STATISTICAL DESIGN OF EXPERIMENTS FOR QUALITY IMPROVEMENT OF FERTILIZER PRODUCTS

Mark J. Anderson (Consulting Engineer, Stat-Ease Corp., 612-378-9449, mark@statease.com)
P. K. Bhattacharjee (Chief Engineer, U. S. Agrichemical Corp.),
Patrick J. Whitcomb (Consulting Engineer, Stat-Ease Corp.))

ABSTRACT

Statistical tools, especially design of experiments (DOE), provide the means for quality improvement of DiAmmonium Phosphate (DAP) and related fertilizer products. Depletion of high-grade phosphate ores in Florida and elsewhere makes it increasingly difficult to meet customer specifications for nitrogen content of DAP. Urea or ammonia can be used as nitrogen supplements, but this adds cost to the final product. This paper lays out a special form of DOE, called two-level factorial design, which can be used to maximize nitrogen content in DAP, and make it less susceptible to impurities in lower-grade phosphates.

PROBLEMS OF THE PROCESS ENGINEER

Here's a list of typical problems we face as process engineers, particularly those of us who work in a mature chemical line:

- Raw material costs are increasing and/or quality is decreasing
- Competitors are cutting product prices
- Profit margins are declining

To solve these problems, we need to get together and identify causes for poor yields quality. An exhaustive list might include dozens of potential variables - many more than we could possibly investigate. Some of these variables can't be controlled: we can at least record their values. Other variables won't be given much priority: we should try to hold these at fixed levels. Still, we're likely to be left with many possible control factors, more than we can deal with one at a time. These can most efficiently be investigated via design of experiments (DOE).

The basics of DOE are well-documented (1). To illustrate the application of DOE to chemical engineering, we will look at phosphate fertilizer production. The case study shows how we can make breakthrough improvements with a simple DOE called two-level factorial design. By restricting the tests to only two levels, we minimize the number of experiments, yet the contrast between levels gives us the necessary driving force for process improvement. Fortunately, software makes it easy to set up and analyze two-level DOEs (2,3). However, like any powerful tool, the more we know about DOE, the better we'll do, so it pays to learn what we can about the statistical aspects.

CASE STUDY

Phosphate fertilizer is produced by combining phosphoric acid with ammonia in a slurry reactor. The end product is DiAmmonium phosphate (DAP) in granular form. Customers desire nitrogen contents of 18 per cent or better, but this can be difficult to achieve in the presence of raw material impurities, especially calcium, iron, aluminum and magnesium. The latter three contaminants are monitored via the metallic elements ratio (MER), which is the ratio of oxides (Fe_2O_3 plus Al_2O_3 plus MgO) to phosphate (P_2O_5). If the reaction does not produce a sufficient nitrogen level, then producers must add relatively expensive supplements such as urea (ammonium nitrate).

Table 1 (below) lists four possible factors each at 2 levels for a hypothetical 16-run DOE on the DAP fertilizer process. The table shows the full factorial test matrix and simulated response for nitrogen content. The design is listed in standard order, however it should be run in random order to protect against uncontrolled lurking variables, such as ambient temperature, which could bias the effects.

Standard Order	A Temp	B Acid to Ammonia	C Agitation	D Impurities (MER)	Nitrogen (Percent)
1	–	–	–	–	17.9
2	+	–	–	–	17.8
3	–	+	–	–	17.6
4	+	+	–	–	17.4
5	–	–	+	–	18.3
6	+	–	+	–	18.2
7	–	+	+	–	17.7
8	+	+	+	–	18.0
9	–	–	–	+	18.4
10	+	–	–	+	18.2
11	–	+	–	+	17.8
12	+	+	–	+	17.9
13	–	–	+	+	18.3
14	+	–	+	+	18.4
15	–	+	+	+	17.8
16	+	+	+	+	17.8

Table 1. Factorial Design Matrix with Response Data (Simulated)

Note the balanced array of plus (high) and minus (low) levels: Each column contains eight pluses and eight minuses. This matrix exhibits a very important statistical property called "orthogonality", which means that factors are not correlated. If you just collected happenstance data from the process, you would never get an array of factors like this. Variables such as temperature and pressure would go up and down together, making it very difficult to tell which one actually affected the quality or yield. Therefore, whenever possible, you should take control of your experiment by doing a DOE, not just collecting data.

USING STATISTICAL PRINCIPLES TO PICK SIGNIFICANT FACTORS

Orthogonal test matrices make it easy to estimate main effects – just subtract the average of responses at the minus levels from the average at the plus level. For example, the D effect comes from the contrast of the first 8 responses to the last 8 responses. Interactions of factors, which often prove to be the key to understanding your process, require a bit more work to estimate, but computer software does this for you very quickly and accurately. In a typical process only about 20 per cent of the main effects and two-factor interactions will be active. You can quickly identify these vital few effects with a computer-generated graph called a "half-normal plot". Figure 1 (below) shows the half-normal plot of effects for the phosphate fertilizer case.

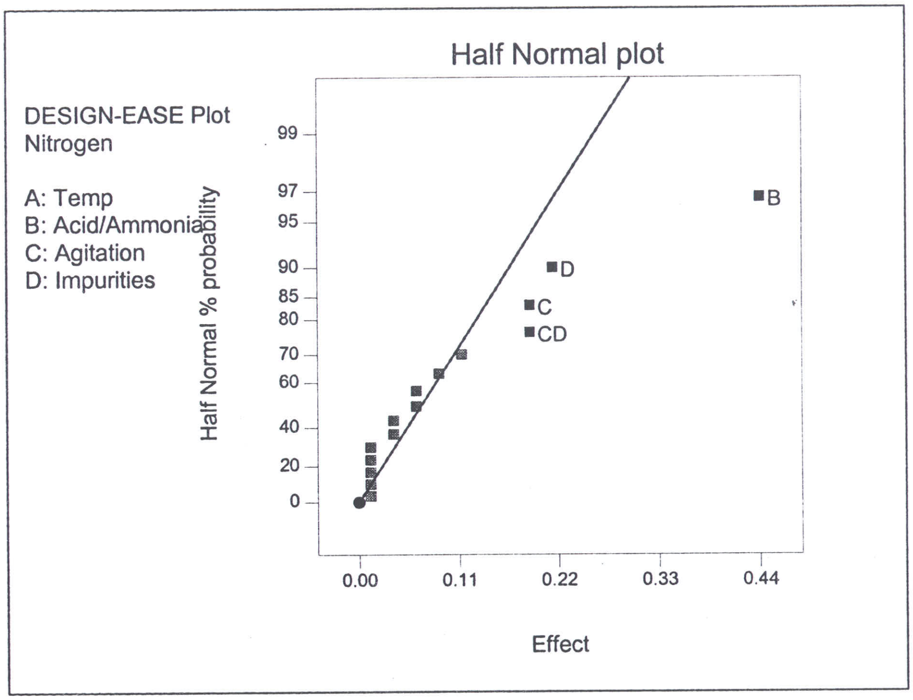

Figure 1. Half-Normal Plot of Effects

With 16 unique experiments, we can estimate 15 effects plus the overall average response. The half-normal plot identifies only the vital few effects which significantly alter the nitrogen response. Factor B is by far the largest effect. Then there's a "family" grouping of intermediate effects: C, D and CD. Nearest the zero level, we see a group of 11 unlabeled effects. These effects have an insignificant impact on the response. They include higher order interactions that rarely occur, namely ABC, ABD, ACD, BCD, ABCD, plus the trivial many inactive main effects and two-factor interactions (A, AB, AC, AD, BC, BD. The Y-axis of the half-normal graph paper is constructed so that normally distributed data falls on a straight line. Therefore, since that near-zero effects fall in a relatively straight line, we can use them for estimation of experimental error. Then, with the aid of computer software, standard statistical analyses such as Analysis of Variance (ANOVA) can be performed to validate the overall outcome and individual effects.

INTERPRETING THE RESULTS

Now you are ready to make your report. In these case, we can start by noting that Factor A was not significant, so you can pick whichever level minimizes cost. Knowing which factors are not important often proves to be the key to success.

Next it will be helpful to make a plot(s) of any significant main effects that are not part of a significant interaction. In the phosphate fertilizer case, only factor B stands alone. The effect of factor B can be seen in Figure 2 (below).

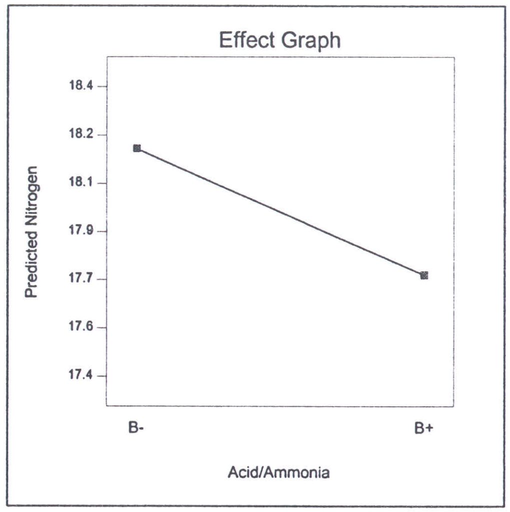

Figure 2. Main Effect Plot for Factor B

Each point on this plot represents a contrast between the response averages at low versus high factor levels. In this case there are eight runs at each level, so the results carry a lot of weight. Going to the low level of B will significantly increase the nitrogen content of the resulting product.

Finally we produce the interaction plot(s). In this case only CD is significant. It's shown in Figure 3 (below).

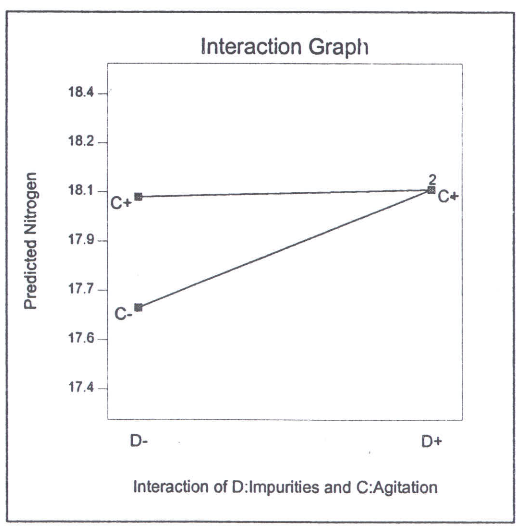

Figure 3. Interpretation Plot for Interaction CD

Notice that the lines on these plots are not parallel. In other words, the effect of one factor depends on the level of the other, so it would be inappropriate to display either of these factors alone. Interestingly factor D (impurity) has a much bigger impact when C (agitation) is at the lower rate. Clearly it's best to go with the high level of agitation to get uniformly high yields, regardless of the level of impurity.

The end result is a process that's robust to impurity levels, producing a consistently high level of purity, at minimum cost. Without the use of DOE, it would be nearly impossible to discover this winning combination. Figure 4 shows the response surface for the CD interaction on nitrogen yield. It dramatically illustrates the quality improvement made possible by changing settings to their optimal levels.

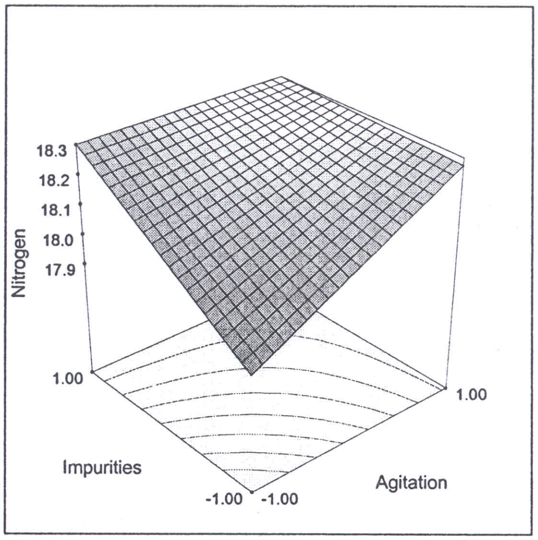

Figure 4. Response Surface Plot for Interaction CD

WHAT'S IN IT FOR US

By equipping ourselves with the basic tools of statistical DOE, we can make the most of "opportunities" such as those presented at the outset of this article. Computer software makes it relatively easy to do. The techniques can be applied to any chemical process to achieve breakthrough improvements in quality and yield.

LITERATURE CITED

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(2) "Mathematics, Statistics" section, *CEP Software Directory*, (supplement to 1/97 issue of *Chemical Engineering Progress*), page 36, AIChE, New York.

(3) Helseth, T.J., et al, *Design-Ease*, Version 5 for Windows ($395), Stat-Ease, Inc, Minneapolis.