Minimizing Background Signals for ICP-MS

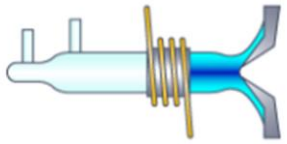
VIAL CLEANLINESS IN BACKGROUND REDUCTION

ALEXANDRA GAFFNEY

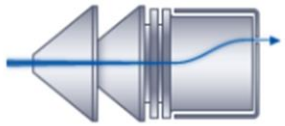
ICP-MS Operational Workflow

Inductively Couple Plasma Mass Spectrometry

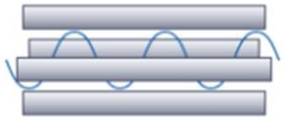
- [ ] Sample Introduction



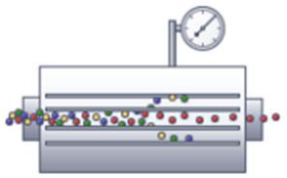
- [ ] Plasma



- [ ] Ion Lenses

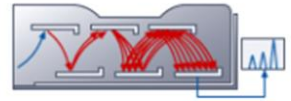


- [ ] Q1



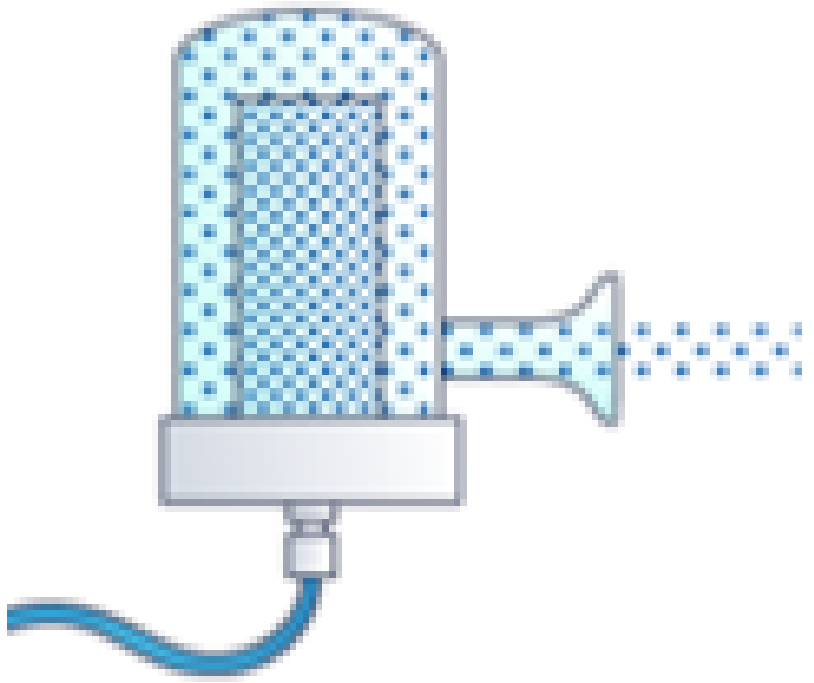
- [ ] Collision/ Reaction Cell

- [ ] Q2



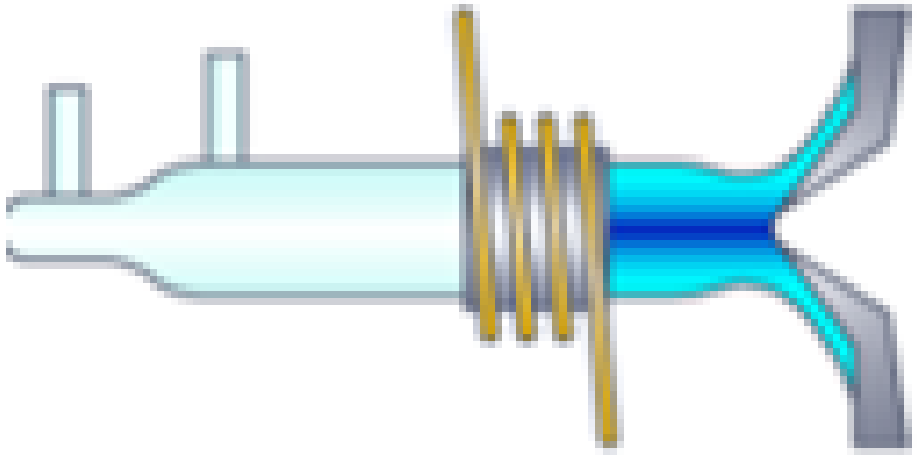
- [ ] Detector

From Sample Introduction to Ion Detection in ICP-MS



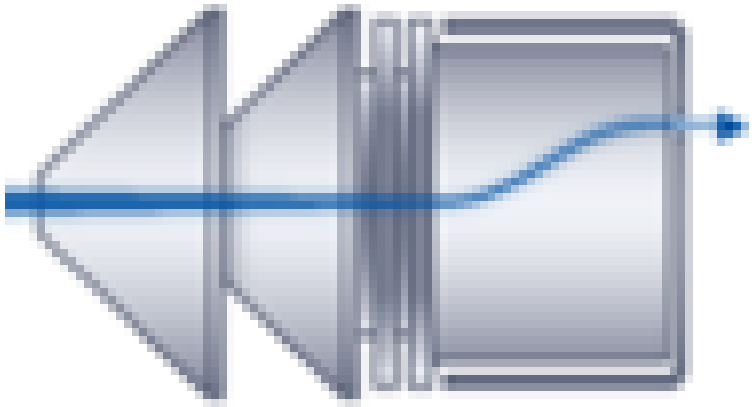
Sample Introduction

From Sample Introduction to Ion Detection in ICP-MS



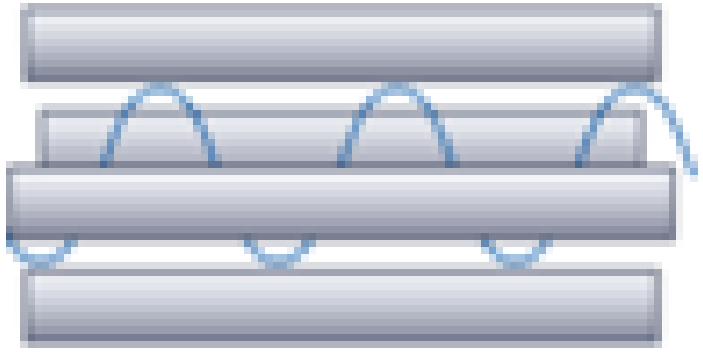
Plasma

From Sample Introduction to Ion Detection in ICP-MS



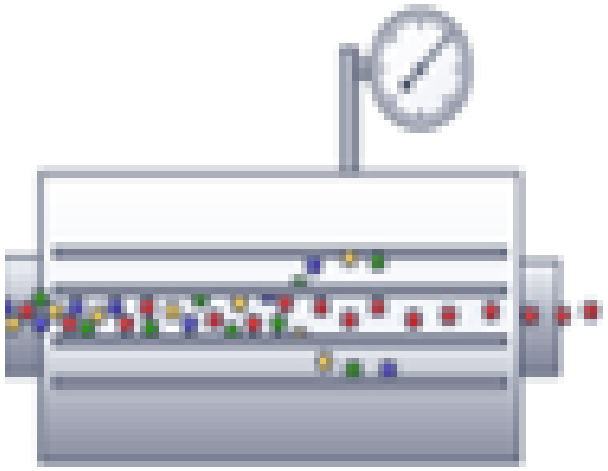
Ion Lenses

From Sample Introduction to Ion Detection in ICP-MS



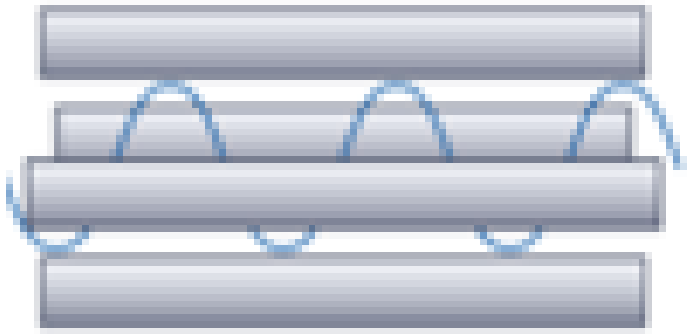
Q1

From Sample Introduction to Ion Detection in ICP-MS



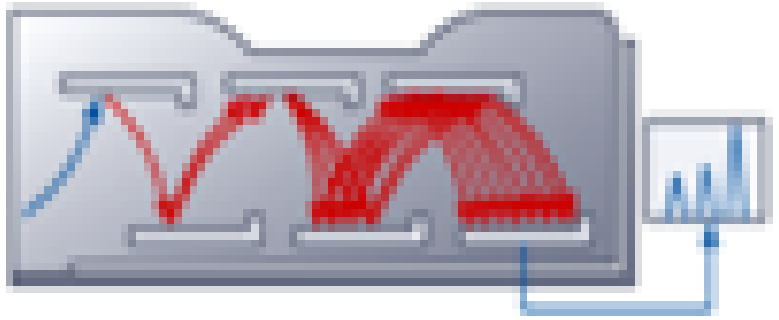
Collision/
Reaction Cell

From Sample Introduction to Ion Detection in ICP-MS



Q2

From Sample Introduction to Ion Detection in ICP-MS



Detector

ICP-MS Background: Sources & Control

Common background sources include:

- Argon-Based polyatomic (e.g., $^{38}Ar^{18}O = {}^{56}Fe$, $^{40}Ar^{35}Cl = {}^{75}As$)
- Acids, Water, Labware
- Sample Carryover
- Environmental contamination (dust, salts, metals)

Types of Background Interference

- **Spectral:** Mass Overlaps from polyatomic, doubly charged, or dimer ions.
- **Non-Spectral:** Sample Matrix effects (e.g., high dissolved solids)
- **Instrumental:** Electronic noise or internal contamination

ICP-MS Background: Importance

Why Background Control Matters:

- Lower Detection Limits
- Improved Accuracy & Precision
- Reduced False Positives
- Reliable ppt-level quantification

Sample Vial Materials In ICP-MS

Good Versus Poor Use Cases at Ultra Trace Levels

GOOD BACKGROUND USES (PP VIALS)

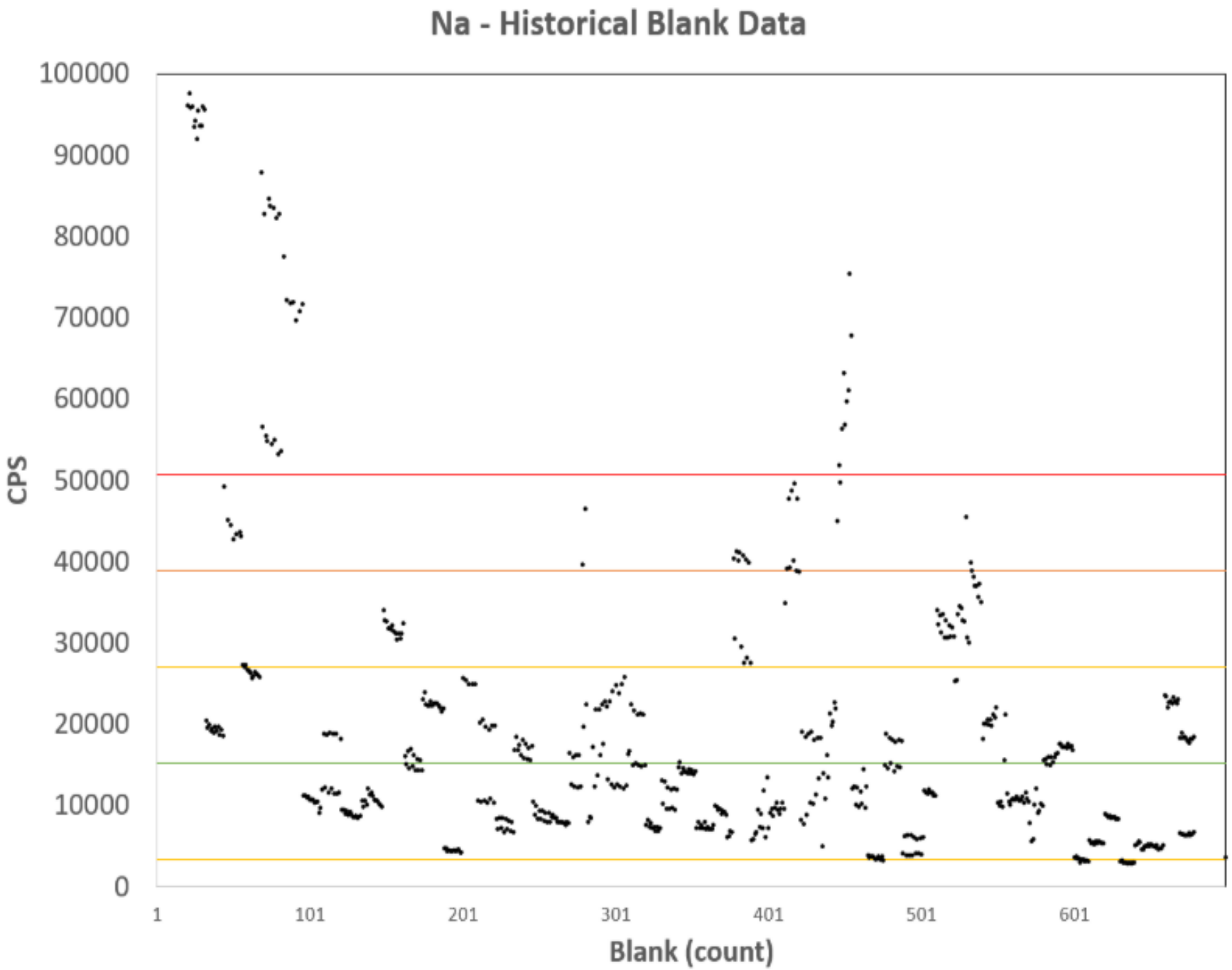
- Low metal background
- Best for ppt-level analysis
- Stable blanks & standards

POOR BACKGROUND USES (PFA & OTHER PLASTICS)

- Higher background variability
- Potential metal leaching
- Not ideal for ppt quantification

Legend

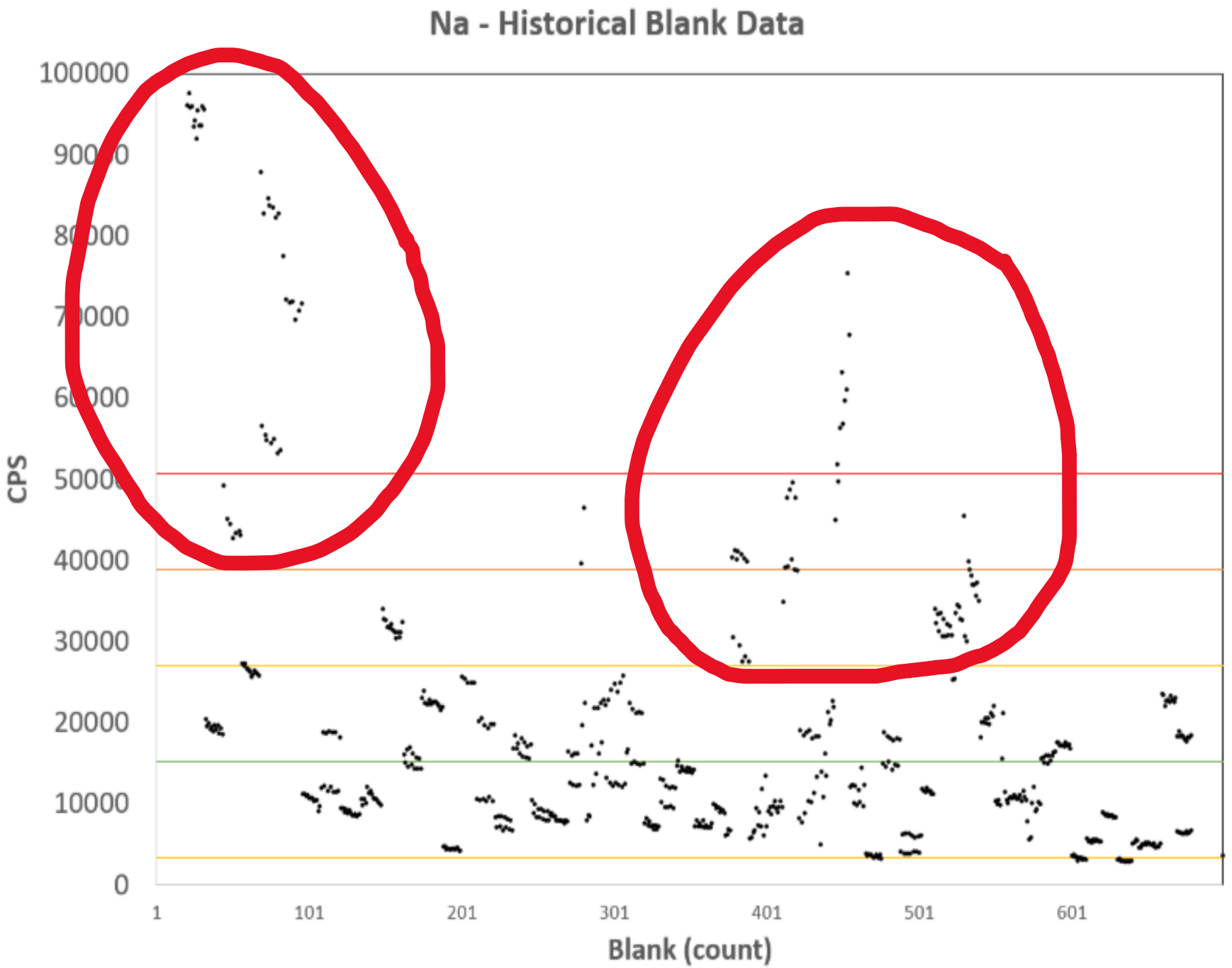
- 23 -> 23 Na [Cool]
- Mean (CPS)
- ''Mean + 2SD'' (CPS)
- ''Mean + 1SD'' (CPS)
- ''Mean - 1SD'' (CPS)
- ''Mean + 3SD'' (CPS)



Data Timeline from March 24th, 2024 – March 18th, 2026

Legend

- 23 -> 23 Na [Cool]
- Mean (CPS)
- ''Mean + 2SD'' (CPS)
- ''Mean + 1SD'' (CPS)
- ''Mean - 1SD'' (CPS)
- ''Mean + 3SD'' (CPS)



Data Timeline from March 24th, 2024 – March 18th, 2026

Background, Variability & Detection Metrics in ICP-MS

- **Noise (3σ)** – Represents natural variability of the blank. Calculated as,

  3σ = 3 * Standard Deviation Blank Signal

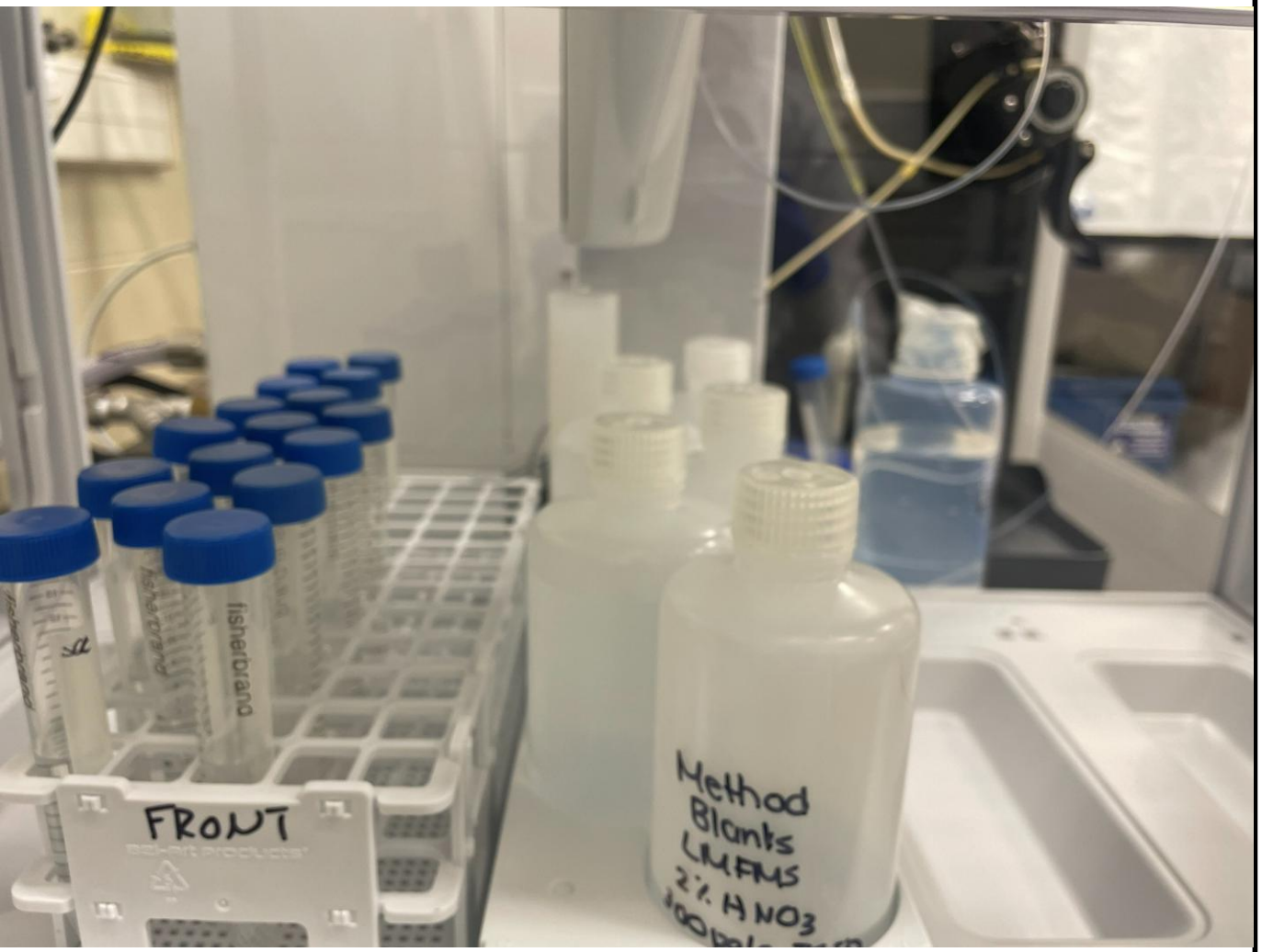
- **Background Equivalent Concentration (BEC)** – Concentration Corresponding to average blank signal. Calculated as,

  BEC = Mean Blank Counts Per Second / Sensitivity (Counts Per Second per concentration)

- **Instrument Detection Limit (IDL)** – Lowest concentration distinguishable from background noise. Calculated as,

  IDL = 3 * Standard Deviation Blank / Sensitivity

Testing Protocols



ICP-MS Sample Introduction System

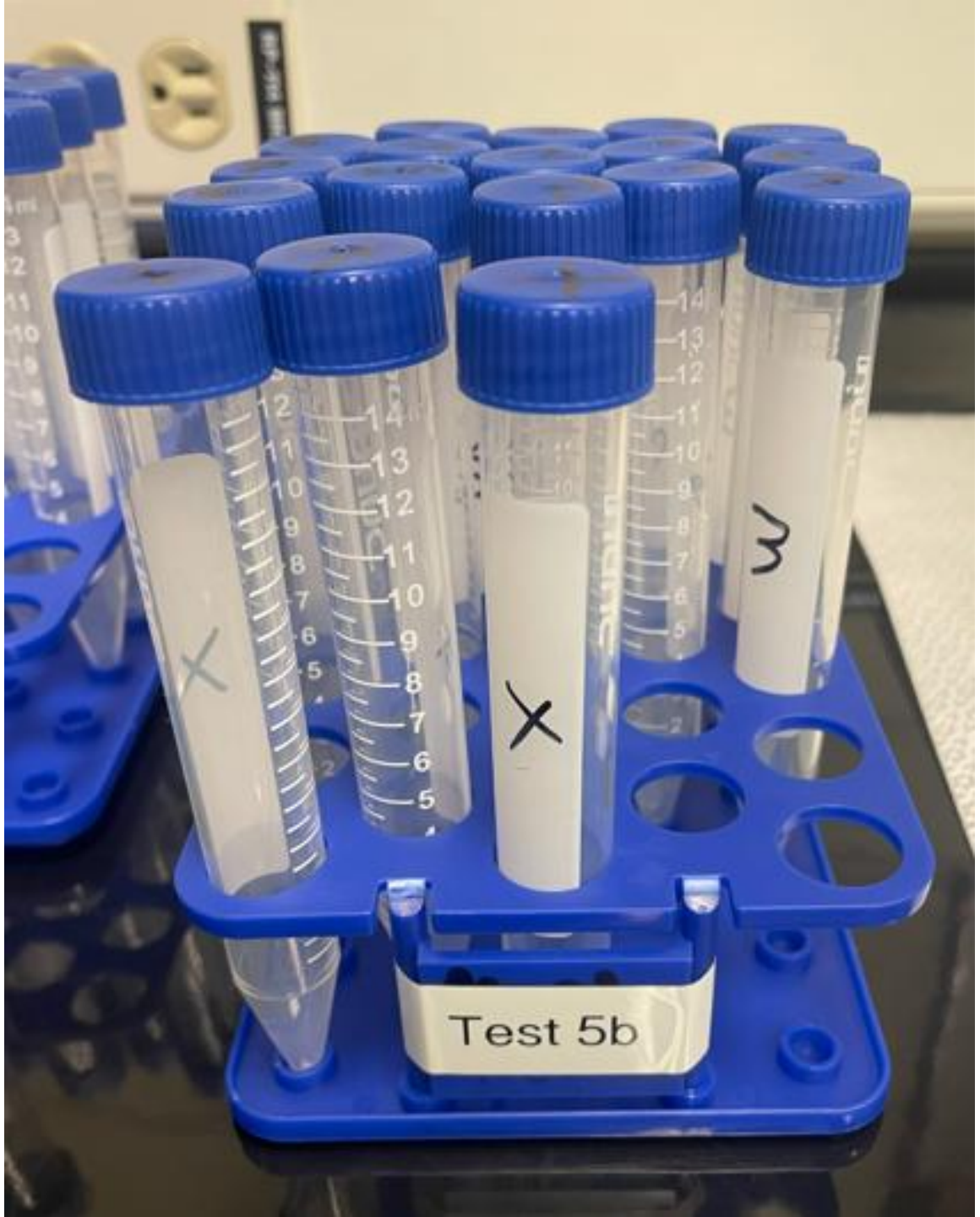
Test Cleaning Series

Cleaning Workflow

- Detergent Wash
- UPW Rinse A
- Acid Leach
- ICP-MS Analysis

Purpose

- Evaluate detergent + acid leach effectiveness for PP sample vials



Test Cleaning Series

Test Cleaning Series (24 h Leach)

- A: (2M HNO_3)
- B: (6M HNO_3)
- C: (4M HNO_3)
- D:(3M HCl; 6M HNO_3)
- E: (1M HCl; 6M HNO_3)
- F: (1M HCl + 6M HNO_3)

Detergent Tested

- Unclean Control (0)
- UPW (1)
- 1% Alconox (2)
- 2% Micro-90 (3)
- 1% Nuclean (4)
- U/S clean (5)

Differences Among Detergents

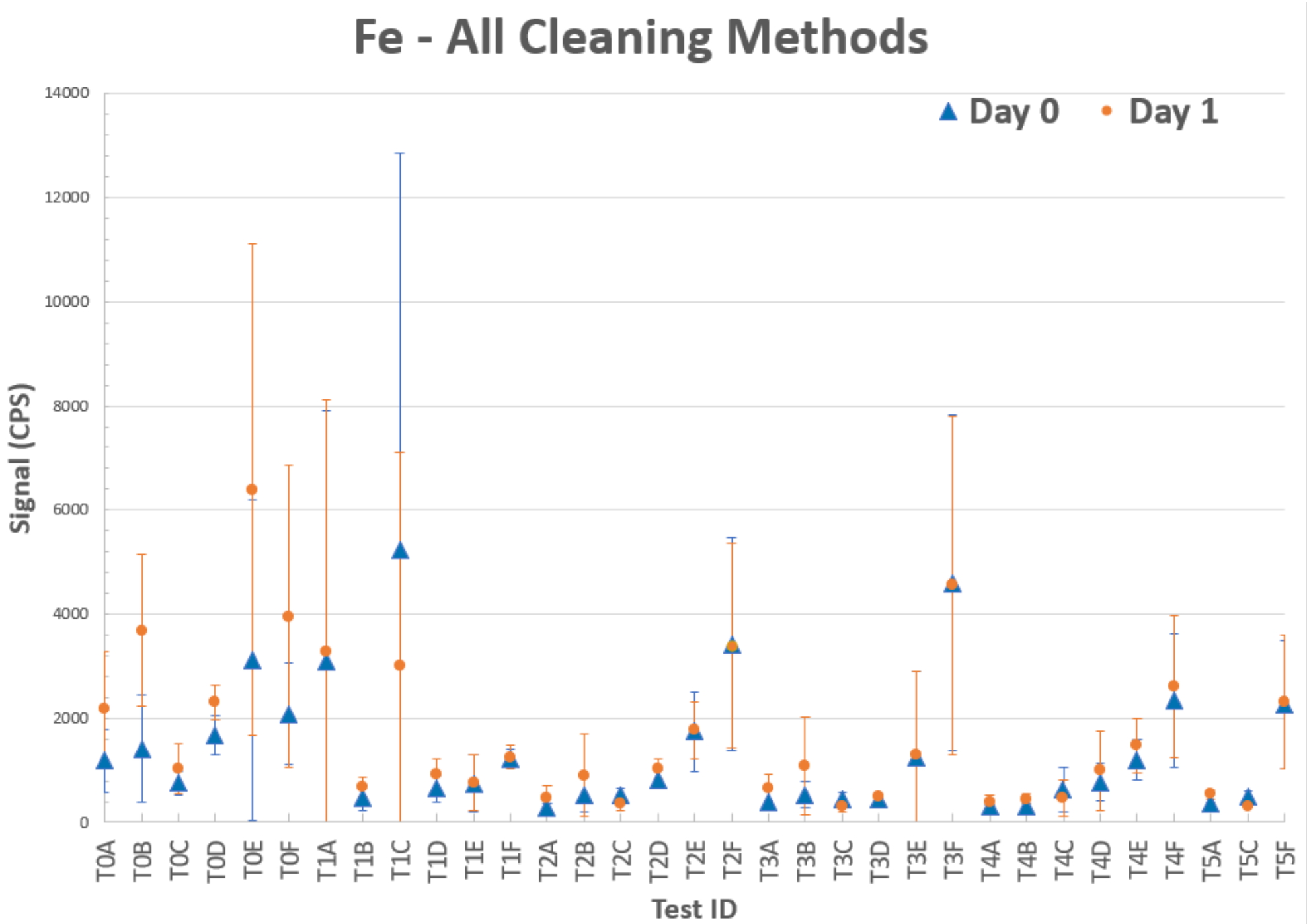
- **Alconox –** Organic Residue Removal
- **Micro-90** – Alkaline Detergent for removing oils, greases, and particulate contamination
- **Nuclean** – Acidic cleaner used to dissolve inorganics, metal oxides, and mineral scale

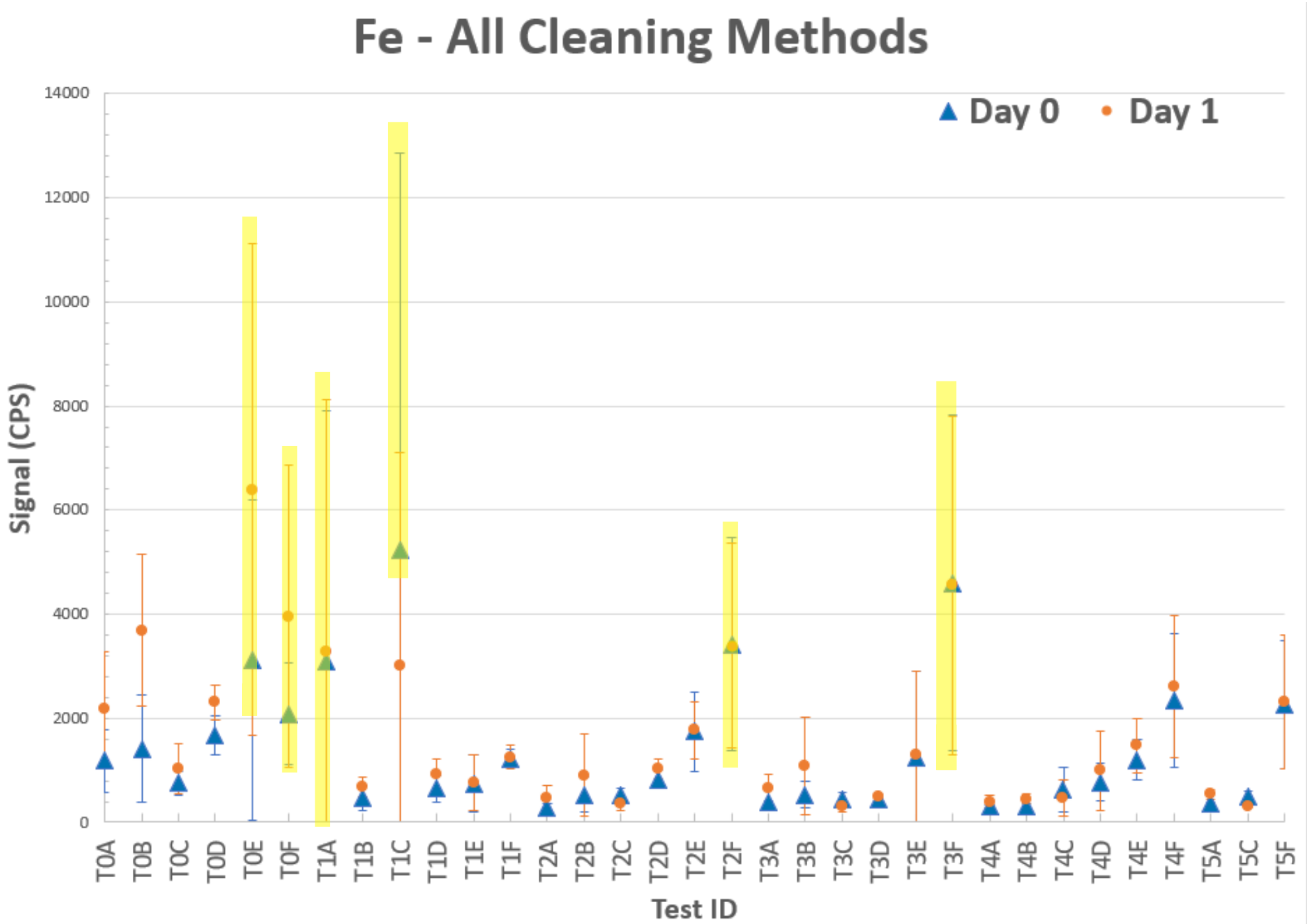
- Testing was completed on **44** analytes. Fe, Cu, Na, and Ca were selected for further evaluation due to their significant reductions in signal counts and lower variability compared to other analytes. Approximately 108 vials were analyzed, representing 6 acid combinations and 6 ''detergents'' with 3 replicate vials per condition; vials were analyzed twice each.

Purpose

- Reduce Contamination
- Minimize Background noise & analytical variability
- Improve detection limits
- Ensure stable baselines
- Support accurate trace-level measurements
- Maintain consistent instrument performance

Results


Fe - All Cleaning Methods
Day 0
Day 1
Signal (CPS)
14000
12000
10000
8000
6000
4000
2000
0
T0A
T0B
T0C
T0D
T0E
T0F
T1A
T1B
T1C
T1D
T1E
T1F
T2A
T2B
T2C
T2D
T2E
T2F
T3A
T3B
T3C
T3D
T3E
T3F
T4A
T4B
T4C
T4D
T4E
T4F
T5A
T5C
T5F
Test ID


Fe - All Cleaning Methods
Day 0
Day 1
Signal (CPS)
14000
12000
10000
8000
6000
4000
2000
0
T0A
T0B
T0C
T0D
T0E
T0F
T1A
T1B
T1C
T1D
T1E
T1F
T2A
T2B
T2C
T2D
T2E
T2F
T3A
T3B
T3C
T3D
T3E
T3F
T4A
T4B
T4C
T4D
T4E
T4F
T5A
T5C
T5F
Test ID

Iron (Fe) Cleaning Performance

- **Best Performance:** Acid B **(6M HNO_3)** & Detergent 4 **(1% Nuclean)**
  - 77.4% Reduction in background cps relative to TØ

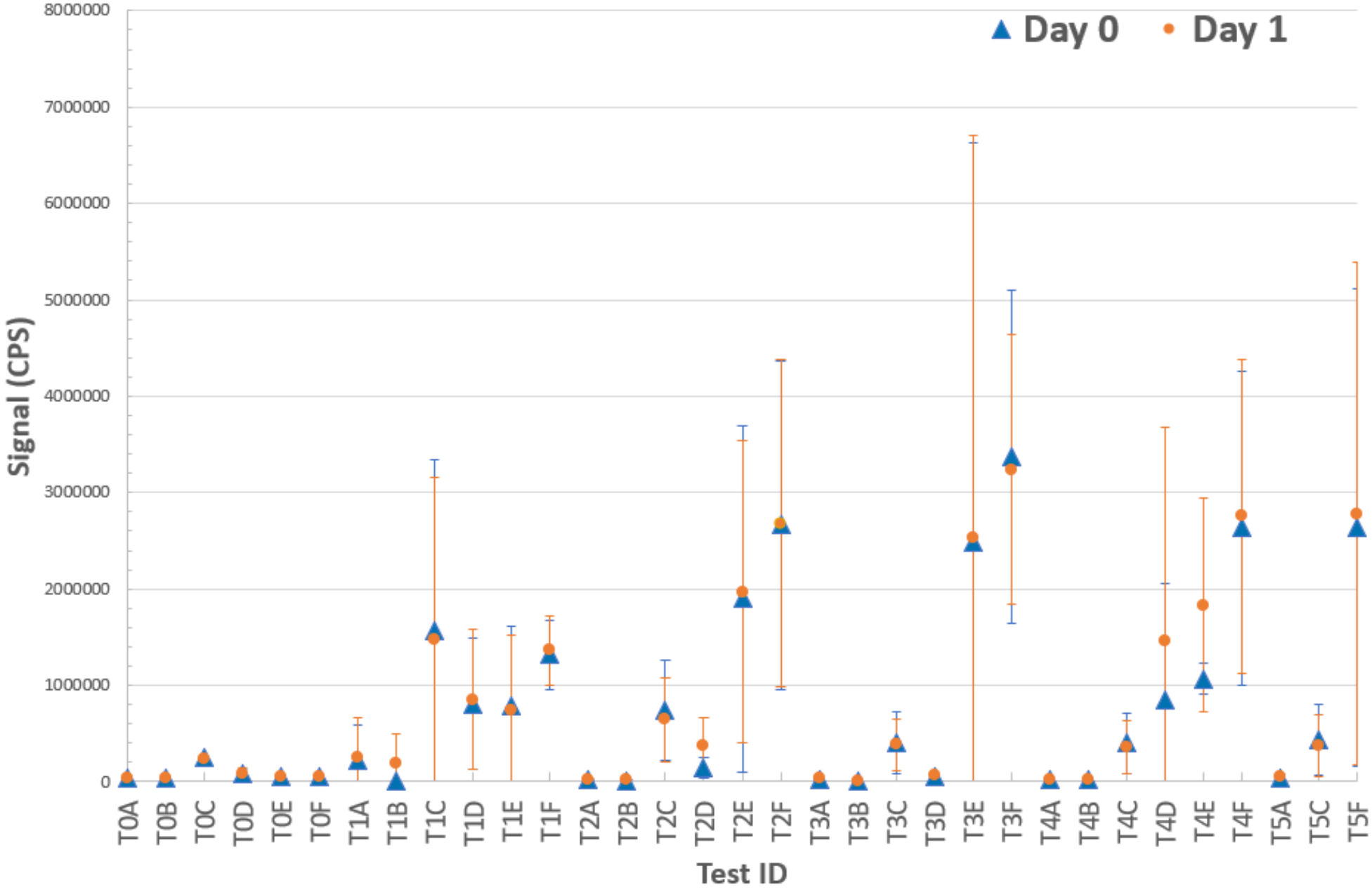
Parameter	TØ (Unclean Vials)	Old Cleaning Method (n=419)	New Cleaning Method (n=3)
Mean (CPS)	1416	570	320
SD (CPS)	1026	52	34
BEC (pg/g)	20.3 pg/g	8.3 pg/g	4.6 pg/g
IDL (og/)	44.2 pg/g	2.3 pg/g	1.5 pg/g

Iron (Fe) Cleaning Performance

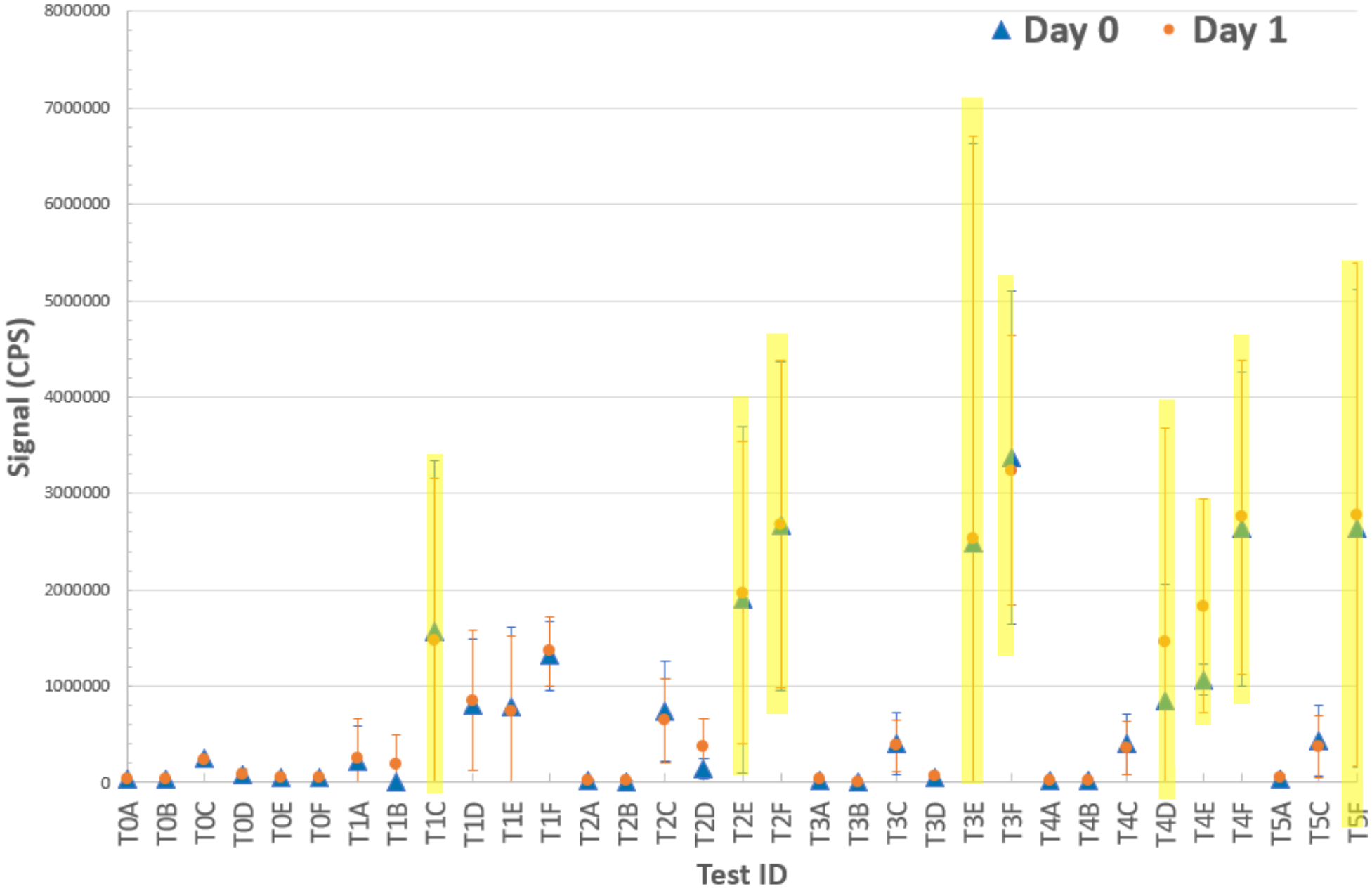
- **Best Performance:** Acid B **(6M HNO_3)** & Detergent 4 (1% Nuclean)
  - 77.4% Reduction in background cps relative to TØ

Parameter	TØ (Unclean Vials)	Old Cleaning Method (419 data points)	New Cleaning Method (3 data points)
Mean CPS	1416	570	320
SD CPS	1026	52	34
BEC	20.3 g/g	8.3 pg/g	4.6 pg/g
IDL	44.2 pg/g	2.3 pg/g	1.5 pg/g

Na - All Cleaning Methods



Na - All Cleaning Methods



Sodium (Na) Cleaning Performance

- **Best Performance:** Acid B **(6M HNO_3)** & Detergent 3 **(2% Micro-90)**
  - 76.3% Reduction in background cps relative to TØ

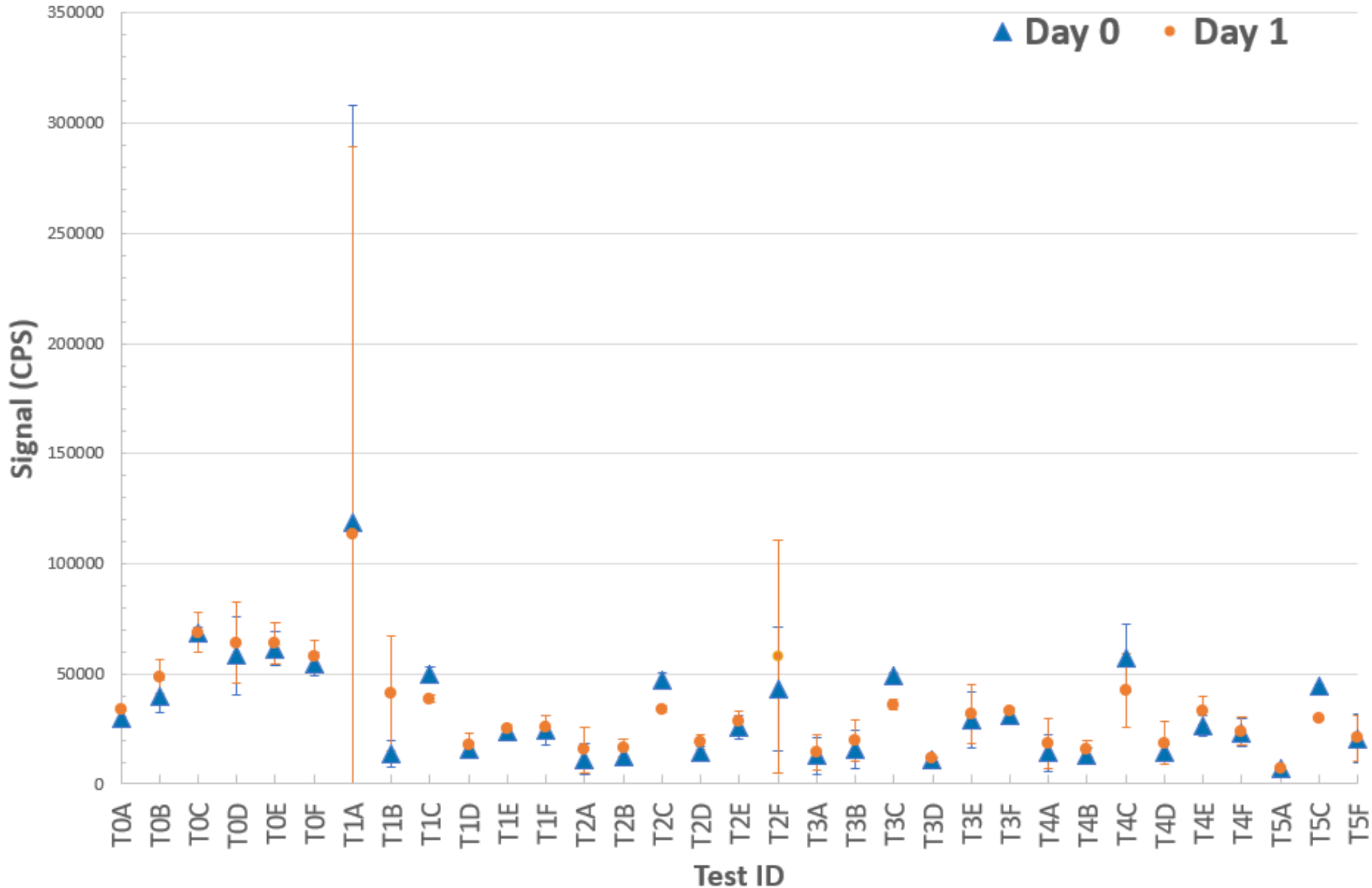
Parameter	TØ (Unclean Vials)	Old Cleaning Method (419 data points)	New Cleaning Method (3 data points)
Mean CPS	34,225	15,000	8000
SD CPS	6061	12,000	760
BEC	108.3 pg/g	47.5 pg/g	25.3 pg/g
IDL	57.6 g/g	114 pg/g	7.22 pg/g

Sodium (Na) Cleaning Performance

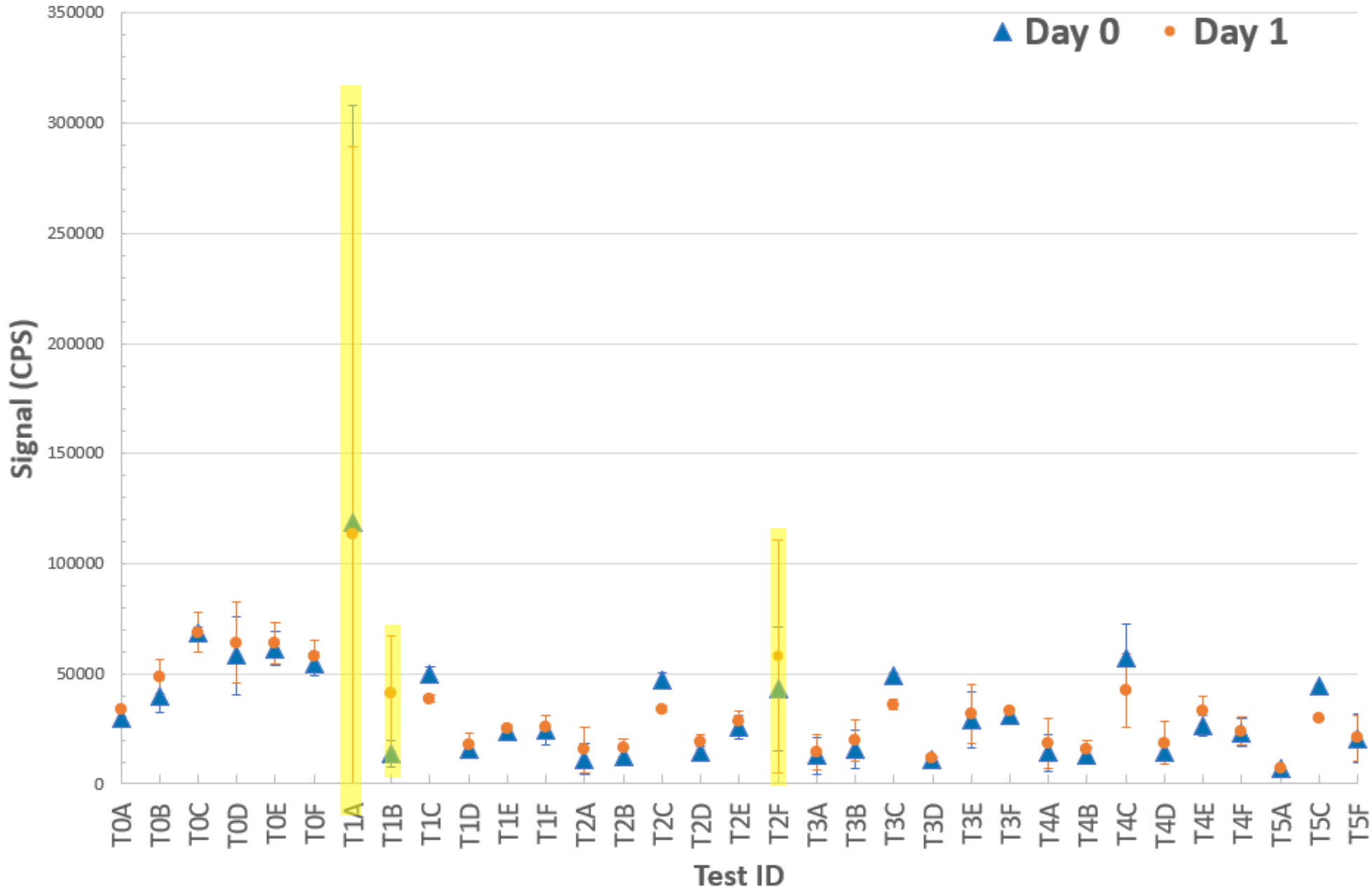
- **Best Performance:** Acid B **(6M HNO_3)** & Detergent 3 (2% Micro-90)
  - 76.3% Reduction in background cps relative to TØ

Parameter	TØ (Unclean Vials)	Old Cleaning Method (419 data points)	New Cleaning Method (3 data points)
Mean CPS	34,225	15,000	8000
SD CPS	6061	12,000	760
BEC	108.3 pg/g	47.5 pg/g	25.3 pg/g
IDL	57.6 g/g	114 pg/g	7.22 pg/g

Ca - All Cleaning Methods



Ca - All Cleaning Methods



Calcium (Ca) Cleaning Performance

- Acid D **(3M HCl + 6M HNO_3)** & Detergent 3 **(2% Micro-90)**
  - 81.6% Reduction in background cps relative to TØ

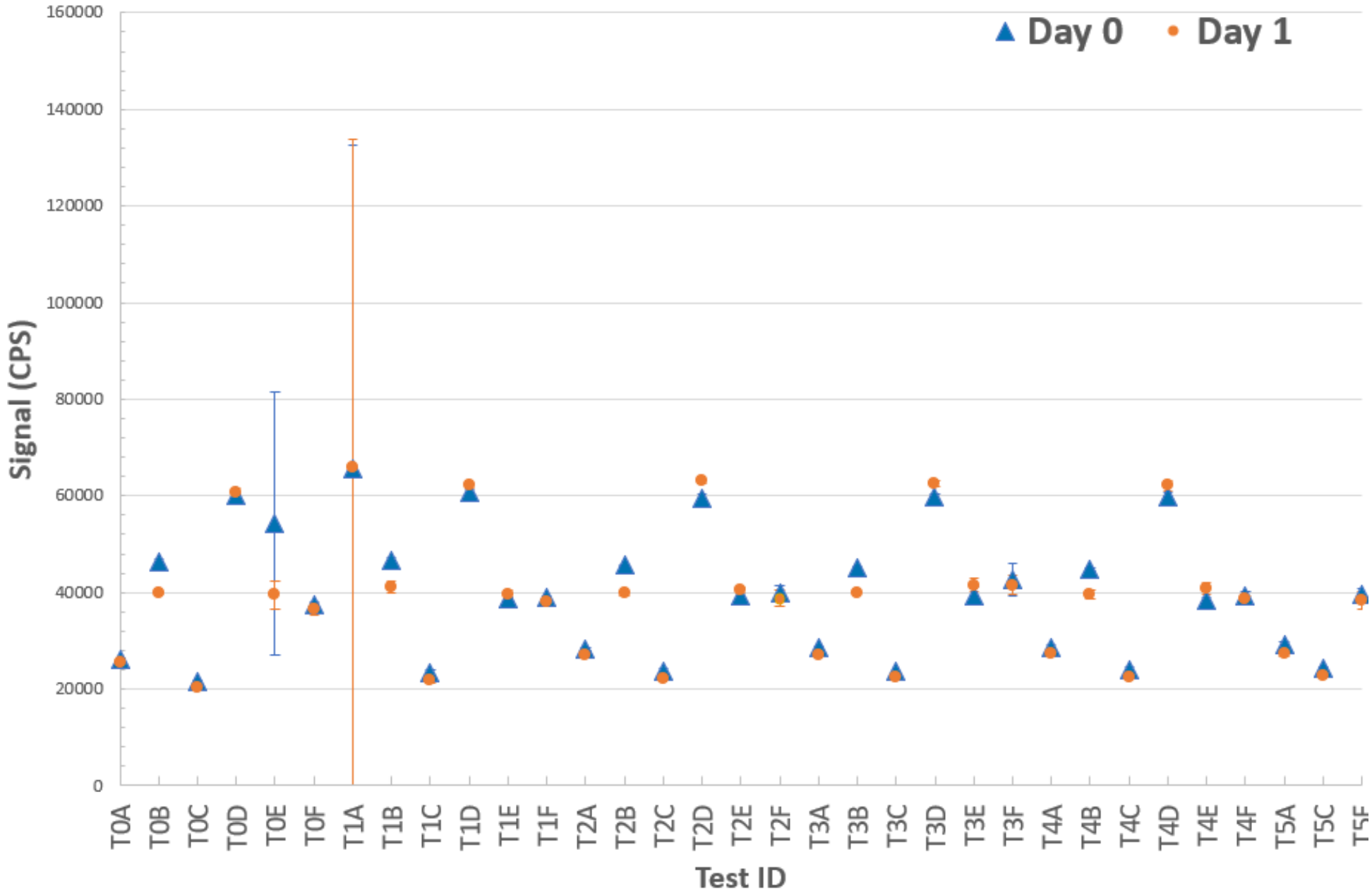
Parameter	TØ (Unclean Vials)	Old Cleaning Method (419 data points)	New Cleaning Method (3 data points)
Mean CPS	58,276	13,000	10,000
SD CPS	17,526	10,000	1000
BEC	979 pg/g	219 pg/g	168 pg/g
IDL	883 pg/g	505 pg/g	50.5 pg/g

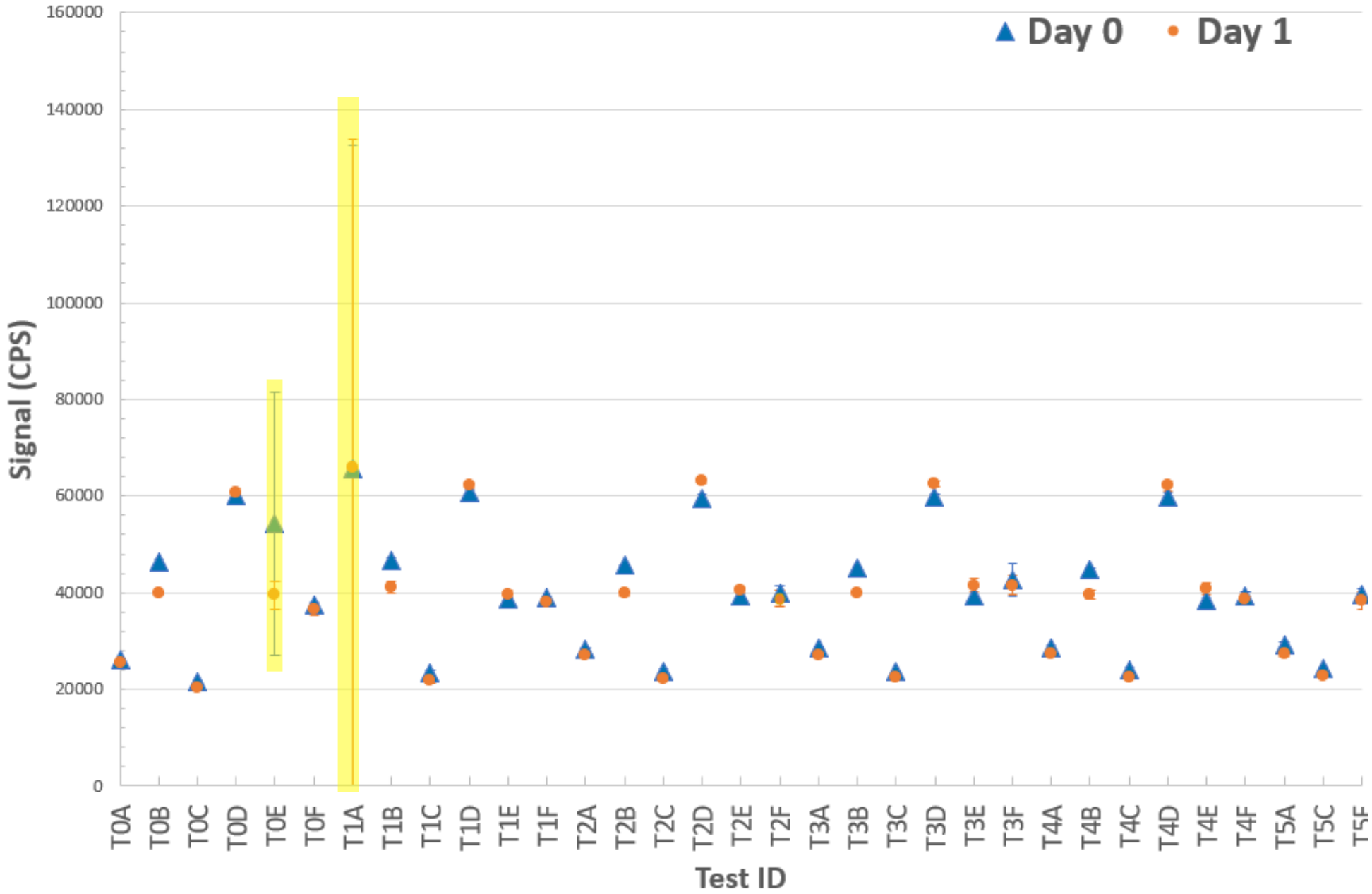
Calcium (Ca) Cleaning Performance

- Acid D **(3M HCl + 6M HNO_3)** & Detergent 3 **(2% Micro-90)**
  - 81.6% Reduction in background cps relative to TØ

Parameter	TØ (Unclean Vials)	Old Cleaning Method (419 data points)	New Cleaning Method (3 data points)
Mean CPS	58,276	13,000	10,000
SD CPS	17,526	10,000	1000
BEC	979 pg/g	219 pg/g	168 pg/g
IDL	883 pg/g	505 pg/g	50.5 pg/g

Cu - All Cleaning Methods




Cu - All Cleaning Methods
Day 0
Day 1
Signal (CPS)
160000
140000
120000
100000
80000
60000
40000
20000
0
T0A
T0B
T0C
T0D
T0E
T0F
T1A
T1B
T1C
T1D
T1E
T1F
T2A
T2B
T2C
T2D
T2E
T2F
T3A
T3B
T3C
T3D
T3E
T3F
T4A
T4B
T4C
T4D
T4E
T4F
T5A
T5C
T5F
Test ID

Copper (Cu) Cleaning Performance

Best Performance: Acid E (1M HCl + 6M HNO_3) & Detergent 3 (2% Micro-90)

- 29.1% Reduction in background cps relative to TØ

Parameter	TØ (Unclean Vials)	Old Cleaning Method (419 data points)	New Cleaning Method (3 data points)
Mean CPS	54,157	34,000	27,209
SD CPS	27,209	17,000	1763
BEC	277 pg/g	180 pg/g	201pg/g
IDL	418 pg/g	270 pg/g	11.6 pg/g

Copper (Cu) Cleaning Performance

Best Performance: Acid E **(1M HCl + 6M HNO_3)** & Detergent 3 **(2% Micro-90)**

- 29.1% Reduction in background cps relative to TØ

Parameter	TØ (Unclean Vials)	Old Cleaning Method (419 data points)	New Cleaning Method (3 data points)
Mean CPS	54,157	34,000	27,209
SD CPS	27,209	17,000	1763
BEC	277 pg/g	180 pg/g	201pg/g
IDL	418 pg/g	270 pg/g	11.6 pg/g

Next Steps & Moving Forward

- Testing Second LOT of Vials to assess reproducibility
- Test for contamination to determine cleaning method effectiveness and establish clear criteria for when vials are no longer acceptable for use
    - 1% Alconox, 1% Nuclean, 2 % Micro-90 with Acids 2M HNO3, 6M HNO3, 3M HCl; 6M HNO3
- Perform MDL assessment for the final cleaning protocol (100 data points across 10 runs over 10 days), followed by LOQ evaluation for analytes

Key Takeaway

- Optimized detergent and acid cleaning
- Reduced background and analytical variability
- Improved reproducibility
- Lower and stable baselines
- Enhanced ICP-MS accuracy
- Supports efficient and sustainable lab practices

Questions?