be possible to get an improved design with at least the pre-specified numbers of degrees of freedom. So, the key point is to generate a starting design that has at least the pre-specified numbers of degrees of freedom for estimating the variance components. As we will see later, the number of degrees of freedom for estimating the sub-plot error variance can be raised by replicating treatments within whole-plots and the number of degrees of freedom for estimating the whole-plot error variance can be raised by replicating the treatments between the whole-plots. So, the technique we suggest to generate a random starting design with at least pre-specified numbers of degrees of freedom is based on replicating the treatments within and over the whole-plots.

Suppose that we need to generate a design with w_p whole-plots each of size k and the pre-specified numbers of degrees of freedom for estimating the

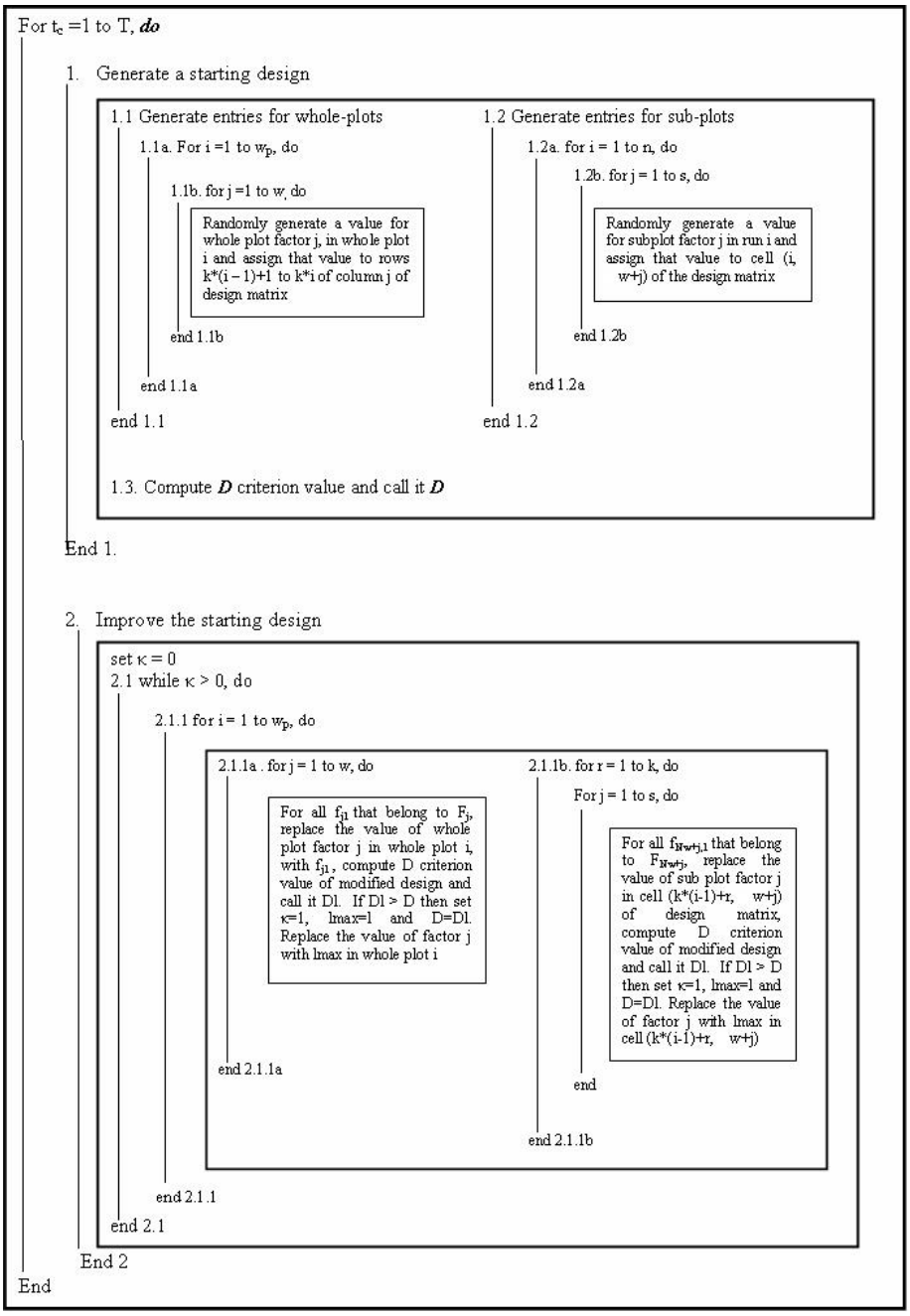

Figure 4.1: Algorithm by Jones & Goos (2007)

(w = number of whole plot factors, s = number of sub plot factors, w_p = number of whole plots, k = whole plot size, b = number of factor levels, F_j = set of values for levels of j^{th} factor, T = total number of starting designs, t_c = number of current starting design)

whole-plot and the sub-pot error variances are u and v respectively, where $0 \leq u \leq (w_p - p_w - 1)$, $0 \leq v \leq (n - p - u)$, n is the number of total experimental runs, p is the number of total parameters in the RS model and p_w is the number of parameters that belong to the HTC factors. Then, to generate the required number of degrees of freedom for estimating the whole-plot error variance, we replicate one of the treatments in $u + 1$ whole-plots. This will give us at least u degrees of freedom for estimating the whole-plot error variance. Then, for generating the pre-specified number of degrees of freedom for estimating the sub-plot error variance, we replicate treatments within the whole-plots. For this, we randomly replicate one of the treatments in each whole-plot a_i times such that it appears at least $a_i + 1$ times in the whole-plot, where $0 \leq a_i \leq (k - 1)$, $i = 1, 2, ..., w_p$ and $\sum_{i=1}^{w_p} a_i = v$. This technique will give us at least v degrees of freedom for estimating the sub-plot error variance (see Lemma 2). Hence the starting design would give us at least the pre-specified numbers of degrees of freedom for obtaining the pure error estimates of the variance components. The remaining required levels in the design for HTC and ETC factors can be generated randomly. Then this starting design can be improved to obtain a D-optimal design with at least the required numbers of degrees of freedom. The improvement is made in terms of an increase in the optimality criterion value.

To suit our situation we slightly modify the optimality criterion. In our situation, the optimality criterion could be

$$\mathcal{D}^* = \mathcal{D} \times \mathcal{D}_u \times \mathcal{D}_v, \tag{4.1}$$

where

$$\mathcal{D} = |\mathbf{X}'\mathbf{\Sigma}^{-1}\mathbf{X}|$$

is the usual D-optimal criterion and

$$\mathcal{D}_u = \begin{cases} 1 & \text{if } d_u \geq u \ ; \\ \frac{1}{(u-d_u)c_1} & \text{otherwise,} \end{cases}$$

and

$$\mathcal{D}_v = \begin{cases} 1 & \text{if } d_v \geq v \ ; \\ \frac{1}{(v-d_v)c_2} & \text{otherwise,} \end{cases}$$

Here d_u and d_v are the available numbers of degrees of freedom in the current design for estimating the whole-plot and sub-plot error variance respectively

and c_1 and c_2 can take values ≥ 1.

To compute the optimality criterion value, we first calculate the usual D criterion value, denoted by $\mathcal{D}$ in our optimality criterion. Then, in the second step, we calculate the available numbers of degrees of freedom in the current design for estimating the variance components, denoted by d_u and d_v. This is done by constructing the $\mathbb{C}_2$ matrix and then by calculating its rank, as described in chapter 2. Since the matrix $\mathbb{C}_2$ depends on the design incidence matrix $\mathbf{N}$, to construct $\mathbb{C}_2$, we first construct $\mathbf{N}$ using the design matrix $\mathbf{X}$ then the matrix $\mathbf{R}$ can easily be constructed using the $\mathbf{N}$ matrix. For a fixed whole-plot size, $\mathbf{K} = k\mathbf{I}$. Then, the $\mathbb{C}_2$ can be constructed using the equation (2.17). Once the $\mathbb{C}_2$ matrix is constructed, we compute d_u and d_v and determine the values for $\mathcal{D}_u$ and $\mathcal{D}_v$. In the third step, we put the values for $\mathcal{D}$, $\mathcal{D}_u$ and $\mathcal{D}_v$ in (4.1) and compute $\mathcal{D}^*$.

Note that if a design has at least the required numbers of degrees freedom then the $\mathcal{D}^*$ criterion would be the usual D-optimal criterion and $\mathcal{D}_u$ and $\mathcal{D}_v$ will have no effect. But if a design has less than the required numbers of degrees of freedom then $\mathcal{D}_u$ and $\mathcal{D}_v$ would cause a decrease in the optimality criterion value. The reason to include these terms in the optimality criterion is that it is quite possible for a design with less than the required numbers of degrees of freedom to have a higher usual D-criterion value as compare to another design that has at least the required numbers of freedom. So, in this situation, a design with less than the required numbers of degrees of freedom would easily replace the design with the required numbers of degrees of freedom which might not be acceptable at all or might be acceptable but under some conditions. By introducing $\mathcal{D}_u$ and $\mathcal{D}_v$ in the criterion we can avoid this situation.

Now the question is to determine the values for c_1 and c_2. These values can be determined under two different situations. First consider the situation where the experimenter is interested to obtain a design with at least the required the numbers of the degrees of freedom for estimating the variance components. Any design with even one degrees of freedom less then the required numbers of degrees of freedom is not acceptable at all. In this situation, we need to determine those values for c_1 and c_2 that sufficiently decrease the optimal criterion value of a design that has less than the required numbers of degrees of freedom, regardless of how many degrees of freedom

are less than the required numbers, in order to ensure that it does not replace a design that has the required numbers of degrees of freedom. For this we can choose

$$c_1 = c_2 = \mathcal{D} + 1.$$

In this case $0 \leq \mathcal{D}^* < 1$ and there will be no chances for a design with less then the required numbers of degrees of freedom to replace another design that has at least the required numbers of degrees of freedom.

Now consider the second situation where the experimenter might consider choosing a design which has one less degree of freedom for estimating the sub-plot (or whole-plot) error variance but it has c_2 (or c_1) times higher criterion value than another design that has the required numbers of degrees of freedom. Then we need to impose a condition that every single decrease in numbers of degree of freedom would be considered as a proportional decrease in the optimality criterion value and the values for c_1 and c_1 will depend on the choice of the experimenter. For example, suppose that for a particular experiment the experimenter wishes to have 3 degrees of freedom available for estimating the whole-plot error variance and 10 degrees of freedom available for estimating the sub-plot error variance. Suppose that a design with the required degrees of freedom has D-criterion value equals to 50. Then the experimenter might like to use another design which has 3 degrees of freedom for estimating the whole-plot error variance and 9 degrees of freedom for estimating the sub-plot error variance but its D-criterion value is greater than 500. In this case $c_2 < 10$.

4.4 Determining the Degrees of Freedom

In this section, we prove that our suggested approach for generating a starting design ensures that the starting design has at least the required numbers of degrees of freedom for estimating the variance components. In Chapter 2, we discussed that a split-plot design can be considered as a general incomplete block design if we ignore the treatment structure. Then, in this case, the numbers of degrees of freedom for estimating the whole-plot error variance are equal to the rank of the matrix $\mathbb{C}_2$, where $\mathbb{C}_2$ is defined in equation (2.17) as

$$\mathbb{C}_2 = \mathbf{K} - \mathbf{N}'\mathbf{R}^{-1}\mathbf{N}. \tag{4.2}$$

We use the following lemmas to prove that if a starting design is generated using the approach suggested in section 4.3 then the starting design would contain at least the required numbers of degrees of freedom for estimating the variance components.

Lemma 1 *In a split-plot design with w_p whole-plots and fixed whole-plot size (k), if w_1 whole-plots have at least one treatment common between them then $d_w \geq w_1 - 1$, where $2 \leq w_1 \leq w_p$ and d_w denotes the numbers of degrees of freedom for estimating the whole-plot error variance.*

Proof

We assume, without loss of generality, that the first w_1 whole-plots have at least one treatment in common and the next $w_2 = w_p - w_1$ whole-plots do not have any treatment common with the first w_1 whole-plots. Under this situation, the matrices $\mathbf{N}$, $\mathbf{K}$ and $\mathbf{R}$ can be written as

$$\mathbf{N} = \begin{bmatrix} \mathbf{N}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{N}_2 \end{bmatrix}, \mathbf{R} = \begin{bmatrix} \mathbf{R}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{R}_2 \end{bmatrix}, \mathbf{K} = \begin{bmatrix} \mathbf{K}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{K}_2 \end{bmatrix},$$

where $\mathbf{N}_1$ is a $(t_1 \times w_1)$ matrix, $\mathbf{N}_2$ is a $(t_2 \times w_2)$ matrix, $\mathbf{K}_1 = k\mathbf{I}_{w_1}$, $\mathbf{K}_2 = k\mathbf{I}_{w_2}$, $\mathbf{R}_1 = diag\begin{bmatrix} r_1 & r_2 & \cdots & r_{t_1} \end{bmatrix}$, $\mathbf{R}_2 = diag\begin{bmatrix} r_{t_1+1} & r_{t_1+2} & \cdots & r_t \end{bmatrix}$, and t_1 and t_2 denote the numbers of distinct treatments applied to w_1 and w_2 whole-plots respectively. Then

$$\mathbf{N}'\mathbf{R}^{-1}\mathbf{N} = \begin{bmatrix} \mathbf{N}_1' & \mathbf{0}' \\ \mathbf{0}' & \mathbf{N}_2' \end{bmatrix} \begin{bmatrix} \mathbf{R}_1^{-1} & \mathbf{0} \\ \mathbf{0} & \mathbf{R}_2^{-1} \end{bmatrix} \begin{bmatrix} \mathbf{N}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{N}_2 \end{bmatrix} \tag{4.3}$$

$$= \begin{bmatrix} \mathbf{N}_1'\mathbf{R}_1^{-1}\mathbf{N}_1 & \mathbf{0}' \\ \mathbf{0}' & \mathbf{N}_2'\mathbf{R}_2^{-1}\mathbf{N}_2 \end{bmatrix}, \tag{4.4}$$

and

$$\mathbb{C}_2 = \mathbf{K} - \mathbf{N}'\mathbf{R}^{-1}\mathbf{N} \tag{4.5}$$

$$= \begin{bmatrix} \mathbf{K}_1 - \mathbf{N}_1'\mathbf{R}_1^{-1}\mathbf{N}_1 & \mathbf{0}' \\ \mathbf{0}' & \mathbf{K}_2 - \mathbf{N}_2'\mathbf{R}_2^{-1}\mathbf{N}_2 \end{bmatrix} \tag{4.6}$$

$$= \begin{bmatrix} \mathbf{C}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{C}_2 \end{bmatrix}, \tag{4.7}$$

where

$$\mathbf{C}_1 = \mathbf{K}_1 - \mathbf{N}_1'\mathbf{R}_1^{-1}\mathbf{N}_1,$$

and

$$\mathbf{C}_2 = \mathbf{K}_2 - \mathbf{N}_2'\mathbf{R}_2^{-1}\mathbf{N}_2.$$

Then

$$rank(\mathbb{C}_2) \geq rank(\mathbf{C}_1). \tag{4.8}$$

The equality will hold if w_2 whole-plots have different and distinct treatments. In that case $\mathbf{C}_2$ will be a matrix of zeros and $rank(\mathbb{C}_2) = rank(\mathbf{C}_1)$.

Now, the matrix $\mathbf{N}_1$ can be written as

$$\mathbf{N_1} = \begin{pmatrix} n_{11} & n_{12} & \cdots & n_{1w_1} \\ n_{21} & n_{22} & \cdots & n_{2w_1} \\ \vdots & \vdots & & \vdots \\ n_{t_1 1} & n_{t_1 2} & \cdots & n_{t_1 w_1} \end{pmatrix}.$$

By applying the definition of a connected design given by Hinkelmann & Kempthorne (2005, p;14)[16], and also given in chapter 2 of this thesis, it can be seen that $\mathbf{N}_1$ represents a connected design. For example, if we consider the case when the w_1 whole-plots have exactly one treatment in common, and we assume that the i^{st} treatment is common between the whole-plots, then all the elements in the i^{th} row of matrix $\mathbf{N}_1$ would be non-zero and it is easy to check that it represents a connected design. From chapter 2, we know that the rank of matrix $\mathbb{C}_2$ for a connected design with w_a blocks is $w_a - 1$. So,

$$rank(\mathbf{C}_1) = w_1 - 1.$$

Then form equation (4.8) we get,

$$rank(\mathbb{C}_2) \geq w_1 - 1. \tag{4.9}$$

Again, the equality will hold when w_2 whole-plots have distinct and different treatments.

In case where more than one treatment is common between the whole-plots, the design would still be a connected design with $rank(\mathbf{C}_1) = w_1 - 1$.

Lemma 2 *Suppose that a split-plot design has w_p whole-plots, each of size k, and w_1 whole-plots have at least one treatment in common such that $d_w = w_1 - 1 = u$. Then, if one of the treatments in whole-plot i appears $a_i + 1$ times within the whole-plot then $d_s \geq \sum_{i=1}^{w_p} a_i$.*

Here $2 \leq w_1 \leq w_p$, $0 \leq a_i \leq (k-1)$, $i = 1, 2, ..., w_p$ and d_w and d_s denotes the numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances respectively.

Proof
From chapter 2, we know that,

$$d_s = n - t - rank(\mathbb{C}_2). \tag{4.10}$$

So,

$$d_s = n - t - u.$$

Since $w_1 = u + 1$ whole-plots have at least one treatment common between them, $t \leq n - u$. Then from equation (4.10) we get

$$d_s \geq 0.$$

Now suppose that one of the treatments in the first whole-plot is replicated $a_1 + 1$ times, where $0 \leq a_1 \leq (k-1)$, then $t = n - u - a_1$ and from equation (4.10) we will get

$$d_s \geq a_1$$

Now suppose that one of the treatments in the 2^{nd} whole-plot is also replicated a_2+ times within the whole-plot. Then $t = n - u - a_1 - a_2$ and it will give us,

$$d_s \geq a_1 + a_2.$$

Similarly suppose that $a_i + 1$ represents the number of times one of the treatment in whole-plot i is replicated within the i^{th} whole-plot, where $0 \leq a_i \leq (k-1)$ and $i = 1, 2, ..., w_p$, then $t \leq n - u - \sum_{i=1}^{w_p} a_i$ and from equation (4.10) we will get

$$d_s \geq \sum_{i=1}^{w_p} a_i.$$

Next we present two different computer search algorithms that work under the new approach for generating a RS split-plot design such that the design has at least the pre-specified numbers of degrees of freedom for estimating the pure error variance components. The first algorithm is a point exchange algorithm and the second algorithm is a coordinate exchange algorithm.

4.5 A Point Exchange Algorithm

The point exchange algorithm, given in this section, searches for an optimal design using a set of candidate points for the HTC and the ETC factors. For our algorithm, we suppose that we have two different sets of candidate points, one for the HTC factors and the other for the the ETC factors. In this case, a set of candidate points can be a 3^w or 3^s full factorial design, where w and s denotes the number of the HTC and the ETC factors involved in the experiment. The algorithm first generates a starting design with the required numbers of degrees of freedom and then improves the starting design to obtain an optimal design. Here, we describe this two stage procedure.

4.5.1 Generating a Starting Design

Let $\mathbf{A_w}$ be a candidate set of points and n_w be the number of candidate points for the HTC factors only and let $\mathbf{A_s}$ be the candidate set of points and n_s be the number of candidate points for the ETC factors only. We call an ETC candidate point to a point available in $\mathbf{A_s}$ and an HTC candidate point to a point available in $\mathbf{A_w}$. We suppose that any combination of the candidate points (of HTC and ETC factors) is acceptable. We assume that we have w_p whole plots with fixed whole-plot size k and u and v are the required numbers of degrees of freedom for estimating the variance components, as mentioned before. Let $w_1 = \min[(w_p - u - 1), (n_w - 1)]$. Then we randomly choose w_1 candidate points from $\mathbf{A_w}$ and randomly assign those points to w_1 whole-plots such that every whole-plot receives a different candidate point. Then, for each of the w_1 whole-plots, we randomly choose k candidate points from $\mathbf{A_s}$ and randomly assign to the k sub-plots within each whole-plot such that every sub-plot in a single whole-plot receives a different candidate point. Hence, we construct a design of w_1 whole-plots with a single treatment replication. This will help to ensure that there are enough numbers of degrees of freedom available for estimating the model parameters.

Then we choose one candidate point from $\mathbf{A_w}$, not assigned to any whole-plot previously, and assign this point to all the remaining $w_2 = w_p - w_1$ whole-plots. Then we choose a candidate point from $\mathbf{A_s}$ and assign it to the k^{th} sub-plot of each of the last $u+1$ whole-plots such that the last $u+1$ whole-plots now have the same combination of HTC and ETC factor levels at the k^{th} sub-plot. This will generate u degrees of freedom for estimating the whole-plot error variance because the $u+1$ whole-plots receive the same treatment combination. Then we randomly choose candidate points from $\mathbf{A_s}$, excluding the point already assigned to the k^{th} sub-plot of each of the $u+1$ whole-plots, and assign them to the remaining sub-plots associated with the w_2 whole-plots with minimum possible replication of points. Let $n_2 = w_2 \times k$, then if $n_2 - u \leq n_s$, then ETC candidate points can be applied with a single replication. If $n_2 - u > n_s$, and let $n_0 = n_2 - u - n_s$, then $n_s - 1$ distinct ETC candidate points are assigned to the first $n_s - 1$ sub-plots associated with the w_2 whole-plots. Then we replicate one of the ETC candidate points n_0 times and assign it to the remaining sub-plots such that this candidate point is now applied to a total of $n_0 + 1$ sub-plots. This would generate n_0 degrees of freedom for estimating the sub-plot error variance. Now our design has u degrees of freedom for estimating the whole-plot error variance and 0 or n_0 degrees of freedom for estimating the sub-plot error variance. If $n_0 < v$, the remaining $v_0 = v - n_0$ degrees of freedom for estimating the sub-plot error variance will be generated by selecting a_i treatments from the i^{th} whole-plot, such that $0 \leq a_i \leq (k-1)$, $\sum_{i=1}^{w_0} a_i = v_0$, $i = 1, 2, ..., w_0$, and $w_0 = n_0/k$ rounded to the next integer, and replacing these treatments by the k treatment of the corresponding whole-plot.

Thus, the starting design will have exactly the pre-specified numbers of degrees of freedom. A design generated by this approach can be partitioned into two designs. One design consisting on first w_1 whole-plots where every whole-plot has a different HTC design point and the second design consists on the last w_2 whole-plots where each whole-plot has the same (but different from the first design) HTC design point and at least one common ETC design point.

4.5.2 Examples of Starting Designs

In this section we give a couple of examples to show how a starting design with the required numbers of degrees of freedom for estimating the variance components is generated using the point exchange algorithm given in the last section. We will consider two different design problems to illustrate two different cases. The first case is when $w_1 = w_p - u - 1$ and the second case is when $w_1 < w_p - u - 1$.

Example 5 *Suppose that we have* 2 *HTC and* 2 *ETC factors, each at three levels, the number of whole-plots is* 9*, each whole-plot has* 3 *sub-plots, the model to be fitted is a second order RS model and pre-specified numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances are* 3 *and* 6 *respectively. We assume that the set of candidate points in each case (for the HTC and for the ETC factors) is a* 3^2 *full factorial design. So* $n_w = n_s = 9$*,* $w1 = w_p - u - 1 = 9 - 3 - 1 = 5$ *and* $w_2 = 9 - 5 = 4$*.*

First we choose 5 *HTC candidate points from* $\mathbf{A_w}$ *and assign these candidate points to the first* 5 *whole-plots such that every whole-plot receives a different candidate point. Then, for each of these* 5 *whole-plot, we randomly choose* 3 *candidate points from* $\mathbf{A_s}$ *and assign these points to the sub-plots such that every sub-plot within a single whole-plot receives a different candidate point. The design at this stage is given in table 4.1.*

In the second step, we generate degrees of freedom for estimating the pure error whole-plot variance. The desired number of degrees of freedom for estimating the whole-plot variance is 3*. So, we apply one of the HTC candidate point (1,1), not applied before, to all of the remaining* 4 *whole-plots. Then we apply one of the ETC candidate points (1,1) to the* 3^{rd} *sub-plot of each of the* 4 *whole-plots. Thus one of the treatments (1,1,1,1) is being replicated in all of the last* 4 *whole-plots. This will generate* $4 - 1 = 3$ *degrees of freedom for estimating the whole-plot variance. Now, we have* $(k-1) \times w_2 = 2 \times 4 = 8$ *remaining sub-plots associated with the last* 4 *whole-plots and* $3^2 - 1 = 9 - 1 = 8$ *ETC candidate points to assign these sub-plots. So we assign the* 8 *ETC candidate points to the* 8 *sub-plots such that every sub-plot receives a different candidate point. The design at this stage is given in table 4.2. As given in chapter* 2*, we construct the matrix* $\mathbb{C}_2$ *for calculating the available numbers of degrees of freedom for estimating the variance components. So, the matrices* $\mathbf{N}$*,* $\mathbf{R}$ *and* $\mathbb{C}_2$*, for this design, are given as,*

Table 4.1: A starting design at the first stage with 5 whole-plots.

whole plot	W_1	W_2	S_1	S_2
1	-1	0	-1	0
1	-1	0	0	1
1	-1	0	0	0
2	0	-1	0	-1
2	0	-1	1	-1
2	0	-1	0	1
3	0	0	1	1
3	0	0	-1	1
3	0	0	1	0
4	1	-1	1	-1
4	1	-1	0	1
4	1	-1	0	0
5	1	0	-1	1
5	1	0	-1	-1
5	1	0	1	1

$$\mathbf{N} = \begin{pmatrix} 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 1 & 1 & 1 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \end{pmatrix},$$

Table 4.2: A starting design at the second stage with the required numbers of degrees of freedom for estimating the whole-plot error variance.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	0	-1	0	6	1	1	0	-1
1	-1	0	0	1	6	1	1	-1	1
1	-1	0	0	0	6	1	1	1	1
2	0	-1	0	-1	7	1	1	0	0
2	0	-1	1	-1	7	1	1	-1	0
2	0	-1	0	1	7	1	1	1	1
3	0	0	1	1	8	1	1	0	1
3	0	0	-1	1	8	1	1	1	0
3	0	0	1	0	8	1	1	1	1
4	1	-1	1	-1	9	1	1	-1	-1
4	1	-1	0	1	9	1	1	1	-1
4	1	-1	0	0	9	1	1	1	1
5	1	0	-1	1					
5	1	0	-1	-1					
5	1	0	1	1					

$$\mathbf{R} = diag\begin{pmatrix} 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 4 & 1 & 1 & 1 & 1 & 1 & 1 \end{pmatrix},$$

and

$$\mathbb{C}_2 = \begin{pmatrix} 0 & 0 & 0 & 0 & 0 & 0.00 & 0.00 & 0.00 & 0.00 \\ 0 & 0 & 0 & 0 & 0 & 0.00 & 0.00 & 0.00 & 0.00 \\ 0 & 0 & 0 & 0 & 0 & 0.00 & 0.00 & 0.00 & 0.00 \\ 0 & 0 & 0 & 0 & 0 & 0.00 & 0.00 & 0.00 & 0.00 \\ 0 & 0 & 0 & 0 & 0 & 0.00 & 0.00 & 0.00 & 0.00 \\ 0 & 0 & 0 & 0 & 0 & 0.75 & -0.25 & -0.25 & -0.25 \\ 0 & 0 & 0 & 0 & 0 & -0.25 & 0.75 & -0.25 & -0.25 \\ 0 & 0 & 0 & 0 & 0 & -0.25 & -0.25 & 0.75 & -0.25 \\ 0 & 0 & 0 & 0 & 0 & -0.25 & -0.25 & -0.25 & 0.75 \end{pmatrix}.$$

Here,

$$rank(\mathbb{C}_2) = 3,$$

so this design gives 3 *degrees of freedom for estimating the whole-plot error variance. Also, for this design* $t = 24$ *and*

$$d_s = n - t - rank(\mathbb{C}_2) = 27 - 24 - 3 = 0.$$

So, this design gives us 0 *degrees of freedom for estimating the sub-plot error variance.*

Now, we need to generate 6 *degrees of freedom for estimating the sub-plot error variance without losing any of the* 3 *degrees of freedom for estimating the whole-plot error variance generated previously. For this we randomly choose* a_i *treatments from the* i^{th} *whole-plot such that* $0 \leq a_i \leq (k-1)$, $\sum_{i=1}^{wp} a_i = v$ *and* $i = 1, 2, ..., 9$ *and replace these treatments by the* 3^{rd} *treatment of the corresponding whole-plot. For this example we have* $a_i = 1$ *for* $i = 1, 4, 5, 9$, $a_i = 2$ *for* $i = 3$ *and* $a_i = 0$ *for* $i = 2, 6, 7, 8$. *So, we replace one treatment from each of the whole-plot number* $1, 4, 5$ *and* 9 *and two treatments from the whole-plot number* 3 *by the* 3^{rd} *treatment of the corresponding whole-plot. This will generate* $v = 6$ *degrees of freedom for estimating the sub-plot error variance. The design at this stage is given in table 4.3 and the matrices* $\mathbf{N}$, $\mathbf{R}$ *and* $\mathbb{C}_2$ *for this design are given as,*

Table 4.3: The starting design at the final stage with the required numbers of degrees of freedom for estimating the variance components.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	0	-1	0	6	1	1	0	-1
1	-1	0	0	0	6	1	1	-1	1
1	-1	0	0	0	6	1	1	1	1
2	0	-1	0	-1	7	1	1	0	0
2	0	-1	1	-1	7	1	1	-1	0
2	0	-1	0	1	7	1	1	1	1
3	0	0	1	0	8	1	1	0	1
3	0	0	1	0	8	1	1	1	0
3	0	0	1	0	8	1	1	1	1
4	1	-1	1	-1	9	1	1	-1	-1
4	1	-1	0	0	9	1	1	1	1
4	1	-1	0	0	9	1	1	1	1
5	1	0	-1	1					
5	1	0	1	1					
5	1	0	1	1					

$$\mathbf{N} = \begin{pmatrix} 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 2 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 2 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 3 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 2 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 1 & 1 & 2 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \end{pmatrix},$$

$$\mathbf{R} = diag\begin{pmatrix} 1 & 2 & 1 & 2 & 3 & 1 & 1 & 1 & 1 & 2 & 1 & 1 & 5 & 1 & 1 & 1 & 1 & 1 \end{pmatrix}.$$

$$\mathbb{C}_2 = \begin{pmatrix} 0 & 0 & 0 & 0 & 0 & 0.0 & 0.0 & 0.0 & 0.0 \\ 0 & 0 & 0 & 0 & 0 & 0.0 & 0.0 & 0.0 & 0.0 \\ 0 & 0 & 0 & 0 & 0 & 0.0 & 0.0 & 0.0 & 0.0 \\ 0 & 0 & 0 & 0 & 0 & 0.0 & 0.0 & 0.0 & 0.0 \\ 0 & 0 & 0 & 0 & 0 & 0.0 & 0.0 & 0.0 & 0.0 \\ 0 & 0 & 0 & 0 & 0 & 0.8 & -0.2 & -0.2 & -0.4 \\ 0 & 0 & 0 & 0 & 0 & -0.2 & 0.8 & -0.2 & -0.4 \\ 0 & 0 & 0 & 0 & 0 & -0.2 & -0.2 & 0.8 & -0.4 \\ 0 & 0 & 0 & 0 & 0 & -0.4 & -0.4 & -0.4 & 1.2 \end{pmatrix}.$$

Here,

$$rank(\mathbb{C}_2) = 3,$$

so this design still gives 3 *degrees of freedom for estimating the whole-plot error variance. But, now* $t = 18$ *and*

$$d_s = n - t - rank(\mathbb{C}_2) = 27 - 18 - 3 = 6.$$

So, this design gives us 6 *degrees of freedom for estimating the sub-plot error variance. Thus the starting design now has the required numbers of degrees of freedom for estimating the variance components.*

Note the structure of the matrix $\mathbb{C}_2$ *in all three cases. In step* 1*, the matrix* $\mathbb{C}_2$ *will always be a zero matrix while in step* 2 *and* 3 *matrix* $\mathbb{C}_2$ *will always be of the form*

$$\mathbb{C}_2 = \begin{pmatrix} \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{C}_c \end{pmatrix},$$

where $\mathbf{C}_c$ *is a matrix of order* $(w_2 \times w_2)$*.*

Example 6 *We consider another design problem where there are* 2 *HTC and* 2 *ETC factors,* $w_p = 12$*,* $k = 4$*,* $u = 2$*,* $v = 22$*, the model to be fitted is a second order RS model and a set of candidate points in each case (for the HTC and for the ETC factors) is a* 3^2 *full factorial design. Here* $w_1 = min\,[(w_p - u - 1), (n_w - 1)] = n_w - 1 = 8$*. So, first we choose* 8 *candidate points from* $\mathbf{A_w}$ *and randomly apply these points to the first* 8 *whole-plots. Then for each of the* 8 *whole-plots, we choose* 4 *candidate points from* $\mathbf{A_s}$ *and randomly assign to the sub-plots associated with the whole-plot such that each sub-plot ina single whole-plot receives a different ETC candidate point. The design at this stage is given in table 4.4.*

Now, we come to the remaining $w_2 = w_p - w_1 = 12 - 8 = 4$ *whole-plots. First we apply one of the HTC candidate points, not applied to the first* 8 *whole-plots, to each of the remaining* 4 *whole-plots. Then we choose one ETC candidate point and assign it to the* 4^{th} *sub-plot of each of the last* $u + 1 = 3$ *whole-plots such that last* 3 *whole-plots now have the same combination of the HTC and the ETC factor levels at the* 4^{th} *sub-plot. This will generate* 2 *degrees of freedom for estimating the whole-plot error variance. Now we have* $16 - 3 = 13$ *remaining sub-plots associated with the last* 4 *whole-plots and* $n_s - 1 = 8$ *distinct ETC candidate points to assign to these* 13 *sub-plots. So, we randomly apply the* 8 *distinct ETC candidate points to the first* 8 *sub-plots associated with the last* 4 *whole-plots and then we randomly choose a ETC candidate point, replicate it* $13 - 8 = 5$ *times and assign it to the each of the remaining* 5 *sub-plots. The design at this stage is given in table 4.5 and matrices* $\mathbf{N}$*,* $\mathbf{R}$ *and* $\mathbb{C}_2$ *for this design are given as,*

Table 4.4: A starting design at the first stage with $w_1 = 8$ whole-plots.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	1	-1	1	1	7	-1	0	1	0
1	1	-1	0	0	7	-1	0	1	1
1	1	-1	0	-1	7	-1	0	-1	-1
1	1	-1	1	-1	7	-1	0	0	1
2	0	1	0	-1	8	-1	1	1	0
2	0	1	0	0	8	-1	1	-1	1
2	0	1	-1	-1	8	-1	1	0	1
2	0	1	1	-1	8	-1	1	-1	-1
3	0	0	1	-1					
3	0	0	-1	-1					
3	0	0	0	-1					
3	0	0	-1	1					
4	0	-1	0	1					
4	0	-1	-1	1					
4	0	-1	-1	0					
4	0	-1	1	1					
5	-1	-1	0	-1					
5	-1	-1	-1	0					
5	-1	-1	1	0					
5	-1	-1	1	-1					
6	1	0	-1	1					
6	1	0	1	1					
6	1	0	0	0					
6	1	0	-1	0					

Table 4.5: A starting design at the second stage.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	1	-1	1	1	7	-1	0	1	0
1	1	-1	0	0	7	-1	0	1	1
1	1	-1	0	-1	7	-1	0	-1	-1
1	1	-1	1	-1	7	-1	0	0	1
2	0	1	0	-1	8	-1	1	1	0
2	0	1	0	0	8	-1	1	-1	1
2	0	1	-1	-1	8	-1	1	0	1
2	0	1	1	-1	8	-1	1	-1	-1
3	0	0	1	-1	9	1	1	0	1
3	0	0	-1	-1	9	1	1	0	-1
3	0	0	0	-1	9	1	1	-1	1
3	0	0	-1	1	9	1	1	1	0
4	0	-1	0	1	10	1	1	-1	0
4	0	-1	-1	1	10	1	1	0	0
4	0	-1	-1	0	10	1	1	-1	-1
4	0	-1	1	1	10	1	1	1	1
5	-1	-1	0	-1	11	1	1	1	-1
5	-1	-1	-1	0	11	1	1	1	-1
5	-1	-1	1	0	11	1	1	1	-1
5	-1	-1	1	-1	11	1	1	1	1
6	1	0	-1	1	12	1	1	1	-1
6	1	0	1	1	12	1	1	1	-1
6	1	0	0	0	12	1	1	1	-1
6	1	0	-1	0	12	1	1	1	1

$$\mathbf{N} = \begin{pmatrix} \mathbf{N}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{N}_2 \end{pmatrix},$$

where

$$\mathbf{N}_1 = \begin{pmatrix} \mathbf{1}_4 & \mathbf{0} & \mathbf{0} & \cdots & \mathbf{0} \\ \mathbf{0} & \mathbf{1}_4 & \mathbf{0} & \cdots & \mathbf{0} \\ \vdots & \vdots & \ddots & & \vdots \\ \mathbf{0} & \mathbf{0} & \mathbf{0} & \cdots & \mathbf{1}_4 \end{pmatrix}_{(32\times 8)},$$

and

$$\mathbf{N}_2 = \begin{pmatrix} 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 1 & 1 & 1 \\ 0 & 0 & 3 & 3 \end{pmatrix},$$

$$\mathbf{R} = \begin{pmatrix} \mathbf{R}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{R}_2 \end{pmatrix},$$

where $\mathbf{R}_1$ *is an identity matrix of order* (32×32) *and*

$$\mathbf{R}_2 = diag\begin{pmatrix} 1 & 1 & 1 & 1 & 1 & 1 & 1 & 3 & 6 \end{pmatrix},$$

and

$$\mathbb{C}_2 = \begin{pmatrix} \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{C}_2 \end{pmatrix},$$

where

$$\mathbf{C}_2 = \begin{pmatrix} 0 & 0 & 0 & 0 \\ 0 & 0.667 & -0.333 & -0.333 \\ 0 & -0.333 & 2.167 & -1.833 \\ 0 & -0.333 & -1.833 & 2.167 \end{pmatrix}.$$

Here $rank(\mathbb{C}_2) = 2$ *and* $t = 41$. *So, the design at this stage generates* 2 *degrees of freedom for estimating the whole-plot error variance and* $48 - 41 - 2 = 5$ *degrees of freedom for estimating the sub-plot error variance. We have* 17 *less than the required degrees of freedom for estimating the sub-plot error variance. For generating* 17 *more degrees of freedom for estimating the split-plot error variance we randomly select* $1, 1, 3, 1, 3, 3, 2$ *and* 3 *(total* 17*) treatments from whole-plot number* $1, 2, 3, 4, 6, 7, 8$ *and* 10 *and replace these treatments with the* 4^{th} *treatment of the corresponding whole-plot. The design incidence matrices* $\mathbf{N}$, $\mathbf{R}$ *and* $\mathbb{C}_2$ *for this design are given below and the design is given in table 4.6.*

$$\mathbf{N} = \begin{pmatrix} \mathbf{N}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{N}_2 \end{pmatrix},$$

where

$$\mathbf{N}_1 = \begin{pmatrix} 2 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 2 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 4 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 2 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 4 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 4 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 3 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \end{pmatrix},$$

Table 4.6: The starting design at the final stage with the required numbers of degrees of freedom for estimating the variance components.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	1	-1	1	-1	7	-1	0	0	1
1	1	-1	0	0	7	-1	0	0	1
1	1	-1	0	-1	7	-1	0	0	1
1	1	-1	1	-1	7	-1	0	0	1
2	0	1	1	-1	8	-1	1	-1	-1
2	0	1	0	0	8	-1	1	-1	-1
2	0	1	-1	-1	8	-1	1	0	1
2	0	1	1	-1	8	-1	1	-1	-1
3	0	0	-1	1	9	1	1	0	1
3	0	0	-1	1	9	1	1	0	-1
3	0	0	-1	1	9	1	1	-1	1
3	0	0	-1	1	9	1	1	1	0
4	0	-1	1	1	10	1	1	1	1
4	0	-1	-1	1	10	1	1	1	1
4	0	-1	-1	0	10	1	1	1	1
4	0	-1	1	1	10	1	1	1	1
5	-1	-1	0	-1	11	1	1	-1	-1
5	-1	-1	-1	0	11	1	1	1	-1
5	-1	-1	1	0	11	1	1	1	-1
5	-1	-1	1	-1	11	1	1	1	1
6	1	0	-1	0	12	1	1	1	-1
6	1	0	-1	0	12	1	1	1	-1
6	1	0	-1	0	12	1	1	1	-1
6	1	0	-1	0	12	1	1	1	1

and

$$\mathbf{N}_2 = \begin{pmatrix} 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 \\ 0 & 4 & 1 & 1 \\ 0 & 0 & 3 & 3 \end{pmatrix}.$$

$$\mathbf{R} = \begin{pmatrix} \mathbf{R}_1 & \mathbf{0} \\ \mathbf{0} & \mathbf{R}_2 \end{pmatrix},$$

where

$$\mathbf{R}_1 = diag\begin{pmatrix} 2 & 1 & 1 & 2 & 1 & 1 & 4 & 2 & 1 & 1 & 1 & 1 & 1 & 1 & 4 & 4 & 3 & 1 \end{pmatrix},$$

and

$$\mathbf{R}_2 = diag\begin{pmatrix} 1 & 1 & 1 & 1 & 6 & 6 \end{pmatrix}.$$

$$\mathbb{C}_2 = \begin{pmatrix} \mathbf{0} & \mathbf{0} \\ \mathbf{0} & \mathbf{C}_2 \end{pmatrix},$$

where

$$\mathbf{C}_2 = \begin{pmatrix} 0 & 0 & 0 & 0 \\ 0 & 1.333 & -0.667 & -0.667 \\ 0 & -0.667 & 2.333 & -1.667 \\ 0 & -0.667 & -1.667 & 2.333 \end{pmatrix}.$$

Here rank$(\mathbb{C}_2) = 2$ *and* $t = 24$. *So, the design at this stage generates* 2 *degrees of freedom for estimating the whole-plot error variance and* $48 - 24 - 2 = 22$ *degrees of freedom for estimating the sub-plot error variance. Hence the starting design has the required numbers of degrees of freedom for estimating the variance components.*

4.5.3 Improving the Starting Design

Once a starting design is generated, its optimality criterion value is computed and then an improvement in terms of an increase in the optimality criterion value is searched by exchanging the design points with the candidate points. The improvement procedure is performed whole-plot by whole-plot. First, the HTC design point in the first whole-plot is replaced by all the points in $\mathbf{A_w}$ one by one. For each replacement, the algorithm calculates the numbers of degrees of freedom, in the new design, for estimating the variance

components by generating the $\mathbb{C}_2$ matrix and then computes the optimality criterion value. If there is an increase in the optimality criterion value, the replacement is saved as a new design otherwise no change is made. Then the ETC design point in the first sub-plot of the first whole-plot is replaced by all the points in $\mathbf{A_s}$ one by one, the optimality criterion value for each replacement is computed and if there is any improvement the change is saved. The same procedure is repeated for the second, third and so on, for the k^{th} sub-plot of the first whole-plot. Hence all the design points in the first whole-plot and the corresponding sub-plots are replaced by all of the corresponding candidate points. Then the same process is repeated for the second, third and so on, for the last whole-plot. Thus the first round of the improvement phase is completed. If at least one improvement in the optimality criterion value is recorded in one complete round then the whole improvement procedure is performed again. When no improvement is recorded in a complete round the algorithm will stop and the best design in terms of the optimality criterion value would be given as the output.

This two stage procedure of generating and then improving a starting design is repeated several times and the best available design with the highest optimality criterion value is selected.

4.5.4 The Algorithm

We assume that we have, the number of whole-plots (w_p), fixed whole-plot size (k), a set of candidate points for HTC factors ($\mathbf{A_w}$), the number of HTC candidate points (n_w), a set of candidate points for the ETC factors ($\mathbf{A_s}$), the number of ETC candidate points (n_s), the number of total experimental runs (n), the required number of degrees of freedom for estimating the whole-plot error variance (u), the required number of degrees of freedom for estimating the sub-plot error variance (v) and a point estimate of the ratio of the variance components. Then the algorithm can be described as:

1. Set
   (a) $w_1 = \min\left((w_p - u - 1), (n_w - 1)\right)$
   (b) $n_1 = k \times w_1$
   (c) $w_2 = w_p - w_1$
   (d) $n_2 = n - n_1$

2. Generate a Starting Design

   (a) Randomly choose w_1 distinct points from $\mathbf{A_w}$ and assign these points to first w_1 whole-plots such that each whole-plot receives a different candidate point.

   (b) For each of the first w_1 whole-plots, randomly choose k distinct points from $\mathbf{A_s}$ and assign these points to the k sub-plots of the corresponding whole-plot such that every sub-plot within a single whole-plot receives a different candidate point.

   (c) Randomly choose a single point from $\mathbf{A_w}$, not used in Step 2.(a), and assign this point to each of the remaining w_2 whole-plots.

   (d) Randomly choose s_0, where s_0 is a point in $\mathbf{A_s}$, and assign this point to the k^{th} sub-plot of each of the last $u+1$ whole-plots.

   (e) Set $m_0 = \min\left((n_2 - u), (n_s - 1)\right)$.

   (f) Randomly choose a set of m_0 distinct points from $\mathbf{A_s}$ (excluding s_0) and call this set $\mathbf{A_{s_a}}$.

   (g) Compute $n_0 = n_2 - u - n_s$.

   (h) If $n_0 > 0$, set $w_0 = w_p - w_a$, $v_0 = v - n_0$ and go the Step 2.(i), else set $w_0 = w_p$, $v_0 = v$ and go to the step 2.(j). (Here $w_a = n_0/k$ rounded to the next integer).

   (i) Randomly choose a point from $\mathbf{A_{s_a}}$, replicate this point n_0 times and add these replicated points in $\mathbf{A_{s_a}}$.

   (j) Randomly assign the candidate points in $\mathbf{A_{s_a}}$ to all the sub-plots associated with the w_2 whole-plots, excluding the k^{th} sub-plot of each of the last $u+1$ whole-plots.

   (k) If $v_0 > 0$, go to the next step else go to Step 3.

   (l) For $(i = 1, 2, ..., w_0)$, randomly choose a_i treatments from the i^{th} whole-plot such that $0 \leq a_i \leq (k-1)$ and $\sum_{i=1}^{w_0} a_i = v_0$, then replace those treatments with the k^{th} treatment of the corresponding whole-plot.

3. Compute the optimality criterion value and call it $\mathcal{D}^*$

4. Improve the Starting Design

   (a) Set $\kappa = 0$.

(b) Set $i = 1$.

(c) Set $j = 1$.

(d) Replace the HTC design point in the i^{th} whole-plot with the j^{th} candidate point in $\mathbf{A_w}$.

(e) Compute the optimality criterion value for the new design and call it $\mathcal{D}_0$.

i. Compute $\mathcal{D}$, the usual D criterion value

ii. Construct matrices $\mathbf{N}$, $\mathbf{R}$ and $\mathbb{C}_2$ to compute d_u and d_v and determine the values for $\mathcal{D}_u$ and $\mathcal{D}_v$

iii. Compute $\mathcal{D}_0 = \mathcal{D} \times \mathcal{D}_u \times \mathcal{D}_v$

(f) If $\mathcal{D}_0 > \mathcal{D}^*$, set $\mathcal{D}^* = \mathcal{D}_0$, $\kappa = 1$ and save this change.

(g) If $j < n_w$, set $j = j + 1$ and go back to Step 4.(d)

(h) Set $l = 1$.

(i) Set $j = 1$.

(j) Replace the ETC design point in the l^{th} sub-plot of the i^{th} whole-plot by the j^{th} point in $\mathbf{A_s}$.

(k) Compute the optimality criterion value for the new design and call it $\mathcal{D}_0$.

i. Compute $\mathcal{D}$, the usual D criterion value

ii. Construct matrices $\mathbf{N}$, $\mathbf{R}$ and $\mathbb{C}_2$ to compute d_u and d_v and determine the values for $\mathcal{D}_u$ and $\mathcal{D}_v$

iii. Compute $\mathcal{D}_0 = \mathcal{D} \times \mathcal{D}_u \times \mathcal{D}_v$

(l) If $\mathcal{D}_0 > \mathcal{D}^*$, set $\mathcal{D}^* = \mathcal{D}_0$, $\kappa = 1$ and save the change in the design.

(m) If $j < n_s$, set $j = j + 1$ and go back to Step 4.(j).

(n) If $l < k$, set $l = l + 1$ and go back to Step 4.(i).

(o) If $i < w_p$, set $i = i + 1$ and go back to Step 4.(c).

(p) If $\kappa > 0$, go back to Step 4.(a).

5. Repeat Step 2 to Step 4.(p) T times, where T is the number of total starting designs.

4.6 A Coordinate Exchange Algorithm

This algorithm is based on Jones & Goos algorithm and generates a starting design by randomly generating the required levels for the HTC and the ETC factors without using a set of candidate points. However, a random starting design generated by Jones & Goos algorithm does not always guarantee the required numbers of degrees of freedom for estimating the variance components. So, we modify their algorithm to generate required numbers of the degrees of freedom in a starting design. The improvement strategy is essentially the same as given in Jones & Goos algorithm except that we use a different optimality criterion.

4.6.1 Generating a Starting Design

For generating a starting design we need all the information given in section 4.2.1 and pre-specified numbers of degrees of freedom for estimating the variance components. Suppose that we want to generate a design with w_p whole-plots, fixed whole-plot size k and the pre-specified numbers of degrees of freedom for estimating the whole-plot and sub-pot error variances are u and v respectively. Then, we first generate a starting design by randomly selecting the levels of the ETC and the HTC factors as suggested by Jones & Goos. Then to generate the required number of degrees of freedom for estimating the whole-plot variance, we choose the i^{th} treatment of the j^{th} whole-plot where $1 \leq i \leq k$, $1 \leq j \leq w_p$ and i and j are chosen randomly. Then we randomly choose u whole-plots, excluding the j^{th} whole-plot from which the i^{th} treatment was chosen previously, and replace the i^{th} treatments in each of the u whole-plot by the previously selected treatment. So, one of the treatments now appears in at least $u + 1$ whole-plots. This will give us at least u degrees of freedom for estimating the whole-plot error variance.

Then, for generating the pre-specified number of degrees of freedom for estimating the sub-plot error variance, we replicate treatments within the whole-plots. For this, we randomly choose a_i treatments from each of the whole-plots, excluding the treatments that were previously replaced for generating the required numbers of degrees of freedom for estimating the whole-plot error variance, such that $0 \leq a_i \leq (k-1)$, $i = 1, 2, ..., w_p$ and $\sum_{i=1}^{w_p} a_i = v$. Then within each whole-plot, we replace the selected a_i treatments, by another treatment in the same whole-plot such that in each whole-plot one

of the treatments appears at least $a_i + 1$ times. This technique will give us at least v degrees of freedom for estimating the sub-plot error variance. Hence the starting design has at least the pre-specified numbers of degrees of freedom for obtaining the pure error estimates of the variance components.

Note that in this approach we first generate a random starting design and then, assuming that the random starting design does not have any degree of freedom available, use two different techniques for generating the pre-specified numbers of degrees of freedom for estimating the variance components. However, if a random starting design already has some degrees of freedom available, these techniques can generate more than the required numbers of degrees of freedom for estimating the variance components. This could possibly result in a singular starting design which does not have enough numbers of degrees of freedom for estimating the model parameters. So, to avoid this situation, we impose a condition in the algorithm that ensures that, in a starting design, there are enough degrees of freedom available for estimating the model parameters. This problem is more likely to occur in relatively small designs where n is not much greater than the number of the model parameters.

Here we give an example to show how a starting design is generated such that it has the pre-specified numbers of degrees of freedom for estimating the variance components.

4.6.2 An Example of a Starting Design

Here, we give a step-by-step example to show how our coordinate exchange algorithm generates a starting design with at least the required numbers of degrees of freedom.

Example 7 *Suppose we want to generate a second order RS Spit-plot design with* 2 *HTC and* 2 *ETC factors each with* 3 *levels, the number of whole-plots is* 9 *with a fixed whole-plot size* 3 *and required numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances are* 3 *and* 6 *respectively. Note that, we do not need a prior point estimate of* η *for generating a starting design. Although, we will need that when we compute the optimality criterion value of a starting design and also at the improvement stage when*

we make exchanges and compute the optimality criterion value for the new design.

Then, in the first step, we generate a random starting design (given in table 4.7) using Jones & Goos algorithm. The matrices $\mathbf{N}$, $\mathbf{R}$ *and* $\mathbb{C}_2$ *for this starting design are given as,*

Table 4.7: Step 1. Random starting design by Jones & Goos algorithm

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	1	1	-1	6	-1	0	0	-1
1	-1	1	-1	0	6	-1	0	0	0
1	-1	1	1	0	6	-1	0	-1	1
2	-1	0	0	-1	7	0	0	-1	1
2	-1	0	-1	-1	7	0	0	0	-1
2	-1	0	1	1	7	0	0	0	0
3	1	1	0	0	8	0	-1	1	-1
3	1	1	-1	-1	8	0	-1	0	1
3	1	1	1	1	8	0	-1	0	0
4	1	0	0	-1	9	0	0	1	1
4	1	0	1	-1	9	0	0	1	1
4	1	0	1	1	9	0	0	1	0
5	-1	1	0	-1					
5	-1	1	0	0					
5	-1	1	1	1					

$$\mathbf{N} = \begin{pmatrix}
1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & 1 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\
0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 2 \\
0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1
\end{pmatrix},$$

$$\mathbf{R} = diag\begin{pmatrix} 1 & 1 & 1 & 2 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 2 & 1 \end{pmatrix},$$

and

$$\mathbb{C}_2 = \begin{pmatrix}
0 & 0.0 & 0 & 0 & 0 & 0.0 & 0 & 0 & 0 \\
0 & 0.5 & 0 & 0 & 0 & -0.5 & 0 & 0 & 0 \\
0 & 0.0 & 0 & 0 & 0 & 0.0 & 0 & 0 & 0 \\
0 & 0.0 & 0 & 0 & 0 & 0.0 & 0 & 0 & 0 \\
0 & 0.0 & 0 & 0 & 0 & 0.0 & 0 & 0 & 0 \\
0 & -0.5 & 0 & 0 & 0 & 0.5 & 0 & 0 & 0 \\
0 & 0.0 & 0 & 0 & 0 & 0.0 & 0 & 0 & 0 \\
0 & 0.0 & 0 & 0 & 0 & 0.0 & 0 & 0 & 0 \\
0 & 0.0 & 0 & 0 & 0 & 0.0 & 0 & 0 & 0
\end{pmatrix}.$$

Here $n = 27$, $t = 25$ *and* $rank(\mathbb{C}_2) = 1$. *So the starting design gives* 1 *degree of freedom for estimating the whole-plot error variance and* 1 *degree of freedom for estimating the sub-plot error variance. Hence the available numbers of degrees of freedom for estimating the variance components are less than the required numbers.*

Then, in the second step, we generate the required numbers of degrees of freedom for estimating the whole-plot error variance by randomly choosing a treatment from a whole-plot and then replicating that treatment in 3 *other whole-plots. For our example, we randomly chose the first treatment of the* 5^{th} *whole-plot and then we randomly chose whole-plot number* 1, 4 *and* 9 *and the first treatment in each of these whole-plots is replaced by the first treatment of whole-plot number* 5. *This technique would give us at least* 3 *degrees of freedom for estimating the whole-plot error variance. The design at this stage is given in table 4.8 and matrices* $\mathbf{N}$, $\mathbf{R}$ *and* $\mathbb{C}_2$ *for this design are given as*

Table 4.8: Step 2. Replicating a treatment between whole-plots for generating degrees of freedom for estimating the whole-plot error variance.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	1	0	-1	6	-1	0	0	-1
1	-1	1	-1	0	6	-1	0	0	0
1	-1	1	1	0	6	-1	0	-1	1
2	-1	0	0	-1	7	0	0	-1	1
2	-1	0	-1	-1	7	0	0	0	-1
2	-1	0	1	1	7	0	0	0	0
3	1	1	0	0	8	0	-1	1	-1
3	1	1	-1	-1	8	0	-1	0	1
3	1	1	1	1	8	0	-1	0	0
4	-1	1	0	-1	9	-1	1	0	-1
4	-1	1	1	-1	9	-1	1	1	1
4	-1	1	1	1	9	-1	1	1	0
5	-1	1	0	-1					
5	-1	1	0	0					
5	-1	1	1	1					

$$\mathbf{N} = \begin{pmatrix} 1 & 0 & 0 & 1 & 1 & 0 & 0 & 0 & 1 \\ 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \\ 0 & 1 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 1 & 0 & 0 & 0 & 1 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \end{pmatrix},$$

$$\mathbf{R} = diag\begin{pmatrix} 4 & 1 & 2 & 2 & 1 & 1 & 1 & 1 & 1 & 1 & 3 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 \end{pmatrix},$$

$$\mathbb{C}_2 = \begin{pmatrix} 1.25 & 0.0 & 0 & -0.25 & -0.25 & 0.0 & 0 & 0 & -0.75 \\ 0.00 & 0.5 & 0 & 0.00 & 0.00 & -0.5 & 0 & 0 & 0.00 \\ 0.00 & 0.0 & 0 & 0.00 & 0.00 & 0.0 & 0 & 0 & 0.00 \\ -0.25 & 0.0 & 0 & 1.42 & -0.58 & 0.0 & 0 & 0 & -0.58 \\ -0.25 & 0.0 & 0 & -0.58 & 1.42 & 0.0 & 0 & 0 & -0.58 \\ 0.00 & -0.5 & 0 & 0.00 & 0.00 & 0.5 & 0 & 0 & 0.00 \\ 0.00 & 0.0 & 0 & 0.00 & 0.00 & 0.0 & 0 & 0 & 0.00 \\ 0.00 & 0.0 & 0 & 0.00 & 0.00 & 0.0 & 0 & 0 & 0.00 \\ -0.75 & 0.0 & 0 & -0.58 & -0.58 & 0.0 & 0 & 0 & 1.92 \end{pmatrix}.$$

Here rank$(\mathbb{C}_2) = 4$. *So we now have the required numbers of degrees of freedom for estimating the whole-plot error variance.*

Next, we generate the required number of degrees of freedom for estimating the sub-plot error variance by replicating treatments within the whole-plots. The required number of degrees of freedom for estimating the sub-plot error variance is 6. *So we randomly choose* a_i *treatments from each of the whole-plots such that* $0 \leq a_i \leq 2$, $i = 1, 2, ..., w_p$, *and* $\sum_{i=1}^{w_p} a_i = 6$ *and then these treatments are replaced by the first treatment of the corresponding whole-plot. In our example we select* $2, 2, 1, 1$ *treatments from whole-plot number* $1, 4, 5$ *and* 9 *respectively and replace these treatments by the first treatment in each whole-plot. This would give us at least* 6 *degrees of freedom for estimating the sub-plot error variance. The design at this final stage is given in table 4.9 and the matrices* $\mathbf{N}$, $\mathbf{R}$ *and* $\mathbb{C}_2$ *are given as*

Table 4.9: Step 3. The starting design at the final stage.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	1	0	-1	6	-1	0	0	-1
1	-1	1	0	-1	6	-1	0	0	0
1	-1	1	0	-1	6	-1	0	-1	1
2	-1	0	0	-1	7	0	0	-1	1
2	-1	0	-1	-1	7	0	0	0	-1
2	-1	0	1	1	7	0	0	0	0
3	1	1	0	0	8	0	-1	1	-1
3	1	1	-1	-1	8	0	-1	1	-1
3	1	1	1	1	8	0	-1	0	0
4	-1	1	0	-1	9	-1	1	0	-1
4	-1	1	0	-1	9	-1	1	0	-1
4	-1	1	0	-1	9	-1	1	1	0
5	-1	1	0	-1					
5	-1	1	0	-1					
5	-1	1	1	1					

$$\mathbf{N} = \begin{pmatrix} 3 & 0 & 0 & 3 & 2 & 0 & 0 & 0 & 2 \\ 0 & 1 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 2 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0 & 0 & 1 \end{pmatrix},$$

$$\mathbf{R} = diag\begin{pmatrix} 10 & 2 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 2 & 1 & 1 \end{pmatrix},$$

and

$$\mathbb{C}_2 = \begin{pmatrix} 2.1 & 0.0 & 0 & -0.9 & -0.6 & 0.0 & 0 & 0 & -0.6 \\ 0.0 & 0.5 & 0 & 0.0 & 0.0 & -0.5 & 0 & 0 & 0.0 \\ 0.0 & 0.0 & 0 & 0.0 & 0.0 & 0.0 & 0 & 0 & 0.0 \\ -0.9 & 0.0 & 0 & 2.1 & -0.6 & 0.0 & 0 & 0 & -0.6 \\ -0.6 & 0.0 & 0 & -0.6 & 1.6 & 0.0 & 0 & 0 & -0.4 \\ 0.0 & -0.5 & 0 & 0.0 & 0.0 & 0.5 & 0 & 0 & 0.0 \\ 0.0 & 0.0 & 0 & 0.0 & 0.0 & 0.0 & 0 & 0 & 0.0 \\ 0.0 & 0.0 & 0 & 0.0 & 0.0 & 0.0 & 0 & 0 & 0.0 \\ -0.6 & 0.0 & 0 & -0.6 & -0.4 & 0.0 & 0 & 0 & 1.6 \end{pmatrix}.$$

Here $rank(\mathbb{C}_2) = 4$, $t = 16$. *So,*

$$d_w = rank(\mathbb{C}_2) = 4,$$

and

$$d_s = n - t - rank(\mathbb{C}_2) = 27 - 16 - 4 = 7.$$

Thus, the starting design has the pre-specified numbers of degrees of freedom for obtaining the pure error estimates of the variance components.

4.6.3 Improving the Starting Design

First, the optimal criterion value for the starting design is computed. Then an improvement in the optimality criterion value is searched by replacing each factor level by all the values, listed in the set of candidate values for those factor levels, one by one. After each replacement, the optimality criterion value is calculated and if there is an improvement the new design replaces the previous design otherwise no change is made. The improvement process is performed whole-plot-by-whole-plot i.e. first all the levels in the first whole-plot are replaced by all the candidate values for those levels one by one and an improvement in the optimality criterion value is searched and, if found, saved. Then the improvement process is performed for the second, third, and so on, for the last whole-plot. Within a whole-plot, the improvement process starts from the first HTC factor and continues until the level of the last HTC factor is replaced by all the candidate values. Then the improvement is searched for the first ETC factor in the first sub-plot and this search continues until the level of the last ETC factor in the first sub-plot is replaced by all the candidate values. Then, using the same technique, the improvement is searched in the second, third, and so on, in the k sub-plot of the whole-plot. One round of the improvement process completes when all the levels, from the first HTC in the first whole-plot to the last ETC factor in the last experimental run, are replaced by their candidate values. If at least a single improvement is recorded in one round, the improvement process is repeated again. Thus, the improvement process continues until there is no further increase in the optimality criterion value.

This two stage procedure, first generating a starting design and then improve it, is repeated for several random starting designs and the best design in terms of the optimality criterion value is chosen at the end.

4.6.4 The Algorithm

For our algorithm we need all the information mentioned in section 4.6.1 as the input information. Then a D-optimal design with the pre-specified numbers of degrees of freedom for estimating the variance components can be generated by following these steps.

1. Generate a random starting design.

2. Randomly choose t_{ij}, where t_{ij} denotes treatment i in whole-plot j. Then randomly choose u other whole-plots, excluding the j^{th} whole-plot chosen before, and in each of the u whole-plots replace the i^{th} treatment by t_{ij}.

3. Randomly chose a_l treatments from whole-plot l, excluding the i^{th} treatment, such that $0 \leq a_l \leq (k-1)$, $l = 1, 2, ..., w_p$, and $\sum_{l=1}^{w_p} a_l = v$ and replace these treatments by the i^{th} treatments of the corresponding whole-plot.

4. Compute d_u and d_v. If $u \leq d_u \leq (w_p - p_w - 1)$ and $v \leq d_v \leq (n - p - d_u)$ then go step 5 otherwise go back to step 1.

5. Compute the optimality criterion value ($\mathcal{D}^*$).

6. Improve the starting design.

7. Repeat step $1 - 6$ T times.

Note that, in the above algorithm u and d_u respectively denote the pre-specified and available number of degrees of freedom in the design for estimating the whole-plot error variance, v and d_v respectively denotes the pre-specified and available number of degrees of freedom in the design for estimating the sub-plot error variance, T denotes the total number of starting designs and subscript i has a fixed value in steps 2 and 3.

As stated earlier, our algorithm is a modified form of Jones & Goos algorithm. So in step 1 and 6 of our algorithm, where we generate a random starting design and improve it, we basically follow the same procedure as given by Jones & Goos and described in fig 4.1 except that in step 6 we compute the value for the optimality criterion given in (4.1). For each coordinate exchange, we not only compute the usual D criterion value for the new design but we also compute the available numbers of degrees of freedom, for estimating the pure error variance components, in the new design by constructing the $\mathbb{C}_2$ matrix.

4.7 Computational results

In this section, we consider some of the design problems already given in the literature for generating a second order RS split-plot designs. We use our

algorithm to generate a design for each of these design problems and then make comparison between the design generated by our algorithm and the design generated by a different methodology. We have chosen three different designs generated by three different approaches for constructing RS split-plot designs. We give the original design and then we generate another design for the same design problem using our design methodology.

4.7.1 A D-Optimal Design

Macharia & Goos (2010)[22] gave a second order D-optimal design with one HTC factor and two ETC factors each at three levels. The design has 5 whole-plots and the fixed whole-plot size is 3. Their design is given in table 4.10.

Table 4.10: A D-optimal design for 1 HTC and 2 ETC factors given by Macharia & Goos (2010)

whole plot	W_1	S_1	S_2
1	-1	-1	0
1	-1	0	1
1	-1	1	-1
2	-1	-1	1
2	-1	-1	-1
2	-1	1	0
3	0	-1	1
3	0	0	0
3	0	1	1
4	1	-1	-1
4	1	0	1
4	1	1	-1
5	1	-1	1
5	1	0	-1
5	1	1	1

This design was generated for $\eta = 1$, where $\eta = \sigma_\gamma^2/\sigma_\epsilon^2$, and gives 0 degrees of freedoms for estimating the whole-plot or sub-plot pure error variances. Using this design as a benchmark, we generated different designs to

see that to what extent the optimality of a design was affected if we forced it to generate some degrees of freedom for obtaining the pure error estimates of the variance components. We generated 15 designs using the same value for η that was used to generate the benchmark design but with different pre-specified numbers of degrees of freedom and checked their D-efficiency. These designs were generated using the statistical software package *R*. To compare the computing time, we used both of the algorithms (the point exchange algorithm and the coordinate exchange algorithm) for generating each design and for each design we used 5000 iterations. The point exchange algorithm executed 5000 iterations in approximately 4 hours whereas the coordinate exchange algorithm took around 2 hours and 30 minutes. So, the coordinate exchange algorithm worked much faster than the point exchange algorithm. The relative D-efficiencies of the designs as compared to the optimal design are given in table 4.16 and the designs are given in tables 4.11 to 4.15.

The results in table 4.16 show that the optimality criterion value (and hence the relative D-efficiency) of a design decreases as we increase the numbers of degrees of freedom for estimating the variance components. But the increase in the sub-plot error degrees of freedom causes more decrease in the optimality criterion value than the increase in the whole-plot error degrees of freedom. For example, we compare design 6 and 8 with design 4. Design 4 was generated for $u = v = 1$. The relative D-efficiency of this design (as compared to the benchmark design) is 98.02%. Design 8 has $v = 2$ and $u = 1$, so this design has one more degree of freedom for estimating the sub-plot error variance as compared to design 4. With a single increase in v the relative D-efficiency decreases to 93.36%. Design 6 has $v = 1$ and $u = 2$. So, this design has the same v as compared to the design 1 but has one more degree of freedom for estimating the whole-plot error variance. The relative D-efficiency for this design, as compared to the design 1 is 95.62% which is greater than the relative D-efficiency of design 8. The same trend can be seen if we compare other designs in the same pattern. The relative D-efficiency decreases more with a single degree of freedom increase in v as compared to the single degree of freedom increase in u. Finally, we compare designs 12, 14 and 15. These three designs have the maximum possible number of degrees of freedom for estimating the variance components i.e. $u + v = 5$. Among these three designs, design with the maximum possible number of whole-plot error degrees of freedom, design 12, has the greater relative D-efficiency as compared to the other two designs whereas the design with the maximum

Table 4.11: D-optimal designs with pre-specified numbers of degrees of freedom for estimating the variance components. Here, u and v denote the numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances respectively.

	$u=0, v=0$			$u=1, v=0$			$u=2, v=0$		
whole plot	W_1	S_1	S_2	W_1	S_1	S_2	W_1	S_1	S_2
1	-1	-1	1	-1	-1	1	-1	-1	1
1	-1	0	-1	-1	0	-1	-1	1	-1
1	-1	1	1	-1	1	1	-1	1	1
2	-1	-1	-1	-1	-1	0	-1	-1	-1
2	-1	0	1	-1	0	1	-1	0	0
2	-1	1	-1	-1	1	-1	-1	1	1
3	0	-1	0	0	-1	-1	0	0	1
3	0	0	1	0	0	1	0	-1	-1
3	0	1	-1	0	1	0	0	1	0
4	1	-1	-1	1	-1	1	1	-1	0
4	1	-1	1	1	1	-1	1	1	-1
4	1	1	0	1	1	1	1	1	1
5	1	-1	0	1	-1	-1	1	-1	1
5	1	1	-1	1	0	0	1	0	-1
5	1	1	1	1	1	1	1	1	1

Table 4.12: D-optimal designs with pre-specified numbers of degrees of freedom for estimating the variance components. Here, u and v denote the numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances respectively.

	$u=0, v=1$			$u=0, v=2$			$u=0, v=3$		
whole plot	W_1	S_1	S_2	W_1	S_1	S_2	W_1	S_1	S_2
1	-1	-1	-1	-1	-1	-1	-1	-1	-1
1	-1	1	1	-1	1	1	-1	1	1
1	-1	1	1	-1	1	1	-1	1	1
2	-1	0	0	-1	-1	1	-1	-1	1
2	-1	-1	1	-1	0	0	-1	-1	1
2	-1	1	-1	-1	1	-1	-1	1	-1
3	0	0	1	0	0	1	0	-1	0
3	0	-1	-1	0	0	1	0	0	1
3	0	1	0	0	1	0	0	0	1
4	1	-1	1	1	-1	0	1	-1	-1
4	1	-1	-1	1	0	-1	1	0	0
4	1	1	-1	1	1	1	1	1	-1
5	1	-1	0	1	-1	-1	1	-1	1
5	1	0	-1	1	-1	1	1	0	-1
5	1	1	1	1	1	-1	1	1	1

Table 4.13: D-optimal designs with pre-specified numbers of degrees of freedom for estimating the variance components. Here, u and v denote the numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances respectively.

	$u=0, v=4$			$u=0, v=5$			$u=1, v=1$		
whole plot	W_1	S_1	S_2	W_1	S_1	S_2	W_1	S_1	S_2
1	-1	-1	1	-1	-1	0	-1	0	1
1	-1	0	0	-1	0	1	-1	-1	-1
1	-1	1	-1	-1	0	1	-1	1	0
2	-1	-1	-1	-1	0	-1	-1	1	-1
2	-1	1	1	-1	1	0	-1	0	0
2	-1	1	1	-1	1	0	-1	-1	1
3	0	0	-1	0	0	0	0	1	1
3	0	0	-1	0	0	0	0	-1	0
3	0	1	0	0	1	1	0	0	-1
4	1	-1	1	1	-1	1	1	1	-1
4	1	-1	1	1	-1	1	1	-1	1
4	1	1	-1	1	-1	-1	1	-1	-1
5	1	-1	-1	1	1	-1	1	-1	-1
5	1	-1	-1	1	1	1	1	1	1
5	1	1	1	1	1	-1	1	-1	1

Table 4.14: D-optimal designs with pre-specified numbers of degrees of freedom for estimating the variance components. Here, u and v denote the numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances respectively.

	$u=1, v=2$			$u=1, v=3$			$u=1, v=4$		
whole plot	W_1	S_1	S_2	W_1	S_1	S_2	W_1	S_1	S_2
1	-1	1	-1	-1	-1	1	-1	1	-1
1	-1	-1	0	-1	-1	1	-1	-1	-1
1	-1	1	1	-1	1	-1	-1	1	1
2	-1	1	0	-1	-1	-1	0	-1	1
2	-1	0	1	-1	1	1	0	0	-1
2	-1	-1	-1	-1	0	0	0	1	0
3	0	0	0	0	0	-1	0	-1	1
3	0	-1	1	0	0	-1	0	0	-1
3	0	0	0	0	-1	0	0	1	0
4	1	1	1	1	-1	-1	1	-1	-1
4	1	-1	-1	1	-1	1	1	0	0
4	1	1	-1	1	1	-1	1	0	0
5	1	-1	1	1	-1	1	1	1	1
5	1	-1	-1	1	1	1	1	1	-1
5	1	1	-1	1	-1	-1	1	1	1

Table 4.15: D-optimal designs with pre-specified numbers of degrees of freedom for estimating the variance components. Here, u and v denote the numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances respectively.

	$u = 2, v = 1$			$u = 2, v = 2$			$u = 2, v = 3$		
whole plot	W_1	S_1	S_2	W_1	S_1	S_2			
1	-1	-1	-1	-1	1	1	-1	-1	-1
1	-1	1	0	-1	0	0	-1	-1	1
1	-1	0	1	-1	-1	-1	-1	1	0
2	-1	1	0	-1	0	0	-1	-1	-1
2	-1	0	-1	-1	1	-1	-1	-1	1
2	-1	-1	1	-1	-1	1	-1	1	0
3	0	0	0	0	0	1	0	-1	0
3	0	1	1	0	0	1	0	0	1
3	0	1	-1	0	1	0	0	1	-1
4	1	-1	-1	1	-1	1	1	-1	-1
4	1	-1	1	1	1	1	1	0	0
4	1	1	1	1	-1	-1	1	1	1
5	1	-1	-1	1	-1	1	1	-1	1
5	1	-1	1	1	1	-1	1	0	0
5	1	1	-1	1	1	1	1	1	1

Table 4.16: Relative D-efficiency for different designs generated for pre-specified numbers of degrees of freedom for estimating the variance components. Here, u and v denote the numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances respectively.

Design	v	u	$D_{R.E}$
1	0	0	100.31%
2	0	1	99.74%
3	0	2	98.25%
4	1	1	98.02%
5	1	0	97.20%
6	1	2	95.62%
7	2	2	93.61%
8	2	1	93.36%
9	2	0	93.25%
10	3	1	89.66%
11	3	0	89.41%
12	3	2	86.11%
13	4	0	83.57%
14	4	1	77.15%
15	5	0	65.60%

possible number of sub-plot error degrees of freedom, design 15, has the minimum relative D-efficiency.

Although all the designs, given in this section, were generated using $\eta = 1$, we have done a robustness study of these designs for different values of η. We generated designs for $\eta = 5$ and $\eta = 10$ and all the designs (design 2 to 15 but not design 1) are robust to these changes in the η value. Some other similar robustness studies for RS split-plot designs are given by Goos & Vandebroek (2001)[14] and Goos & Vandebroek (2003)[15].

4.7.2 The Strength of the Ceramic Pipe Experiment

Vining et al (2005)[28] gave an example involving ceramic pipes. An engineer wishes to estimate the relation of the strength of ceramic pipes (response variable) with some other factors of interest (the explanatory variables). Those factors are temperature 1, temperature 2, amount of binder in the formulation and grinding speed of the batch. Among these four factors, temperature 1 (W_1) and temperature 2 (W_2) are the HTC factors and amount of binder in the formulation (S_1) and grinding speed of the batch (S_2) are the ETC factors. The authors present a second order RS split-plot design with 12 whole-plots and a fixed whole-plot size of 4 for performing the experiment. The design is given in table 4.17.

Some interesting properties of this design are that the OLS and GLS estimates of the regression coefficients are identical and the design provides 2 degrees of freedom for obtaining pure error estimates for the whole-plot error variance and 21 degrees of freedom for sub-plot error variance. They estimated $\sigma^2_\epsilon = 0.09348$ and $\sigma^2_\gamma = 0.52828$.

We used our coordinate algorithm to generate two different designs for this design problem using the same values of the variance components as estimated by Vining et al (2005). For each design, we used 5000 iterations and our programme (written in R language and run on a laptop machine) took approximately 24 hours to execute 5000 iterations. These designs are given in tables 4.18 and 4.19. The design given in table 4.18 gives 4 degrees of freedom for estimating the whole-plot error variance and 21 for estimating the sub-plot error variance. So, our design has more degrees of freedom for obtaining the pure error estimates of the variance components as given in

Table 4.17: An equivalent estimation design for 2 HTC and 2 ETC factors given by Vining et al (2005)

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	-1	-1	-1	6	0	-1	0	0
1	-1	-1	1	-1	6	0	-1	0	0
1	-1	-1	-1	1	6	0	-1	0	0
1	-1	-1	1	1	6	0	-1	0	0
2	1	-1	-1	-1	7	0	1	0	0
2	1	-1	1	-1	7	0	1	0	0
2	1	-1	-1	1	7	0	1	0	0
2	1	-1	1	1	7	0	1	0	0
3	-1	1	-1	-1	8	0	0	-1	0
3	-1	1	1	-1	8	0	0	1	0
3	-1	1	-1	1	8	0	0	0	-1
3	-1	1	1	1	8	0	0	0	1
4	1	1	-1	-1	9	0	0	0	0
4	1	1	1	-1	9	0	0	0	0
4	1	1	-1	1	9	0	0	0	0
4	1	1	1	1	9	0	0	0	0
5	-1	0	0	0	10	0	0	0	0
5	-1	0	0	0	10	0	0	0	0
5	-1	0	0	0	10	0	0	0	0
5	-1	0	0	0	10	0	0	0	0
6	1	0	0	0	12	0	0	0	0
6	1	0	0	0	12	0	0	0	0
6	1	0	0	0	12	0	0	0	0
6	1	0	0	0	12	0	0	0	0

the design proposed by Vining et al (2005). But the relative D-efficiency of our design is 166.46% when compared to their design. The second design given in table 4.19 gives 6 degrees of freedom (maximum possible) for estimating the whole-plot error variance and 21 degrees of freedom for estimating the sub-plot error variance. The relative D-efficiency of our design, with the maximum possible number of degrees of freedom for estimating the whole-plot error variance, is 173.84% when compared to the original design. One might think that the equivalent estimation design has an extra quality of equivalence of OLS and GLS estimates that our design does not have but this property might not be very appealing for an experimenter who is interested in more efficient use of resources. Furthermore, as we have mentioned before, in order to draw inferences about the model parameters one would have to estimate the variance components because the variance-covariance matrix of the model estimates depends on the variance components.

4.7.3 The Freeze-Dried Coffee Experiment

Gilmour et al (2000)[12] reported the dried coffee experiment that is described in Example 3 of section 1.3. The design suggested by the authors is given in table 2.2. This design, generated by using the algorithm by Trinca & Gilmour (2001)[27], gives 3 degrees of freedom for estimating the whole-plot error variance and 0 degrees of freedom for estimating the sub-plot error variance, under the randomization based method. We used our coordinate exchange algorithm to generate two different designs (with different pre-specified numbers of degrees of freedom for estimating the variance components) for the same design problem. For each design we used $\eta = 1$ and run 5000 iterations. Our algorithm (written is R language and executed on a laptop machine) took around 26 hours to run 5000 iterations. For the first design, the required numbers of degrees of freedom for estimating the whole-plot and sub-plot error variances are 3 and 3 respectively and for second design the required numbers are 3 and 4. The two designs are given in table 4.20. The Relative D-efficiency for the first design with respect to the design given by Gilmour et al (2000)[12] is 140.52% and the relative D-efficiency for the second design is 139.96%. This should not be a surprise because their design was not constructed by using the D-optimality criterion.

Table 4.18: A D-optimal design for 2 HTC and 2 ETC factors with $u = 4$ and $v = 21$.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	-1	1	1	7	0	1	-1	1
1	-1	-1	1	-1	7	0	1	-1	1
1	-1	-1	-1	-1	7	0	1	-1	1
1	-1	-1	-1	1	7	0	1	1	0
2	-1	-1	1	-1	8	1	-1	-1	-1
2	-1	-1	1	1	8	1	-1	-1	1
2	-1	-1	-1	1	8	1	-1	1	1
2	-1	-1	-1	-1	8	1	-1	1	-1
3	-1	0	-1	1	9	1	-1	1	-1
3	-1	0	-1	1	9	1	-1	1	1
3	-1	0	-1	1	9	1	-1	-1	-1
3	-1	0	-1	1	9	1	-1	-1	1
4	-1	1	-1	-1	10	1	0	0	-1
4	-1	1	0	0	10	1	0	0	-1
4	-1	1	0	0	10	1	0	0	-1
4	-1	1	1	1	10	1	0	-1	0
5	-1	1	1	1	11	1	1	1	-1
5	-1	1	0	0	11	1	1	1	1
5	-1	1	1	-1	11	1	1	0	1
5	-1	1	-1	-1	11	1	1	-1	-1
6	0	0	0	-1	12	1	1	0	1
6	0	0	-1	0	12	1	1	-1	-1
6	0	0	-1	0	12	1	1	1	-1
6	0	0	-1	0	12	1	1	1	1

Table 4.19: A D-optimal design for 2 HTC and 2 ETC factors with $u = 6$ and $v = 21$.

whole plot	W_1	W_2	S_1	S_2	whole plot	W_1	W_2	S_1	S_2
1	-1	-1	1	1	7	0	1	0	-1
1	-1	-1	-1	-1	7	0	1	0	-1
1	-1	-1	0	0	7	0	1	-1	0
1	-1	-1	1	-1	7	0	1	1	1
2	-1	-1	-1	-1	8	1	-1	-1	-1
2	-1	-1	0	0	8	1	-1	1	-1
2	-1	-1	1	-1	8	1	-1	1	1
2	-1	-1	1	1	8	1	-1	-1	1
3	-1	0	-1	1	9	1	-1	1	-1
3	-1	0	-1	1	9	1	-1	-1	-1
3	-1	0	1	0	9	1	-1	1	1
3	-1	0	-1	-1	9	1	-1	-1	1
4	-1	0	1	0	10	1	-1	-1	1
4	-1	0	-1	-1	10	1	-1	1	-1
4	-1	0	1	0	10	1	-1	1	1
4	-1	0	-1	1	10	1	-1	-1	-1
5	-1	1	0	1	11	1	1	1	1
5	-1	1	-1	0	11	1	1	-1	-1
5	-1	1	0	1	11	1	1	-1	1
5	-1	1	1	-1	11	1	1	1	-1
6	0	1	1	1	12	1	1	-1	-1
6	0	1	0	-1	12	1	1	-1	1
6	0	1	-1	0	12	1	1	1	-1
6	0	1	-1	0	12	1	1	1	1

Table 4.20: Two new designs with pre-specified numbers of degrees of freedom for estimating the variance components for the Dried Coffee experiment

	$u = 3$, $v = 3$					$u = 3$, $v = 4$				
whole plot	W_1	S_1	S_2	S_3	S_4	W_1	S_1	S_2	S_3	S_4
1	-1	-1	-1	-1	-1	-1	-1	-1	1	-1
1	-1	-1	-1	1	1	-1	1	1	1	-1
1	-1	-1	1	-1	1	-1	-1	1	1	1
1	-1	-1	1	1	-1	-1	-1	1	-1	-1
1	-1	1	1	1	1	-1	1	-1	1	1
2	-1	-1	1	-1	1	0	1	-1	0	0
2	-1	1	-1	-1	1	0	0	1	0	-1
2	-1	1	1	1	1	0	0	0	-1	-1
2	-1	1	-1	1	-1	0	-1	0	1	0
2	-1	1	1	-1	-1	0	-1	1	-1	1
3	0	-1	1	-1	-1	-1	1	-1	1	1
3	0	0	1	0	0	-1	1	-1	-1	-1
3	0	-1	-1	1	0	-1	1	1	-1	1
3	0	-1	0	0	1	-1	-1	1	-1	-1
3	0	1	-1	-1	0	-1	-1	-1	-1	1
4	0	0	0	-1	1	1	-1	-1	-1	-1
4	0	0	1	0	0	1	1	0	1	-1
4	0	-1	-1	1	0	1	0	1	-1	0
4	0	-1	1	-1	-1	1	1	-1	-1	1
4	0	1	-1	0	-1	1	1	1	1	1
5	1	-1	0	0	0	1	-1	-1	1	1
5	1	0	-1	-1	-1	1	-1	1	1	-1
5	1	1	1	1	-1	1	1	1	-1	-1
5	1	1	-1	1	1	1	1	1	1	1
5	1	1	1	-1	1	1	0	-1	1	-1
6	1	-1	-1	-1	1	1	-1	-1	1	1
6	1	-1	-1	1	-1	1	-1	1	1	-1
6	1	1	-1	1	1	1	-1	0	0	1
6	1	1	0	-1	-1	1	1	0	1	-1
6	1	-1	1	1	1	1	-1	-1	-1	-1

Chapter 5

Concluding Remarks

in this thesis, we noted that for many industrial experiments the interest lies in getting some understanding of a process and (or) its optimization. These objectives are often achieved by estimating the relationship between the input (factors) and the output (response) of that process through a linear regression model. A second order linear regression model is often used for this purpose. Most of the designs of experiments available to cope with this situation assume that for every experimental run each factor level is re-set. But there might be a situation where it is not possible for some of the input factors to change their level settings after each experimental runs. Such factors are called the Hard-to-Change factors and the others are called the Easy-to-Change factors. So, a Split-plot type experimental structure is then used when the levels of the HTC factors are applied to large experimental units and the levels of the ETC factors are applied to small experimental units such that, for several consecutive runs, the levels of the HTC factors remain constant forming the whole-plots of the experiment while the levels of the ETC factors can change from run to run forming the sub-plots of the experiment. The corresponding model in this case is the linear mixed model.

We further discussed that the variance components of that model are used to estimate the model parameters and also to draw inferences about the model parameters. So, the estimation of the variance components plays an important role. Different estimation techniques are available in the literature for estimating the variance components. A general approach is to use REML, however, as pointed out by some authors, it might not work well for non-orthogonal Split-plot design with only a small number of whole-plots,

because in that case there are often no degrees of freedom available for estimating the whole-plot error variance and hence it is estimated as 0 which can lead to erroneous results. This leads us to think of a better alternative that is called the randomization based approach. This approach gives us the unbiased pure error estimates of the variance components using a full treatment model and, hence, can be used to check the lack of fit of the response surface model.

We also discussed the role of the numbers of degrees of freedom for estimating the variance components. The estimates obtained by using 5 or more degrees of freedom are generally considered more reliable then those obtained by using only a few degrees of freedom. Also, the numbers of degrees of freedom play a role in testing the significance of the model parameters. So, it is recommended to use appropriate numbers of degrees of freedom for estimating the variance components.

Then we looked at some of the design construction methodologies already available in the literature for constructing RS Split-plot designs. The equivalent estimation design methodology allows the equivalence of the OLS and the GLS estimates of the model parameters. Two different techniques, for constructing equivalent estimation designs are available. The VKM design methodology gives the pure error estimates of the variance components but constructs inefficient designs. The MWP design methodology constructs designs with the minimum number of whole-plots but does not provide pure error estimates of the variance components. D-optimal, D-efficient equivalent estimation and a stratum-by-stratum design construction methodologies generate efficient designs but rarely allow the estimation of the pure error variance components. We noted that none of these design methodologies allows the estimation of the pure error variance components with the prespecified numbers of degrees of freedom. So, we have discussed the need of a design construction methodology that can allow the pure error estimates of the variance components with the pre-specified numbers of degrees of freedom but with the efficient use of the resources.

We described that, by ignoring the treatment structure, Split-plot designs can be seen as general incomplete block designs where the whole-plots now serve as blocks. We found some standard results in the literature, known as Yates' procedure, for obtaining the pure error estimates of the variance com-

ponents of the model for general Incomplete block designs with the related numbers of degrees of freedom. We used and modified those results to develop a new design construction methodology that would generate a second order D-optimal RS Split-plot design such that it has the pre-specified numbers of degrees of freedom for estimating the pure error variance components.

We presented two different computer based search algorithms for generating second order D-optimal RS Split-plot designs with the pre-specified numbers of degrees of freedom for estimating the pure error variance components. The first algorithm is a point exchange algorithm while the second one is a coordinate exchange algorithm. The point exchange algorithm first generates a starting design and then improves the starting design using a set of candidate points for the HTC factors and one for the ETC factors. The coordinate exchange algorithm generates a starting design by randomly generating all the required levels and then improves the starting design using the set of candidate values for factor levels.

Then, we used our design methodology to generate designs for some of the design problems already available in the literature and compare our design with the previously generated designs. When compared to a D-optimal design, we found that the relative D-efficiency of the design decreased when we increased the numbers of degrees of degrees of freedom for estimating the variance components. However, the increase in the number of sub-plot error degrees of freedom caused more decrease in the relative D-efficiency of the design then the increase in the number of whole-plot error degrees of freedom. Sometimes, an increase in the number of whole-plot error degrees of freedom resulted in an increase in the relative D-efficiency of the design. Although, the designs, generated by our algorithms with different pre-specified numbers of degrees of freedom for estimating the variance components, were generated for $\eta = 1$, we checked their robustness to different values of η and we found that these designs were robust for $\eta = 5$ and also for $\eta = 10$.

Comparison with an equivalent estimation design showed that our algorithm generated a much more efficient design with a greater number of degrees of freedom for estimating the variance components. Although our design did not allow the equivalence of the OLS and the GLS estimates of the model parameters, we noticed that one had to estimate the variance components in order to draw inferences about the model parameters. In that

case, the equivalence property might not be very appealing for an experimenter who is interested in the efficient use of the resources. Comparison with a design constructed by a stratum-by-stratum design methodology also revealed a greater relative D-efficiency for our design with a greater numbers of degrees of freedom for estimating the variance components, however, the stratum-by-stratum design was not constructed using the D-optimality criterion.

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