NREL
NATIONAL RENEWABLE ENERGY LABORATORY

Introduction to Statistically Designed Experiments

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Objectives

- Give some basic background information on statistically designed experiments
- Demonstrate the advantages of using statistically designed experiments[1]

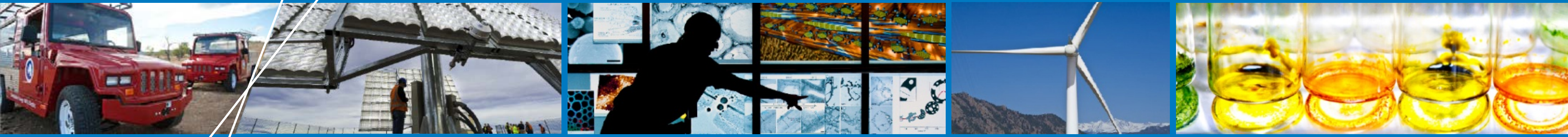
[1] Often referred to as design of experiments (DOE or DOX), or experimental design.

A Few Thoughts

- Many companies/organizations do experiments for
  - ✓ Process improvement
  - ✓ Product development
  - ✓ Marketing strategies
- We need to plan our experiments as efficiently and effectively as possible
- Statistically designed experiments have a proven track record (Mullin 2013 in *Chemical & Engineering News*)
  - ✓ Conclusions supported by statistical tests
  - ✓ Substantial reduction in total experiments
- *Why are we still planning experiments like it's the 19th century?*

Outline

- Research Problems
- The Linear Model
- Key Types of Designs
    - Full Factorial
    - Fractional Factorial
    - Response Surface
- Sequential Approach
- Summary



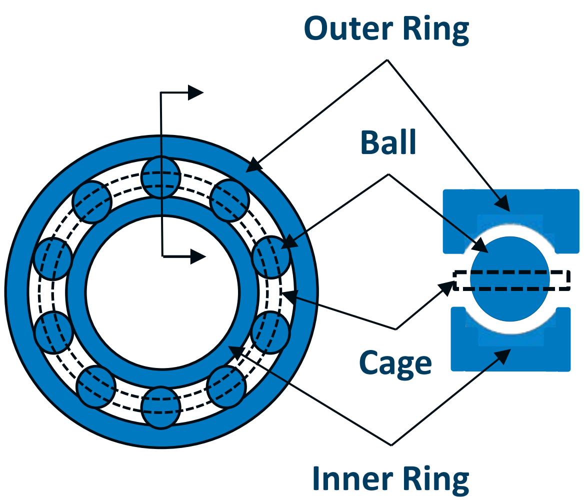
Research Problems

Research Problems

Is a dependent variable (Y) affected by multiple independent variables (Xs)?

Objective: Increase bearing lifetime (Hellstrand 1989)

Y	Bearing lifetime (hours)



X_1	Inner ring heat treatment
X_2	Outer ring osculation (ratio between ball diameter and outer ring raceway radius)
X_3	Cage design

Research Problems

Objective: Hydrogen cyanide (HCN) removal in diesel exhaust gas (Zhao et al. 2006)

Y	HCN conversion

X_1	Propene (C_3H_6) conc. (ppm)
X_2	Nitric oxide (NO) conc. (ppm)
X_3	Temperature (°C)
X_4	Gas hourly space velocity (h^{-1})

$$HCN + NO + O_2 \xrightarrow[\text{Pt catalyst}]{} N_2 + N_2O + NO_2 + CO + H_2O + CO_2$$

Research Problems

Objective: For prototype power sensor, evaluate how appliances being "ON" or "OFF" affects the sensor measurement bias

Y	Sensor bias

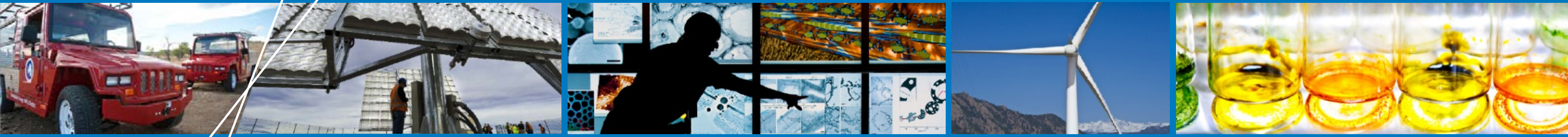
Photo by Dennis Schroeder, NREL 35602

Less expensive monitoring of home energy consumption

X_1	Refrigerator
X_2	Dishwasher
X_3	Clothes washer
X_4	Lighting incandescent
X_5	Lighting LEDs
X_6	Lighting CFLs
X_7	Television
X_8	Range

All combinations: $2^8 = 256$ experiments

Fractional factorial: <u>16 experiments</u>



The Linear Model

The Linear Model

“All models are wrong but some are useful” – George Box

Linear model used as an approximation for statistical testing

$$Y = \underbrace{\boxed{\beta_0}}_{\text{Intercept}} + \underbrace{\boxed{\beta_1 X_1 + \beta_2 X_2}}_{\text{Main effects}} + \underbrace{\boxed{\beta_3 X_1 X_2}}_{\text{Interaction}} + \underbrace{\boxed{\beta_4 X_1^2 + \beta_5 X_2^2}}_{\text{Polynomial terms to test curvature}} + \underbrace{\boxed{\varepsilon}}_{\text{Error}}$$

Which terms are included in a model depends on the experimental design

The Linear Model

Fictitious Case Study

Zaphod Beeblebrox decides to put his two heads to good use.

Insul-spray – a clear spray on coating for buildings that provides additional thermal resistance (TR)

Objective: TR value of at least 40 (°F ft^2 hr)/Btu

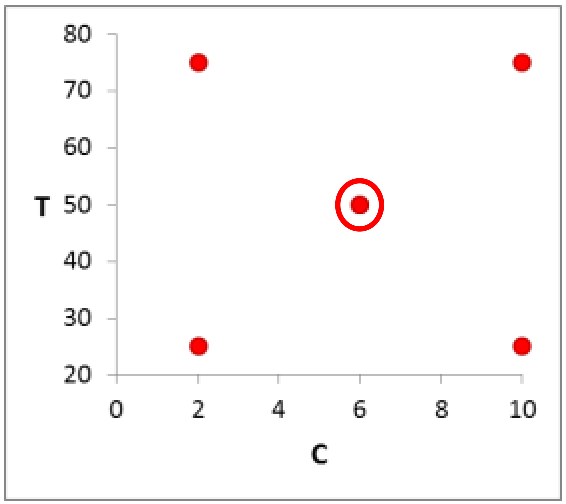
TR	**Thermal resistance**

C	**Special additive conc. (ppm)**
T	**Temperature (°C)**

Photo by Warren Gretz, NREL 00956

The Linear Model

Experimental Design



Experimental Plan and Results

Trial	Actual C	Actual T	Coded C	Coded T	TR
1	2	25	-1	-1	8.3
2	10	25	1	-1	44.7
3	2	75	-1	1	0.5
4	10	75	1	1	27.2
5	6	50	0	0	16.0
6	6	50	0	0	17.5

Trial run order should be randomized

$$TR = \beta_0 + \boxed{\beta_C C + \beta_T T} + \boxed{\beta_{CT} CT} + \boxed{\beta_{CC} C^2} + \varepsilon$$

Main effects

Interaction

Polynomial term to test curvature
Caution: C^2 confounded with T^2

The Linear Model

Statistical hypotheses tests for multiple variables

MLR on coded C and T

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	16.8	0.8	22.3	0.028
C	15.8	0.5	29.8	0.021
T	-6.3	0.5	-11.9	0.053
C^2	3.4	0.9	3.7	0.167
C*T	-2.4	0.5	-4.6	0.137

Residual standard error: 1.06 on 1 degrees of freedom

Multiple R-squared: 0.999, Adjusted R-squared: 0.995

F-statistic: 265 on 4 and 1 DF, p-value: 0.046

Reduced model

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	19.0	1.5	12.7	0.001
C	15.8	1.8	8.6	0.003
T	-6.3	1.8	-3.5	0.041

Residual standard error: 3.66 on 3 degrees of freedom

Multiple R-squared: 0.966, Adjusted R-squared: 0.944

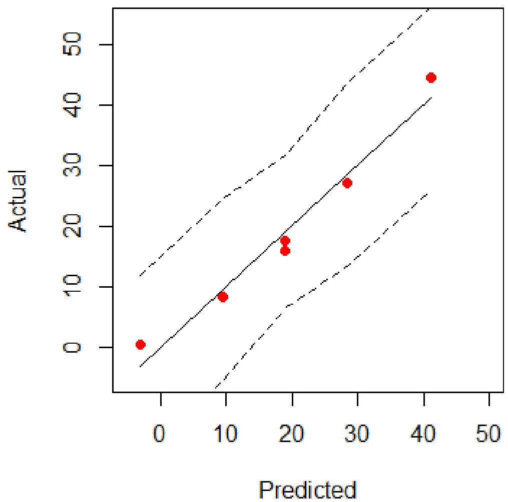
F-statistic: 43 on 2 and 3 DF, p-value: 0.00618

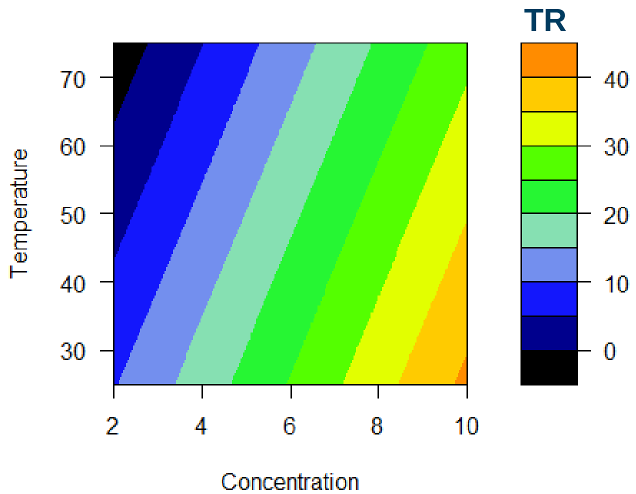
ANOVA can also be applied

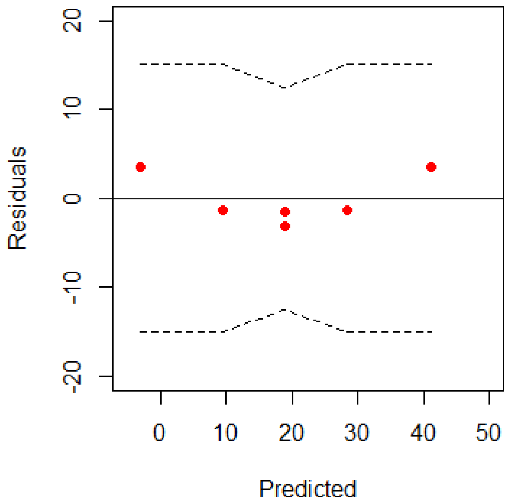
Conclusion:

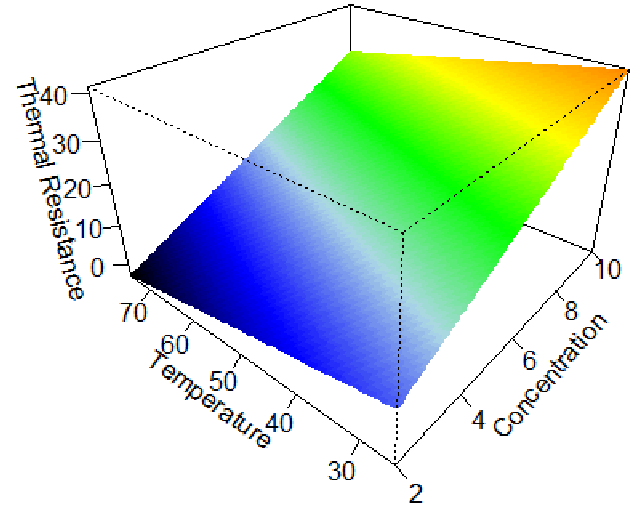
C and T significantly affect TR

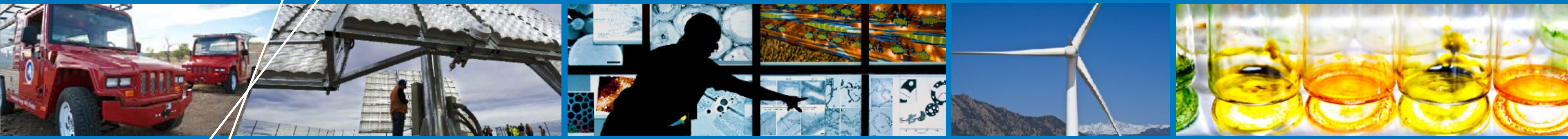
Additional diagnostics and plots









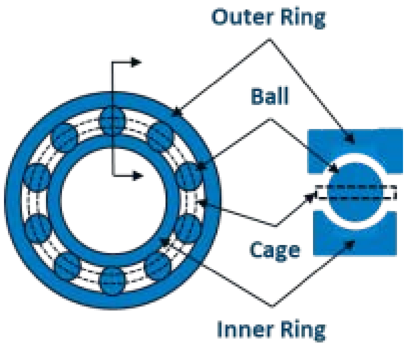


Types of Designs

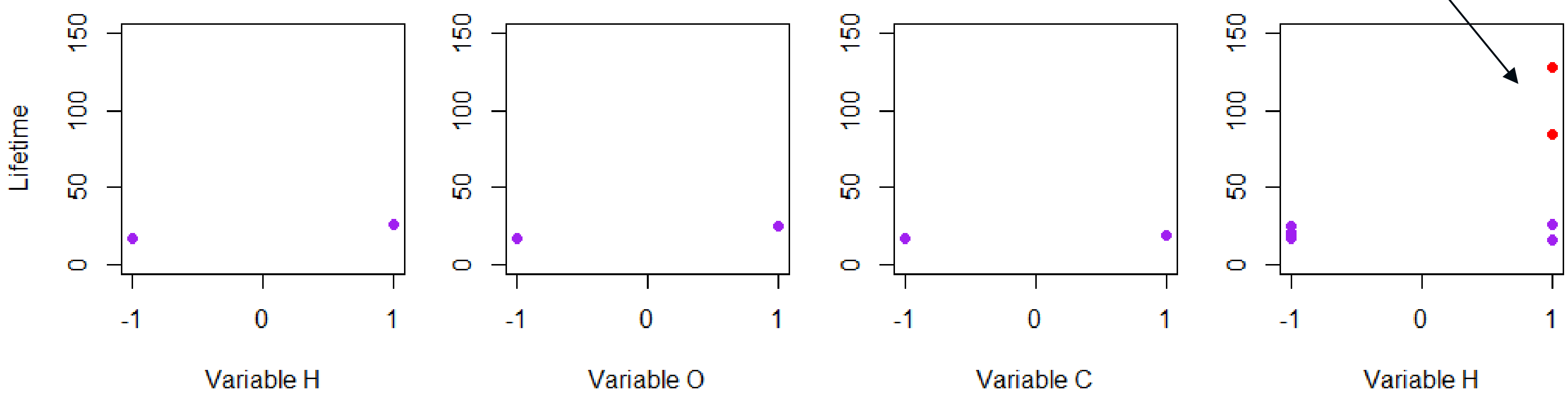
Full Factorial

Objective: Increase bearing lifetime (Hellstrand 1989)

H	**Inner ring heat treatment**
O	**Outer ring osculation**
C	**Cage design**



H and O Modified



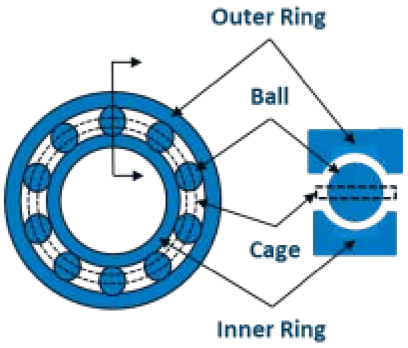
-1 = Standard
1 = Modified

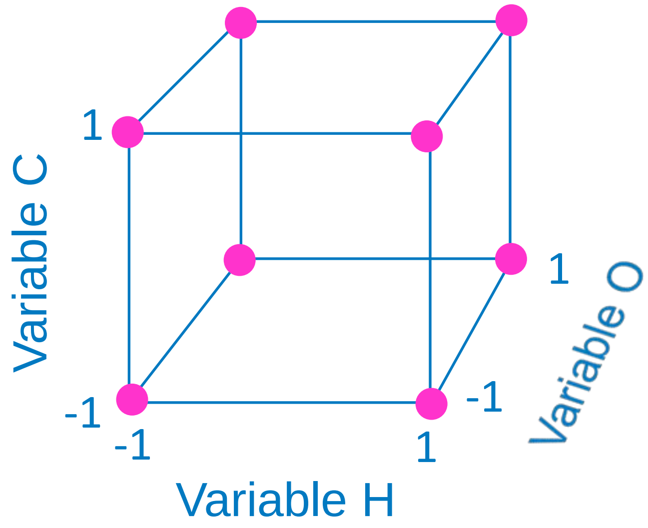
This is an interaction!

Full Factorial

Objective: Increase bearing lifetime (Hellstrand 1989)

H	**Inner ring heat treatment**
O	**Outer ring osculation**
C	**Cage design**





Trial	H	O	C	LT
1	-1	-1	-1	17
2	1	-1	-1	26
3	-1	1	-1	25
4	**1**	**1**	-1	**85**
5	-1	-1	1	19
6	1	-1	1	16
7	-1	1	1	21
8	**1**	**1**	1	**128**

-1 = Standard
1 = Modified

For 2 level designs:
(trial conditions) = 2^k
where k = number of variables

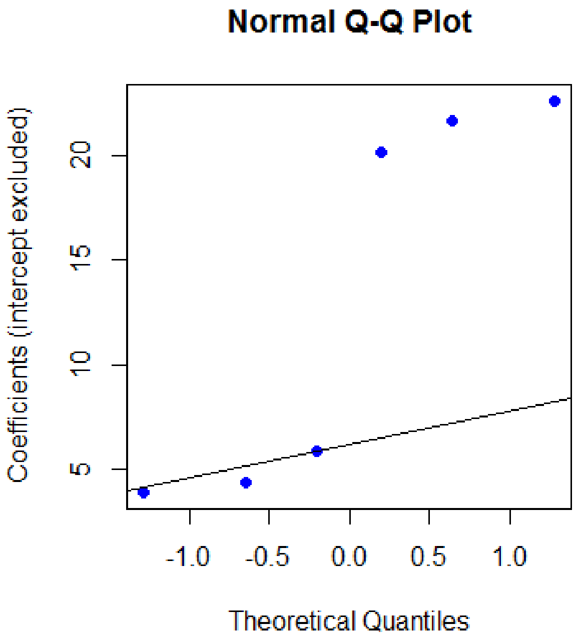
Full Factorial

MLR

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	42.1	7.4	5.7	0.110
H	**21.6**	**7.4**	**2.9**	**0.210**
O	**22.6**	**7.4**	**3.1**	**0.200**
C	3.9	7.4	0.5	0.690
H*O	**20.1**	**7.4**	**2.7**	**0.220**
H*C	4.4	7.4	0.6	0.660
O*C	5.9	7.4	0.8	0.570

Reduced model

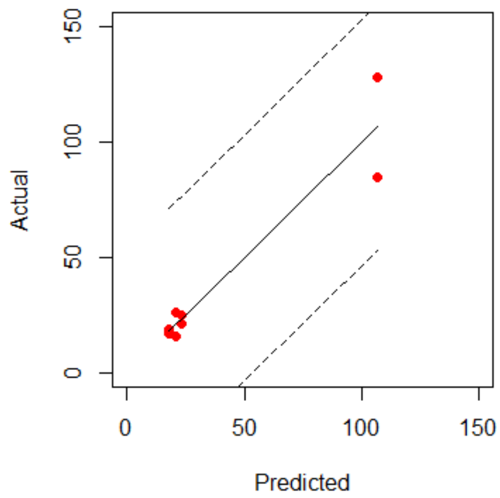
Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	42.1	5.6	7.6	0.002
H	**21.6**	**5.6**	**3.9**	**0.018**
O	**22.6**	**5.6**	**4.1**	**0.015**
H*O	**20.1**	**5.6**	**3.6**	**0.022**

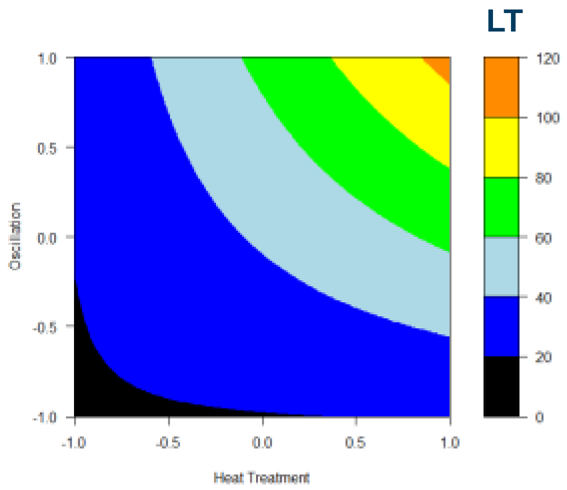


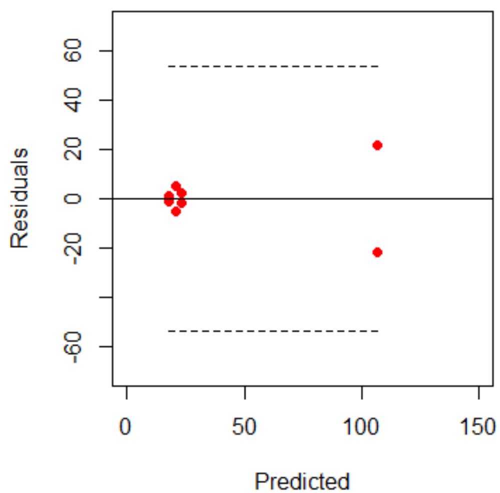
Conclusion:
H, O, and H*O significantly affect LT

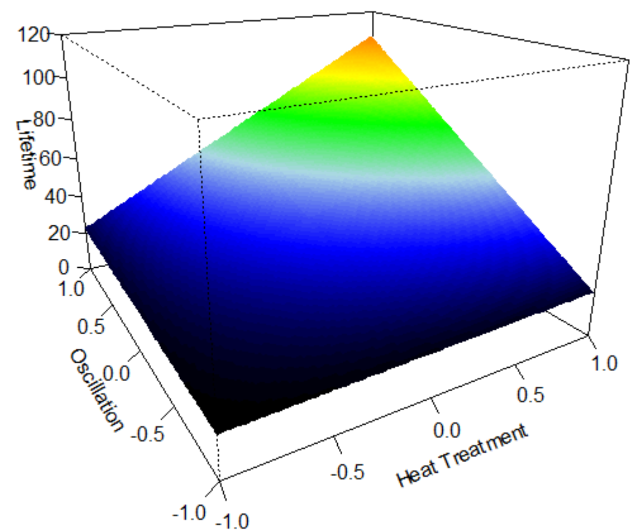
Full Factorial

Plots for bearing lifetime analysis



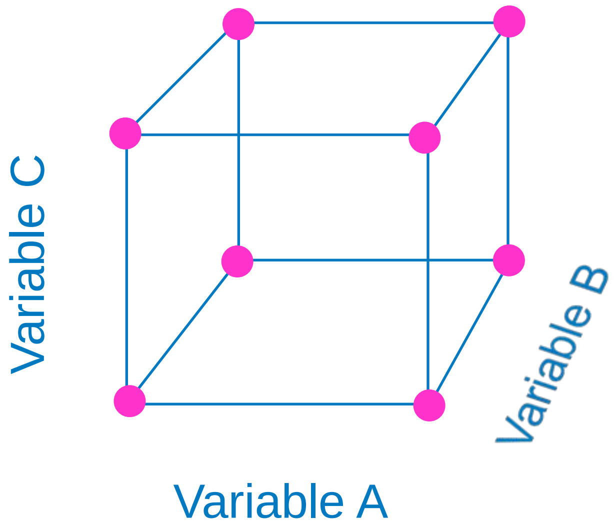




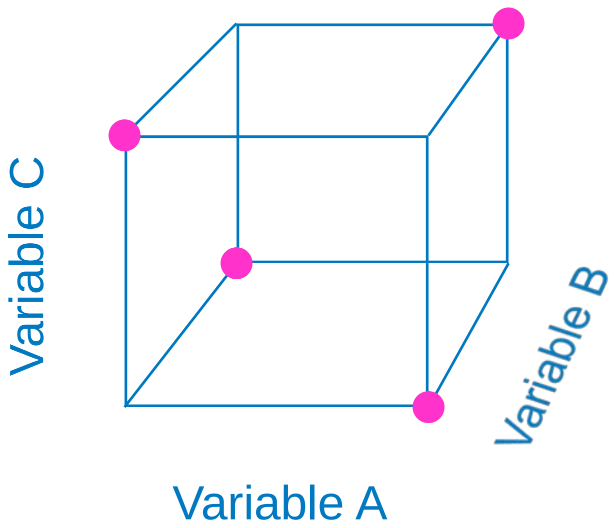


Fractional Factorial

Full Factorial
8 trial conditions



Fractional Factorial
4 trial conditions



Center point condition often added to test for curvature

Fractional Factorial

Objective: Hydrogen cyanide (HCN) removal in diesel exhaust gas

(Zhao et al. 2006)

$$HCN + NO + O_2 \xrightarrow[\text{Pt catalyst}]{} N_2 + N_2O + NO_2 + CO + H_2O + CO_2$$

P	Propene (C_3H_6) conc. (ppm)
NO	Nitric oxide (NO) conc. (ppm)
T	Temperature (°C)
G	Gas hourly space velocity (h^{-1})

	Actual				Coded				
Trial	Propene	Nitric Oxide	Temp	GHSV	P	NO	T	G	HCN conversion
1	90	13	165	30300	-1	-1	-1	-1	0.585
2	504	13	165	87870	1	-1	-1	1	0.159
3	90	52	165	87870	-1	1	-1	1	0.204
4	504	52	165	30300	1	1	-1	-1	0.400
5	90	13	277	87870	-1	-1	1	1	0.857
6	504	13	277	30300	1	-1	1	-1	0.951
7	90	52	277	30300	-1	1	1	-1	0.950
8	504	52	277	87870	1	1	1	1	0.840

Full Factorial: 2^4 = 16 experiments

Fractional factorial: 8 experiments

Fractional Factorial

MLR

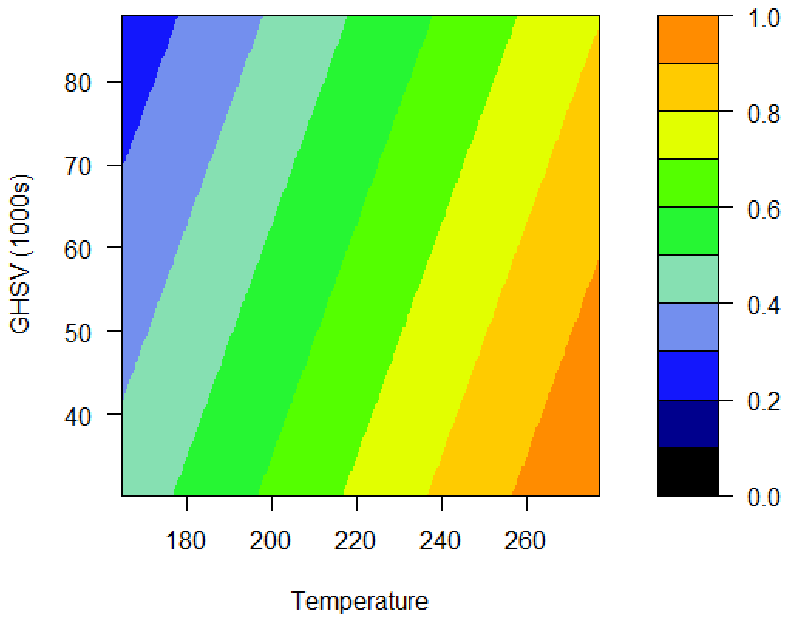
Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	0.618	0.022	28.4	0.001
P	-0.031	0.022	-1.4	0.293
NO	-0.020	0.022	-0.9	0.460
T	**0.281**	**0.022**	**12.9**	**0.006**
G	**-0.103**	**0.022**	**-4.7**	**0.042**
T*G	0.052	0.022	2.4	0.139

Reduced model

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	0.618	0.032	19.5	0.000
T	**0.281**	**0.032**	**8.9**	**0.000**
G	**-0.103**	**0.032**	**-3.3**	**0.022**

Conclusion:

T and G significantly affect HCN conversion



Response Surface

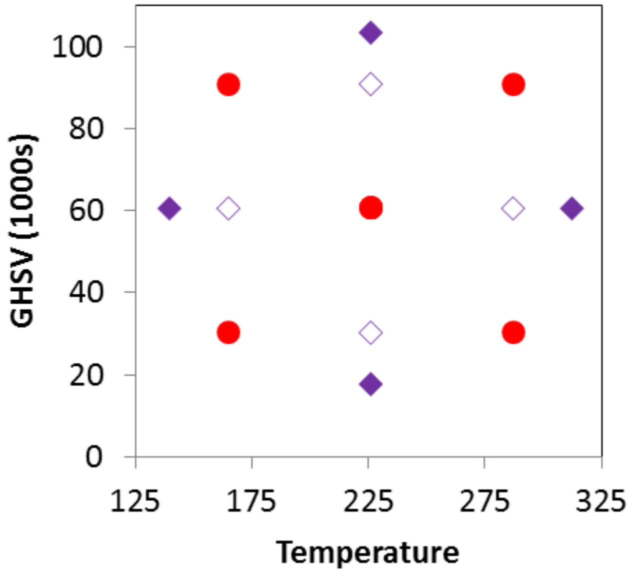
Objective: Hydrogen cyanide (HCN) removal in diesel exhaust gas

(Zhao et al. 2006)

$$HCN + NO + O_2 \xrightarrow[\text{Pt catalyst}]{} N_2 + N_2O + NO_2 + CO + H_2O + CO_2$$

T	Temperature (°C)
G	Gas hourly space velocity (h^{-1})

Experimental Design



	Actual		Coded		
Trial	Temp	GHSV	T	G	HCN conversion
1	165	30300	-1	-1	0.452
2	287	30300	1	-1	0.949
3	165	90900	-1	1	0.175
4	287	90900	1	1	0.855
5	165	60600	-1	0	0.235
6	287	60600	1	0	0.894
7	226	30300	0	-1	0.934
8	226	90900	0	1	0.663
9	226	60600	0	0	0.782
10	226	60600	0	0	0.785
11	226	60600	0	0	0.784

Propene = 250 ppm

Nitric Oxide = 30 ppm

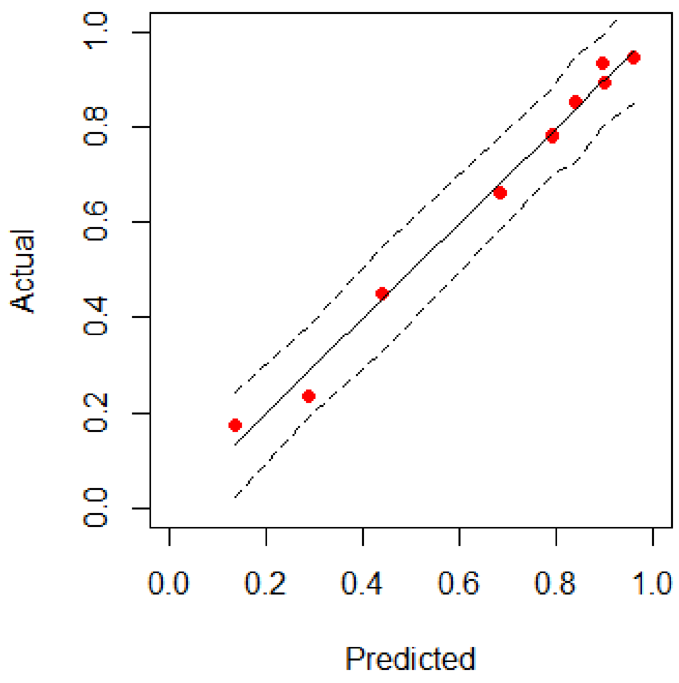
Response Surface

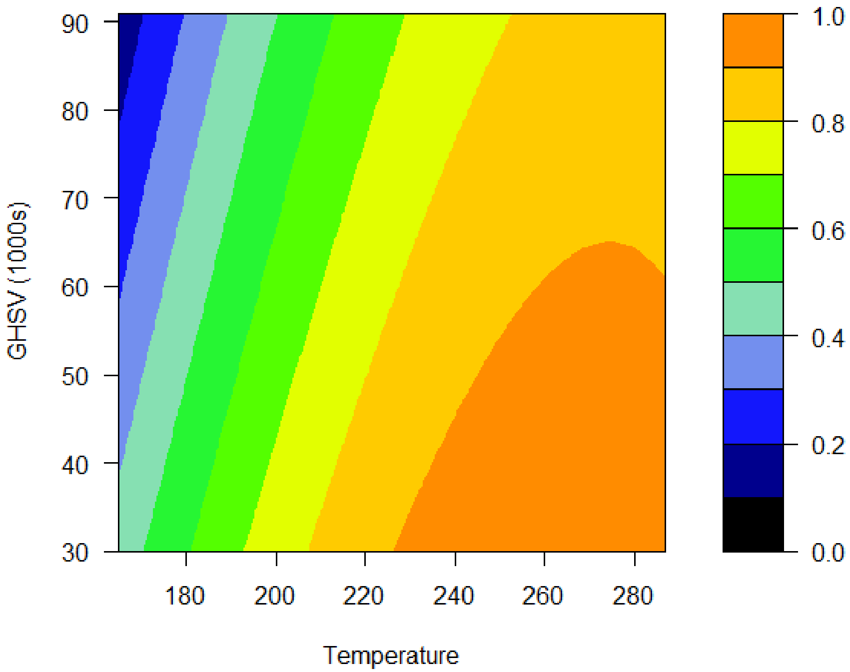
MLR Reduced model

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	0.790	0.015	52.2	0.000
T	**0.306**	**0.014**	**22.2**	**0.000**
G	**-0.107**	**0.014**	**-7.8**	**0.000**
T^2	**-0.196**	**0.021**	**-9.6**	**0.000**
T*G	**0.046**	**0.017**	**2.7**	**0.035**

Conclusion:

T, G, T*G and T^2 significantly affect HCN conversion





Sequential Approach

Sequential Approach

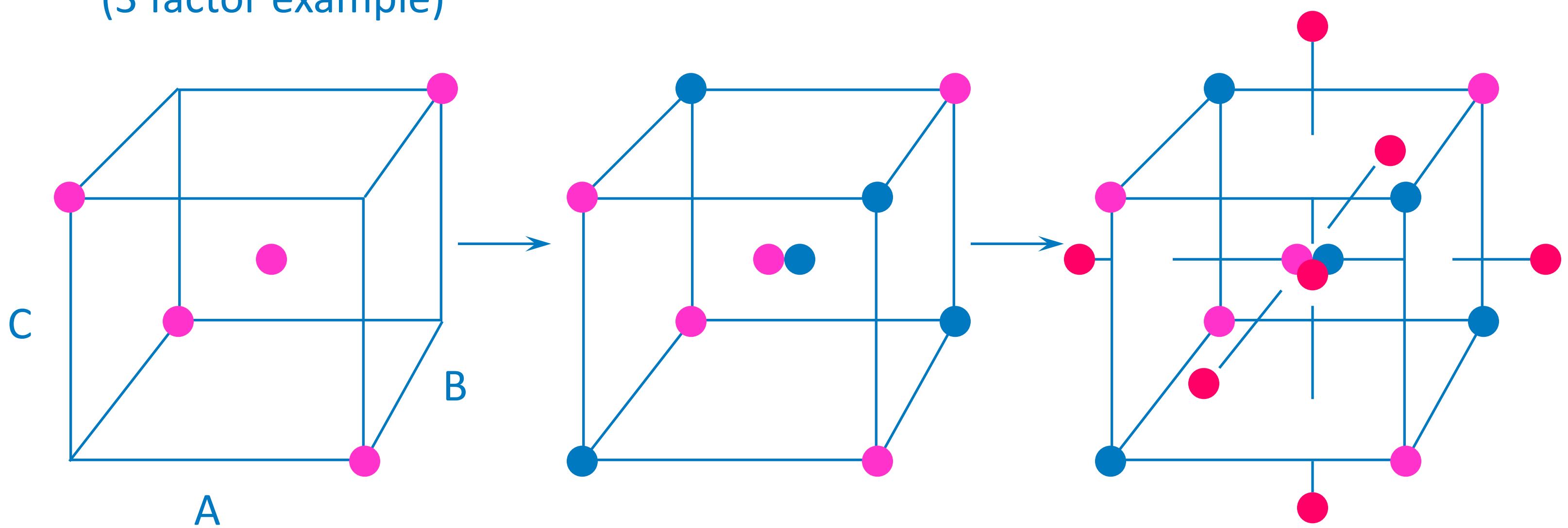
General concepts of sequential approach
(Box and Bisgaard 1997)

- Basic idea is to break up overall experimental plan into a few complementary experimental plans
- Start simple (fractional factorials) - 25% rule (25-40% of allotted time/effort in first designed experiment)
- Modify additional experiments based on what is learned from prior experiments
- **BE FLEXIBLE**!!!

General concepts of sequential approach

(Box and Bisgaard 1997)

First DOE (3 factor example) | **Second DOE** | **Third DOE**



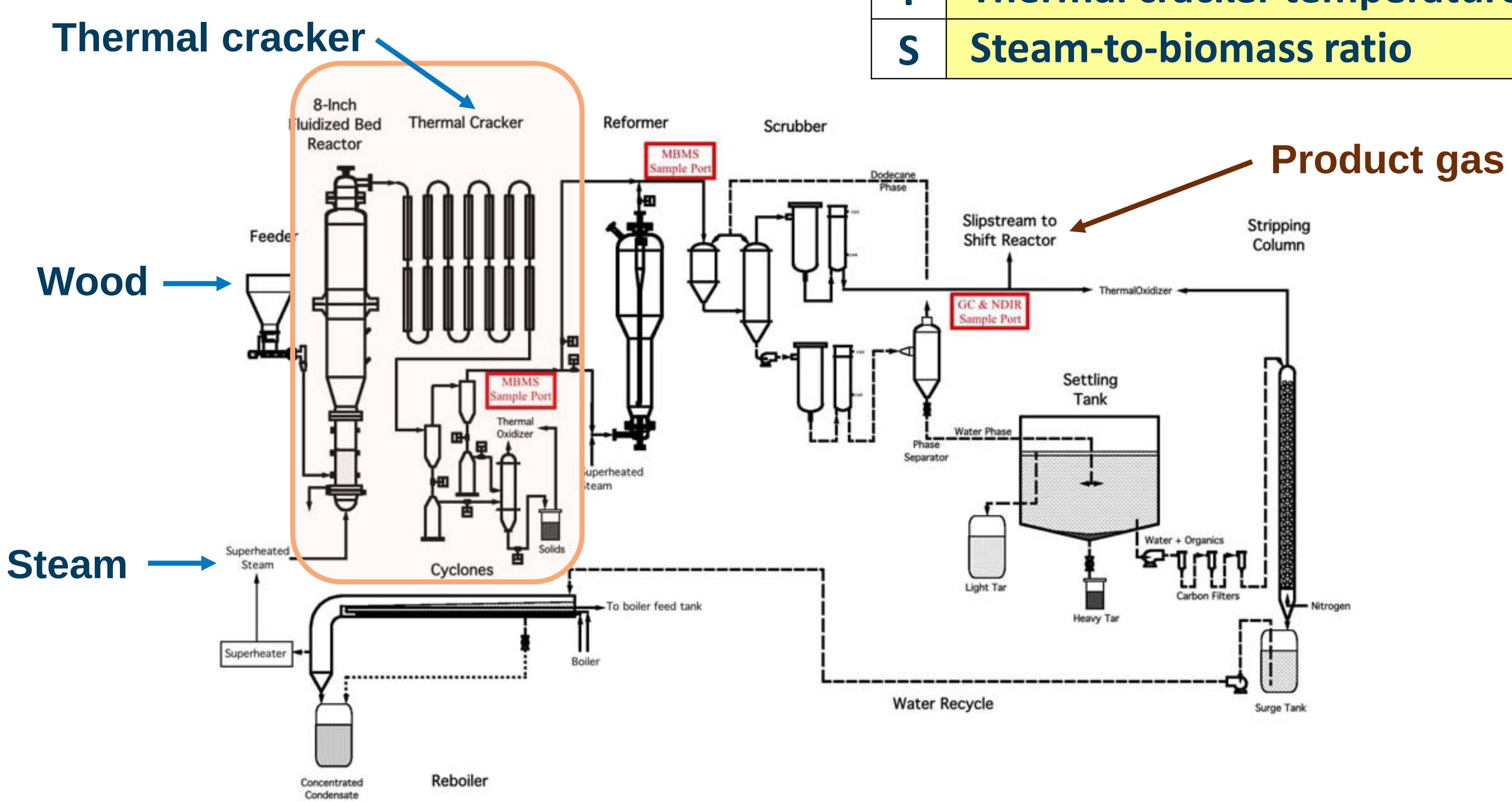
fractional factorial | complete full factorial | **response surface**

Sequential Approach

Objective: Verify the technical and economic performance of hydrogen production from a biomass gasification process (Hrdlicka et al. 2008)

H2	**H_2 (% volume in product gas)**

W	**Wood type (oak and pine)**
T	**Thermal cracker temperature**
S	**Steam-to-biomass ratio**

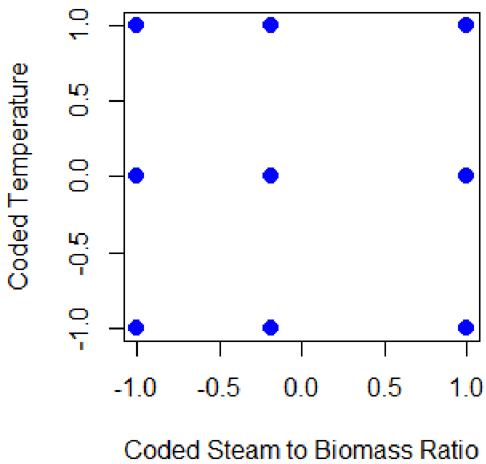


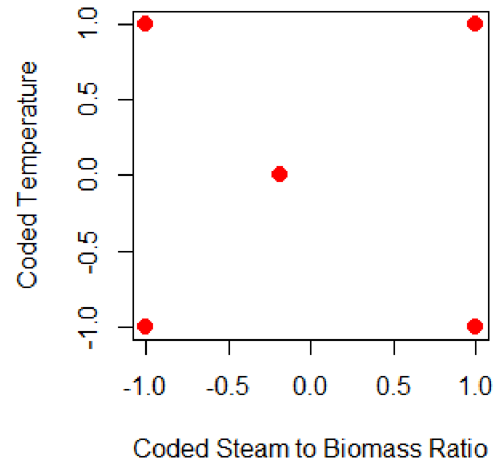
Source: http://www.nrel.gov/docs/fy09osti/44557.pdf

Sequential Approach: Parametric Gasification

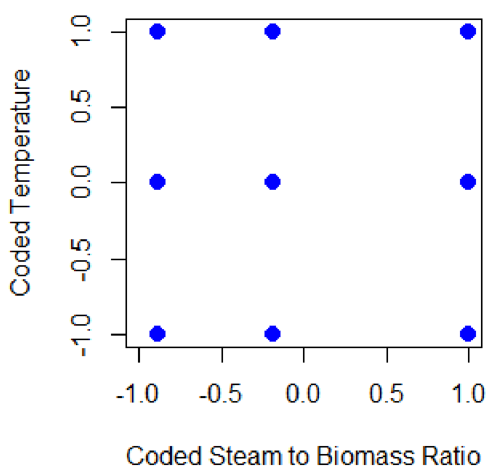
As Completed | Sequential Approach

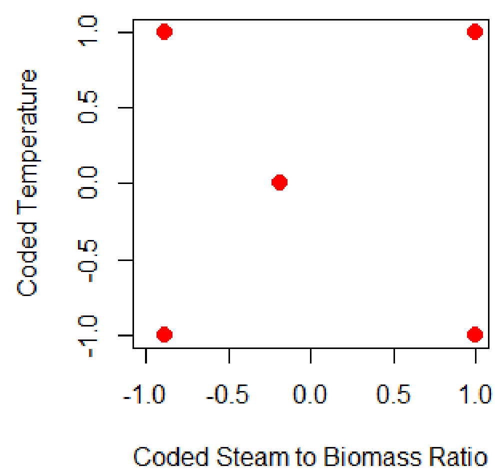
Oak





Pine





2x3x3 = 18 + 5 reps = 23

2x2x2 = 8 + 2 cp + 1 reps = 11

Sequential Approach

MLR As Completed

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	34.7	1.2	29.4	0.000
W	**3.4**	**0.5**	**6.3**	**0.000**
T	**1.7**	**0.7**	**2.4**	**0.034**
S	1.0	0.7	1.5	0.154
T^2	0.7	1.1	0.6	0.530
S^2	-1.0	1.3	-0.8	0.448
W*T	-0.7	0.7	-0.9	0.374
W*S	-0.5	0.7	-0.7	0.473
T*S	0.7	0.9	0.8	0.427

Reduced model

↓

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	34.5	0.5	64.2	0.000
W	**3.4**	**0.5**	**6.4**	**0.000**
T	**1.8**	**0.7**	**2.6**	**0.017**

MLR Sequential Approach

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	34.3	1.6	21.6	0.000
W	**2.8**	**0.8**	**3.4**	**0.044**
T	**2.2**	**1.0**	**2.3**	**0.106**
S	0.8	1.0	0.8	0.499
T^2	0.2	1.9	0.1	0.907
W*T	-1.1	1.0	-1.2	0.332
W*S	-1.1	1.0	-1.1	0.335
T*S	0.9	1.0	0.9	0.435

Reduced model

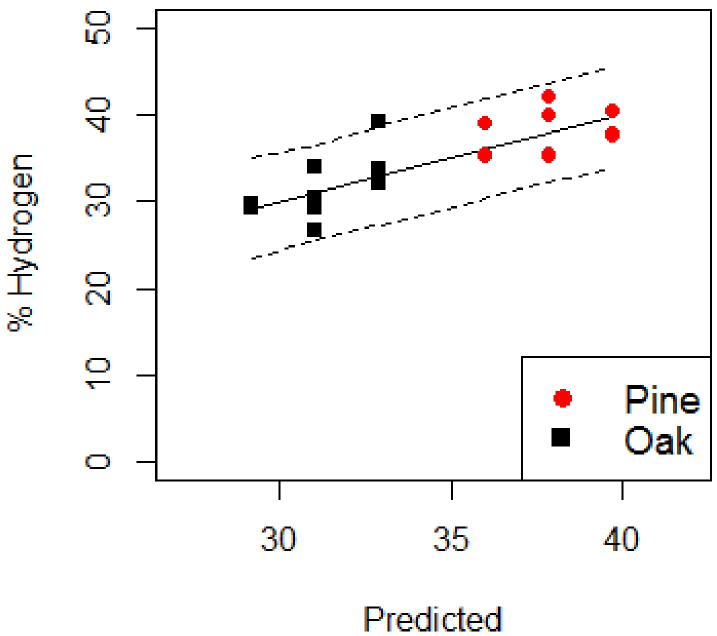
↓

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	34.4	0.8	44.8	0.000
W	**2.8**	**0.8**	**3.7**	**0.006**
T	**2.2**	**0.9**	**2.5**	**0.040**

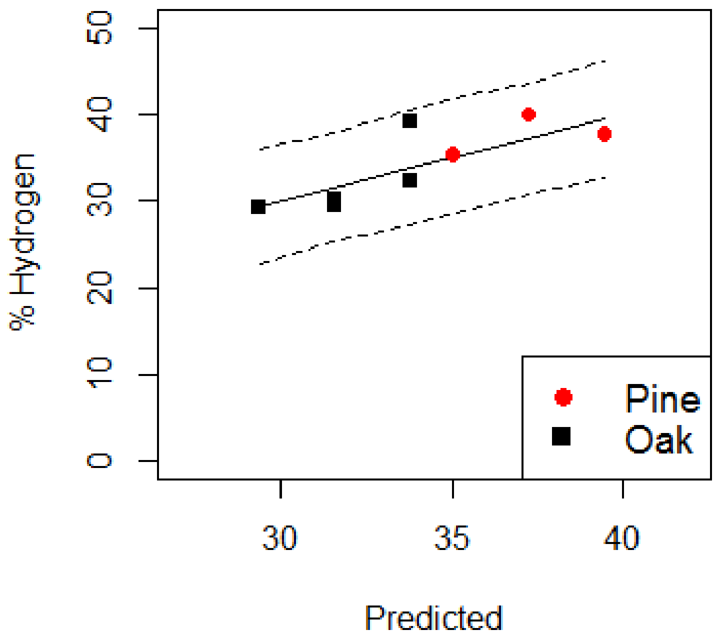
Conclusion: W and T significantly affect H_2 production

Sequential Approach

MLR As Completed



MLR Sequential Approach



- **Comparable results even with substantial variability**
- **Selective replication could have been done for sequential approach**

Summary

- Statistically designed experiments offer many advantages:
  - Statistical hypothesis test for multiple independent variables
  - Detect ***interactions***
  - Reduce number of experimental trials
  - Resolve correlation between independent variables
- Sequential approach can further reduce the number of experiments and still provide conclusive results
- Statistically designed experiments have been in use approximately 90 years

Thank you!

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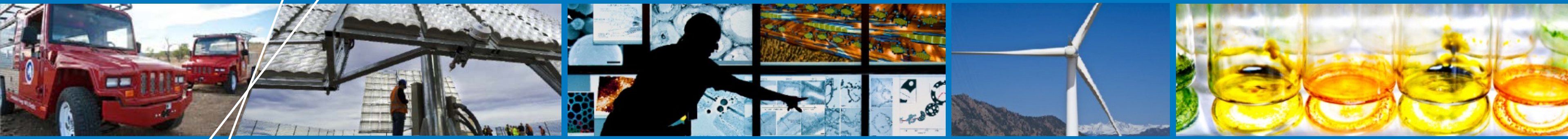

Supplemental Slides

References

Box, G.E.P. and Bisgaard, S. (1997). *Design of Experiments for Discovery, Improvement and Robustness: Going Beyond the Basic Principles*. The College of Engineering, University of Wisconsin, Madison, WI.

Hellstrand, C. (1989). *The Necessity of Modern Quality Improvement and Some Experience With its Implementation in the Manufacture of Rolling Bearings*. The College of Engineering, University of Wisconsin, Madison, WI. Accessed January 11, 2012: http://cqpi.engr.wisc.edu/system/files/r035.pdf.

Hrdlicka, J.; Feik, C.; Carpenter, D.; Pomeroy, M. (2008). *Parametric Gasification of Oak and Pine Feedstocks Using the TCPDU and Slipstream Water-Gas Shift Catalysis*. Golden, CO: National Renewable Energy Laboratory, NREL/TP-510-44557.

Mullin, R. (2013). "Design Of Experiments Makes A Comeback." *Chemical & Engineering News*, Volume 91 Issue 13, pp 25-28. Accessed November 19, 2015: http://cen.acs.org/articles/91/i13/Design-Experiments-Makes-Comeback.html?h=2027056365.

Zhao, H; Tonkyn, R.; Barlow, S.; Peden, C; Koel, B. (2006). "Fractional Factorial Study of HCN Removal over 0.5% Pt/Al2O3 Catalyst: Effects of Temperature, Gas Flow Rate, and Reactant Partial Pressure." *Industrial & Engineering Chemistry Research,* Volume 45, pp 934-939.

Bibliography

Box, G.E.P., Hunter, W.G. and Hunter, J.S. (1978). *Statistics for Experimenters*. John Wiley & Sons.

Deming, W.E. (1986). *Out of the Crisis*. Massachusetts Institute of Technology, Cambridge, MA.

Gunst, R.F. and Mason, R.L. (1991). *Volume 14: How to Construct Fractional Factorial Experiments*. ASQ Quality Press, Milwaukee, WI.

Montgomery, D.C. (1997). *Design and Analysis of Experiments, Fourth Edition*. John Wiley & Sons.

NIST (2015). *Engineering Statistics Handbook*. Accessed December 1, 2015: http://www.itl.nist.gov/div898/handbook/index.htm.

R Core Team (2014). "R: A Language and Environment for Statistical Computing." Vienna, Austria: R Foundation for Statistical Computing. Accessed April 6, 2015: http://www.R-project.org/.

Rodríguez, G. (2013). "Introducing R." Princeton University, Accessed January 13, 2014: http://data.princeton.edu/R/linearModels.html.

More on Parametric Gasification Experiment

MLR Model with Steam to Biomass Ratio (S)

Variable	Estimate	Std. Error	t value	Pr(>|t|)
(Intercept)	34.4	0.5	66.9	0.000
W	3.4	0.5	6.6	0.000
T	1.8	0.7	2.6	0.017
S	1.0	0.6	1.7	**0.108**

Residual standard error: 2.44 on 19 degrees of freedom
Multiple R-squared: 0.735, Adjusted R-squared: 0.693
F-statistic: 17.5 on 3 and 19 DF, p-value: 1.05e-05