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# Design of experiments

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## Session 1

Introduction

- Why experimental design

Factorial design

- Design matrix
- Model equation = coefficients
- Residual
- Response contour

# Session 2

Factorial design

- Research problem
- Design matrix
- Model equation = coefficients
- Degrees of freedom
- Predicted response
- Residual
- ANOVA
- $R^2$
- Response contour



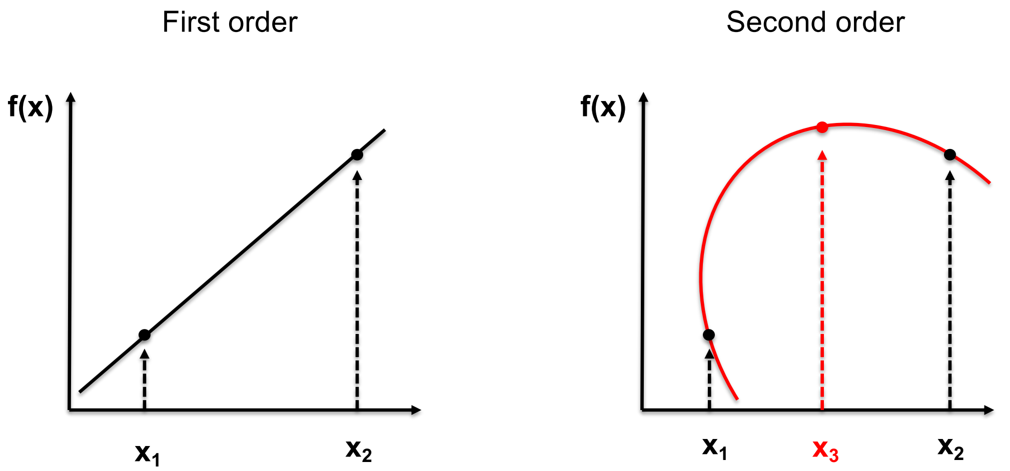
# Session 3

Composite designs

Second order models

Stationary points

# Higher order models

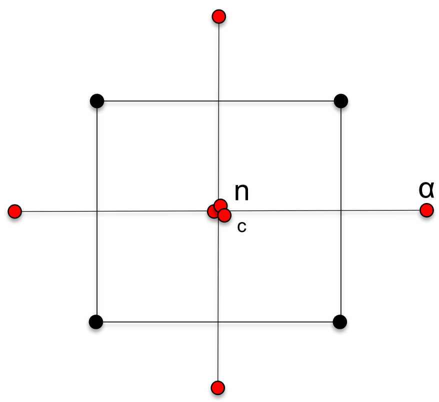





# Optimization designs

Second order models through

- Center points
- Axial points
- Different designs



# Central composite designs

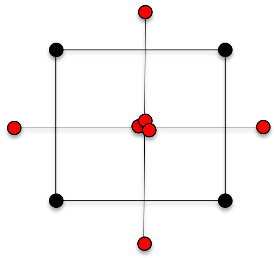
Center points

- Pure error (lack of fit)
- Curvature

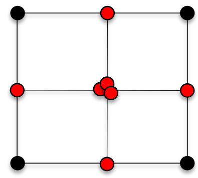
Axial points

- Quadratic terms

Spherical design
α > 1



Cuboidal design
α = 1





# Some alternatives

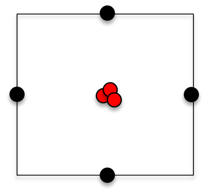
Center points

- Pure error (lack of fit)
- Curvature

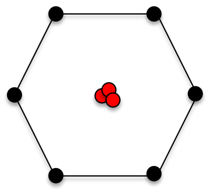
Axial points

- Quadratic terms

Box Behnken
design



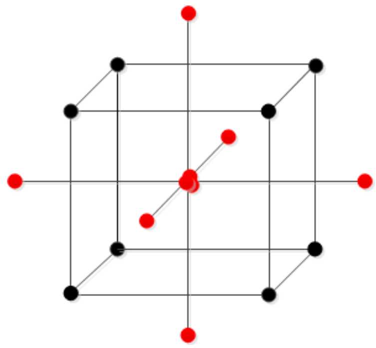
Doehlert design



# Optimization designs

Things to consider when choosing a design

- Pure error (lack of fit)
- Estimated error distribution
- Area of operability
- Control over factor levels





# Models

Factorial designs

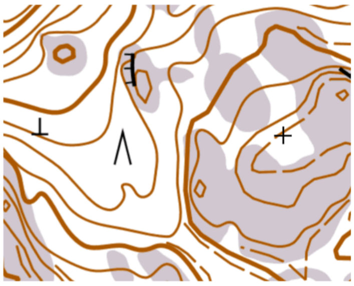
- Main effects
- Main effects + interactions

Optimization designs

- Main effects + interactions + quadratic terms
- $y = \beta_0 + \beta_i x_i + \beta_j x_j + \beta_{ij} x_i x_j + \ldots + \beta_{jj} x_j^2 + \varepsilon$

# Models

Complicated vs. simple function?



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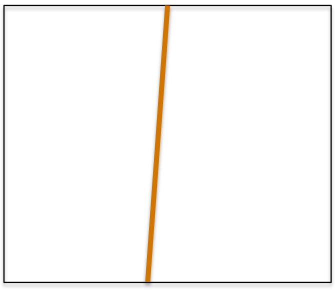
vs.



→ Start far enough to create variation, but close enough to use a simple function



# Design matrix

With two factors

Factorial: rows 1–4; Axial: rows 5–8; Center points: rows 9–11

| N:o | $x_1$ | $x_2$ | $x_{12}$ | $x_{11}$ | $x_{22}$ |
|---|---|---|---|---|---|
| 1 | -1 | -1 | 1 | | 1 |
| 2 | 1 | -1 | -1 | | 1 |
| 3 | -1 | 1 | -1 | | 1 |
| 4 | 1 | 1 | 1 | | 1 |
| 5 | -α | 0 | 0 | | 0 |
| 6 | α | 0 | 0 | | 0 |
| 7 | 0 | -α | 0 | | $\alpha^2$ |
| 8 | 0 | α | 0 | | $\alpha^2$ |
| 9 | 0 | 0 | 0 | | 0 |
| 10 | 0 | 0 | 0 | | 0 |
| 11 | 0 | 0 | 0 | | 0 |

# Research problem

A central composite design was performed for a tire tread compound and tire abrasion index was measured as a response

- Two factors $x_1$ and $x_2$
- Axial distance 1.633
- N:o of center points 4

| Variable | Variable levels | | | | |
|---|---|---|---|---|---|
| $x_1$ | -1.633 | -1 | 0 | 1 | 1.633 |
| $x_2$ | -1.633 | -1 | 0 | 1 | 1.633 |

Myers et al., Response Surface Methodology, 3rd ed., 2009, 275.

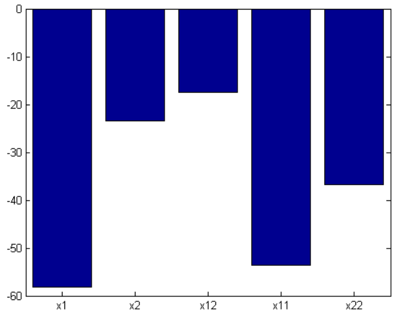


# Design matrix

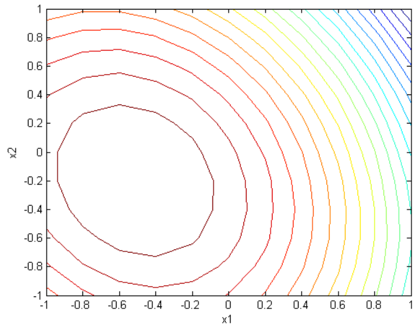
| | N:o | $x_1$ | $x_2$ | $x_{12}$ | $x_{11}$ | $x_{22}$ | y |
|---|---|---|---|---|---|---|---|
| Factorial | 1 | -1 | -1 | 1 | 1 | 1 | 270 |
| | 2 | 1 | -1 | -1 | 1 | 1 | 270 |
| | 3 | -1 | 1 | -1 | 1 | 1 | 310 |
| | 4 | 1 | 1 | 1 | 1 | 1 | 240 |
| Axial | 5 | -1.633 | 0 | 0 | 2.667 | 0 | 550 |
| | 6 | 1.633 | 0 | 0 | 2.667 | 0 | 260 |
| | 7 | 0 | -1.633 | 0 | 0 | 2.667 | 520 |
| | 8 | 0 | 1.633 | 0 | 0 | 2.667 | 380 |
| Center points | 9 | 0 | 0 | 0 | 0 | 0 | 520 |
| | 10 | 0 | 0 | 0 | 0 | 0 | 290 |
| | 11 | 0 | 0 | 0 | 0 | 0 | 580 |
| | 12 | 0 | 0 | 0 | 0 | 0 | 590 |

# Model

Unrefined coefficients



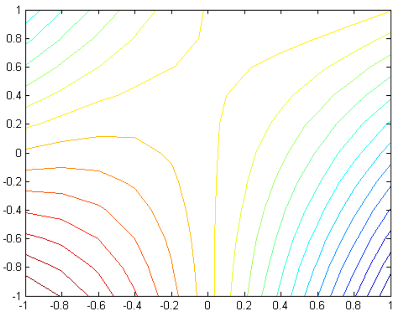

Contour



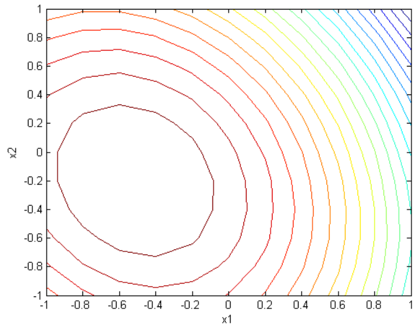



# Quadratic models

Can show stationary points



Saddle point




Maximum/minimum

# Quadratic models

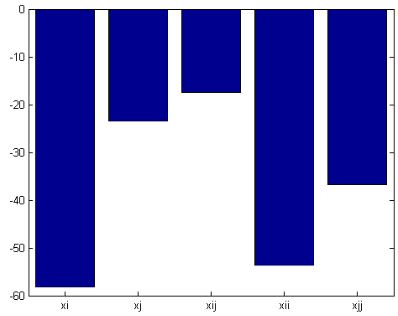
Stationary point can be described

Quadratic regression equation

$$y = \beta_0 + \beta_i x_i + \beta_j x_j + \beta_{ij} x_i x_j + \ldots + \beta_{jj} x_j^2 + \varepsilon$$

Derivation $\frac{\partial \hat{y}}{\partial x_i} = \frac{\partial \hat{y}}{\partial x_j} = 0$ results in

$$\left.\begin{aligned} \beta_i + \beta_{ij} x_j + 2\beta_{ii} x_i = 0 \\ \beta_j + \beta_{ij} x_i + 2\beta_{jj} x_j = 0 \end{aligned}\right\} \quad [\ x_i \quad x_j\ ]$$






# Quadratic models

For determining a stationary point

$\hat{y} = b_0 + \mathbf{x'b} + \mathbf{x'Bx}$ where

$$\mathbf{x'} = [\ x_1 \quad x_2 \quad \cdots \quad x_k\ ],\ \mathbf{b} = \begin{bmatrix} b_1 \\ b_2 \\ \vdots \\ b_k \end{bmatrix},\ \text{and } \mathbf{B} = \begin{bmatrix} b_{11} & b_{12}/2 & \cdots & b_{1k}/2 \\ & b_{22} & \cdots & b_{2k}/2 \\ & & \ddots & \vdots \\ sym. & & & b_{kk} \end{bmatrix}$$

→ location and character

# Quadratic models

Stationary point location

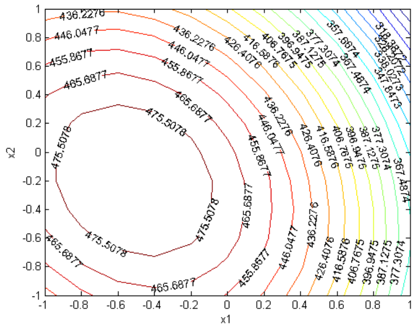
$$\frac{\partial \hat{y}}{\partial \mathbf{x}} = \mathbf{b} + 2\mathbf{B}\mathbf{x} = 0$$

$$\mathbf{x}_s = -\frac{1}{2}\mathbf{B}^{-1}\mathbf{b}$$

From the previous example

$$\mathbf{x}_s = \begin{bmatrix} x_1 & x_2 \end{bmatrix} = \begin{bmatrix} -0.5 & -0.2 \end{bmatrix}$$

$$\hat{y}_s = 485.8$$





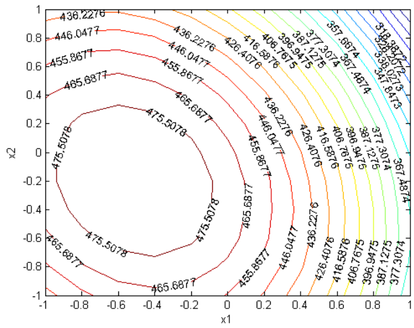

# Quadratic models

Stationary point character

$$\mathbf{B} = \begin{bmatrix} b_{11} & b_{12}/2 & \cdots & b_{1k}/2 \\ & b_{22} & \cdots & b_{2k}/2 \\ & & \ddots & \vdots \\ sym. & & & b_{kk} \end{bmatrix}$$

Eigenvalues ($\lambda_1, \lambda_2, \ldots, \lambda_k$)

- all < 0 → maximum
- all > 0 → minimum
- mixed in sign → saddle point




$$\begin{cases} \lambda_1 = -57.3 \\ \lambda_2 = -33.0 \end{cases}$$

# Research problem

A researcher wanted to maximize the yield of carbohydrates during an extraction process. She performed a total of 17 experiments based on a central composite design ($\alpha$ = 1) with three variables and three center points. Determine a regression model and use it to optimize yield.

| Variable | min | max |
|---|---|---|
| Temp | 150 | 190 |
| Time | 30 | 180 |
| L/S ratio | 4 | 6 |



# Session 3

Composite designs

Second order models

Stationary points

# Nomenclature

Center point

Axial point / star point

Stationary point

Saddle point

Minimum

Maximum