



### DOE for Ruggedness Testing

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#### **Talking Points**



- ➤ Design of experiment (DOE) tools provide confidence that newly developed systems will withstand multifactor field conditions.
- ➤ The ASTM E1169 Standard Practice for Conducting Ruggedness Tests provides a tried-and-true protocol, not just for methods, but for processes and products.
- ➤ Low-resolution designs such as Plackett-Burmans, provide a go/no-go test based on <u>practical importance</u> of the observed effects (significance not an issue initially).
- ➤ Foldovers deliver a way forward for resolving what causes significant failures in ruggedness.

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#### Agenda



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- Ruggedness testing methodology
- Moisture method mess-up
- ➤ Memory wire fails on basis of range
- > Foldover reveals pH sensor issues
- > Take-homes

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#### Ruggedness Testing Methodology\*



- ➤ Vary key factors over ranges that are expected to be encountered during normal use of a "system" (process, product, method...).
- > This application of DOE boils down to a go/no-go test.
- > Ideally:
  - ➤ None of the factors emerge statistically significant, with
  - > All potential effects being unimportant.
- \*(Introduced by W. J. Youden & E. H. Steiner, *Statistical Manual of the AOAC*, Association of Official Analytical Chemists, Washington D.C., 1975.)

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# ASTM E1169 Standard Practice for Conducting Ruggedness Tests



ASTM E1169 advocates Plackett-Burman designs (PB's) for:

- ✓ Simplicity Two levels only
- ✓ Flexibility Multiples of 4 rather than standard powers of 2
- ✓ Efficiency Minimum runs (Resolution III) suffice for a pass/fail test on up to N-1 factors (saturated)
- √ Adaptability Fold-over resolves main effects (III => IV)
- ✓ Convenience Can easily lay out by hand from seed rows (std run 1):

```
 \begin{array}{lll} N=&8^*&+1,+1,+1,-1,+1,-1,+1&& (\underline{2^{nd}\ row}:+1,\,+1,+1,+1,-1,+1,-1)\\ N=&12&+1,+1,-1,+1,+1,+1,-1,-1,-1,+1,-1\\ N=&16^*&+1,+1,+1,+1,+1,-1,+1,+1,-1,-1,+1,-1,-1,-1\\ N=&20&+1,+1,-1,-1,+1,+1,+1,+1,+1,-1,+1,-1,+1,-1,-1,-1,+1,+1,-1\\ Second row shifts one place to the right, rotating in the last sign to the beginning. Continue cycling N-2 times until reaching the final row—this being all minus. \\ *PS: "Geometric" PBs—N being a power of 2, are equivalent to <math>2^{k\cdot p} factorials. \\ \end{array}
```

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## Story Behind Plackett and Burman



Plackett and Burman invented their experiment designs during WWII for development of proximity fuses for anti-aircraft shells.

The British Ministry of Supply kept them secluded in a Scottish castle under guard. Data would come to the castle and matrices would come out to be delivered to engineers in manufacturing. The story was that this effort was as secret as the storied Ultra secret code-breaking effort.

The PB designs remained classified until after the war when in 1946 the inventors published "The Design of Optimum Multifactorial Experiments" in *Biometrika* (33, 305-325).

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### Moisture Method Mess-Up



During manufacturing startup of a new adhesive, <u>all</u> the product failed due to high moisture—per tests from the plant QC testing. R&D engineers were called in to fix the process. When they set samples back to the Analytical lab, results showed all moistures in spec! Although too late for all the product dumped, the engineers ran a ruggedness test on the moisture method as done by QC (low) versus what Analytical did:

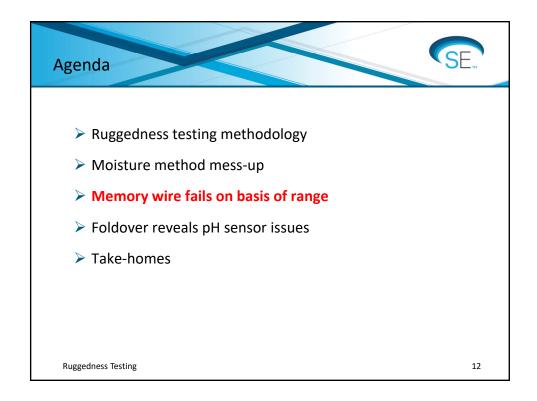
Factor	Units	Low Level (-)	High Level (+)
Reagent		used	new
Reac time	min	0	15
n-Heptane	ml	190	210
Dis time	min	45	90
Dis rate	drops/sec	2	6
Aniline	ml	8	12
Hydration		ca 2	ca 5

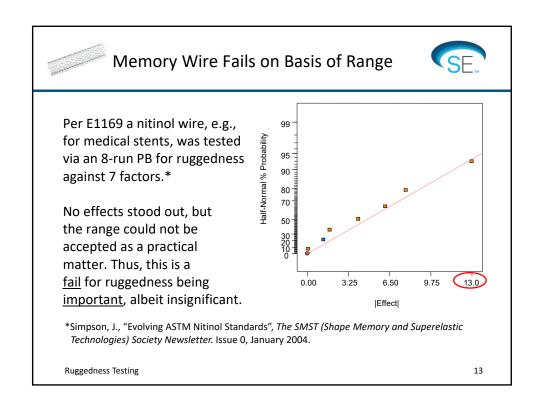
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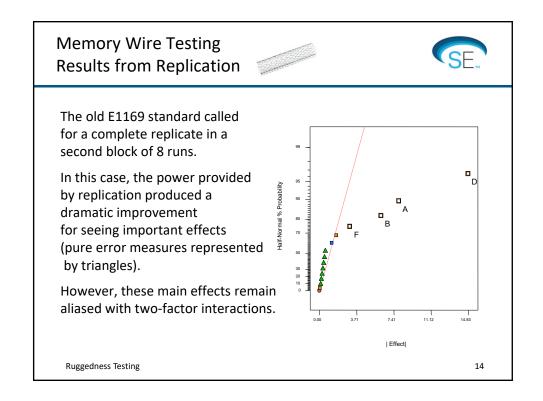
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#### Moisture Method Ruggedness Test Results per Half-Normal Plot of Effects Half-Normal Plot Shapiro-Wilk test W-value = 0.882 p-value = 0.280 A: Reagent B: Reac time 99 [A] Half-Normal % Probability 95 90 Fail! Effect too important to ignore. To save money, QC recycled the 80 70 reagent (factor A), not knowing it azeotroped during distillation, thus 50 re-introducing water. Oops! But this remains provisional due to A 10 being aliased with BD+CE+FG. 0.15 0.61 |Effect| Ruggedness Testing

#### Example of Complete Foldover\* \*(easily done with Design-Expert® software Design Tools ©) Std Blk Α В c D G [A]=A+BD+CE+FG (Res III) used Upgraded to new [A]=A+BCG+BEF+CDF+DEG used Results confirm A (reagent) new used being the culprit used new new A-Reagent used new used used new used Ruggedness Testing







#### Aliasing in PB Design Unresolved by Replication



$$[A] = A - BF - CD - EG$$

$$[B] = B - AF - CG - DE$$

$$[D] = D - AC - BE - FG$$

$$[F] = F - AB - CE - DG$$

Could the smallest effect [F] be [AB], a good possibility based on heredity? Or perhaps correct model may be some other combination of main effects and interactions. There's no way to know due to aliasing! A foldover would have been a far better investment in 8 more runs than a simple rep.

PS: The current Ell69 protocol calls for foldover, not replication. ©

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# Re Ruggedness Test Outcome: Importance vs Significance



#### Many are unclear on this difference!

		Significant	
		No	<u>Yes</u>
tant	No	<b>©</b>	<b>=</b>
Important	<u>Yes</u>	<b>⊗</b>	\!/ ⊗

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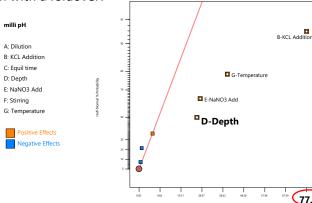
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#### Foldover Reveals pH Sensor Issues Results from First Block of Runs



Results from the first block of 8 runs on 7 factors show that this instrument is not rugged to field conditions—too broad a range.

Continue on with a foldover.

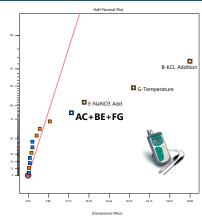


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#### Foldover Reveals pH Sensor Issues Second Block of Runs Dealiases Main Effects



Main effect of D is now revealed to be its aliased interaction AC+BE+FG. A semifold\* (easily done with Design-Expert's Design Tools) would nail this down—most likely to BE based on heredity (B and E being main effects).



\*Anderson & Whitcomb, "How To Save Runs, Yet Reveal Breakthrough Interactions by Doing Only A Semifoldover on Medium-Resolution Screening Designs", 55<sup>th</sup> Annual Quality Congress, ASQ, https://cdnm.statease.com/pubs/semifold.pdf.

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# Take-Homes DOE for Ruggedness Testing



- ➤ Design of experiment (DOE) tools provide confidence that newly developed systems will withstand multifactor field conditions.
- The ASTM E1169 Standard Practice for Conducting Ruggedness Tests provides a tried-and-true protocol, not just for methods, but for processes and products.
- Low-resolution designs such as Plackett-Burmans, provide a go/no-go test based on <u>practical importance</u> of the observed effects (significance not an issue initially). My preference is the 12-run option being more powerful than only doing 8.
- Foldovers deliver a way forward for resolving what causes significant failures in ruggedness.



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