Gauge R&R (JMP) of Xray photoelectron spectroscopy to monitor a coating process

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Sukti Chatterjee
CTO, Applied Materials

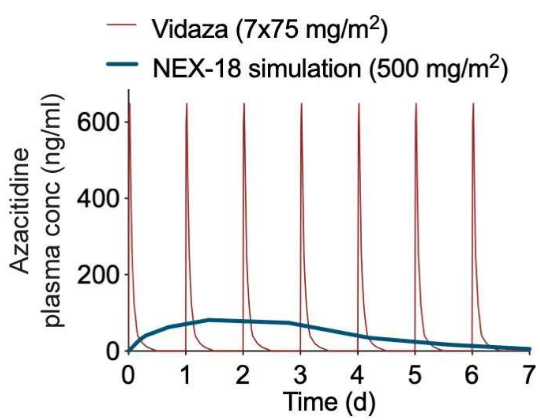
APPLIED MATERIALS®
make possible

AGENDA

- Background & Problem statement
- Operation definition & data collection plan
- MSA components analysis
- Plan for MSA components improvement

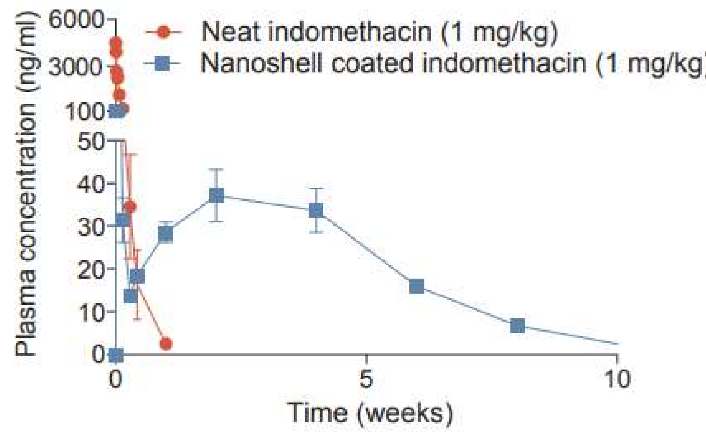
Background

In therapeutic areas (ex. antibiotic, drug/alcohol addiction), daily Injections cause side effects and patients skipping medication**.**



https://nanexa.com/en/nex-18/

To solve patient's adherence issue, **a potential approach to tailored release** of drug



https://www.sciencedirect.com/science/article/pii/S0939641118312049?ref=cra_js_challenge&fr=RR-1

Team developed AlOx barrier layer that forms a shell around the API particle & controls the release of the drug. Characteristics (**composition** & thickness) of oxide layer can customize the release.

Reporting noise analysis (GRR) of composition (O/Al ratio) measurements

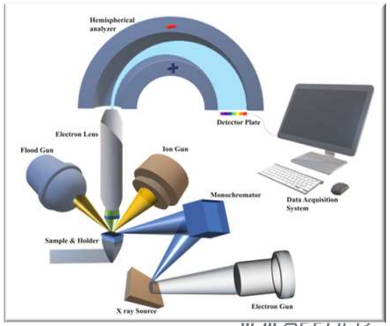
Problem statement / objective and measurement device

- **Problem statement**: **Measure AlOx coating composition (spec O/Al ratio: 1.2-2.3)**
- **Objective**: To determine analysis (XPS) method is adequate to differentiate AlOx process variation
  Determine GRR of XPS for AlOx composition analysis

- **Measurement device**
  - X-Ray Photoelectron Spectroscopy (XPS) is used to determine quantitative atomic composition
  - XPS measures the kinetic energy of the photoelectrons emitted from elements and counts the electrons
- **Measurement parameters**
  - Counts of electrons for each elements
    - Accounts the presence of elements
    - What other elements bonded with it

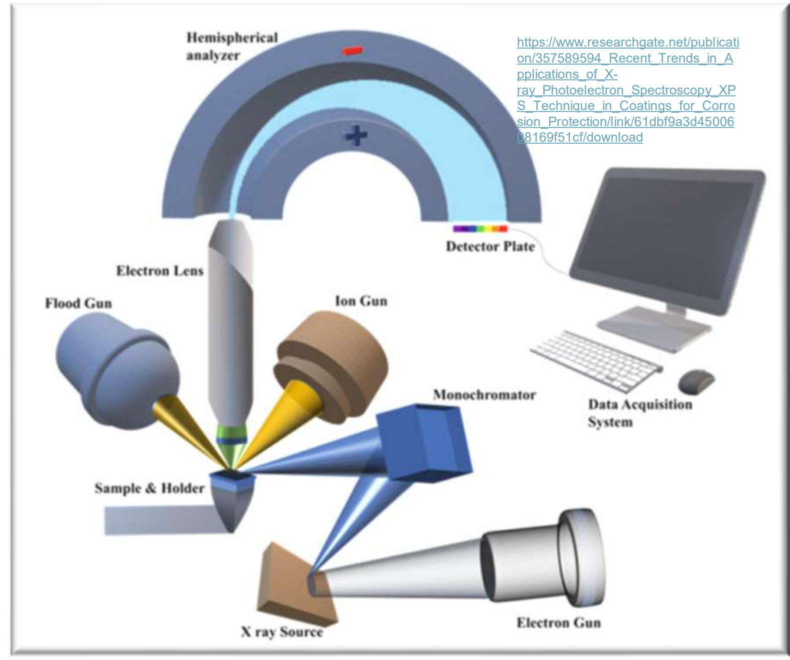
Source of errors	Impact on GRR components
Calibration	Reproducibility
Electron counts	Repeatability & reproducibility
Analysis	Reproducibility

https://www.researchgate.net/publication/357589594_Recent_Trends_in_Applications_of_X-ray_Photoelectron_Spectroscopy_XPS_Technique_in_Coatings_for_Corrosion_Protection/link/61dbf9a3d4500608169f51cf/download



XPS and its detection capability

- Measures energy and electron counts
  - Assess the binding energies (BE) of core-level electrons and the chemical affinity of an atom
- Source of errors in XPS analysis
  - background/baseline corrections
  - Electron counts
  - Peak deconvolution

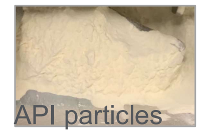


- Recent development to eliminate XPS analysis errors
  - background/baseline corrections
    - eliminate inelastically scattered electrons interference in measurement & improve in accuracy to identify peak position & counts
  - Electron counts
    - Flood gun: neutralize the surface charge during data acquisition
    - Ion gun: clean surface before measurement to eliminate effect of contamination
    - Hemispherical analyzer: different energy electrons arrive at different positions in the radial direction that improve binding energy resolution
  - Peak deconvolution
    - X-ray emission, charge neutralization, resolution, peak fitting software

Operation definition

#	Task	Operation
	Subject	Measure composition of AlOx coating (O/Al ratio)
1.	Baseline correction	Automatic
2.	Calibration	XPS spectra adjusted with carbon peaks (C1s = 284.8 eV, C-C,H)
3.	XPS scan	XPS survey & high-resolution scan,
4.	Analysis	Peak fitting, quantize at% and determine O/Al ratio

Coating

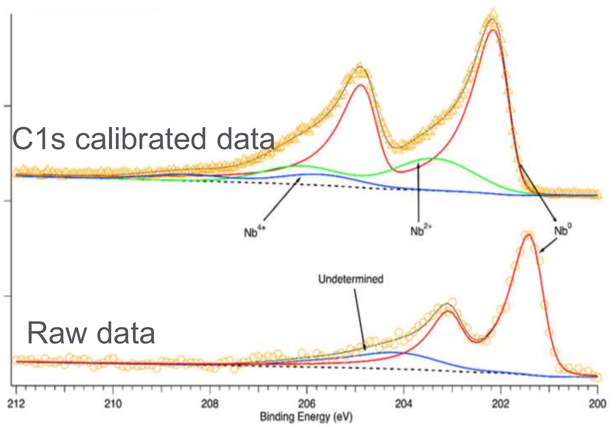


Substrate not for XPS measurement

Substrates for XPS measurement

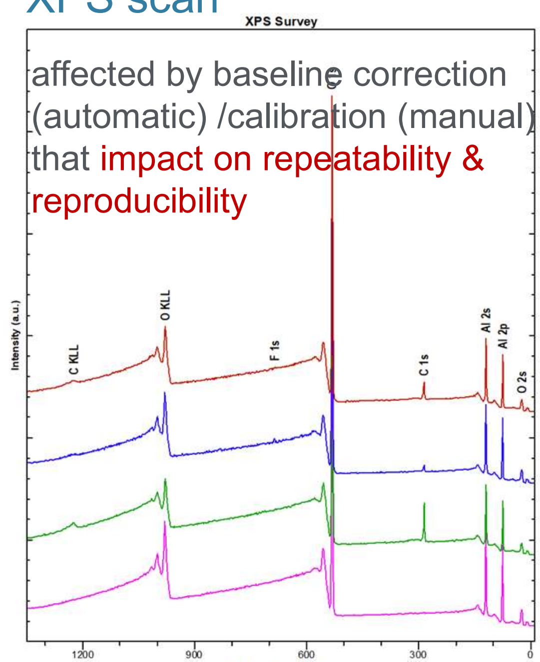
Calibration with C-peak

No calibration sample available
Human error associated impact on reproducibility



https://mmrc.caltech.edu/XPS%20Info/Practical%20Guides%20to%20XPS/XPS%20guide%20%20Curve%20fitting.pdf

XPS scan



affected by baseline correction (automatic) /calibration (manual) that impact on repeatability & reproducibility

Analysis

Peak fitting semi-automatic.
impact on reproducibility

Quantize at%

O%	Al%
66.8	32.4

O/Al
2.06

Most of state-of-the-art XPS tools are semiautomatic and human error is associated with sample loading, calibration and peak fitting

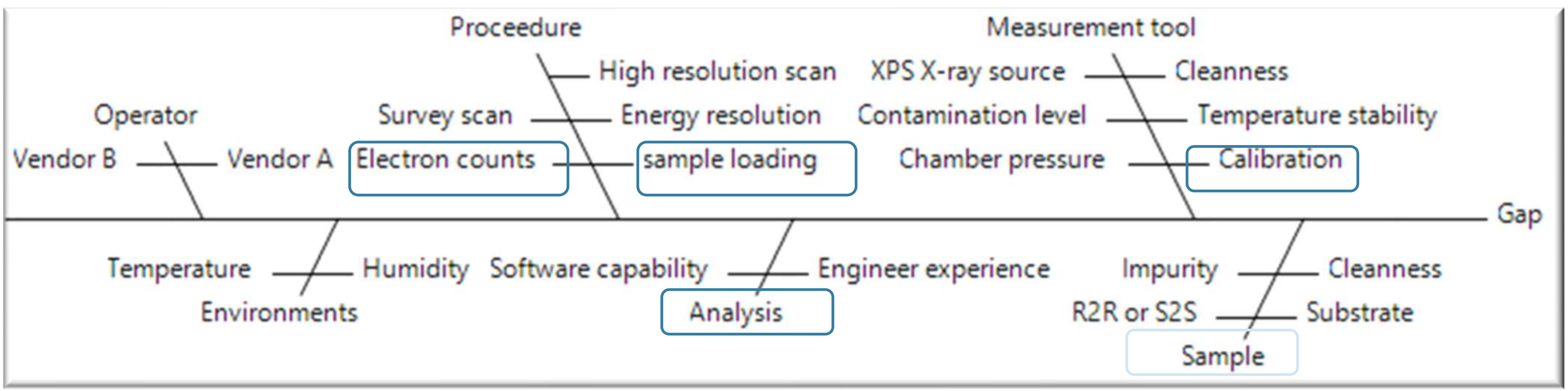
APPLIED MATERIALS

MSA C&E diagram

Parameters that impact on GRR of XPS for AlOx composition analysis

- System calibration: XPS scan affected by calibration and under or overestimate elements at% (impact on reproducibility)
- XPS scan (electron counts): Effect on peak position as well peak area depending on baseline correction & calibration (impact on repeatability & reproducibility)
- Analysis - Peak fitting: Generate error in the proportion of elements at% (Impact on reproducibility)
- Sample: process variation with substrate

JMP Platform: Analyze > Quality and Process > Diagram



APPLIED MATERIALS

Data Collection plan

- Sampling Method:
  - » 2 sites for XPS analysis (O/Al ratio)
  - » 6 samples (parts) (S0, S1, S2, A0, A1, A2)
  - » 4 replicates of each sample measured at each site
- Expected outcome:
  - » 2 sites get similar results
  - » Sample (part) not interact with site
  - » XPS method is adequate to differentiate process variation (O/Al ratio)
- Risk assessment
  - » O/Al ratio degradation with time

Total 48 measurements

A3 AMAT example_SC more data in MSE design style - JMP

File Edit Tables Rows Cols DOE Analyze Graph Tools View Window Help

7/0 Cols · 48/0 Rows

	Sample ID	Site	substrate type	O%	Al%	O/Al
	A0-1, A0-2, A0-3, A0-4, A1-1, 19 others	Site A, Site B	API pallet, Si	68.6 – 53.2	39.1 – 31.2	2.1 – 1.49
1	A0-1	Site A	API pallet	64.1	31.2	2.05
2	A0-2	Site A	API pallet	63.1	32.9	1.92
3	A0-3	Site A	API pallet	64.8	31.9	2.03
4	A0-4	Site A	API pallet	65	32.2	2.02
5	A0-1	Site B	API pallet	64.8	32.9	1.97
6	A0-2	Site B	API pallet	65.2	33.5	1.95
7	A0-3	Site B	API pallet	66.8	34.5	1.94
8	A0-4	Site B	API pallet	65.4	32.5	2.01
9	A1-1	Site A	API pallet	57.6	36.1	1.60
10	A1-2	Site A	API pallet	55	34.5	1.59
11	A1-3	Site A	API pallet	58	39	1.49
12	A1-4	Site A	API pallet	56.2	36.7	1.53
13	A1-1	Site B	API pallet	57.8	38.9	1.49
14	A1-2	Site B	API pallet	58	39	1.49
15	A1-3	Site B	API pallet	59.2	38.1	1.55
16	A1-4	Site B	API pallet	57.4	37.4	1.53

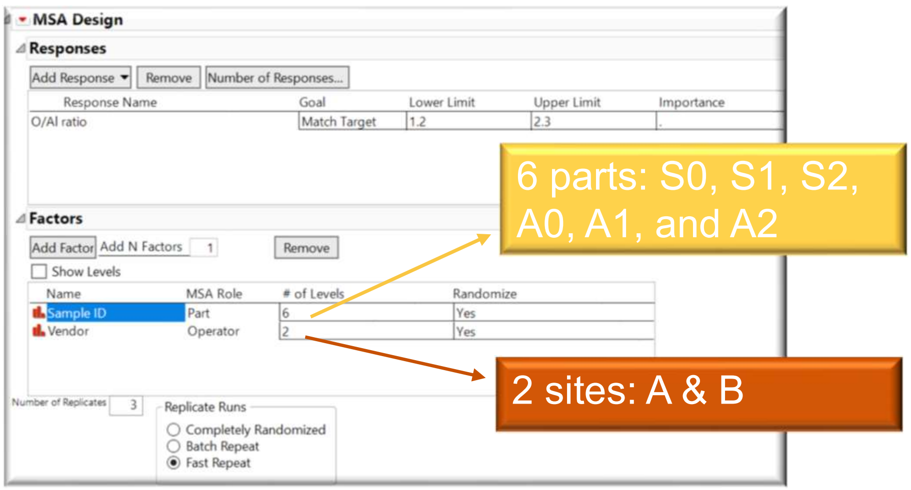
4 replicates

evaluations done

MSA design

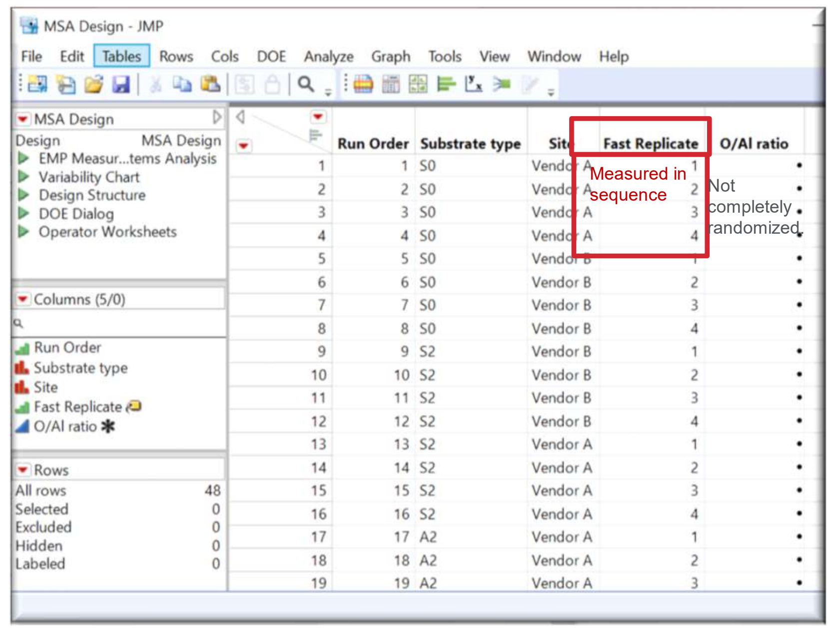
Substrate type & site
both are **Crossed factors**

- MSA design window



  - » Not be able to use completely randomized option – risk: minimizing noise
  - » Fast repeat option – Not changing sample replicate # - risk: sample degradation
    - – Compare 1st and 4th replicates to retire sampling risk

- MSA fast repeat table: **48 rows**
  - ▸ Sample size is more than necessary from the perspective of power



APPLIED MATERIALS

Sequence of JMP analysis for MSA

#	Task to do	Application to use	JMP Platform
1.	Data distribution of MSA samples	Descriptive and inferential statistics	Distribution & Fit Y by X
2.	Data variability – common cause vs. special cause	I-MR & One-way ANOVA	Control charts & Fit Y by X
3.	GRR components	Gauge R&R method	Variability
4.	Process capability (Cp) with GRR	ICC vs P/T – EMP method	Measurement system analysis
5.	Improve GRR components	Box plot, density ellipse, Fit line & matched pairs	Distribution, Fit Y by X & Specialized modeling

APPLIED MATERIALS

MSA sample distribution

Spec limits: O/Al - 1.2-2.3

- JMP analysis identified overall data distribution is bimodal. Data from each section has normal distribution

- Sample selection (Bimodal) may impact on GRR components

P/T	P/TV	P/PV	Misclassification
No	Yes	Yes	Yes

Misclassification Probabilities

Lower Tolerance = 1.2, Upper Tolerance = 2.3, Grand Mean = 1.709167

Description	Probability
P(Good part is falsely rejected)	0.00640770
P(Bad part is falsely accepted)	0.16233021
P(Part is good and is rejected)	0.00626913
P(Part is bad and is accepted)	0.00351035
P(Part is good)	0.97837523

Less impacted by sample distribution

More impacted by sample distribution

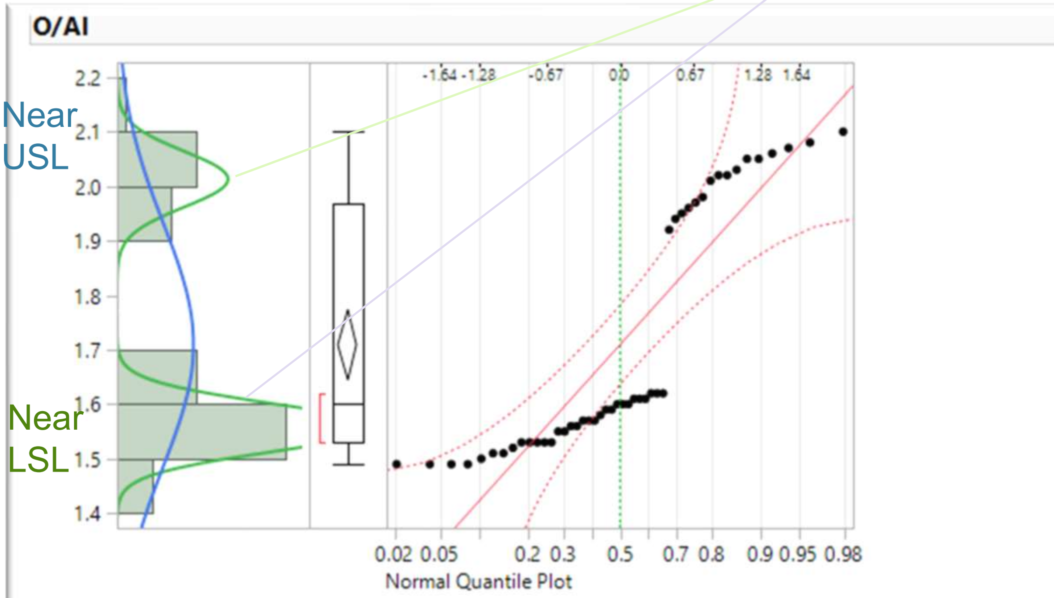
- To minimize risk for sample distribution our GRR focus is on P/T ratio
  - » Next time we prefer sample selection with uniform distribution

2 parts
4 replicates
2 sites
Fast repeat

Near USL

4 parts
4 replicates
2 sites
Fast repeat

Near LSL



Compare Distributions

Show	Distribution	AICc	AICc Weight	.2 .4 .6 .8	BIC	-2*LogLikelihood
☑	Normal 2 Mixture	-86.44188	1		-78.51445	-97.87045
☑	Normal	-4.894691	0		-1.418956	-9.161358

APPLIED MATERIALS

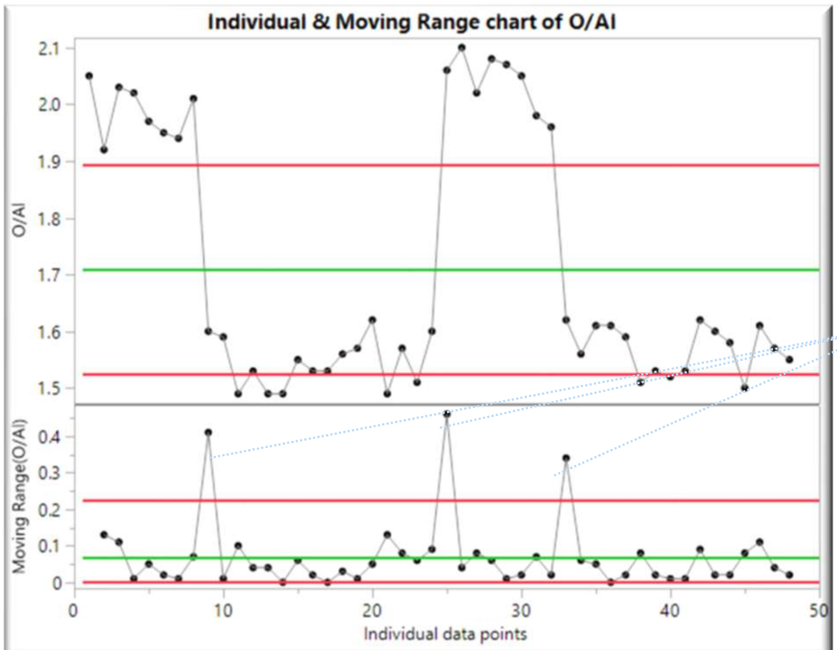
Variability in O/AI I Identify candidate for subgrouping

JMP Platform: Analyze > Quality and Process > Control Chart Builder (LHS) & Variability (RHS)

To identify special cause of variation

I-MR chart

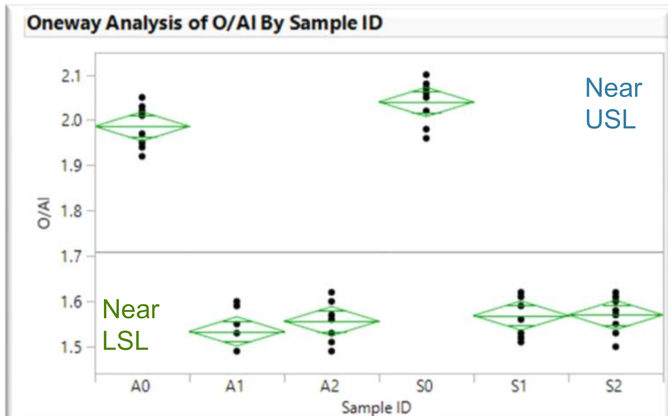
Mix up common cause variation (equipment variation or repeatability) and special cause variation (site or part variation), control limits here are meaningless. Need subgrouping with special cause



Type II shift

One-way ANOVA

Is part variation special cause?



Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample ID	5	2.2357167	0.447143	218.7539	<.0001*
Error	42	0.0858500	0.002044		

Variance Components

Component	Var Component	% of Total	20 40 60 80	Sqrt(Var Comp)
Sample ID	0.05563741	96.5		0.23588
Within	0.00204405	3.5		0.04521
Total	0.05768146	100.0		0.24017

Part variation is a special cause

A candidate for subgrouping

P<0.05, parts are significantly different

Part to part variation is much higher than within part variation

Variability in O/AI | Identify candidate for subgrouping

JMP Platform: Analyze > Quality and Process > Control Chart Builder (LHS) & Variability (RHS)

To identify special cause of variation

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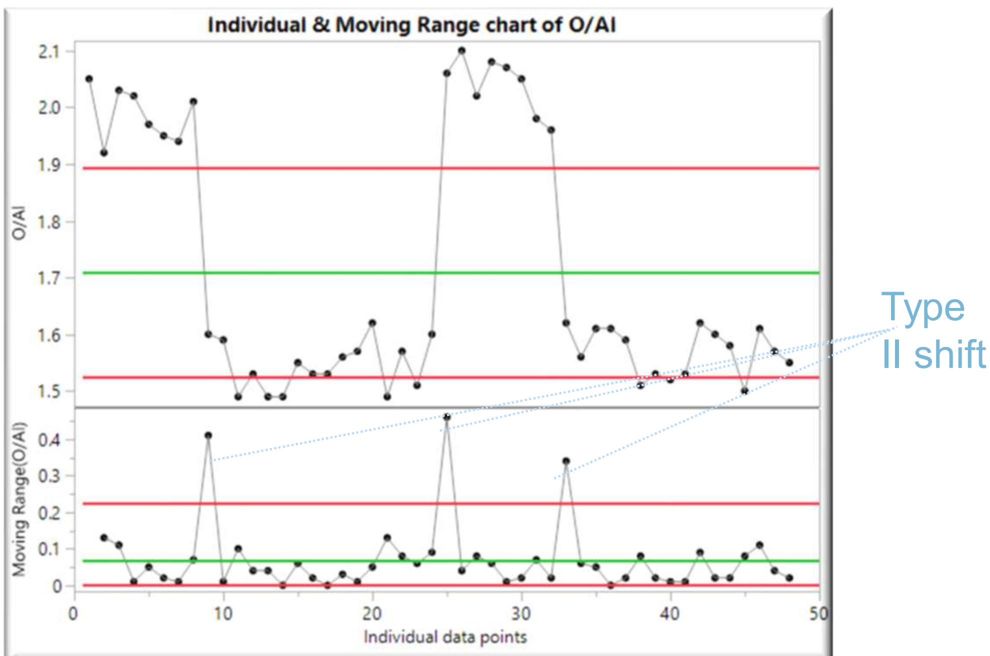
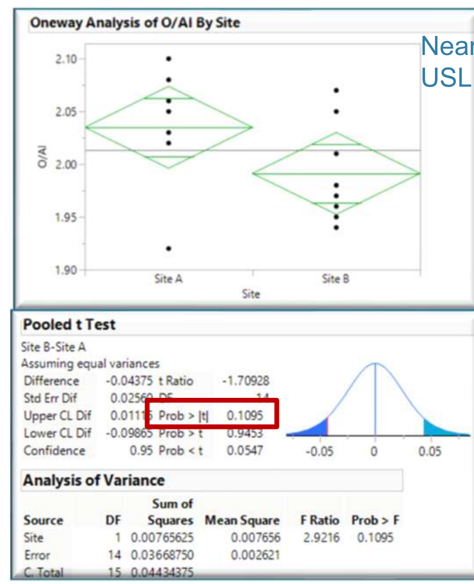
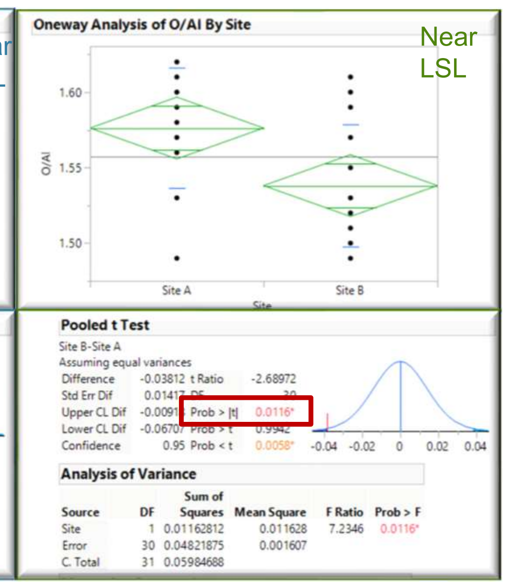


One-way ANOVA

Is site variation special cause?





Either P> 0.05 (no evidence to reject) or P <0.01(marginally reject)

Site variation is marginal, not a good candidate for subgrouping. Part variation is better candidate

JMP Platform: Analyze > Quality and Process > Control Chart Builder

Variability in O/AI I subgrouping with part by site

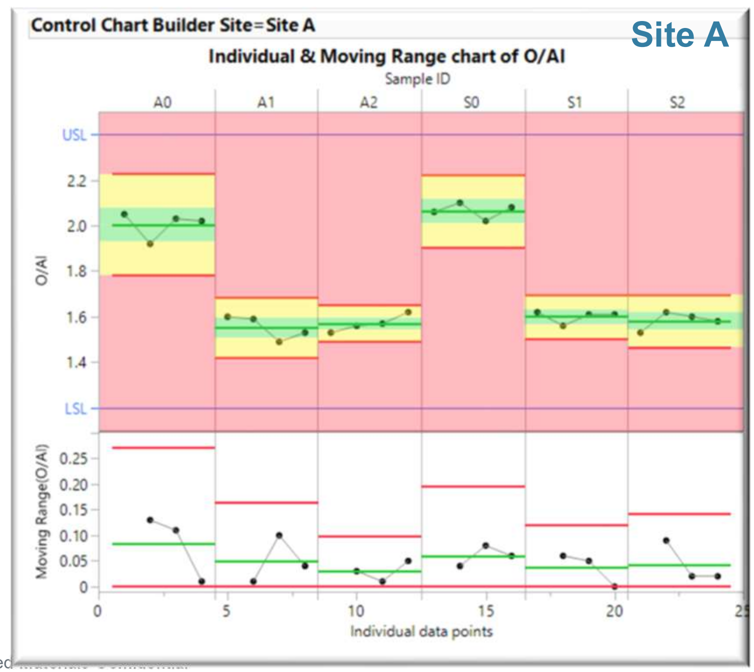
Visualize of repeatability & reproducibility

I-MR chart with phase option (part ID)

» Limited data points (4 only) in each phase. No data point outside control limit

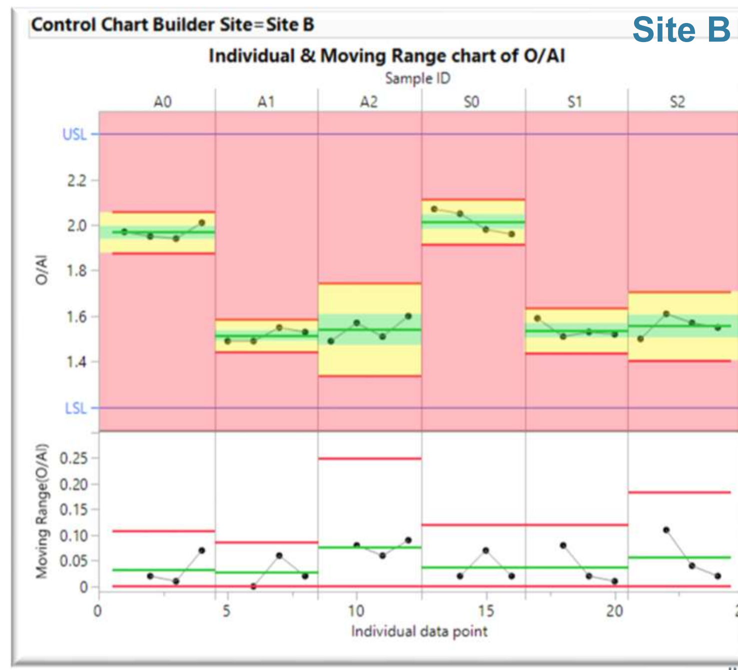
» Bottom charts – moving range indicates variability in each subgroup and forms control limits of upper chart

» Upper charts – control limit varies with phase and site – indications of variation in repeatability and reproducibility



Near USL

Near LSL



Near USL

Near LSL

APPLIED MATERIALS

JMP Platform: Analyze > Quality and Process > Variability

GRR analysis

Repeatability is dominating error over reproducibility

Main Effect (ANOVA without Interaction)

Gauge R&R

Measurement Source		Variation (6*StdDev)	% of Tolerance		which is 6*sqrt of
Repeatability	(EV)	0.2419131	21.99	Equipment Variation	V(Within)
Reproducibility	(AV)	0.1623625	14.76	Appraiser Variation	V(Operator)
Operator		0.1623625	14.76		V(Operator)
Gauge R&R	(RR)	0.2913478	26.49	Measurement Variation	V(Within) + V(Operator)
Part Variation	(PV)	1.4159201	128.72	Part Variation	V(Sample ID)
Total Variation	(TV)	1.4455841	131.42	Total Variation	V(Within) + V(Operator) + V(Sample ID)

Summary and Gauge R&R Statistics

6	k
20.1543	% Gauge R&R = 100*(RR/TV)
0.20577	Precision to Part Variation = RR/PV
6	Number of Distinct Categories = Floor(sqrt(2)*(PV/RR))
1.2	Lower Tolerance (LT)
2.3	Upper Tolerance (UT)
1.1	Tolerance = UT-LT
0.26486	Precision/Tolerance Ratio = RR/(UT-LT)

Using last column 'Sample ID' for Part.

Variance Components for Gauge R&R

Component	Var Component	% of Total	20 40 60 80
Gauge R&R	0.00235788	4.06	
Repeatability	0.00162561	2.80	
Reproducibility	0.00073227	1.26	
Part-to-Part	0.05568972	95.94	

Misclassification Probabilities

Lower Tolerance = 1.2, Upper Tolerance = 2.3, Grand Mean = 1.709167

Description	Probability
P(Good part is falsely rejected)	0.00640770
P(Bad part is falsely accepted)	0.16233021
P(Part is good and is rejected)	0.00626913
P(Part is bad and is accepted)	0.00351035
P(Part is good)	0.97837523

Type I error (α)
Type II error (β)

It's customer call. Have option to improve it by improving repeatability

Crossed (ANOVA with Interaction)

Gauge R&R

Measurement Source		Variation (6*StdDev)	% of Tolerance		which is 6*sqrt of
Repeatability	(EV)	0.2348226	21.35	Equipment Variation	V(Within)
Reproducibility	(AV)	0.1107092	10.06	Appraiser Variation	V(Operator) + V(Operator*Sample ID)
Operator		0.0887197	8.07		V(Operator)
Operator*Sample ID		0.0662219	6.02		V(Operator*Sample ID)
Gauge R&R	(RR)	0.2596116	23.60	Measurement Variation	V(Within) + V(Operator) + V(Operator*Sample ID)
Part Variation	(PV)	1.1940197	108.55	Part Variation	V(Sample ID)
Total Variation	(TV)	1.2219171	111.08	Total Variation	V(Within) + V(Operator) + V(Operator*Sample ID) + V(Sample ID)

Summary and Gauge R&R Statistics

6	k
21.2463	% Gauge R&R = 100*(RR/TV)
0.21743	Precision to Part Variation = RR/PV
6	Number of Distinct Categories = Floor(sqrt(2)*(PV/RR))
1.2	Lower Tolerance (LT)
2.3	Upper Tolerance (UT)
1.1	Tolerance = UT-LT
0.23601	Precision/Tolerance Ratio = RR/(UT-LT)

Using last column 'Sample ID' for Part.

Variance Components for Gauge R&R

Component	Var Component	% of Total	20 40 60 80
Gauge R&R	0.00187217	4.51	
Repeatability	0.00153171	3.69	
Reproducibility	0.00034046	0.82	
Part-to-Part	0.03960231	95.49	

Misclassification Probabilities

Lower Tolerance = 1.2, Upper Tolerance = 2.3, Grand Mean = 1.709167

Description	Probability
P(Good part is falsely rejected)	0.00257780
P(Bad part is falsely accepted)	0.18430970
P(Part is good and is rejected)	0.00256040
P(Part is bad and is accepted)	0.00124391
P(Part is good)	0.99325100

GRR marginally passed. Type II error is 18%, we have option to improve GRR

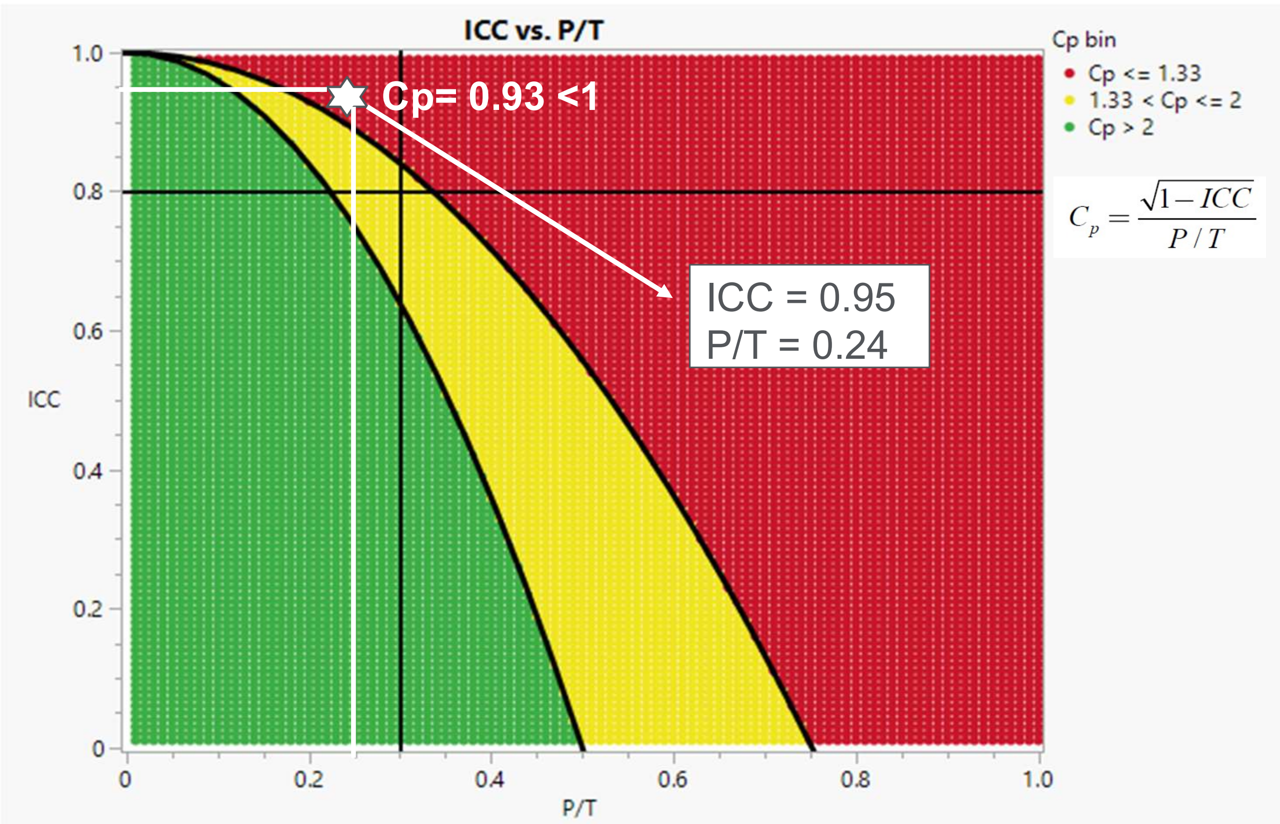
- **P/T Ratio: 26%**
  - **(Repeatability= 22%)**
- **P/TV Ratio: 20%**

- **P/T Ratio: 24%**
  - **(Vendor*Part Interaction= 6%)**
- **P/TV Ratio: 21%**

Relation of process capability (Cp) with GRR

JMP Platform: Analyze > Quality and Process > Measurement system analysis

ICC vs P/T



Intraclass correlation co-efficient

$$ICC = \frac{\sigma^2_{part}}{\sigma^2_{part} + \sigma^2_{gauge}}$$

Part variance in total variance

$$P/T = \frac{6\hat{\sigma}_{gauge}}{USL - LSL}$$

Spec based GRR

- Cp <1 (red zone)
- ICC is high and P/T is 24%
- To improve Cp into yellow zone, P to be improved
  - Repeatability to be improved since it is major error factor

APPLIED MATERIALS

JMP Platform: Analyze > Quality and Process > Measurement System Analysis

EMP analysis

- Main vs. Crossed model: parameters values are not changing much since part*vendor interaction is minor

Main effect (ANOVA without Interaction)

EMP Results

EMP Test	Results	Description
Test-Retest Error	0.0403	Within Error
Degrees of Freedom	41	Amount of information used to estimate within error
Probable Error	0.0272	Median error for a single measurement
Intraclass Correlation (no bias)	0.9716	Proportion of variation attributed to part variation without including bias factors
Intraclass Correlation (with bias)	0.9594	Proportion of variation attributed to part variation with bias factors
Bias Impact	0.0123	Amount by which the bias factors reduce the intraclass correlation

System	Classification
Current (with bias)	First Class
Potential (no bias)	First Class

Monitor Classification Legend

Classification	Intraclass Correlation	Attenuation of Process Signal	Probability of Warning, Test 1 Only*	Probability of Warning, Tests 1-4*
First Class	0.80 - 1.00	Less than 11%	0.99 - 1.00	1.00
Second Class	0.50 - 0.80	11% - 29%	0.88 - 0.99	1.00
Third Class	0.20 - 0.50	29% - 55%	0.40 - 0.88	0.92 - 1.00
Fourth Class	0.00 - 0.20	More than 55%	0.03 - 0.40	0.08 - 0.92

*** Probability of warning for a 3 standard error shift within 10 subgroups using Wheeler's tests, which correspond to Nelson's tests 1, 2, 5, and 6.**

Effective Resolution

Source		Value	Description
Probable Error	(PE)	0.0272	Median error for a single measurement
Lower Bound Increment	(0.1*PE)	0.0027	Measurement increment should not be below this value
Smallest Effective Increment	(0.22*PE)	0.006	Measurement increment is more effective above this value
Current Measurement Increment	(MI)	0.01	Measurement increment estimated from data (in tenths)
Largest Effective Increment	(2.2*PE)	0.0598	Measurement increment is more effective below this value

Action: Use as is

Reason: The measurement increment of 0.01 is effective.

Crossed effect (ANOVA with Interaction)

EMP Results

EMP Test	Results	Description
Test-Retest Error	0.0445	Within Error
Degrees of Freedom	33.098	Amount of information used to estimate within error
Probable Error	0.03	Median error for a single measurement
Intraclass Correlation (no bias)	0.9473	Proportion of variation attributed to part variation without including bias factors
Intraclass Correlation (with bias)	0.9296	Proportion of variation attributed to part variation with bias factors
Bias Impact	0.0177	Amount by which the bias factors reduce the intraclass correlation

System	Classification
Current (with bias)	First Class
Potential (no bias)	First Class

Monitor Classification Legend

Classification	Intraclass Correlation	Attenuation of Process Signal	Probability of Warning, Test 1 Only*	Probability of Warning, Tests 1-4*
First Class	0.80 - 1.00	Less than 11%	0.99 - 1.00	1.00
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Third Class	0.20 - 0.50	29% - 55%	0.40 - 0.88	0.92 - 1.00
Fourth Class	0.00 - 0.20	More than 55%	0.03 - 0.40	0.08 - 0.92

*** Probability of warning for a 3 standard error shift within 10 subgroups using Wheeler's tests, which correspond to Nelson's tests 1, 2, 5, and 6.**

Effective Resolution

Source		Value	Description
Probable Error	(PE)	0.03	Median error for a single measurement
Lower Bound Increment	(0.1*PE)	0.003	Measurement increment should not be below this value
Smallest Effective Increment	(0.22*PE)	0.0066	Measurement increment is more effective above this value
Current Measurement Increment	(MI)	0.01	Measurement increment estimated from data (in tenths)
Largest Effective Increment	(2.2*PE)	0.0661	Measurement increment is more effective below this value

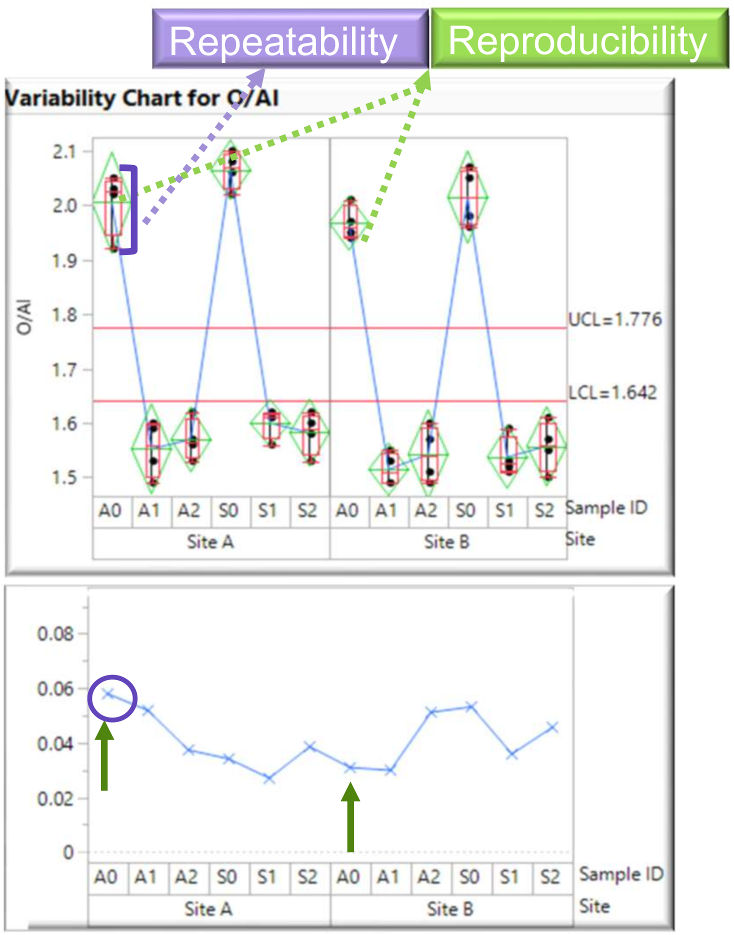
Action: Use as is

Reason: The measurement increment of 0.01 is effective.

Variation in O/Al ratio I Site (Operator)

Repeatability & reproducibility in variability chart

JMP Platform: Analyze > Quality and Process > Variability



Source of measurement error

Repeatability impacted by	Reproducibility impacted by
Base line correction	Calibration
Detector difference	Analysis

Variability chart and Analysis of variance confirmed Repeatability is bigger problem than reproducibility

» To improve GRR, need root cause analysis of repeatability

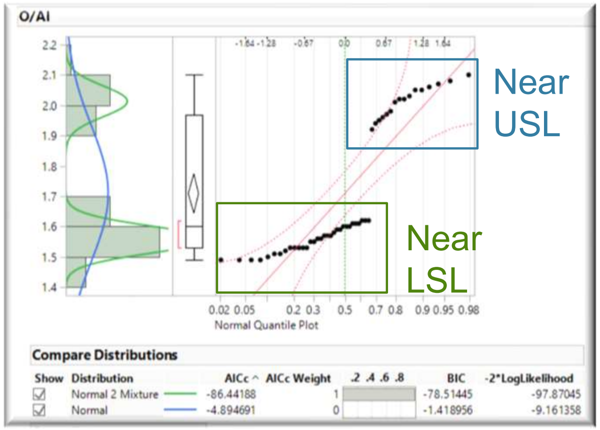
Analysis of Variance

Source	DF	SS	Mean Square	F Ratio	Prob > F
Site	1	0.0192	0.0192	11.811	0.0014*
Sample ID	5	2.235717	0.44714	275.062	<.0001*
Within	41	0.06665	0.00163		
Total	47	2.321567	0.0494		

P<0.05 indicates site to site variation in analysis

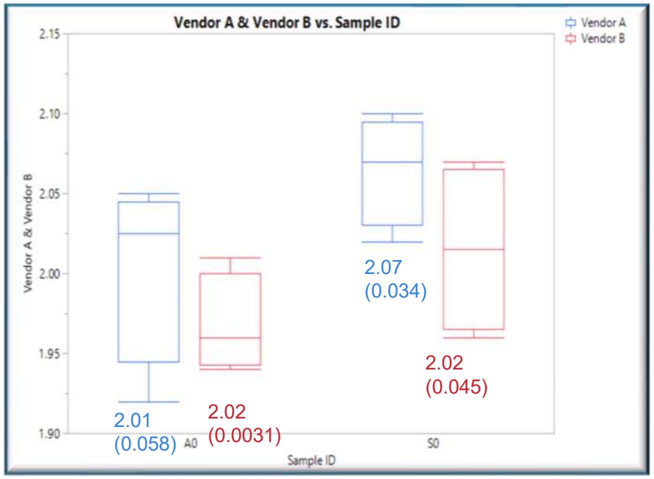
Root cause analysis I Why high repeatability

Compare repeatability variation @ part and site



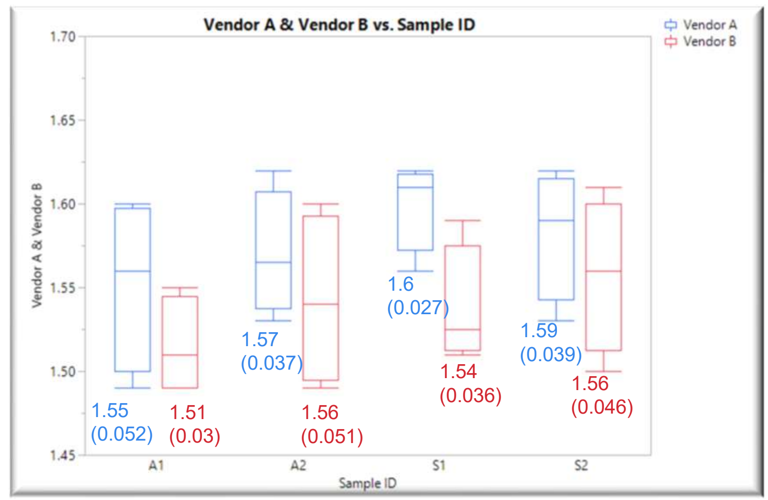
JMP Platform: Analyze > Distribution

Near USL



Standard deviation ranges from 0.03-0.06 for replicate samples

Near LSL



JMP Platform: Graph > graph Builder

Repeatability varies from part to part and Site to site

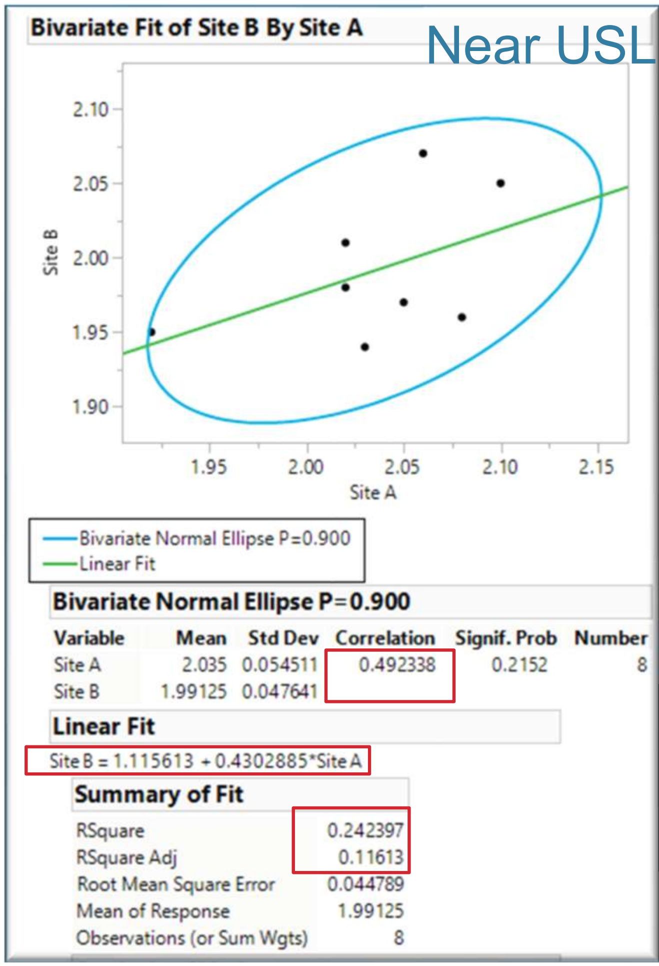
Site variation could be due to:

1. Calibration
2. XPS scan
3. Analysis

JMP Platform: Analyze > Fit Y by X

Root cause analysis I Relationship of Site A and Site B

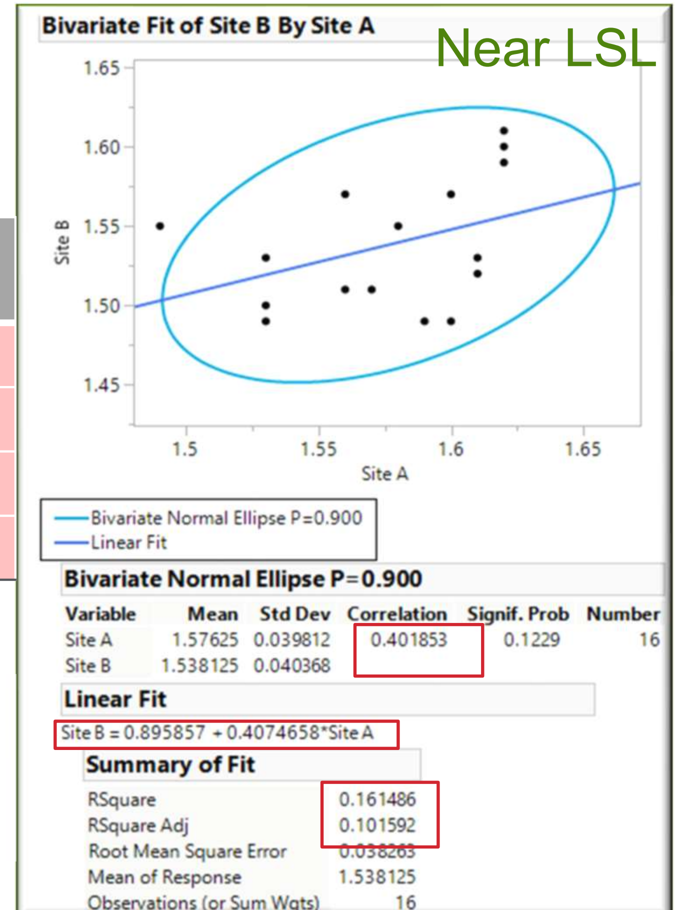
Test correlation and linear fit: site A vs. site B



Poor relationship between Site A & B analysis

Parameter	Success criteria	Status
Correlation	> 0.9	<0.5
Linear fit intercept	0	>0.9
Linear fit slope	1	<0.4
RSquare	>0.9	<0.3

Variation in repeatability impact on site-to-site variation (reproducibility)

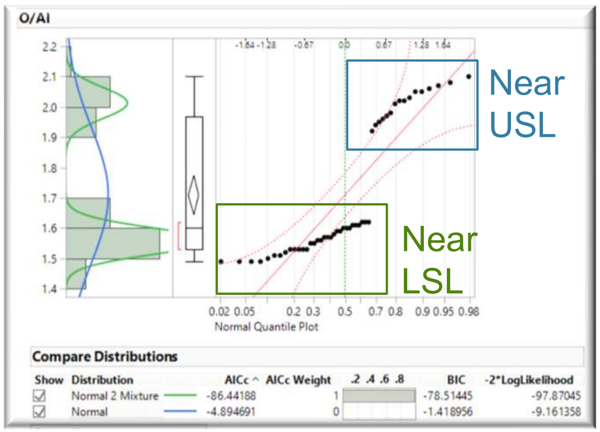


Root cause analysis | Compare each measurement

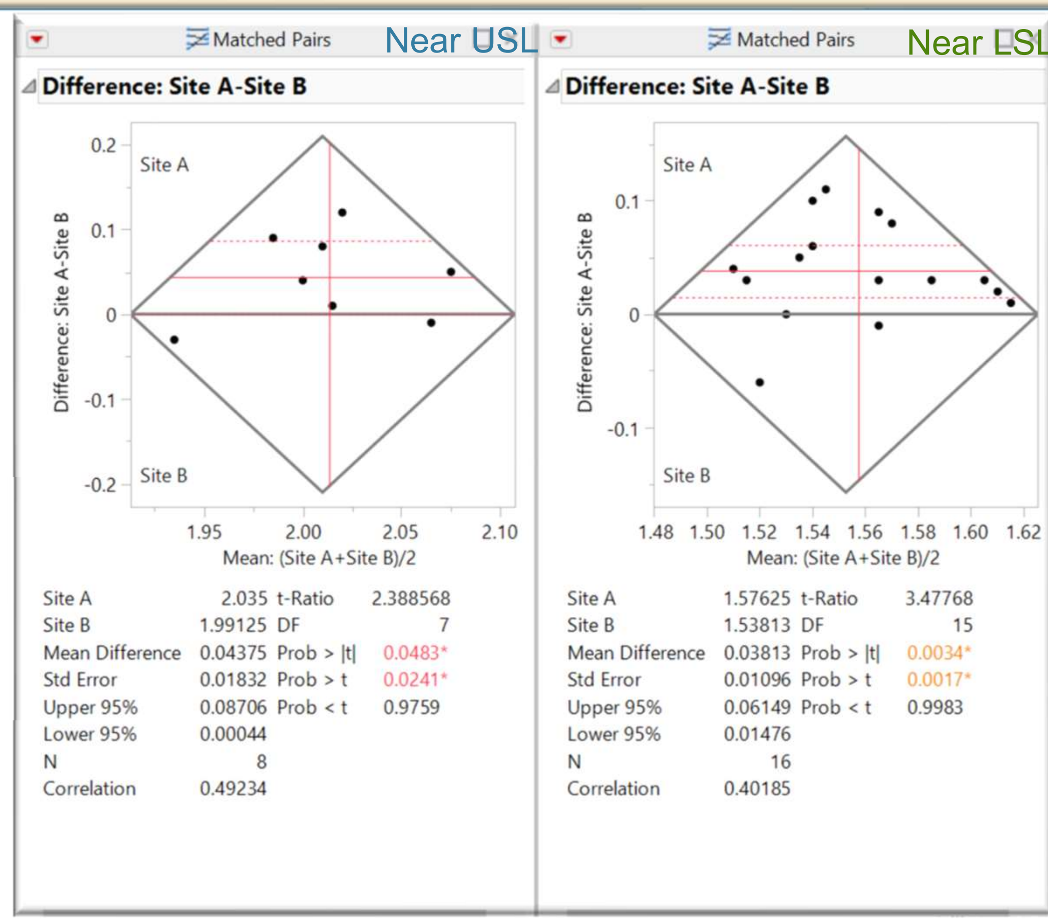
JMP Platform: Analyze > Specialized Modeling > Matched Pairs

Difference in each measurement – Site A vs. Site B

In majority Site A measured data is higher than Site B



JMP Platform: Analyze > Distribution



Matched pairs model

- » $P<0.05$

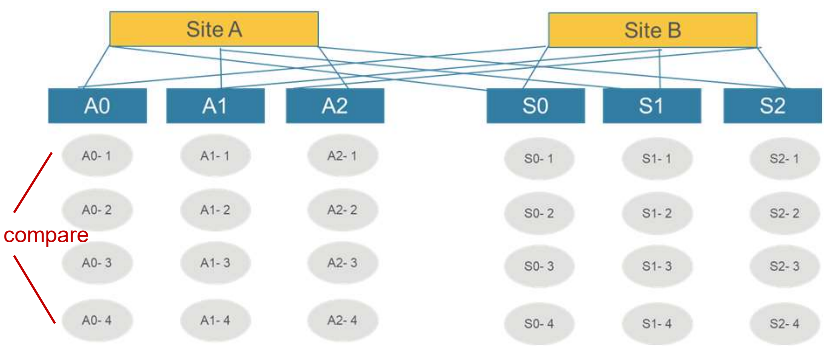
Site A measurement significantly different from Site B

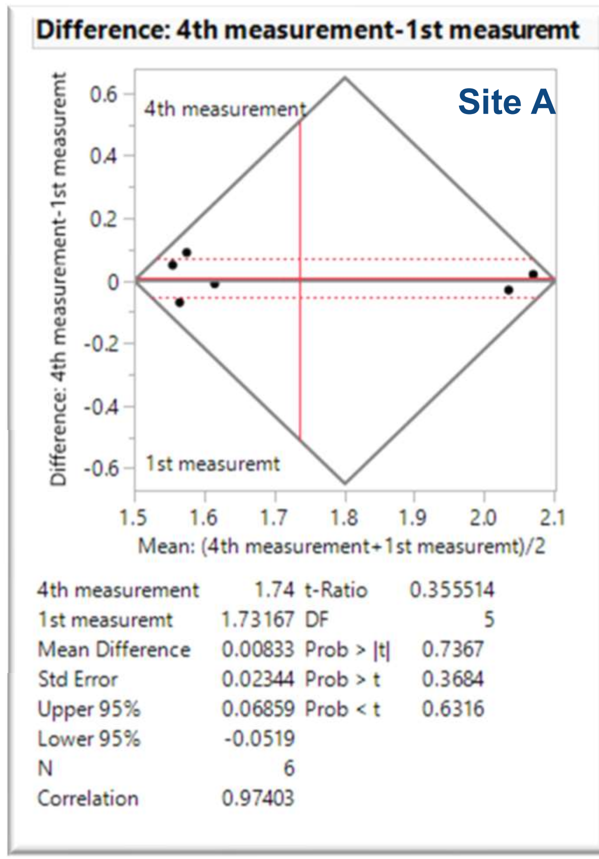
Part and site interaction is observed

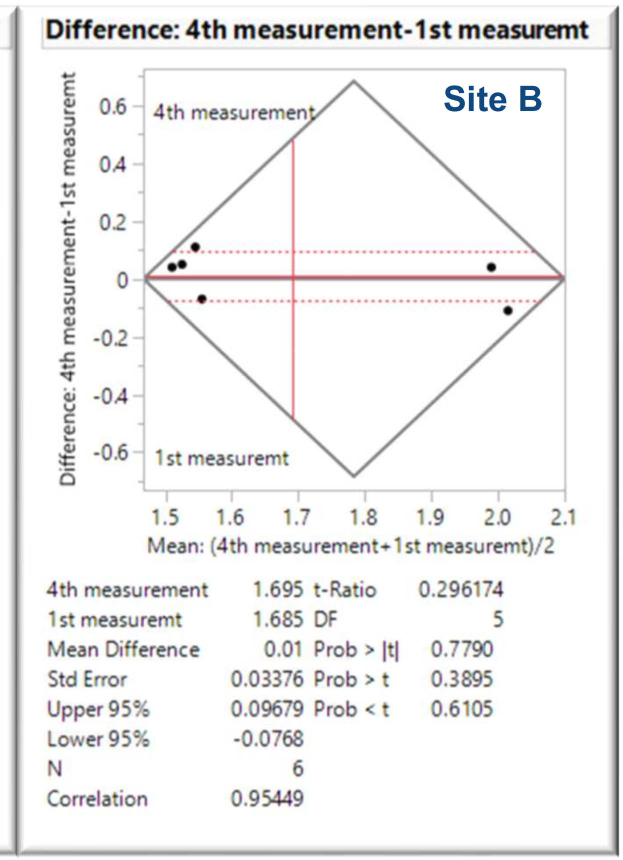
Root cause analysis I Risk of part degradation

JMP Platform: Analyze > Specialized Modeling > Matched Pairs

1st vs. 4th replicate – Site A and Site B







Matched pairs model

- P>0.05
- Eliminated risk of part degration

No evidence of part degradation between 1st and 4th replicates

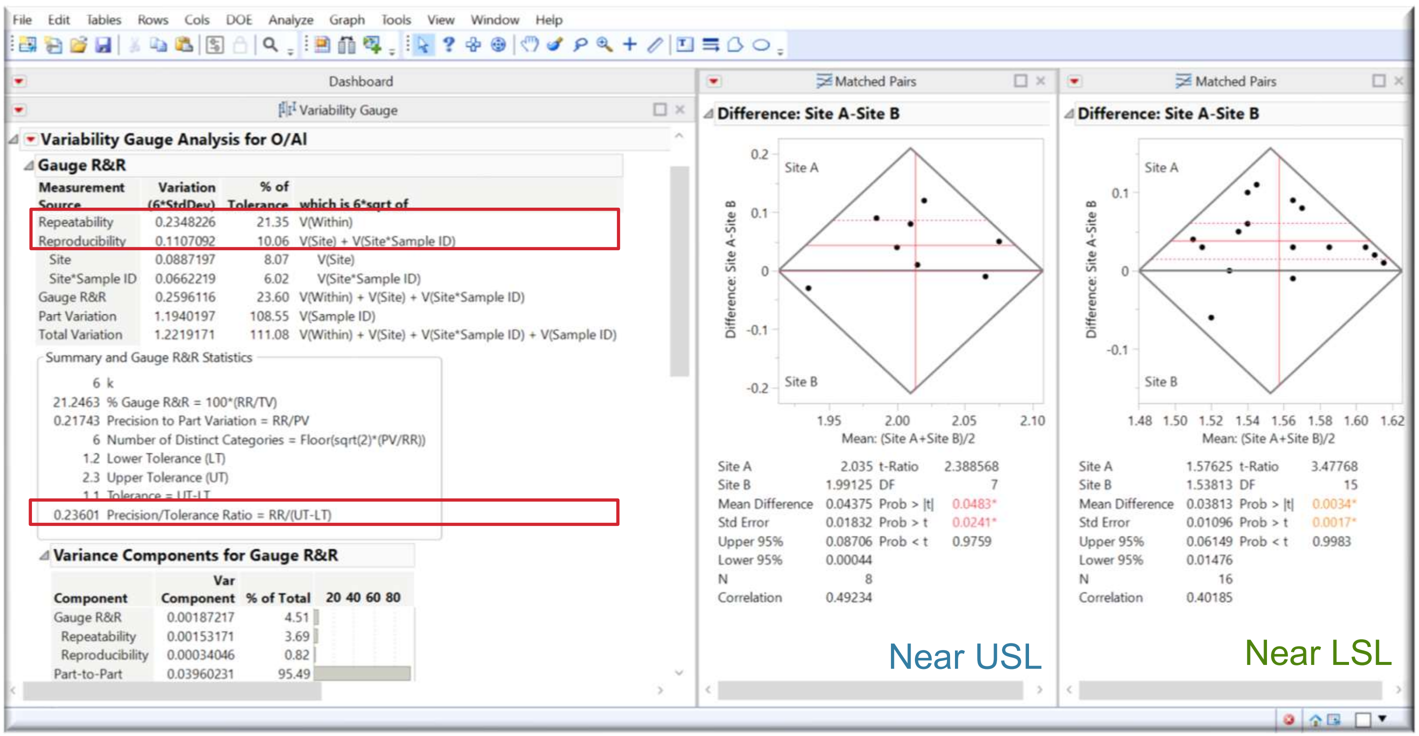
APPLIED MATERIALS

JMP Platform: File > Dashboard

Root cause analysis I Dashboard Summary

Why repeatability is a key problem

- P/T (24%) & repeatability (21%) are high since repeatability varies part to part and site to site



APPLIED MATERIALS

MSA improvement plan

- **With Site**
  - » Issue # 1: Repeatability
  - » Issue # 2: Part – Site interaction

    - Source of errors
      - background/baseline corrections
      - Electron counts
      - Peak deconvolution

    - Discuss to set up calibration sample
    - One set of samples measure in regular time interval

- **With process team**
  - » Issue # 3: MSA sample collection
  - » Issue # 4: Part to part repetability

    - MSA samples at two spec limits could underestimate MSA components (repeatability & reproducibility) – requirement of MSA samples throughout the whole spec

    - Validate thermal map for process uniformity

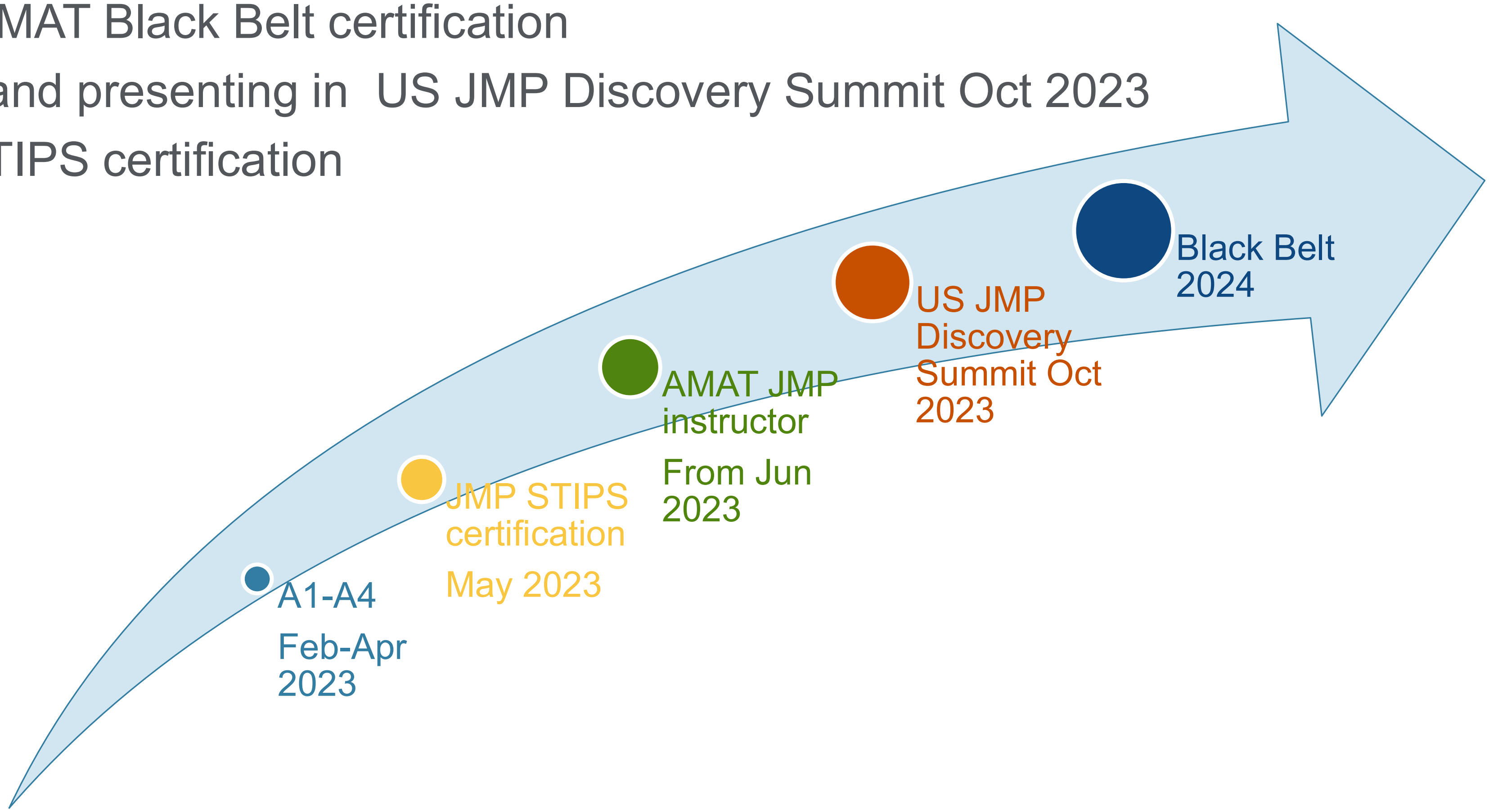
Learnings & Impact

- **Learnings – data driven measurement method validation**
  - Separating out signal variation from noise variation
  - Identification of specific GRR figure of merit to justify measurement method
  - Misclassification risks related with MSA components
  - Root cause analysis for improving MSA

- **Improve culture & practices – data driven decision making**
  - As a regular practice, apply JMP analysis to all the programs involved for improving project quality, cost and time
  - Promote data driven decision (JMP) making in Advance Technology Group

On going

- Enrolled for AMAT Black Belt certification
- Participating and presenting in US JMP Discovery Summit Oct 2023
- Completed STIPS certification



APPLIED MATERIALS®

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