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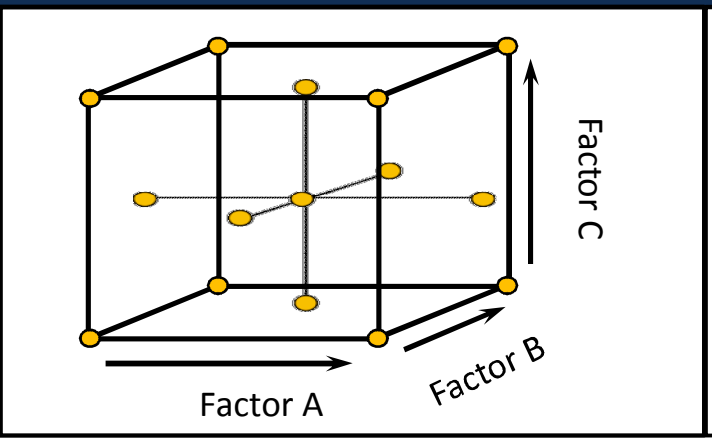

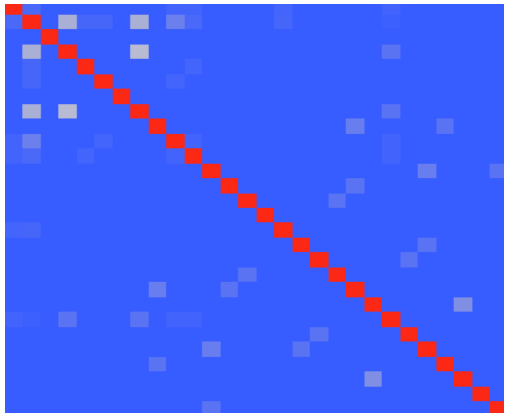

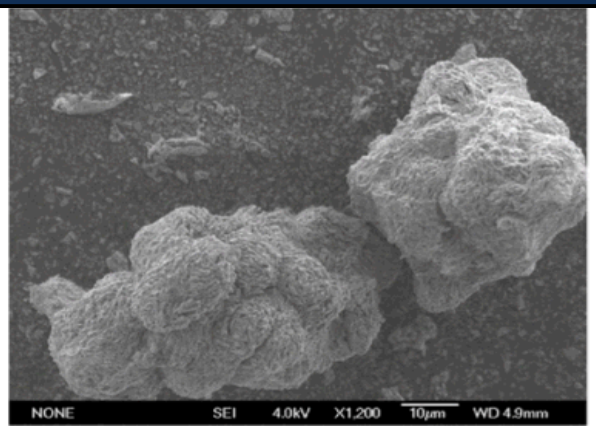

Experimental Design

NTNFC Monthly Webinar Series

John Lewis, Adah Zhang – SNL

Christine Anderson-Cook – LANL

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Seed Questions

1. How do we infer that there are missing/more important signatures from our experiments? What in our experimental design might lead us to make such an inference?
2. How do we design experiments to optimally identify connection between process variable(s) and product observable(s)?
3. What is the “right” mix between experimentalists, theoreticians, and production experts to develop the experimental approach to signature identification?
4. Within the context of our limited laboratory experimental scope, how do we address the changes that occur in a process when going from lab scale work to production scale work?
5. How might we approach the experimental design of an SEM round-robin to calibrate our instruments across our enterprise and standardize the results and reporting thereof?
6. What approaches to experimental design are applied by our laboratories and how might we optimize our experimental work?

Purpose of Talk

- Statistical perspective on experimental design
  - General steps to design an experiment
  - Contributions/responsibilities of all parties:
    - SME's
    - Experimentalists
    - Statisticians

- Connection to seed questions
  - Some guidance to help answer them
  - Definitive answers not necessarily available

What is a Designed Experiment?

Planned series of tests where systematic changes are made to inputs **so we can identify sources of change in outputs**

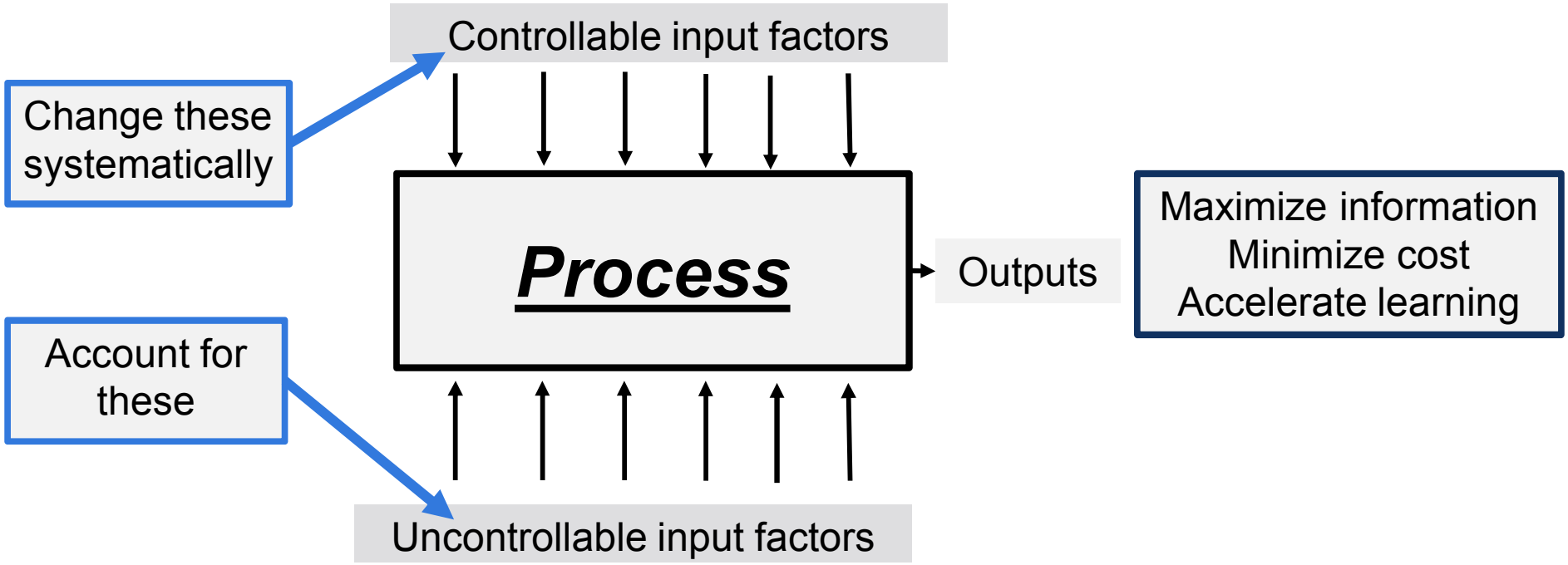


Figure adapted from: Montgomery, D.C. Design and analysis of Experiments

What is a Designed Experiment?

- Ultimately – it's a list of tests in a certain order

Test (Run)	Factor 1	Factor 2	Factor 3	….
1	Level 1	Level 1	Level 1	….
2	Level 1	Level 2	Level 2	….
3	Level 2	Level 2	Level 1	….
4	Level 1	Level 1	Level 1	….
….	….	….	….	….

- Chosen to match the objectives
- Order matters: often randomized to mitigate unknown effects
- Can be run sequentially
  - Information from first set of tests can inform the choice of a next set of tests

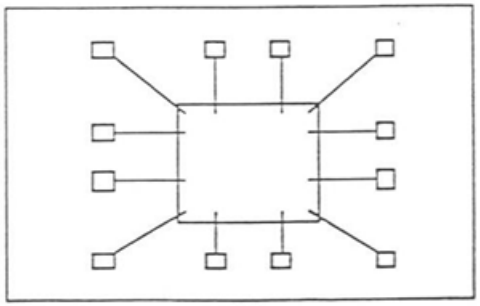
All Experiments are Designed…

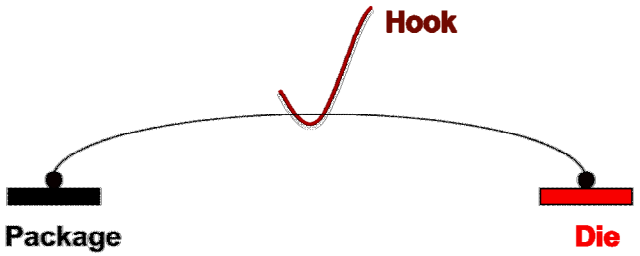
Some poorly …

Example of a poorly designed experiment

Wire Bonding (WB) of Integrated Circuits (IC)

- Goal/Objective: Determine the sources of variation in bond strength





Data for ***11,000*** pull tests across many

- Devices
- Operators doing the wire bonding
- Operator doing the pull test

Poorly Designed Experiment Example

- **Experimental Protocol**
  - Operators chose their bonding machine (each had favorites)
  - They pull-tested the wires they themselves bonded
- **Experiment did not control the bonder, bonding machine, or puller.**
- **Observed differences in bond strength could be due to:**
  1. Bonding operator
  2. Testing/Pulling operator
  3. Bonding machine
  4. A combination of the above three
- *Follow-up designed experiment de-confounded effects with ~75% less data*
- Risks of poorly designed experiments
  - extra cost, confounding,…

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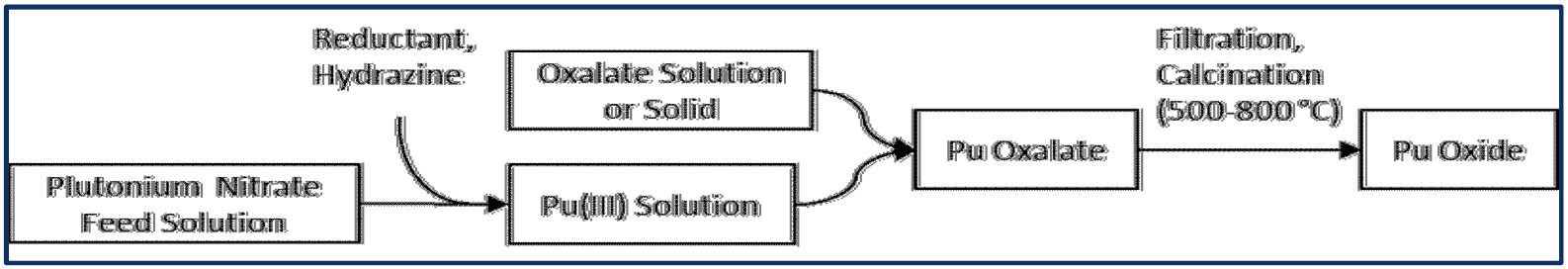
General Steps to take when designing an experiment…

Designing Experiments

1. Clearly define objectives

Example: Pu (III) Oxalate Bench-Scale Study



- Identify signatures of nuclear forensic value that can be related to the processing conditions used to produce them
- Inform choice of processing conditions for the large-scale study

Designing Experiments

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2. List response variables and factors

Factors

Oxalate feed:
Solution (0.9M) & Solid

Reagent add./dig. timing:
0/40, 20/20, 40/0

Nitric acid concentration:
1M, 2M, 3M

Mixing sequence:
Direct & Reverse

Precipitation Temperature:
30°C & 50°C

Feed concentration of Pu:
10g/L, 30g/L, 50g/L

Responses

Many – some not known yet.
Size distributions (SEM)
XRD strain
XRD crystallite size
Pycnom density
…

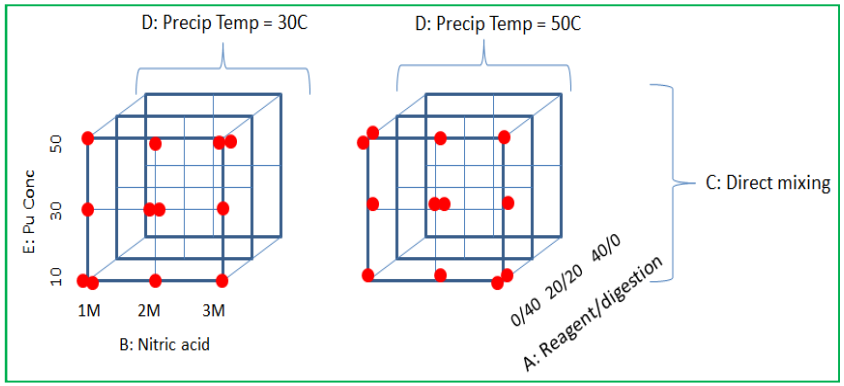
Considerations

- Final list of factors chosen from much larger list
  - Expert opinion, costs, goals – trying to do too much can be detrimental
- Controllability of the factors (both varying and fixed)
- # of levels to use
- Ability to measure (e.g., SEM's)

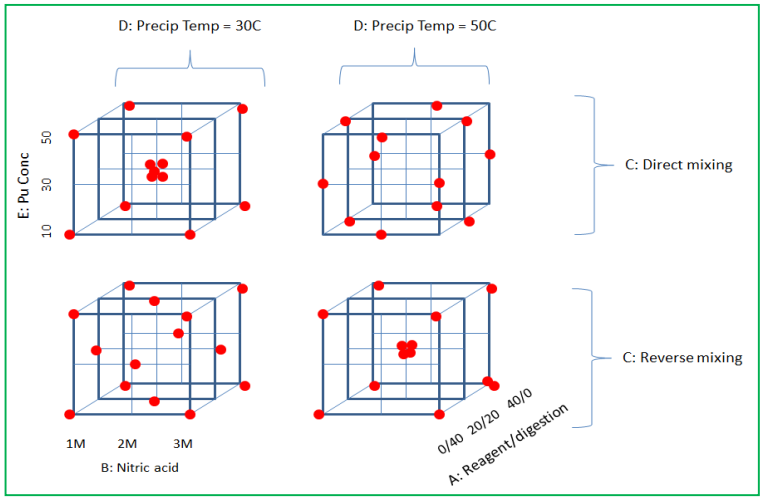
Designing Experiments

3. Design the Experiment

Oxalate Feed Solid



Oxalate feed in solution



- Connect objectives with statistical theory
  - I-optimality – minimize average prediction variance
- Some good properties
  - Tests spread across space
  - Replicates

Considerations

- Run order
  - Material Availability

Connections to seed questions 2 and 3

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2. How to design experiments to optimally identify connection between process variable(s) and product observable(s)?

3. What is the "right" mix between experimentalists, theoreticians, and production experts to develop the experimental approach to signature identification?

- Modified terminology – SME, experimentalists, and statisticians

How to? And Right 'Mix'?

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1. Clearly defined objectives

SME/Experimentalists

- Brings problem/goals to table

Statisticians

- Maps goals to statistical objectives - what data is needed to achieve the objectives?

How to? And Right 'Mix'?

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2. List response variables and factors

SME/Experimentalists

- Creates these (usually long) lists
- Prioritized factors using perceived effects

Statisticians

- # of levels needed
- sample size

How to? And Right 'Mix'?

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3. Design the Experiment

SME/Experimentalists

- Constraints?
- Feasibility of proposed designs

Statisticians

- Objective based (e.g. optimal design)
- Provide options: evaluate pros/cons

Iterative Nature of Experimentation

Iterative Nature of Experiments

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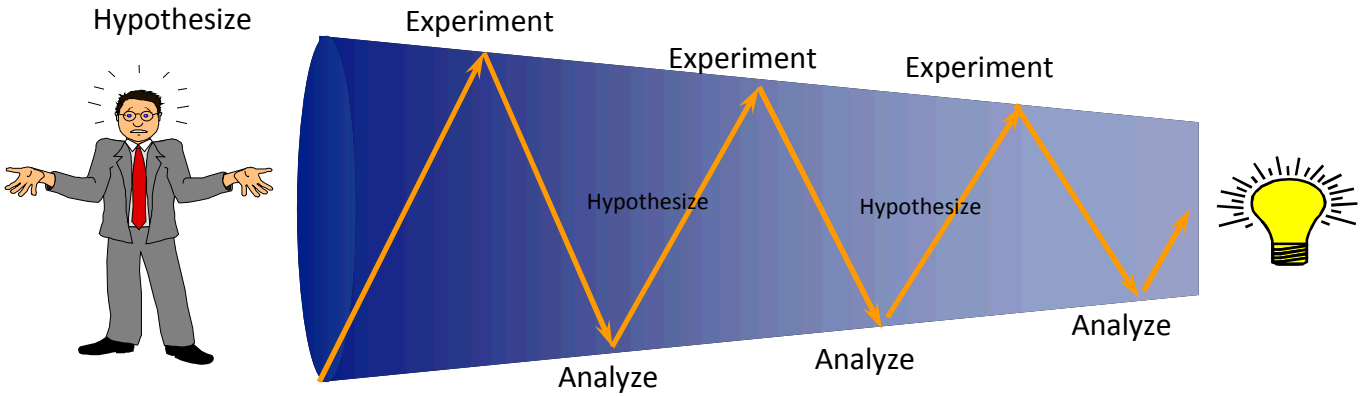
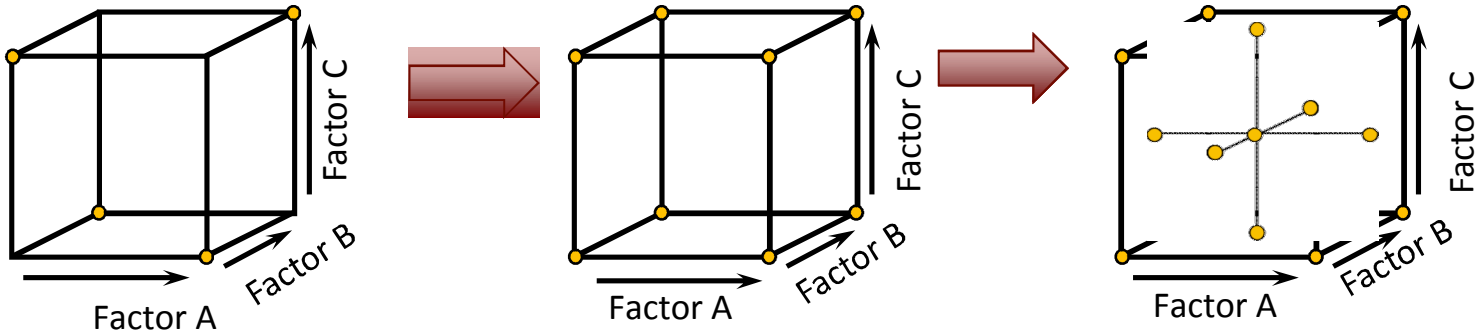
Experimentation is part of *The Scientific Method*



Smaller experimental efforts precede and inform larger efforts



Narrow down long list of potential factors

Understand large directional effects of each variable

Pin down input-output relationship further

If feasible: results from each new test can be used to choose the next test (sequential designs)

Seed Question 4

4. Within the context of our limited laboratory experimental scope, how do we address the changes that occur in a process when going from lab scale work to production scale work?

Bench-Scale vs. Lab-Scale

Approach being taken in Pu Signatures Project

Pu (III) Oxalate Bench-Scale Study

- 76-run design, ~10g per test
- Understand as much as possible

Informs the larger Lab-scale study

- Smaller set of runs
- ~200g per test

Compare

- Look for empirical differences
- Match to what science suggests

Iterative approach is the only feasible approach

Seed Question 1

How do we infer that there are missing/more important signatures from our experiments? What in our experimental design might lead us to make such an inference?

Note on terminology

- Signature = set of measurements with NF value

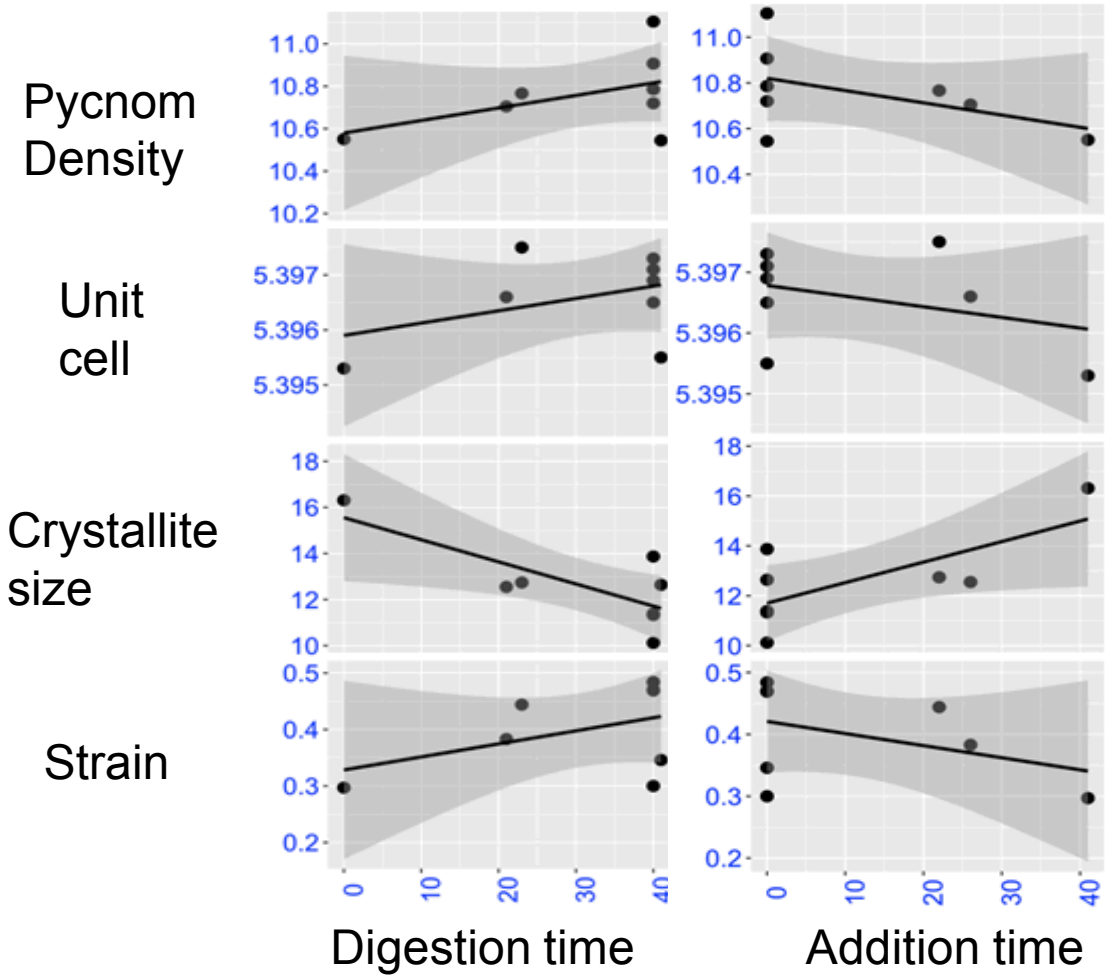
We cannot evaluate if we don't have data

Wise to start with as rich a set of responses as possible

- Range of factors, responses, using principled DoEx
- Can always down-select later

Data Analysis

- Important measurements/signatures have strong relationships with the processing parameters



First set of data from Pu Bench Scale Study:

- Strong relationships with digestion and addition time?
- Flat lines would indicate little or no linear relationship
- Still too early to be confident

Data Analysis

- Multiple measurements in combination can be more informative than individual measurements

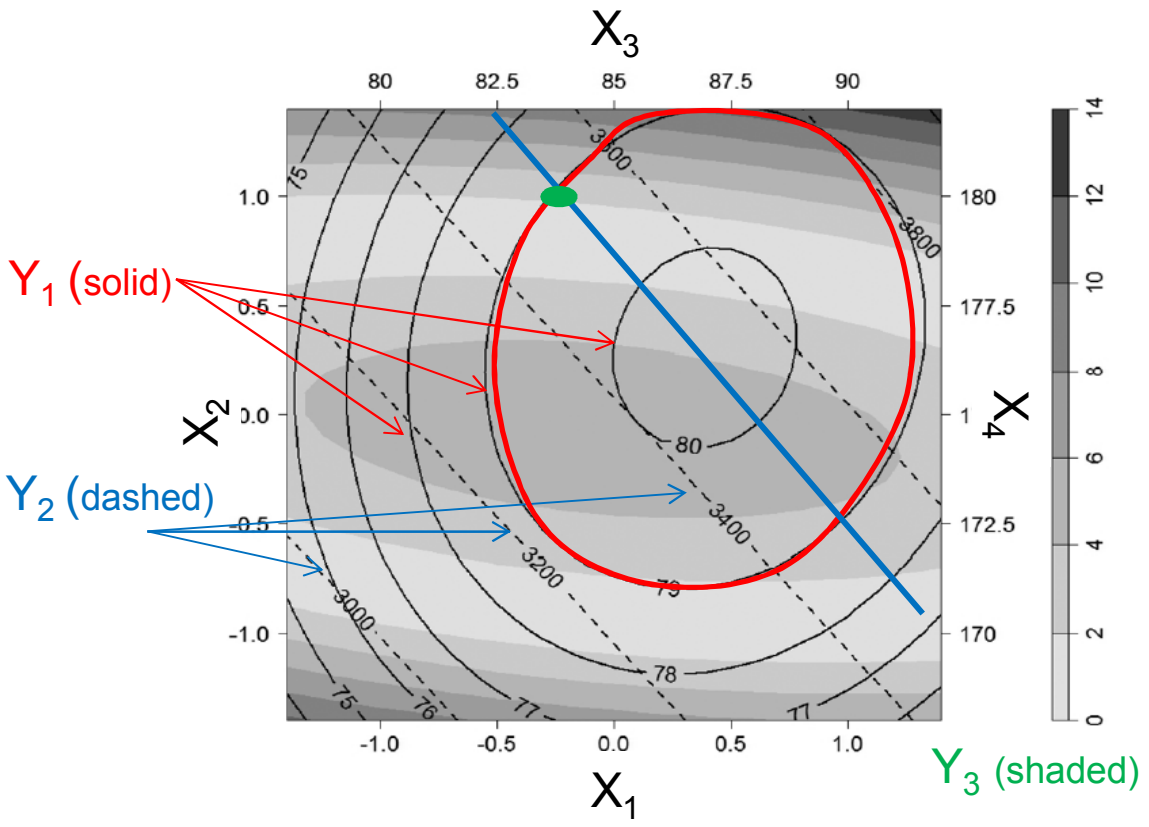
Need enough measurements to obtain unique solutions

Models calibrated with initial experiment:

$Y_1 \approx f_1(X_1,\ldots,X_4)$
$Y_2 \approx f_2(X_1,\ldots,X_4)$
$Y_3 \approx f_3(X_1,\ldots,X_4)$

Example:
New observation
$(Y_1,Y_2,Y_3) = (79,3500,2)$

Suggests $(X_1,X_2,X_3,X_4) \approx$
$(-0.2,1.0,84,180)$

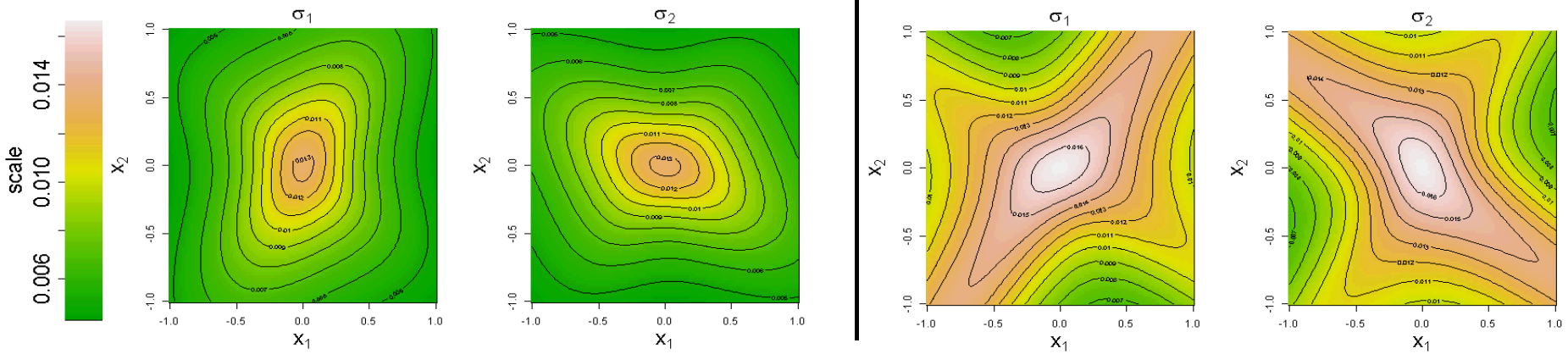


Data Analysis

- Multiple measurements in combination can be more informative than individual measurements

Don't necessarily need *all* measurements

$S = \{1,2,\ldots,16\}$ $S = \{9,10,11,12\}$



- Prediction variance under different signature sets
- Average variance 1.6x larger – but with ¼ of the measurements.

E.V. Thomas, J.R. Lewis, C.M. Anderson-Cook, T. Burr, M.S. Hamada, Selecting an informative/discriminating multivariate response for inverse prediction. (2017). Journal of Quality Technology.

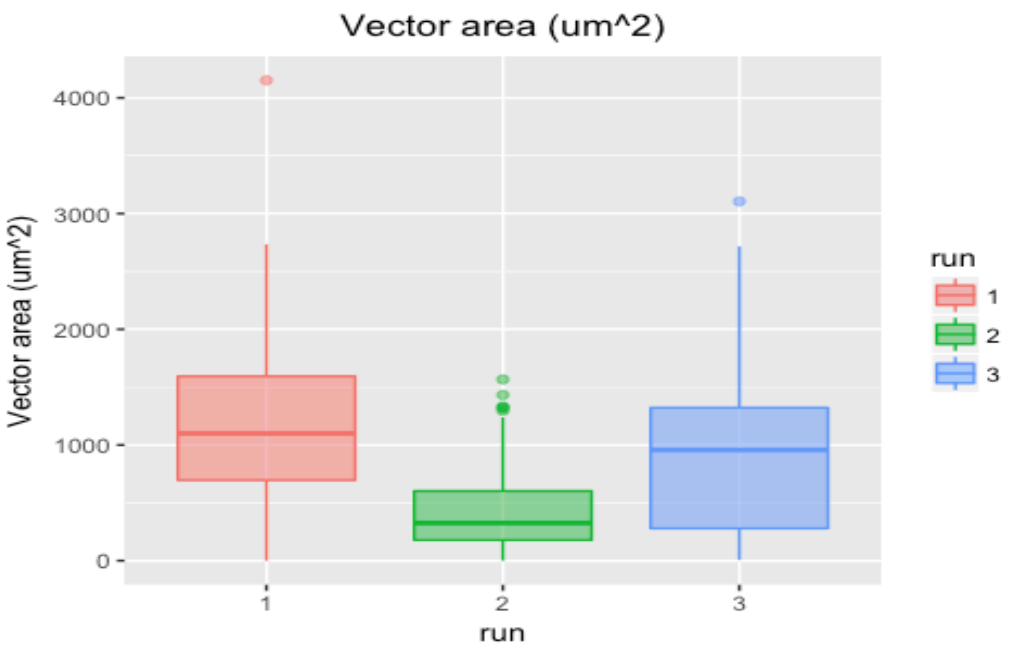
Summary of Question 1

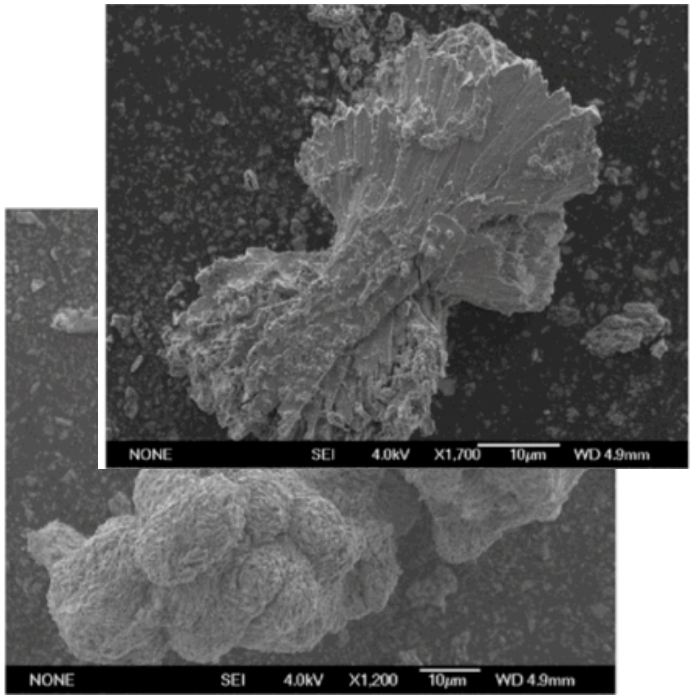
- Analysis of experimental data can:
  - Identify empirical relationships for the inverse prediction
  - Identify important measurements/signatures
- Poor predictive performance?
  - Could be a sign of missing measurements/signatures
  - Requires SMEs to relate empirical results to scientific knowledge
- Experimental design is important
  - Better design -> better information from data analysis
- Design philosophy for signature discovery
  - Span factor space where inverse model predictions are to be applied
  - Allow development of accurate models of the relationships

Seed Question 5

5. How might we approach the experimental design of an SEM round-robin to calibrate our instruments across our enterprise and standardize the results and reporting thereof?

SEM Round-Robin Studies





Are the differences we see across runs due to:

- varying processing conditions?
- the measurement system?

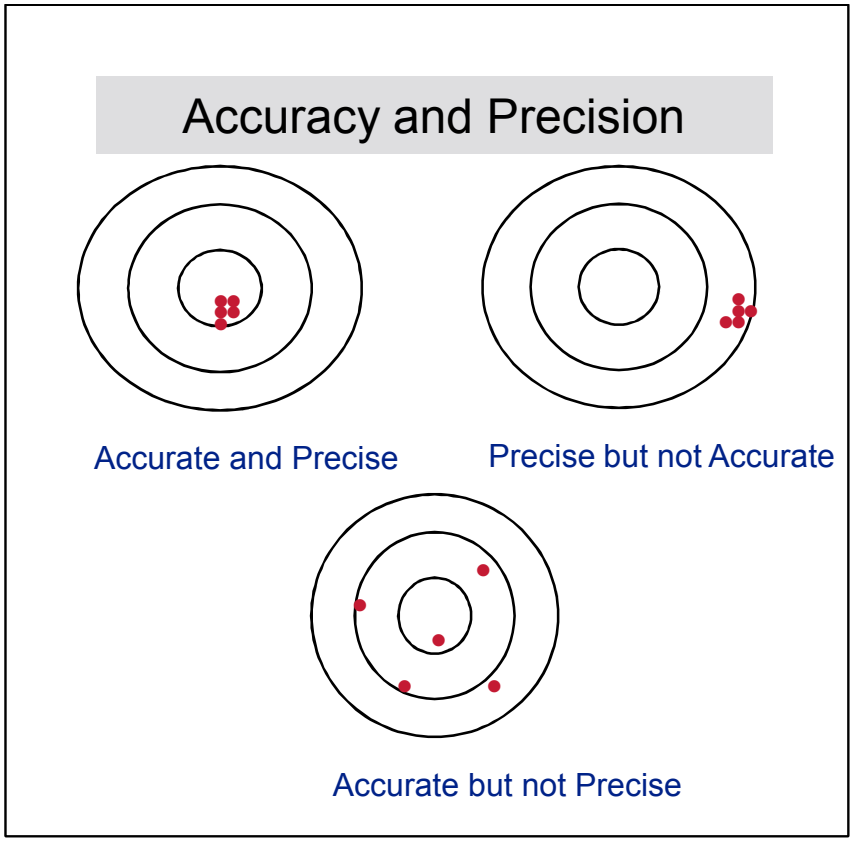
→

DoEx principles can be utilized

Measurement Capability Studies

Essential Question: Can we measure what we want to measure precisely/accurately enough?

- Two sources of variation in measurements
  - Differences caused by treatments
  - **Imperfections in taking measurements**
- Objectives:
  - To understand the capability of a *measurement system*
  - Identify factors that influence the measurement performance

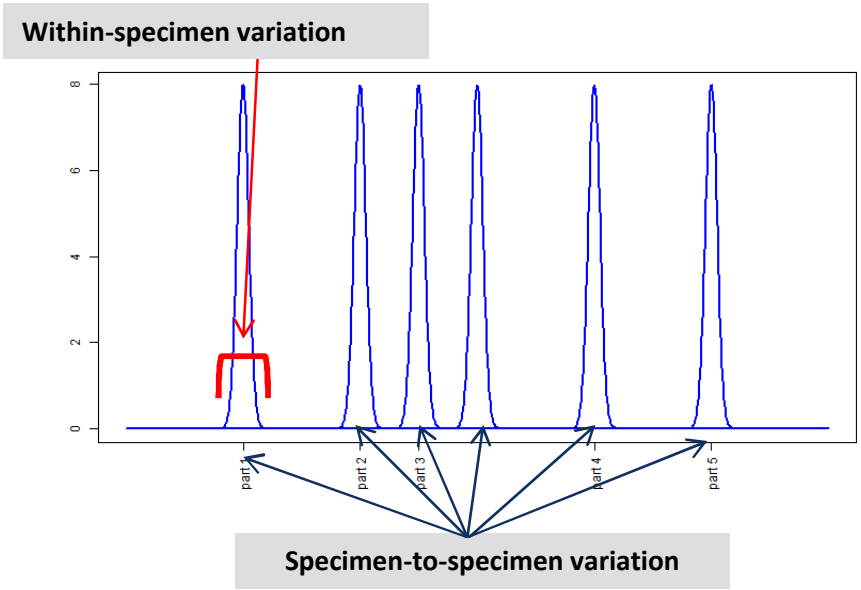


Example: R&R Studies

Objective

Determine how much of the observed variation is due to the measurement system (e. g.: SEM, MAMA software, etc.)

- DoEx: full factorial experiment with factors
  - Pu Specimen
  - Operators (Could be sample preparer, SEM operator, or a combination)
- Replication is needed: each operator measures each specimen multiple times



Idea

- Want to attribute specimen-to-specimen variation to production parameters.
- Need within-specimen variation to be small compared to s specimen-to-specimen variation.

R&R = Repeatability and reproducibility

Summary

- Experimental design is a collaborative process
  - SME/Experimenters have valuable domain knowledge
  - Statisticians have valuable design and statistical knowledge
- Designs should be tailored to the objectives of the study
- Seed questions: Provided at least partial answers to all but one
  - 6. What approaches to experimental design are applied by our laboratories?…
    - We are not sure…
  - and how might we optimize our experimental work?
    - At least one piece of this optimization should be the use of principled experimental design
    - Webinar was meant as a very quick intro to this topic

Back up slides

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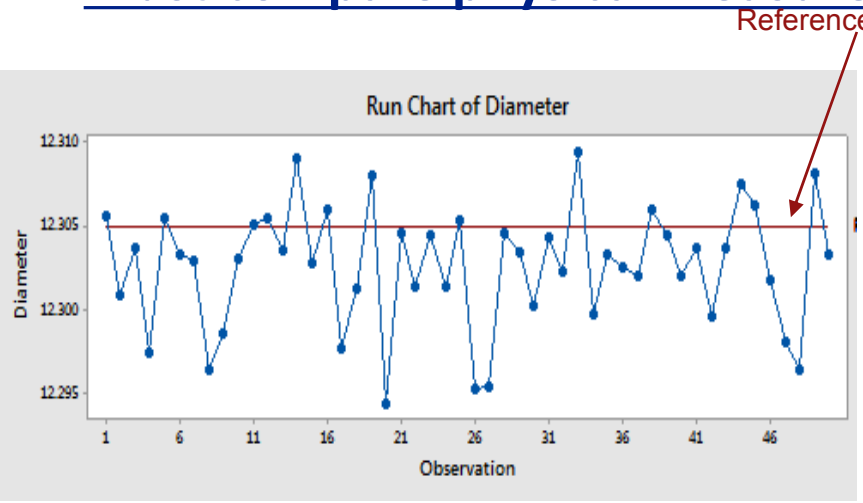
Accuracy

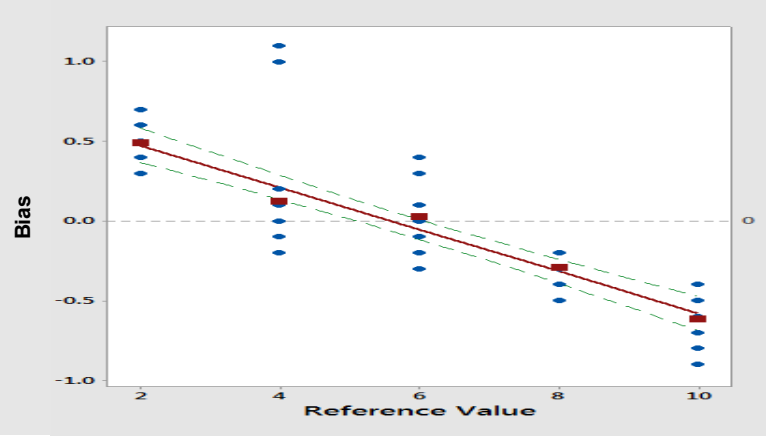
Accuracy: difference between the measurement and the true value.

Components of accuracy

- **Bias -** The difference between the observed average measurement and a master (true) value.
- **Linearity -** Measure of how the size of the part affects the accuracy of the measurement system. It is the difference in the observed accuracy values through the expected range of measurements.

Must compare physical measurements to know reference values





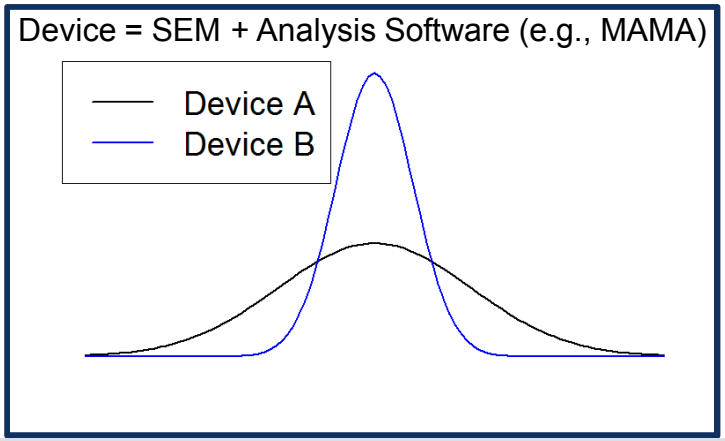
Leads to the question: are there such reference materials for SEM measurements?

Precision

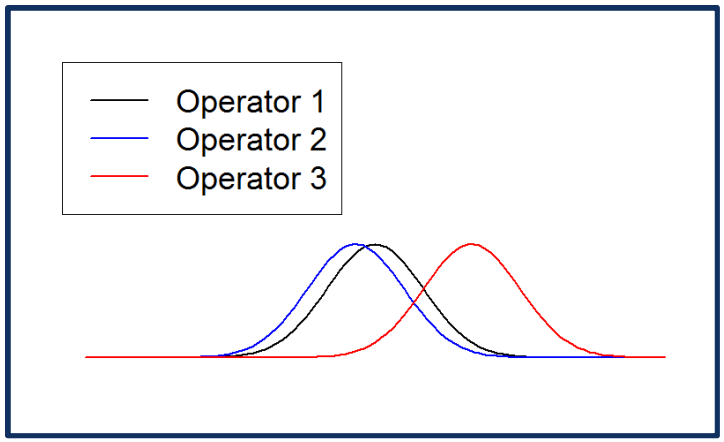
Precision: variation when you measure the same sample repeatedly with the same device.

Components of Precision in SEM measurements

- **Repeatability**- Variation that occurs when the same SEM operator/sample preparer repeatedly implements the entire measurement process with the same tools (sample prep, SEM, MAMA software)
- **Reproducibility** - Variation that occurs when different SEM operators/sample preparers implement the entire measurement process with the same tools.



Same operator measures the same sample several times with two different SEM devices - device B is more repeatable than device A because it has less variation



Several operators measure the same sample several times. Variation in average measurements between operators 1 and 2 is much less than between operators 1 and 3 – The device demonstrates poor reproducibility