New-User Intro to Design-Expert® Software

Richard Williams
BSChE, MSM, Six Sigma MBB

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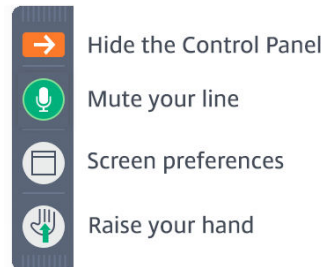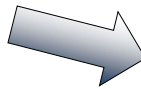
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Goals

- Why consider design of experiment (DOE) methods
- Introduction to Design-Expert by demonstration ("demo")
- Awareness of applications and design types
- Next steps

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Design of Experiments Software and Training
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STAT-EASE 360

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Agenda

➢ DOE – What's in it for me? (WIIFM)

➢ Factorial demo—characterizing vital interactions

➢ Response surface method (RSM) demo—optimization

➢ Mixture design–specialized tools to hit the sweet spot

➢ Optimal (custom) demo – designs for every challenge, e.g., combining mixture and amount

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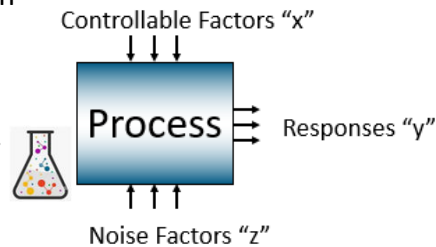
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DOE WIIFM:
What is a Designed Experiment?

> Design of experiments (DOE) is a planned approach for determining cause and effect relationships

> It benefits from multifactor testing rather than evaluating one factor at a time (OFAT)

> DOE can identify factor interactions!

Controllable Factors "x"

Process

Responses "y"

Noise Factors "z"

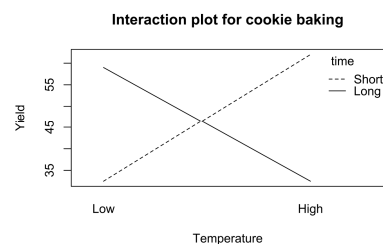DX Software Intro for New Users

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DOE WIIFM
Why do design of experiments?

> OFAT experimentation cannot identify factor interactions which frequently play a key role in process improvement, e.g., time and temperature affecting your yield of cookies—too much of both will burn them, not enough leaves them unbaked.*

> DOE multifactorial experimentation by its parallel processing provides more information in fewer runs than OFAT (an inefficient and old-fashioned serial method)

> DOE statistics provide confidence in the findings

Interaction plot for cookie baking

Yield

55

45

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time

--- Short
— Long

Low   High

Temperature

*Source of graphic: https://en.wikipedia.org/wiki/Interaction_(statistics)
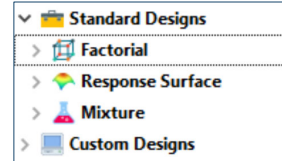
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DOE WIIFM
SCO Strategy of Experimentation

Design-Expert provides a full array of designs and analysis tools to address three overarching goals:

- ➢ Screen for vital few process factors or mixture components
- ➢ Characterize critical interactions
- ➢ Optimize for peak performance

∨ 📦 Standard Designs
> 🔲 Factorial
> 🔶 Response Surface
> 🔺 Mixture
> 🖥 Custom Designs

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DOE Process

1. Define objective in terms of measurable responses
2. Select the input factors to study
3. Select your design (the layout of factor levels run-by-run)
4. Conduct the experiment (the hard part!)
5. Analyze the results using your software's statistical tools
6. Diagnose the residuals for abnormalities and outliers
7. Interpret the outcomes using model graphs
8. Optimize factors for maximum desirability of multiple responses
9. Test your predictive model via confirmation runs
10. Celebrate!

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Reactor Case Study
2^5 Full Factorial

Find the conditions that maximize yield of a chemical reaction.

There are five key factors that the engineers chose:

A. Feed rate, 10-15 l/min
B. Catalyst level, 1-2 wt %
C. Agitation rate, 100-120 rpm
D. Temperature, 140-180 deg C
E. Atmosphere, nitrogen or air*
   *Saves time not having to batten down the hatch.
   But will the air ruin the reaction? Let's find out!*

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Reactor Case Study
2^5 Full Factorial

> Objective: Maximize yield

> Parameters for calculating power
> *Ensures that design will likely succeed if*

❖ Signal (Δy) = 3 %
   Any improvement less than this will be unimportant

❖ Noise (σ) = 2 %
   The variability in the reaction, sampling and testing

rsw reactor.dxpx
Rebuild (show power!) and analyze

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Factorial Design Summary
Things to Remember

- Full-factorial designs test all possible factor combinations. They characterize <u>all</u> effects: main, two-factor interactions (2fi's), etc.
- Power assesses the probability an experimental design will identify important effects. It is driven by the signal-to-noise ratio (Δy/σ). Ideally power will be in the range of 80 to 95%, i.e., with just the right number of runs, not too few and not too many.

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Factorial Design Summary
Tip 💡

- Consider saving time and resources via fractionation. For example, the 32-run reactor experiment could be cut in half and still characterize 2fi's. But be wary of loss of power and resolution. Green is good , yellow OK (for screening) and red best avoided (other than ruggedness testing)

Runs	2	3	4	5	6	7
4	2^2	2^{3-1}_{III}				
8		2^3	2^{4-1}_{IV}	2^{5-2}_{III}	2^{6-3}_{III}	2^{7-4}_{III}
16			2^4	2^{5-1}_{V}	2^{6-2}_{IV}	2^{7-3}_{IV}
32				2^5	2^{6-1}_{VI}	2^{7-2}_{IV}
64					2^6	2^{7-1}_{VII}

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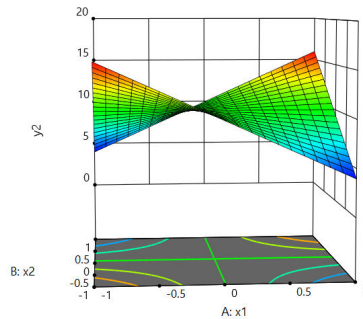➢ Optimal (custom) demo – designs for every challenge, e.g., combining mixture and amount
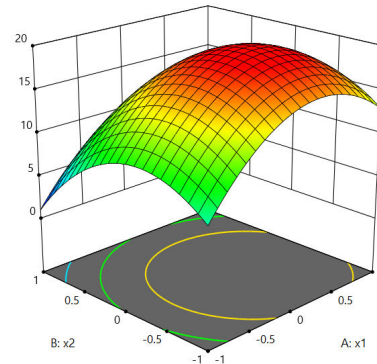
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Response Surface Method Designs
Handling "Pure" Curvature

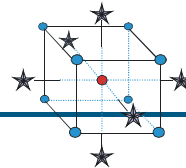Two-factor interactions
twists in the plane
$y=10+6AB$

Squared terms
symmetric curvature
$y=19+3A-3B-6A^2-6B^2$

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Reaction Case Study
RSM (Central Composite Design)

Engineers hope to find conditions that maximize conversion while also keeping activity within a target range.

There are three key factors you think are important

A. Time, 40-50 minutes

B. Temperature, 80-90 deg C

C. Catalyst, 2-3 wt %

Goals: Achieve greater than 80% conversion and maintain activity between 60 and 66, with 63 being ideal.

The engineers believe the optimal conditions will be in the interior of the design space.
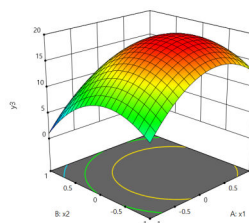
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Reaction Case Study
RSM (Central Composite Design)

➢ Objective: maximize conversion and target activity

➢ Responses: Conversion %, Activity

➢ Goals:

  ➢ Conversion >80%

  ➢ Activity Target 63 +/- 3

RSW RXN-a.dxpx
Rebuild, model & optimize.

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RSM Design Summary
Things to Remember

▪ RSM designs are useful when curvature (non-linearity) is anticipated within the design space

▪ The model terms include quadratic (or higher) terms in addition to the main effect and interaction terms from factorial designs

▪ More runs will be required for RSM designs than for factorials

▪ Numerical and graphical optimization tools in DX find the optimal conditions to satisfy one or more criteria

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Stat-Ease Training:
Sharpen Up Your DOE Skills

Modern DOE for Process Optimization

Discover how to use factorial and RSM designs to gain process understanding and optimize your products.

Individuals	Teams (6+ people)
Improve your DOE skills	Choose your date & time
Topics applicable to both novice and advanced practitioners	Add company case studies

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What Makes a Mixture?
Two Conditions

1. The factors are ingredients of a mixture.

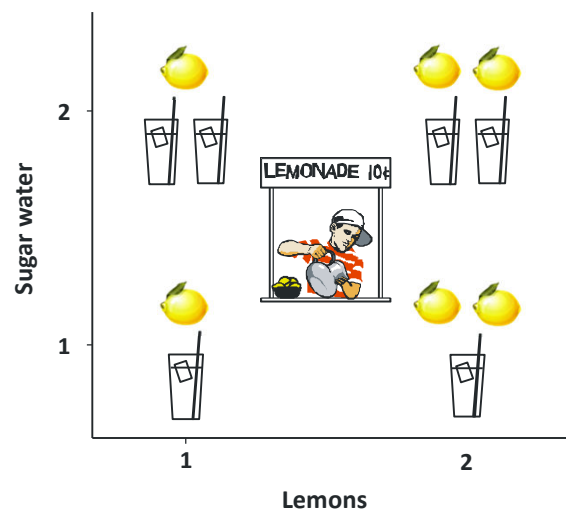2. The response is a function of proportions; not amounts.

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

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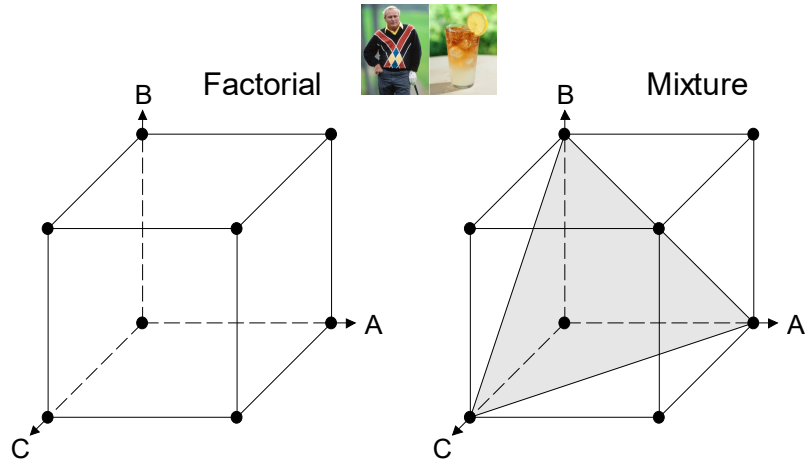
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Lemonade
Taste Dependent on Proportions

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Three-Component Mixture
(e.g., an Arnie Palmer—tea added to lemonade)

Factorial

Mixture

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Mixture Amount Experiments
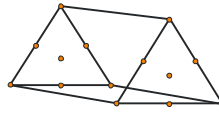
A mixture-amount experiment is used when the response depends on both composition and amount of the mixture:

☑ Application of fertilizer - - Yield is a function of both the proportions of ingredients and the amount of fertilizer applied.

☑ Paint - - Coverage is a function of both the proportions of ingredients and the thickness of the coating applied.

☑ Medicine - - Effectiveness is a function of both the proportions of ingredients and the dosage prescribed.

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Mixture-Amount Experiment
Ibuprofen Coating

Pharmaceutical formulators must optimize the composition and the amount of latex coating on a controlled-release ibuprofen. The total solids content is 31% w/v, comprised of three components:

A) EA [Ethylacrylate] (7 to 20%)
B) MMA [Methylmethacrylate] (7 to 20%)
C) TEC [Triethyl Citrate] (4 to 14%)

The amount of latex coated on to a fixed number of tablets is :

D) Coating (15 to 25 ml)

The responses are the cumulative % of drug dissolved in 1 and 12 hours.

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Mixture-Amount Experiment
Ibuprofen Coating

> Objective: Optimize coating formulation and amount

> Goals:

❖ Dissolve 10-30% (target 20%) in 1 hour
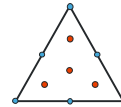
❖ Dissolve 80-100% (target 90%) in 12 hours

rsw ibuprofen-a
Rebuild, model & optimize.

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Mixture (Combined) Design Summary
Things to Remember

- Mixtures have a built-in constraint: the formulation must sum to a total

- Although mainly used for optimization a la RSM designs, because of the constrained total, special approaches are needed to build DOE's for mixtures and model the results

- Design-Expert can create custom (optimal) designs that combine mixture components and process factors (e.g., time, temperature, amount, etc.)

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Stat-Ease Training:
Sharpen Up Your DOE Skills

Mixture Design for Optimal Formulations

*Master formulation studies, from screening to optimization.
Mixture-process combined designs provide even more in-depth
information.*

Individuals	Teams (6+ people)
Improve your DOE skills	Choose your date & time
Topics applicable to both novice and advanced practitioners	Add company case studies

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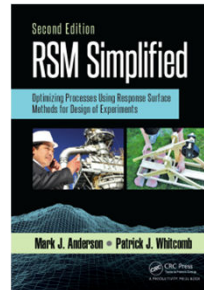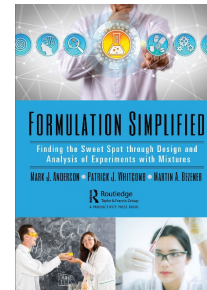DX Software Intro for New Users

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Summary

- A DOE is a planned, multifactor, approach to efficiently and effectively determine cause and effect relationships.

- DOE's statistics provide power and credibility to the conclusions.

- Design-Expert software, specialized for DOE, provides a wide variety of design types and modeling tools to address virtually all experimental needs.

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References*
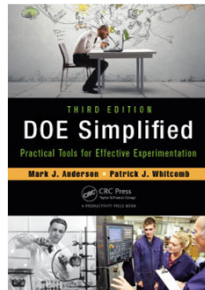
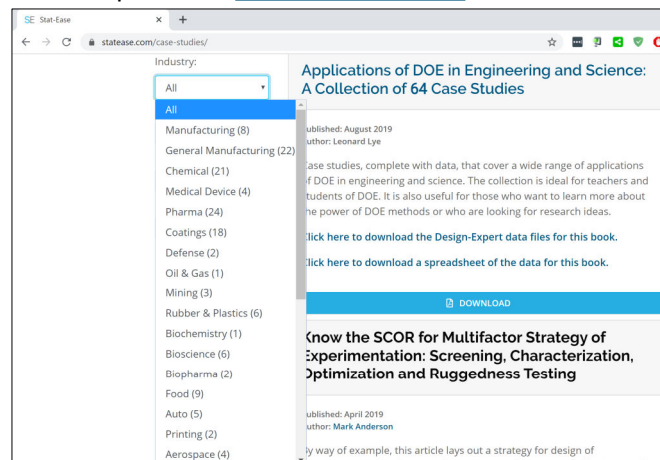*3rd edition 2015*   *2nd edition 2016*   *1st edition 2018*

* Taylor & Francis/CRC/ Productivity Press New York, NY.

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Inspiring industry-specific success stories

❑ Case studies posted at www.statease.com under Publications

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  - ❑ Wealth of white papers and other pubs at www.statease.com.
- ❑ In Design-Expert software press the lightbulb for Screen Tips, view reports in annotated mode, look for context-sensitive Help (right-click) or search the Help system.
- ❑ Tutorials within the Design Expert Help tab plus accompanying data sets

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Richard Williams
stathelp@statease.com