Keys to Successful DOE

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Maximizing this educational opportunity

Welcome everyone! To make the most from this webinar:

- Attendees on mute
- Questions addressed afterward
- Send further questions to stathelp@statease.com

Presentation posted: www.statease.com/webinars/ and
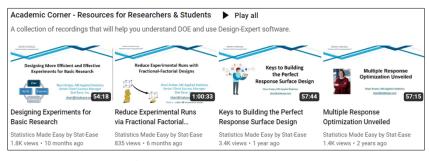
▶ YouTube   Statistics Made Easy by Stat-Ease

Stat-Ease YouTube Channel

Easy source for more content: **StatisticsMadeEasybyStatEase**

On this channel, explore playlists on DOE topics:

Academic Corner - Resources for Researchers & Students   ▶ Play all

A collection of recordings that will help you understand DOE and use Design-Expert software.

Designing Experiments for Basic Research	Reduce Experimental Runs via Fractional Factorial...	Keys to Building the Perfect Response Surface Design	Multiple Response Optimization Unveiled
Statistics Made Easy by Stat-Ease	Statistics Made Easy by Stat-Ease	Statistics Made Easy by Stat-Ease	Statistics Made Easy by Stat-Ease
1.8K views • 10 months ago	835 views • 6 months ago	3.4K views • 1 year ago	1.4K views • 2 years ago

There will be many references to videos on this site!

Keys to Successful DOE

Keys to Successful DOE

Design of experiments is an **ART**, as well as a science. Over time we have identified some key concepts and tools that, when used properly, lead to highly successful DOE's. We will discuss a few key concepts for **planning** the experiment, and then keys to **analyzing** the data.

Planning the DOE	Analyzing the results
Define objective	Selection of model terms
Measure responses	Residual diagnostics
Size the design	Interpreting results
Ordering of runs	Confirmation runs

Keys to Successful DOE

Agenda: Keys to Successful DOE - Planning

➢ **Defining the objective**

➢ Measuring the responses

➢ Sizing the design

➢ Ordering the runs

Plan

Verify

Execute

Analyze
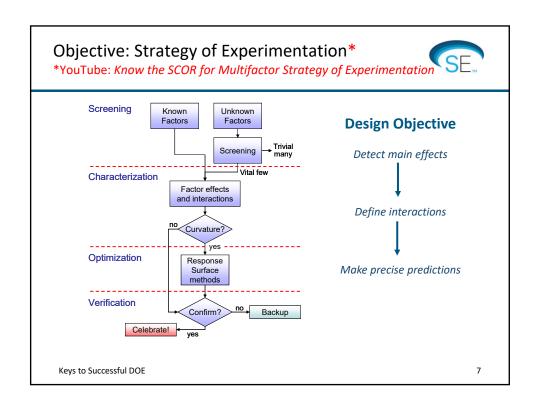
Planning the Experiment - Objective

What kind of information is needed from this experiment?
How many runs can you afford?

Consider your *strategy of experimentation* at this stage – are you doing:

- Screening
- Characterization
- Optimization

Choose a design that will provide the information you need.

Objective: Strategy of Experimentation*
*YouTube: *Know the SCOR for Multifactor Strategy of Experimentation*

Screening

Known Factors		Unknown Factors

Screening → Trivial many

Vital few

Characterization

Factor effects and interactions

no — Curvature?

yes

Optimization

Response Surface methods

Verification

Confirm? — no → Backup

Celebrate! yes

Design Objective

Detect main effects

↓

Define interactions

↓

Make precise predictions

Planning the Experiment
Choosing the Design – Use DOE Software!!

- Screening – assuming that interactions exist in the system, use a design that keeps main effect information **unbiased** from two-factor interaction effects.
  - Resolution IV (yellow), Res V (green) or better designs
  - NOT Res III (red), Plackett-Burman or Taguchi designs

- Characterization – use a design that can estimate all two-factor interactions (Full factorial or Resolution V+).
  - Consider adding center points to test for non-linearity (curvature)

- Optimization – use a response surface design like central composite, Box-Behnken, or optimal (custom) design.

- Formulations – use a mixture design for either screening or characterization/optimization (likely an optimal design).

Planning the Experiment
Choosing the Design – Use DOE Software!!

Why use DOE Software to design/build your experiment?

- Allows you to choose from modern design options that may be better than those used by previous researchers

- Defaults will provide a better design than what you would create by hand or using a textbook template

- Reminds you to consider adding center points, blocking, replicates

- Allows a check of power (or precision) to size the design properly

- Final design layout provided in random run order

- Easy to add response data (copy/paste to Excel if desired)

*Most DOE **analysis** problems are caused by errors made in **building** the designs.*
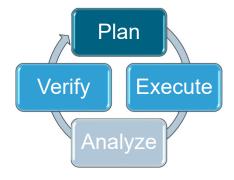

Agenda: Keys to Successful DOE - *Planning*

- ➢ Defining the objective
- ➢ **Measuring the responses**
- ➢ Sizing the design
- ➢ Ordering the runs

Plan

Verify

Execute

Analyze

Planning the Experiment - Responses

Determine which **response(s)** will be measured – make sure the measurement system will produce accurate results.

1. Continuous numeric – best!

2. Rating scale (1-5 or 1-9) – experimental data should span the full range

3. Pass/Fail – this is a binomial response, so will require 75-100 runs/samples to do model fitting

The DOE results should span the full range of "good" product to "bad" product in order to create a strong predictive model.

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Agenda: Keys to Successful DOE - Planning

➢ Defining the objective
➢ Measuring the responses
➢ **Sizing the design**
➢ Ordering the runs

Plan

Verify   Execute

Analyze

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Planning the Experiment
Check for sample size – sizing the design*

Confirm your DOE includes enough runs to get results (appropriate for its type and purpose):

- Factorial Designs – size for Power.
  The power to detect each individual effect/coefficient is key.
- Response Surface (RSM)/Mixture design – size for Precision.
  The ability to predict the mean response with a specified amount of precision.

1. For Power (factorial designs), define:
   - Δy : minimum change in the response you want the DOE to detect
   - s: standard deviation of the response (historical data)
   - Size to achieve power 80% or greater

*YouTube: *How Many Runs Do I need? Using Power & Precision to Size DOE's*

Planning the Experiment
Check for sample size – sizing the design*

2. For Precision using the Fraction of Design Space tool (FDS) (RSM and Mixture designs), define:
   - d: precision of the predicted mean (predicted y **+/- d**)
   - s: standard deviation of the response (historical data)
   - Size to achieve FDS 80% or greater

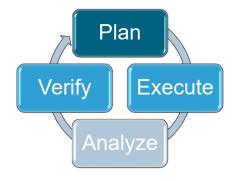*With this info, you can answer the question...How many runs are needed?*

Agenda: Keys to Successful DOE - Planning

- ➤ Defining the objective
- ➤ Measuring the responses
- ➤ Sizing the design
- ➤ **Ordering the runs**

Plan

Verify   Execute

Analyze

Planning the Experiment – Ordering the Runs

Run order matters!

Random is best! Protects against external variables inadvertently biasing the factor effects. (Chemical degradation, humidity change, etc.)

Std	A	B	C	
1	−	−	−	y_1
2	+	−	−	y_2
3	−	+	−	y_3
4	+	+	−	y_4
5	−	−	+	y_5
6	+	−	+	y_6
7	−	+	+	y_7
8	+	+	+	y_8

$$\text{Effect}(\Delta y) = \frac{\sum y_+}{n_+} - \frac{\sum y_-}{n_-}$$

If runs are completed in standard order (or sorted by any column), then

Effect estimate = Factor effect + other unknown effect

NOT unbiased and potentially wrong

Planning the Experiment – Ordering the Runs

How to deal with restrictions on randomization:

1. Blocking – tool to statistically order the runs to protect against bias from a variable that you are NOT studying but is also not constant. *Examples: day-to-day variation, multiple analysts*

2. Split-plot designs* – design option when you must change the run order based on a factor setting to make the design feasible to run. This requires a special analysis to correctly account for the hard-to-change versus easy-to-change factors.
YouTube: Split Plot Pros and Cons: Dealing with a Hard to Change Factor

RANDOMIZE! If you can't randomize, then use the proper tool to restrict the randomization. Use DOE software to do it right!
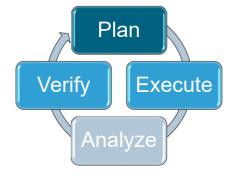

Agenda: Keys to Successful DOE - Analysis

➢ **Selecting the model**
➢ Residual diagnostics
➢ Interpreting results
➢ Confirmation runs

Plan

Verify

Execute

Analyze

Analyzing the data – Selecting the model

Selecting the model is the **MOST IMPORTANT** part of data analysis!!

- Only include terms that are statistically significant
- Or required to maintain model hierarchy
  (If AB is in the model, parent terms A and B should be included)

All other terms are pooled together to estimate the residual (error) of the system.

The polynomial model is used to create the model graphs, predict future results, and optimize the system. If the model is wrong, everything is wrong.

Analyzing the data – Selecting the model
Factorial Design

Configure | Effects | ANOVA | Diagnostics | Model Graphs
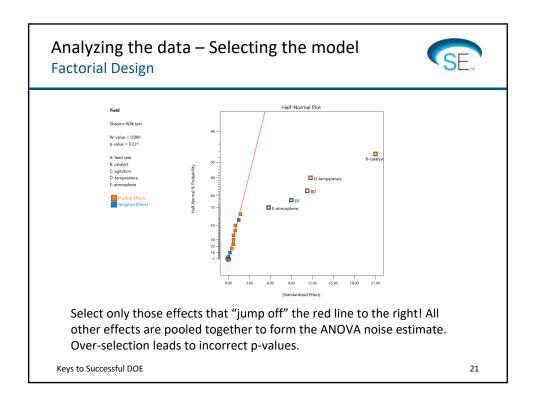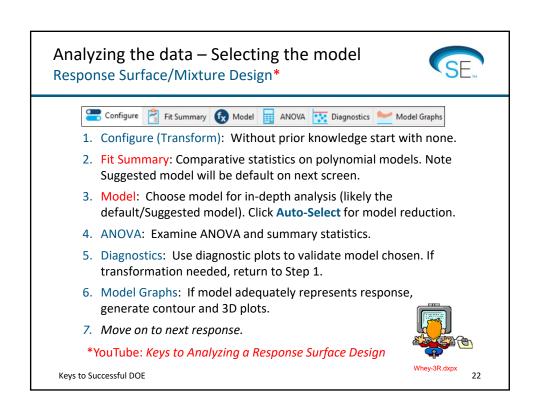
1. Configure (Transform): Without prior knowledge start with none.
2. Effects*: Use **Half-Normal plot** to select effects to include in model, leaving the remainder to be pooled together for the residual.
3. ANOVA: Examine ANOVA and summary statistics.
4. Diagnostics: Use diagnostic graphs to validate model chosen. If transformation needed, return to Step 1.
5. Model Graphs: If model adequately represents response, generate contour and 3D plots.
6. *Move on to next response.*

*YouTube: *Graphical Selection of Factorial Effects*

Reactor half-fraction.dxpx

Analyzing the data – Selecting the model
Factorial Design

Yield

Shapiro-Wilk test

W-value = 0.900
p-value = 0.221

A: feed rate
B: catalyst
C: agitation
D: temperature
E: atmosphere

Positive Effects
Negative Effects

Half-Normal Plot

B-catalyst

D-temperature

BD

DC

E-atmosphere

Half-Normal % Probability

|Standardized Effect|

Select only those effects that "jump off" the red line to the right! All other effects are pooled together to form the ANOVA noise estimate. Over-selection leads to incorrect p-values.

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Analyzing the data – Selecting the model
Response Surface/Mixture Design*

Configure | Fit Summary | Model | ANOVA | Diagnostics | Model Graphs

1. Configure (Transform): Without prior knowledge start with none.
2. Fit Summary: Comparative statistics on polynomial models. Note Suggested model will be default on next screen.
3. Model: Choose model for in-depth analysis (likely the default/Suggested model). Click **Auto-Select** for model reduction.
4. ANOVA: Examine ANOVA and summary statistics.
5. Diagnostics: Use diagnostic plots to validate model chosen. If transformation needed, return to Step 1.
6. Model Graphs: If model adequately represents response, generate contour and 3D plots.
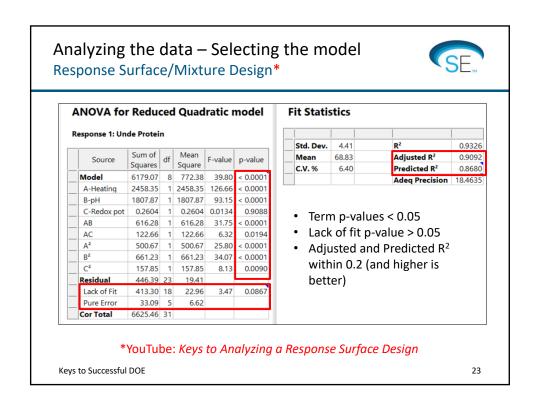7. *Move on to next response.*

 *YouTube: *Keys to Analyzing a Response Surface Design*

Whey-3R.dxpx

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Analyzing the data – Selecting the model
Response Surface/Mixture Design*

ANOVA for Reduced Quadratic model

Response 1: Unde Protein

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	6179.07	8	772.38	39.80	< 0.0001
A-Heating	2458.35	1	2458.35	126.66	< 0.0001
B-pH	1807.87	1	1807.87	93.15	< 0.0001
C-Redox pot	0.2604	1	0.2604	0.0134	0.9088
AB	616.28	1	616.28	31.75	< 0.0001
AC	122.66	1	122.66	6.32	0.0194
A^2	500.67	1	500.67	25.80	< 0.0001
B^2	661.23	1	661.23	34.07	< 0.0001
C^2	157.85	1	157.85	8.13	0.0090
Residual	446.39	23	19.41		
Lack of Fit	413.30	18	22.96	3.47	0.0867
Pure Error	33.09	5	6.62		
Cor Total	6625.46	31			

Fit Statistics

Std. Dev.	4.41	R^2	0.9326
Mean	68.83	**Adjusted R^2**	0.9092
C.V. %	6.40	**Predicted R^2**	0.8680
		Adeq Precision	18.4635

- Term p-values < 0.05
- Lack of fit p-value > 0.05
- Adjusted and Predicted R^2 within 0.2 (and higher is better)

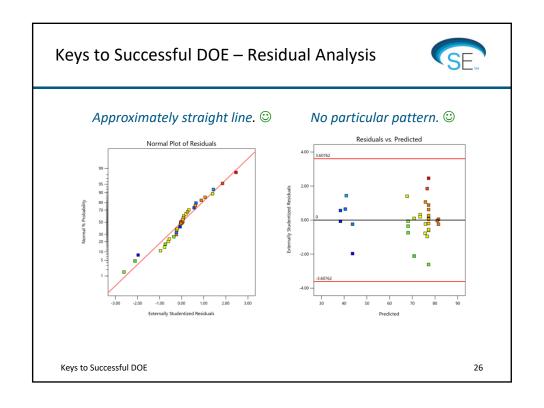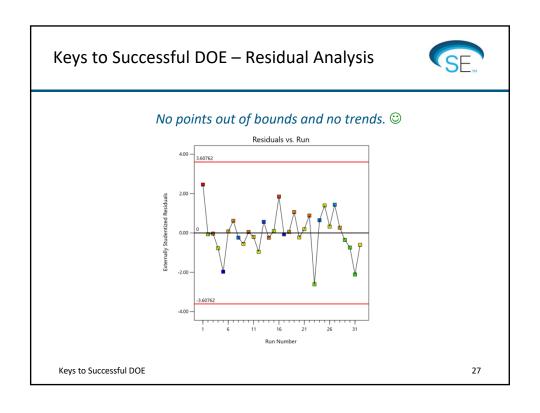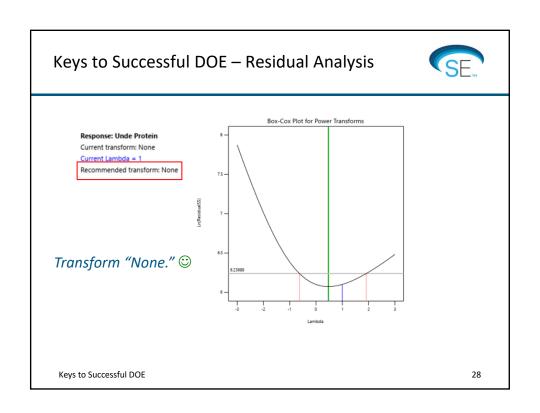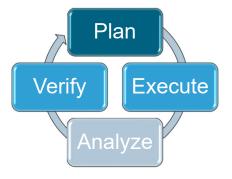*YouTube: *Keys to Analyzing a Response Surface Design*

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Agenda: Keys to Successful DOE - Analysis

- ➢ Selecting the model
- ➢ **Residual diagnostics**
- ➢ Interpreting results
- ➢ Confirmation runs

Plan

Verify

Execute

Analyze

Keys to Successful DOE – Residual Analysis

```
  ┌─────────────────┐       ┌─────────────────┐       ┌─────────────────┐
  │      Data       │       │    Analysis     │       │      Model      │
  │ (Observed Values)│ ───▶ │                 │ ───▶ │(Predicted Values)│
  │       y_i       │       │  Filter Signal  │       │       ŷ_i       │
  │  Signal + Noise │       │                 │       │     Signal      │
  └─────────────────┘       └─────────────────┘       └─────────────────┘
```

$$\text{Residuals}$$
$$\text{(Observed - Predicted)}$$
$$e_i = y_i - \hat{y}_i$$
$$\text{Noise}$$

Independent $N(0, s^2)$

There should be no "signal" remaining in the residuals, i.e. no visible patterns.

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Keys to Successful DOE – Residual Analysis

Approximately straight line. ☺   *No particular pattern.* ☺

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Keys to Successful DOE – Residual Analysis

No points out of bounds and no trends. ☺

Residuals vs. Run

Keys to Successful DOE – Residual Analysis

Response: Unde Protein
Current transform: None
Current Lambda = 1
Recommended transform: None

Box-Cox Plot for Power Transforms

Transform "None." ☺

Agenda: Keys to Successful DOE - Analysis

- ➢ Selecting the model
- ➢ Residual diagnostics
- ➢ **Interpreting results**
- ➢ Confirmation runs

Plan

Verify   Execute
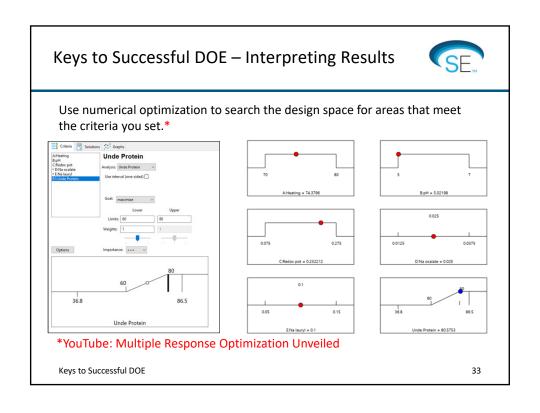
Analyze

Key: Interpreting & Telling the Story

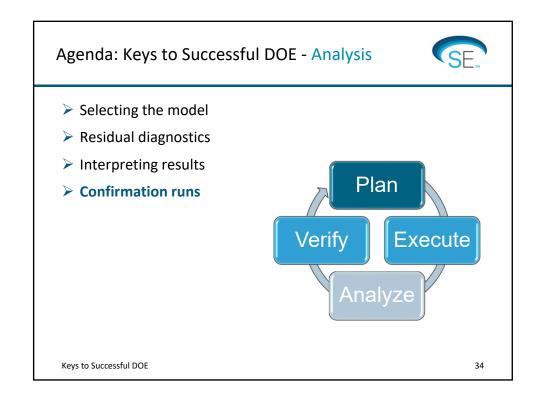Reminder: What were the original objectives of the experiment?

1. Review the model graphs.
   a. Which factors are NOT statistically significant? How is this information important to the business?
   b. What new process understanding do you get?

2. Consider numerical optimization.
   a. Change the limits to reflect customer specifications.
   b. Getting many results means that you have a robust process, and you can tighten the limits to focus more on an optimal area.

Keys to Successful DOE – Interpreting Results

High Yield: Catalyst +, Temperature +, use Nitrogen
Expected Yield: 90%

Keys to Successful DOE – Interpreting Results

There is an area of the design space that gives maximum response – optimization will give many good "solutions", not just 1 solution.

Keys to Successful DOE – Interpreting Results

Use numerical optimization to search the design space for areas that meet the criteria you set.*

*YouTube: Multiple Response Optimization Unveiled

Agenda: Keys to Successful DOE - Analysis

➤ Selecting the model
➤ Residual diagnostics
➤ Interpreting results
➤ **Confirmation runs**

Keys to Successful DOE – Confirmation Runs

Final steps after analysis:

1. Interpret the model graphs using subject matter knowledge.

2. Use numerical and/or graphical optimization to find a "sweet spot"

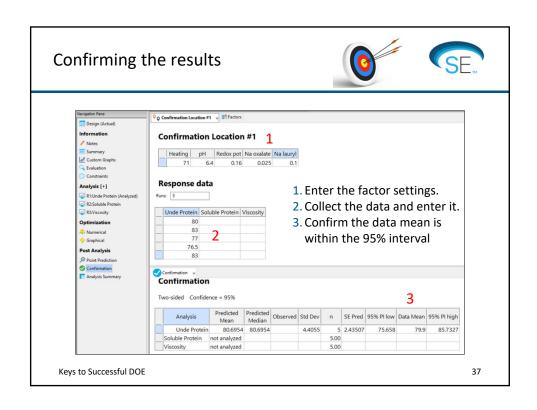3. **Confirm/verify the results – do the predictions work?**

4. Write a report

Confirming the results
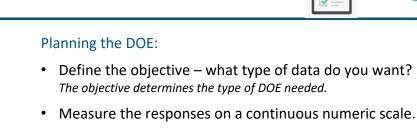
1. Gather **one single observation** at the optimal settings.
   - Does 1 run provide a good estimate of the process?

2. Gather **several observations** at the optimal settings.
   - Use Confirmation node - If the mean is within the adjusted prediction interval, then the model has been confirmed.

3. Run a small **experiment** around the optimal settings.
   - Treat the optimal setting like a center point. Pick a small range for each factor. Compare the predictions at the center point in the new experiment to the prediction interval calculated by the previous analysis. Useful to get some sensitivity analysis in addition to confirming the previous model.

Confirming the results

1. Enter the factor settings.
2. Collect the data and enter it.
3. Confirm the data mean is within the 95% interval

Recap: Keys to Successful DOE

Planning the DOE:

- Define the objective – what type of data do you want?
  The objective determines the type of DOE needed.

- Measure the responses on a continuous numeric scale.

- Check power or precision of the design to confirm that the sample size is large enough.

- Run the design in random order or change the design to accommodate any restrictions.

Recap: Keys to Successful DOE

Analyzing the data:

- Select only significant model terms and leave the remainder in the residual. Don't overfit the model!

- Check residual diagnostics and Box-Cox plot for anomalies.

- Use model graphs in combination with optimization tools to gain process understanding and tell the story of the data.

- Go back to the process and complete confirmation runs to verify that the results are real.

Foundational DOE Training

Modern DOE for Process Optimization

Mixture Design for Optimal Formulations

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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Thank you for listening!

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