Sensitivity Study of the NIST 500 mm Guarded-Hot-Plate Apparatus: A Methodology Based on Orthogonal Designs

Robert Zarr and James Filliben National Institute of Standards and Technology

November 6, 2014

EMRP THERMO: Metrology for Thermal Protection Materials G15 CS4, National Physical Laboratory, Teddington UK (via teleconference)

Organized by:
National Physical Laboratory



Motivation

2012 ASTM C16 Workshop

NIST Technical Note 1764

High-Temperature Guarded-Hot-Plate and Pipe Measurements: 2nd Operators Workshop (March 19-20, 2012) Co-sponsored by ASTM Committee C16 on Thermal Insulation

> Robert Zarr Thomas Whitaker Frank Tyler

http://dx.doi.org/10.6028/NIST.TN.1764



National Institute of Standards and Technology U.S. Department of Commerce



Workshop Recommendation #2

2) Attendees should consider conducting "in-house" sensitivity studies (also known by ASTM as "ruggedness tests")

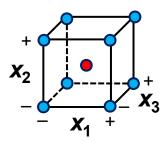
Introduction to Design of Experiment (DEX)

James Filliben, NIST Mathematical Statistician

- Orthogonal Factorial Design Full (2^k) or Fractional (2^{k-p})
 - Advantages:
 - Optimum design for examining multiple factors (screening process)
 - Balanced every factor setting occurs the same number of times
 - Randomized minimizes bias (i.e., drift protection over time)
 - Allows detection of interaction effects (not possible with one-factor-ata-time design)
 - Disadvantage: $n = 2^k$; n increases geometrically with k (time ↑, cost ↑)
 - Disadvantage: $n = 2^{k-p}$; confounding (cannot estimate all effects separately)

Example:

- Full factorial hypothetical small experiment
 - 3 factors (k = 3): x_1 , x_2 , x_3
 - 2 settings: coded as –1 and +1
- Number of runs, $n = 2^k = 2^3 = 8 + 1$ (center point)



2⁶⁻² Fractional Factorial Design (½ fractionated)

Number of runs (i.e., tests)

- 6 controllable factors (k = 6): x_1 thru x_6
- $-n = 2^{k-p} = 2^{6-2} = 16$ runs (about 3 weeks)

Response, y_i

- n = 16 values

$$y_i = \lambda = \frac{Q L_{avg}}{A \Delta T_{specimen}}$$

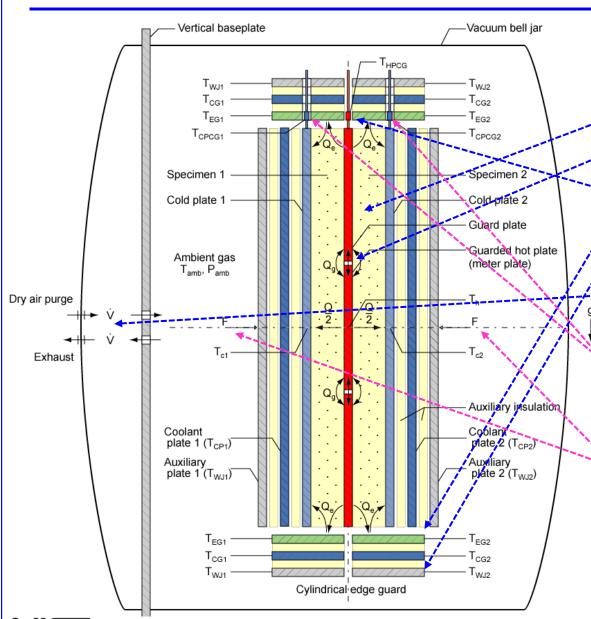
Underlying model (2⁶⁻²)

- 6 main effects (β_i)
- 15 two-term interactions (β_{ij})

$$y = \beta_0 + \frac{1}{2} \begin{bmatrix} \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_5 x_5 + \beta_6 x_6 + \\ \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{14} x_1 x_4 + \beta_{15} x_1 x_5 + \beta_{16} x_1 x_6 + \\ \beta_{23} x_2 x_3 + \beta_{24} x_2 x_4 + \beta_{25} x_2 x_5 + \beta_{26} x_2 x_6 + \\ \beta_{34} x_3 x_4 + \beta_{35} x_3 x_5 + \beta_{36} x_3 x_6 + \\ \beta_{45} x_4 x_5 + \beta_{46} x_4 x_6 + \\ \beta_{56} x_5 x_6 \end{bmatrix} + e$$

Factor Assignments

"All variables are *not* created equal; some can be varied more easily than others."
G.J. Hahn (1977)



Controlled factors

$$x_1 = \Delta T_{\text{specimen}}$$

$$x_2 = \Delta T_{\text{gap}}$$

$$x_3 = \Delta T_{\text{hot plate conn. guard}}$$

$$x_4 = \Delta T_{\text{edge guard}}$$

$$x_5 = \Delta T_{\text{water jacket}}$$

$$x_6 = V$$

Fixed factors

$$x_7 = \Delta T_{\text{cold plate conn. guard}} = 0 \text{ K}$$

$$x_8 = T_m = 310 \text{ K}$$

$$x_9 = L_{avg} = 26 \text{ mm}$$

$$x_{10} = F = 159 \text{ N} \implies 810 \text{ Pa}$$

Uncontrolled factors (recorded)

 x_{12} : T_{DP} : 205 K to 210 K

 x_{13} : T_{amb} : 293.1 K to 294.5 K

 x_{14} : P_{amb} : 99.22 kPa to 100.63 kPa

National Institute of Standards and Technology

Factors - Coded Settings and Descriptions

Factor	Coded settings				
Factor	-1 (low)	0 (center)	+1 (high)		
$x_1, \Delta T_{\text{specimen}}$	20 K	25 K	30 K		
$x_2, \Delta T_{gap}$	–0.25 K, guard cooler	0 K	+0.25 K, guard hotter		
x_3 , $\Delta T_{\text{hot-plate conn. guard}}$	–0.50 K, guard cooler	0 K	+0.50 K, guard hotter		
x_4 , $\Delta T_{\text{edge guard}}$	–2 K, guard cooler	0 K	+2 K, guard hotter		
$x_5, \Delta T_{\text{water jacket}}$	–2 K, guard cooler	0 K	+2 K, guard hotter		
x ₆ , [♥] (dry-air purge)	0 m ³ /h (Off)	0.7 m ³ /h	1.4 m ³ /h (Full open)		
x_7 , $\Delta T_{cold-plate\ conn.\ guard}$	0 K (fixed)				
$x_8, \Delta T_{mean}$	310 K (fixed)				
x ₉ , L _{avg}	26 mm (fixed)				
x ₁₀ , F	159 N (fixed)				

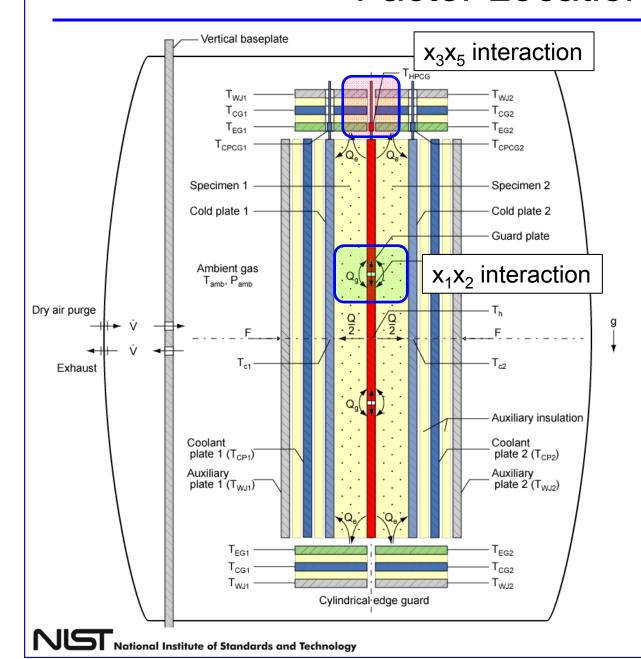
Results (Yates order)

Response, y

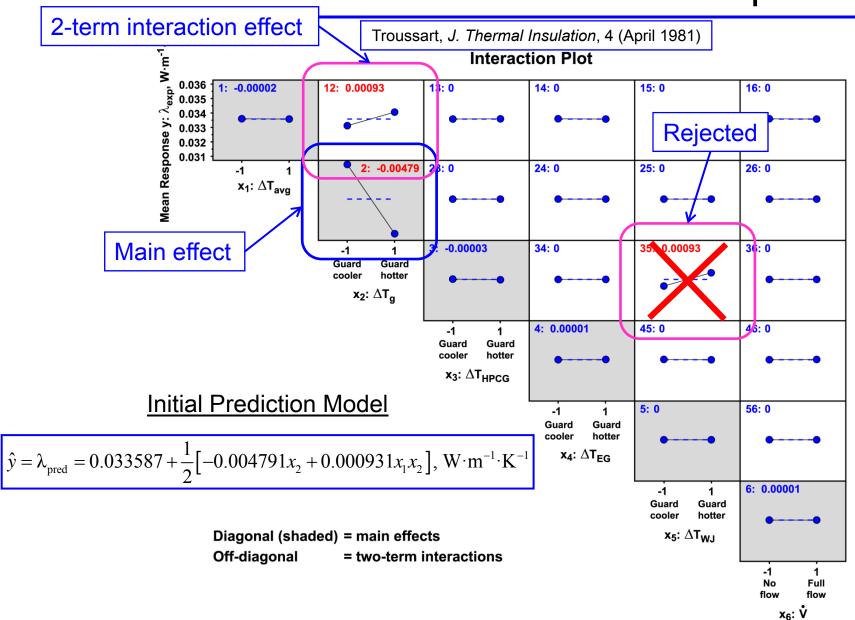
Run	<i>X</i> ₁ (ΔΤ _{spec.})	<i>X</i> ₂ (ΔT _{gap})	<i>X</i> ₃ (ΔΤ _{HPCG})	<i>X</i> ₄ (ΔΤ _{EG})	X ₅ (ΔT _{WJ})	<i>X</i> ₆ (V)	λ _{exp} W/(m·K)
1	_		_	1	_	1	0.03647
2	+	١	1	1	+	1	0.03552
3	_	+	1	1	+	+	0.03075
4	+	+	1	1	-	+	0.03166
5	_		+	1	+	+	0.03644
6	+	ı	+	1	+	+	0.03549
7	_	+	+	1	-	1	0.03071
8	+	+	+	1	-	1	0.03163
9	_		-	+	-	+	0.03648
10	+	ı	1	+	+	+	0.03552
11	_	+	_	+	+	1	0.03076
12	+	+	_	+	_	_	0.03166
13	_		+	+	+	_	0.03644
14	+		+	+	_	_	0.03549
15	_	+	+	+	_	+	0.03073
16	+	+	+	+	+	+	0.03164

7

Factor Locations



Main Effects and Interaction Multi-plot



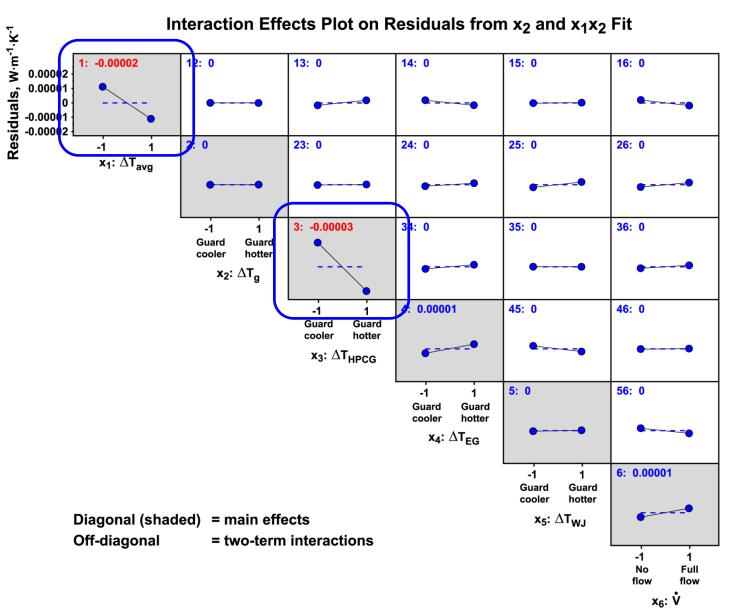
Further Examination of Data

- The effect of dominant factors in a sensitivity study
 - Factor x₂: ΔT_g and the interaction term x₁x₂ dominate the other effects by several orders of magnitude
 - When one (or two) factor dominants, it is prudent to check the other factors thoroughly for significance and possible inclusion in the predictive model
 - Method:
 - 1) Yates analysis; and,
 - 2) Graphical interaction plot on the residuals from a model including only the obviously dominant x_2 and x_1x_2 terms
- Yates analysis least squares fit of an additive model consisting of:
 - Main effects (6)
 - Appropriate interaction terms

Yates Analysis

Identifier	Estimate (W·m ⁻¹ ·K ⁻¹)	<i>t</i> -value	S _{res} (W·m⁻¹·K⁻¹)	
Mean	0.033587		0.002520	
2	-0.004791	-1377.7	0.000498	
12	0.000931	267.9	0.000023	
3	-0.000034	-9.7	0.000015	
1	-0.000022	-6.4	0.000007	
4	0.0000065	1.9	0.0000063	
6	0.0000063	1.8	0.0000051	
16	-0.0000038	-1.1	0.0000047	
13	0.0000036	1	0.0000041	
14	-0.0000034	-1	0.0000035	
34	0.0000030	0.9	0.0000028	
24	0.0000020	0.6	0.0000024	
124	-0.0000017	-0.5	0.0000020	
134	0.0000015	0.4	0.0000010	
5	0.0000006	0.2	0.0000008	
23	0.0000004	0.1	0.0000000	

Interaction Effects Plot on Residuals from Fit of x_2 and x_1x_2



12

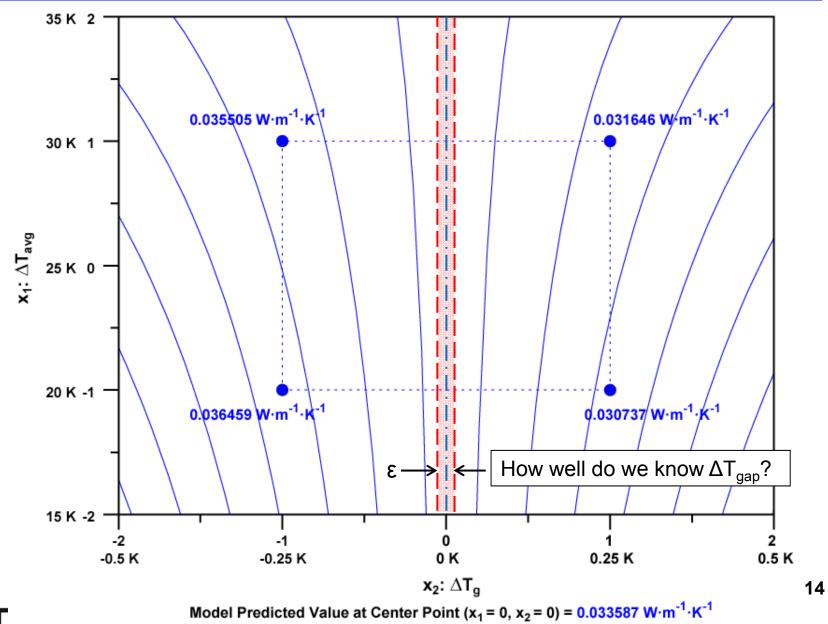
Discussion

Final Prediction Model

$$\hat{y} = \lambda_{\text{pred}} = 0.033587 + \frac{1}{2} \left[-0.004791 x_2 + 0.000931 x_1 x_2 - 0.000034 x_3 - 0.000022 x_1 \right], \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$$

- where x_i are coded (-1 or +1)
- Guard gap imbalance most important; interaction with ΔT also important
- Localized heat flows can be important → Is active guarding of sensor leads required?
- Suggests that ΔT should be specified in inter-laboratory comparisons (and standard test methods)
- Example typical operating conditions:
 - $x_2 = x_3 = 0$
 - Let $x_1 = -1$ (20 K); $\lambda_{pred} = 0.033587 + 0.000022 = 0.033609 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$
 - Let x_1 = +1 (30 K); λ_{pred} = 0.033587 0.000022 = 0.033565 W·m⁻¹·K⁻¹

Contour Plot of Dominant Factors (x_2 : ΔT_g and x_1 : ΔT_{spec})



Sensitivity Study Conclusions

Factor rank (most important)

- 1) Main effect: x_2 : (ΔT_{qap})
- 2) Two-term interaction: x_1x_2 : ($\Delta T_{\text{specimen}} \Delta T_{\text{gap}}$)
- Two other small main effects
 - 3) x_3 : ($\Delta T_{\text{hot plate conn. guard}}$) local heat flow
 - 4) x_1 : ($\Delta T_{\text{specimen}}$)

Results valid over:

- Range of the varied factors (x_1 thru x_6)
- Fixed setting of other factors (x_7 thru x_{11})
 - o $T_m = 310 \text{ K}$
 - \circ L = 26 mm
 - o Material: fibrous-glass board

Future work at NIST:

- Additional sensitivity study tests (staged as time permits) are recommended for other temperatures, materials, and thicknesses
- Publish manuscript in ASTM JOTE