Experimental design and optimisation (4): Plackett–Burman designs

Analytical Methods Committee, AMCTB No 55

Good experimental design is important in many studies of analytical and other chemical processes. Complete factorial designs, which study all the factors (experimental variables) a ecting the system response, using at least two levels (values) for each factor, can give rise to an unacceptably large number of trial experiments. This is because even apparently simple processes ma15.4(es)TvoF-337.11537.1537h156(i)13.9m(es).je(y)-2231 acsabes3

times at the higher level and four times at the lower. The e ect of each factor is then readily determined from the expression:

$$2[\sum(y+) - \sum(y-)]/N$$

where N is the total number of experiments, eight in this case. The (+) terms are the responses when a given factor is at its high level, and the (−) terms re ect the responses for that factor set to its low level. It can be shown that the e ects for the main factors determined in this way are not confounded with each other (see AMCTB 36).

An example

In this example the experimental output is the uorescence intensity (arbitrary units) of a single sample material, measured using four di erent spectrometer excitation and emission spectral bandwidths and wavelengths, factors A–D. We need an 8-experiment PB design, so there are three dummy factors, labelled d1, d2, and d3, included alternately in Table 1.

From these results we can see that, for example, the e ect of factor A is 0.25(10 + 9 + 10 + 8 − 9 − 7 − 7 − 7) = +1.75. Similarly it can be shown that the e ects of B, C, and D are +0.25, −1.25 and +0.75 respectively. Clearly a negative e ect, as obtained here with factor C, means that moving that factor from a high to low value *c ea e* the system response (uorescence intensity in this case) rather than decreasing it. The e ects of the dummy factors d1, d2 and d3, are found by the same method to be +0.75, +0.25, and +0.25 respectively.

How signi cant is each factor?

Simple ANOVA-related calculations will enable us to assess the signi cance of the "real" factors. For each factor the sum of squares (SS) in a two-level design is given by:

$$SS = N \times (\quad a \quad)^2/4$$

The sums of squares for A, B, C, and D are thus 6.125, 0.125, 3.125, and 1.125 respectively. Each of these sums of squares has just one degree of freedom, so their mean square values (*.e.*, variances) are the same as the SS ones. The sums of squares for the dummy factors d1, d2, and d3 are similarly found to be 1.125, 0.125, and 0.125 respectively. The mean sum of squares for these estimates of the random measurement errors is thus 0.458: this has three degrees of freedom as there are three dummy variables. Each of the individual factors A–D can now be compared with this estimated random error using a one-tailed F-test at the $= 0.05$ signi cance level. So for factor A the value of F is 6.125/0.458 = 13.37. The critical value of $F_{1,3}$ at $= 0.05$ is 10.13, so we can conclude that the e ect of changing the level of factor A is signi cant. The same approach shows that factors B, C and D seem to have no signi cant e ect. Such calculations are in practice performed using suitable so tware such as Minitab , so once the trial experiments are complete the conclusions can be drawn at once.

Plackett–Burman in action

PB designs have been used in an enormous variety of chemical and biochemical studies, synthetic as well as analytical. Spectroscopy, electrochemistry and chromatography have all proved to be fertile elds for their application in measurement science. In practice, designs with 12 and 20 runs seem to have been most popular. This may be because PB designs where 4 is a power of 2 [*.e.* = 2, 4, 8 *e c.*] are exactly equivalent to some other fractional factorial designs, so PB methods confer no advantage. Moreover performing (for example) 12 experiments rather than 8 will provide extra dummy factors, hence better estimates of the measurement error and of the possible signi cance of the real factors. The potential for further applications of PB designs is clear, especially in the development of new or improved analytical methods. Evolutionary methods such as simplex optimisation can be used to nd the best combination of factor levels, but when an optimum set of

Table 1

	FACTORS							
EXPERIMENT	A	d1	B	d2	C	d3	D	Result
1	+	−	−	+	−	+	+	10
2	+	+	−	−	+	−	+	9
3	+	+	+	−	−	+	−	10
4	−	+	+	+	−	−	+	9
5	+	−	+	+	+	−	−	8
6	−	+	−	+	+	+	−	7
7	−	−	+	−	+	+	+	7
8	−	−	−	−	−	−	−	7
Eff	+1.75	+0.75	+0.25	+0.25	−1.25	+0.25	+0.75	*
	6.125	1.125	0.125	0.125	3.125	0.125	1.125	*
F-	13.4	*	0.3	*	6.8	*	2.5	*

conditions has been found in this way we still need to know whether the analytical results are unduly sensitive to small changes in any of the factors.

However, the popularity of PB methods comes with a significant health warning. PB designs are ideal for screening purposes in systems where it is desired to identify a few main factors affecting the outcome, *assuming effects are specified*. Theory shows that while the main factors in a PB design are not confounded, there is strong confounding between the main factors and any two-factor interactions that may arise. So if there are significant interactions, PB methods could provide misleading results. In recent years much attention has been given to diagnostic approaches for revealing interactions in PB designs. These are beyond the scope of this paper; but it is worth noting that if dummy factors seem to have unexpectedly high effect values, this might be a sign that interactions are indeed present.

This Technical Brief, drafted by J.N. Miller, was prepared for the Analytical Methods Committee by the Statistical Subcommittee.