STATeaser

ABOUT STAT-EASE® SOFTWARE, TRAINING, & CONSULTING FOR DOE

Workshop Schedule

Experiment Design Made Easy (EDME)

June 18-19, 2013: Minneapolis, MN*
August 12-13 2013: Minneapolis, MN*
September 16-17, 2013: San Francisco, CA*
October 22-23, 2013: Minneapolis, MN
$1295 ($1095 each, 3 or more)

Response Surface Methods for Process Optimization (RSM)

June 20-21, 2013: Minneapolis, MN*
September 18-19, 2013: San Francisco, CA*
$1295 ($1095 each, 3 or more)

Mixture Design for Optimal Formulations (MIX)

August 14-15, 2013: Minneapolis, MN*
November 5-6, 2013: Minneapolis, MN*
$1295 ($1095 each, 3 or more)

Advanced Formulations: Combining Mixture & Process Variables (MIX2)

November 7-8, 2013: Minneapolis, MN*
$1495 ($1195 each, 3 or more)

PreDOE: Basic Statistics for Experimenters Online Course

Free (a $95 value). Learn more at: http://www.statease.com/clas_pre.html.

*Attend the EDME/RSM, EDME/MIX, or MIX/MIX2 workshops in the same week and save $395 on tuition!

Workshops limited to 16. Multiclass discounts are available. Contact Elicia Bechard at 612.746.2038 or workshops@statease.com.

UK Boffins Pull Off Brilliant DOE on Beer

Being a student of beer,* I greatly enjoyed Paul Nelson's presentation on pouring last summer at our Fourth European DOE User Meeting. Paul, the Technical Director and co-founder of PRISMTC (see sidebar), detailed an ingenious experiment that revealed the secrets to the perfect pour for a pint of brew. To get an idea of the lengths the PRISMTC crew** went to, just look at the machine they made to be precise in meeting the settings specified by their experimental design.

They focused on two responses—head height (foam after pour), and life of the beer (time to go flat). Using a response surface method (RSM) design created by Design-Expert® software, the experimenters studied four factors:

A. Dispense time (15 - 30 seconds),
B. Glass angle (45- 90 degrees)
C. Pour Height (0 – 150 millimeters)
D. Beer Type (Premium versus Value)

Note that the last variable is categorical (an 'either-or' choice), but that can be dealt with in a variety of ways, including optimal design, by our program.

For the rest of the story I refer you to PRISMTC's write-up at http://www.prismtc.co.uk/quality-beer-design/,*** where not only do you get all the details, but also a tool to try your hand at this delicate operation via a virtual pint pourer. Amazing!
—Mark Anderson, mark@statease.com

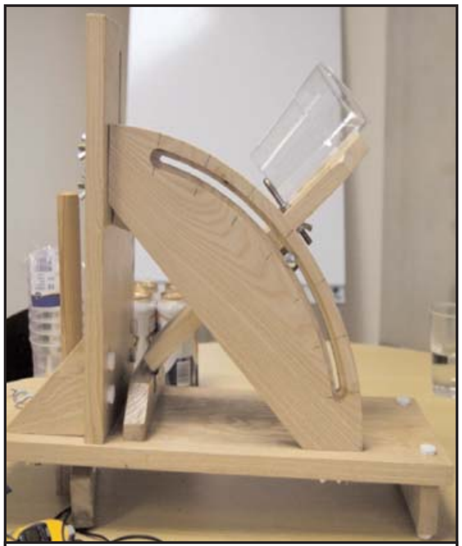

Figure 1: Device for controlling angle of the pour

*(Homage to the Guinness brewer who published his development of sample statistics under the pseudonym "Student.")
**(Stuart Wilson and Andrew Macpherson)
***(Save typing into your browser by using this shortened link: http://is.gd/beerfoam)

P.S. Coincidentally, last summer I attended an outdoor wedding that featured a beer wagon so revelers could just pull themselves a draft brew straight out of the barrels from a line of spigots mounted on the side. That was all well and good except that I could only draw out foam. Luckily a more experienced party-goer knew the trick

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of angling the glass just right so one got mainly liquid with a nice inch of foam at its head. Now if I could only get my hands on the PRISMTC device for receiving beer at the proper angle. Then I would come better prepared when the wagon rolls up the next time around.

PRISMTC, co-founded in 2000 by Paul Nelson (mentioned previously) and Ian Macpherson—master statisticians who came out of the pharmaceutical industry—is the new reseller in United Kingdom for Stat-Ease software. Their clients range from early phase start-ups to large, multinational corporations. They currently employ 12 professionals and associates who work with experimenters in R&D (product development) and manufacturing (process improvement). The PRISMTC team, managed by Iain Wilson, provides a full range of learning solutions for design of experiments and other statistical tools.

The Peril of Parts & the Failure of Fillers as Excuses to Dodge Mixture Design

Definitions of a mixture from A Primer on Mixture Design: What's In It for Formulators?(www.statease.com/pubs/MIXprimer.pdf):

"Mixtures are combinations of ingredients (components) that together produce an end product having one or more properties of interest." —John Cornell & Greg Piepel

"What makes a mixture?
1. *The factors are ingredients.*
2. *The response is a function of proportions, not amounts.*

Given these two conditions, fixing the total (an equality constraint) facilitates modeling of the response as a function of component proportions." —Pat Whitcomb

Given how easy Design-Expert software makes it, mixture design is the go-to tool for formulation DOE—not factorials or response surface methods (RSM). Unfortunately many formulators get schooled on factorials/RSM and then go this route as a bypass. Here are the primary excuses (actual quotes!):

- [Regarding us advising a three-component mixture design.] *"I am a Design-Expert user doing a chemical formulation DOE study. I have two ingredients to study in relation by parts to the base polymer (100 parts). So I don't think it is a mixture design."*
- [Regarding us advising a four-component mixture design.] *"The meeting with the process engineers was interesting. They did not see a clear reason why the 2^3 factorial was inappropriate. They told me that the filler was "like the ocean" compared to the amounts of the other three components and so they didn't see it as a variable to have in the model."*

This white paper dispels these misconceptions, which come down to "parts" and/or "filler"—both being elements of the following real-life cases.

Case 1—An RSM experiment done by parts

Many years ago we worked with a client who learned RSM. Naturally they were very excited by the possibilities of this powerful statistical tool and applied it immediately to the following recipe for a sealant such as you might use at home to keep water from leaking out of your shower:

A. Plasticizer varied from 50 to 100 parts
B. Filler (inorganic) varied from 100 to 250 parts
C. All other ingredients held at 57 parts
D. Polymer set at constant level of 100 parts

They set up a 13-run central composite design (RSM) on the filler and plasticizer. Figure 1 (on page 3) shows the experiment in standard order with the four-run two-level (2^2) factorial core first (1-4), then the four axial points (5-8) and five center points (9-13).

Unfortunately, because the total amount going into the reactor is changing, the proportions of the other ingredients (C) and polymer (D) also vary: They really are not fixed as first thought. This becomes obvious by re-drawing the bar chart in terms of the percentages of materials—see Figure 2 (on page 3). The ingredient ranges are:

A. Filler 23% to 55%
B. Plasticizer 11% to 28%
C. Polymer 19% to 33%
D. Other 11% to 19%

The factors studied are ingredients and performance is a function of proportions: This is a mixture! Fortunately this persuasive presentation helped the formulators see the light. Via a three-component mixture design they varied:

A. Filler 25% to 55%
B. Plasticizer 11% to 28%
C. Polymer 20% to 33%

The remainder of 14% is held constant in all thirteen blends.

To wrap up this case of mistaken identity—what should be a mixture design, not an RSM—take a look at the bar graph in Figure 3 (on page 3). It displays the more appropriate design on three components that make up 86% of the fixed total.

The mixture design controls the proportions of the ingredients and accounts for them in a specialized regression model

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called a "Scheffé polynomial." In this case all the important properties depended on proportions of ingredients, not amounts, so shifting from RSM to mixture design proved to be the secret to success. See the mixture design in Figure 4 below.

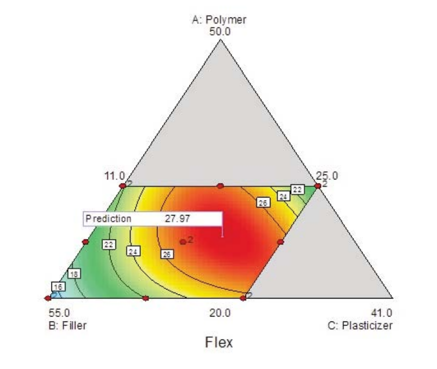

Figure 4: A three-component mixture design on the sealant

Case 2—Filler 'like an ocean' relative to 'active' ingredients

My colleague Pat Whitcomb once worked on a combination of chemicals called "dopants" that created various colors on a cathode ray tube (CRT), such as those used in televisions before flat panels took over. These were mixed in quantities of parts per billion into an inert filler. Naturally the experimenters figured they'd simply run a factorial design on the dopants. However, due to the dependency of color on proportions of these ingredients, this approach failed miserably. Think of mixing one blue and one yellow versus two blues and two yellows—either way the picture goes green. Pat helped these CRT developers re-analyze their results using a mixture model. Unfortunately, by not employing the right tool for this problem—a mixture design—the experimenters failed to uncover the optimal combinations of components, thus they were able to achieve only limited success.

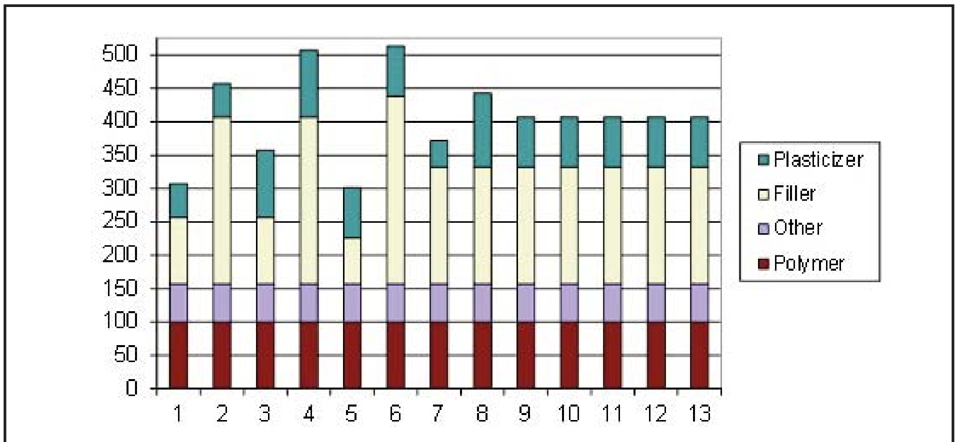

Figure 1: Experiment laid out by parts

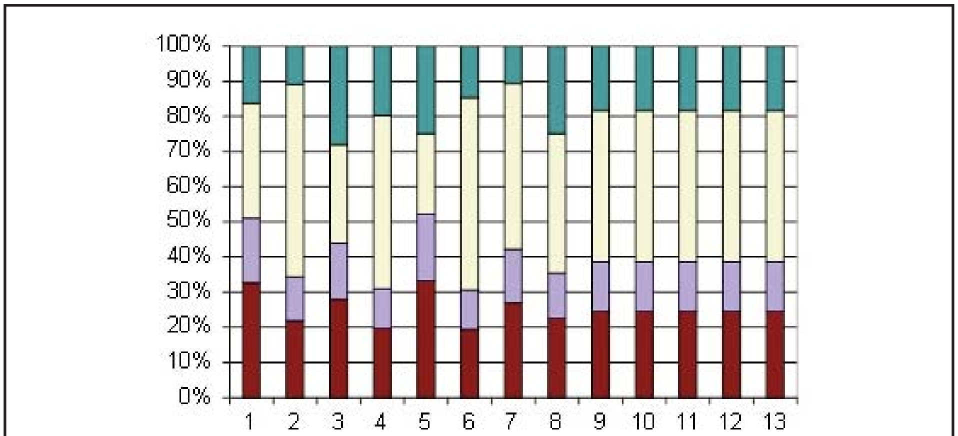

Figure 2: Ingredients recalculated by percentage

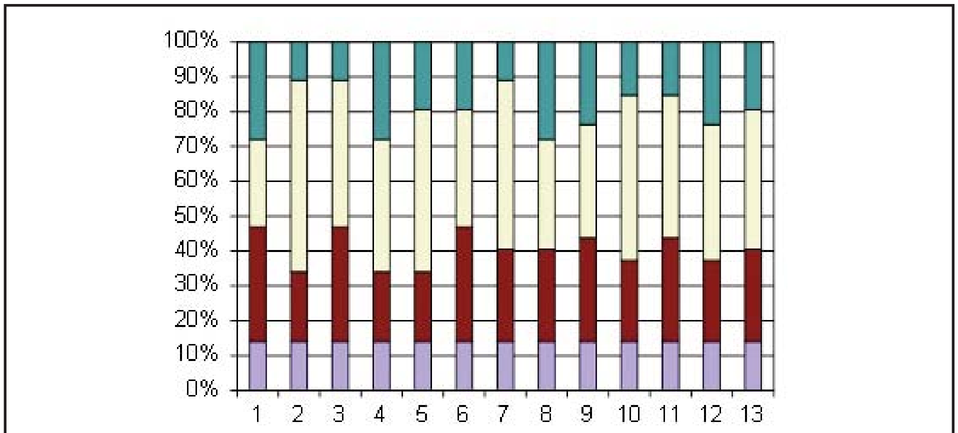

Figure 3: Three-component mixture design shown by percentages

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Conclusion

Being a chemical engineer who worked firsthand on many formulations and aided scores of experimenters in their product development, I find the word "filler" to be an oxymoron—these materials would not be included in the recipe if they did not perform some function, if only to keep extremely potent "active" ingredients diluted. Also, the approach of mixing things by parts has never appealed to me because it creates differing totals batch by batch—very inconvenient when you can only fit so much in the kettle. I prefer fixing the total and then using a mixture design—filler and all. It's easy with Design-Expert software and most effective for finding the formulation's sweet spot.
—Mark Anderson, mark@statease.com

P.S. For those who are determined to use response surface methods for formulation experiments, see "Applying RSM to Mixtures", Chapter 11, RSM Simplified, Anderson & Whitcomb (www.statease.com/rsm_simplified.html).

Pat Whitcomb Wins Award

05/13

At their annual dinner on February 22, the Minnesota Federation of Engineering, Science and Technology Societies (MFESTS) and the Minnesota Society of Professional Engineers awarded Stat-Ease Founder, Patrick Whitcomb, the *Charles W. Britzius Distinguished Engineer Award*. He earned this honor by being technically outstanding in his professional field and by making significant contributions to society through efforts in education and community affairs.

Before starting his own business in 1982, Pat worked as a chemical engineer, quality assurance manager, and plant manager. He was honored in 1982 with *Minnesota's Young Chemical Engineer of the Year* award. In addition to co-authoring design of experiments (DOE) software and books, Pat is a highly-rated teacher and presenter. He has won the *Shewell Award* for best presentation at the Fall Technical Conference on three occasions!

Congratulations, Pat, on another well-deserved recognition.