# Introduction to Design of Experiments for UV/EB Scientists and Engineers

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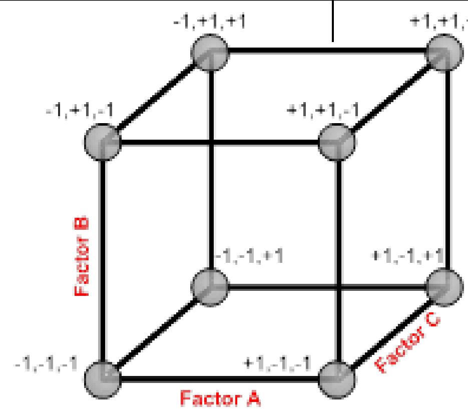
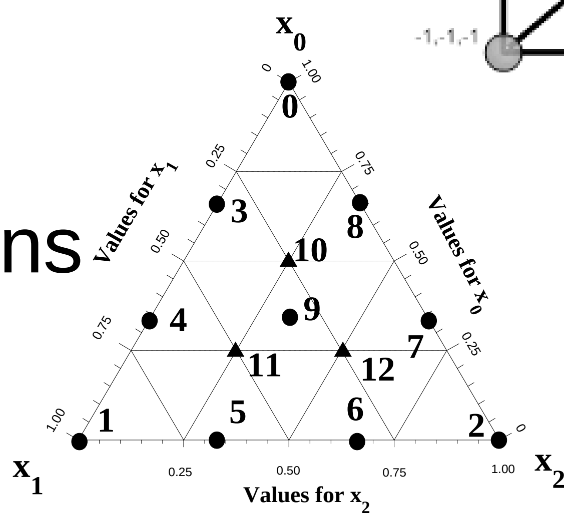
# Outline

- Introduction and Motivation
- Steps in DOE
- DOE Basics
- Screening Designs
- Model Building Designs
- Mixture Designs




10 Design Conditions ( ● )
3 Check Conditions ( ▲ )

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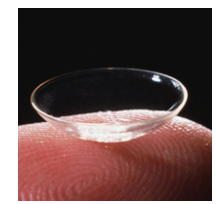
# (Statistical) Design of Experiments- DOE

Careful and efficient plan for data collection and analysis to test hypotheses – maximize information with fewest experiments

## Intentions

- Product/ Process Improvement
- Lower Product Variability
- Improve Process Robustness
- Expand Scientific Knowledge

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# Advantages of DOE

- Can simultaneously consider many factors (independent variables) and their interactions*
  - Identify significant factors and interactions within limits of experimental error
  - Find factor values to optimize a response (dependent variable)
  - Give direction for a specific response
  - Find best overall conditions for several responses

* Factor produces a different response when a second factor is changed.

# DOE Background

- Jacques Hadamard (1865 – 1963)
  - Mathematician
  - Discovered Independent Matrices

- Sir Ronald Fisher (1890 – 1962)
  - Statistician and Geneticist
  - Applied Hadamard's Matrices to Develop Multilevel Full Factorial Designs



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# Foundation of Experimental Designs

- All data can be modeled to a mathematical relationship (e.g. a line or a polynomial)
- DOE helps in choosing experimental factor values that provide optimal information per point.
- Ideally, factor response relationships are simple, but often higher degree polynomials must be used to obtain accurate approximations.

# Model Building

- Using DOE techniques, coefficients of the polynomial can be estimated.

$$R = b_0 + b_1x_1 + b_2x_2 + b_3x_1x_2 + \ldots$$

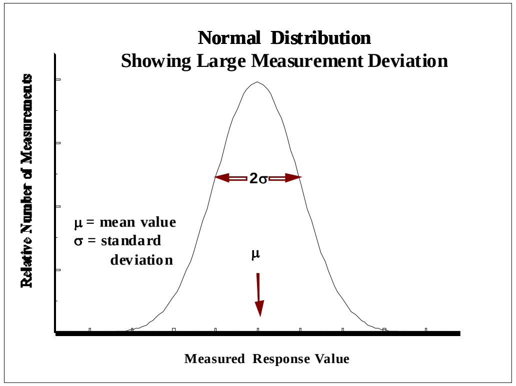
- The magnitude of the coefficients are used to determine which factors and/or interactions are important.
- Models are only accurate using factor space established in DOE.

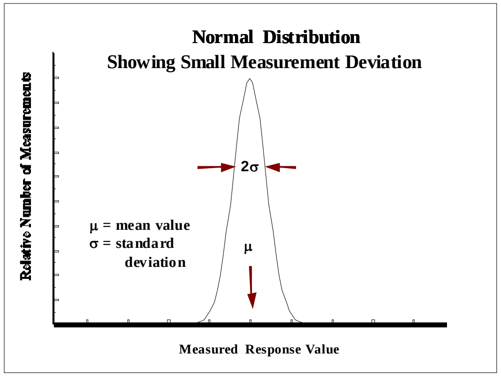
# Steps Involved in an Experimental Design

1. Do your homework.
2. Define problem statement (quantitatively).
3. Establish experimental objectives.
4. Select responses and expected ranges.
5. Select factors and levels.
6. Determine resources (use only 35% in first DOE)
7. Select design type and analysis strategy.
8. Randomize experimental runs.
9. Conduct experiments.
10. DOE Analysis.
11. Make conclusions and predictions.
12. Set up new DOE if needed.

# Role of Error

- Noise or error is always present in any experimental design.
- If large, error can prevent any meaningful conclusions (garbage in → garbage out)
- Random error must be minimized and systematic error eliminated as much as possible.
- If relative standard deviation (std dev/mean) is greater than about 8%, information will be difficult to obtain using DOE.



# Coded Factor Values

- Designs use coded values to represent factors

| Photoinitiator % | Real Space | Factor Space |
|---|---|---|
| Level 1 | 0.50% | -1 |
| Level 2 | 1.00% | 0 |
| Level 3 | 1.50% | 1 |

- In coded space, all factors have the same range, and thus the same magnitude.
- Coded space enables a better comparison of the relative importance of factors influencing the response.

# DOE Lingo

- Design (layout): Complete specification of experimental test runs, including blocking, randomization, repeat tests, replication, and the assignment of factor-level combinations to experimental units.
- Factor: A controllable experimental variable that is thought to influence the response.
- Level: Specific value of a factor (i.e. -1, 0 ,1 in coded space).
- Response: Outcome or result of an experiment.
- Experimental region (factor space): All possible factor-level combinations for which experimentation is possible.
- Interaction: Existence of joint factor effects in which the effect of each factor depends on the levels of the other factors.
- Confounding: One or more effects that cannot unambiguously be attributed to a single factor or interaction.
- Covariate: An uncontrollable variable that influences the response but is unaffected by any other experimental factors.
- Effect: Change in the average response between two factor-level combinations or between two experimental conditions.

# Photocuring Design

- Select response(s)
  - Conversion
  - Adhesion strength
- Select factors and ranges
  - Photoinitiator concentration
    - 0.5-1.5%
  - Temperature
    - 60-80°F
  - Oligomer concentration
    - 60-80 %
  - Belt speed (light dose)
  - Phase of the moon

| Run No. | Photoinitiator % | Temperature (°F) |
|---|---|---|
| 1 | 0.50% | 60 |
| 2 | 0.50% | 80 |
| 3 | 1.50% | 60 |
| 4 | 1.50% | 80 |

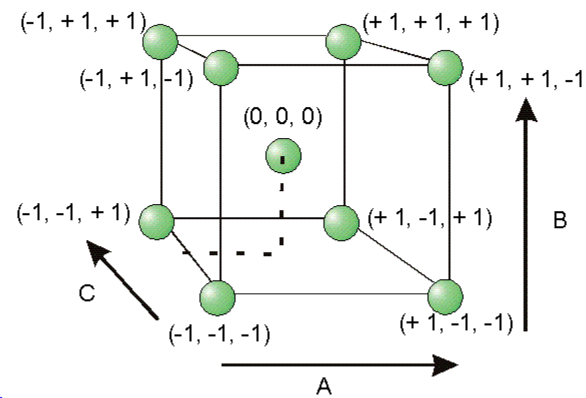
| Run No. | Photoinitiator % | Temperature (°F) |
|---|---|---|
| 1 | -1 | -1 |
| 2 | -1 | 1 |
| 3 | 1 | -1 |
| 4 | 1 | 1 |

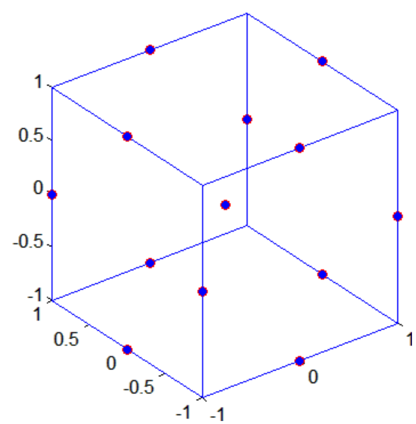
# General Design Types

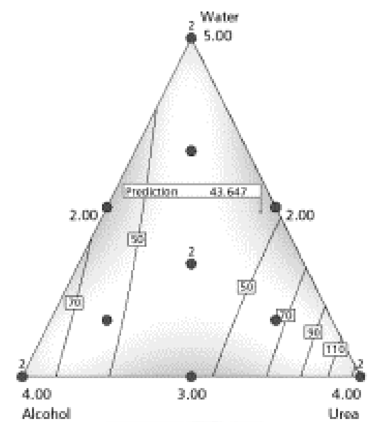
- Screening Designs - Used to identify major factors (and interactions) important in determining a response from many possible factors.
- Model Building Designs - Establish a function that estimates the effects of major factors and interactions on response.
- Simplex or Mixture Designs - Variable components always sum to constant value (e.g. 1).

# Common Designs

- Screening
  - Fractional Factorial
  - Plackett Burman
  - Taguchi
- Model Building
  - Full Factorial
  - Box Behnken
  - Central Composite
  - Equiradial
- Mixture Designs
  - Simplex Lattice
  - Simplex Centroid



# Screening Designs

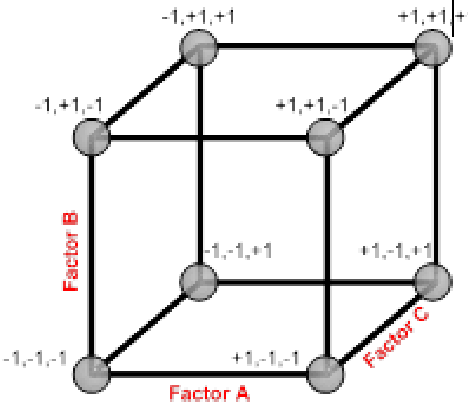
- Screening designs are intended to find the few significant factors from a list of many potential factors.
- The design is a typically a subset of a full factorial design.
- Use screening designs when you have many factors to consider Even when the experimental goal is to eventually fit a response surface model, the first experiment should be a screening design when there are many factors to consider.
- Interactions can be determined using some screening designs, but are typically confounded with other factors and/or interactions.
- Fractional factorial, Plackett-Burman, Taguchi

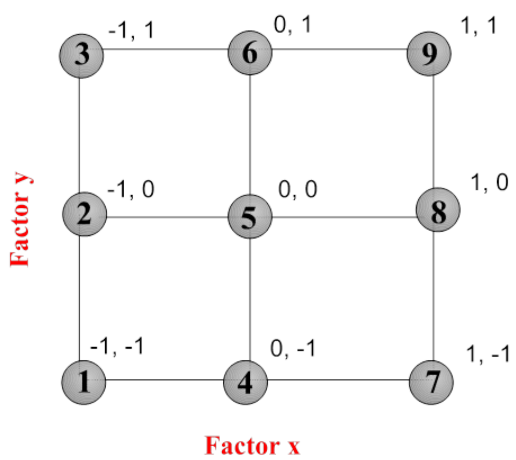
Plackett–Burman 8-Run Matrix

| Treatment Combinations | Factors A | B | C | D | E | F | G |
|---|---|---|---|---|---|---|---|
| 1 | + | - | - | + | - | + | + |
| 2 | + | + | - | - | + | - | + |
| 3 | + | + | + | - | - | + | - |
| 4 | - | + | + | + | - | - | + |
| 5 | + | - | + | + | + | - | - |
| 6 | - | + | - | + | + | + | - |
| 7 | - | - | + | - | + | + | + |
| 8 | - | - | - | - | - | - | - |

# Full Factorial

- Responses involve k factors.
- Each factor has L levels. Total test conditions = $L^k$
- No confounding of factors or interactions.
- Two level designs used to determine which of many factors are important.
- High level designs (L>2) are used to study a few important factors and develop response models.
- Maximum number of experiments.

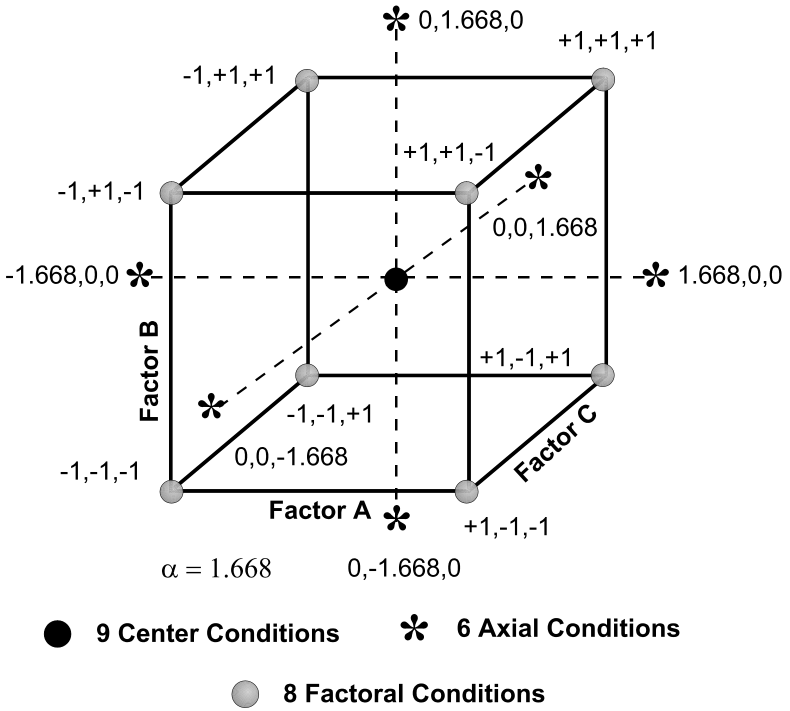


# Central Composite Design

- Sequential Design
  - 2 Level Factorial (Linear)
  - Center Points
  - Axial Points
- If linear model is not adequate, axial points are added to build non-linear model.

Central Composite Design for 3 Factors



# Equiradial Design

- All exterior points on circle or sphere.
  - Pentagonal
  - Hexagonal
  - Octagonal
  - Decagonal
  - Icosahedral

## Hexagonal Design

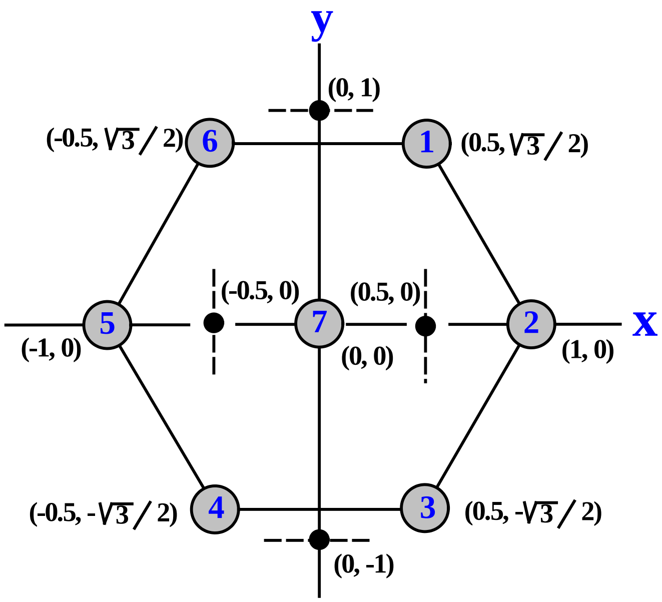



Figure shows x-y coded plane space.

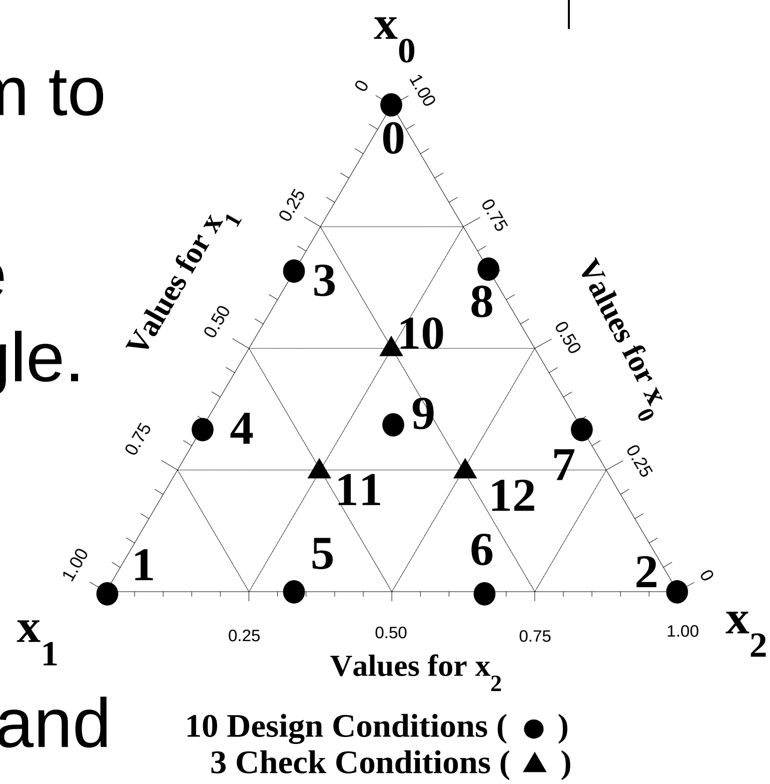
Numbers in gray circles are test conditions.

**Test Design Matrix in Coded Variable Space**

| Test Condition | x | y |
|---|---|---|
| 1 | 0.5 | $\sqrt{3}/2$ |
| 2 | 1 | 0 |
| 3 | 0.5 | $-\sqrt{3}/2$ |
| 4 | -0.5 | $-\sqrt{3}/2$ |
| 5 | -1 | 0 |
| 6 | -0.5 | $\sqrt{3}/2$ |
| 7 | 0 | 0 |
| 8 | 0 | 0 |
| 9 | 0 | 0 |
| 10 | 0 | 0 |
| 11 | 0 | 0 |
| 12 | 0 | 0 |

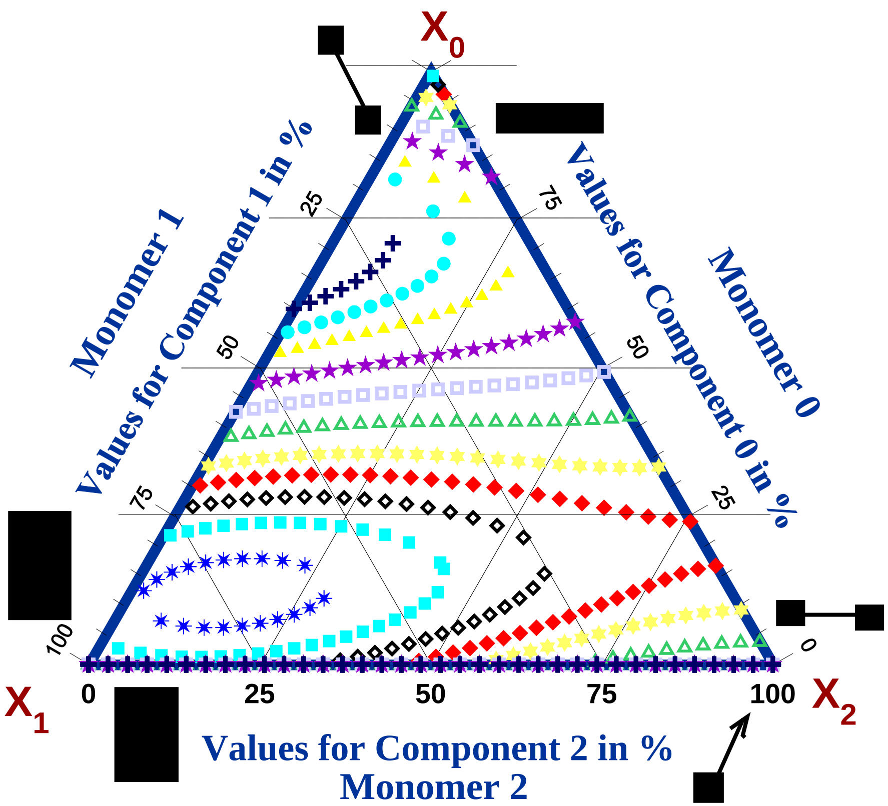
# Mixture Designs

- Components must always sum to same amount.
- Three component designs are shown on an equilateral triangle.
- Four components designs are shown on a tetrahedron.
- Cubic model for 3 and 4 components only requires 10 and 14 experiments respectively.



# Simplex Lattice Mixture Design

**TRIANGLE DIAGRAM**
**Coating Viscosity in poise Vs.**
**Polymer Thickener Composition**




------ Response-----

| Symbol | Value (poise) |
|---|---|
| ✚ | 1.40 |
| ● | 1.50 |
| ▲ | 1.60 |
| ★ | 1.75 |
| □ | 1.90 |
| △ | 2.05 |
| ✸ | 2.20 |
| ◆ | 2.30 |
| ◇ | 2.40 |
| ■ | 2.50 |
| ✳ | 2.60 |

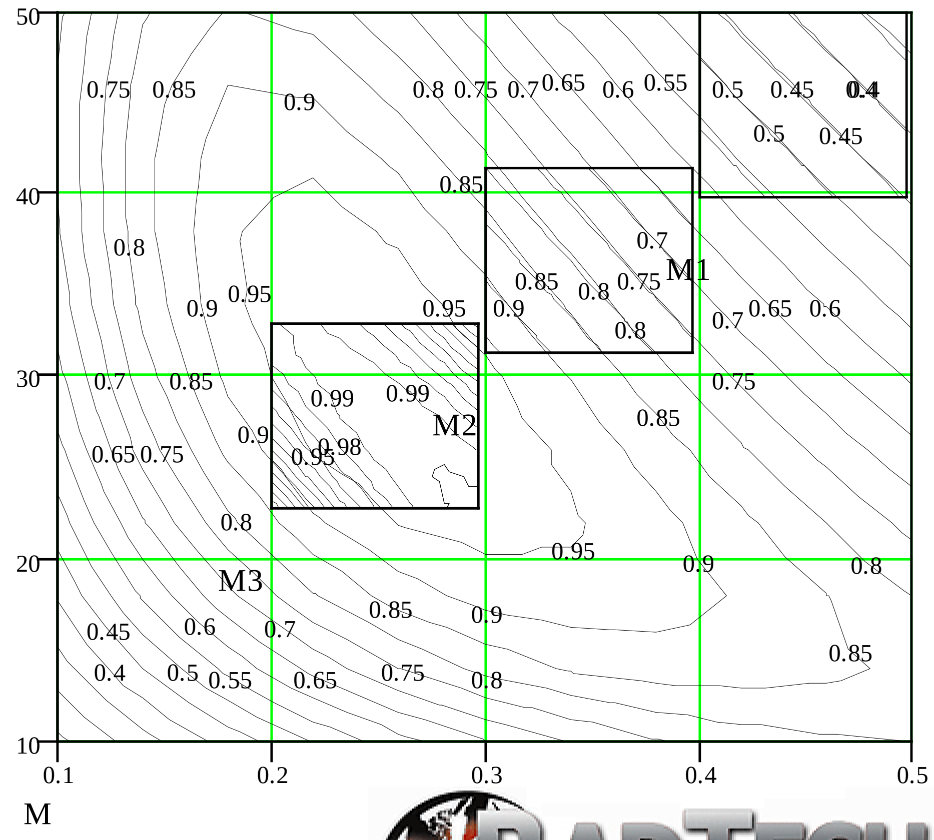
Shay, G. D., A. F. Rich, "Urethane-Functional Alkali-Soluble Associative Latex Thickners," J. Coat.Tech., 58, 43-53, 1986.

# Response Surface Analysis

- A response surface model can be used to optimize responses based on specified criteria.
- DOE software provides easily produced graphs and analysis.
- Software programs include:
  - Design Ease/Expert
  - Mini-tab
  - JMP and other statistics packages.

RESPONSE SURFACE CONTOUR PLOT

R = f ( x, y )



# Summary

- Design of Experiments is a valuable tool to maximize the amount of information with a minimal number of experiments.
- While numerous DOE frameworks exist, all operate using similar principles.
- Use sequential series of experiments
  - Screening Design
  - Model Building Design
  - Confirmation Runs

# Short Course at Radtech 2016

- Design of Experiments for UV/EB Formulation Scientists
- Monday, May 16th 6:30 – 9:00 pm
- Tuesday, May 17th noon – 2:00 pm
- Enrollment capped at 30 attendees
- DOE designs, analysis, and background will covered in much greater detail
- Demonstration of software
- Hands on activities (and prizes!).
- Register at [www.radtech.org](http://www.radtech.org)

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RADTECH UV+EB 2016 TECHNOLOGY EXPO AND CONFERENCE May 16-18, 2016 Hyatt Regency, O'Hare - Chicago, Illinois

The world's largest event dedicated to the educational, technical, and scientific advancement of ultraviolet (UV) and electron beam (EB) technologies.

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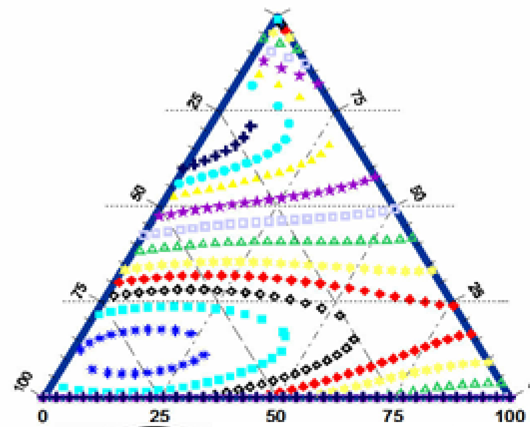
# Design of Experiments – A Two Day In House Short Course (with Roger Hester)

- In-depth instruction regarding background, motivation, and details of experimental design
- Attendees set up a DOE which will be evaluated by the instructors
- Three hands-on DOE labs
- Software demonstration and training
- Statistics tutorial
- 1.5 CEU credits from University of Iowa

Comments from previous attendees include:

- *The instructors did a very good job in presenting the material.*
- *I would recommend this course as essential for any DOE user.*
- *Very engaging instructors; held my interest throughout.*
- *As exciting as DOE can be!*
- *The most useful DOE course I have taken (this is my third or fourth).*

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