



Automating the Solid Phase Extraction of PFAS for a Range of Methods and Matrices



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Presentation Outline

1

PFAS Extraction Methods

2

Automation Considerations

3

Drinking Water
(533, 537.1)

4

Non-potable Water
(ISO 21675, EPA 1633)

5

Soil
(In-house)

SPE-03

8-Channel Automated SPE System



PFAS SPE Methods

Matrices	Methods
Drinking Water	EPA Method 537.1 18 compounds EPA Method 533 25 compounds



PFAS SPE Methods

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Drinking Water	EPA Method 537.1 18 compounds EPA Method 533 25 compounds
Non-Potable Water	Proprietary Methods >40 compounds ISO 21675 (water) >30 compounds EPA Method 1633 40 compounds

PFAS SPE Methods

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Drinking Water	EPA Method 537.1 18 compounds EPA Method 533 25 compounds
Non-Potable Water	Proprietary Methods >40 compounds ISO 21675 (water) >30 compounds EPA Method 1633 40 compounds
Solids/Tissues/Soil	SPE Clean Up > 40 compounds EPA Method 1633 40 compounds

Addressing Challenges of Manual SPE



Manual SPE



Addressing Challenges of Manual SPE



Manual SPE



SPE-03 (8-Channel)

Considerations of PFAS Automation

1. Size and Efficiency

- Compact footprint
- 8 samples in parallel

Valves in conventional design



Patented multi-channel valve



Considerations of PFAS Automation

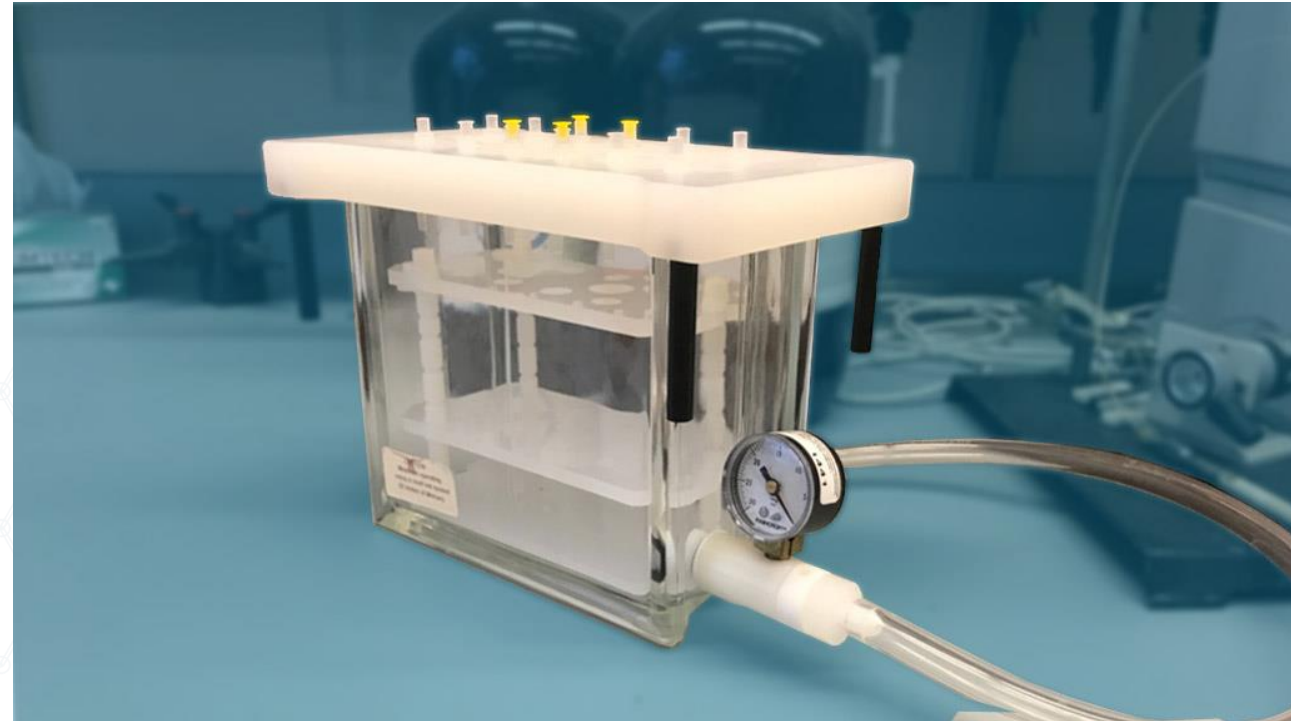
1. Size and Efficiency

- Compact footprint
- 8 samples in parallel

2. Flow control and Cartridge Clogging

Vacuum Manifold

- One shared vacuum source
- Non-uniform flow
- Constant supervision
- Clogs easily



Considerations of PFAS Automation

1. Size and Efficiency

- Compact footprint
- 8 samples in parallel

2. Flow control and Cartridge Clogging

- Positive-pressure syringe pumps

SPE-03 Pumps

- Positive pressure
- Resistant to clogging
- Uniform flow across all samples
- Sorbent does not go dry



Considerations of PFAS Automation

1. Size and Efficiency

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2. Flow control and Cartridge Clogging

- Positive-pressure syringe pumps
- Inline filters

Inline Filters

- Keep particulates out
- Streamlined with extraction
- PFAS-free, good recovery



Considerations of PFAS Automation

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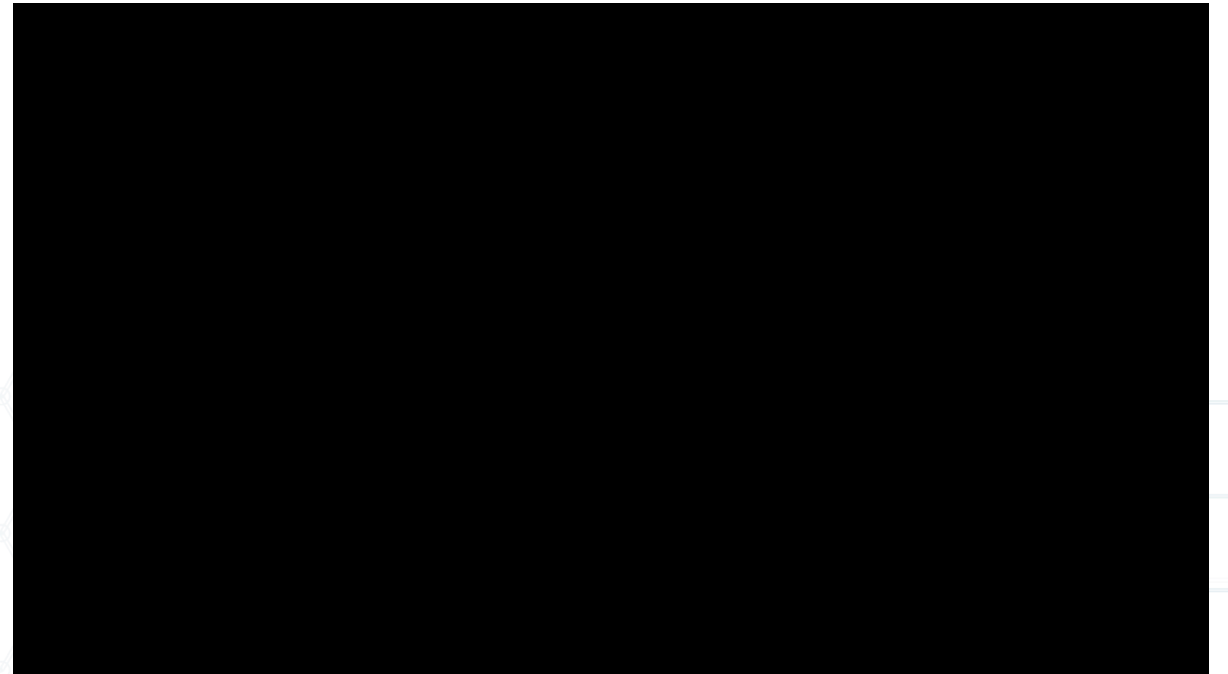
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3. Bottle Rinsing

- Integrated rack



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- Built-in resonators



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- Integrated rack
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- MOD-00P (Volume-Matrix Plus)



Results and Discussion



Drinking Water



Non-potable Water



Soil

Results and Discussion – Drinking Water



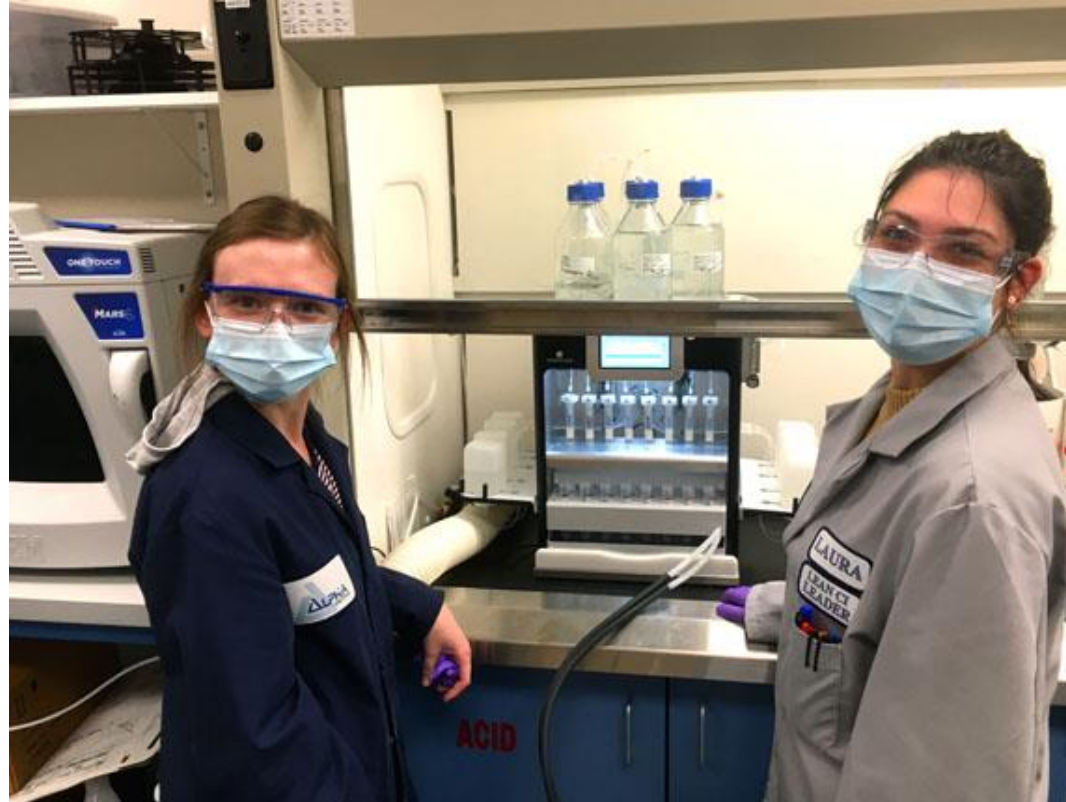
Drinking Water

1. EPA Method 533
2. EPA Method 537.1

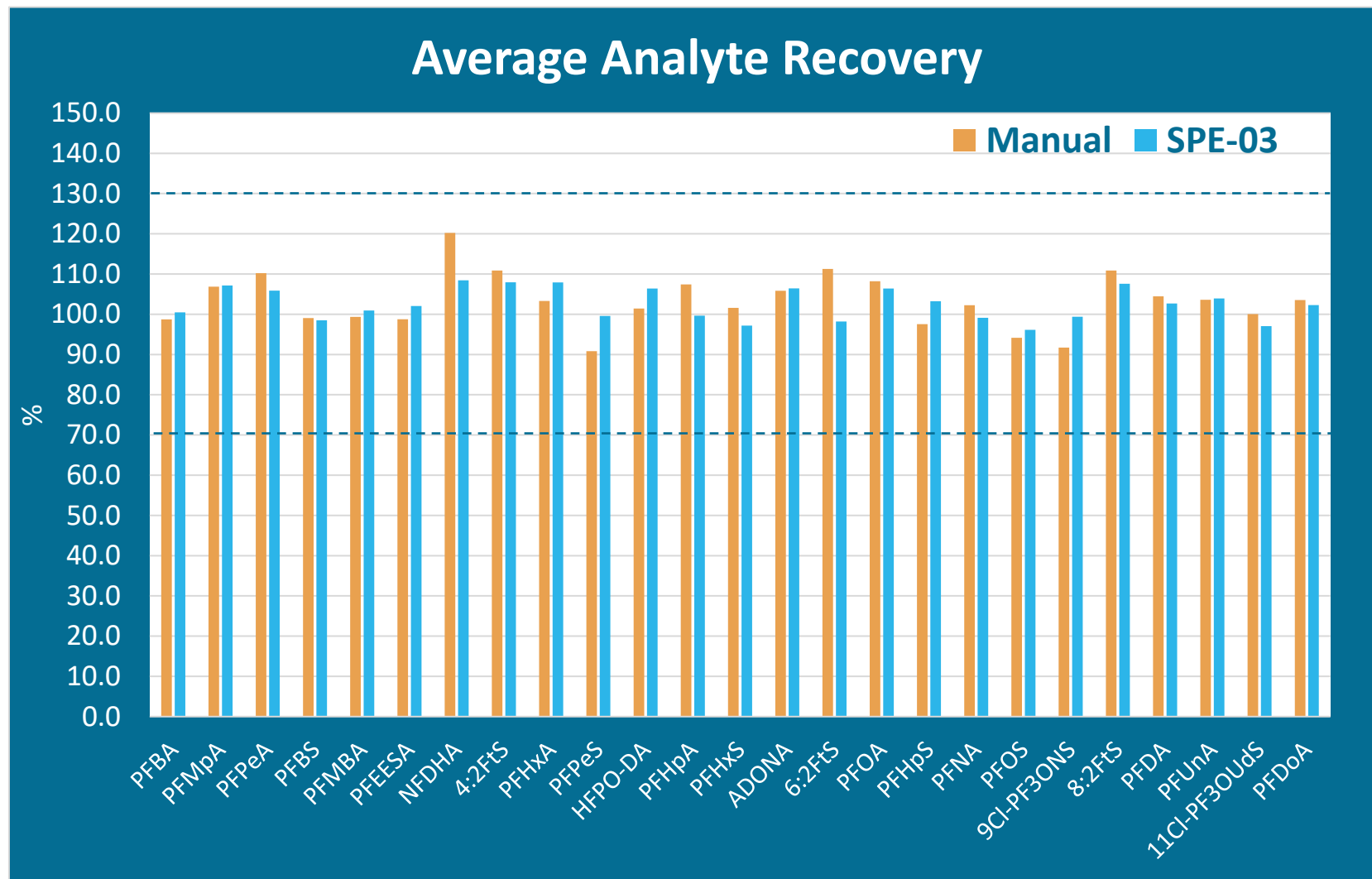


Method 533 - Data Collection

- Field extraction data from **Alpha Analytical**, Massachusetts, March to April 2021
- Including both **SPE-03** and **manual extraction**



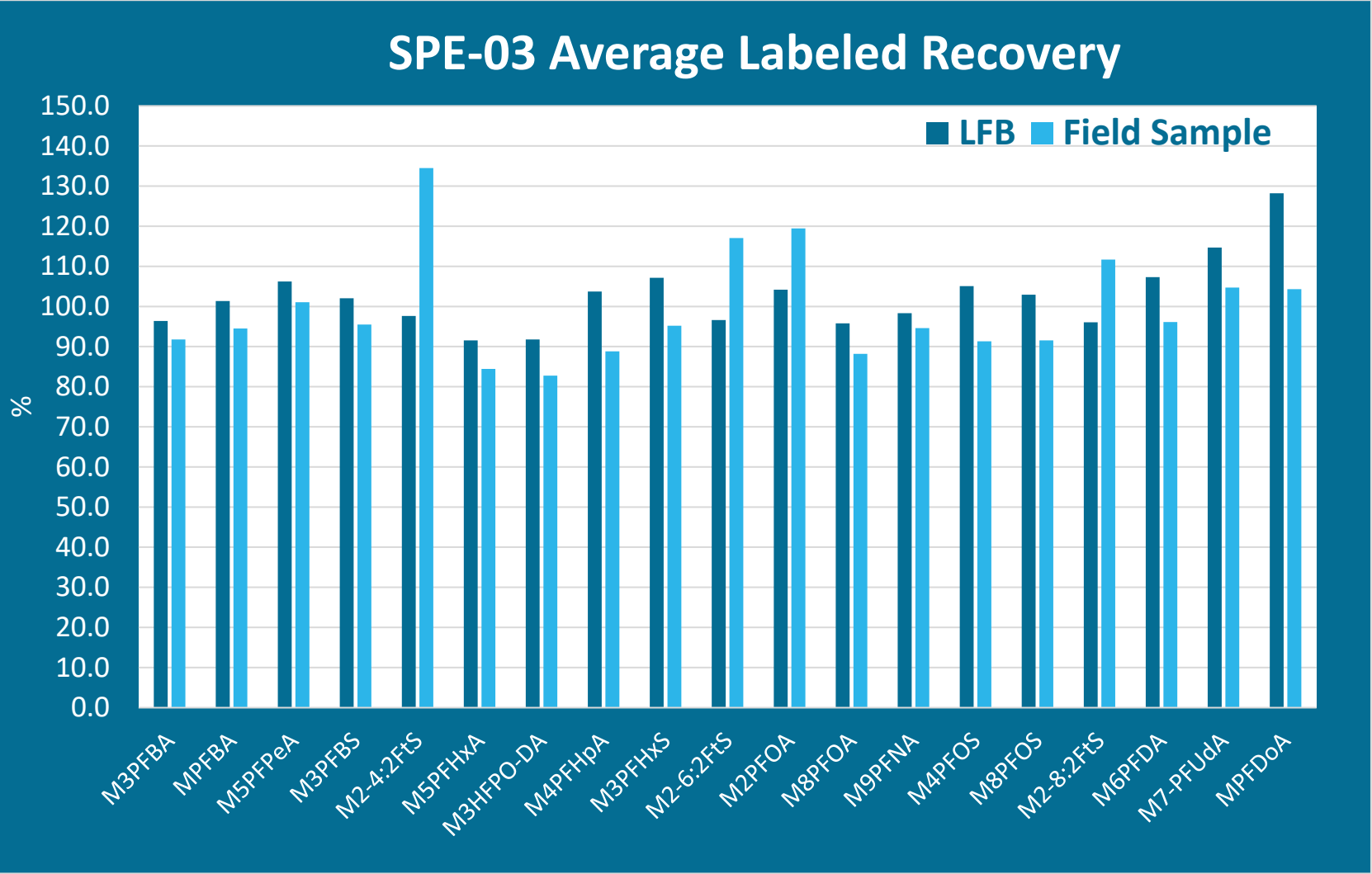
Method 533 - Results – Accuracy



Manual and SPE-03 LFB Recovery

- N = 8
- SPE-03: 95% to 110%
- Manual: 90% to 120%

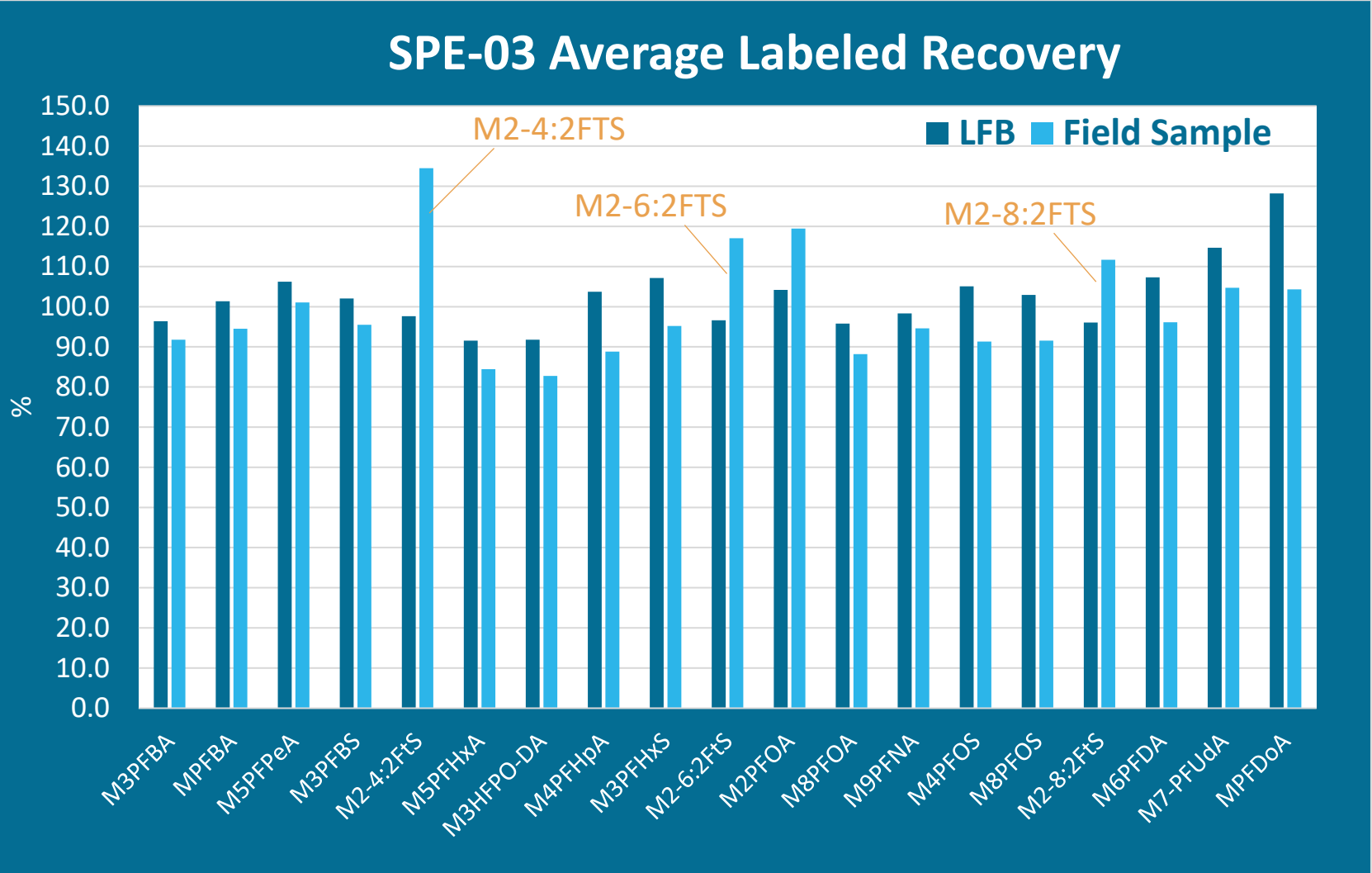
Method 533 - Results – Matrix Tolerance



LFB vs Field Sample Labeled Recovery on SPE-03

- N = 24 field samples

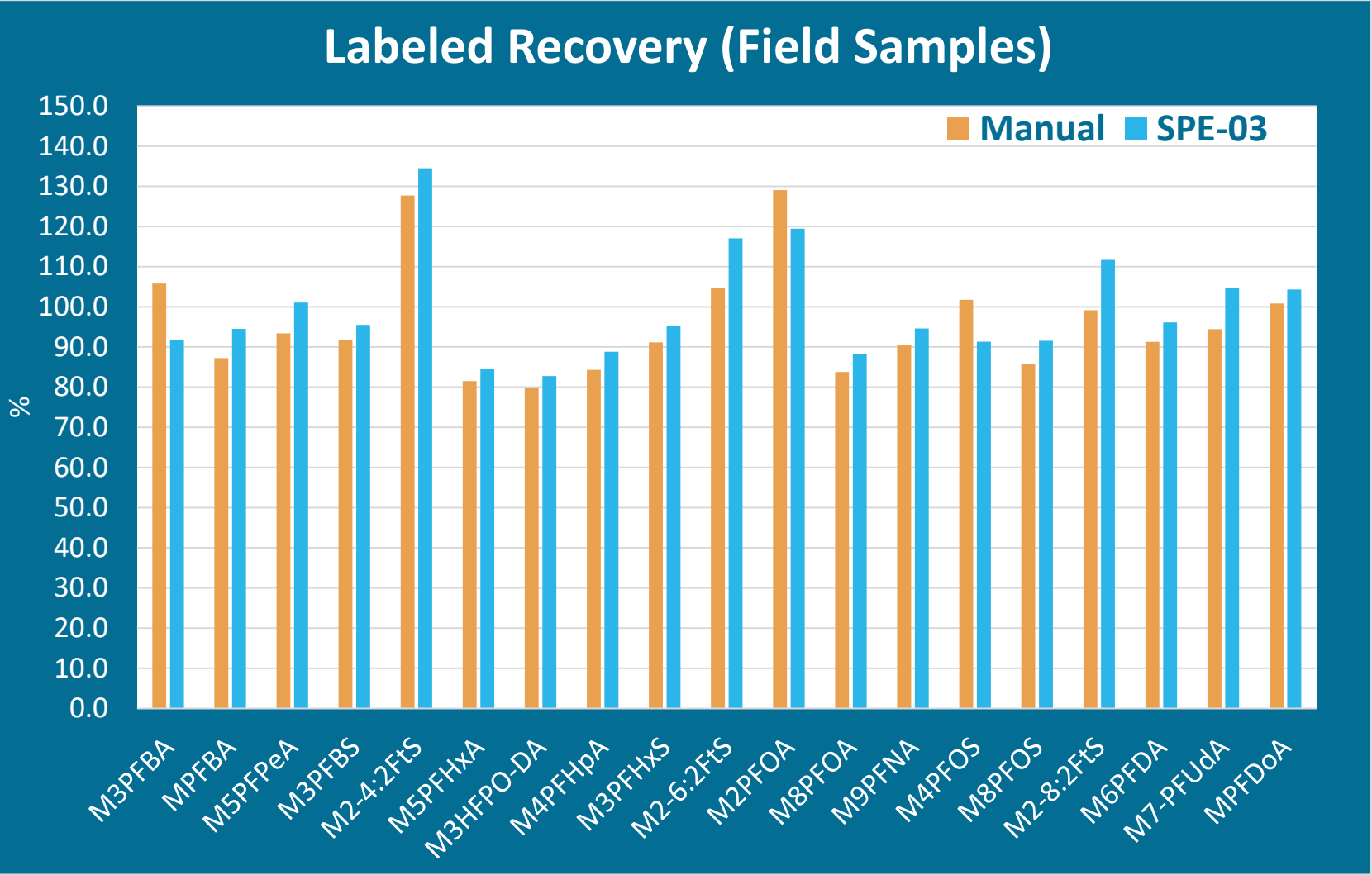
Method 533 - Results – Matrix Tolerance



LFB vs Field Sample Labeled Recovery on SPE-03

- N = 24 field samples
- Matrix enhancement on FTS isotopes

Method 533 - Results – Matrix Tolerance



Manual vs SPE-03
Labeled Recovery

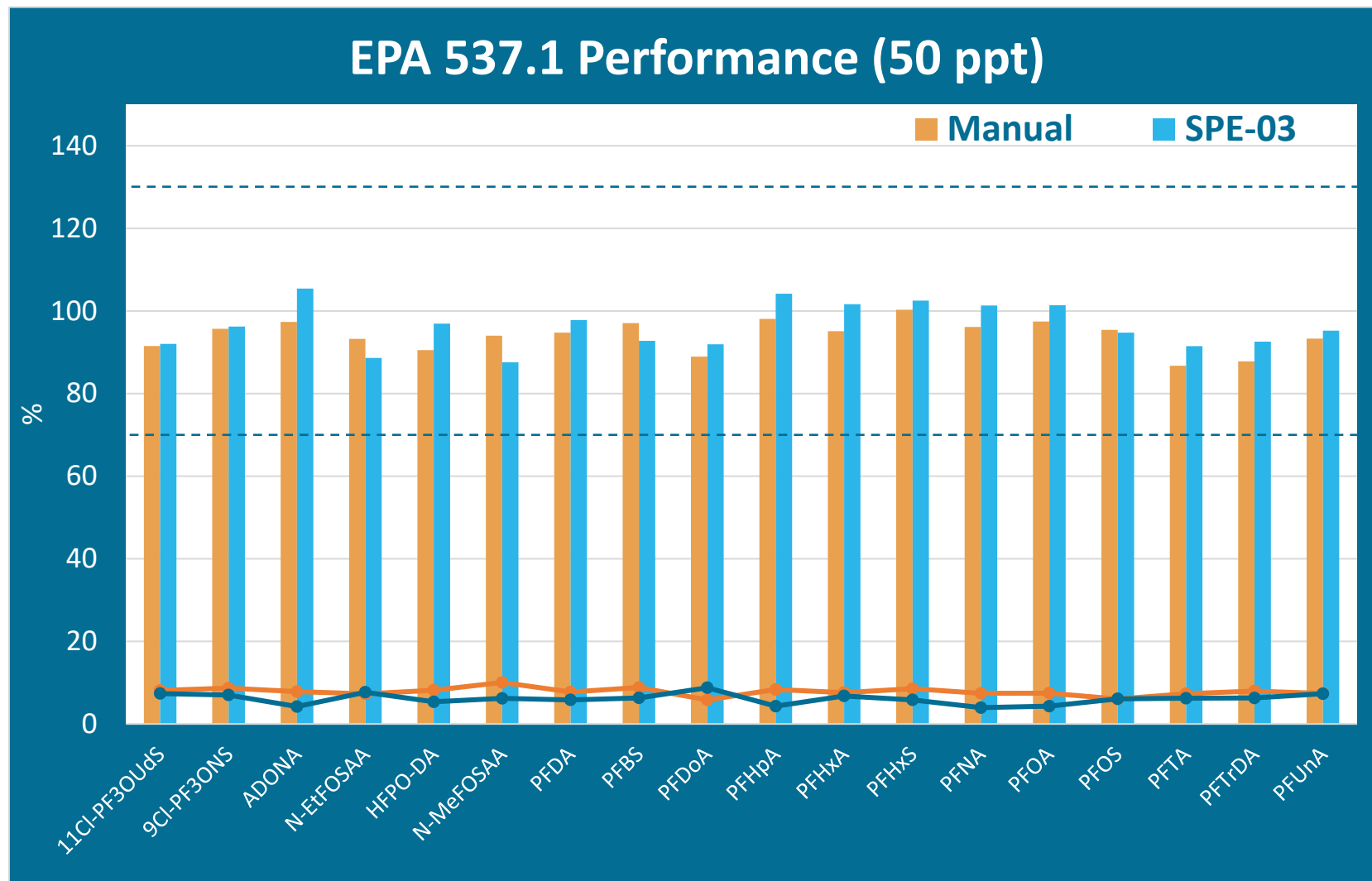
- Similar matrix effects

Method 537.1 - Data Collection

- Extraction data from **Orange County Water District (OCWD)** California
- **Manual extraction** results from August to December 2020
- **Automated extraction** results from January to March 2021



Method 537.1 - Results – 50 ppt

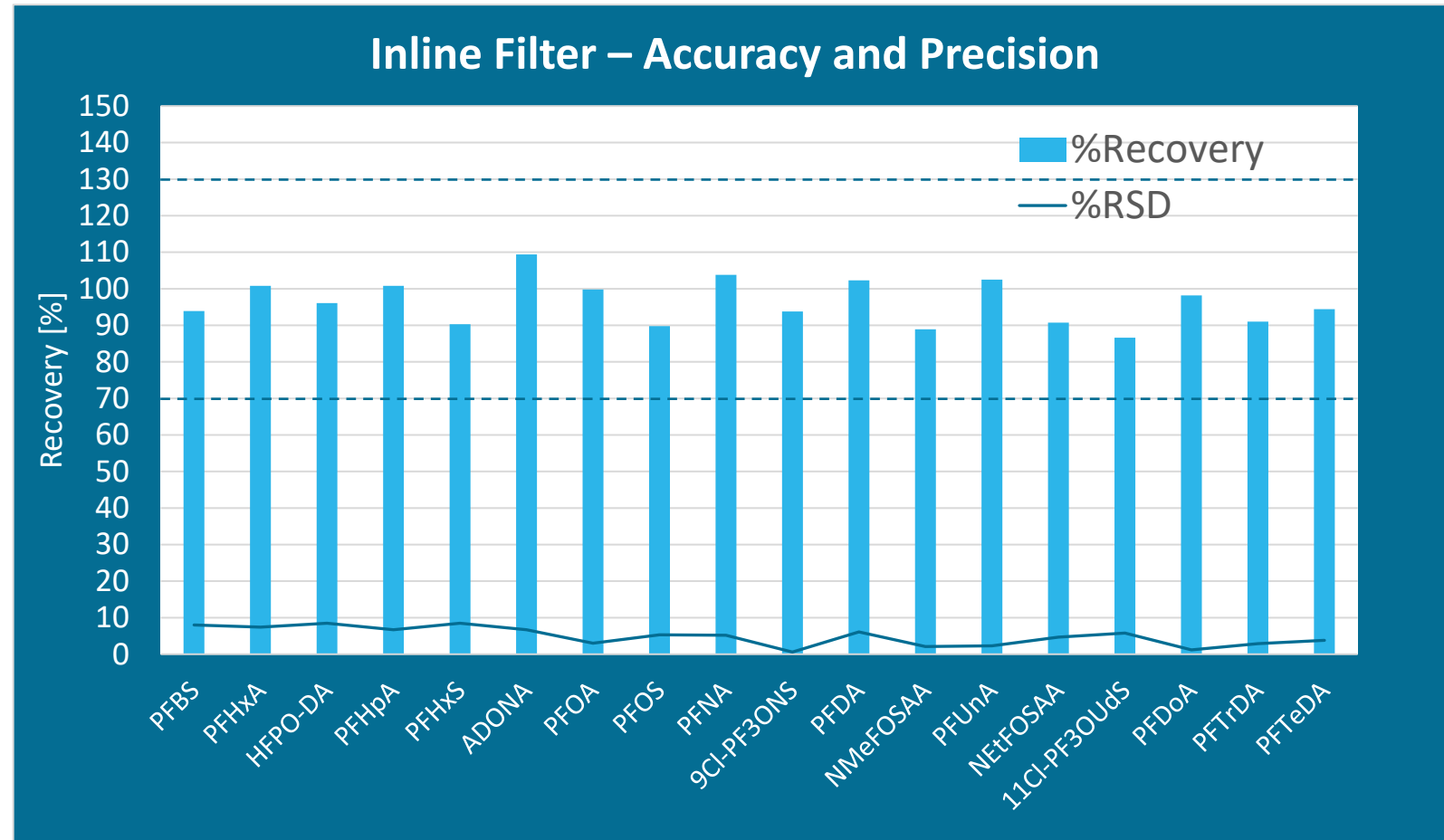
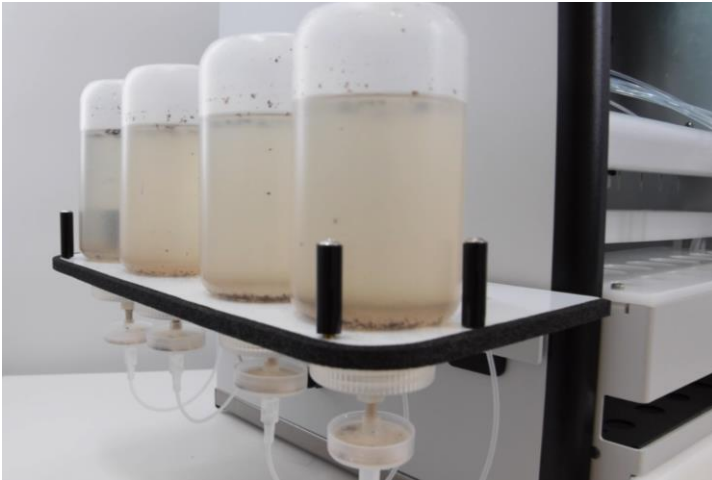


Manual vs SPE-03 LFB

- N = 19 at 50 ppt
- SPE-03: 88% to 105% recovery
- Manual: 87% to 100% recovery
- Similar recoveries and RSD

Method 537.1 – Using Inline Filter

- Using **inline filters** for **dirty samples**
- N = 4 x 20 ng/L spikes



Results and Discussion – Non-potable Water



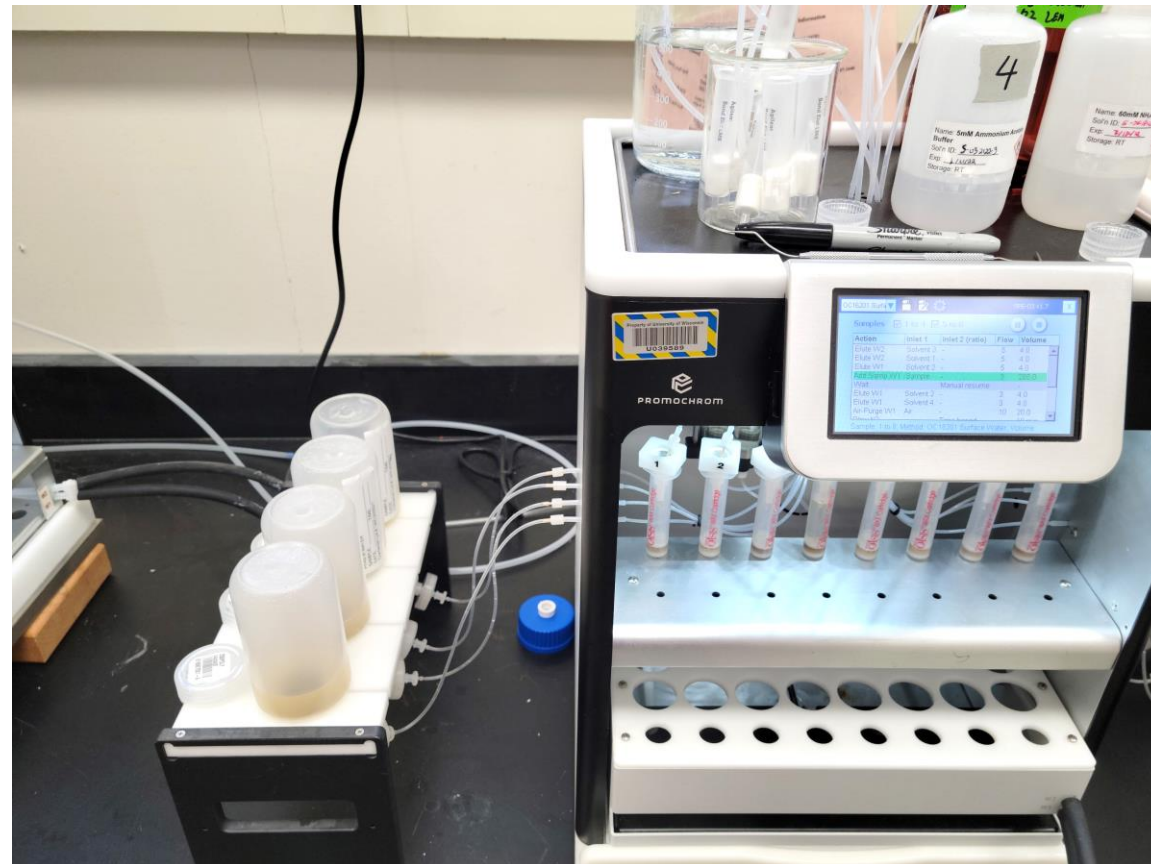
Non-potable Water

1. ISO 21675
2. Draft EPA Method 1633

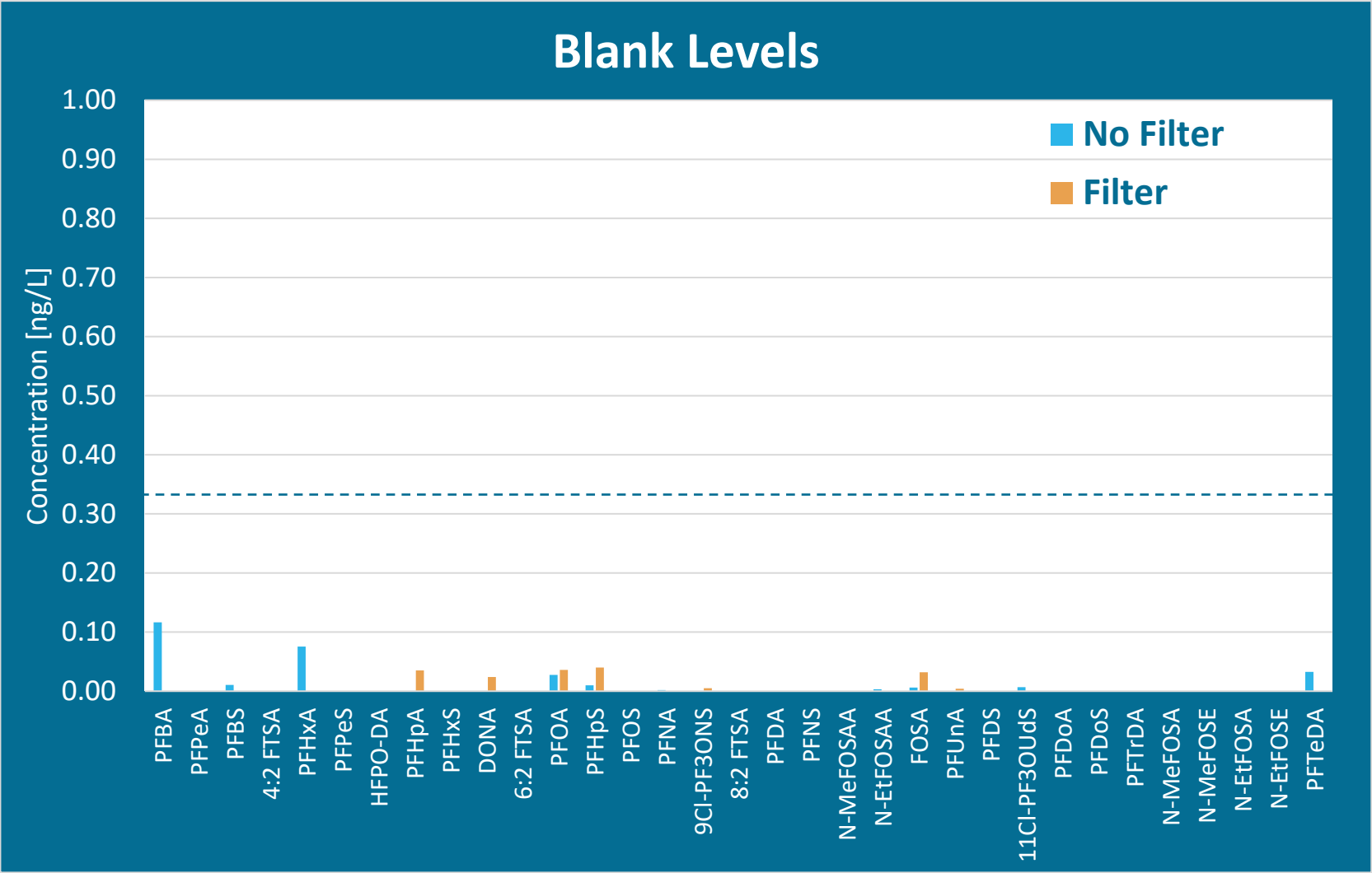


ISO 21675 – Data Collection

- Blank and LFB and field data for 33 compounds from the Wisconsin Lab of Hygiene, 2022 to 2021
- **SPE-03** and **manual extraction** field results



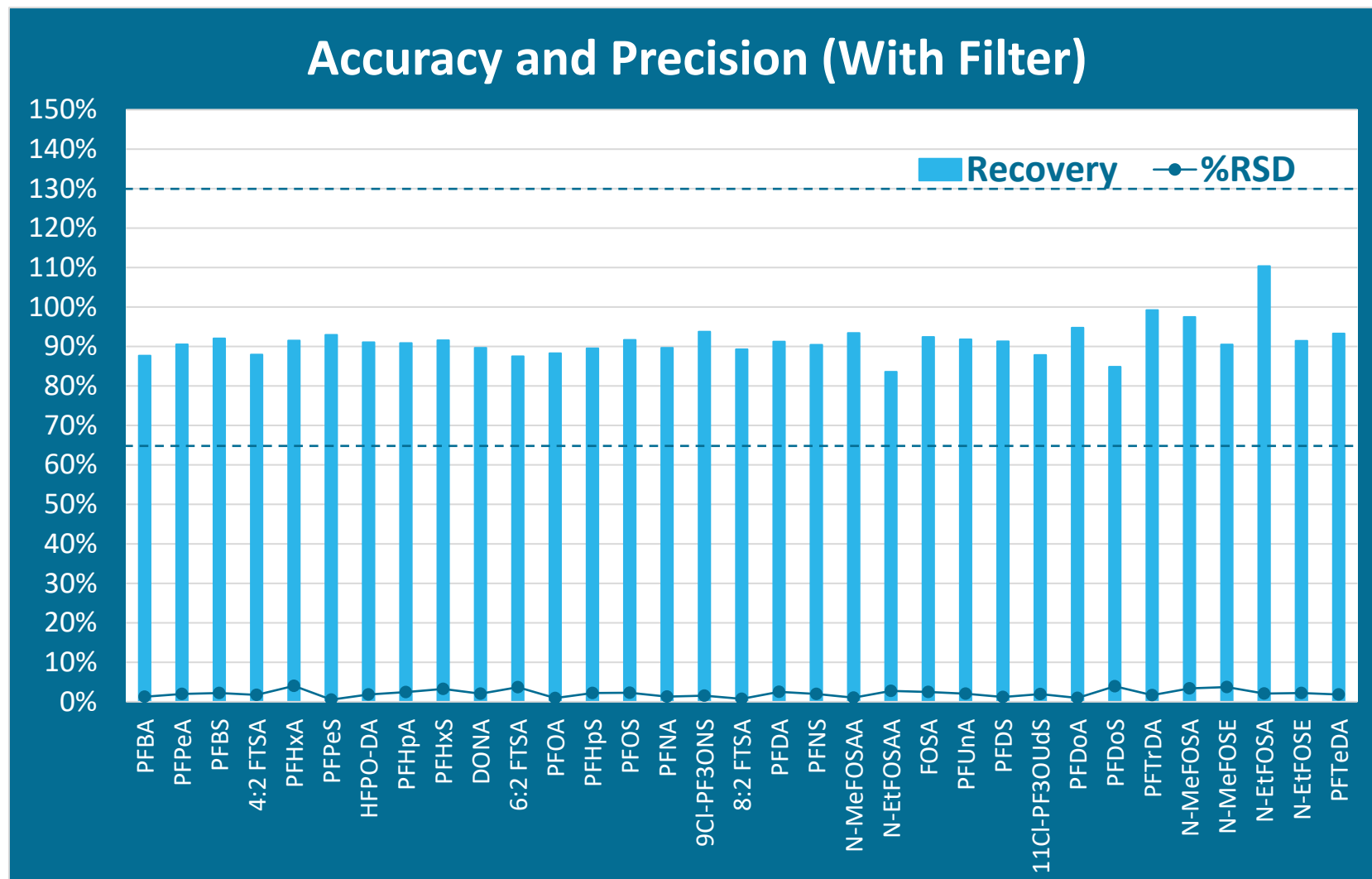
ISO 21675 – Results – Background



SPE-03 and Filter LRB

- No filter: N = 8
- Filter: N = 1
- Method limit: 0.33 ng/L

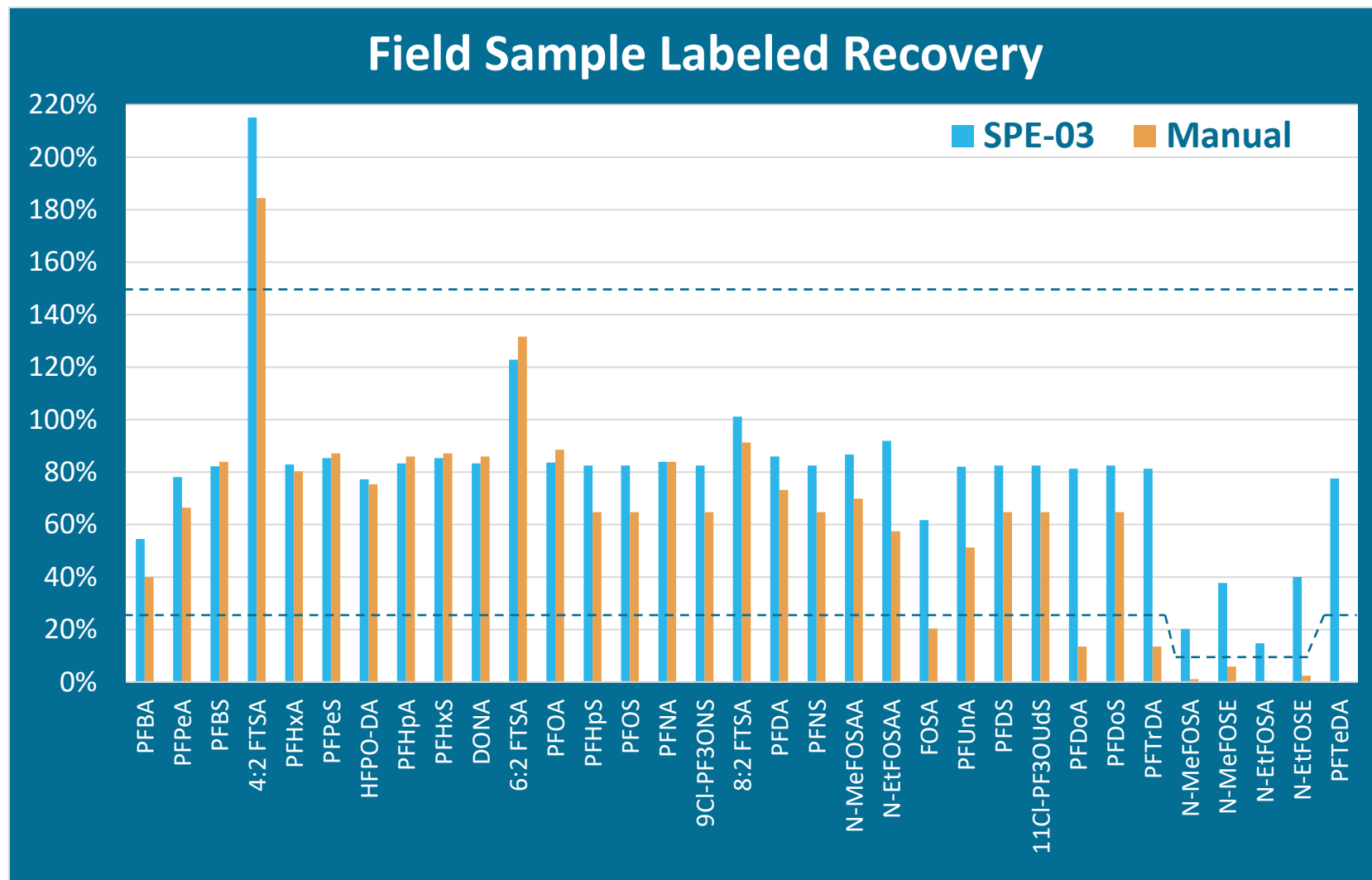
ISO 21675 – Results – Accuracy and Precision



SPE-03 + Filter LFB

- N = 4 at 8 ng/L
- Method limit: 65% to 130%
- SPE-03
 - 84% to 110% recovery
 - $\leq 5\%$ RSD

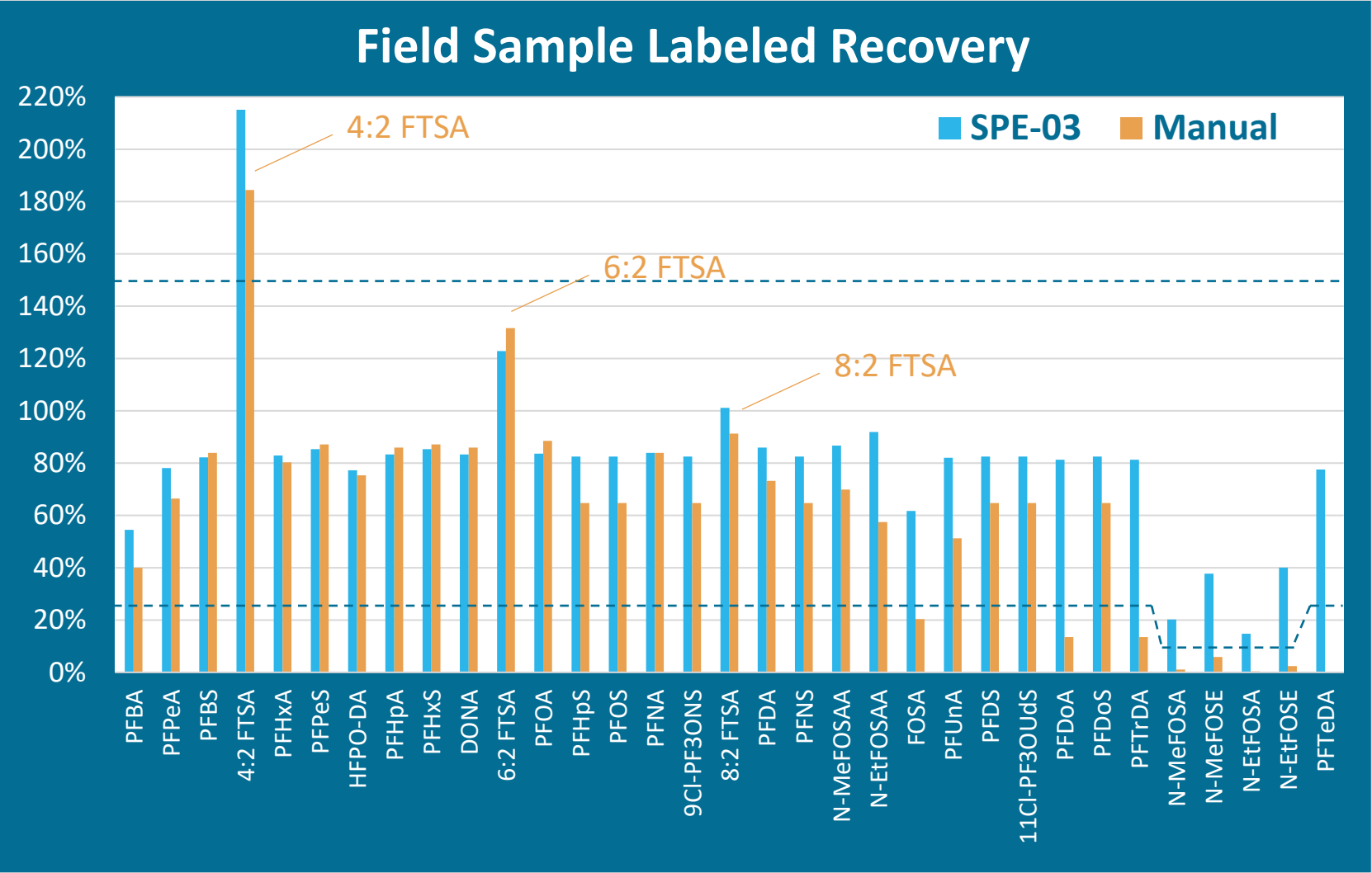
ISO 21675 – Results – Matrix Effects



SPE-03 vs Manual Labeled Recovery

- Method limits:
 - 25% to 150%
 - 10% for Me/Et FOSA and FOSE

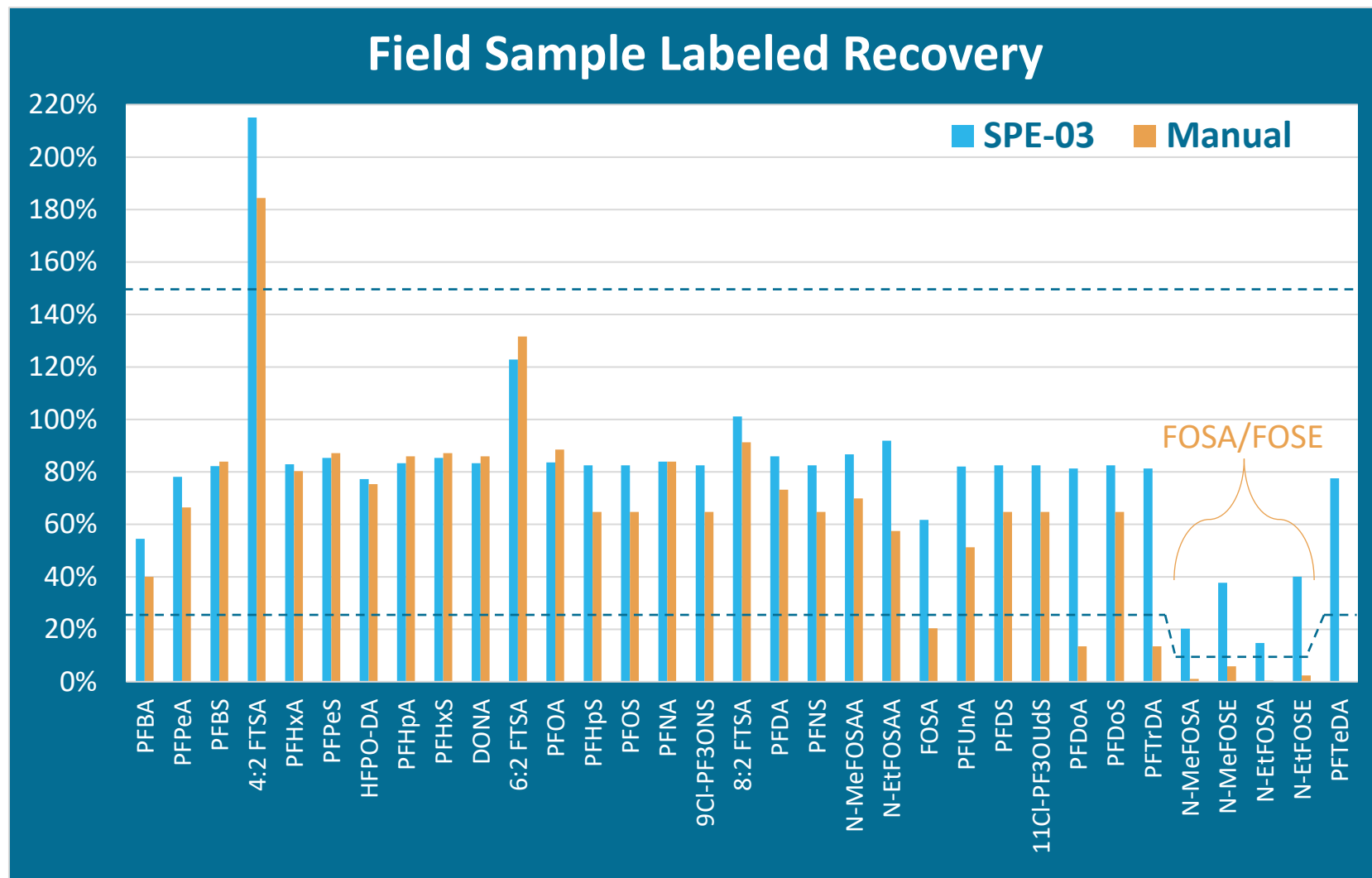
ISO 21675 – Results – Matrix Effects



SPE-03 vs Manual Labeled Recovery

- Matrix enhancement on the FTSA isotopes

ISO 21675 – Results – Matrix Effects



SPE-03 vs Manual Labeled Recovery

- Lower recovery on manual extraction for more adsorbent compounds

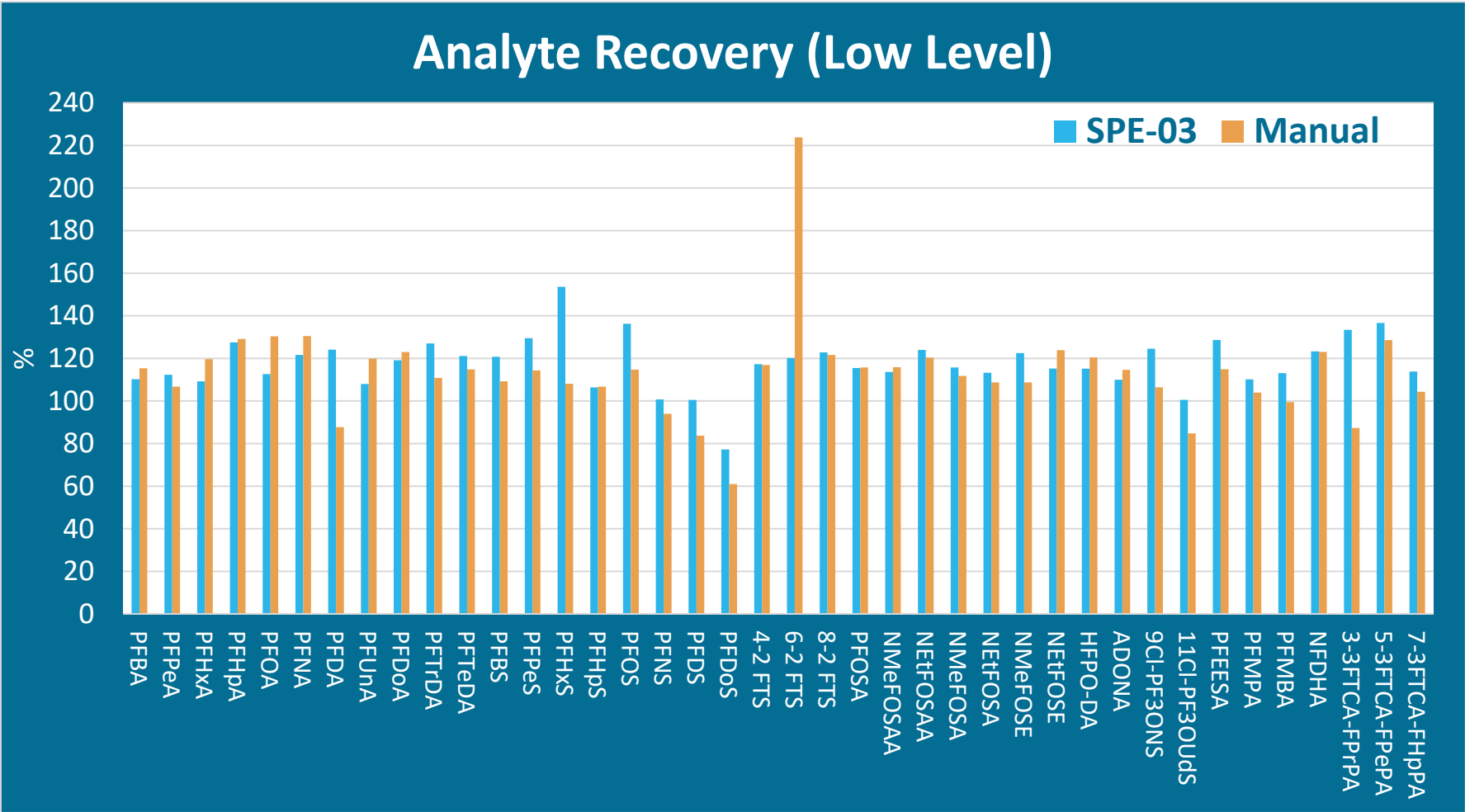
Method 1633 – Data Collection

- IDC/MDL data from **Maryland Department of Health (MDH)**, Feb 2022
- **SPE-03** (Volume-Matrix Plus configuration) and **manual extraction** LFB results



Data cited from Frost A., Hu X., Cao Z., and S. Urban. **Automating sample extraction for high-throughput environmental chemistry testing**. APHL Annual Conference Proceedings 2022. The work at **MDH** performed only covers research for its own benefits and does not constitute endorsement of any particular commercial product.

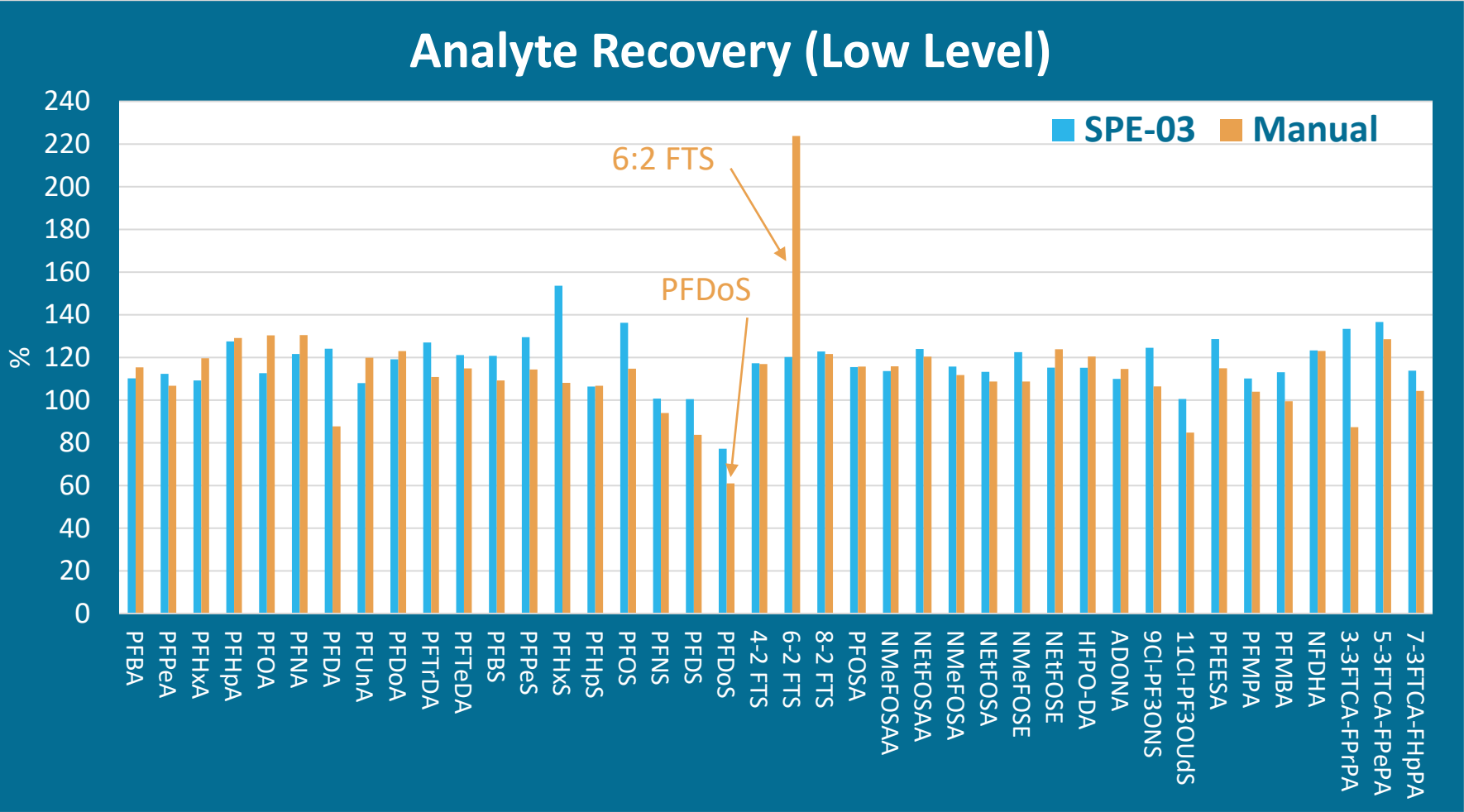
Method 1633 – Analyte Performance (Low level)



SPE03 vs Manual

- N = 4, 0.2 to 5 ppt
- SPE03: 77% to 154%
- Manual: 61% to 224%

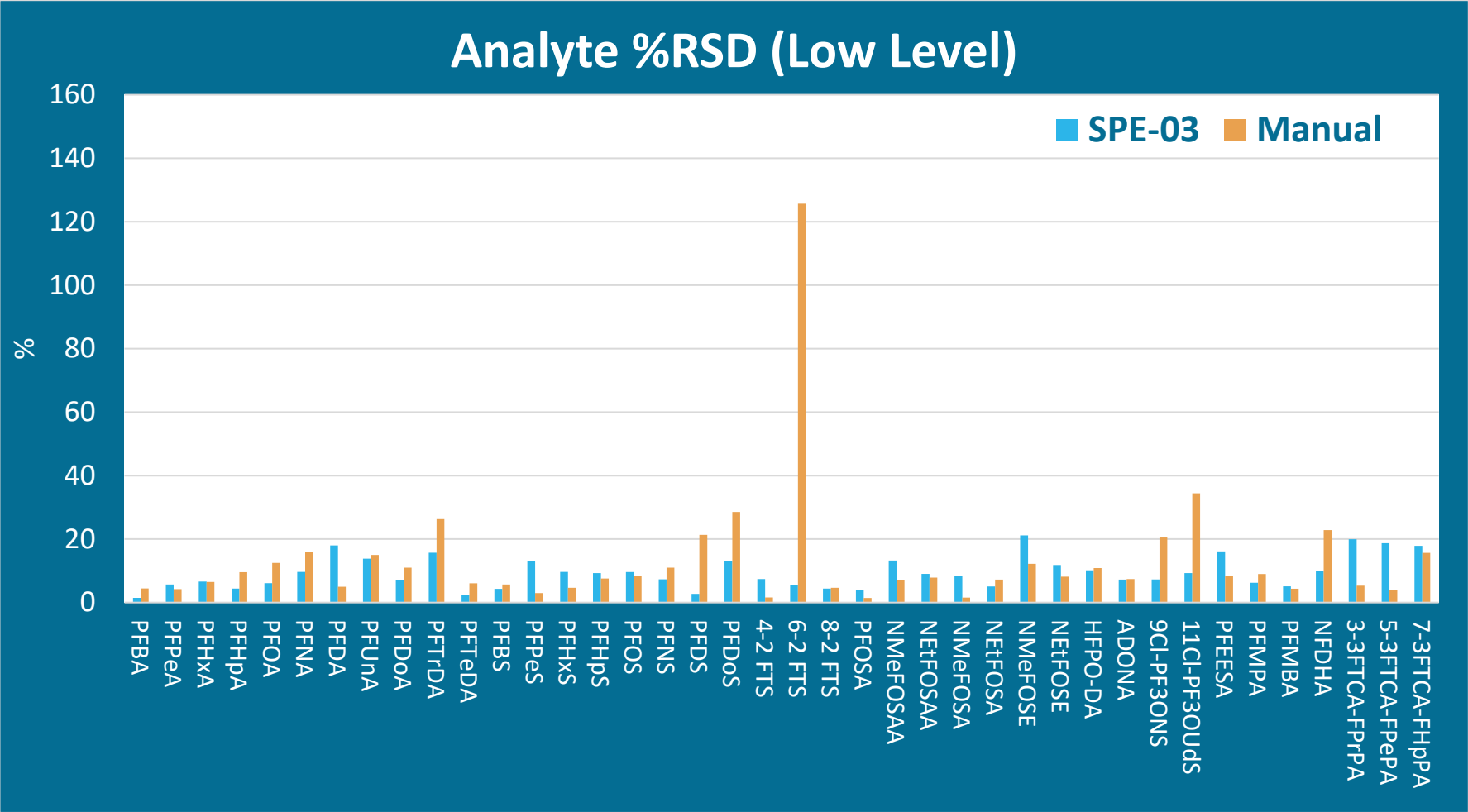
Method 1633 – Analyte Performance (Low level)



SPE03 vs Manual

- Higher variation on manual:
 - PFDoS
 - 6:2 FTS

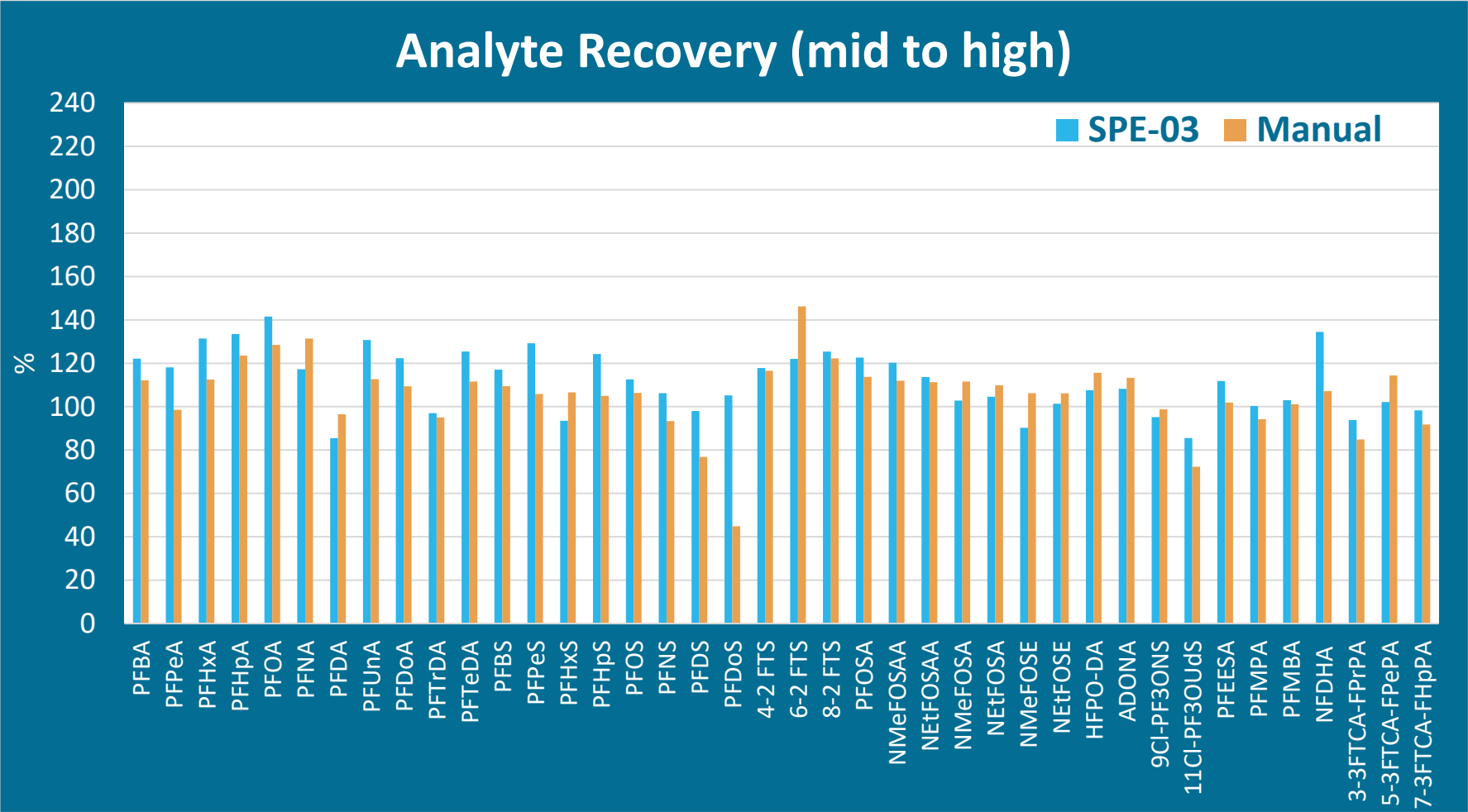
Method 1633 – Analyte Performance (Low level)



SPE03 vs Manual

- N = 4, 0.2 to 5 ppt
- SPE03: <21.2%
- Manual:
 - 125% for 6:2 FTS
 - <34.4% others

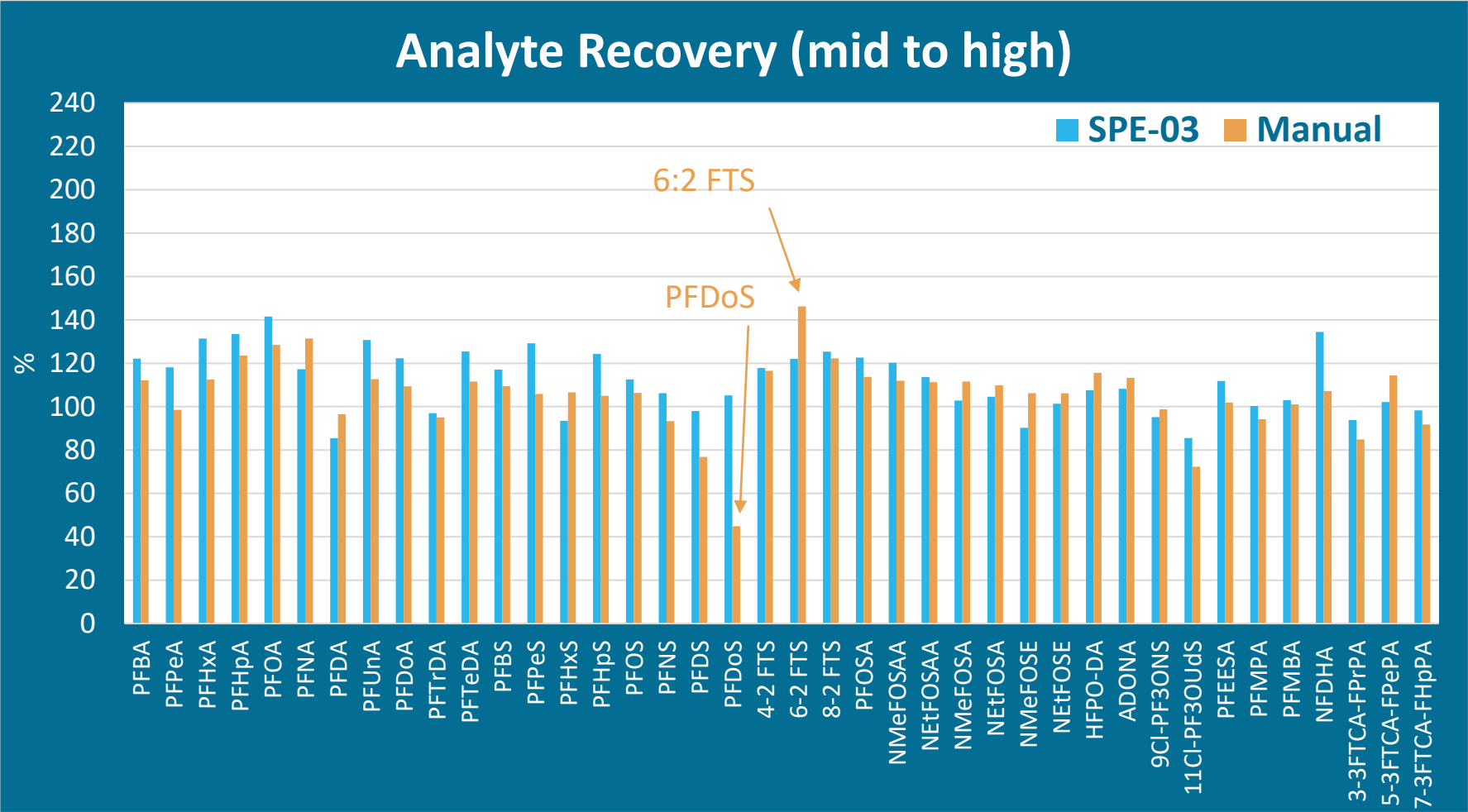
Method 1633 – Analyte Performance (Other Levels)



SPE03 vs Manual

- N = 4, mid to high
- SPE03: 86% to 142%
- Manual: 45% to 146%

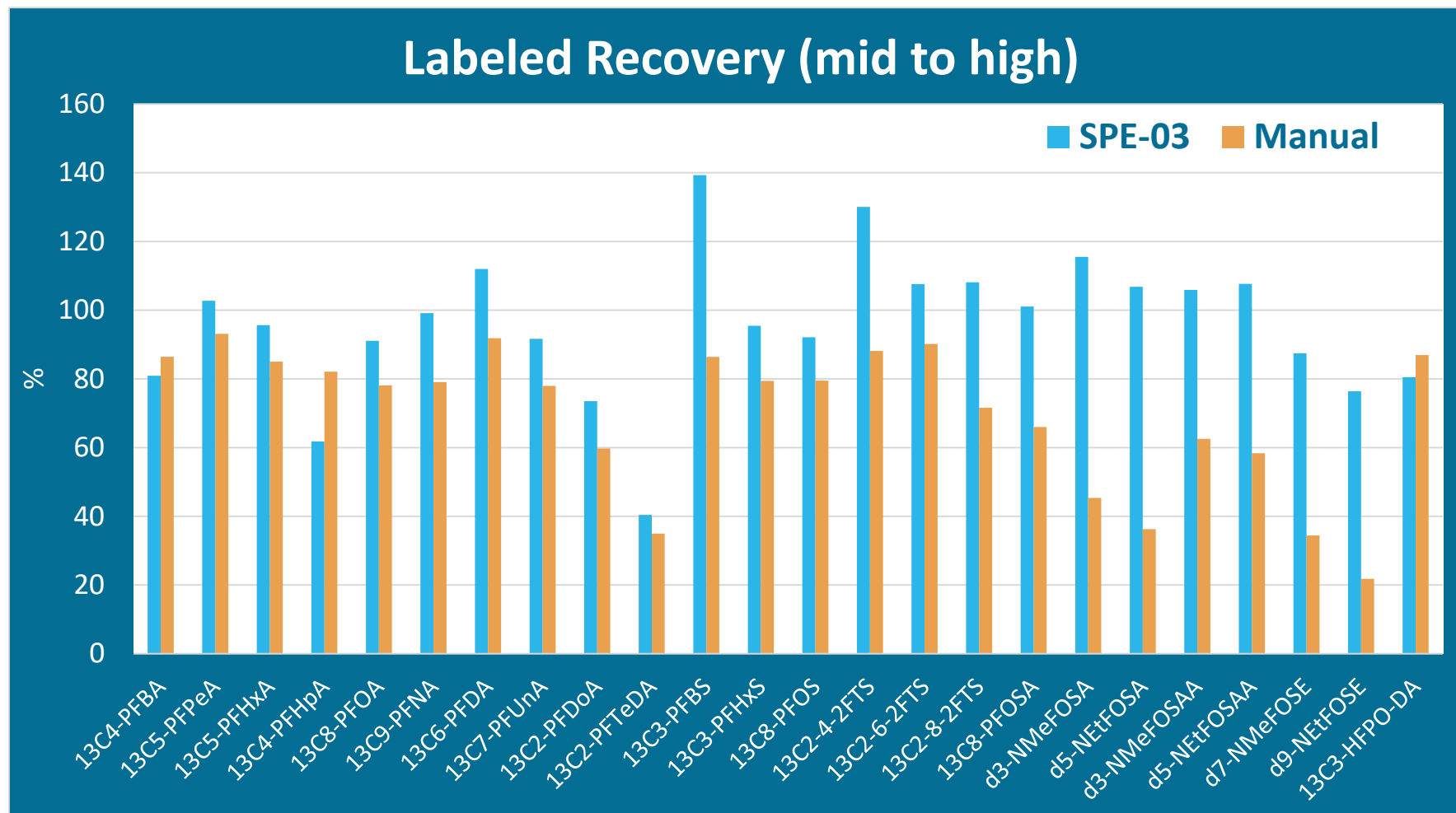
Method 1633 – Analyte Performance (Other Levels)



SPE03 vs Manual

- Higher variation on manual:
 - PFDoS
 - 6:2 FTS (better than low level)

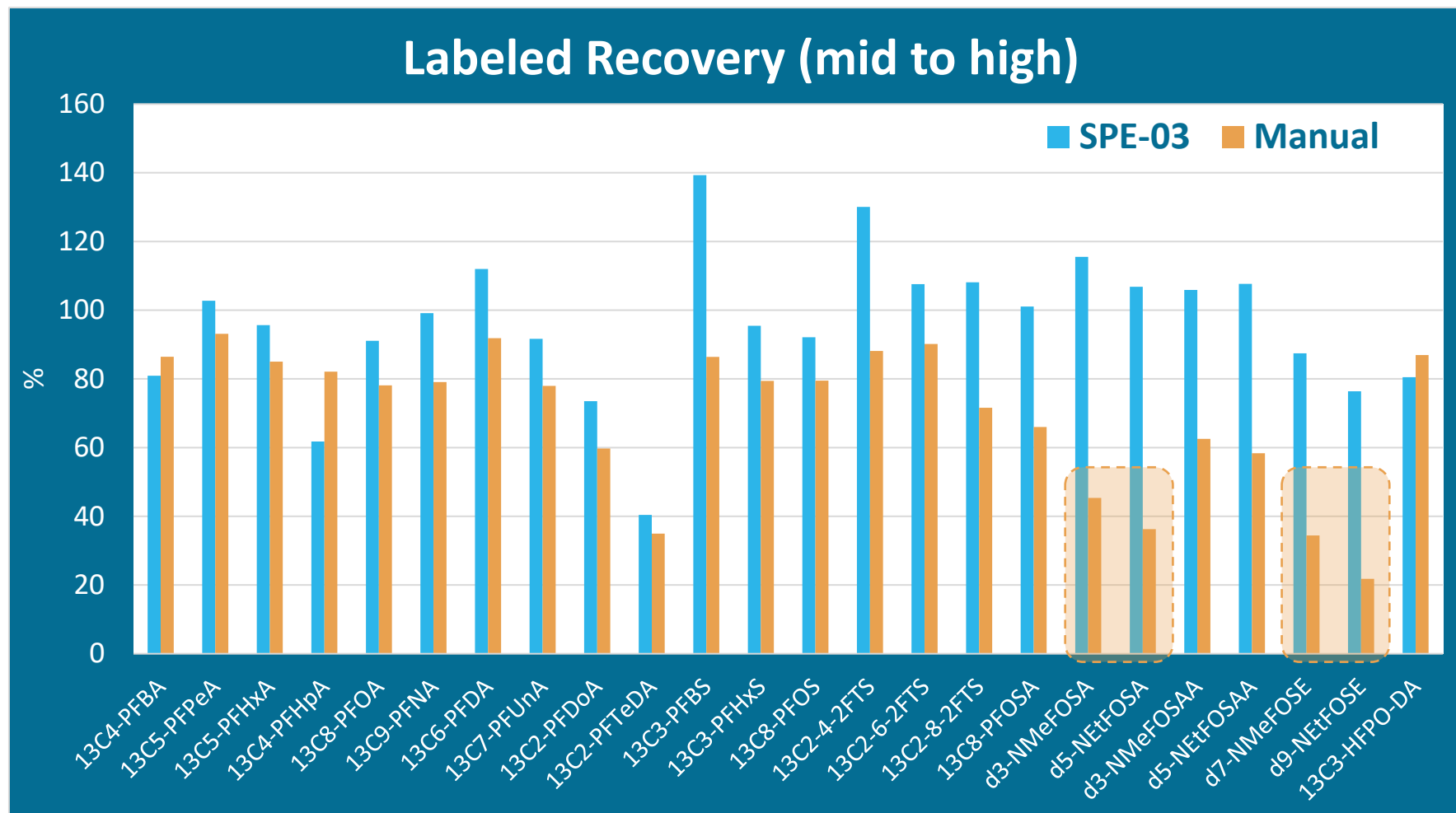
Method 1633 – Labeled Compound Performance (Other levels)



SPE03 vs Manual

- N = 4, mid to high
- SPE03: 40% to 140%
- Manual: 22% to 87%

Method 1633 – Labeled Compound Performance (Other levels)



SPE03 vs Manual

- Lower isotope recoveries on manual extraction, especially FOSA and FOSE

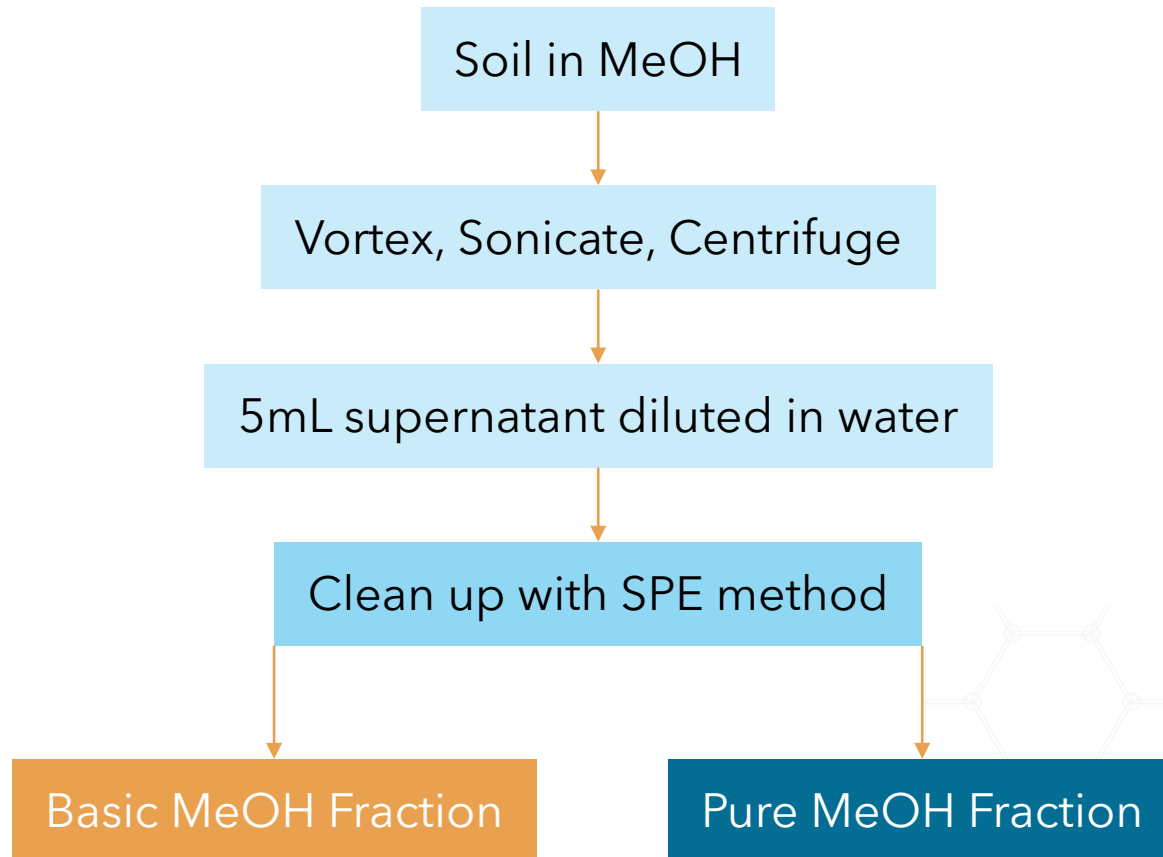
Soil – Data Collection

- Data from **Alpha Analytical, Massachusetts**, Aug 2020
- 36 acidic + 7 neutral PFAS compounds

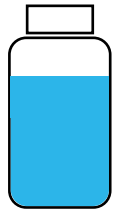


Soil

Soil Extract Clean Up Procedure

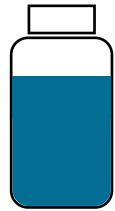


Soil Extract Clean Up



MB

Used for
background check



LCS

Used for recovery validation



LCSD



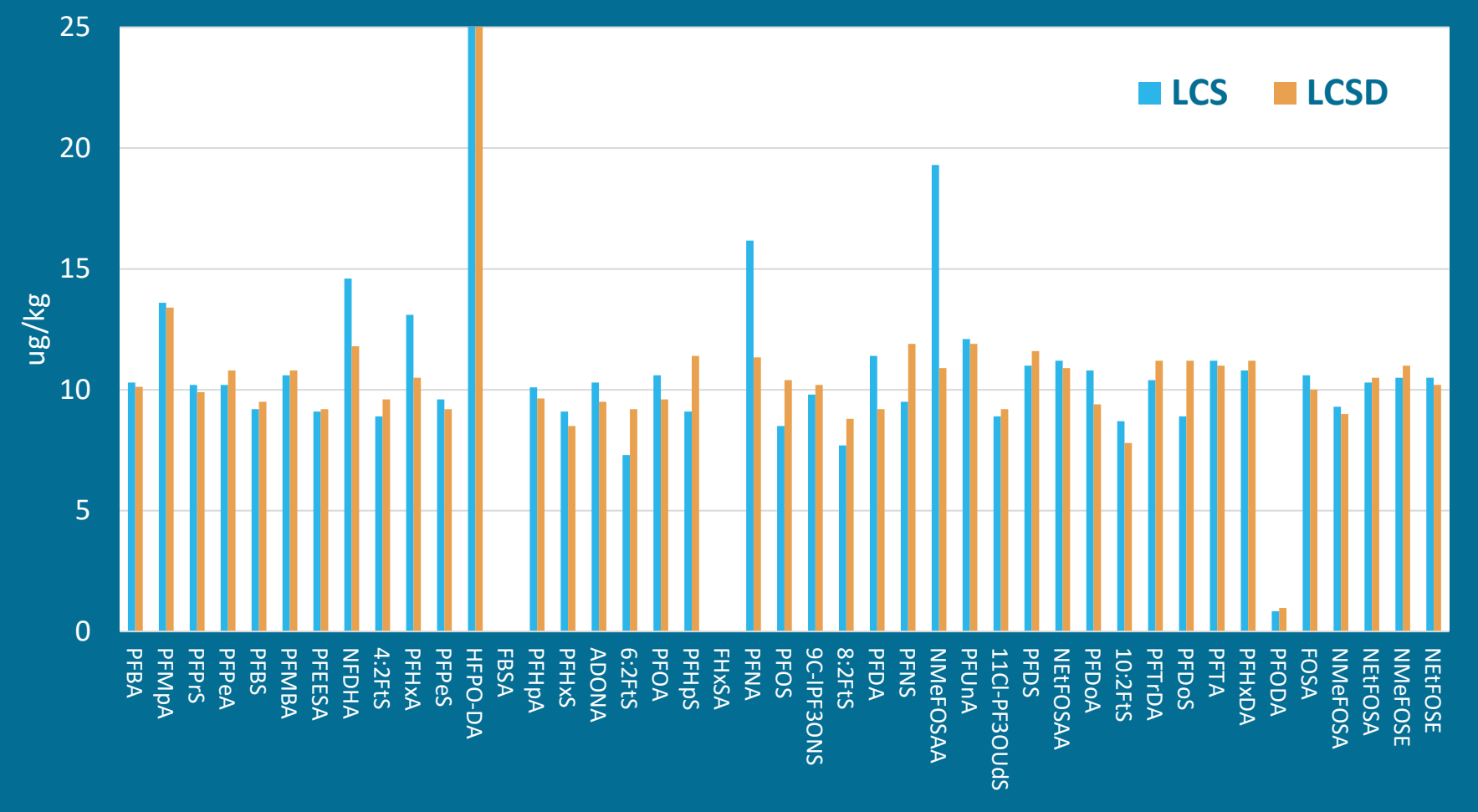
QC

QC sample from
ERA to validate
accuracy from 3rd
party



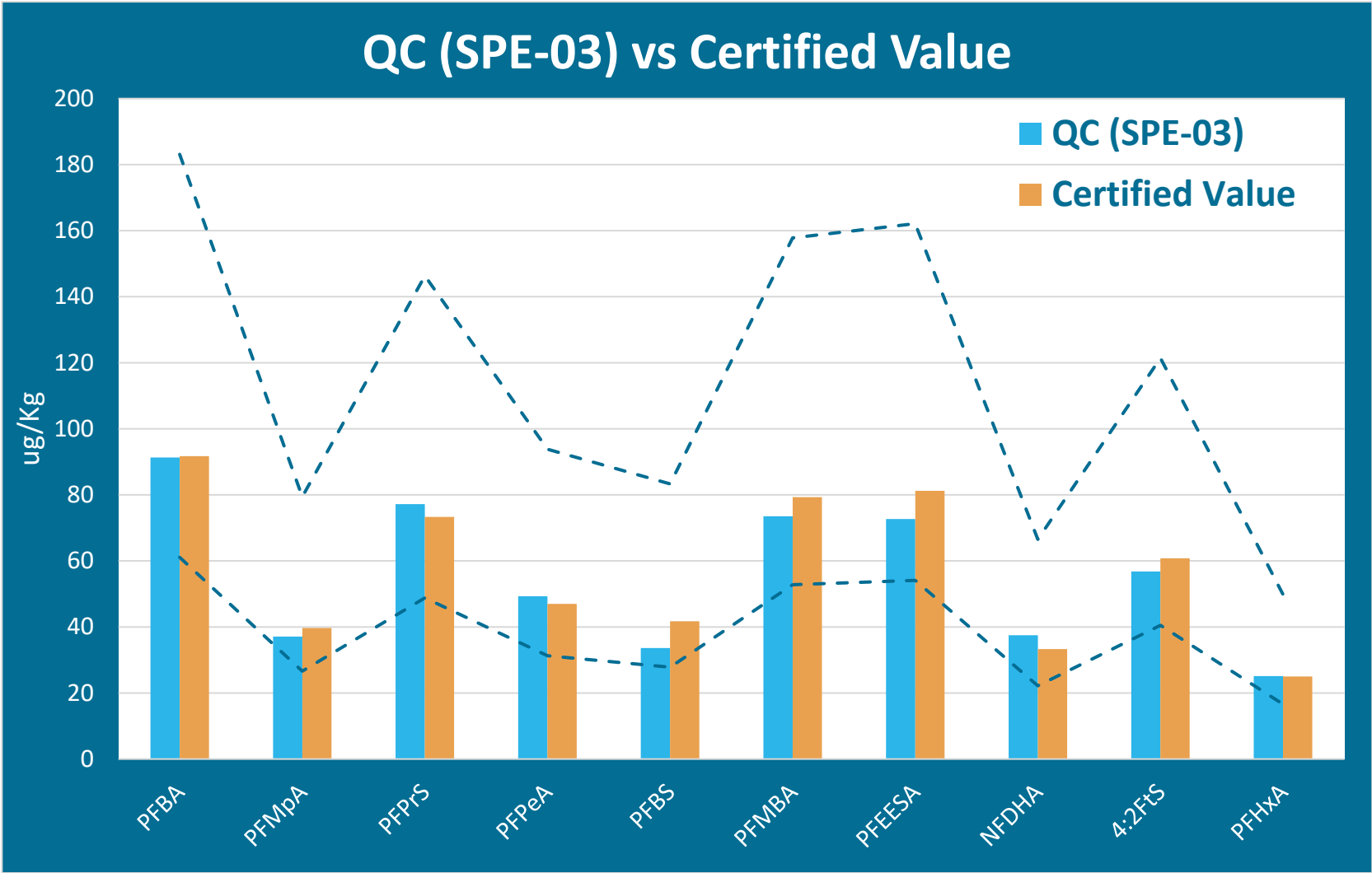
Soil Extract Clean Up – Native Compound Recovery

LCS Recoveries



- Spike = 10 ug/kg
- 4 compounds a bit high for LCS
- Similar levels for other compounds

Soil Extract Clean Up - Accuracy

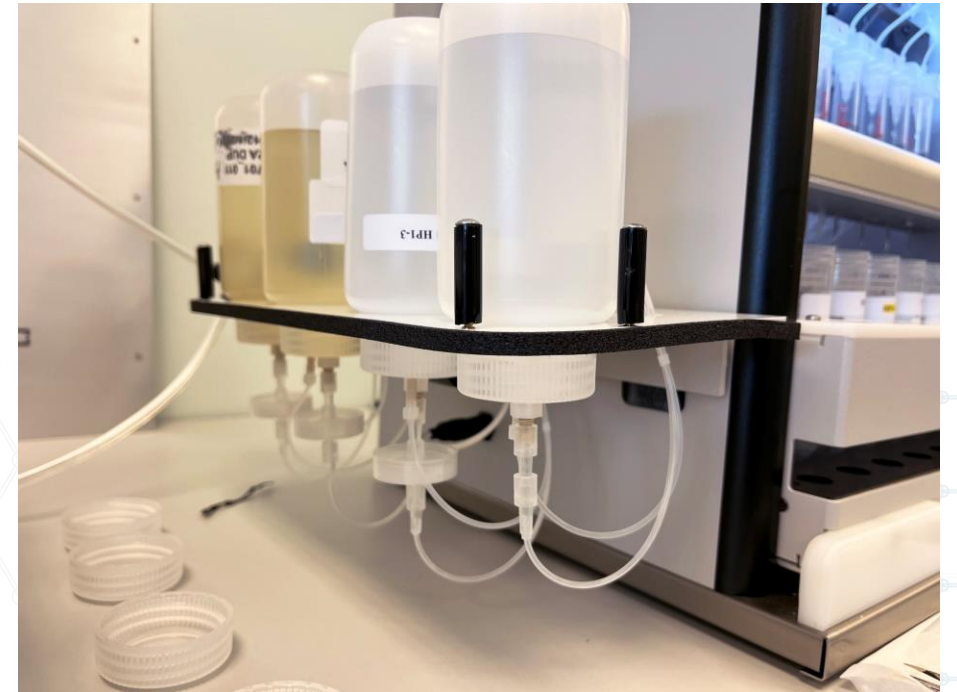


- QC Sample from ERA cleaned up on SPE-03
- Very close to certified values
- Well-within low and high range bounds

Conclusion

1. Successful extraction automation for

- Drinking water
- Non-drinking water
- Soil



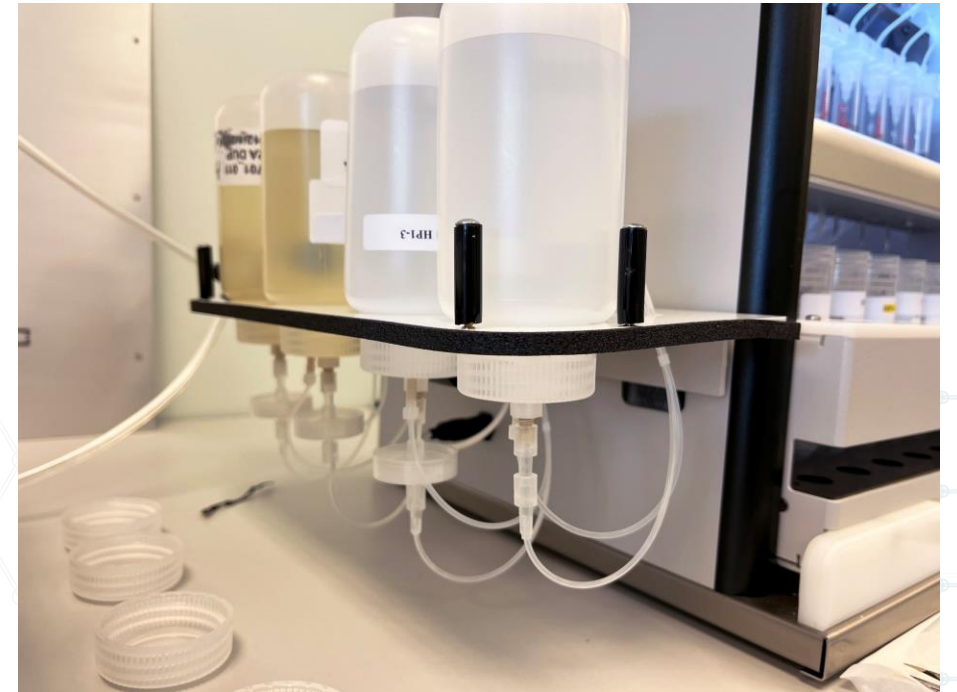
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 - Drinking water
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2. Considerations for non-potables extraction
 - Flow control
 - Anti-clogging
 - Automatic bottle rinse



Conclusion

1. Successful extraction automation for
 - Drinking water
 - Non-drinking water
 - Soil
2. Considerations for non-potables extraction
 - Flow control
 - Anti-clogging
 - Automatic bottle rinse
3. Benefits of automation
 - Quick deployment
 - Better use of human resources



Acknowledgements



Questions?

- ian_wan@promochrom.com
- www.promochrom.com/pfas-extractions



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