# Quality Systems:
# Risk and Maturation of Technology

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20221018

**Outline-**

- What is Quality?
- Intentional vs. unintentional variability
- Confident decision making
- Risk assessment & mitigation
- Drivers of quality
- Case studies
- Additional references

# What is Quality?

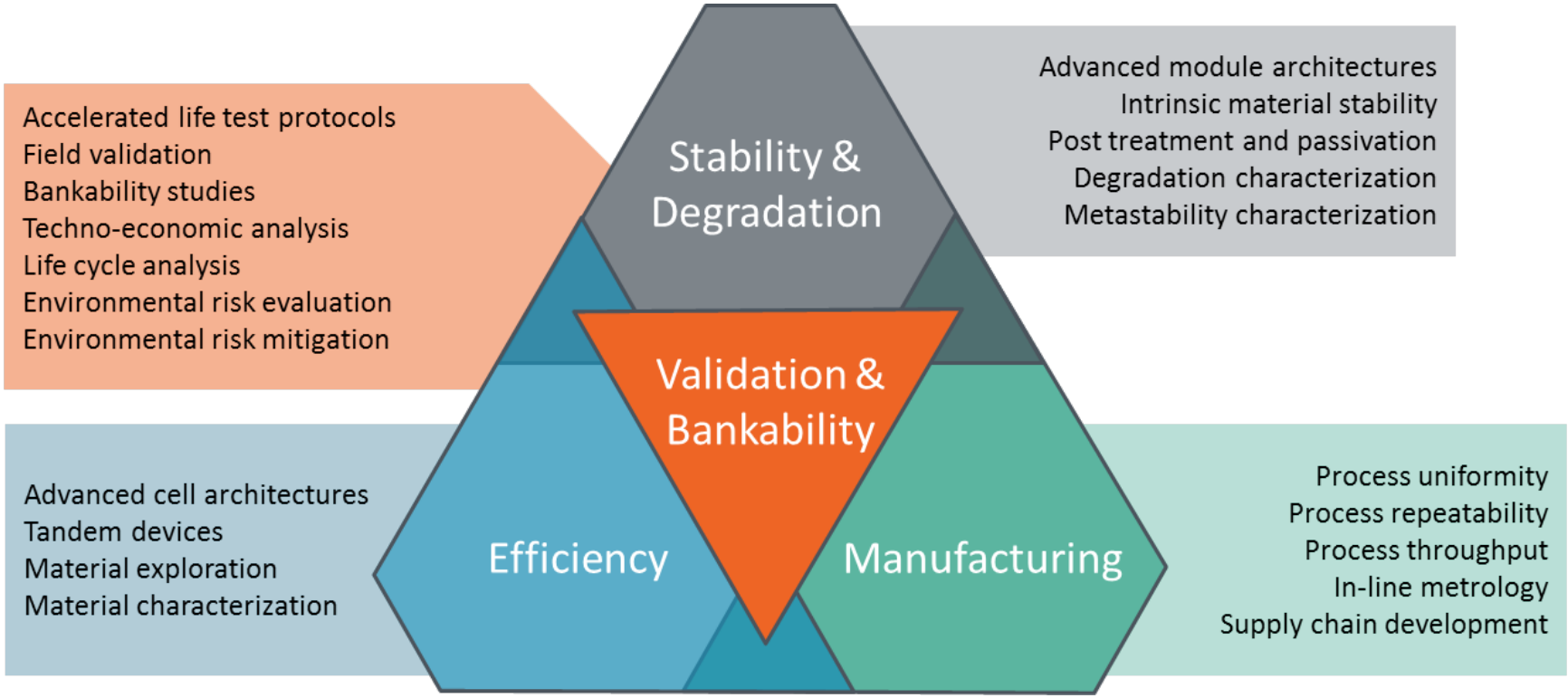
"***Quality is a fitness for use.***"
— Juran

# What is Perovskite PV Quality?

Efficient Durable Environmentally Benign Bankable

**meets expectations / no surprises**

# Perovskite RD&D Challenges

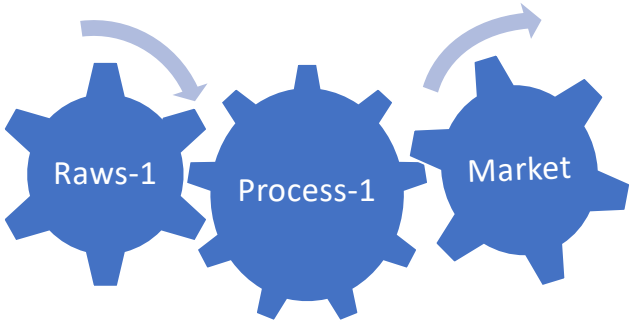



**All challenges must be simultaneously met for commercial competitiveness**

# Quality Toolbox

- Quality is naively engineered late in development
- Development makes early decisions in tools, sourcing, formulation and process, and sees the process can (mis)behaviors (e.g., defects, cliffs etc.)- Early quality aids decision making
- Project/Program management
  - Theory of Constraints, buffer chart
  - Team roles, “distraction factors”
  - Effective meetings, deliverables, collaboration
  - **“Copy Exactly!”** no improvements until baseline is stable.
- Risk Assessment & Mitigation Methodology
  - Cradle-to-grave version control
    - Raws, intermediates, process steps, documents, specs, recipes, “bluebook”
  - ConOps, RA matrix, FMEA, FEA
  - Design of Experiments, SPC, Structured Problem Solving
  - Tool & process matching, “Round-Robin”
  - Testing: In-line/off-line, reliability/accelerated reliability

The Quality Management Plan is a self-improving system

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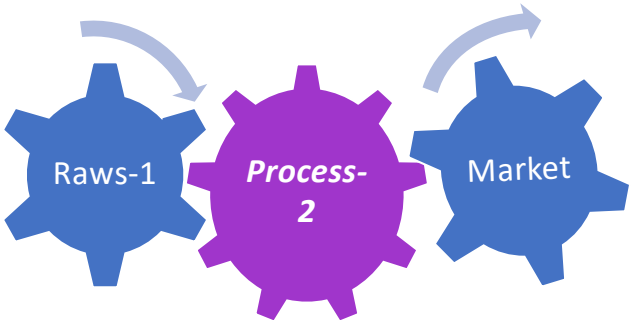
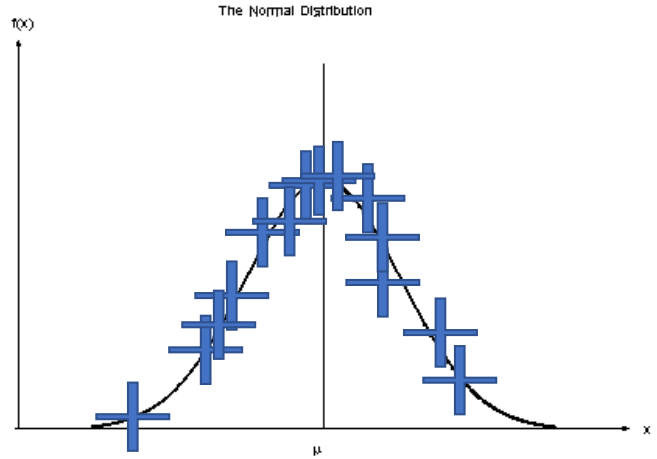
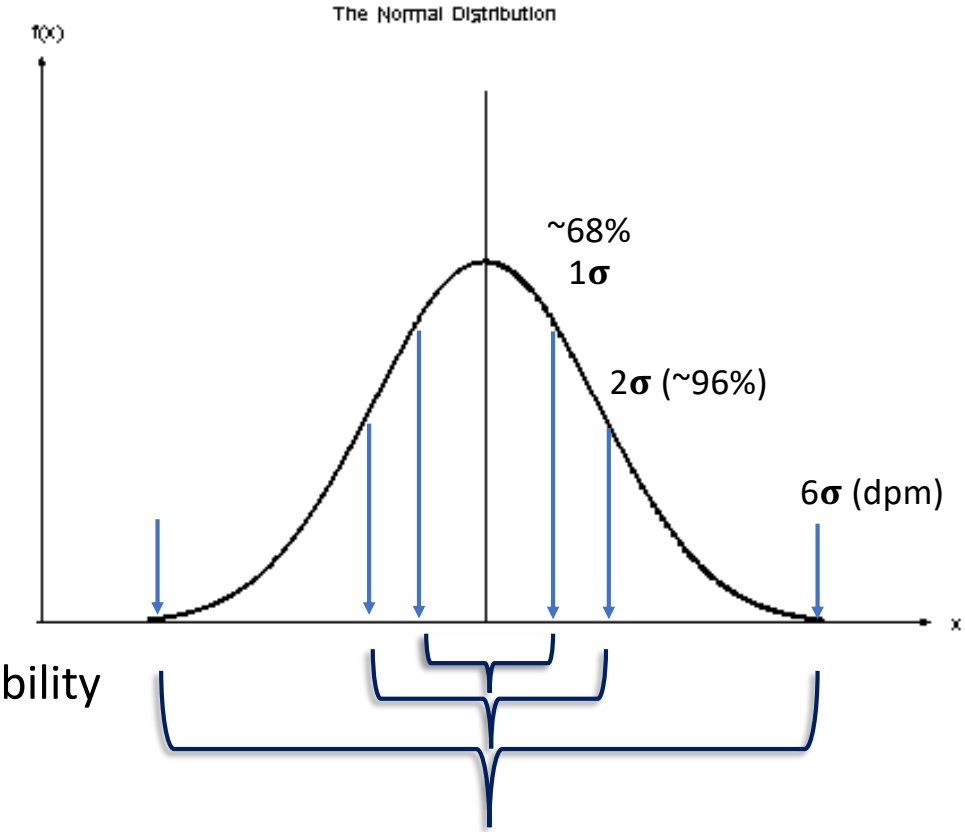


## Unintentional Variability- “Excursions”

*“molecules do not care”- Prof. Doug Taber*

- Inputs → Outputs
- Error is everywhere!
- Inputs + s → Outputs + **σ**




- Nested interdependent subsets→ confounded!
- Materials purity, Tool precision → process capability
  - Raw materials lot tracking, quality testing,
  - storage, handling and shelf-life validation
  - Process & product characterization (metrology)

***“Black Swans”*** = research **and development**

# Are they equal? →Student's t-test

- Two sets of independent samples
- Both normally distributed data
- Both sets have similar variance

$$t = \frac{\bar{X}_1 - \bar{X}_2}{\sqrt{\left(\frac{(N_1 - 1)s_1^2 + (N_2 - 1)s_2^2}{N_1 + N_2 - 2}\right)\left(\frac{1}{N_1} + \frac{1}{N_2}\right)}}$$

Pooled Std Dev

Typically Automated (software)

P

N-1

| df \ α | 0.25 | 0.1 | 0.05 | 0.025 |
|---|---|---|---|---|
| 1 | 1.000 | 3.078 | 6.314 | 12.706 |
| 2 | 0.816 | 1.886 | 2.920 | 4.303 |
| 3 | 0.765 | 1.638 | 2.353 | 3.182 |
| 4 | 0.741 | 1.533 | 2.132 | 2.776 |
| 5 | 0.727 | 1.476 | 2.015 | 2.571 |
| 6 | 0.718 | 1.440 | 1.943 | 2.447 |
| 7 | 0.711 | 1.415 | 1.895 | 2.365 |
| 8 | 0.706 | 1.397 | 1.860 | 2.306 |
| 9 | 0.703 | 1.383 | 1.833 | 2.262 |
| 10 | 0.700 | 1.372 | 1.812 | 2.228 |

- Null Hypothesis is that the means are equal
  H0: u1 - u2 = 0,
  u1 i& u2 are the means two sets
  df=n-1
- If $t_{test} > t_{table}$ H0 is rejected → the means are different
- Two risks of being wrong: **α** & **β**
- Low numbers of small measurements are likely to increase noise
- **Larger numbers of trials reduces noise and can tease out subtle effects**

If N is too small, then risk of overinterpreting noise increases

# Intentional Variability- "Experimentation"

*(molecules still do not care)*

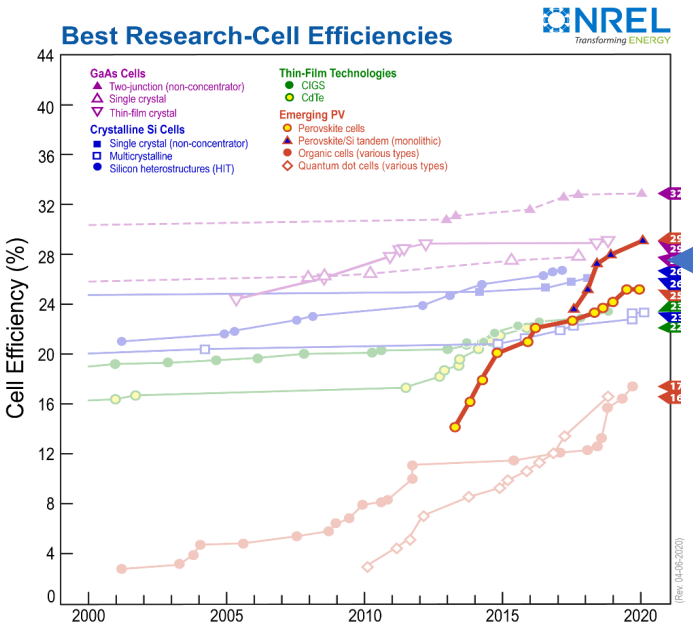
- As N increases, S→ **σ**
- Smaller sample sizes underestimate variability, confidence (sensitivity, resolution) is reduced
- For any given N, confidence can be assigned based on resolution needed
- For single sided tests 95% confidence can be resolved for 1 **σ** differences at N=6 (2-sided, n=13)
- Tighter resolution assigns higher risk

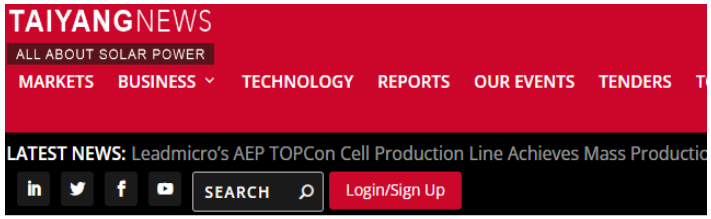
**NIST on choosing sample size vs. risk-**
https://www.itl.nist.gov/div898/handbook/prc/section2/prc222.htm

**Sample Size Table for Two-Sided Tests**

| $\alpha$ | $\beta$ | $\delta$=0.5$\sigma$ | $\delta$=1.0$\sigma$ | $\delta$=1.5$\sigma$ |
|---|---|---|---|---|
| 0.01 | 0.01 | 98 | 25 | 11 |
| 0.01 | 0.05 | 73 | 18 | 8 |
| 0.01 | 0.10 | 61 | 15 | 7 |
| 0.01 | 0.20 | 47 | 12 | 6 |
| 0.01 | 0.50 | 27 | 7 | 3 |
| 0.05 | 0.01 | 75 | 19 | 9 |
| 0.05 | 0.05 | 53 | 13 | 6 |
| 0.05 | 0.10 | 43 | 11 | 5 |
| 0.05 | 0.20 | 33 | 8 | 4 |
| 0.05 | 0.50 | 16 | 4 | 3 |
| 0.10 | 0.01 | 65 | 16 | 8 |
| 0.10 | 0.05 | 45 | 11 | 5 |
| 0.10 | 0.10 | 35 | 9 | 4 |
| 0.10 | 0.20 | 25 | 7 | 3 |
| 0.10 | 0.50 | 11 | 3 | 3 |
| 0.20 | 0.01 | 53 | 14 | 6 |
| 0.20 | 0.05 | 35 | 9 | 4 |
| 0.20 | 0.10 | 27 | 7 | 3 |
| 0.20 | 0.20 | 19 | 5 | 3 |
| 0.20 | 0.50 | 7 | 3 | 3 |

# Additives for Stability and Efficiency

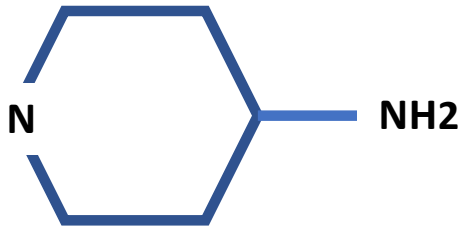



**9/1/2022**

- **η = 24%**

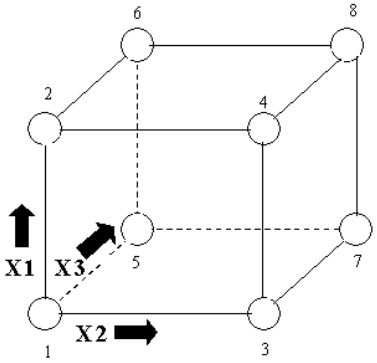
**T87= 2400 h (3-APy)**



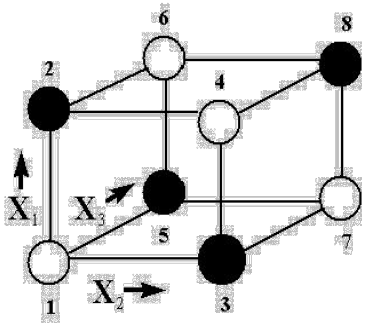

- Passivation adds process steps, maybe a supplier?
- Formulation's behavior on tool, in bottle, in storage may differ
- Control of process implies capable metrology and monitored baseline
- Un-forecasted changes: order of operations, raw lots, atalyst, container cleaning, HVAC
- Precision targeting

Minor recipe ingredients/treatments can have outsized influence upon performance

# Confident Decision Making: Statistically Driven Experimental Plan

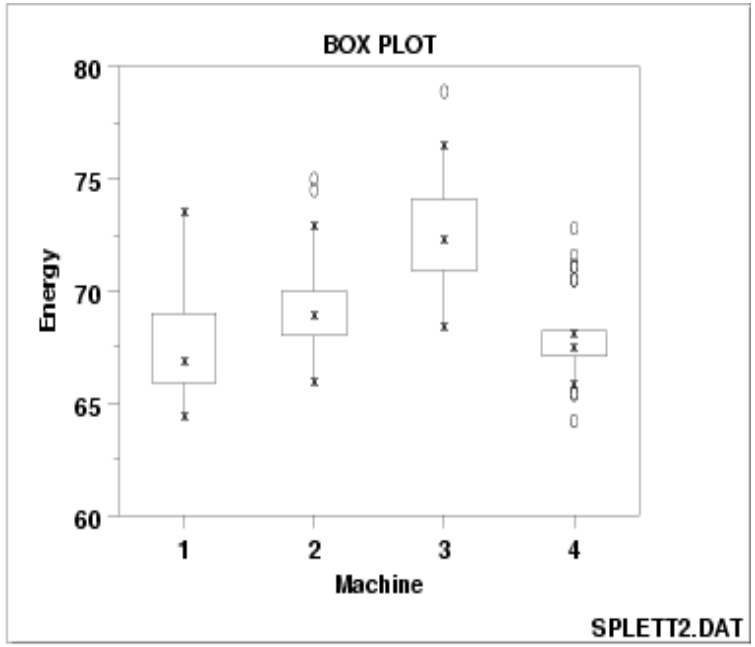



Full Factorial




Half Factorial

- Design of Experiments (the "other" DOE) uses statistical criterial to define sampling plans and experiment.
- Results obtained balance economy vs. risk.
- DOE is used to test a subset of variables combinations (partial factorial).
- Choices made are based on how likely variables are to be important and to interact independently with each other.



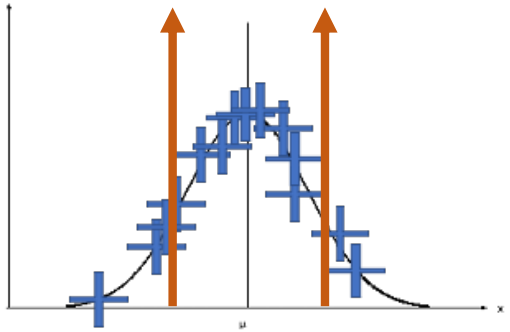

Anova (Analysis Of Variation)

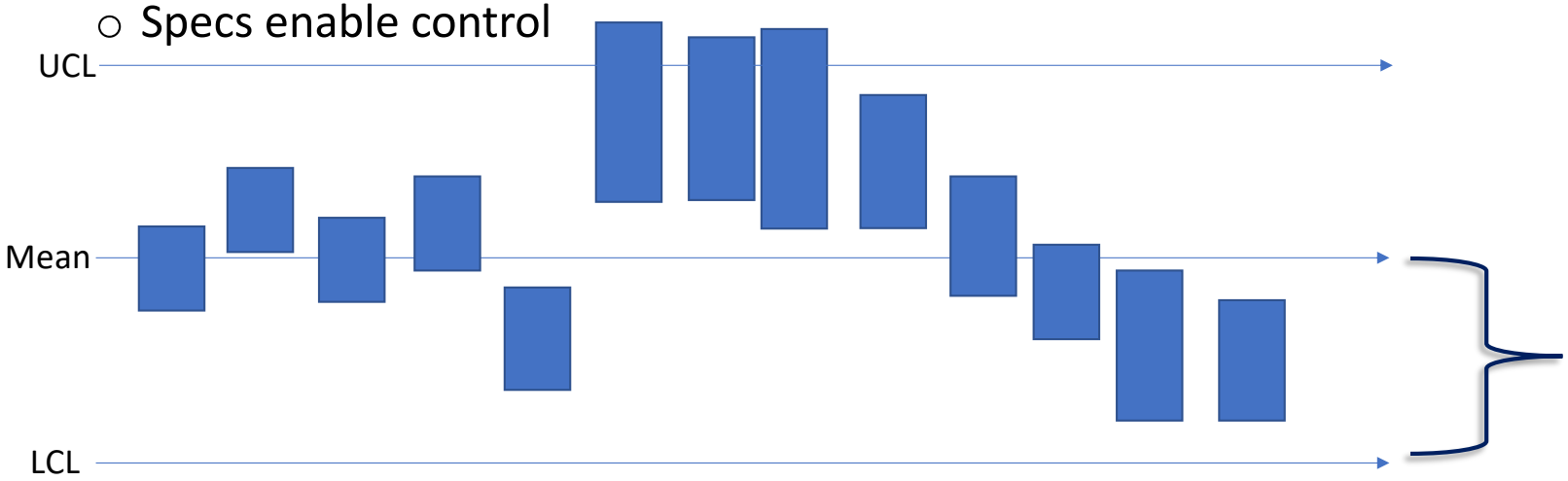
**Statistically driven experimental plans** enable results with known confidence to minimize risk

# Drivers of Quality- Specifications

- A Control Chart plots data lot by lot
  - Within the systems for which it is designed
  - Based on samples taken & tools used
- A "specification" can be defined from process data & negotiated with supplier
  - Raw, intermediate, process, product…
  - Based on samples taken & tools used
  - Specs enable control

Spec defines "good enough"






UCL & LCL: often 3$\sigma$ from a 15 lots baseline

# Drivers of Quality- Risk Assessment

**Technical :**

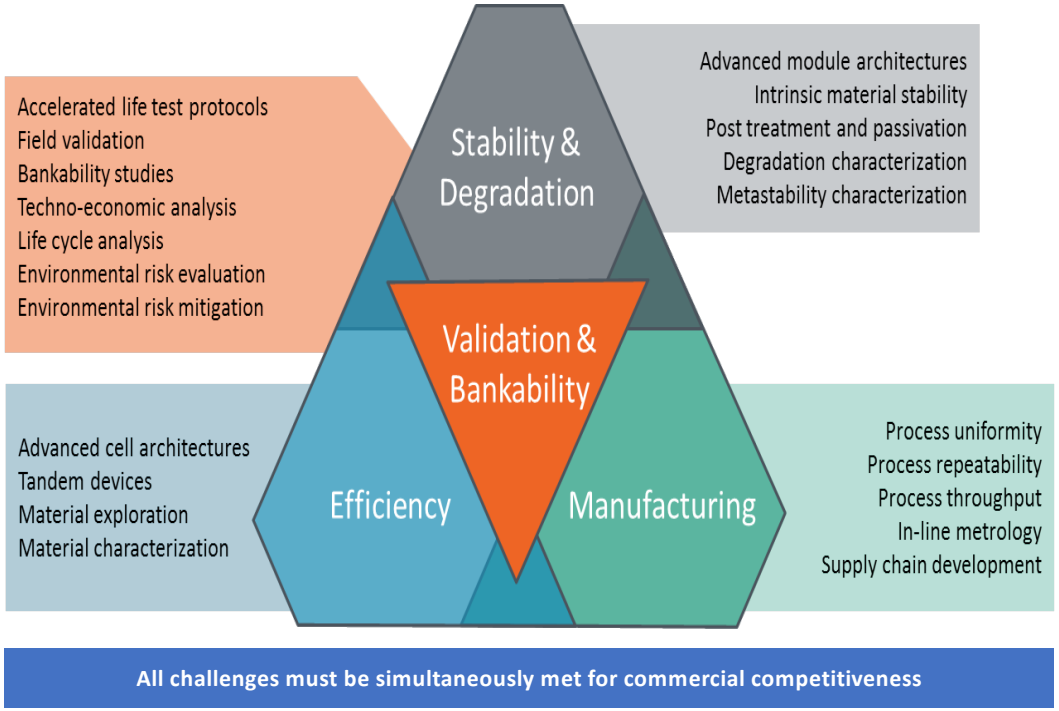
- **η** with Durability
- Scalability
- Manufacturability
- Safety

**Business :**

- Validation and trust
- Environmental/EOL
- Scaling
- Supply Chain

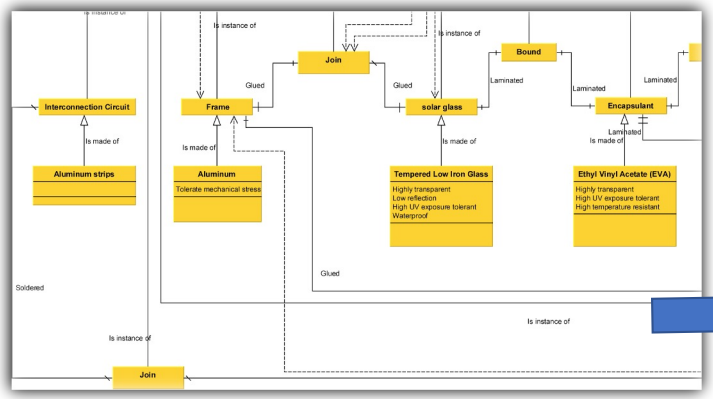
**Market :**

- MVP – first minimum viable products
- Resilience – technical vs. business success
- Finance – seeks lower risk path
- Timing – Fast paced environment
- Horizon – challenges from competition



# Edrivers of Quality: Copy Exactly! & FMEA

- Failure modes and effects analysis (**FMEA**) can be helpful for prototype and process design.
- Ranks possible points of failure (risk assessment) for action
- Scale-up challenge is matching performance in different sizes of process equipment & energies.
- Tool matching for metrology can challenging between collaborators
- Technology transfer nearly always involves tool matching.
- Exactly copying tools, recipes, sources, conditions and scales is very valuable for tech transfer



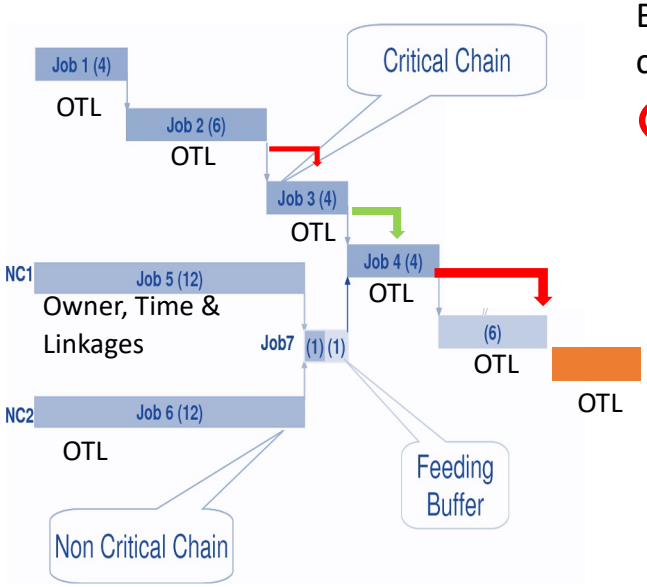

**Risk Priority Number**
RPN = Product of (Severity x Occurrence x Detection)
Each is scored 1 to 10

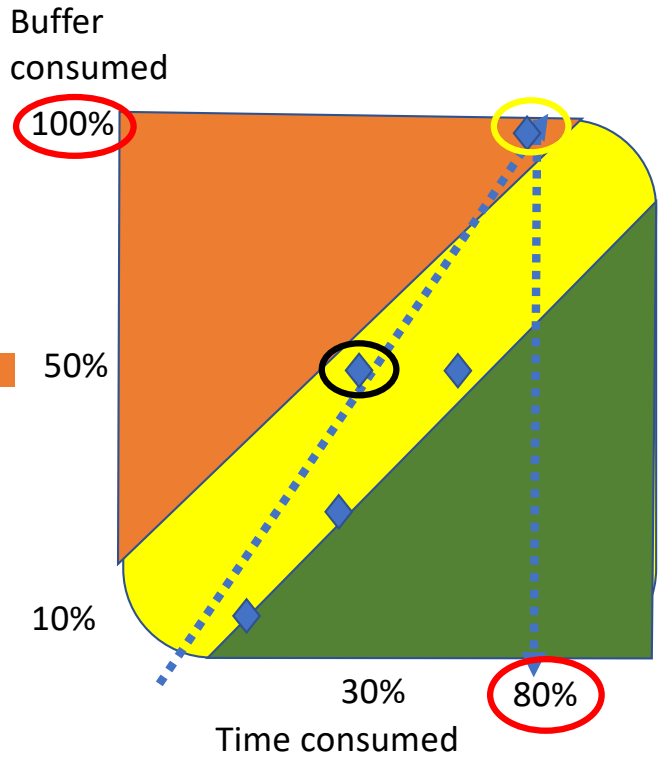
| Product FMEA | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Prepared By: Gustavo L. C. Cavenaghi | | | | | | | | | FMEA No: 03 | | |
| Date: 11/02/2013 | | | | | | | | | Component: PV Module | | |
| Item | Function | Potential Failure Mode | Potential Effects or Failure | Svrty | Potential Causes of Failure | Ocrnc | Current Controls for Prevention/ Detection | Dtctn | RPN | Recommended Action | Responsibility and Target Completion Date |
| Glass | Protect the cells from environment while allowing light penetration and provide a rigid structure | Crack | Loss of the product | 10 | Mechanical stress, overheat or freeze | 4 | Material selection | 6 | 240 | Use of standarized materials only, Knowledge of material operation limits, care while handling, inspection | Process engineers, techinical crew |

RPNs > 125 need action plan to < 100.
RPNs > 250 w/no credible action plan need escalated

# Drivers of Quality: Project Management

- Sets of tasks often moving in parallel threads (paths, chains)
  1) building and testing new material in devices
  2) developing models, fundraising, communications, subsequent plans
- Theory of constraint (**TOC**) sorts task dependencies (chains) , identifies parallel & critical chain, minimum time required (**Tr**), and time allowed (**Ta**)
  1) Project Ta = Project Tr + Project buffer
  2) **Project Ta > ∑ (Task Tr + Task error)**
- **Gantt chart** diagrams interdependencies and tracks progress against schedule (PAS)
- Tasks have time, owners and dependencies; owners have distractions
- Errors in time become cumulative




**Buffer table shows risk to completion**

## Case Study 1 : Transistor “Nubs”

- **Project:** New photosensitive polymer coating defect at limit of instrumental detection
- **Excursion:** a defect was a small bump on side of transistor’s gate
- Contained within supplier & sub-supplier lots and
- Root identified as operator A vs. B → monomer reactivity and order of operations

## Case Study 2&3 : Siloxane scaling

- **Project:** New siloxane-based material scaleup
- **Excursion**: a defect in test performance with new sub-supplier lots
- Contained in qualification
- Root identified as completely different distillation parameters
- **Excursion:** freezer failure
- Contained after lots in process
- Root cause identified as chart paper not checked or changed despite spec and system
- In both cases systems were in place and ignored

## Summary

- Researchers spend time and other resources to identify signals in noise
- Uncertainty erodes quality and renders a project less resilient, less valuable
- Total Quality embraces problem solving statistics and planning wholistically
- Understanding raw materials characteristics (purity, storage, shelf life, handling) is vital as is process characterization and tool matching
- Scaling and reproduction via *Exact Copy works!*
- T-test with appropriate N & P can define signals (ANOVA for larger sets of data)
- Partial factorial designed experiments give confidence with savings over full matrix

***Some pet peeves:***

*How many tests/units will you include in that experiment?*
*How will you know you have made a significant difference?*
*What is the error that you expect?*
*What tool are you planning at the new site?*
*What is your qual plan*
*Do you have a control chart for that?*

# Backup Materials

## Resources:

Statistics & Design of Experiments

- https://www.theanalysisfactor.com/confusing-statistical-terms-1-alpha-and-beta/
- https://www.statisticshowto.com/probability-and-statistics/t-test/
- https://www.six-sigma-material.com/t-distribution.html
- https://www.graphpad.com/quickcalcs/ttest1.cfm
- https://www.itl.nist.gov/div898/handbook/pri/pri.htm
- https://www.itl.nist.gov/div898/handbook/prc/section2/prc222.htm
- https://sciencing.com/determine-sample-size-6507705.html
- https://www.itl.nist.gov/div898/software/dataplot/index.htm

FMEA & CE!

- https://ieeexplore.ieee.org/document/6648239
- https://digital.library.adelaide.edu.au/dspace/bitstream/2440/98411/3/hdl_98411.pdf
- https://citeseerx.ist.psu.edu/viewdoc/download?doi=10.1.1.1038.4189&rep=rep1&type=pdf
- https://dspace.mit.edu/bitstream/handle/1721.1/50399/40607000-MIT.pdf;sequence=2
- https://supplier.intel.com/construction/changecontrol/
- https://www.iqasystem.com/news/risk-priority-number/

Project Management

- https://www.pomsmeetings.org/confpapers/011/011-0754.pdf
- https://csbweb01.uncw.edu/people/rosenl/classes/OPS100/A%20Critical%20Look%20at%20Critical%20Chain%20Project%20Management.pdf
- https://pmworldlibrary.net/wp-content/uploads/2015/09/pmwj38-Sep2015-Ghaffari-Emsley-research-on-critical-chain-second-edition.pdf
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