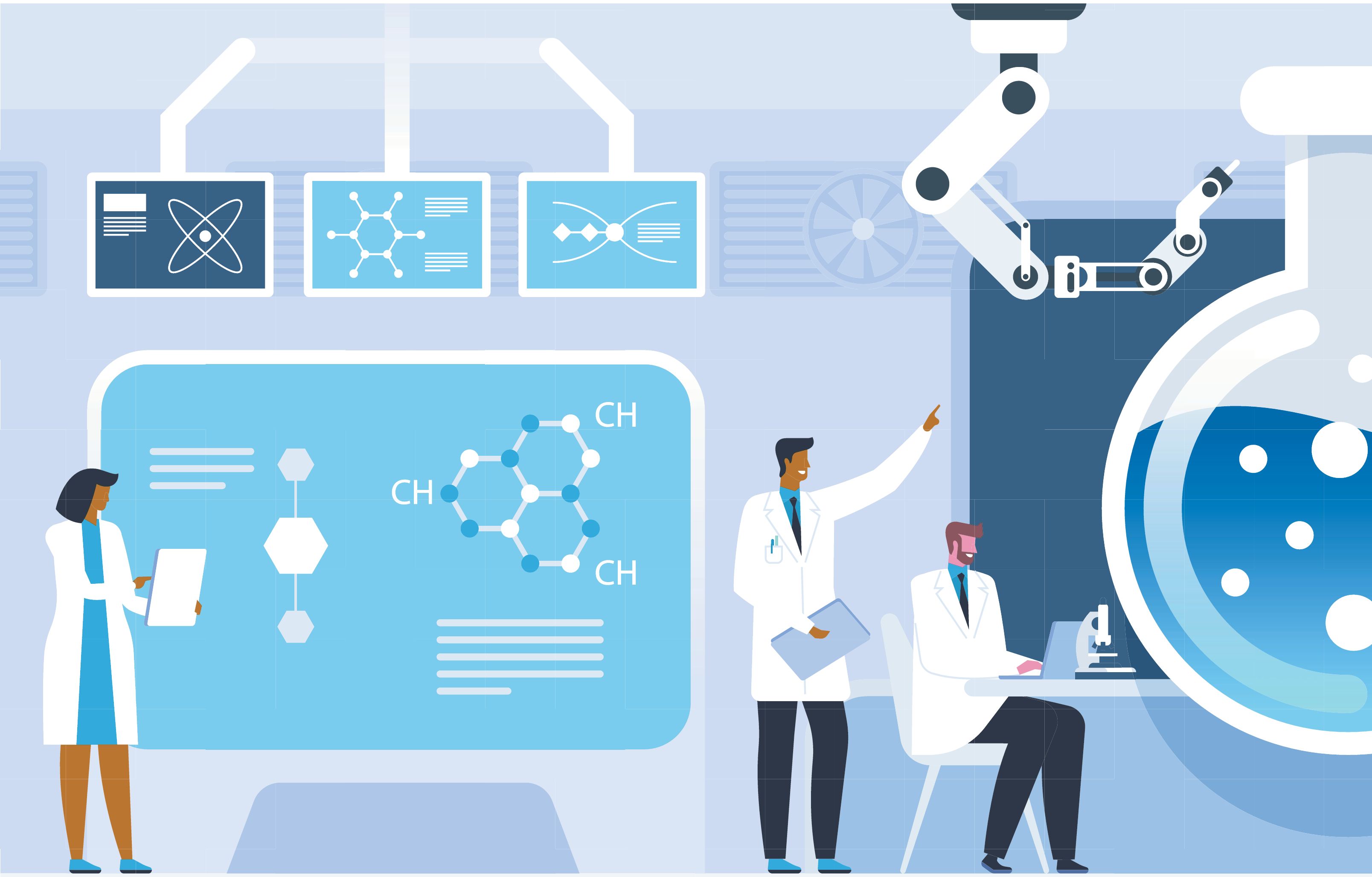
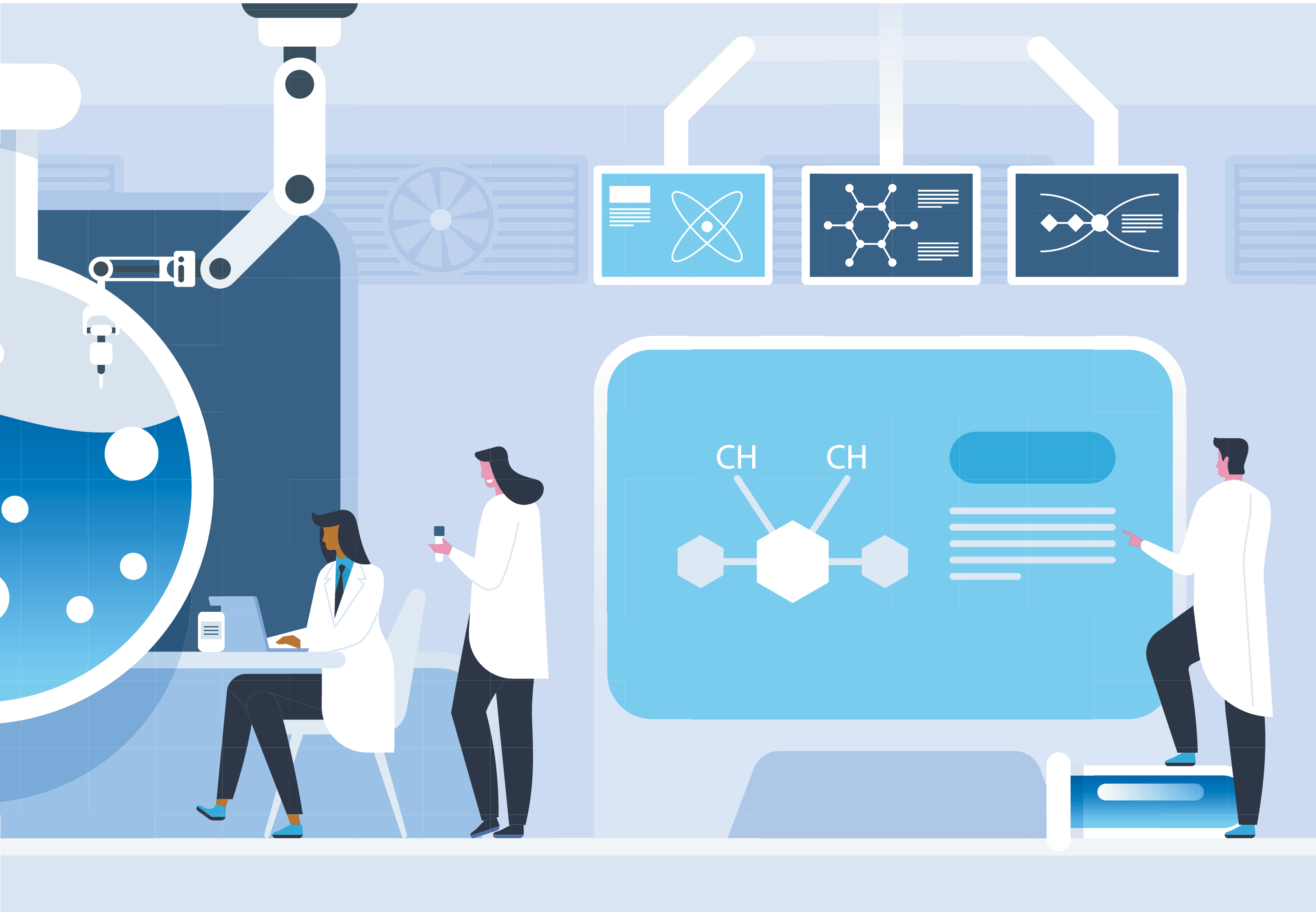


Design of Experiments Pinpoints

Lubricant development can be a frustrating process, especially in the case of formulations. Many commercial products can have 20 or more components. With so many ways for multicomponent blending effects to impact performance, it is difficult to optimize formulations.

Fortunately, a multifactor testing method called design of experiments can be used to simultaneously change input variables in a statistically optimal layout and unveil significant effects from a minimal number of experimental runs.

It is not uncommon for existing products to be slightly modified for the purpose of improving performance or reducing costs. Oftentimes these changes will have the desired effect with no unexpected impacts on a product's critical behaviors. However, there are some cases in which a seemingly simple modification may result in perplexing



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Better Products

By Jason Pandolfo

problems with performance, the roots of which may not be easy to determine, much less fix.

In 2018, Quaker Houghton, a global manufacturer of industrial process fluids based in Conshohocken, Pennsylvania, modified one of its metalworking fluids to improve performance traits and reduce raw material costs. The modification met all of the company's goals except for machinability, a key attribute of metalworking fluids.

Final testing determined that the product's lubricity had unexpectedly worsened due to the changes. Since lubricity is an indicator of machining performance, this discovery was troubling.

Shortly after the fluid's decline in performance was verified, the laboratory began to investigate the cause of the lubricity issue and determined that a branch of applied statistics known as design of experiments would be the most effective and efficient means of exploring the case. Quaker Houghton chemists were trained in this technique and used it to design statistically advantageous studies that provided understanding of the underlying issue as well as a path to optimizing lubricity.

Table 1. Factors and Levels for First Design of Experiments

Factor	Category	Factor Level	
		Low (Product A)	High (Product B)
A	Carrier oil	More expensive	Less expensive
B	Amine package	Lower alkalinity	Higher alkalinity
C	Ethoxylated emulsifier	More expensive	Less expensive
D	Additional emulsifier	0%	1%

Source for all: Quaker Houghton

The Problem

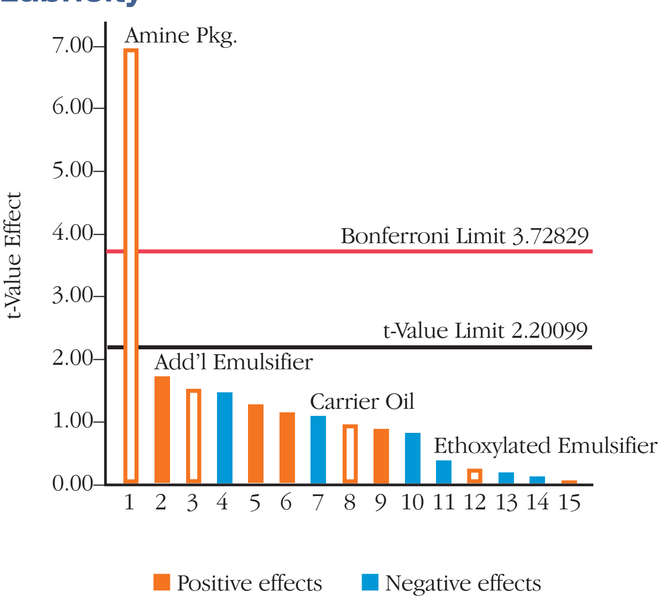
The original formulation, Product A, needed an overall reduction of raw material costs and increased product stability. The new formulation, Product B, aimed to achieve this with a similar but less expensive carrier oil, as well as a similar but less expensive ethoxylated emulsifier and 1 percent of an additional stabilizing emulsifier. Product B also needed a higher level of reserve alkalinity, so it incorporated a 25 percent increase in amine content. No change was made in lubricity additives.

The laboratory did not expect any major performance differences from this straightforward modification to Product A. However, Product B's tapping torque, a measure of machinability, was 15 percent higher than Product A. This indicated that Product B had worse lubricity. Considering that the lubricity additive packages remained the same, this result perplexed the lab team and required further study.

To investigate the root cause of the increased tapping torque, the lab team used Design-Expert software from Stat-Ease Inc. to design an experiment around the four primary ingredients. (See Table 1.)

Due to resource constraints in the laboratory, it was not possible to replicate the samples that were run in the experiment. However, the software showed that the experiment provided adequate ability to resolve any multi-factor interactions on tapping torque.

Figure 1. Pareto Chart of Effects on Lubricity



After analyzing the tapping torque for each of the 16 formulations, the software's specialized factorial-analysis tools revealed that Factor B, which was the change to higher alkalinity in the amine package, caused a significant increase in the tapping torque. The Pareto plot (Figure 1), red-lined at 95 percent confidence, illustrates the results of the experiment.

The amine package was ranked first, which signifies that it had the strongest effect on the fluid's lubricity relative to the other main and multi-factor effects. The three other main effects are labeled only for reference's sake. They fall far short of the significance threshold and therefore do not impact the response.

Exploring the Amine Package

The impact of changing the amines was not expected, as amines are not generally regarded as key lubricity drivers. The next logical step was to experiment with individual amines to see how they might be manipulated to reduce tapping torque while keeping all other specifications in line. Design-Expert provides specialized tools for design of experiments for mixtures, which allowed the lab team to set up a second experiment to study six amines, labeled A through F.

Amines A, B and C were used in the initial reformulation, Product B. The lab team added three new amines to the experiment—D, E and F—to see if they might help identify drivers of increased tapping torque.

Adding water as a component allowed the team to vary the amount of amines in a formulation while still ensuring that the sum of all components added up to 100 percent (the "unity" rule of mixture designs). In effect, the amines as a whole were tested over a range from 67.95 to 135.90 total milliequivalents. This approach allowed the project team to assess the performance impact of the total amine level in addition to that of individual amines.

Anticipating nonlinear blending effects—that is, possible synergisms or antagonisms between components—the lab team chose a custom design for a quadratic mixture model, which examined the effects of four different factors on the formulation. The design also allowed the team to examine the effects that two components had on each other within a mixture.

They bolstered the experiment with 10 check blends and five replicates. The check blends tested the precision of the predictive models derived from the experiment design and assured

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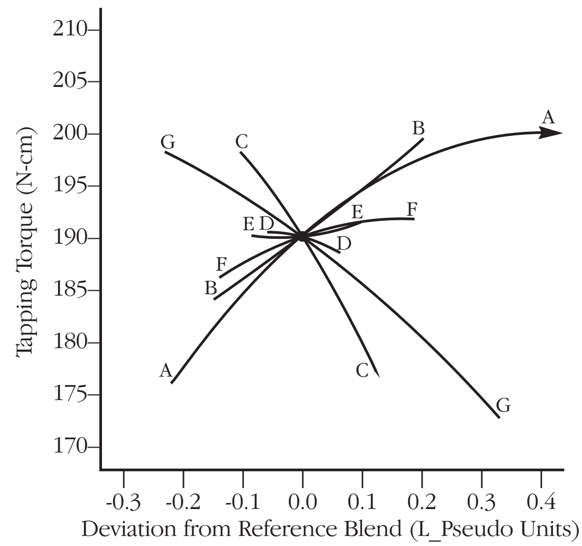
that the results would hold up throughout the region of interest for the factors.

The results for tapping torque exhibited a strong statistical fit to the designed-for quadratic model (greater than 0.95 R^2). This confirmed that although amines are critical for controlling alkalinity of metalworking fluids, they also affect machinability.

The trace plot in Figure 2 reveals that Amine A created the greatest increase in tapping torque and reduction in machinability, followed by Amine B. It also shows that Amine C could be increased to counteract the negative impact of Amines A and B. The relatively flat traces of new Amines D, E and F indicated that they created small effects on tapping torque, but they remained in play as substitutes.

The water portion of the formulation (G) produced the most revealing trace, showing that more water in the formulation (and therefore a smaller proportion of amines) created a substantial decrease in tapping torque. In other words, higher overall levels of amines degrade machining performance.

Figure 2. Tapping Torque Trace Plot



In this trace plot, moving to the right from the center point shows the effect of increasing the amount of an individual amine while proportionally lowering the other components. G is the water component.

Pinpointing a New Formulation

Traditional one-factor-at-a-time methods of experimentation are extremely inefficient but can identify a solution by hit or miss. Multifactor design of experiments, however, produces an extremely valuable predictive model that one-factor methods cannot provide. In this case, the model enabled Quaker Houghton's formulators to "dial in" the tapping torque by manipulating amines while also taking into account their costs and all other critical-to-quality attributes that were measured and modeled.

This sweet spot can be adjusted as needed to produce variations in metalworking fluids as requested by customers. Since it is possible to generate any number of amine combinations and use the software to predict performance, a tremendous amount of guesswork can be removed from the product development process.

Keeping the optimization focused only on minimizing tapping torque led to a new formulation that contained Amine C as well as Amines E and F, which were substituted for Amines A and B.

The impact of this study was immediate and long-lasting. First, the learnings from the factorial investigation ensured that the role that amine packages play in lubricity will not be taken for granted in the future. The follow-on mixture design is a durable model that will enable development chemists to design amine packages for optimal lubricity in similar product matrices. This translates to the development of products with the best possible machining behavior.

Furthermore, the cost function added to the model allows for increased performance and more favorable raw material costs. Quaker Houghton, for instance, gained a model that can potentially save many man-hours of development time.

This case study also highlights the value of design of experiments in the commercial research and development environment. The design of experiments toolbox provided a means for unmistakably identifying the source of the problem. As an iterative process, the follow-on experiment further explored the combined impact of several drivers, revealing interesting effects for individual components. The time that was invested yielded high-impact results that were backed by statistics.

All told, compared to those who remain mired in old-fashioned one-factor experiments, practitioners of design of experiments can learn more about their products with reliable and statistically valid results that are often delivered in a shorter amount of time. ∎

Jason Pandolfo is a metalworking fluid development chemist for Quaker Houghton. He holds a Master of Science in chemistry from Drexel University and a Bachelor of Science in chemistry from the University of the Sciences in Philadelphia. In addition to his career at Quaker Houghton, Pandolfo serves as an engineering duty officer in the United States Navy Reserve.