CHAPTER 20

Interpretation of a Fitted Model

A mechanistic model is usually derived on the assumption that changes in the operating variables cause changes in the response variable. An empirical model fitted to process data also provides a description of the relationship between changes in selected operating variables and corresponding changes in the response variable but this relationship may not be a cause and effect relationship. Operating data can display systematic associations that defy logical explanation. Sometimes such relationships can be produced by what Box [1] has called a "lurking variable" which affects two or more variables simultaneously, thereby producing a systematic association between the affected variables. Extreme examples of such meaningless relationships in other fields have been cited by Huff [2].

Other obstacles prevent proper interpretation of routine operating data. In normal process operation, important operating variables are often held in tight control. Under these circumstances the effects of such operating variables on the response may be completely masked by random error. Sometimes routine changes in process variables are deliberately correlated. For example, a reduction in temperature may be regularly accompanied by a reduction pressure. In such cases it will be impossible to extract the individual effects of these operating variables on the response.

For these reasons, as Box [1] has advised, "to find out what happens to a system when you interfere with it, you have to interfere with it (not just passively observe it)".

"Interference" with a system to obtain specific information is accomplished most effectively and efficiently using experimental plans or strategies which are discussed in the following sections.

REFERENCES

[1] Box, G.E.P., (1966), "Use and abuse of regression", Technometrics, 8, pp 625-629.

[2] Huff, D. (1954), How to Lie with Statistics, Norton.

CHAPTER 21

Planning Experiments in Process Modeling

In Sections 12 to 19 it has been demonstrated that attempting to find an adequate linear model form to represent a set of data is not a straightforward operation. Even when an adequate fitted model with a minimum number of terms has been found, the result may be unsatisfactory. The precision of the fitted model may be so poor that it is of no value.

The precision of parameter estimates in a linear model is expressed by their covariance matrix $\left(\mathbf{X}^{\mathrm{T}}\mathbf{X}\right)^{-1}\sigma^2$. This matrix is completely determined by two characteristics of the data, σ^2, the variance of the random error, and $\mathbf{X}$, the matrix of values of the independent variables in the fitted model, which in turn results from the sets of operating conditions tested. The matrix $\mathbf{X}$ also controls the degree of correlation among parameter estimates, thereby influencing both numerical accuracy of the least squares calculations and the extent to which parameter values can be assessed independently of one another. $\mathbf{X}$ and σ^2 also determine the precision of predicted response values.

It is clear that a reduction in the random error variance σ^2 will reduce the variances of the individual parameter estimates, although it will not affect their correlations. Reduction in σ^2 will also improve the precision of predicted responses. Methods for achieving improved control in testing procedures to achieve smaller experimental error are the concern of all experimenters. No comments will be made here on this important undertaking.

What will be discussed in this section and succeeding sections is the influence of the choice of operating conditions, that is, the matrix $\mathbf{X}$ for a proposed model, on the precision of the fitted model. Strategies for planning experimental programs to achieve specified objectives in process modeling are part of a field of study which statisticians call experimental design.

Much has been written about statistical experimental design and most introductory statistics textbooks contain at least one or two sections on this topic. The emphasis in many texts is an assessment of differences in the effects of two or more treatments on specific process characteristics. Analysis of variance is usually used to analyze the results of these experiments.

In these notes, interest in experimental design will be restricted to the efficient development of mathematical models for processes. The chief tool for analysis of the data will be least squares model fitting.

Even for this restricted area of application the choice of an experimental design procedure will of course depend upon the modeling objective. Hooke [1, pp. 91-93] describes some specific objectives that call for very different design strategies. One common objective is to design a specified number of tests to obtain parameter estimates in a proposed model form that have minimum variances. Another common objective is to obtain smallest possible variances for predicted responses for a proposed model form within a specified operating region. In general, these two objectives require different types of experimental designs.

Whatever the design objective may be, it is important to keep in mind that the choice of experimental strategy will also depend on the form of model or models being considered. To illustrate this point the straight line model

$$E(Y) = \beta_0 + \beta_1 x \tag{21.1}$$

will again be used. Ordinarily there will be more interest in the parameter β_1 than the parameter β_0, since β_1 describes the rate of change of the response with respect to the operating variable x. From expression (18 2), the variance of the estimate β_1 obtained from n data can be shown to be

$$\operatorname{var}\left(\hat{\beta}_i\right) = \frac{\sigma^2}{\sum_{u=1}^{n} (x_u - \bar{x})^2} \tag{21.2}$$

where $\bar{x}$ is the sample mean of the n values of the operating variable.

Knowing before any data have been collected that the variance of β_1 will be given by (21.2), regardless of the values of x, it is a simple matter to specify the values of the operating variable for n tests to achieve a β_1 that has minimum variance. In virtually all practical situations feasible values of an operating variable are constrained to fall between an upper limit x^U and a lower limit x^L. To minimize the variance of β_1, the operating conditions for n tests should be divided as evenly as possible between x^U and x^L.

Concentrating all tests at only two operating conditions may not appeal to some investigators. They may be concerned about the absence of intermediate test conditions from which the adequacy of the straight line model form may be assessed. This is another example of different design objectives leading to different experimental strategies. The preceding paragraph has described an optimal experimental design procedure for obtaining a minimum variance estimate of β_1. If the objective is to test the adequacy of the model form (21.1) against a specific alternative model form, then another experimental design procedure must be employed [2].

To discover other advantages of dividing operating conditions equally between x^U and x^L, the straight line model will be expressed in the form (11.12),

$$E(Y) = \gamma_0 + \gamma_1(x - \bar{x})$$

where $\bar{x}$ is again the sample mean of the n values of the operating variable. To simplify the presentation, n will be considered to be an even number.

In addition to providing a minimum variance estimate of γ_1 (where $\gamma_1 = \beta_1$ in (21.1)), it can be confirmed that this experimental design procedure produces estimates of γ_0 and γ_1 that are uncorrelated. The size of the elliptical joint confidence region (18.6) for γ_0 and γ_1 (or, equivalently, for β_0 and β_1) can be shown to be inversely proportional to the square root of the determinant $|\mathbf{X}^T\mathbf{X}|$. Since this design strategy maximizes $|\mathbf{X}^T\mathbf{X}|$, it produces parameter estimates whose joint precision is maximized.

Extensions of this particular design objective will be discussed in subsequent sections. In all cases it must be kept in mind that the benefits achieved are specific to the particular model form being considered.

REFERENCES

[1] Hooke, R., (1963), Introduction to Scientific Inference, Holden-Day, San Francisco.

[2] Box, G.E.P. and Draper, N.R., (1959), Journal of American Statistical Association, 54, pp. 622-654.

CHAPTER 22

Two Level Factorial Designs

The experimental strategy of distributing n operating conditions equally at the upper and lower limits for a single operating variable when the proposed model form is a straight line is now extended to the case of two operating variables whose influence on the response variable is to be described by a first degree polynomial,

$$E(Y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 \tag{22.1}$$

To simplify notation and subsequent calculations x_1 and x_2 in (22.1) will be dimensionless coded variables defined as follows:

$$x_i = \frac{\begin{pmatrix}\text{value of operating}\\ \text{variable } i\end{pmatrix} - \frac{1}{2}\begin{pmatrix}\text{it's upper} & + & \text{it's lower}\\ \text{limit} & & \text{limit}\end{pmatrix}}{\frac{1}{2}\begin{pmatrix}\text{it's upper} & - & \text{it's lower}\\ \text{limit} & & \text{limit}\end{pmatrix}} \tag{22.2}$$

for $i = 1,2$.

If each operating variable is to be tested at only two conditions, then using the coding (22.2) there are four possible combinations of operating conditions as shown in table 22.1.

Table 22.1 - A two level factorial design for two operating variables.

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

The set of four test conditions which comprise all possible combinations of values of the operating variables is called a *two level factorial design for two operating variables*. It is a special case of the general factorial design involving k operating variables in which m_1

values, or levels, of the first operating variable can be tested, m_2 values of the second operating variable, and so on.

A factorial design in this general situation would require $\prod_{i=1}^{k} m_i$ test conditions, this being the number of possible combinations of values of the k operating variables.

For $n = 4$ tests the experimental design in table 22.1 provides estimates of β_0, β_1 and β_2 in model (22.1) that have individually minimum variances. In addition this design yields uncorrelated estimates of the three parameters.

There is another even more important benefit that is available from this design. It provides information about any possible interaction between the two coded operating variables x_1 and x_2. Two operating variables are said to interact if the size of the influence of one of these operating variables on the response depends upon the value of the other operating variable. The following simple example illustrates this concept.

Suppose that as a first stage in a study of the dependence of the tensile strength of a synthetic fibre upon the quenching temperature and the draw ratio, four tests are conducted using the experimental design in table 22.1. If the results were as shown in figure 22.1(a), then the two operating variables, quenching temperature and draw ratio, would be said to interact since the effect of a change in the quenching temperature on the tensile strength clearly depends upon the value of the draw ratio. On the other hand, if the results were as shown in Figure 22.1(b) then no interaction between these two operating variables would be said to exist since the effect of a change in the quenching temperature on the tensile strength is the same for both values of the draw ratio.

In the case of no interaction (Figure 22.1(b)) the model (22.1) could be expected to provide a reasonable representation of the data. If interaction exists (Figure 22.1(a)), then it may be accounted for by the addition, of a cross product term in the two operating variables,

$$E(Y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 \qquad (22.3)$$

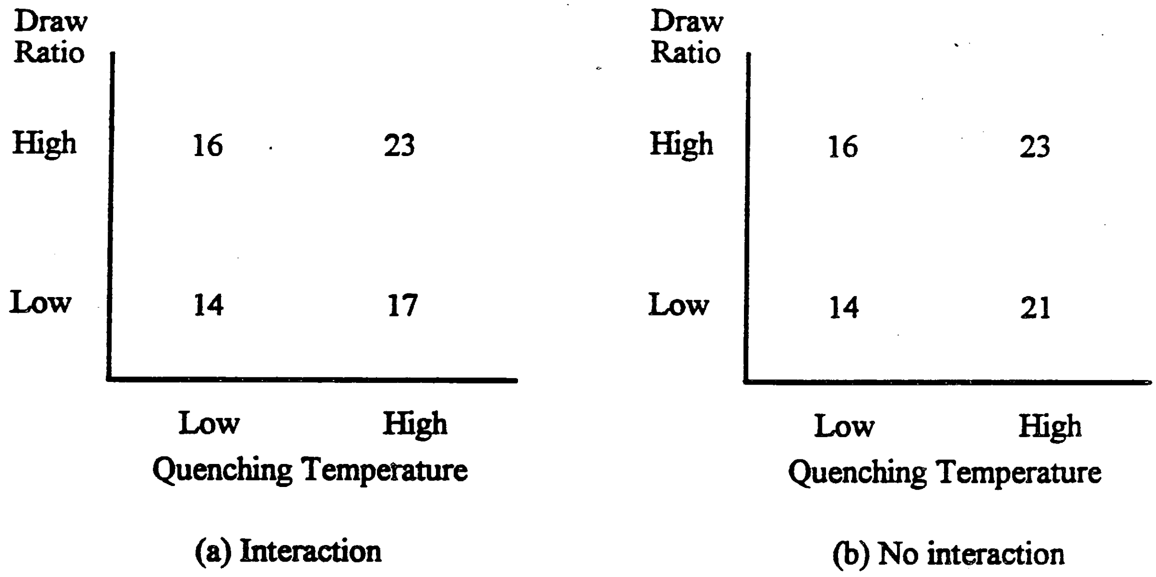

Figure 22.1 - An illustration of interaction. Values of tensile strength in kg are shown at each set of operating conditions

Since model (22.3) has as many parameters as there are tests in this example, it will fit the data exactly, leaving no possibility of testing its inadequacy. If replicate tests are carried out at the four sets of operating conditions or if an external estimate of pure error variance is available, then a confidence interval for each of the four parameters can be calculated. Deletion of individual terms can also be tested, opening up the possibility of a test of adequacy of a reduced model.

Two level factorial designs are not intended to provide definitive models of process behaviour. Indeed, few process investigators would be content to test only two values of each operating variable during a study. The primary use of a two level factorial design is as a *screening tool* to detect from a number of potentially important operating variables those that have strong influences, either individually, i.e. $\beta_i x_i$ or jointly, i.e. $\beta_{ij} x_i x_j$, on the response. The fact that the variances of the estimates of all four coefficients in model (22.3) are minimized by using the two level factorial design in table 22.1 is of secondary importance. The primary benefit is the information provided about the possible interaction between the two operating variables.

Extension of two level factorial designs to study more than two operating variables is straightforward. A two level factorial design for k operating variables is denoted as a 2^k design, indicating that k variables are being tested, each at two levels. The notation 2^k also identifies the number of tests required. Independent estimates of each of the 2^k parameters in the following "complete" model are available from this design,

$$E(Y) = \beta_0 + \beta_1 x_1 + \cdots + \beta_k x_k \\ + \beta_{12} x_1 x_2 + \cdots + \beta_{k-1,k} x_{k-1} x_k \\ + \beta_{123} x_1 x_2 x_3 + \cdots + \beta_{k-2,k-1,k} x_{k-2} x_{k-1} x_k \\ + \cdots \\ + \beta_{12\cdots k} x_1 x_2 \cdots x_k \quad (22.4)$$

In practice, interaction terms in (22.4) involving more than two operating variables are often neglected since their influence on the response is usually small. Deletion of these higher order terms also provides residual information for testing the adequacy of a fitted model.

Two level factorial designs do not yield information about possible quadratic behaviour of individual operating variables, that is, terms such as $\beta_{ii} x_i^2$. Because each variable is tested only at two values, only a straight line, i.e. a first order term $\beta_i x_i$, can be uniquely obtained for each variable. There are an infinite number of parabolas, i.e. curves involving both $\beta_i x_i$ and $\beta_{ii} x_i^2$, that can pass through two points. To obtain this type of information about a single operating variable at least three distinct values of the variable must be tested. Experimental designs that provide this information will be discussed in a later section .

The following example illustrates the information available from a two level factorial design and the precision of that information.

In the first stage in a process yield study, three operating variables, temperature, type of catalyst. and pressure, are selected. Two types of catalyst, denoted as A and B, are to be evaluated. Two temperature values, 200°C and 260°C, and two pressure values, 240 psi and 270 psi, are chosen as limits for this initial investigation.

Temperature and pressure are *quantitative variables,* that is, their values can be ranked on a numerical scale. Catalyst type is a *qualitative variable* as it has no such ranking. Both types of variables can be accommodated in a two level factorial design.

The coding (22.2) can be applied directly to the two quantitative variables, producing coded values of 1 and -1 for each of these variables. To maintain consistency among the three operating variables the catalyst type can be coded in an arbitrary fashion such as

$$\text{coded catalyst type} = \begin{cases} -1 & \text{for catalyst A} \\ 1 & \text{for catalyst B} \end{cases}$$

* To choose upper and lower limits for an experiment, one should look at literature related to the experiment to make an educated guess about ~~the~~ the appropriate limits.

A 2^3 experimental design for this initial investigation is shown in table 22.2. A measured yield value for each test is also shown. An external estimate of pure error variance is available, $\hat{\sigma}^2 = 5.2$ with 6 degrees of freedom.

Randomize!

Table 22.2 - A 2^3 experimental design

Coded Temperature x_1	Coded Catalyst Type x_2	Coded Pressure x_3	Measured Yield (%) y
-1	-1	-1	60
1	-1	-1	72
-1	1	-1	54
1	1	-1	68
-1	-1	1	52
1	-1	1	83
-1	1	1	45
1	1	1	80

Before describing the analysis of the data in table 22.2, a comment will be made about the order of execution of the eight tests.

As will be demonstrated later in this example, a change in catalyst from type A to type B appears to have a significant effect on the process yield. Since these eight tests are active interference with the process rather than merely passive observation [1], a natural conclusion is that a change in catalyst type *causes* a change in yield. However, unless precaution is taken in the order in which the eight tests are carried out, this conclusion may not be warranted; the "effect" may be merely a coincidental association of the type discussed in earlier sections.

For example, if the eight tests were executed in the order shown in table 22.2, all of the tests using catalyst type A may have been carried out by one operator while the tests using catalyst type B were carried out by another operator. If the first operator was more proficient in operating the process than the second operator, a change in process yield that coincided with a change in catalyst type may well have been due mainly to the change in operators.

Since the pattern of spurious correlations may be more complex than that just described, some overall safeguard is required. The most effective protection against unknown trends is to carry out the tests in *randomized order*. The selected order of execution of a set of tests is random if every possible order has an equal chance of being chosen. This could be accomplished for this example by putting eight pieces of paper, numbered from 1 to 8 to correspond to the order shown in table 22.2, in a box and carrying out runs in the order in which numbers are drawn.

Two arguments against randomization are often heard. The first is that a randomized order may in fact coincide with changes in an important unknown variable anyway. However, the probability of such a coincidence is minimized by always selecting the order of tests at random. The second objection is that it is inconvenient or unnecessarily costly to conduct runs in a random order. This objection is valid only if there is assurance that all important operating variables, other than those being manipulated deliberately, have reasonably constant values throughout the set of tests. This ideal situation is rarely achieved in practice. The purpose of randomizing the order of tests is to assure the validity of conclusions as far as possible.

The complete model (22.3) will be fitted to the data in table 22.2. For $k = 3$ operating variables, the model is

$$\begin{aligned} E(Y) = \beta_0 &+ \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 \\ &+ \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \\ &+ \beta_{123} x_1 x_2 x_3 \end{aligned} \tag{22.5}$$

Least squares estimates of the 8 parameters $\boldsymbol{\beta}$ are given by

$$\hat{\boldsymbol{\beta}} = \left(\mathbf{X}^{T}\mathbf{X}\right)^{-1}\mathbf{X}^{T}\mathbf{Y} \tag{22.6}$$

where

$$\mathbf{Y} = \begin{bmatrix} 60 \\ 72 \\ 54 \\ 68 \\ 52 \\ 83 \\ 45 \\ 80 \end{bmatrix} \qquad \boldsymbol{\beta} = \begin{bmatrix} \beta_0 \\ \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_{12} \\ \beta_{13} \\ \beta_{23} \\ \beta_{123} \end{bmatrix}$$

and

These are the values we set

$$
\begin{array}{cc}
 & \begin{array}{cccccccc} \beta_0 & x_1 & x_2 & x_3 & x_1x_2 & x_1x_3 & x_2x_3 & x_1x_2x_3 \end{array} \\
\mathbf{X} = & \begin{bmatrix}
1 & -1 & -1 & -1 & 1 & 1 & 1 & -1 \\
1 & 1 & -1 & -1 & -1 & -1 & 1 & 1 \\
1 & -1 & 1 & -1 & -1 & 1 & -1 & 1 \\
1 & 1 & 1 & -1 & 1 & -1 & -1 & -1 \\
1 & -1 & -1 & 1 & 1 & -1 & -1 & 1 \\
1 & 1 & -1 & 1 & -1 & 1 & -1 & -1 \\
1 & -1 & 1 & 1 & -1 & -1 & 1 & -1 \\
1 & 1 & 1 & 1 & 1 & 1 & 1 & 1
\end{bmatrix}
\end{array}
$$

The elements of the columns of $\mathbf{X}$ associated with the interaction terms $x_1x_2, \cdots, x_1x_2x_3$ are the products of the corresponding elements in the columns associated with x_1, x_2 and x_3.

Now

$$
\mathbf{X}^T\mathbf{X} = \begin{bmatrix}
8 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
 & 8 & 0 & 0 & 0 & 0 & 0 & 0 \\
 & & 8 & 0 & 0 & 0 & 0 & 0 \\
 & & & 8 & 0 & 0 & 0 & 0 \\
 & & & & 8 & 0 & 0 & 0 \\
 & & & & & 8 & 0 & 0 \\
 & & & & & & 8 & 0 \\
 & & & & & & & 8
\end{bmatrix}
$$

so that

$$
(\mathbf{X}^T\mathbf{X})^{-1} = \begin{bmatrix}
1/8 & 0 & 0 & 0 & 0 & 0 & 0 & 0 \\
 & 1/8 & 0 & 0 & 0 & 0 & 0 & 0 \\
 & & 1/8 & 0 & 0 & 0 & 0 & 0 \\
 & & & 1/8 & 0 & 0 & 0 & 0 \\
 & & & & 1/8 & 0 & 0 & 0 \\
 & & & & & 1/8 & 0 & 0 \\
 & & & & & & 1/8 & 0 \\
 & & & & & & & 1/8
\end{bmatrix}
$$

and

$$\mathbf{X}^{\mathrm{T}}\mathbf{Y} = \begin{bmatrix} \sum_{u=1}^{8} y_u \\ \sum_{u=1}^{8} x_{u1} y_u \\ \sum_{u=1}^{8} x_{u2} y_u \\ \sum_{u=1}^{8} x_{u3} y_u \\ \sum_{u=1}^{8} x_{u1} x_{u2} y_u \\ \sum_{u=1}^{8} x_{u1} x_{u3} y_u \\ \sum_{u=1}^{8} x_{u2} x_{u3} y_u \\ \sum_{u=1}^{8} x_{u1} x_{u2} x_{u3} y_u \end{bmatrix}$$

where x_{u1}, x_{u2}, x_{u3} and y_u are values of the respective variables for test u.

Using (22.6), the least squares parameter estimates are then

$$\hat{\boldsymbol{\beta}} = \begin{bmatrix} \hat{\beta}_0 \\ \hat{\beta}_1 \\ \hat{\beta}_2 \\ \hat{\beta}_3 \\ \hat{\beta}_{12} \\ \hat{\beta}_{13} \\ \hat{\beta}_{23} \\ \hat{\beta}_{123} \end{bmatrix} = \begin{bmatrix} \sum_{u=1}^{8} y_u \Big/ 8 \\ \sum_{u=1}^{8} x_{u1} y_u \Big/ 8 \\ \sum_{u=1}^{8} x_{u2} y_u \Big/ 8 \\ \sum_{u=1}^{8} x_{u3} y_u \Big/ 8 \\ \sum_{u=1}^{8} x_{u1} x_{u2} y_u \Big/ 8 \\ \sum_{u=1}^{8} x_{u1} x_{u3} y_u \Big/ 8 \\ \sum_{u=1}^{8} x_{u2} x_{u3} y_u \Big/ 8 \\ \sum_{u=1}^{8} x_{u1} x_{u2} x_{u3} y_u \Big/ 8 \end{bmatrix} \qquad (22.7)$$

Some of the advantages of using a two level factorial design are now clear. Because $\mathbf{X}^T\mathbf{X}$ is a diagonal matrix, each of the parameter estimates in (22.7) can be calculated independently of all of the other parameter estimates. That is, the parameter estimates are mutually uncorrelated. Furthermore, since the variances of the parameter estimates are the diagonal elements of $\left(\mathbf{X}^T\mathbf{X}\right)^{-1}\sigma^2$, it is clear that a 2^k design produces parameter estimates with equal variances, namely $\sigma^2/2^k$. This is the minimum variance achievable from 2^k tests within the specified operating region.

Using the matrices $\mathbf{X}$ and $\mathbf{Y}$ for this example, the least squares fitted model of form (22.5) is

Full Fitted model

$$\hat{y} = 64.25 + 11.5x_1 - 2.5x_2 + 0.75x_3 + 0.75x_1x_2 + 5.0x_1x_3 + 0.25x_1x_2x_3 \qquad (22.8)$$

Because $\hat{\beta}_{23} = 0$, there is no term in x_2x_3 in (22.8).

The fitted model (22.8) provides an exact fit to the data for this example since model (22.5) contains as many parameters as there are tests. Therefore the adequacy of this model cannot be tested. Possible deletions of terms in (22.8) can be investigated however. Because of the absence of correlation among all parameter estimates, there are a number of ways of assessing the significance of each term independently.

One method is to use the available external estimate of pure error variance to calculate a confidence interval for each term. If a confidence interval reveals that zero is a plausible value for a parameter, then that term may be deleted from the fitted model. * Notice that because all parameter estimates in (22.8) are uncorrelated, setting one or more parameters to zero will not alter the least squares estimates of the remaining parameters.

As pointed out earlier in this example, the variance of each of the parameter estimates in (22.8) is $\sigma^2/8$. Using the external estimate $\hat{\sigma}^2 = 5.2$ with 6 degrees of freedom, a 95 per cent confidence interval for the true value of each parameter is

$$\hat{\beta} \pm t_{6,0.025}\sqrt{\frac{5.2}{8}} = \hat{\beta} \pm 1.97.$$

Applying this confidence interval to each of the parameter estimates in (22.8) it is seen that the parameters β_3, β_{12}, β_{23} and β_{123} are all plausibly zero. The reduced fitted model is then

$$\hat{y} = 64.25 + 11.5x_1 - 2.5x_2 + 5.0x_1x_3 \qquad (22.9)$$

* always do replicates (center point is best for this purpose)

The significance of individual terms in (22.8) can also be tested using the ratio Q defined in equation (17.1). Once again, however, the lack of correlation among all the parameter estimates leads to a special result.

From equation (16.7),

$$\mathbf{e}^{\mathrm{T}}\mathbf{e} = \mathbf{Y}^{\mathrm{T}}\mathbf{Y} - \hat{\boldsymbol{\beta}}^{\mathrm{T}}\mathbf{X}^{\mathrm{T}}\mathbf{Y}$$

and for model (22.5)

$$\hat{\boldsymbol{\beta}}^{\mathrm{T}}\mathbf{X}^{\mathrm{T}}\mathbf{Y} = 8\hat{\beta}_0^2 + 8\hat{\beta}_1^2 + 8\hat{\beta}_2^2 + 8\hat{\beta}_3^2 + 8\hat{\beta}_{12}^2 + 8\hat{\beta}_{13}^2 + 8\hat{\beta}_{23}^2 + 8\hat{\beta}_{123}^2 \qquad (22.10)$$

Because each term on the right hand side of (22.10) is independent of the other terms (a condition not generally achieved unless an orthogonal experimental design has been used), the increase in the residual sum of squares produced by deleting any one term β is equal to $8\hat{\beta}^2$. Thus the significance of each term in (22.8) can be tested by comparing the value of the ratio Q in the following form

$$Q = \frac{8\hat{\beta}^2}{\hat{\sigma}^2}$$

with the value of $F_{1,\nu}$ at the desired probability level.

Using $\hat{\sigma}^2 = 5.2$ with 6 degrees of freedom and a significance level of 0.05, the reduced model (22.9) is again obtained.

The residuals from the fitted model (22.9) in the order corresponding to the data in table 22.2 are -0.25, -1.25, -1.25, -0.25, 1.75, -0.25, -0.25, 1.75. Tests of this fitted model using the significance tests for model inadequacy reveal no evidence of inadequacy.

The reduced fitted model (22.9) can be expressed in terms of the original (uncoded) operating variables as

$$\begin{aligned}\text{Predicted Yield} = 64.25 &+ 11.5\left(\frac{\text{temperature} - 230}{30}\right)\\ &-2.5\left(\begin{matrix}-1 \text{ if catalyst A used}\\ +1 \text{ if catalyst B used}\end{matrix}\right)\\ &+5.0\left(\frac{(\text{temperature} - 230)}{30}\frac{(\text{pressure} - 255)}{15}\right)\end{aligned}$$

this code is purely qualitative it is meant only as an "either or" type of indicator

* It may also be advantageous to do Q ratio tests.

$$\text{Predicted Yield} = 627.75 - 2.45(\text{temperature}) - 2.55(\text{pressure}) + 0.0111(\text{temperature})(\text{pressure}) + \begin{pmatrix} +2.5 \text{ if catalyst A is used} \\ -2.5 \text{ if catalyst B is used} \end{pmatrix}$$

From this fitted model it can be seen that the apparent effect of changing from catalyst A to catalyst B is to reduce the process yield by 5 per cent. Because no significant interaction was found between catalyst type and either of the other two operating variables, it can be concluded that within this operating region the influence of a charge in catalyst type is unaffected by the values of temperature and pressure.

The influence of temperature and pressure on the process yield must be considered jointly because of the large interaction between them. Their joint effect can be represented most simply by a diagram such as figure 22.3. The process yields shown are the averages of the two tests carried out at each combination of temperature and pressure. Differences in the effects of a change in temperature on the process yield at the two pressures is clearly demonstrated.

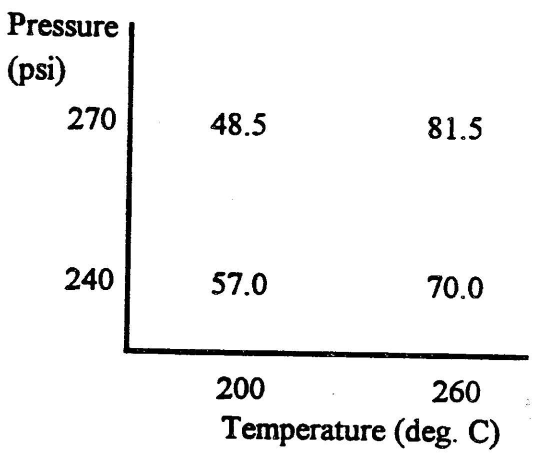

Figure 22.3 - Joint effect of temperature and pressure on process yield

Two level factorial designs are most useful for screening studies involving up to four operating variables. For situations with more than four operating variables the number of tests required, 2^k, can be prohibitive. Furthermore, much of the information generated in these situations is of little interest, for example, interactions involving three, four and even higher numbers of operating variables. A useful class of subsets or *fractions* of two level factorial designs permit economy in the number of tests without loss of important information about the effects of operating variables on the response.

* Often times only 2 factor interactions are important.

CHAPTER 23

Two Level Fractional Factorial Designs

If only a subset of the possible 2^k tests in a complete two level factorial design for k operating variables is executed, it is obvious that some loss of information must be incurred It would be desirable to select an appropriate fraction of the complete 2^k design, that is, an appropriate *fractional factorial design*, so that important pieces of information are still retained even though some unimportant pieces of information are lost. Unfortunately, information from fractional factorial designs is available only as mixtures of the individual pieces of information available from complete factorial designs. Skillful selection of a fractional factorial design can result in each mixture, consisting of one potentially important piece of information combined with one or more pieces of information that is judged *a priori* to be unimportant. If the analysis of data from a fractional factorial design produces ambiguous results, a further fractional factorial design, planned specifically to resolve those ambiguities, may be carried out.

To demonstrate how mixtures of pieces of information arise in fractional factorial designs, the following case is considered. Suppose there are four operating variables of interest in a screening study and restricted resources permit only eight tests to be carried out. If each operating variable is tested at only two values, represented by -1 and 1 in the coded notation introduced in the preceding section, then one solution is to select eight of the sixteen tests of a 2^4 design. The resulting design is called a *half-fraction of a* 2^4 *design* and denoted more concisely as a 2^{4-1} design. Using this notation a subset of four tests from a 2^4 design would be denoted as a 2^{4-2} design.

There are 12870 different subsets of eight tests that can be selected from the sixteen tests of a 2^4 design, each yielding different mixtures of information. The particular subset chosen for this demonstration is that for which the four factor interaction term $x_1x_2x_3x_4$ has the value 1. These eight tests are shown in table 23.1.

Table 23.1 - A 2^{4-1} design

x_1	x_2	x_3	x_4		$x_1x_2x_3x_4$
−1	−1	−1	−1		1
1	−1	−1	1	→ these	1
−1	1	−1	1	are just	1
1	1	−1	−1	the product	1
−1	−1	1	1	of $x_1x_2x_3$.	1
1	−1	1	−1		1
−1	1	1	−1		1
1	1	1	1		1

The model

$$E(Y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 \tag{23.1}$$

can be fitted to the data obtained from this 2^{4-1} design and the five parameter estimates will be mutually uncorrelated. The least squares estimate of β_1, for example, is

$$\frac{\sum_{u=1}^{8} x_{u1} y_u}{8}$$

which, if tests are numbered in the order displayed in table 23.1, is the linear combination l_1 where

$$l_1 = \frac{1}{8}\left(-y_1 + y_2 - y_3 + y_4 - y_5 + y_6 - y_7 + y_8\right) \tag{23.2}$$

However, if the term $\beta_1 x_1$ in model (23.1) is replaced by the interaction term $\beta_{234} x_2 x_3 x_4$, then, because the value of $x_2x_3x_4$ is identical to the value of x_1 for each of the eight tests in table 23.1, the least squares estimate of β_{234} will also be given by expression (23.2).

Since in these eight tests, changes in the variable x_1 coincide exactly with changes in the variable $x_2x_3x_4$, the resulting effect of these changes on the response is in fact the *sum* of the individual effects of x_1 and $x_2x_3x_4$. Because of the perfect correlation between changes in x_1 and changes in $x_2x_3x_4$, it is impossible to separate the effects of these two variables. Thus l_1 is in fact an estimate of $(\beta_1 + \beta_{234})$, the sum of the parameters for the two variables.

In this situation, the variables x_1 and $x_2x_3x_4$ are said to be *confounded.* Another way of describing their relationship is that x_1 is an *alias* of $x_2x_3x_4$, and vice versa. In all fractional factorial designs every variable is confounded with one or more other variables.

The additional aliases arising from the 2^{4-1} design in table 23.1 could be discovered by constructing the columns of values of all the interaction terms involving x_1, x_2, x_3 or x_4. It would be found, for example, that, x_2 is an alias of $x_1x_3x_4$, x_2x_3 is an alias of x_1x_4, and so on. However, there is a more direct way of determining the overall alias structure for a fractional factorial design.

If a column consisting entirely of "1's" is denoted as *I*, then because the values of the variable $x_1x_2x_3x_4$ from the 2^{4-1} design in table 23.1 are also 1's, this design can be identified by the *defining relation* $I = x_1x_2x_3x_4$. This designation is unique as there is only one of the 12870 subsets of eight runs from a 2^4 design for which the interaction $x_1x_2x_3x_4$ has the value 1 for all eight runs. The complementary 2^{4-1} design, for which $x_1x_2x_3x_4$ has the value -1 for all eight runs, has the defining relation $I = -x_1x_2x_3x_4$ indicating that corresponding elements of the I and $x_1x_2x_3x_4$ columns are equal in magnitude but opposite in sign.

The defining relation can he used to identify the alias of any variable arising from a design. Since the identification procedure involves multiplying both sides of the defining relation by a variable, this operation is now described. For any fractional factorial design, multiplication of variables x_i and x_j means the creation of a column of values, each of which is the product of corresponding elements of the x_i and x_j columns in that design. Two special cases deserve mention.

First, multiplication of any variable by *I* leaves the column of values for that variable unchanged. For example,

$$x_1 \cdot I = x_1$$

and

$$x_2x_3 \cdot I = x_2x_3.$$

Second, multiplication of any variable by itself produces a column of 1's, that is; a column identical to *I*. For example,

$$x_3 \cdot x_3 = x_3^2 = I$$

and

$$(x_1 x_3 x_4)^2 = I.$$

Now for illustration, $I = x_1 x_2 x_3 x_4$, the defining relation for the 2^{4-1} design in table 23.1, is used to identify the aliases of x_4 and $x_1 x_2$. To determine the alias of x_4, both sides of this defining relation are multiplied by x_4, yielding

$$x_4 \cdot I = x_4 \cdot x_1 x_2 x_3 x_4 \quad .$$

which can be expressed more simply using the above multiplication definition as

$$x_4 = x_1 x_2 x_3.$$

Therefore, for this design, x_4 is an alias of $x_1 x_2 x_3$.

The alias of $x_1 x_2$ is obtained by multiplying both sides of the defining relation by $x_1 x_2$, yielding

$$x_1 x_2 = x_3 x_4.$$

Therefore, for this design, $x_1 x_2$ is an alias of $x_3 x_4$. Further application of this identification procedure to identify other alias relationships follows the same pattern.

Because of the several aliases arising from a fractional factorial design, it is of interest to identify the various combinations of parameters that can be estimated from the response data obtained by executing the design.

A design involving eight tests yields at most eight: separate parameter estimates. For the 2^{4-1} design with $I = x_1 x_2 x_3 x_4$ (table 23.1), the following eight estimates are available:

l_1, which estimates $(\beta_1 + \beta_{234})$
l_2, which estimates $(\beta_2 + \beta_{134})$
l_3, which estimates $(\beta_3 + \beta_{124})$
l_4, which estimates $(\beta_4 + \beta_{123})$
l_{12}, which estimates $(\beta_{12} + \beta_{34})$
l_{13}, which estimates $(\beta_{13} + \beta_{24})$
l_{14}, which estimates $(\beta_{14} + \beta_{23})$
l_0, which estimates $(\beta_0 + \beta_{1234})$

The definition. of l_1 was given in equation (23 2). The other linear combinations are defined analogously. For example,

$$l_{12} = \frac{\sum_{u=1}^{8} x_{u1} x_{u2} y_u}{8}$$

and

$$l_0 = \frac{\sum_{u=1}^{8} y_u}{8}.$$

Interpretation of these estimates depends upon what one is willing to assume about individual interactions. For example, if it is assumed *a priori* that interactions involving more than two operating variables are negligible, then a large value of l_3 would suggest that β_3 was large. Ambiguities can arise when individual operating variables are confounded with two variable interactions or when two variable interactions; are confounded with one another. As will be shown later in this section, such ambiguities can be resolved by carrying out one or more additional fractional factorial designs. It is clear, for example, that combining the information from the 2^{4-1} design $I = x_1 x_2 x_3 x_4$ with the information from the 2^{4-1} design $I = -x_1 x_2 x_3 x_4$ must provide all of the individual pieces of information available from the 2^4 design in variables x_1, x_2, x_3 and x_4.

Ambiguity arising from a fractional factorial design can be minimized by choosing it's defining relation so that as far as possible, potentially important variables are confounded with potentially unimportant variables. The effect of the choice of defining relation can be illustrated with a 2^{4-1} design. If the defining relation $I = x_1$ is selected, then the value of x_1 remains fixed at the value 1 for all eight tests. Consequently no information about the effect on the response of changing x_1 is provided. If the defining relation $I = x_1x_2$ is selected, then x_1 is confounded with x_2 and their individual effects on the response cannot be separated. The defining relation $I = x_1x_2x_3$ would confound each of the three operating variables x_1, x_2 and x_3 with a two variable interaction. If any one of the interactions x_1x_2, x_1x_3 or x_2x_3 is of appreciable size, it will not be possible to estimate the influence on the response of the individual operating variable which is confounded with that .interaction. Since the effects of individual operating variables, that: is terms such as $\beta_1x_1, \beta_2x_2, \beta_3x_3$ and β_4x_4, are of primary interest in a screening study, the relation $I = x_1x_2x_3x_4$ is the best choice since each of these terms is confounded with a three variable interaction and interactions involving three or more operating variables are almost always of negligible size.

A defining relation can also be used to determine the values of the operating variables for each of the tests in a fractional factorial design. For the 2^{4-1} design $I = x_1x_2x_3x_4$, for example, it has been shown that $x_4 = x_1x_2x_3$. One method of identifying the values of the four operating variables for the eight runs is to write down a 2^3 design in the variables x_1, x_2 and x_3 and then set the value of x_4 for each test equal to the value of the interaction $x_1x_2x_3$. This procedure has been used in table 23.1.

From the foregoing discussion of a 2^{4-1} design, it is clear that a 2^{k-1} design can be constructed using any variable to split a 2^k design into two halves. To minimize ambiguities in the information produced, however, the highest order interaction among the k operating variables is usually the best choice for the defining relation. Thus for a 2^{5-1} design, for example, the defining relation $I = x_1x_2x_3x_4x_5$ should be used.

For situations involving more than six operating variables even a 2^{k-1} design produces some information that is unlikely to be important. Combinations of parameters associated only with high order interaction terms are seldom of interest. Further economy in the number of tests is possible by using smaller fractions of the 2^k design. The general notation for these designs is 2^{k-q} where $q = 1, \cdots, k-1$.

In discussing 2^{k-q} designs it is useful to introduce the concept of *resolution* [1,2]. The three classes of 2^{k-q} designs most commonly employed are the following.

Resolution III designs are 2^{k-q} designs for which

(i) no individual operating variable, such as x_1, is confounded with any other individual operating variable, such as x_2 and

(ii) at least one individual operating variable is confounded with a two variable interaction.

An example of a resolution III design is the 2^{3-1}_{III} design $I = x_1x_2x_3$. Notice that the resolution of a design is written as a subscript in Roman numerals.

Resolution IV designs are 2^{k-q} designs for which

(i) no individual operating variable is confounded with any other individual operating variable or with any two variable interaction and

(ii) at least one two variable interaction is confounded with another two variable interaction.

An example is the 2^{4-1}_{IV} design $I = x_1x_2x_3x_4$.

Resolution V designs are 2^{k-q} designs for which

(i) no individual operating variable is confounded with any other individual operating variable or with any two variable interaction and

(ii) no two variable interaction is confounded with another two variable interaction and

(iii) at least one two variable interaction is confounded with a three variable interaction.

An example is the 2^{5-1}_{V} design $I = x_1x_2x_3x_4x_5$.

* The resolution of a 2^{k-q} design is the smallest order interaction appearing in the defining relation for the design.

To demonstrate the construction of a smaller fraction of a 2^k design, a 2^{5-2}_{III} design is now developed. The notation 2^{5-2}_{III} indicates a resolution III design of eight tests involving five operating variables, each at two levels. To begin, a 2^3 design, in any three of the five operating variables is written down. Variables x_1, x_2 and x_3 have been chosen in table 23.2.

*The smallest # of terms in any of your

Table 23.2 - A 2_{III}^{5-2} design.

x_1	x_2	x_3	x_4	x_5
−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

The two remaining operating variables are now confounded with any of the interactions among x_1, x_2 and x_3. For example, in table 23.2, x_4 is confounded with $x_1x_2x_3$ and x_5 is confounded with x_2x_3. Now since $x_4 = x_1x_2x_3$, it follows that $I = x_1x_2x_3x_4$. Similarly since $x_5 = x_2x_3$, it follows that $I = x_2x_3x_5$.

Each of the interactions $x_1x_2x_3x_4$ and $x_2x_3x_5$ is called a *generator* of this design. In general, any interaction that is "equal to" I for a 2^{k-q} design is a generator for that design. Now since $I = x_1x_2x_3x_4$ and $I = x_2x_3x_5$, the product of these two relationships, $I = x_1x_2^2x_3^2x_4x_5 = x_1x_4x_5$, reveals a third generator, $x_1x_4x_5$. When a design has more than one generator, it's defining relation must include each generator and all possible products of the generators. For this 2_{III}^{5-2} design, the defining relation is $I = x_1x_2x_3x_4 = x_2x_3x_5 = x_1x_4x_5$. Because the smallest order interaction in this defining relation is a three variable interaction, the resolution of this design is III.

From this defining relation it is apparent that this deign yields the following eight estimates. Interactions involving more than two operating variables have been ignored.

l_1, which estimates $(\beta_1 + \beta_{45})$
l_2, which estimates $(\beta_2 + \beta_{35})$
l_3, which estimates $(\beta_3 + \beta_{25})$
l_4, which estimates $(\beta_4 + \beta_{15})$
l_5, which estimates $(\beta_5 + \beta_{23} + \beta_{14})$
l_{12}, which estimates $(\beta_{12} + \beta_{34})$
l_{13}, which estimates $(\beta_{13} + \beta_{24})$
l_0, which estimates β_0

the "+1" is used find the constant term (it is not a parameter associated with an operating variable)

There exists a particularly economical group of resolution III designs known as *saturated designs*. With a design from this group, k operating variables can be investigated simultaneously in $(k+1)$ tests. First the case in which $(k+1)$ is a power of 2 will be discussed. Then the more general case in which $(k+1)$ is a multiple of 4 will be considered.

Ore popular saturated design is the 2^{7-4}_{III} design which can provide independent estimates of all of the parameters in the model

$$E(Y)=\beta_0+\beta_1x_1+\beta_2x_2+\beta_3x_3+\beta_4x_4+\beta_5x_5+\beta_6x_6+\beta_7x_7$$

although each of these parameters is confounded with a number of two variable interactions.

Construction of a 2^{7-4}_{III} design follows the pattern described above for a 2^{5-2}_{III} design. First a 2^3 design in three of the seven operating variables, x_1, x_2 and x_3, is written down. Each of the remaining operating variables, x_4, x_5, x_6 and x_7 is confounded with an interaction among x_1, x_2 and x_3. If the aliases $x_4=x_1x_2$, $x_5=x_1x_3$, $x_6=x_2x_3$ and $x_7=x_1x_2x_3$ are used, then the resulting design is that shown in table 23.3.

Table 23.3 - A saturated 2^{7-4}_{III} design.

x_1	x_2	x_3	x_4	x_5	x_6	x_7
−1	−1	−1	1	1	1	−1
1	−1	−1	−1	−1	1	1
−1	1	−1	−1	1	−1	1
1	1	−1	1	−1	−1	−1
−1	−1	1	1	−1	−1	1
1	−1	1	−1	1	−1	−1
−1	1	1	−1	−1	1	−1
1	1	1	1	1	1	1

From the four basic generators $x_1x_2x_4$, $x_1x_3x_5$, $x_2x_3x_6$ and $x_1x_2x_3x_7$ arising from the choice of aliases, the following defining relation for this design can be formed,

In a saturated design, ~~the~~ the number extra confounded terms is equal to the total number of combinations of unconfounded variables (excluding single term confounding, i.e. only ~~the~~ two factor confounding and up is considered).

2^{7-4}_{III} example in class.

*

$I = x_1x_2x_4 = x_1x_3x_5 = x_2x_3x_6 = x_1x_2x_3x_7$ (taking basic generators one at a time)

$= x_2x_3x_4x_5 = x_1x_3x_4x_6 = x_3x_4x_7 = x_1x_2x_5x_6 = x_2x_5x_7 = x_1x_6x_7$ (products of two basic generators)

$= x_4x_5x_6 = x_1x_4x_5x_7 = x_2x_4x_6x_7 = x_3x_5x_6x_7$ (products of three basic generators)

$= x_1x_2x_3x_4x_5x_6x_7$ (products of four basic generators)

Notice that the smallest order interaction in this defining relation is three, verifying that the resolution of the design is indeed III.

Again ignoring interactions involving more than two operating variables, the following eight estimates can be obtained from this design.

l_1, which estimates $(\beta_1 + \beta_{24} + \beta_{35} + \beta_{67})$
l_2, which estimates $(\beta_2 + \beta_{14} + \beta_{36} + \beta_{57})$
l_3, which estimates $(\beta_3 + \beta_{15} + \beta_{26} + \beta_{47})$
l_4, which estimates $(\beta_4 + \beta_{12} + \beta_{56} + \beta_{37})$
l_5, which estimates $(\beta_5 + \beta_{13} + \beta_{46} + \beta_{27})$
l_6, which estimates $(\beta_6 + \beta_{23} + \beta_{45} + \beta_{17})$
l_7, which estimates $(\beta_7 + \beta_{34} + \beta_{25} + \beta_{16})$
l_0, which estimates β_0

Interpretation of results from a saturated design may be ambiguous because each operating variable is confounded with a number of two variable interactions. As will be shown later in this section, ambiguities can be partially resolved by carrying out another saturated design from the same "family", that is, a design for which the signs of all values of one or more of the operating variables .are reversed. Thus saturated designs are more useful for the first step in a study of several operating variables. Before describing this procedure further, some additional examples of saturated designs are given.

Besides the 2^{7-4}_{III} design, commonly used saturated designs include 2^{3-1}_{III}, 2^{15-11}_{III} and 2^{31-26}_{III}. Choice of a defining relation for each of the latter three designs follows the pattern described above for the 2^{7-4}_{III} design. Each of the interactions involving the first $(k-q)$ operating variables is confounded with one of the remaining q operating variables.

Plackett and Burman [3] have developed saturated designs for experiments having a number of tests equal to a multiple of 4 (except for the case of 92 tests). For those cases in which the number of tests is equal to a power of 2, the Plackett and Burman designs are equivalent to those obtained by the procedures described above. For other cases, particularly experiments involving a study of eleven variables in twelve tests, or nineteen variables in twenty tests, the Plackett and Burman designs have been found to be very effective [4]. The Plackett and Burman saturated design for eleven variables is shown in table 23.4.

Table 23.4 - A saturated design for eleven operating variables [3]

x_1	x_2	x_3	x_4	x_5	x_6	x_7	x_8	x_9	x_{10}	x_{11}
1	1	−1.	1	1	1	−1	−1	−1	1	−1
−1	1	1	−1	1	1	1	−1	−1	−1	1
1	−1	1	1	−1	1	1	1	−1	−1	−1
−1	1	−1	1	1	−1	1	1	1	−1	−1
−1	−1	1	−1	1	1	−1	1	1	1	−1
−1	−1	−1	1	−1	1	1	−1	1	1	1
1	−1	−1	−1	1	−1	1	1	−1	1	1
1	1	−1	−1	−1	1	−1	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

As indicated earlier, ambiguities in the interpretation of results from a 2^{k-q} design may be resolved by carrying out another 2^{k-q} design from the same family. One useful choice for a second design is that for which the signs of all of the values for one operating variable are reversed. If interactions involving three or more operating variables are again ignored, then it can be shown [1] that combining the results of this second design with those of the original design yields unconfounded estimates of the effect of that operating variable and each two variable interaction involving that variable. Another useful choice for a second design is that for which the signs of the values for all of the operating variables are reversed. The use of this procedure is illustrated by the following example which was discussed by Box and Hunter [1].

During the startup of a new process in a chemical plant, trouble was encountered in a filtration operation. Filtration was requiring about 70 minutes per batch instead of 40 minutes, the required time for a similar operation at other plant sites. An investigation was undertaken to identify the operating variables that affected filtration time and to determine how these variables might be altered in order to reduce the filtration time. The operating variables selected for the initial study are shown in table 23.5. The low levels represent the operating conditions prior to this screening study. The high levels are changes in operating conditions chosen to identify which, if any, of these seven operating variables affected the filtration time. It will be noted that four of the operating variables, x_1, x_2, x_5 and x_7 are qualitative variables.

Table 23.5 - Operating variables for the filtration study.

Operating Variable		Level -1	Level 1
x_1,	water supply	municipal reservoir	well
x_2,	raw material	made on site	made at another site
x_3,	filtration temperature	low	high
x_4,	holdup time	short	long
x_5,	recycle	included	omitted
x_6,	rate of addition of NaOH	fast	slow
x_7,	type of filter cloth	new	old

As a first step in the study, a 2^{7-4}_{III} design was employed because of it's economy in tests and its facility for use as a building block for further tests that might be required. Basic generators chosen for the design were $x_1x_2x_3x_4$, $x_1x_2x_5$, $x_1x_3x_6$ and $x_2x_3x_7$, producing the following defining relation:

$$\begin{aligned} I &= x_1x_2x_3x_4 = x_1x_2x_5 = x_1x_3x_6 = x_2x_3x_7 \\ &= x_3x_4x_5 = x_2x_4x_6 = x_1x_4x_7 = x_2x_3x_5x_6 = x_1x_3x_5x_7 = x_1x_2x_6x_7 \\ &= x_1x_4x_5x_6 = x_2x_4x_5x_7 = x_3x_4x_6x_7 = x_5x_6x_7 = x_1x_2x_3x_4x_5x_6x_7 \end{aligned} \tag{23.3}$$

The design and the measured steady state filtration time for each test are shown in table 23.6.

Table 23.6 - Results from a 2_{III}^{7-4} filtration design

x_1	x_2	x_3	x_4	x_5	x_6	x_7	y (min.)
−1	−1	−1	−1	1	1	1	68.4
1	−1	−1	1	−1	−1	1	77.7
−1	1	−1	1	−1	1	−1	66.4
1	1	−1	−1	1	−1	−1	81.0
−1	−1	1	1	1	−1	−1	78.6
1	−1	1	−1	−1	1	−1	41.2
−1	1	1	−1	−1	−1	1	68.7
1	1	1	1	1	1	1	38.7

When these results were examined, there may well have been a temptation to conclude that either the sixth test or the eighth test in table 23.6 had resolved the problem since both tests produced filtration times in the order of 40 minutes, the target figure. As will be shown shortly, a conclusion that changes in x_1, x_3 and x_6 produced this favourable result is only one of several possible interpretations of these data. In any case, before making a change in such an important operating variable as water supply (x_1), other interpretations would have had to be assessed. From the defining relation for this design it can be confirmed that the following eight estimates are obtained Interactions involving more than two operating variables have been ignored. Because further tests were carried out in this study, a second subscript has been added to these estimates to denote that they arise from the first set of tests.

l_{11}, an estimate of $(\beta_1 + \beta_{25} + \beta_{36} + \beta_{47})$, $= -5.4$
l_{21}, an estimate of $(\beta_2 + \beta_{15} + \beta_{37} + \beta_{46})$, $= -1.4$
l_{31}, an estimate of $(\beta_3 + \beta_{16} + \beta_{27} + \beta_{45})$, $= -8.3$
l_{41}, an estimate of $(\beta_4 + \beta_{35} + \beta_{26} + \beta_{17})$, $= 0.3$
l_{51}, an estimate of $(\beta_5 + \beta_{12} + \beta_{34} + \beta_{67})$, $= 1.6$
l_{61}, an estimate of $(\beta_6 + \beta_{13} + \beta_{24} + \beta_{57})$, $= -11.4$
l_{71}, an estimate of $(\beta_7 + \beta_{23} + \beta_{14} + \beta_{56})$, $= -1.7$
l_{01}, an estimate of β_0, $= 65.1$

Because no estimate of the pure error variance was reported [1], interpretation of these estimates can be made only on the basis of their relative magnitudes. Among the coefficients of operating variables, the estimates l_6, l_3 and l_1 are much larger in magnitude than the other estimates. A number of alternative interpretations are possible, including the following.

A simple explanation of these three large estimates might be that only the terms $\beta_1 x_1$, $\beta_3 x_3$ and $\beta_6 x_6$ are important, all two variable interactions being of negligible size. Another explanation might be that only operating variables x_1 and x_3 are affecting the filtration time, their influence being explained by terms $\beta_1 x_1$, $\beta_3 x_3$ and $\beta_{13} x_1 x_3$. A third possibility is that only operating variables x_1 and x_6 are important, their effect being accounted for by terms $\beta_1 x_1$, $\beta_6 x_6$ and $\beta_{16} x_1 x_6$. Another alternative is that only operating variables x_3 and x_6; are causing 'the response to change, via terms $\beta_3 x_3$, $\beta_6 x_6$ and $\beta_{36} x_3 x_6$. Many other explanations are possible as well.

Because of the ambiguity in interpreting the results of these tests, a second set of eight tests was conducted using a 2_{III}^{7-4} design formed from the design in table 23.6 by reversing the signs of all values for all seven operating variables. This second design is shown in table 23.7 along with the measured filtration times obtained for these additional tests.

Table 23.7 - Results from a second 2_{III}^{7-4} filtration design

x_1	x_2	x_3	x_4	x_5	x_6	x_7	y (min.)
1	1	1	1	−1	−1	−1	66.7
−1	1	1	−1	1	1	−1	65.0
1	−1	1	−1	1	−1	1	86.4
−1	−1	1	1	−1	1	1	61.9
1	1	−1	−1	−1	1	1	47.8
−1	1	−1	1	1	−1	1	59.0
1	−1	−1	1	1	1	−1	42.6
−1	−1	−1	−1	−1	−1	−1	67.6

Switching the signs of all values for one operating variable x_i in a 2_{III}^{7-4} design is equivalent to replacing x_i with $-x_i$. Because of the manner in which this second 2_{III}^{7-4} design has been constructed from the first 2_{III}^{7-4} design, its defining relation can be obtained by replacing every operating variable x_i in (23.3), the defining relation for the first design, by $-x_i$. The resulting defining relation is

$$\begin{aligned} I &= x_1x_2x_3x_4 = -x_1x_2x_5 = -x_1x_3x_6 = -x_2x_3x_7 \\ &= -x_3x_4x_5 = -x_2x_4x_6 = -x_1x_4x_7 = x_2x_3x_5x_6 = x_1x_3x_5x_7 = x_1x_2x_6x_7 \\ &= x_1x_4x_5x_6 = x_2x_4x_5x_7 = x_3x_4x_6x_7 = -x_5x_6x_7 = -x_1x_2x_3x_4x_5x_6x_7 \end{aligned} \tag{23.4}$$

From the defining relation (23.4), again ignoring interactions involving more than two operating variables, it can be confirmed that the following eight estimates are obtained from the second set of tests.

l_{12}, an estimate of $(\beta_1 - \beta_{25} - \beta_{36} - \beta_{47})$, $= -1.3$
l_{22}, an estimate of $(\beta_2 - \beta_{15} - \beta_{37} - \beta_{46})$, $= -2.5$
l_{32}, an estimate of $(\beta_3 - \beta_{16} - \beta_{27} - \beta_{45})$, $= 7.9$
l_{42}, an estimate of $(\beta_4 - \beta_{35} - \beta_{26} - \beta_{17})$, $= -4.6$
l_{52}, an estimate of $(\beta_5 - \beta_{12} - \beta_{34} - \beta_{67})$, $= 1.1$
l_{62}, an estimate of $(\beta_6 - \beta_{13} - \beta_{24} - \beta_{57})$, $= -7.8$
l_{72}, an estimate of $(\beta_7 - \beta_{23} - \beta_{14} - \beta_{56})$, $= 1.7$
l_{02}, an estimate of β_0, $= 62.1$

The results from the first set of eight tests can be combined with those from the second set of eight tests to yield the following sixteen estimates.

$(l_{11} + l_{12})/2$, an estimate of β_1, $= -3.4$
$(l_{21} + l_{22})/2$, an estimate of β_2, $= -2.0$
$(l_{31} + l_{32})/2$, an estimate of β_3, $= -0.2$
$(l_{41} + l_{42})/2$, an estimate of β_4, $= -2.2$
$(l_{51} + l_{52})/2$, an estimate of β_5, $= 1.4$
$(l_{61} + l_{62})/2$, an estimate of β_6, $= -9.6$
$(l_{71} + l_{72})/2$, an estimate of β_7, $= 0.0$
$(l_{01} + l_{02})/2$, an estimate of β_0, $= 63.6$
$(l_{11} - l_{12})/2$, an estimate of $(\beta_{25} + \beta_{36} + \beta_{47})$, $= -2.1$
$(l_{21} - l_{22})/2$, an estimate of $(\beta_{15} + \beta_{37} + \beta_{46})$, $= 0.6$
$(l_{31} - l_{32})/2$, an estimate of $(\beta_{16} + \beta_{27} + \beta_{45})$, $= -8.1$
$(l_{41} - l_{42})/2$, an estimate of $(\beta_{35} + \beta_{26} + \beta_{17})$, $= 2.5$
$(l_{51} - l_{52})/2$, an estimate of $(\beta_{12} + \beta_{34} + \beta_{67})$, $= 0.3$
$(l_{61} - l_{62})/2$, an estimate of $(\beta_{13} + \beta_{24} + \beta_{57})$, $= -1.8$
$(l_{71} - l_{72})/2$, an estimate of $(\beta_{23} + \beta_{14} + \beta_{56})$, $= -1.7$
$(l_{01} - l_{02})/2$, an estimate of the block effect, $= -1.5$

The *block effect* is the difference in average response values between the two sets of eight runs. Had it been large, it would have indicated the presence of some other variables, beyond the seven being studied, whose change between the two sets of tests strongly affected the filtration time.

Among the above sixteen estimates, the largest is -9.6, an estimate of β_6 and -8 1, an estimate of the linear combination of two variable interactions $(\beta_{16} + \beta_{27} + \beta_{45})$.. The next largest estimate is -3.4, an estimate of β_1. The investigators concluded that operating variables x_1 and x_6 alone, the water supply and the rate of addition of caustic soda, affected the filtration time, and the estimate -8.1 occurred primarily because of the interaction x_1x_6. Even at this stage, of course, other interpretations are possible. For example, the estimate -8.1 might have been due to the interaction x_2x_7 and/or the interaction x_4x_5. It is noted, however, that the estimates of $\beta_2, \beta_7, \beta_4$ and β_5 are all relatively small and, although this does not necessarily mean. that interactions, among these variables must also be small, this is often the case.

The investigators' interpretation can be summarized conveniently by figure 23.1 which shows the average filtration time obtained at each of the four sets of operating conditions. Evidence of the large negative interaction between the two variables is very strong.

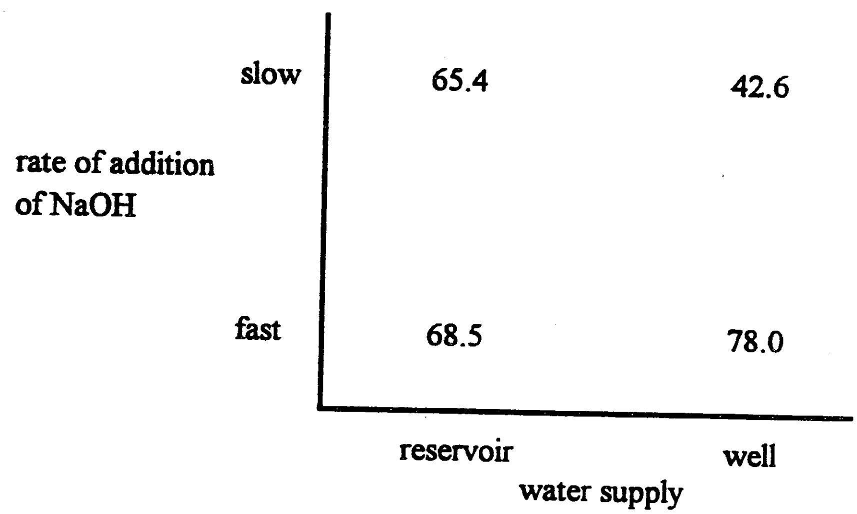

Figure 23.1 - Results of filtration study.

The corrective action implied by these results was to change the water supply from the municipal reservoir to the well and reduce the rate of addition of caustic soda. These changes were made and satisfactory filtration times close to 40 minutes were obtained in subsequent plant operation.

2^{k-q} designs of resolution IV or V are preferred in some screening studies because the effects of individual operating variables are not confounded with two variable interactions. An example of each of these classes of fractional factorial designs is now given.

A 2_{IV}^{7-3} design is shown in table 23.8. It has been constructed by forming a 2^4 design in operating variables x_1, x_2, x_3 and x_5, then confounding x_4 with $x_1x_2x_3$, x_6 with $x_2x_3x_5$ and x_7 with $x_1x_3x_5$. The defining relation is

$$\begin{aligned} I &= x_1x_2x_3x_4 = x_2x_3x_5x_6 = x_1x_3x_5x_7 = x_1x_4x_5x_6 \\ &= x_2x_4x_5x_7 = x_1x_2x_6x_7 = x_3x_4x_6x_7 \end{aligned} \tag{23.5}$$

This design is in fact the combination of the two sets of eight runs used in the filtration example. The seven generators in (23.5) are those that appear in both (23.3) and (23.4). In constructing a resolution IV design it is important to write out the full defining relation to confirm that it contains no interaction involving fewer than four operating variables.

Table 23.8 - A 2_{IV}^{7-3} design.

x_1	x_2	x_3	x_4	x_5	x_6	x_7
−1	−1	−1	−1	−1	−1	−1
1	−1	−1	1	−1	−1	1
−1	1	−1	1	−1	1	−1
1	1	−1	−1	−1	1	1
−1	−1	1	1	−1	1	1
1	−1	1	−1	−1	1	−1
−1	1	1	−1	−1	−1	1
1	1	1	1	−1	−1	−1
−1	−1	−1	−1	1	1	1
1	−1	−1	1	1	1	−1
−1	1	−1	1	1	−1	1
1	1	−1	−1	1	−1	−1
−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

A 2_{V}^{8-2} design can be constructed as follows. First a 2^6 design in any six of the eight operating variables, say x_1, x_2, x_3, x_4, x_5 and x_6, is written down. Each of the remaining two operating variables, x_7 and x_8, is confounded with an interaction involving at least four of the variables x_1, x_2, x_3, x_4, x_5 and x_6. Once again, the full defining relation for the design should be written out to confirm that it contains only interactions involving five or more operating variables. If x_7 is confounded with $x_1x_2x_3x_4$ and x_8 is confounded with $x_1x_2x_5x_6$, then the resulting defining relation is

$$I = x_1x_2x_3x_4x_7 = x_1x_2x_5x_6x_8 = x_3x_4x_5x_6x_7x_8 .$$

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CHAPTER 24

Using a Steepest Ascent Strategy in Process Investigations

The experimental design strategies discussed in chapters 22 and 23 are most useful in the initial stages of a process investigation. Their effectiveness in identifying operating variables that strongly influence the response has been demonstrated. Efficient estimates of individual and joint effects of these variables are also obtained from these designs. Although this information is basic to gaining a quantitative understanding of the operation of a process, it rarely satisfies the ultimate goals of a study.

One common objective in a process investigation is the development of an adequate mathematical model. for the behaviour of a process within a defined operating region. More than two levels of each operating variable must be tested to ensure that this is achieved. Experimental strategies for developing more comprehensive empirical models of process behaviour are described in the next section.

Another common objective is the improvement of process performance, perhaps leading eventually to the location of optimum operating conditions. Many algorithms for optimization of a mathematical function have been proposed [1], but almost all of them operate on the assumption that the response variable is error free. In process improvement studies involving the collection and interpretation of measured data, account must be taken not only of the effect of experimental error, but of the adequacy of the fitted model.

In this section, the steepest ascent strategy for process improvement is described. Like many optimum seeking strategies, it is sequential in nature, individual tests being planned on the basis of information derived from preceding tests. A graphical representation of this strategy is shown in figure 24.1. The dotted curves in that figure are contours of constant response values. Together they represent a surface describing the relationship. between the response and the two operating variables. The concept of a response surface, introduced by Box and Wilson [2], is a useful one even for situations involving more than two operating variables.

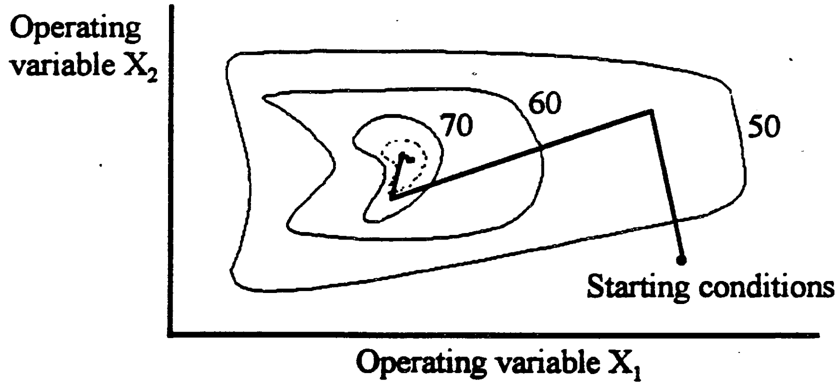

Figure 24.1 - Climbing a response surface by steepest ascent.

In the early stages of a process investigation, the nature of the response surface is unknown. A few tests may have been carried out from which some information is available about promising directions for changes in operating conditions. The steepest ascent direction at any point is that for which greatest increase in the response is expected per unit change in operating conditions. As indicated in figure 24.1, this direction is orthogonal to the contour passing through the point from which changes are made. If an increase in the response is desired, then changes in operating conditions should be made in this direction. Eventually a point will be reached at which the response is no longer improving. Another set of tests must then be carried out to determine a new direction of steepest ascent. This direction is then followed and so on.

Eventually, perhaps even after the first set of tests, the slope of the response surface will begin to approach zero, indicating that a stationary region of the surface has been reached. This may be close to an optimum or it may be a ridge or a plateau. Further tests are necessary to identify the nature of a stationary region and strategies for planning these tests are described in the next section.

Use of a steepest ascent strategy in an experimental situation is now illustrated by an example based on a study reported by Box [3] and also described by Davies [4]. In a two stage reaction

$$A + B \rightarrow C$$
$$A + C \rightarrow D$$

the objective is to increase the yield of the product C while maintaining the yield of D below 20 per cent. The average yield of C during recent operation has been 49.4 per cent with an error variance of 1.1 at steady state conditions. This variance value is based upon a sufficiently large number of measurements that it can be regarded as a "known" value.

Of five operating variables initially selected for study, only the three shown in table 24.1 were found to affect the yield of C significantly in the defined operating region. In arriving at this conclusion from a group of two level tests, it must be recognized that the parameter estimate associated with an individual operating variable may be close to zero for any of the following reasons:

(i) The two levels for this operating variable may have straddled a conditional maximum or minimum of the response.
(ii) The two levels may have been so closely spaced that any effect of the operating variable on the response was masked by experimental error.
(iii) Changes in this operating variable over the range of interest have no appreciable effect on the response.

Only by testing the operating variable in question at additional more widely spaced levels can the correct explanation be found.

Table 24.1 - Operating variables in a yield study.

Operating Variable	Low Level	High Level
Temperature	150°C	160°C
Initial concentration of A	40%	45%
Reaction time	10 hours	13 hours

Eight tests were carried out using a 2^3 design with operating variable levels shown in table 24.1. With coded variables defined as

$$
\begin{aligned}
x_1 &= \frac{(\text{temperature} - 155)}{5} \\
x_2 &= \frac{(\text{concentration of A} - 42.5)}{2.5} \\
x_3 &= \frac{(\text{reaction time} - 11.5)}{1.5}
\end{aligned}
\qquad (24.1)
$$

the results from these tests are shown in table 24.2. The tests were carried out in random order, not in the order shown in this table.

Table 24.2 - Yield results from a 2^3 design.

x_1	x_2	x_3	measured yield of C (%) y
−1	−1	−1	47.1
1	−1	−1	37.9
−1	1	−1	45.5
1	1	−1	36.5
−1	−1	1	44.0
1	−1	1	38.4
−1	1	1	42.6
1	1	1	33.4

As shown in chapter 22, uncorrelated estimates of all of the parameters in the model

$$\begin{aligned} E(Y) = \beta_0 &+ \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 \\ &+ \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \\ &+ \beta_{123} x_1 x_2 x_3 \end{aligned} \tag{24.2}$$

can be obtained from these data. A confidence interval for the true value of each parameter can also be calculated. Because the pure error variance value, 1.1, is "known", a 95 per cent confidence interval for any parameter β in (24.2) is given by the expression

$$\hat{\beta} \pm 1.96\sqrt{\frac{1.1}{8}} = \hat{\beta} \pm 0.73.$$

Parameter estimates and individual 95 per cent confidence intervals are given in table 24.3.

Table 24.3 - Parameter estimates from a 2^3 design.

Parameter	95 per cent confidence interval
β_0	40.68 ± 0.73
β_1	-4.13 ± 0.73
β_2	-1.18 ± 0.73
β_3	-1.08 ± 0.73
β_{12}	-0.43 ± 0.73
β_{13}	0.43 ± 0.73
β_{23}	-0.43 ± 0.73
β_{123}	-0.48 ± 0.73

These parameter estimates can be interpreted in groups. $\hat{\beta}_0 = 40.68$ is an estimate of the yield of C at the centre point of the 2^3 design, $(x_1, x_2, x_3) = (0,0,0)$. If his point is close to the standard conditions at which an average yield of C of 49.4 per cent has been obtained then, as shown in the profile in figure 24.2, the response surface must be somewhat dome shaped within the operating region tested. If the response surface were more like a plane, then the value $\hat{\beta}_0$ should have been closer to 49.4.

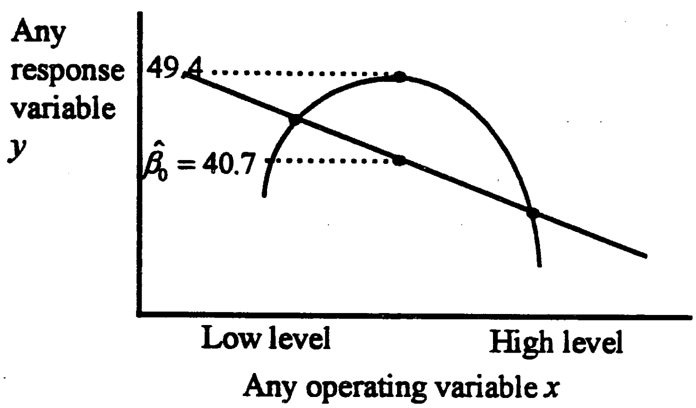

Figure 24.2 - A profile of the response surface.

As seen in table 24.3, the four interaction terms are all of negligible size, suggesting that any curvature in the response surface must be due to quadratic behaviour in one or more of the three operating variables.

The three first order terms, $\beta_1 x_1$, $\beta_2 x_2$ and $\beta_3 x_3$, are all distinctly non-zero. Their negative signs indicate that the levels of all three operating variables should be reduced in order to increase the yield of C.

Use of a steepest ascent strategy to change operating conditions is reasonable only if the relationship between the response and the operating variables can be represented adequately by a plane. From the results of these eight tests the fitted plane

$$\hat{y} = 40.68 - 4.13x_1 - 1.18x_2 - 1.08x_3 \tag{24.3}$$

does appear to be a reasonable model within the operating region defined in table 24.1 even though there is the evidence of curvature near the centre of this region as described above.

The path of steepest ascent from the centre point of the design can be calculated as shown in table 24.4. The centre point and the value of one coded unit for each operating variable have been defined in the coding definitions (24.1). The third line of the table is the steepest ascent direction expressed in terms of the coded operating variables and the fourth line gives this direction in terms of the original units of the operating variables. For example, 4.13 coded units of temperature is $4.13(5) = 20.65\,°C$. In the fifth line, the steepest ascent direction. is expressed in. terms of reasonably sized changes in the three operating variables, in this case, changes corresponding to 10°C decreases in temperature. The remaining lines in the table show values for the three operating variables along the direction of steepest ascent, moving from the centre point of the design.

Table 24.4 - Calculation of path of steepest ascent

	Temperature (°C)	Concentration of A (%)	Reaction time (hrs)
Design centre point	155	42.5	11.5
Value of one coded unit	5	2.5	1.5
Steepest ascent direction (coded units)	-4.13	-1.18	-1.08
Steepest ascent direction (original units)	-20.65	-2.95	-1.62
Steepest ascent direction (original units)	-10	-1.43	-0.78
Steepest ascent path	155	42.5	11.5
	145	41.1	10.7
	135	39.6	9.9
	125	38.2	9.2
	115	36.8	8.4

Having determined the direction of steepest ascent, it must now be decided how far along this path the next test should be carried out. The fitted linear model (24.3) predicts an ever increasing yield of C as the distance from the design centre point increases, but of course the adequacy of this model has been tested only within the operating region defined in table 24.1. The dangers of excessive extrapolation using an empirical model were discussed in section 19. In this example, too large an extrapolation would overshoot the maximum yield conditions. Too small a step along the steepest ascent path would increase the number of tests required to achieve maximum increase in the yield of C. In practice, this decision must be made subjectively using all relevant information about the process. Tests are made at a succession of operating conditions along the path of steepest ascent until the yield of C ceases to increase.

In this study, the three tests shown in table 24.5 were carried out with the results indicated.

Table 24.5 - Tests along the path of steepest ascent

Step	Temperature (°C)	Concentration of A (%)	Reaction time (hrs)	Measured yield of C (%)
1	145	41.1	10.7	50.8
2	125	38.2	9.2	54.0
3	115	36.8	8.4	51.5

The sharp reduction in yield obtained at step 3 indicated that further moves along this path would be unprofitable. A new direction of steepest ascent was required and another 2^3 design was used to determine it. The centre point operating conditions for this design were those for step 2 in the previous ascent exploration, the most successful conditions found to this time. Because of the suspected curvature in the response surface representing the first eight tests, the size of a coded unit for each operating variable was reduced for this new design in the hope that a plane approximation to the surface might again be adequate. The results of this second set of eight tests are shown in table 24.6. The tests were again carried out in random order.

Table 24.6 - A second 2^3 design.

Temperature (C)	Concentration of A (%)	Reaction time (hrs)	Measured yield of C (%)
122	36.8	8.2	43.9
128	36.8	8.2	58.6
122	39.6	8.2	55.5
128	39.6	8.2	56.6
122	36.8	10.2	51.3
128	36.8	10.2	56.0
122	39.6	10.2	56.8
128	39.6	10.2	50.6

Model (24.2) was fitted to these data with the following results,

$$\hat{y} = 53.7 + 1.8x_1 + 1.2x_2 + (0)x_3 - 3.1x_1x_2 - 2.2x_1x_3 - 1.2x_2x_3 + 0.4x_1x_2x_3$$

95 per cent confidence limits for the true value of each parameter are again ± 0.73.

The fact that the coefficients of the two variable interaction terms are now significantly non-zero, and in fact larger in magnitude than those of the individual operating variables, is evidence that a plane cannot provide an adequate representation of the response surface in the operating region defined by these tests. A path of steepest ascent can no longer be easily determined as it will change with every change in an operating variable. Additional. tests at other levels of the operating variables are required to characterize the shape of the response surface in this region. Strategies for planning these additional tests are described in the next chapter.

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CHAPTER 25

*use raw centerpoint to check if variance is changing with operating conditions.

Exploring a Stationary Region

If a polynomial model is to be used to describe the behaviour of a response function in a stationary region, then the polynomial must be of at least second degree. For k operating variables, a second degree polynomial model has the form

$$\begin{aligned} E(Y) = \beta_0 &+ \beta_1 x_1 + \beta_2 x_2 + \cdots + \beta_k x_k \\ &+ \beta_{11} x_1^2 + \beta_{22} x_2^2 + \cdots + \beta_{kk} x_k^2 \\ &+ \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \cdots + \beta_{k-1,k} x_{k-1} x_k \end{aligned} \tag{25.1}$$

Such a model form can represent a local optimum, a saddle point or various types of ridges.

In order to estimate the coefficients of the squared terms, such as $\beta_{11}x_1^2$ in model (25.1), at least three values of each operating variable must be tested. Experimental designs for models of the general form (25.1) are *called second order designs*. In view of the success of two-level factorial designs for first order models, it might be expected that three-level factorial designs would be similarly effective for second order models. This is not true in general. Although three-level factorial designs are efficient in situations involving two or three operating variables, the numbers of runs required to estimate the coefficients in model form (25.1) for larger numbers of operating variables are unacceptably large.

* Box and Behnken [1] have proposed a useful class of three-level fractional factorial designs for estimating the coefficients in a second degree polynomial model. Three of these designs are shown below.

The notation can be explained using the design for three operating variables as an example. The first line, $x_1 = \pm 1, x_2 = \pm 1, x_3 = 0$ represents four experimental runs,

x_1	x_2	x_3
−1	−1	0
1	−1	0
−1	1	0
1	1	0

The fourth line represents three runs at the centre point of the design. An application of the Box and Behnken design for four operating variables has been described by Bacon [2].

Design for 3 Operating Variables

x_1	x_2	x_3	No. of runs
±1	±1	0	4
±1	0	±1	4
0	±1	±1	4
0	0	0	3
			Total 15 runs

Design for 4 Operating Variables

x_1	x_2	x_3	x_4	No. of runs
±1	±1	0	0	4
0	0	±1	±1	4
±1	0	0	±1	4
0	±1	±1	0	4
0	±1	0	±1	4
0	0	0	0	3
				Total 27 runs

Design for 7 Operating Variables

x_1	x_2	x_3	x_4	x_5	x_6	x_7	No. of runs
0	0	0	±1	±1	±1	0	8
±1	0	0	0	0	±1	±1	8
0	±1	0	0	±1	0	±1	8
±1	±1	0	±1	0	0	0	8
0	0	±1	±1	0	0	±1	8
±1	0	±1	0	±1	0	0	8
0	±1	±1	0	0	±1	0	8
0	0	0	0	0	0	0	6
							Total 62 runs

Box and Wilson [3], in the first paper on response surface methodology to appear in the literature, introduced the *central composite design* for second order models. As its name suggests, this design consists of components, one of which is a two-level factorial or fractional factorial design. Construction of a central composite design for the case of three operating variables is illustrated in figure 25.1. Values of the operating variables are quoted in coded form. As indicated in this figure, the complete design is formed by adding axial points and centre points to a two-level factorial design. The axial points are situated in pairs along the axis of each operating variable, each member of a pair lying α coded units from the centre of the design. Optimal values for α and the number of centre points for different numbers of operating variables have been proposed by Box and Hunter [4] using two different criteria, *orthogonality* and *uniform precision*.

An experimental design is said to be "orthogonal" if it yields an $\mathbf{X}^T\mathbf{X}$ matrix that is diagonal for the model considered. As can be easily verified, a diagonal $\mathbf{X}^T\mathbf{X}$ matrix is impossible to attain for a model written in the form

$$\begin{aligned} y = \beta_0 &+ \beta_1 x_1 + \beta_2 x_2 + \cdots + \beta_k x_k \\ &+ \beta_{11} x_1^2 + \beta_{22} x_2^2 + \cdots + \beta_{kk} x_k^2 \\ &+ \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \cdots + \beta_{k-1,k} x_{k-1} x_k \\ &+ \varepsilon \end{aligned} \tag{25.2}$$

even if the variables $x_1, x_2, \cdots, x_k$ are considered in their usual scaled form, because the quadratic coefficients β_{11}, β_{22}, etc. are correlated with each other and with β_0.

Let us assume that the k original operating variables are $\xi_1, \xi_2, \cdots, \xi_k$ and that the corresponding scaled variables $x_1, x_2, \cdots, x_k$ are defined as

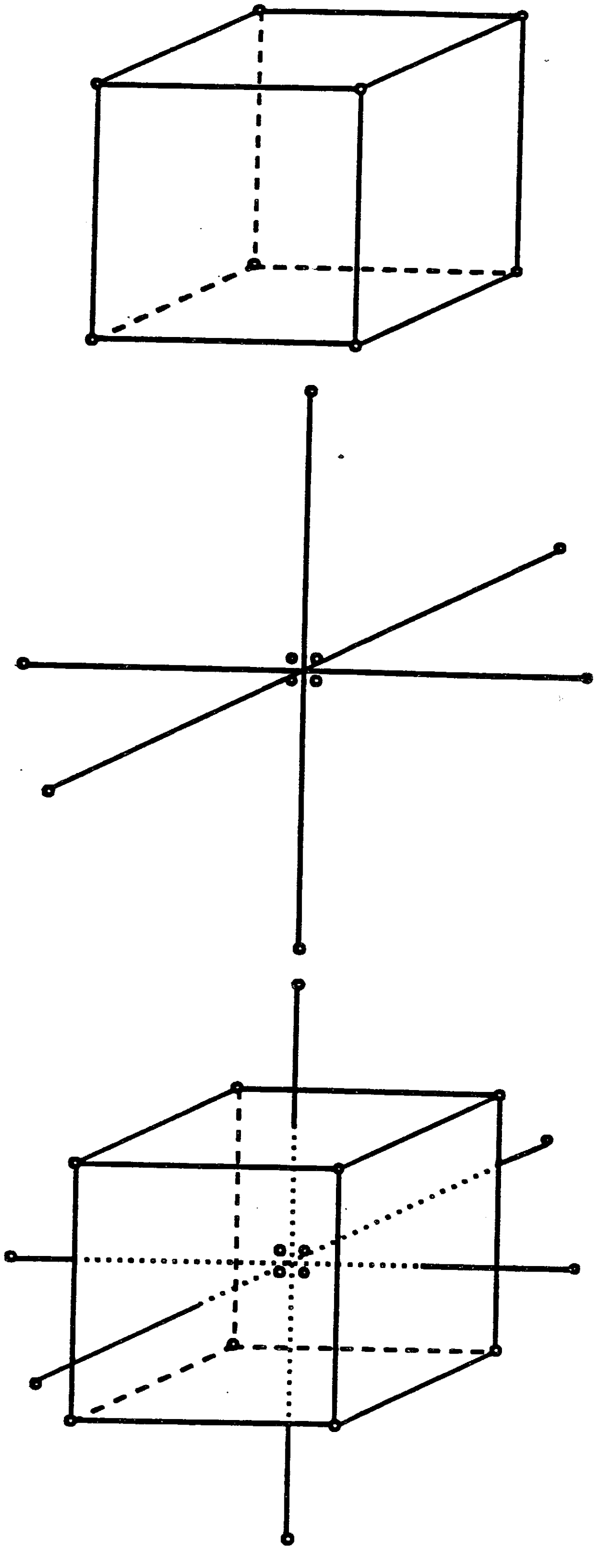
$$x_j = \frac{\xi_j - \bar{\xi}_j}{\sqrt{\dfrac{\sum_{u=1}^{n} \left(\xi_{uj} - \bar{\xi}_j\right)^2}{n}}}, \qquad j = 1,2,\cdots,k$$

where ξ_{uj} is the value of the operating variable ξ_j for run $u, u = 1,2,\cdots,n$ and

$$\bar{\xi}_j = \frac{\sum_{u=1}^{n} \xi_{uj}}{n}, \; j = 1,2,\cdots,k.$$

Now it is possible to express model (25.2) in an alternative but equivalent form involving *orthogonal polynomials* of the quadratic variables. For the case of only two operating variables, x_1 and x_2, model (25.2) can be rewritten as

$$y = \gamma_0 + \gamma_1 x_1 + \gamma_2 x_2 + \gamma_{11} w_1 + \gamma_{22} w_2 + \gamma_{12} x_1 x_2 + \varepsilon \tag{25.3}$$



Experimental Runs

X_1	X_2	X_3
-1	-1	-1
1	-1	-1
-1	1	-1
1	1	-1
-1	-1	1
1	-1	1
-1	1	1
1	1	1
α	0	0
$-\alpha$	0	0
0	α	0
0	$-\alpha$	0
0	0	α
0	0	$-\alpha$
0	0	0
0	0	0
0	0	0
0	0	0

-1	-1	-1
1	-1	-1
-1	1	-1
1	1	-1
-1	-1	1
1	-1	1
-1	1	1
1	1	1
α	0	0
$-\alpha$	0	0
0	α	0
0	$-\alpha$	0
0	0	α
0	0	$-\alpha$
0	0	0
0	0	0
0	0	0
0	0	0

Figure 25.1 - A Central Composite Design for three operating variables.

$$
\begin{aligned}
w_1 &= x_1^2 - [111]x_1 - 1 \\
w_2 &= x_2^2 - [222]x_2 - 1 \\
\gamma_0 &= \beta_0 + \beta_{11} + \beta_{22}
\end{aligned}
$$

where
$$
\begin{aligned}
\gamma_1 &= \beta_1 + [111]\beta_{11} \\
\gamma_2 &= \beta_2 + [222]\beta_{22} \\
\gamma_{11} &= \beta_{11} \\
\gamma_{22} &= \beta_{22} \\
\gamma_{12} &= \beta_{12}
\end{aligned}
$$

and
$$
[111] = \frac{\sum_{u=1}^{n} x_{1u}^3}{n}
$$
$$
[222] = \frac{\sum_{u=1}^{n} x_{2u}^3}{n}
$$

and n = number of data points used to fit the model (25.3).

If model (25.2) is written in the form (25.3), then, for an appropriate selection of run conditions (i.e. for an appropriately selected experimental design), the "new" $\mathbf{X}^{\mathrm{T}}\mathbf{X}$ matrix involving x_1, x_2, w_1, w_2 and x_1x_2, will be diagonal so that estimates of all the γ 's in (25.3) will be uncorrelated. The estimates of the β's in (25.2) can be found directly from the estimates of the γ 's.

It is then possible to speak of "orthogonal" designs for a model such as (25.2) if this model is transformed into model (25.3) using orthogonal polynomials. A fuller discussion of this concept is given by Box and Hunter [4].

Box and Hunter have also introduced the concept of *rotatability*. In simple terms, a *rotatable* design is one that produces estimated response values with equal variances at all points equidistant from the centre of the design.

For example, if a rotatable design has been used to gather experimental data using two operating variables, x_1 and x_2, then

$\hat{y}$ for $x_1 = 1$ and $x_2 = 1$

will have the same variance as

$\hat{y}$ for $x_1 = -0.5$ and $x_2 = 1.32$

rotatable = same precision for all estimates
@ given distance from the center point

this doesn't mean variance is constant

since both of these operating conditions are the same distance from the centre of the design, $x_1 = x_2 = 0$.

By suitable location of the points in a second order design, both rotatability and *uniform precision* can be achieved. Uniform precision is somewhat of a misnomer because what is meant in fact is a rotatable design that produces predicted responses at the design centre (i.e. $x_1 = x_2 = 0$) and at all points at distance 1 from the design centre (e.g. $x_1 = 0.3, x_2 = 0.95$) with equal variance. That is,

$$\mathrm{var}(\hat{y} \text{ at } x_1 = x_2 = 0) = \mathrm{var}(\hat{y} \text{ at any } x_1 \text{ and } x_2 \text{ such that } x_1^2 + x_2^2 = 1)$$

Uniform precision is accomplished in a rotatable design by inclusion of an appropriate number of centre points.

Table 25.1 [4] gives the following characteristics for a number of central composite designs,

(i) n_f, the number of design points in the factorial portion,

(ii) n_a, the number of design points in the star portion,

(iii) n_c, the number of centre points for uniform precision (up) and orthogonality (orth),

(iv) N, the total number of design points, $n_f + n_a + n_c$, and

(v) α, the distance of star points from the design centre.

Table 25.1 - Characteristics of some Central Composite Designs.

k (no. of factors)		2	3	4	5	$5\frac{1}{2}$ fraction	6	$6\frac{1}{2}$ fraction
n_f		4	8	16	32	16	64	32
n_a		4	6	8	10	10	12	12
n_c	(up)	5	6	7	10	6	15	9
	(orth)	8	9	12	17	10	24	15
N	(up)	13	20	31	52	32	91	53
	(orth)	16	23	36	59	36	100	59
α		1.414	1.682	2.000	2.378	2.000	2.828	2.378

For 2-level designs (originally...)

In the yield study discussed in section 24, the results of the second 2^3 design indicated the need for additional experimental runs at other operating conditions to

α cannot be made/determined for "qualitative" variables account for the curvature in the response surface. The additional runs chosen were six axial points and four centre points, which, in combination with the eight points of the last 2^3 design, formed a central composite design. These additional runs and the measured response values that were obtained are shown in table 25.2. Although the value of $\alpha = 1.68$ for the axial points is optimal, the number of centre points is not. This should not be particularly surprising since the optimal properties of central composite designs were discovered some six years after this initial application.

Table 25.2 - Additional runs for the yield study.

	Temperature C (C)	Concentration of A (%)	Reaction time (hrs)	Measured yield of (%)
star points	130	38.1	9.1	51.0
	120	38.1	9.1	43.7
	125	40.6	9.1	54.9
	125	35.6	9.1	53.0
	125	38.1	10.8	55.8
	125	38.1	7.4	57.8
center points	125	38.1	9.1	54.9
	125	38.1	9.1	53.2
	125	38.1	9.1	55.3
	125	38.1	9.1	54.1

The model

$$E(Y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \qquad (25.4)$$

was fitted to the data from the 18 runs of this central composite design (i.e. the data in table 24.6 combined with the data in table 25.2) using the same coded values of the operating variables,

a "stationary ridge" data distribution is good because it gives us the ability to switch some of the operating variables and still maintain optimal output.

$$x_1 = \frac{(\text{temperature} - 1525)}{53}$$

$$x_2 = \frac{(\text{concentration of A} - 38.1)}{1.5}.$$

$$x_3 = \frac{(\text{reaction time} - 9.1)}{1.0}$$

The following fitted model was obtained,

$$\begin{aligned} \hat{y} = 56.38 &+ 1.71x_1 + 0.78x_2 + 0.03x_3 \\ &-2.84x_1^2 - 1.13x_2^2 + 0.32x_3^2 \\ &-2.19x_1x_2 - 1.58x_1x_3 - 0.58x_2x_3 \end{aligned} \tag{25.5}$$

and tests for lack of fit showed this model to be an adequate representation of these data.

An estimated stationary point can be determined by equating each of the derivatives of equation (25.5) with respect to $E(Y) = \hat{y}_s + \lambda_1 x_1^2 + \lambda_2 x_2^2 + \cdots + \lambda_k x_k^2$ to zero. This stationary point may be an estimate of an optimum or it may be a saddle point or a point on a ridge. To determine the nature of the response surface behaviour in the neighbourhood of an estimated stationary point, a *canonical transformation* of the fitted response surface expression may be used.

The canonical form of a second degree polynomial in k variables is an expression of that polynomial in terms of k new variables which coincide with the principal axes of the original polynomial representation. More specifically, the general model form (25 1) is expressed in its canonical form

$$E(Y) = \hat{y}_s + \lambda_1 z_1^2 + \lambda_2 z_2^2 + \cdots + \lambda_k z_k^2 \tag{25.6}$$

where $\hat{y}_s$ is the estimated response value at the stationary point and

$z_1, \cdots, z_k$ are variables defining the principal axes of the second degree polynomial response surface, each z being a linear combination of $x_1, \cdots, x_k$.

Thus, $z_1 = \cdots = z_k = 0$ is the location of the stationary point. The λ's are coefficients whose evaluation will be explained shortly.

The behaviour of a second degree polynomial expressed in the form (25.6) can be visualized directly. The size of the increase in $E(Y)$ for a move away from the stationary

1) translate the axis to the ~~stationary~~ point
2) Rotate the original axis so that they coincide with the principal axis.

point along any particular z direction is given by the coefficient λ for that z variable. Decreases in $E(Y)$ are associated with negative λ values. Thus if all λ's are negative, the stationary point is the location of a maximum of the fitted response function. If all λ's are positive, the stationary point is the location of a minimum. If both positive and negative λ's occur in the canonical form, then the stationary point is a saddle point and the values of the λ's describe the nature of that saddle region.

A canonical transformation of a fitted second degree polynomial model consists of two steps,

(i) a *translation* of the origin from the centre of the design $(x_1 = \cdots = x_k = 0)$ to the stationary point $z_1 = \cdots = z_k = 0$,

(ii) an *orthonormal rotation* of the original co-ordinate axes $(x_1, \cdots, x_k)$ to coincide with the principal axes $(z_1, \cdots, z_k)$ of the fitted model.

The mechanics of these two steps are now illustrated using the fitted model (25.5) for the example being discussed.

Model (25.5) can be expressed in matrix notation as

$$\hat{y} = \hat{\beta}_0 + \mathbf{x}^{\mathrm{T}}\mathbf{b} + \mathbf{x}^{\mathrm{T}}\mathbf{C}\mathbf{x}, \tag{25.7}$$

where in this case

$$\mathbf{x}^{\mathrm{T}} = (x_1 \quad x_2 \quad x_3)$$

$$\hat{\beta}_0 = 50.68$$

$$\mathbf{b} = \begin{bmatrix} \hat{\beta}_1 \\ \hat{\beta}_2 \\ \hat{\beta}_3 \end{bmatrix} = \begin{bmatrix} 1.71 \\ 0.78 \\ 0.03 \end{bmatrix} \Bigg\} \text{ Main effect parameters}$$

$$\mathbf{C} = \begin{bmatrix} \hat{\beta}_{11} & \frac{1}{2}\hat{\beta}_{12} & \frac{1}{2}\hat{\beta}_{13} \\ \frac{1}{2}\hat{\beta}_{12} & \hat{\beta}_{22} & \frac{1}{2}\hat{\beta}_{23} \\ \frac{1}{2}\hat{\beta}_{13} & \frac{1}{2}\hat{\beta}_{23} & \hat{\beta}_{33} \end{bmatrix} = \begin{bmatrix} -2.84 & -1.10 & -0.79 \\ -1.10 & -1.13 & -0.29 \\ -0.79 & -0.29 & 0.32 \end{bmatrix}.$$

As stated above, the stationary point

$$\mathbf{x}_s^{\mathrm{T}} = (x_{1s} \quad x_{2s} \quad x_{3s})$$

is that point for which

$$\frac{\partial \hat{y}}{\partial x_i} = 0 \qquad \text{for } i = 1, 2, 3. \tag{25.8}$$

In matrix notation, equation (25.8) can be expressed as

$$\mathbf{b} + 2\mathbf{C}\mathbf{x}_s = \mathbf{0}$$

so that

Know how to take a 2x2 matrix inverse

$$\mathbf{x}_s = -\frac{\mathbf{b}^{\mathrm{T}}\mathbf{C}^{-1}}{2} = \begin{pmatrix} 0.88 & 0.04 & -2.14 \end{pmatrix} \tag{25.9}$$

The co-ordinate value $x_{3s} = -2.14$ is well outside the region of the central composite design, suggesting that the response surface within the design region may have ridge characteristics. A sketch of this type of situation for two operating variables is given in figure 25.2.

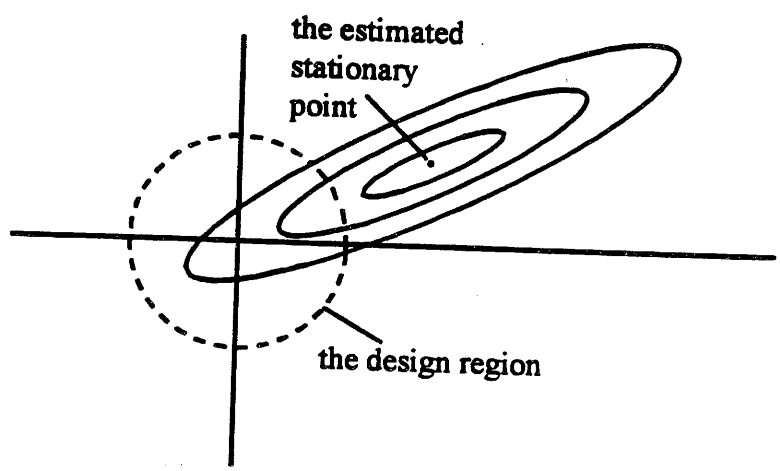


Figure 25.2 - An apparent ridge system within the experimental design region.

The estimated response value $\hat{y}_s$ at the stationary point is obtained by substituting the co-ordinates of the stationary point in the fitted model (25.7),

$$\hat{y}_s = \hat{\beta}_0 + \mathbf{x}_s^{\mathrm{T}}\mathbf{b} + \mathbf{x}_s^{\mathrm{T}}\mathbf{C}\mathbf{x}_s = 57.12. \tag{25.10}$$

Subtracting equation (25.10) from (25.7),

$$\hat{y} = \hat{y}_s + \mathbf{x}^{\mathrm{T}}\mathbf{b} + \mathbf{x}^{\mathrm{T}}\mathbf{C}\mathbf{x} - \mathbf{x}_s^{\mathrm{T}}\mathbf{b} - \mathbf{x}_s^{\mathrm{T}}\mathbf{C}\mathbf{x}_s,$$

and substituting the following expression for $\mathbf{b}$ obtained from equation (25.9),

$$\mathbf{b} = -2\mathbf{C}\mathbf{x}_s,$$

the fitted model can be written as

$$\hat{y} = \hat{y}_s + (\mathbf{x} - \mathbf{x}_s)^{\mathrm{T}}\mathbf{C}(\mathbf{x} - \mathbf{x}_s). \tag{25.11}$$

The linear terms in $\mathbf{x}$ that were present in the fitted model (25.7) have been eliminated in expression (25.11) by a translation of the origin from $\mathbf{x} = \mathbf{0}$ to $\mathbf{x} = \mathbf{x}_s$.

The cross product terms in the x's can now be eliminated by an orthonormal rotation of the co-ordinate axes. This is accomplished by evaluating the eigenvalues and associated eigenvectors of the matrix $\mathbf{C}$. If the eigenvalues of $\mathbf{C}$ are denoted as λ_1, λ_2 and λ_3, and the three associated eigenvectors

$Cu_i = \lambda_i u_i$

$$\begin{aligned} \mathbf{u}_1^{\mathrm{T}} &= (u_{11} \quad u_{12} \quad u_{13}) \\ \mathbf{u}_2^{\mathrm{T}} &= (u_{21} \quad u_{22} \quad u_{23}) \\ \mathbf{u}_3^{\mathrm{T}} &= (u_{31} \quad u_{32} \quad u_{33}) \end{aligned}$$

$\underbrace{|C - \lambda I| = 0}_{\text{determinant}}$

are scaled so that $\sum_{j=1}^{3} u_{ij}^2 = 1$ for $i = 1, 2, 3$, then an orthonormal matrix $\mathbf{U}$ can be formed whose rows are the eigenvectors of $\mathbf{C}$. Because each of the eigenvectors has been defined as having unit length,

$$\mathbf{U}^{\mathrm{T}}\mathbf{U} = \mathbf{U}\mathbf{U}^{\mathrm{T}} = \mathbf{I} \tag{25.12}$$

* You <u>must</u> normalize the eigenvectors

where **I** is the identity matrix.

Using the relationship (25.12), the model (25.11) can be written as

$$\begin{aligned}\hat{y} &= \hat{y}_s + (\mathbf{x}-\mathbf{x}_s)^{\mathrm{T}}\mathbf{U}^{\mathrm{T}}\mathbf{UCU}^{\mathrm{T}}\mathbf{U}(\mathbf{x}-\mathbf{x}_s) \\ &= \hat{y}_s + \mathbf{z}^{\mathrm{T}}\mathbf{UCU}^{\mathrm{T}}\mathbf{z}\end{aligned} \tag{25.13}$$

where

$$\begin{aligned}\mathbf{z}^{\mathrm{T}} &= \begin{pmatrix} z_1 & z_2 & z_3 \end{pmatrix} \\ &= (\mathbf{x}-\mathbf{x}_s)^{\mathrm{T}}\mathbf{U}^{\mathrm{T}}\end{aligned} \tag{25.14}$$

Recalling the definition of an eigenvector,

$$\mathbf{u}_i^{\mathrm{T}}\mathbf{C} = \lambda_i \mathbf{u}_i^{\mathrm{T}}$$

for $i = 1, 2, 3$, the model (25.13) can be expressed as

$$\begin{aligned}\hat{y} &= \hat{y}_s + \mathbf{z}^{\mathrm{T}}\begin{bmatrix} \lambda_1 & 0 & 0 \\ 0 & \lambda_2 & 0 \\ 0 & 0 & \lambda_3 \end{bmatrix}\mathbf{z} \\ &= \hat{y}_s + \lambda_1 z_1^2 + \lambda_2 z_2^2 + \lambda_3 z_3^2\end{aligned} \tag{25.15}$$

sign of the and magnitudes

which is the canonical form defined in equation (25.6).

The three eigenvalues of the matrix **C** are the three solutions of the equation

$$|\mathbf{C} - \lambda\mathbf{I}| = 0$$

For this example the eigenvalues and eigenvectors of **C** are as follows,

Eigenvalue		Eigenvector
$\lambda_1 = -0.10$	$\mathbf{u}_1^{\mathrm{T}} =$	$(\ 0.29 \quad -0.04 \quad -0.96)$
$\lambda_2 = -0.61$	$\mathbf{u}_2^{\mathrm{T}} =$	$(-0.39 \quad 0.91 \quad -0.16)$
$\lambda_3 = -3.59$	$\mathbf{u}_3^{\mathrm{T}} =$	$(\ 0.87 \quad 0.42 \quad 0.25)$

The canonical form of the fitted model (25.5) can then be written as

$$\hat{y} = 57.12 - 0.10z_1^2 - 0.61z_2^2 - 3.59z_3^2 \tag{25.16}$$

where

$$\begin{aligned} z_1 &= 0.29(x_1 - 0.88) - 0.04(x_2 - 0.04) - 0.96(x_3 + 2.14) \\ z_2 &= -0.39(x_1 - 0.88) + 0.91(x_2 - 0.04) - 0.16(x_3 + 2.14). \\ z_3 &= 0.87(x_1 - 0.88) + 0.42(x_2 - 0.04) + 0.25(x_3 + 2.14) \end{aligned}$$

Model (25.16) indicates that the response surface is dome shaped in the stationary region and that a unique maximum response occurs at the stationary point. However the response surface is very flat along the z_1 and z_2 axes relative to its behaviour along the z_3 axis. If the terms in z_1 and z_2 are deleted from expression (25.16), since they have relatively minor effects on the estimated response, then an approximate fitted model is

$$\hat{y} = 57.12 - 3.59z_3^2,$$

which can be rewritten as

$$z_3 = \pm\sqrt{\frac{\hat{y} - 57.12}{-3.59}}. \tag{25.17}$$

For any specified value $\hat{y} = \hat{y}_*$ less than 57.12, the expression (25.17) represents a pair of parallel planes in the z co-ordinate system, both of which are perpendicular to the z_3 axis. The estimated yield of product C at all points on each of these planes is $\hat{y}_*$. The estimated yield of product C attains its maximum value of 57.12% at $z_3 = 0$ and decreases symmetrically in both directions along the z_3 axis away from $z_3 = 0$. Thus, as shown in figure 25.3, the fitted response surface in this stationary region behaves very nearly like a stationary ridge system of two-dimensional planes .

Because the plane $z_3 = 0$ lies outside the region of experimentation, some confirmatory tests must be carried out in the neighbourhood of the point $z_1 = z_2 = z_3 = 0$ to ensure that the model (25.17) does in fact describe the process behaviour in that region. Tests of this sort were carried out and they confirmed the adequacy of the approximate model (25.17).

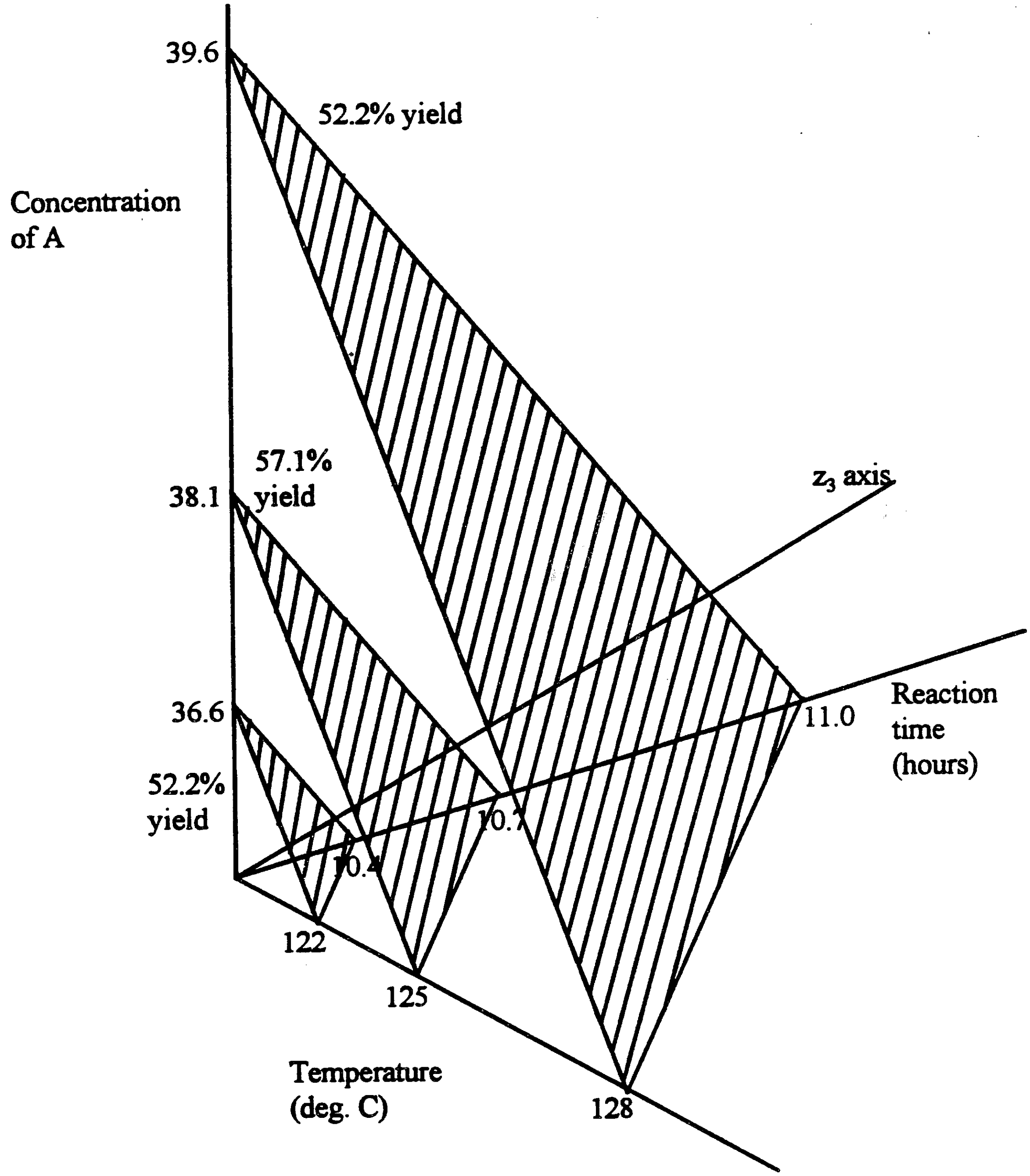


Figure 25.3 - Approximate response surface behaviour in the stationary region

* This stuff relates to suggesting new levels of operating variables ~~to~~ while ~~min~~ minimizing the change in optimal set point (Know this stuff for the exam)

Using the approximate fitted response function (25.17), the yield of product C is estimated to be a maximum anywhere on the plane $z_3 = 0$. Using the relationship between z_3 and the x's, this plane has the equation

$$0.87x_1 + 0.42x_2 + 0.25x_3 = 0.25,$$

or in terms of the three original operating variables, the plane is defined as

$$0.29(\text{temperature}) + 0.28(\text{conc. of A}) + 0.25(\text{reaction time}) = 49.45. \tag{25.18}$$

The equation (25.18) identifies sets of alternative operating conditions that are expected to produce a yield of C close to 57%. A plane of optimum conditions has obvious advantages over a single optimum point. If, for example, the cost of component A increases, it may be preferable to decrease the concentration of A while increasing the operating temperature and/or the reaction time to maintain the yield of product C at its maximum value.

This study has resulted in

(i) a substantial increase in yield of product C,
(ii) location of approximate optimum operating conditions and
(iii) identification of the nature of the response surface in the region of the optimum.

If accurate mechanistic information about the relationship between the yield of C and the three operating variables had been available, these results might have been obtained more directly. In the absence of complete mechanistic information, a sequential empirical strategy of steepest ascent followed by exploration of the stationary region can produce the above results in an efficient manner. Although the fitted models at each stage are only approximate descriptions of the true response function, they can sometimes provide clues about underlying physical mechanisms, leading to increased understanding of the process under study.

The primary objective of this study was to maximize the yield of product C. In addition, a constraint was imposed; the yield of byproduct D was to be less than 20%. Satisfying this constraint required determining the relationship between the yield of D and the three operating variables and this was accomplished as follows.

At each of the experimental runs described above, two measured response values were recorded, the yield of product C and the yield of byproduct D. Using the results from the central composite design, a fitted response surface for the yield of D was obtained. The intersection of this surface with the approximately optimum plane for the yield of C is shown in figure 25.4. The operating region for which the constraint on D is satisfied is identified in that figure.

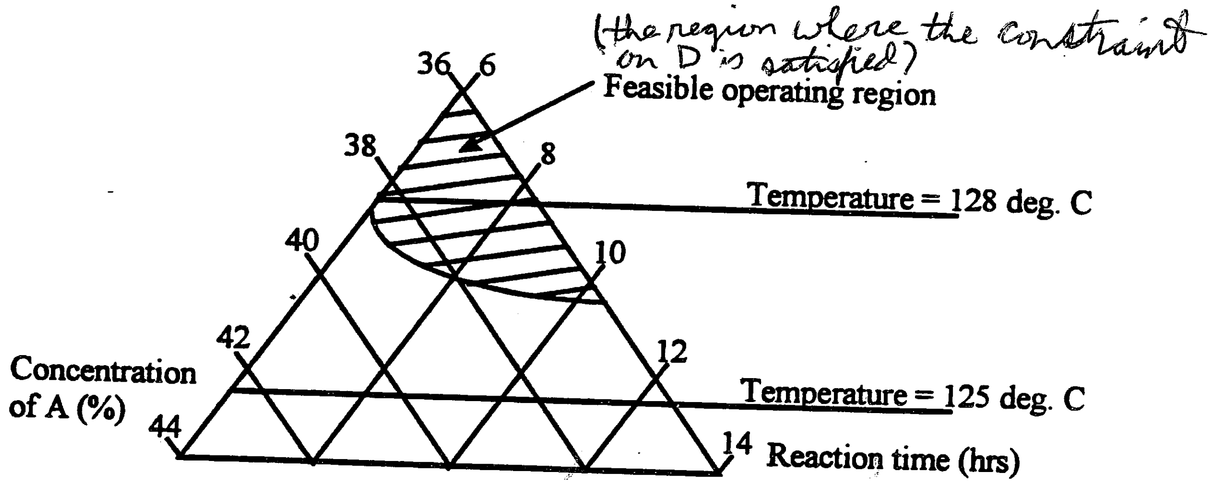


Figure 25.4 - Feasible operating region.

Overlaying response surface representations in the manner shown above can be a useful way of identifying operating conditions that satisfy conditions imposed on more than one response variable, as long as the number of operating variables is not greater than three. This technique has proved to be particularly effective in the development of new products. Tidwell [5] has described an interesting example of the application of this methodology to a chemical process involving three operating variables and two response variables. Of course if constraints are too stringent, there may be no acceptable set of operating conditions.

A survey of applications of response surface methods has been prepared by Hunter and Hill [6].

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APPENDIX G

Blocking in Factorial Designs

BLOCKING

In the previous examples where foldover designs were combined with initial designs, an estimate of a *block effect* was mentioned. This section will explore the importance and value of blocking in an experimental design strategy.

Definition

A block can be defined as a portion of the experiment that is expected to be more homogeneous than the whole. That is, are there blocks of experimental runs that have been or can be executed under the same set of conditions and do these conditions change such that they have had or may have an observable effect on the experimental response(s) between blocks?

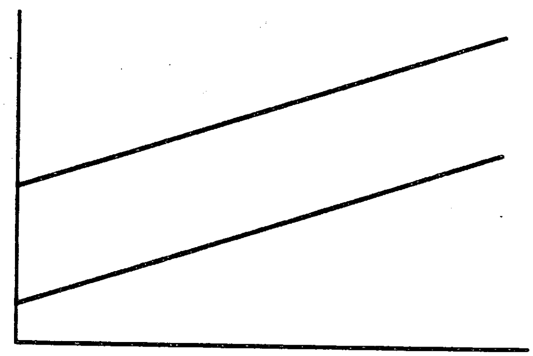
Ideally, we would like to execute our experiment in an environment where nothing changes except the experimental factors that we are actively varying through the course of the experiment. In practice, this is seldom achieved. Uncontrollable noise factors (temperature, humidity, etc.) can be recorded and included in the design matrix as a covariate to assess their impact on the results. Randomizing the run order will serve to ~~smooth out~~ randomly distribute any systematic effects these noise factors may have, thereby minimizing the likelihood that such systematic effects will be confounded with the effects of primary interest. Factors that can be fixed at a constant value during the course of the experiment should be fixed. However, there are several instances where fixed factors may vary over the course of the experiment despite our best effort to fix them. Examples are

- the amount of raw material required necessitates that it be drawn from more than one lot or supplier,
- the amount of time required to run the experiment necessitates that it be executed over a number of shifts or days,
- different operators, equipment set-ups or measuring tools may be used during the experiment.

The necessity for estimating the effect a block may have on experimental results usually arises in two situations. The first is where a set of experimental runs is combined with one or more previous sets of experimental runs in a sequential design strategy. An example of this is adding a foldover design to an initial experimental design. The fact that the subsequent experiment was executed at a later date and time (perhaps with different personnel involved) necessitates that the effect of any differences between the two experiments (blocks of runs) be estimated. The second situation arises where it is known beforehand that, due to limited resources, the experiment can only be executed homogeneously in blocks.

Blocking Between Completed Experiments

In the two examples on foldovers, we saw that $(l_{01} - l_{02})/2$ was an estimate of the block effect between the two experiments. All things being equal, any differences between the two experiments should only result in a constant shift up or down in the average response. That is, the true effects that the experimental factors have on the response should remain the same. This is like shifting a straight line upwards or downwards, the intercept (average) changes, but the slope (effect) does not.



In essence, we are assuming that the block effect, if any, does not interact in any way with the experimental factors. If we denote the block effect by B, then we assume that all $B\,x_i = 0$.

What about the confounding pattern? We do not want our estimate of the block effect between experiments to be confounded with any potentially important experimental effect. That is, we do not want $\hat{B}$ to be an estimate of (B + x_i) or (B + $x_i x_j$). The block effect and it's relationship to the confounding pattern is illustrated through the two examples on foldovers.

Example 1 - Reversing the Sign of x_1

To distinguish the two experiments as two blocks, the experimental runs from the initial experiment (runs 1 through 8) can be denoted by the roman numeral I and those from the foldover (runs 9 through 16) by II as follows,

x_1	x_2	x_3	x_4	x_5	x_6	x_7		
−1	−1	−1	−1	1	1	1	(1)	I
1	−1	−1	−1	1	1	1	(9)	II
−1	1	−1	−1	1	−1	−1	(12)	II
1	1	−1	−1	1	−1	−1	(4)	I
−1	−1	1	−1	−1	1	−1	(14)	II
1	−1	1	−1	−1	1	−1	(6)	I
−1	1	1	−1	−1	−1	1	(7)	I
1	1	1	−1	−1	−1	1	(15)	II
−1	−1	−1	1	−1	−1	1	(10)	II
1	−1	−1	1	−1	−1	1	(2)	I
−1	1	−1	1	−1	1	−1	(3)	I
1	1	−1	1	−1	1	−1	(11)	II
−1	−1	1	1	1	−1	−1	(5)	I
1	−1	1	1	1	−1	−1	(13)	II
−1	1	1	1	1	1	1	(16)	II
1	1	1	1	1	1	1	(8)	I

It can be verified that the pattern of alternating I's and II's in the block column is confounded with the interactions $x_1x_2x_3x_4$, $x_1x_2x_5$, $x_1x_3x_6$, $x_1x_4x_7$, $x_1x_3x_5x_7$, $x_1x_2x_6x_7$, $x_1x_4x_5x_6$ and $x_1x_2x_3x_4x_5x_6x_7$. These happen to be the interactions in the defining relations for the initial and foldover design that did *not* match. We see that the block factor is only aliased with third and higher order interactions. Hence, our estimate of the block effect is not confounded with any main effects or second-order interactions.

Example 2 - Reversing the Signs of all x_i

Following the same coding as in the previous example, we can identify the two blocks in the combined design as follows,

x_1	x_2	x_3	x_4	x_5	x_6	x_7		
−1	−1	−1	−1	−1	−1	−1	*(16)	II
1	−1	−1	1	−1	−1	1	(2)	I
−1	1	−1	1	−1	1	−1	(3)	I
1	1	−1	−1	−1	1	1	*(13)	II
−1	−1	1	1	−1	1	1	*(12)	II
1	−1	1	−1	−1	1	−1	(6)	I
−1	1	1	−1	−1	−1	1	(7)	I
1	1	1	1	−1	−1	−1	*(9)	II
−1	−1	−1	−1	1	1	1	(1)	I
1	−1	−1	1	1	1	−1	*(15)	II
−1	1	−1	1	1	−1	1	*(14)	II
1	1	−1	−1	1	−1	−1	(4)	I
−1	−1	1	1	1	−1	−1	(5)	I
1	−1	1	−1	1	−1	1	*(11)	II
−1	1	1	−1	1	1	−1	*(10)	II
1	1	1	1	1	1	1	(8)	I

Again, it can be verified that the pattern of the blocking column is confounded with the terms in the defining relations that did not match, namely, $x_1x_2x_5$, $x_1x_3x_6$, $x_2x_3x_7$, $x_3x_4x_5$, $x_2x_4x_6$, $x_1x_4x_7$ and $x_5x_6x_7$. Once again, our estimate of the block effect is not confounded with any main effects or second-order interactions.

Summary

It can be seen from the two previous examples that the block effect is confounded with those effects corresponding to the *non-matching* components from the defining relations of the combined experiments. In both examples, the block effect was confounded only with third and higher order interactions, which are assumed to be negligible.

Blocking Within an Experiment

If it has been determined beforehand that an experiment cannot be run under the same conditions due to resource limitations or time constraints, then it is advisable to run the experiment in blocks and allow for the estimation of the block effects in the analysis of the results. Failure to do so can result in less precise estimates of the main effects and interactions because the variance introduced by the block effect is left unaccounted for. The order of the runs within each block should be randomized to minimize unknown systematic effects from corrupting the experiment. The order in which the blocks of runs themselves are executed should also be randomized.

Example 1

Suppose that the eight runs in a 2^3 full factorial design cannot be carried out under homogeneous conditions. In this investigation a mixture of adhesive glue is required to assemble components for the experiment and must be prepared beforehand. The glue has a curing time that will only allow four runs to be assembled at a time. Hence, between blocks of four runs, there is the potential that something different in the preparation of the glue will affect the results. The effect of the glue can be blocked by confounding it with the third-order interaction between the three experimental factors as follows.

run	x_1	x_2	x_3	x_1x_2	x_1x_3	x_2x_3	$x_1x_2x_3$	block
1	−1	−1	−1	1	1	1	−1	I
2	1	−1	−1	−1	−1	1	1	II
3	−1	1	−1	−1	1	−1	1	II
4	1	1	−1	1	−1	−1	−1	I
5	−1	−1	1	1	−1	−1	1	II
6	1	−1	1	−1	1	−1	−1	I
7	−1	1	1	−1	−1	1	−1	I
8	1	1	1	1	1	1	1	II

Thus, runs 1, 4, 6 and 7 constitute the runs to be carried out in one block with one glue mixture and runs 2, 3, 5 and 8 constitute the runs to be carried out in the second block with the other glue mixture. Remember that the intent is to keep the glue as uniform or homogeneous as possible between the blocks, but should they be different enough so as to have an impact on the results, then we will at least be able to estimate it and determine its significance. We will also have more precise estimates of the main effects and second-order interactions than would otherwise be the case. In essence, the blocking factor is like a fourth experimental factor that has been generated from the relationship $B = x_1x_2x_3$ but which does not interact with the other experimental factors. If it was suspected that the glue did interact with the other experimental factors, then it should be included as an experimental factor and not a blocking factor.

Example 2

Suppose that four factors are being investigated in a 2^{4-1}_{III} fractional factorial design. As in the previous example, the glue constrains us to run the experiment in blocks of four runs. The following design strategy is put forward for this study.

run	x_1	x_2	x_3	$x_4 = x_1x_2x_3$	$B = x_1x_2$
1	−1	−1	−1	−1	II
2	1	−1	−1	1	I
3	−1	1	−1	1	I
4	1	1	−1	−1	II
5	−1	−1	1	1	II
6	1	−1	1	−1	I
7	−1	1	1	−1	I
8	1	1	1	1	II

So, runs 2, 3, 6 and 7 form one block and runs 1, 4, 5 and 8 form the other block. In this example, the fourth factor column was generated from $x_1x_2x_3$ as is usually the case in a half-fraction of a 2^3 design. We are then forced to generate the block effect by confounding it with one of the second-order interactions. Thus, depending upon the resolution of the fractional factorial design, blocking factors may be confounded with second-order interactions.

The confounding pattern (up to second-order interactions) for the above design based on the defining relation $I = x_1x_2x_3x_4 = Bx_1x_2 = Bx_3x_4$ is

$x_1 = Bx_2$	$B = x_1x_2 = x_3x_4$
$x_2 = Bx_1$	$x_1x_3 = x_2x_4$
$x_3 = Bx_4$	$x_1x_4 = x_2x_3$
$x_4 = Bx_3$	$x_0 = x_1x_2x_3x_4 = Bx_1x_2 = Bx_3x_4$

The significance of assuming that the blocking factor does not interact with any of the experimental factors is evident from the above confounding pattern where each of the main effects is confounded with the interaction between the blocking and an experimental factor.

Again, the run order within each of these blocks should be randomized.

Example 3

This example highlights the importance of selecting the appropriate confounding relationships for block factors. Suppose that instead of being able to assemble four runs with a single mixture of glue, we are limited to two runs. Thus, we require four blocks, each of size two runs. What type of blocking arrangement can be made with a 2^3 full factorial design?

A Poor Design

One possibility is to confound one blocking factor with $x_1x_2x_3$, as was done in example 1, and a second blocking factor with one of the second-order interactions, say x_1x_2. This yields the following design,

run	x_1	x_2	x_3	$B_1 = x_1x_2x_3$	$B_2 = x_1x_2$	block
1	−1	−1	−1	−1	1	I
2	1	−1	−1	1	−1	II
3	−1	1	−1	1	−1	II
4	1	1	−1	−1	1	I
5	−1	−1	1	1	1	III
6	1	−1	1	−1	−1	IV
7	−1	1	1	−1	−1	IV
8	1	1	1	1	1	III

where

block I corresponds to	$B_1 = -1, B_2 = +1$	(runs 1,4),
block II corresponds to	$B_1 = +1, B_2 = -1$	(runs 2,3),
block III corresponds to	$B_1 = +1, B_2 = +1$	(runs 5,8),
block IV corresponds to	$B_1 = -1, B_2 = -1$	(runs 6,7).

Note that we have only specified two blocking factors, yet there are four blocks of runs which requires three degrees of freedom. The third degree of freedom is associated with the blocking factor obtained from the multiplication $B_1B_2 = x_1x_2x_3 \cdot x_1x_2 = x_3$. Thus, this design confounds the main effect due to x_3 with block differences.

A Better Design

A better confounding arrangement is obtained by confounding the two block factors with any two of the second-order interactions. The third degree of freedom is then confounded with the third second-order interaction. If we choose $B_1 = x_1x_2$ and $B_2 = x_1x_3$, then we have $B_1B_2 = x_1x_2 \cdot x_1x_3 = x_2x_3$. Now, block effects are only confounded with second-order interactions. This yields the following design,

run	x_1	x_2	x_3	$B_1 = x_1x_2$	$B_2 = x_1x_3$	block
1	−1	−1	−1	1	1	I
2	1	−1	−1	−1	−1	II
3	−1	1	−1	−1	1	III
4	1	1	−1	1	−1	IV
5	−1	−1	1	1	−1	IV
6	1	−1	1	−1	1	III
7	−1	1	1	−1	−1	II
8	1	1	1	1	1	I

where

block I corresponds to	$B_1 = +1, B_2 = +1$	(runs 1,8),
block II corresponds to	$B_1 = -1, B_2 = -1$	(runs 2,7),
block III corresponds to	$B_1 = -1, B_2 = +1$	(runs 3,6),
block IV corresponds to	$B_1 = +1, B_2 = -1$	(runs 4,5).

Summary

Again, depending on the resolution of the design, blocking factors may be confounded with second-order interactions. Depending upon the objective of the investigation, this may or may not be acceptable. The value of understanding the pattern of confounding, whether or not blocking is present, is that a determination can be made beforehand as to whether the experimental objectives will be satisfied and if they will not, then further resources will have to be allocated.

Impact of Failing to Block

This example will serve to demonstrate the impact of *not* including a blocking factor in the analysis of a 2^3 experimental design. In the design below, runs 1, 4, 6, 7, 9 and 10 were executed on one day and runs 2, 3, 5, 8, 11 and 12 were executed on the following day. Thus, "day" is a blocking factor that has been confounded with the $x_1x_2x_3$ interaction. The runs were randomized within each block.

run	x_1	x_2	x_3	block
1	−1	−1	−1	I
2	1	−1	−1	II
3	−1	1	−1	II
4	1	1	−1	I
5	−1	−1	1	II
6	1	−1	1	I
7	−1	1	1	I
8	1	1	1	II
9	0	0	0	I
10	0	0	0	I
11	0	0	0	II
12	0	0	0	II

$\Rightarrow$

block	run	x_1	x_2	x_3
I	1	−1	−1	−1
I	4	1	1	−1
I	6	1	−1	1
I	7	−1	1	1
I	9	0	0	0
I	10	0	0	0
II	2	1	−1	−1
II	3	−1	1	−1
II	5	−1	−1	1
II	8	1	1	1
II	11	0	0	0
II	12	0	0	0

The experimental results are,

run	x_1	x_2	x_3	y
1	−1	−1	−1	55
4	1	1	−1	67
6	1	−1	1	40
7	−1	1	1	36
9	0	0	0	51
10	0	0	0	49
2	1	−1	−1	72
3	−1	1	−1	63
5	−1	−1	1	47
8	1	1	1	53
11	0	0	0	58
12	0	0	0	62

The model to be fit is

$$E(Y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 .$$

A Poor Analysis

The solution to $\hat{\beta} = (\mathbf{X}^T\mathbf{X})^{-1}\mathbf{X}^T\mathbf{Y}$ yields the following parameter estimates,

$$\hat{\beta}_0 = 54.4$$
$$\hat{\beta}_1 = 3.9$$
$$\hat{\beta}_2 = 0.6$$
$$\hat{\beta}_3 = -10.1$$

An internal estimate of the pure error variance determined from the four centre-point replicates is $\hat{\sigma}^2 = 54.25$. Thus, the covariance matrix for the parameter estimates can be used to find that

$$s_{\hat{\beta}_0} = \sqrt{\frac{36.7}{12}} = 1.75$$
$$s_{\hat{\beta}_1} = s_{\hat{\beta}_2} = s_{\hat{\beta}_3} = \sqrt{\frac{36.7}{8}} = 2.14$$

95% confidence intervals can then be constructed using $\hat{\beta}_i \pm t_{3,0.025} s_{\hat{\beta}_i}$ where $t_{3,0.025} = 3.182$,

$$\beta_0 \in [48.8, 60.0]$$
$$\beta_1 \in [-2.9, 10.7]$$
$$\beta_2 \in [-6.2, 7.4]$$
$$\beta_3 \in [-16.9, -3.3]$$

Based on our interpretation of the confidence intervals, we conclude that the reduced model form $\hat{y} = 54.4 - 10.1x_3$ adequately fits the response data. It should be noted that no significant lack of fit was detected. The R^2 for this fitted model was 0.751 which means that this model explains 75.1% of the variation in the experimental data.

A Better Analysis

Suspecting that there may be a significant effect between experimental blocks, the model to be fit is modified to include a term for a blocking factor,

$$E(Y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \mathrm{B}.$$

The addition of the block term can be accommodated in the analysis matrix as follows,

x_0	x_1	x_2	x_3	B
1	−1	−1	−1	1
1	1	1	−1	1
1	1	−1	1	1
1	−1	1	1	1
1	0	0	0	1
1	0	0	0	1
1	1	−1	−1	2
1	−1	1	−1	2
1	−1	−1	1	2
1	1	1	1	2
1	0	0	0	2
1	0	0	0	2

The solution to $\hat{\boldsymbol{\beta}} = \left(\mathbf{X}^T\mathbf{X}\right)^{-1}\mathbf{X}^T\mathbf{Y}$ yields the following parameter estimates,

$$\begin{aligned}
\hat{\beta}_0 &= 54.4 \\
\hat{\beta}_1 &= 3.9 \\
\hat{\beta}_2 &= 0.6 \\
\hat{\beta}_3 &= -10.1 \\
\hat{\mathrm{B}}(1) &= -4.8 \\
\hat{\mathrm{B}}(2) &= +4.8
\end{aligned}$$

The estimate of the block effect is interpreted as follows; when B=1, subtract 4.8 from the predicted response and when B=2, add 4.8 to the predicted response,

$$\begin{aligned}
\hat{y} = 54.7 + 3.9x_1 + 0.6x_2 - 10.1x_3 &- 4.8\left(\mathrm{B}=1\right) \\
&+ 4.8\left(\mathrm{B}=2\right)
\end{aligned}$$

An internal estimate of the variance is obtained by pooling the two estimates from each block,

$$s_p^2 = \frac{2.0 + 8.0}{1 + 1} = 5.0\,.$$

Note that this estimate is considerably smaller than the last estimate of 36.7. This is because the overall block effect that shifts the mean response by a total of 9.6 units is not included in the variance calculations. Consequently, the variance of the parameter estimates is much smaller which will produce tighter 95% confidence intervals as follows,

$$s_{\hat{\beta}_0} = s_{\hat{\mathrm{B}}} = \sqrt{\frac{5.0}{12}} = 0.65$$

$$s_{\hat{\beta}_1} = s_{\hat{\beta}_2} = s_{\hat{\beta}_3} = \sqrt{\frac{5.0}{8}} = 0.79$$

95% confidence intervals can then be constructed using $\hat{\beta}_i \pm t_{2,0.025} s_{\hat{\beta}_i}$ where $t_{2,0.025} = 4.303$,

$$\begin{aligned} &\beta_0 \in [51.6, 57.2] \\ &\beta_1 \in [0.5, 7.3] \\ &\beta_2 \in [-2.8, 4.0] \\ &\beta_3 \in [-13.5, -6.7] \\ &|\mathrm{B}| \in [2, 7.6] \end{aligned}$$

We now conclude that only the x_2 term is not significant and end up with the following reduced model form,

$$\begin{aligned} \hat{y} = 54.7 + 3.9x_1 - 10.1x_3 &- 4.8(\mathrm{B}=1) \\ &+ 4.8(\mathrm{B}=2)\,. \end{aligned}$$

The R^2 for this model was 0.996 which means that this model explains 99.6% of the variation in the experimental data. This is significantly better than the 75.1% that the previous fitted model explained.

Note

Note that the inclusion of the blocking term in the model to be fit did not alter the estimates of the other model terms. This goes back to the statement that a constant block effect only shifts the mean response and does not alter the effect that an experimental factor has. Also, because the blocking factors are included as orthogonal components of the other terms, the mathematics dictates that their inclusion will not alter any other parameter estimates.

Blocking and Centre-Points and Replicates

As the previous example illustrates, centre-points should be distributed evenly in each block of the design. If replicates of the experimental runs are also present, then they should be randomized along with the other runs within the block in which the replicated runs appear. If the addition of replicated run exceeds the maximum block size (number of runs) that can be accommodated in a block, then they should be added to the design as extra blocks.

References

[1] Box, G.E.P. and Hunter, J.S. (1961), Technometrics, 3, 311-351.