A Mixture Design Planning Process

(Sizing Mixture & RSM Designs)

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Sizing Mixture & RSM Designs

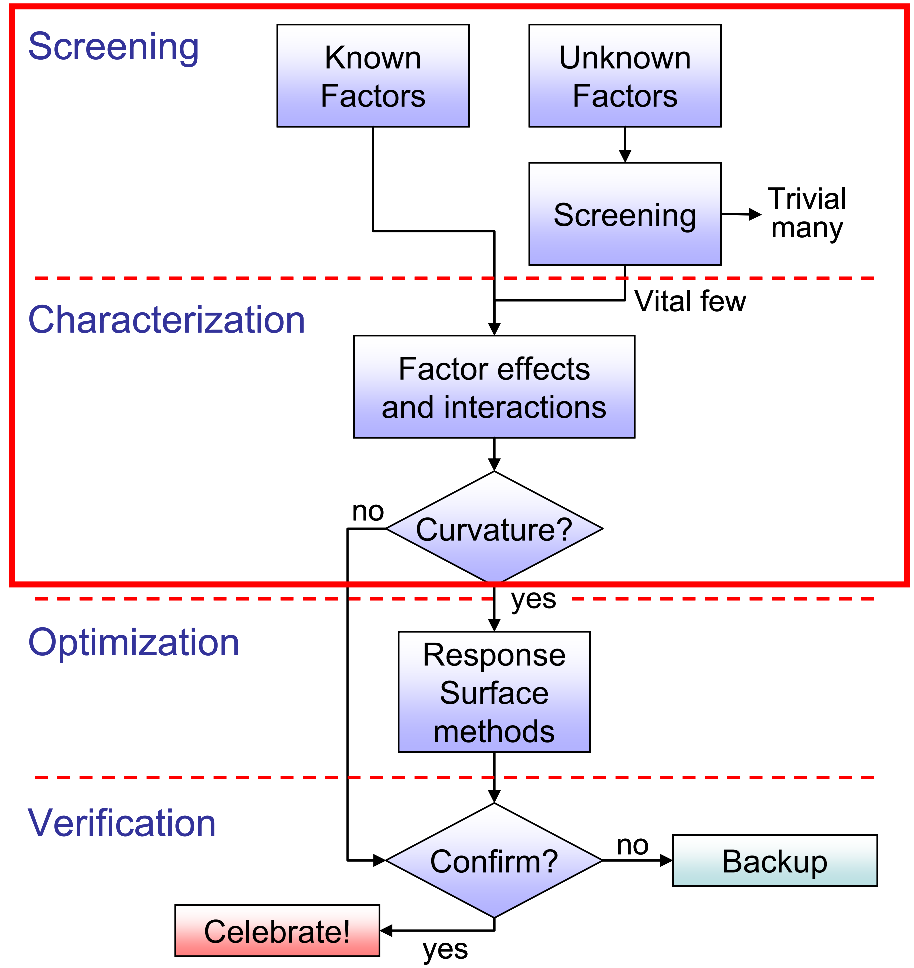
- **Review – Power to size factorial designs.**
- Introduce FDS
- Sizing designs for precision
  - Three component constrained mixture
- Sizing designs to detect a difference
- Conclusion

Sizing Factorial Designs

During screening and characterization the emphasis is on identifying factor effects.

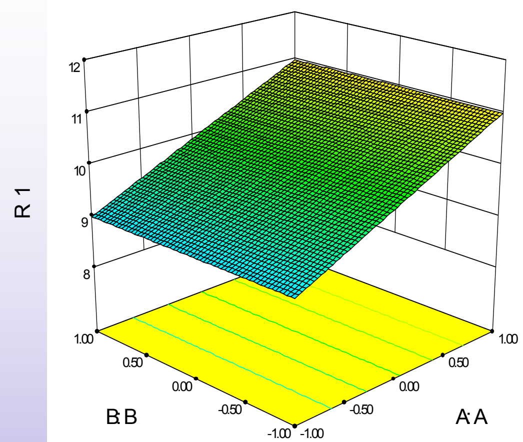
What are the important design factors?

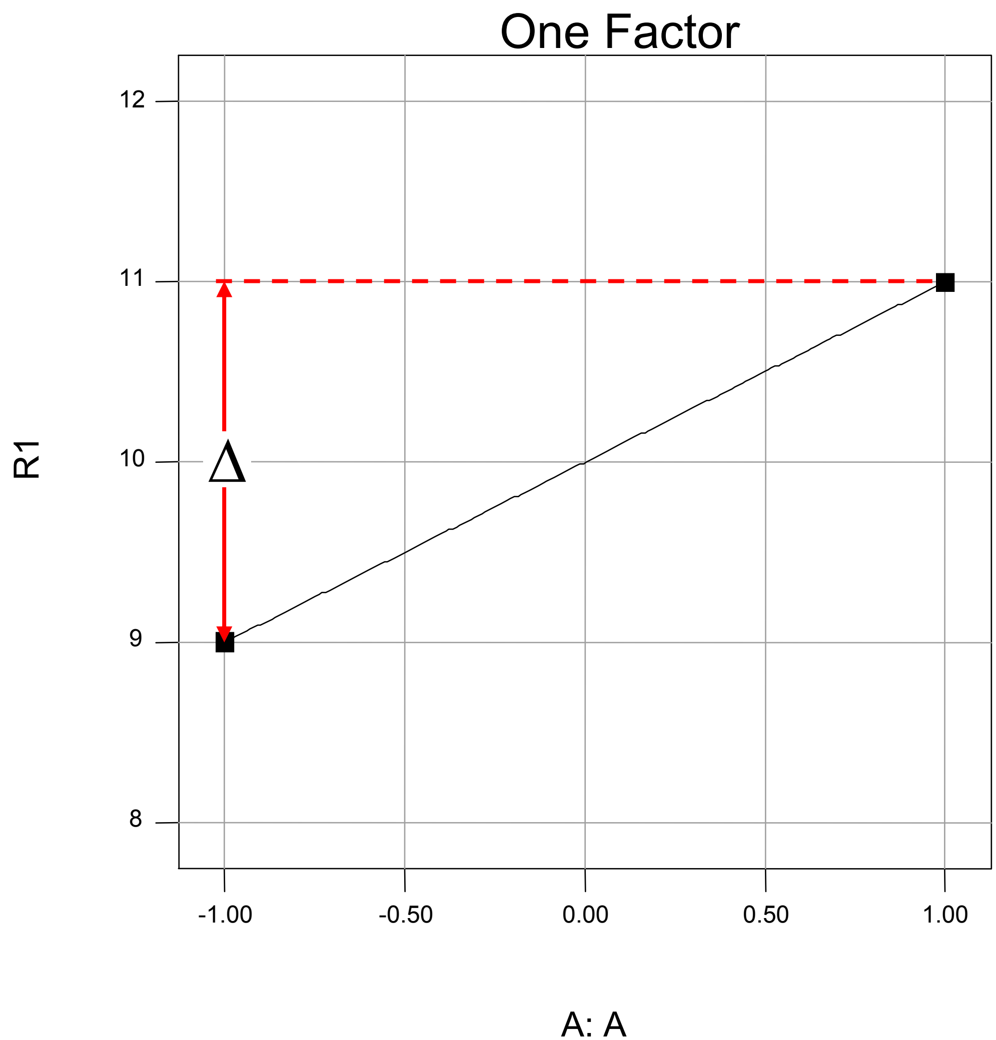
For this purpose power is an ideal metric to evaluate design suitability.



Factorial Design – Power

2^3 Full Factorial Δ=2 and σ=1





One Replicate of 2^3 Full Factorial

$C = (X^T X)^{-1}$ matrix

The design determines the standard error of the coefficient:

$$C = \begin{pmatrix} 0.125 & 0 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0.125 & 0 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0.125 & 0 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0.125 & 0 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0.125 & 0 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0.125 & 0 \\ 0 & 0 & 0 & 0 & 0 & 0 & 0.125 \end{pmatrix}$$

$$\text{t-value}_i = \frac{\beta_i}{SE(\beta_i)} = \frac{\beta_i}{\sqrt{c_{ii}\hat{\sigma}^2}} = \frac{\beta_i}{\sqrt{(0.125)\hat{\sigma}^2}}$$

NonCentrality Parameter

2^3 Full Factorial Δ=2 and σ=1

The reference t distribution assumes the null hypothesis of $\Delta = 0$. The noncentrality parameter (2.828) defines the t distribution under the alternate hypothesis of $\Delta = 2$.

$$\text{noncentrality}_i = \frac{\beta_i}{\sqrt{c_{ii}\hat{\sigma}^2}} = \frac{\Delta_i / 2}{\sqrt{c_{ii}\hat{\sigma}^2}}$$

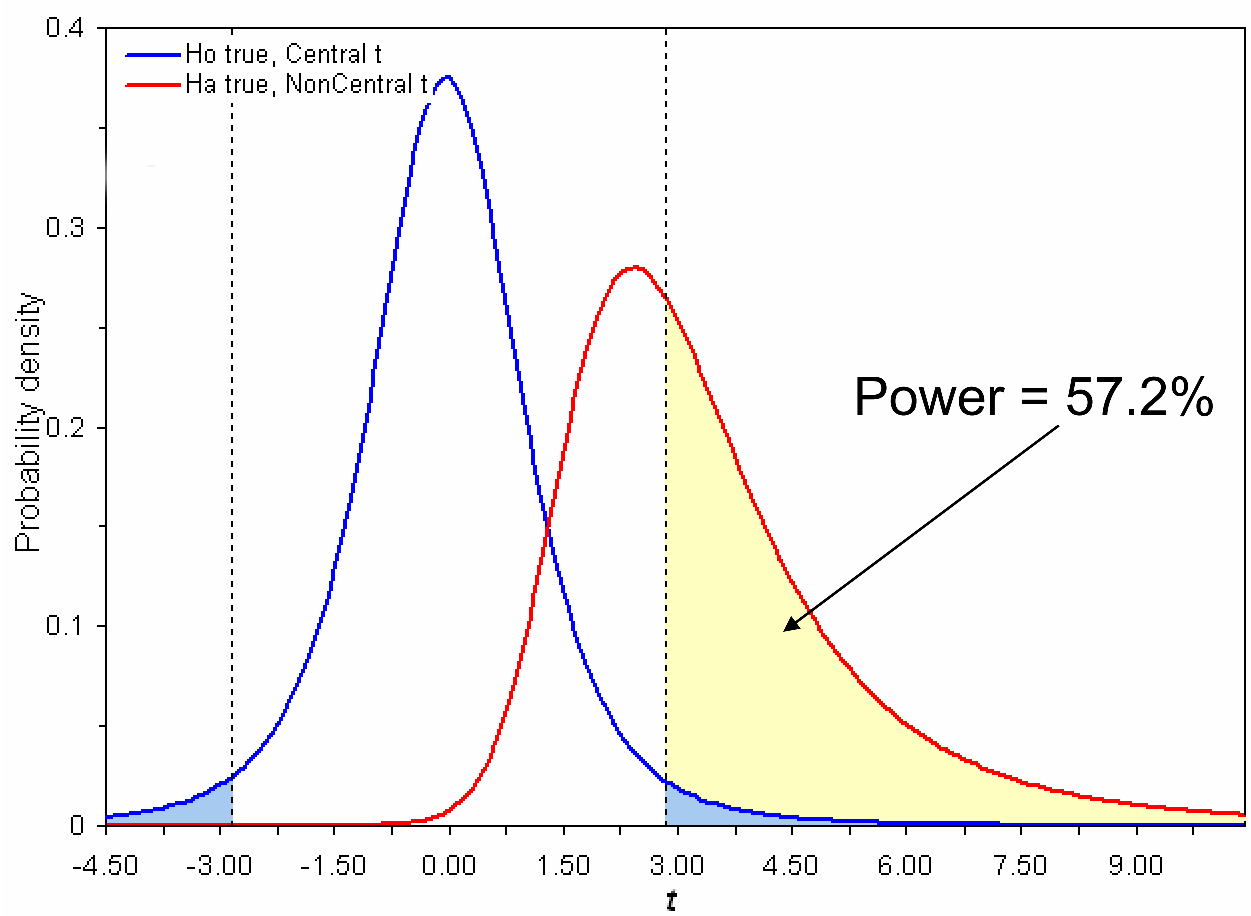
$$= \frac{1}{\sqrt{(0.125)(1)^2}}$$

$$= \frac{1}{0.3536} = 2.828$$

Factorial Design – Power

2^3 Full Factorial Δ=2 and σ=1

noncentral $t_{\alpha=0.05,df=4}$ with noncentrality parameter of 2.828



Factorial Design – Power

2^3 Full Factorial Δ=2 and σ=1

One Replicate

Power is reported at a 5.0% alpha level to detect the specified signal/noise ratio. Recommended power is at least 80%

Signal (delta) = 2.00 Noise (sigma) = 1.00 Signal/Noise (delta/sigma) = 2.00

A	B	C
57.2 %	**57.2 %**	**57.2 %**

Two Replicates

Power is reported at a 5.0% alpha level to detect the specified signal/noise ratio. Recommended power is at least 80%.

Signal (delta) = 2.00 Noise (sigma) = 1.00 Signal/Noise (delta/sigma) = 2.00

A	B	C
95.6 %	**95.6 %**	**95.6 %**

Conclusions

Factorial DOE
During screening and characterization (factorials) emphasis is on identifying factor effects. What are the important design factors? For this purpose power is an ideal metric to evaluate design suitability.

Sizing Mixture & RSM Designs

- Review – Power to size factorial designs.
- **Introduce FDS**
- Sizing designs for precision
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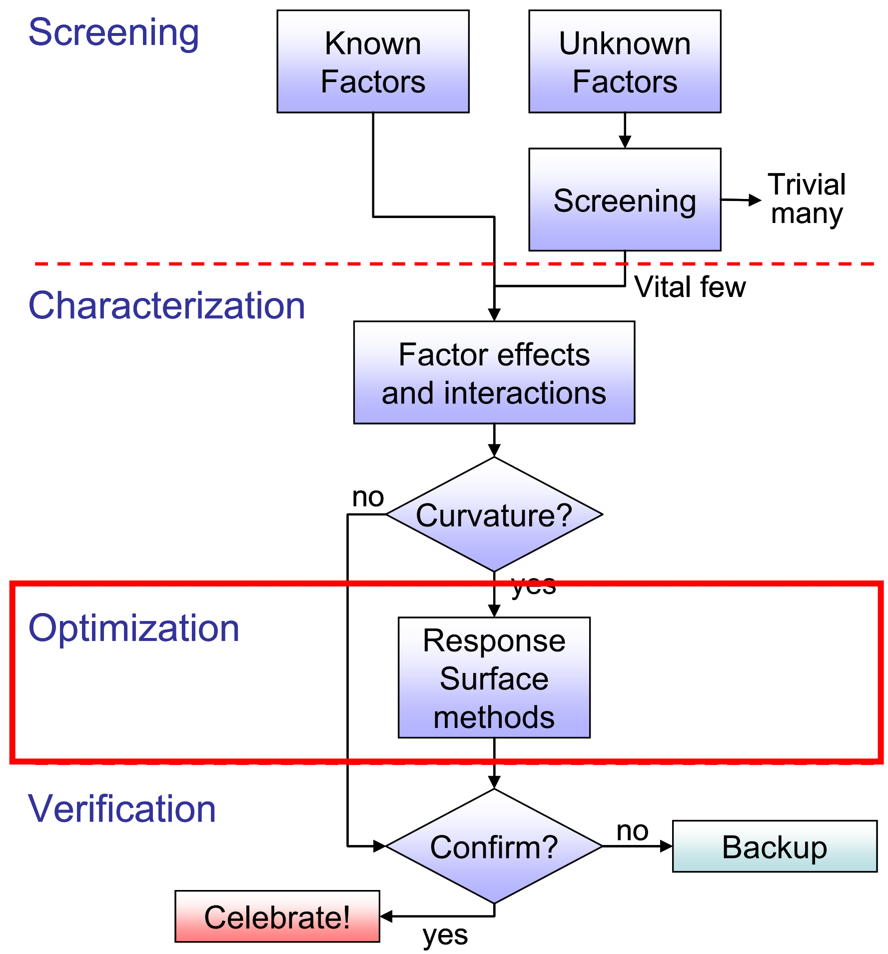
- Conclusion

Sizing Response Surface Designs

When the goal is optimization emphasis is the fitted surface.

How well does the surface represent true behavior?

For this purpose precision (FDS) is an good metric to evaluate design suitability. *(Assuming model adequacy; i.e. insignificant lack of fit.)*



Design Properties

1. Estimate the designed for polynomial well.
2. Give sufficient information to allow a test for lack of fit.
   - ☑ Have more unique design points than coefficients in the model.
   - ☑ Provide an estimate of “pure” error.
3. Remain insensitive to outliers, influential values and bias from model misspecification.
4. Be robust to errors in control of the component levels.
5. Provide a check on model assumptions, e.g., normality of errors.
6. **Generate useful information throughout the region of interest, i.e., provide a good distribution of $\sqrt{\mathrm{Var}\left(\hat{Y}\right)/\sigma^2}$.**
7. Do not contain an excessively large number of trials.

Fraction of Design Space:

- Calculates the volume of the design space having a prediction variance (PV) less than or equal to a specified value.
- The ratio of this volume to the total volume of the design volume is the fraction of design space.
- Produces a single plot showing the cumulative fraction of the design space on the x-axis (from zero to one) versus the PV on the y-axis.

FDS – PV
Fraction of Design Space

Prediction Variance:

$$PV(x_0) = \frac{\operatorname{var}\left(\hat{\bar{y}}_0\right)}{s^2} = x_0^T \left(X^T X\right)^{-1} x_0$$

PV is a function of:

- x_0 – the location in the design space (i.e. the x coordinates for all model terms).
- X – the experimental design (i.e. where the runs are in the design space).

FDS – StdErr
Fraction of Design Space

Prediction standard error of the expected value:

$$PV\left(x_0\right) = \frac{\operatorname{var}\left(\hat{\bar{y}}_0\right)}{s^2} = x_0^T\left(X^T X\right)^{-1} x_0$$

$$StdErr\left(x_0\right) = \frac{s_{\hat{\bar{y}}_0}}{s} = \sqrt{PV\left(x_0\right)}$$

FDS Plot

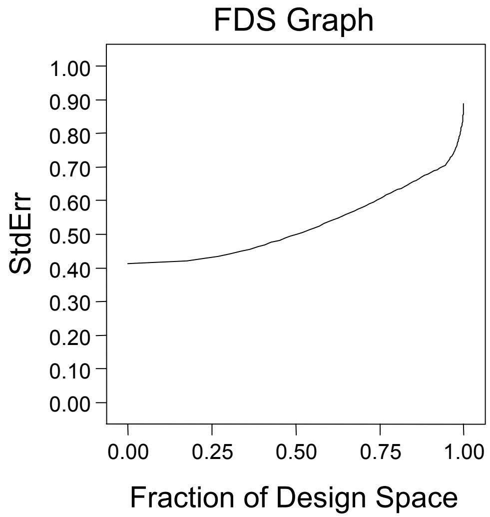
Fraction of Design Space

1. Pick random points in the design space.

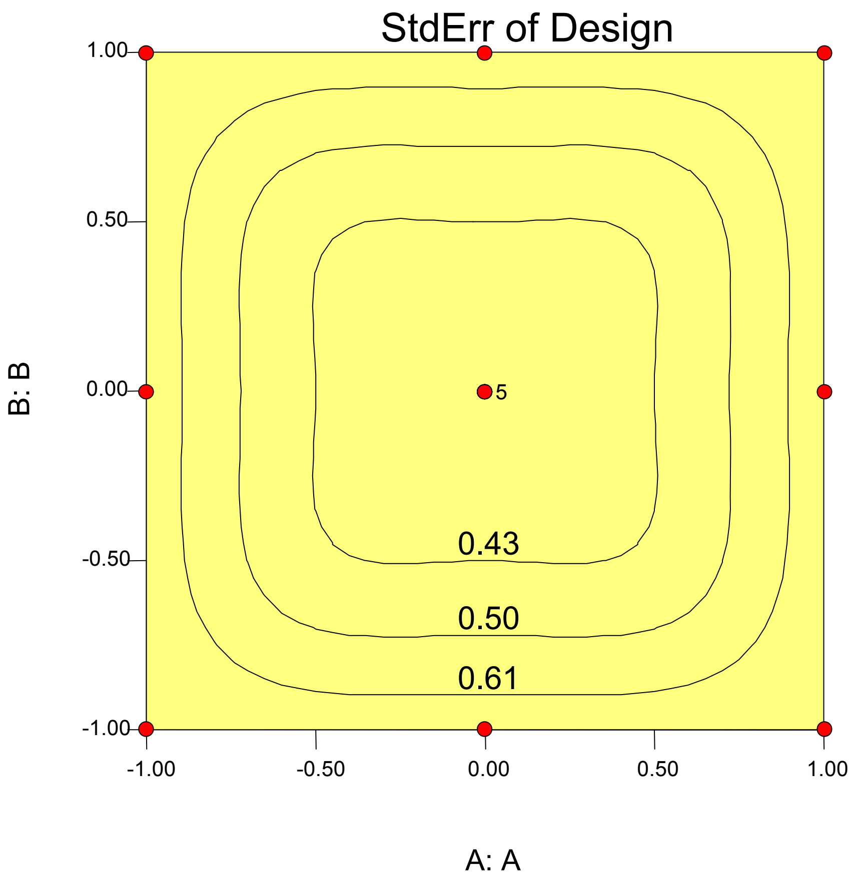
2. Calculate the standard error of the expected value

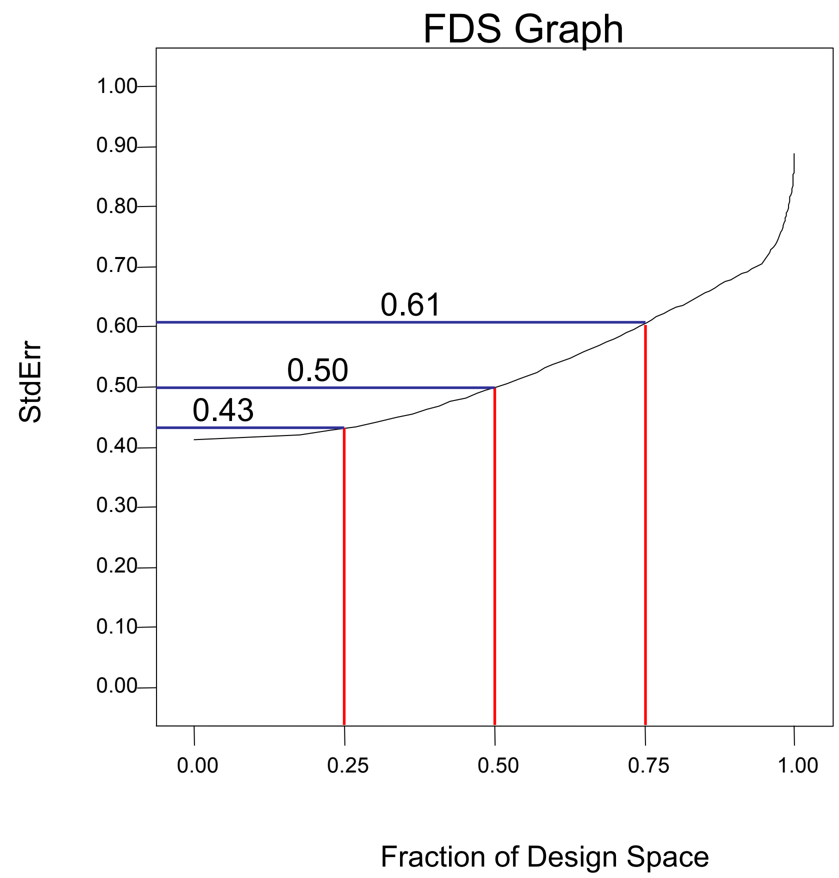
$$SE_{\hat{y}_0} = s\sqrt{x_0^T \left(X^T X\right)^{-1} x_0}$$

3. Plot the standard error as a fraction of the design space.



FDS – StdErr
Two-Factor Face Centered CCD





Sizing Mixture & RSM Designs

- Review – Power to size factorial designs.
- Introduce FDS
- **Sizing designs for precision**
  - **Three component constrained mixture**
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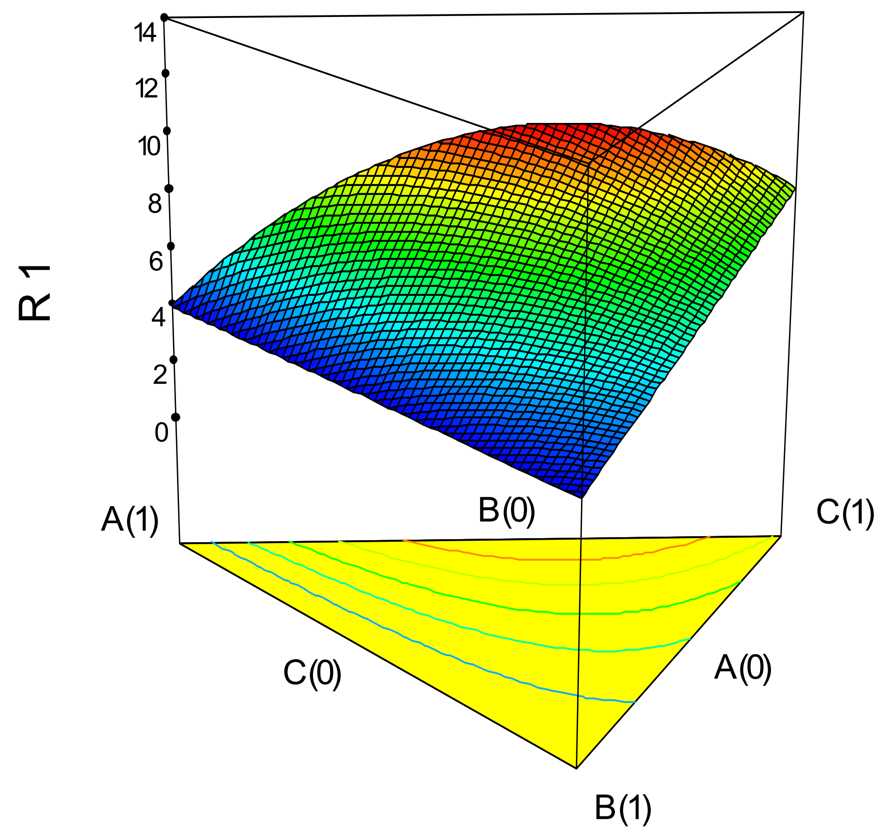
$$\hat{y} = 4A + 4B + 8C + 16AC$$

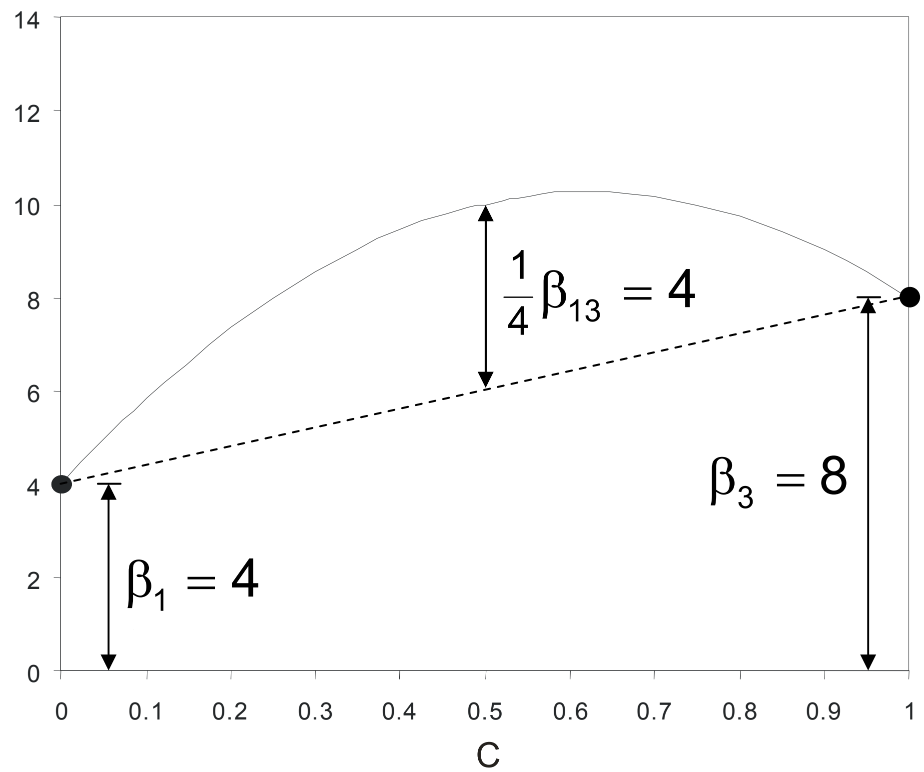
Most of the action occurs on the A-C edge because of the AC coefficient of 16. The quadratic coefficient of 16 means that the response is 4 units higher at A=0.5, C=0.5 than one would expect with linear blending.



Mixture Simplex
Quadratic Model

$$\hat{y} = 4A + 4B + 8C + 16AC$$

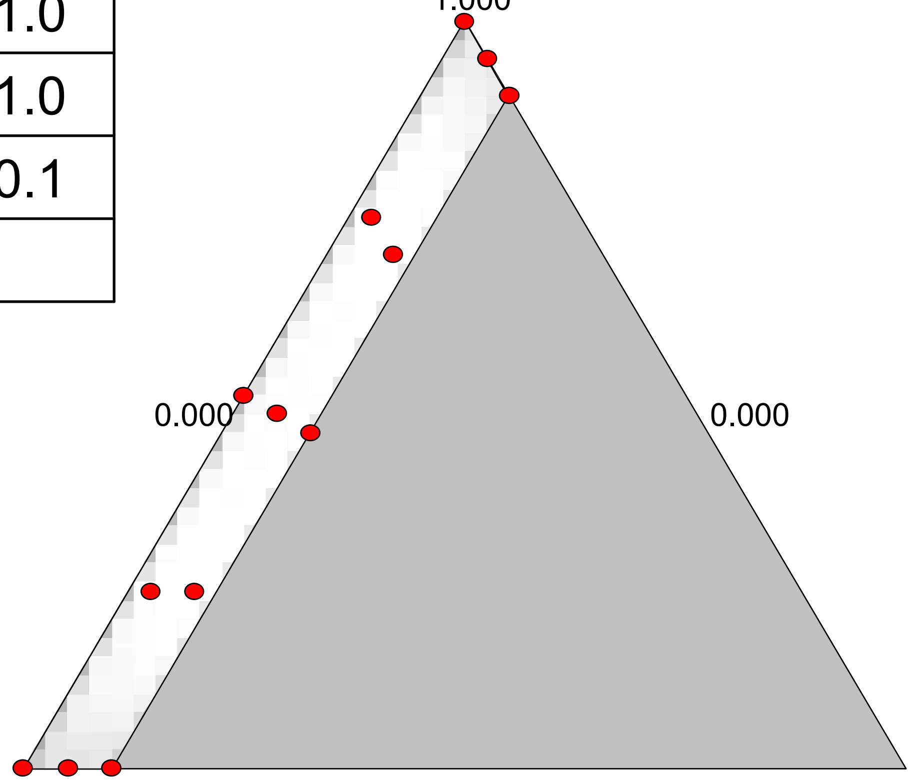
This illustrates the quadratic blending along the A-C edge as C increases from zero to one.



Mixture Constrained

Design for Quadratic Model

0.0	≤	A	≤	1.0
0.0	≤	B	≤	1.0
0.0	≤	C	≤	0.1
Total = 1.0				



Mixture Constrained
Quadratic Model (1 replicate)

				Power at 5 % alpha level for effect of		
Term	StdErr*	VIF	R_i-Squared	1 s	2 s	3 s
A	0.87	3.44	0.7094	5.0 %	5.0 %	5.0 %
B	0.87	3.44	0.7094	5.0 %	5.0 %	5.0 %
C	233.34	2722.29	0.9996	5.0 %	5.0 %	5.0 %
AB	2.86	2.19	0.5442	22.9 %	67.3 %	94.7 %
AC	258.98	1092.98	0.9991	5.0 %	5.0 %	5.0 %
BC	258.98	1092.98	0.9991	5.0 %	5.0 %	5.0 %

*Basis Std. Dev. = 1.0

Power is bottomed out at alpha!

Define Precision

Half Width of the Confidence Interval

6. Generate useful information throughout the region of interest.

Question: Will predictions, using the quadratic model from this design, be precise enough for our purposes?

- To know the truth requires an infinite number of runs; most likely this will exceed our budget.
- So the question is how precisely do we need to estimate the response?
- The trade off is more precision requires more runs.

Define Precision

Half Width of the Confidence Interval

Confidence interval on the expected value:

The mean response is estimated and the precision of the estimate is quantified by a confidence interval:

$$\hat{\bar{y}} \pm t_{\alpha/2, df}\left(s_{\hat{\bar{y}}}\right)$$

We will use half-width of the confidence interval (δ) to define the precision desired:

$$\delta = t_{\alpha/2, df}\left(s_{\hat{\bar{y}}}\right)$$

What Precision is Needed?
Confidence Interval Half-Width

Half-width of confidence interval: δ

Input standard deviation estimate: s

$$\hat{y} \pm \delta$$

$$\delta = t_{\alpha/2, df}\left(s_{\hat{y}}\right)$$

$$s_{\hat{y}} = s\sqrt{x_0^T \left(X^T X\right)^{-1} x_0}$$

$$StdErr\left(FDS\right) = \frac{s_{\hat{y}}}{s} = \sqrt{x_0^T \left(X^T X\right)^{-1} x_0}$$

- Want quadratic surface to represent the true response value within 10 with 95% confidence.
- The overall standard deviation this response is 7.8.

For 95% confidence $t_{.05/2,7} = 2.365, \ \delta = 10 \ \ \& \ \ s = 7.8$

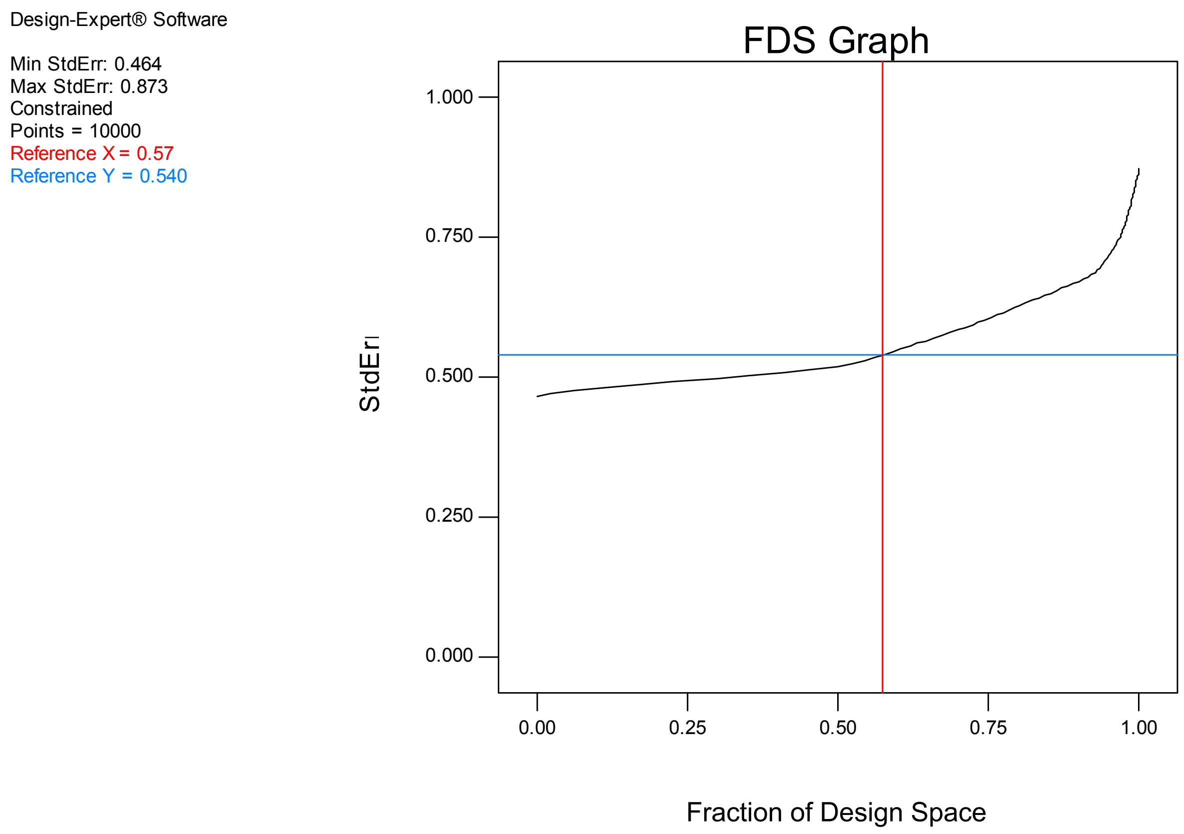
$$\delta = t_{\alpha/2,df}\left(s_{\hat{y}}\right) \quad 10 = 2.365\left(s_{\hat{y}}\right) \quad s_{\hat{y}} = 4.23$$

$$StdErr\left(FDS\right) = \frac{s_{\hat{y}}}{s} = \frac{4.23}{7.8} = 0.54$$

Mixture Constrained

Quadratic Model (1 replicate)

57% of the design space has StdErr ≤ 0.54



Mixture Constrained

Quadratic Model (2 replicates)

				Power at 5 % alpha level for effect of		
Term	StdErr*	VIF	R_i-Squared	1 s	2 s	3 s
A	0.62	3.44	0.7094	5.0 %	5.0 %	5.0 %
B	0.62	3.44	0.7094	5.0 %	5.0 %	5.0 %
C	164.99	2722.29	0.9996	5.0 %	5.0 %	5.0 %
AB	2.02	2.19	0.5442	47.1 %	96.5 %	99.9 %
AC	183.12	1092.98	0.9991	5.0 %	5.0 %	5.0 %
BC	183.12	1092.98	0.9991	5.0 %	5.0 %	5.0 %

*Basis Std. Dev. = 1.0

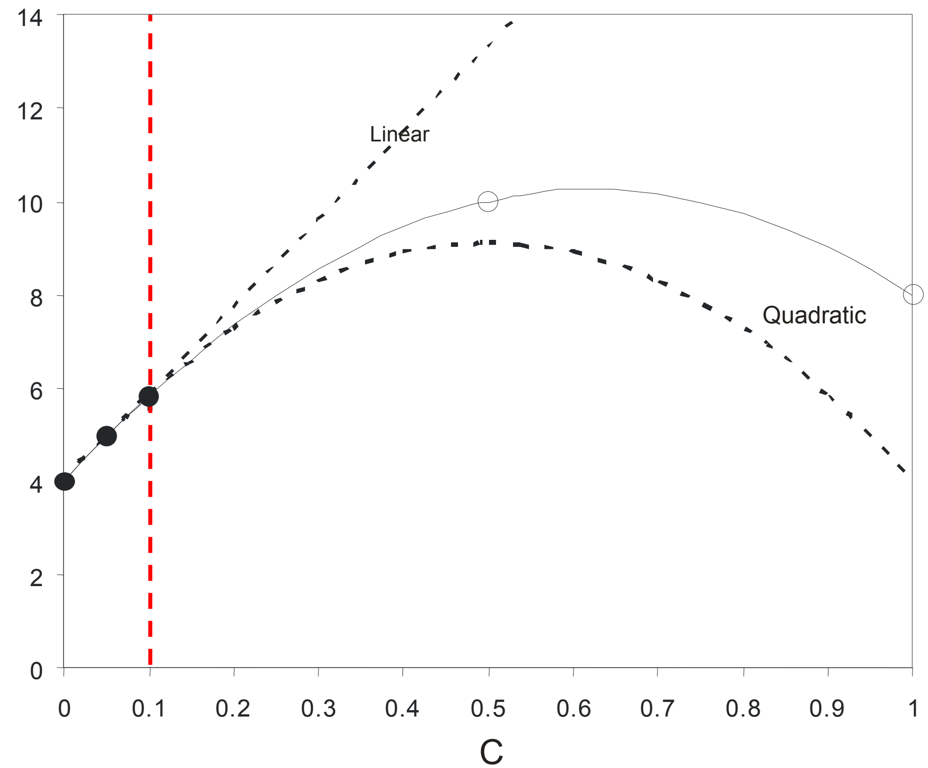
Adding a replicate does little to increase power!

Mixture Constrained

Why didn't Power Increase?

Quadratic, linear or combinations of them model response.

Model Coefficients are Correlated!



Individual coefficients can not be resolved!

Mixture Constrained
Quadratic Model (2 replicates)

- Want quadratic surface to represent the true response value within 10 with 95% confidence.
- The overall standard deviation this response is 7.8.

For 95% confidence $t_{.05/2,20} = 2.086,\ \delta = 10\ \ \&\ \ s = 7.8$

$$\delta = t_{\alpha/2,df}\left(s_{\hat{y}}\right) \quad 10 = 2.086\left(s_{\hat{y}}\right) \quad s_{\hat{y}} = 4.79$$

$$StdErr\left(FDS\right) = \frac{s_{\hat{y}}}{s} = \frac{4.79}{7.8} = 0.61$$

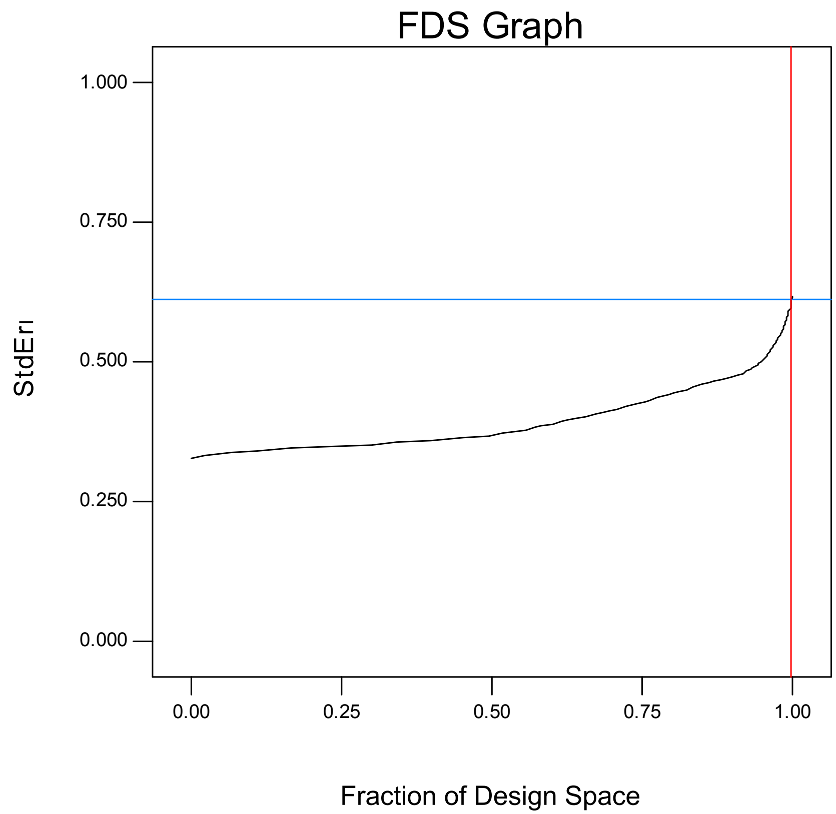
Mixture Constrained

Quadratic Model (2 replicates)

100% of the design space has StdErr ≤ 0.61

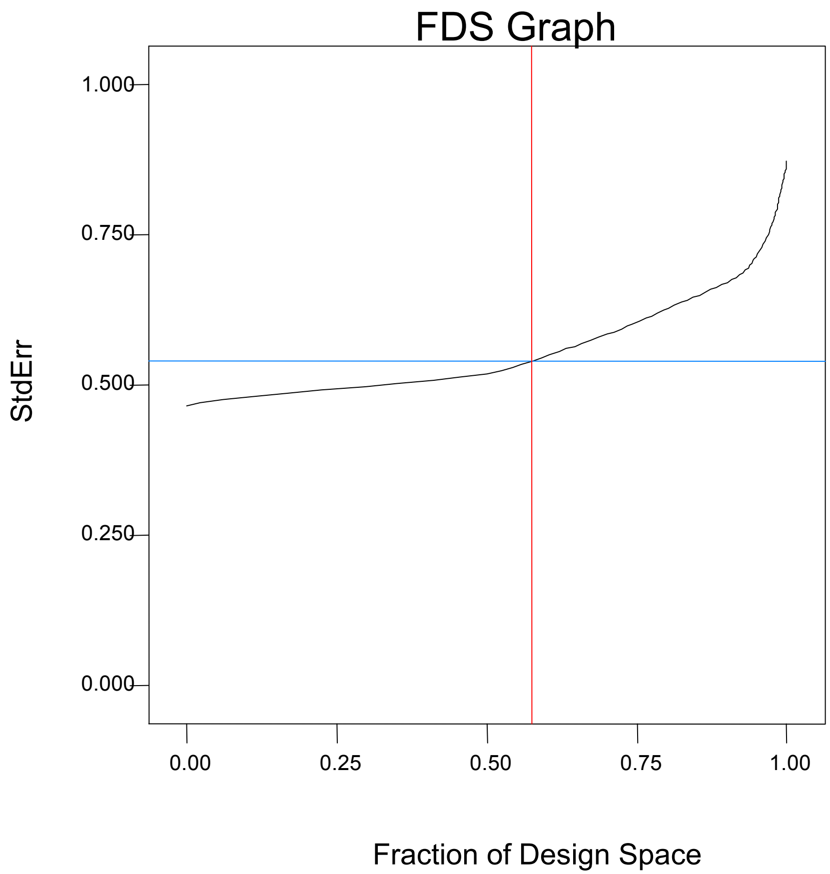
Design-Expert® Software

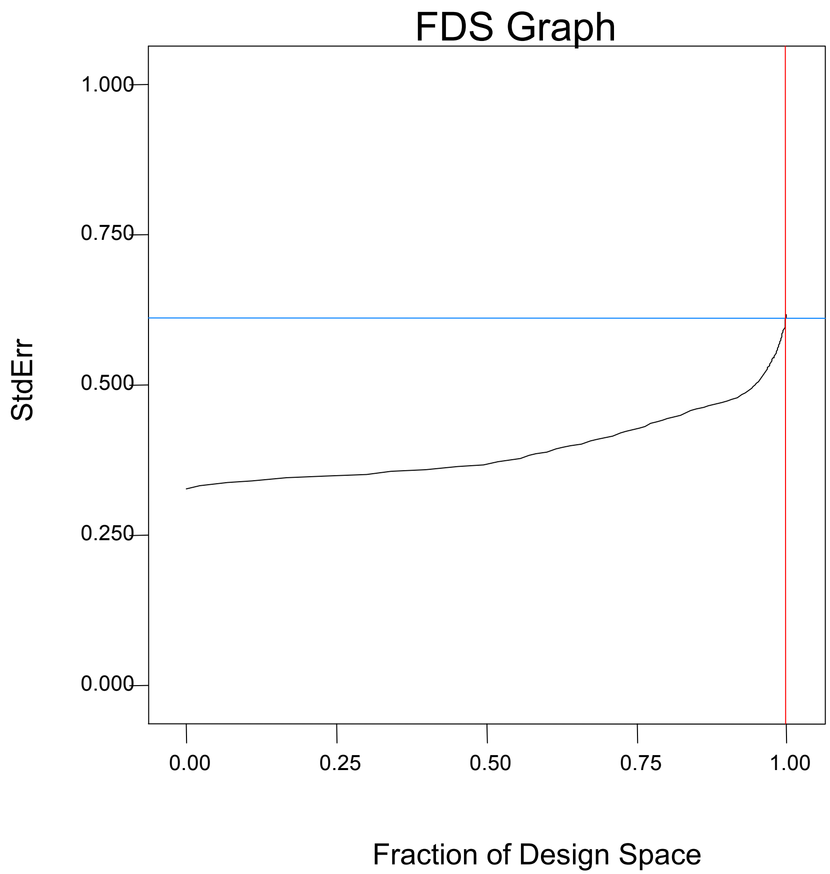
Min StdErr: 0.328
Max StdErr: 0.617
Constrained
Points = 10000
Reference X = 1.00
Reference Y = 0.611



Mixture Constrained

Quadratic Model (1 & 2 replicates)





FDS improves*; even though power doesn't!

* doubling the design reduces StdErr by the sqrt(2)

Precision Depends On

- The size of the confidence interval half-width (δ):

  A larger half-width (δ) increases the FDS.

- The size of the experimental error σ:

  A smaller σ increases the FDS.

- The α risk chosen:

  A larger α increases the FDS.

- Choose design appropriate to the problem:

  Size the design for the precision required.

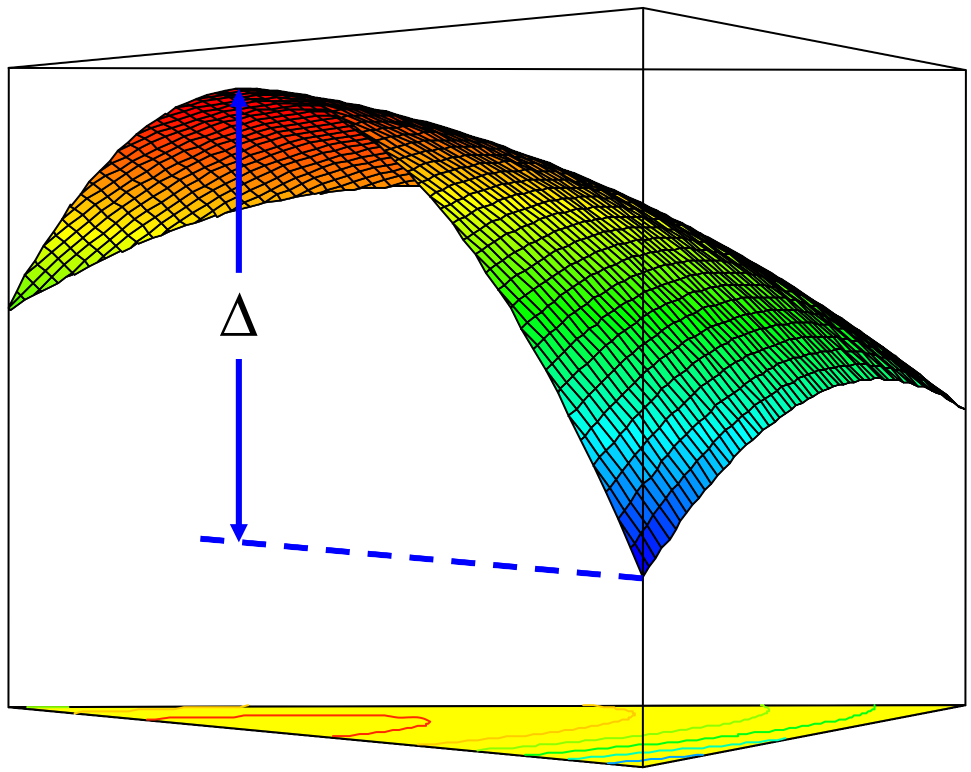
Conclusions

Factorial DOE	Mixture Design and Response Surface Methods
During screening and characterization (factorials) emphasis is on identifying factor effects.	When the goal is optimization (usually the case for mixture design & RSM) emphasis is on the fitted surface.
What are the important design factors?	How well does the surface represent true behavior?
For this purpose power is an ideal metric to evaluate design suitability.	For this purpose precision (FDS) is a good metric to evaluate design suitability.

Sizing Mixture & RSM Designs

- Review – Power to size factorial designs.
- Introduce FDS
- Sizing designs for precision
  - Three component constrained mixture
- **Sizing designs to detect a difference**
- Conclusion

Detecting a Difference of Δ
wherever it may occur



What is the probability of finding a difference ≥ Δ
if it occurs between any two points in the design space?

FPDS Plot

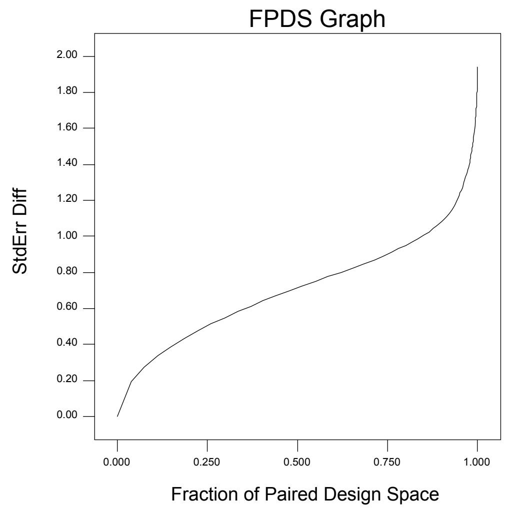
Fraction of Paired Design Space

1. Pick random pairs of points in the design space.

2. Calculate the standard error of the difference

$$SE_{\hat{y}_1-\hat{y}_2} = s\sqrt{x_1^T\left(X^TX\right)^{-1}x_1 + x_2^T\left(X^TX\right)^{-1}x_2 - 2\left(x_1^T\left(X^TX\right)^{-1}x_2\right)}$$

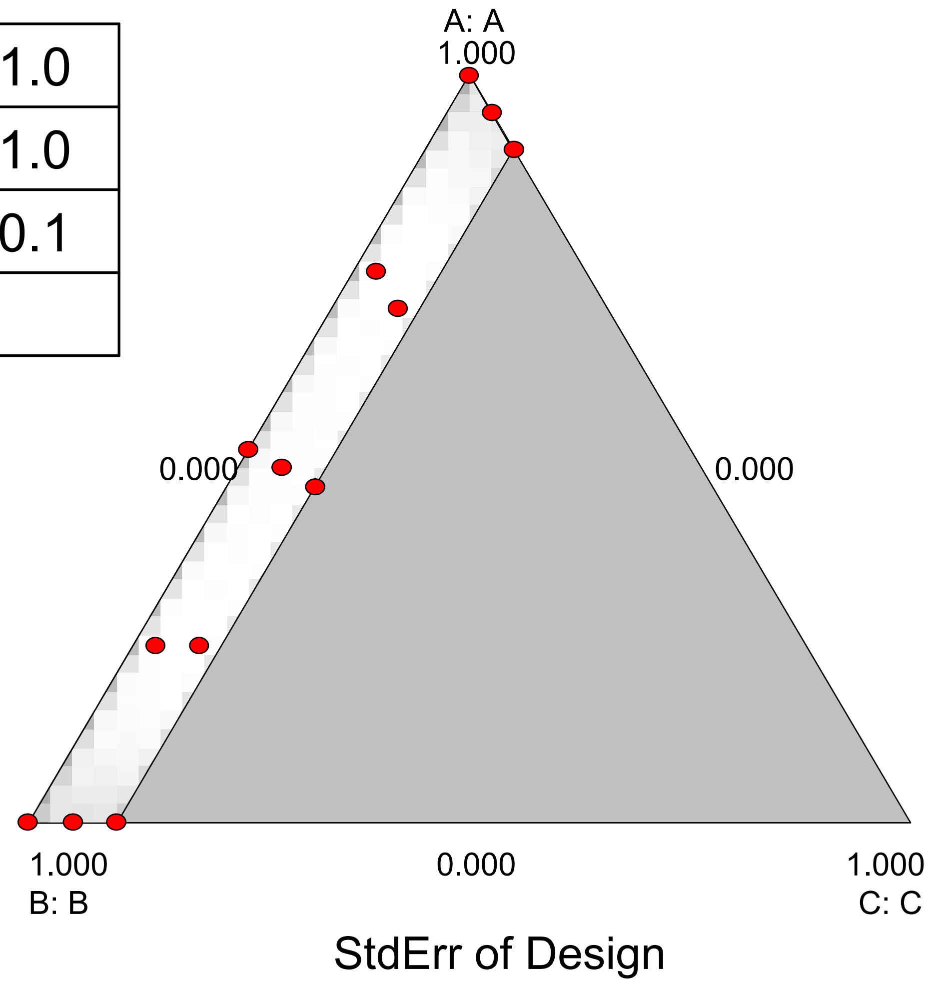
3. Plot the standard error as a fraction of the design space.



Revisit – Mixture Constrained

Design for Quadratic Model

0.0	≤	A	≤	1.0
0.0	≤	B	≤	1.0
0.0	≤	C	≤	0.1
Total = 1.0				



Mixture Constrained

Quadratic Model (1 replicate)

- Want to detect a difference of 10 on a quadratic surface with 95% confidence.
- The overall standard deviation this response is 7.8.

For 95% confidence $t_{.05/2,7} = 2.365,\ \Delta = 10\ \ \&\ \ s = 7.8$

$$\Delta = t_{\alpha/2,df}\left(s_{\Delta}\right) \quad 10 = 2.365\left(s_{\Delta}\right) \quad s_{\Delta} = 4.23$$

$$StdErr\left(FPDS\right) = \frac{s_{\Delta}}{s} = \frac{4.23}{7.8} = 0.54$$

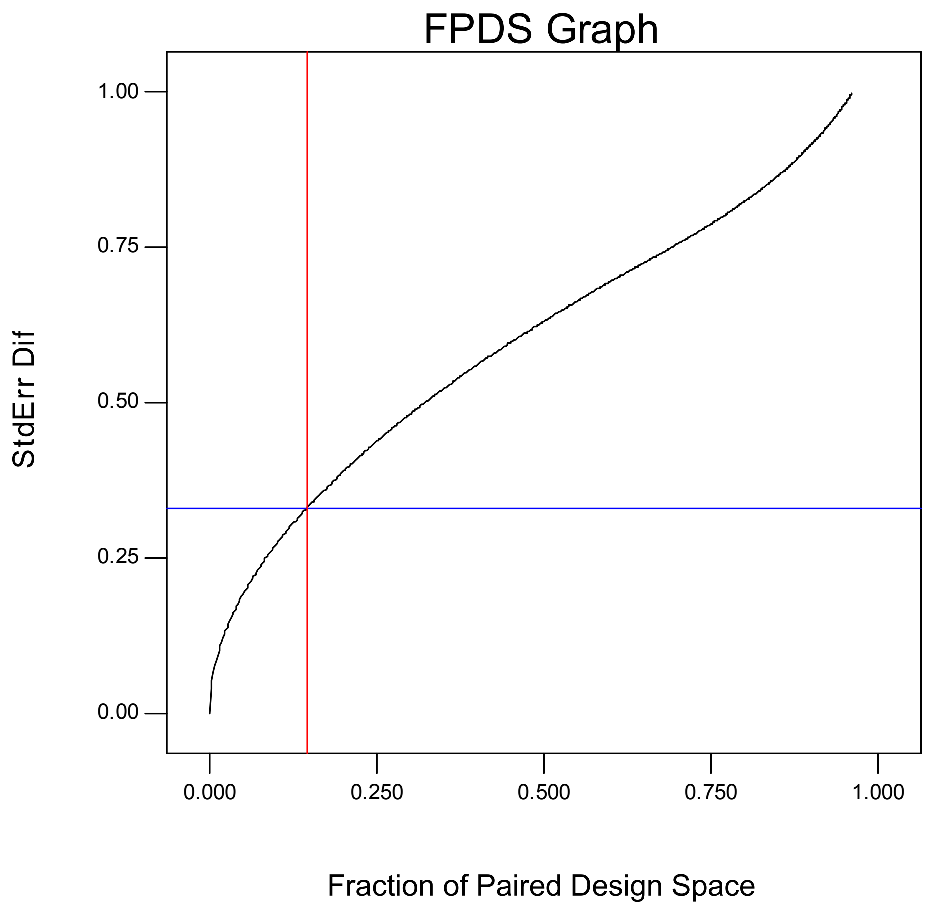
Mixture Constrained

Quadratic Model (1 replicate)

33% of the design space has StdErr ≤ 0.54

Design-Expert® Software

Min StdErr Diff: 0.00
Max StdErr Diff: 1.21
Constrained
Pairs = 100000
t(0.05/2,7) = 2.36462
Reference X = 0.144
Reference Y = 0.33



Mixture Constrained

Quadratic Model (2 replicates)

- Want to detect a difference of 10 on a quadratic surface with 95% confidence.
- The overall standard deviation this response is 7.8.

For 95% confidence $t_{.05/2,7} = 2.086,\ \Delta = 10\ \ \&\ \ s = 7.8$

$$\Delta = t_{\alpha/2,df}\left(s_\Delta\right) \quad 10 = 2.086\left(s_\Delta\right) \quad s_\Delta = 4.79$$

$$StdErr\left(FPDS\right) = \frac{s_\Delta}{s} = \frac{4.79}{7.8} = 0.61$$

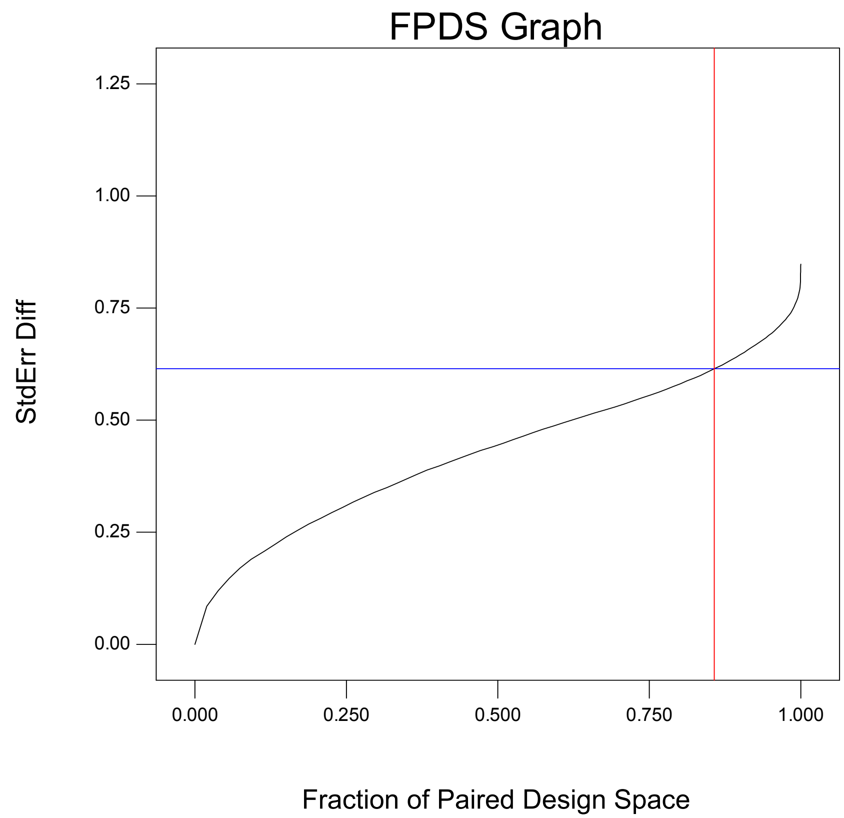
Mixture Constrained

Quadratic Model (2 replicates)

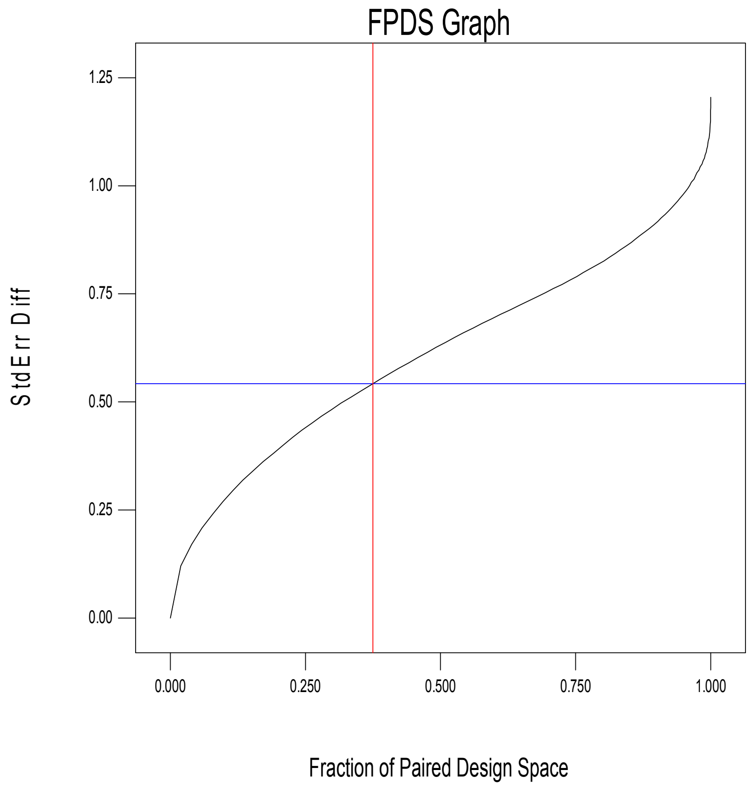
86% of the design space has StdErr ≤ 0.61

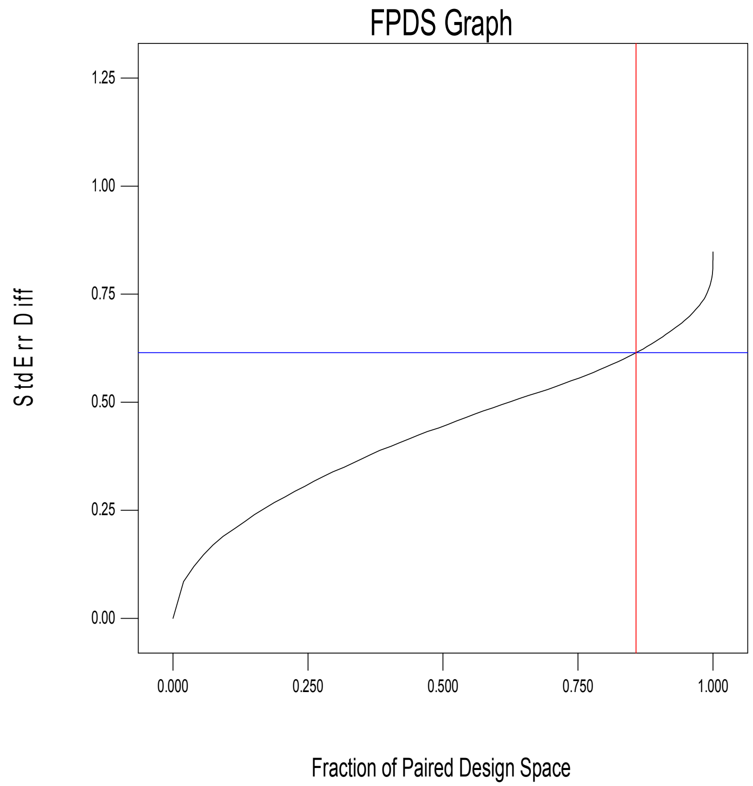
Design-Expert® Software

Min StdErr Diff: 0.00
Max StdErr Diff: 0.85
Constrained
Points = 50000
t(0.05/2,20) = 2.08596
Reference X = 0.857
Reference Y = 0.61



Mixture Constrained
Quadratic Model (1 & 2 replicates)





FPDS improvement

Detecting a Difference of Δ Depends On

- The size of the difference (Δ):
  A larger difference (Δ) increases the FPDS.
- The size of the experimental error σ:
  A smaller σ increases the FPDS
- The α risk chosen:
  A larger α increases the FPDS.
- Choose design appropriate to the problem:
  Size the design to detect a difference of interest.

Conclusions

Factorial DOE	Mixture Design and Response Surface Methods
During screening and characterization (factorials) emphasis is on identifying factor effects.	When the goal is to detect a difference emphasis is on the fitted surface rather than the factors.
What are the important design factors?	How well does the design support finding a difference?
For this purpose power is an ideal metric to evaluate design suitability.	For this purpose (FPDS) is a good metric to evaluate design suitability.

Sizing Mixture & RSM Designs

- Review – Power to size factorial designs.
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- **Conclusion**

Conclusion

FDS *(with confidence interval half width, δ)*

and

FPDS *(with the difference of interest, Δ)*

are better metrics than power for evaluating response surface and mixture designs; particularly constrained designs with non-orthogonal models.

Special Thanks

For aid implementing FDS plots:

- Christine M. Anderson-Cook
- Heidi B. Goldfarb
- Douglas Montgomery

Thank You for your attention!

References

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