IMPROVE Steering Committee Meeting

Virtual November 2025

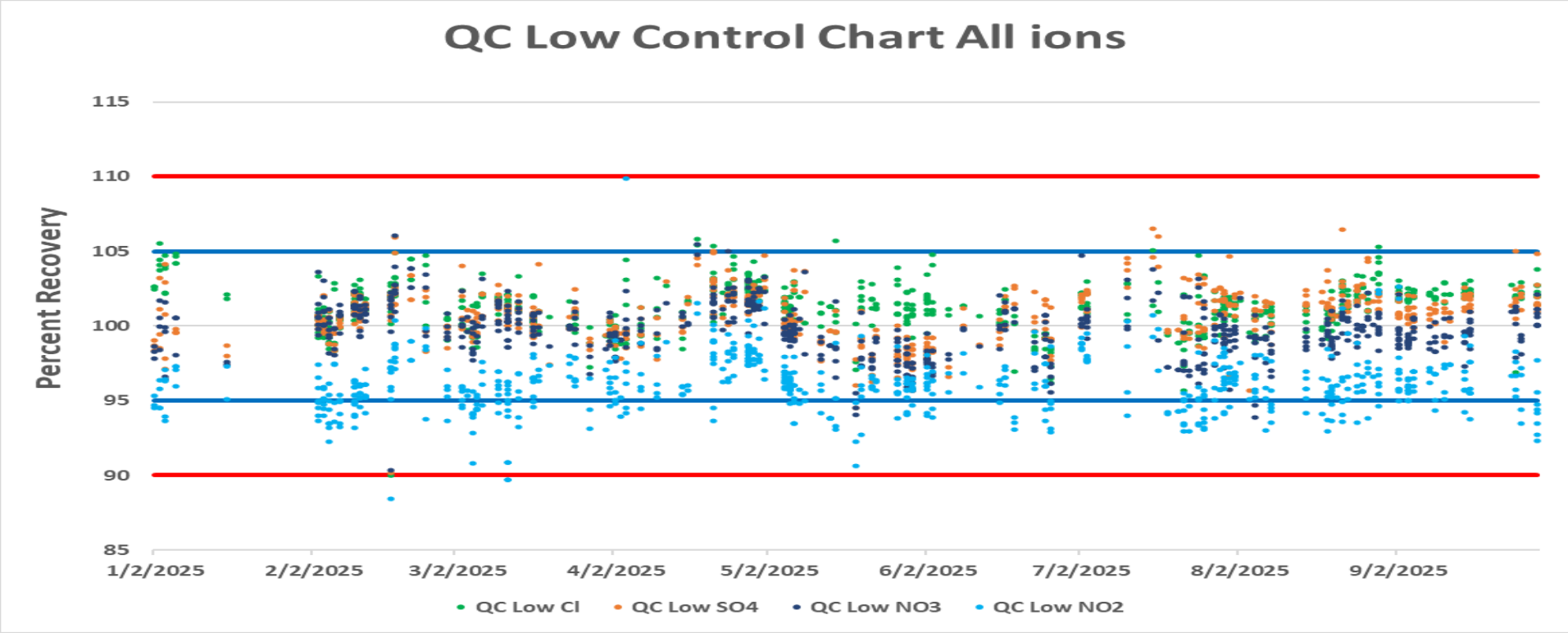
Tracy Dombek, Ions Report

Minimum Detection Limits & Process

- Extract nylon filters in 20 mL of DI water.
- Calibrate systems daily using primary stock standards.
- Quality Controls using secondary source standards before and after every ten samples analyzed.
- Duplicates at a rate of 3 per batch of 50 samples.
- Perform matrix spikes at a rate of 2 per batch of 50 samples.
- Random reanalysis of 5% of the sample total.
- Re-extraction of filters to evaluate extraction efficiencies.

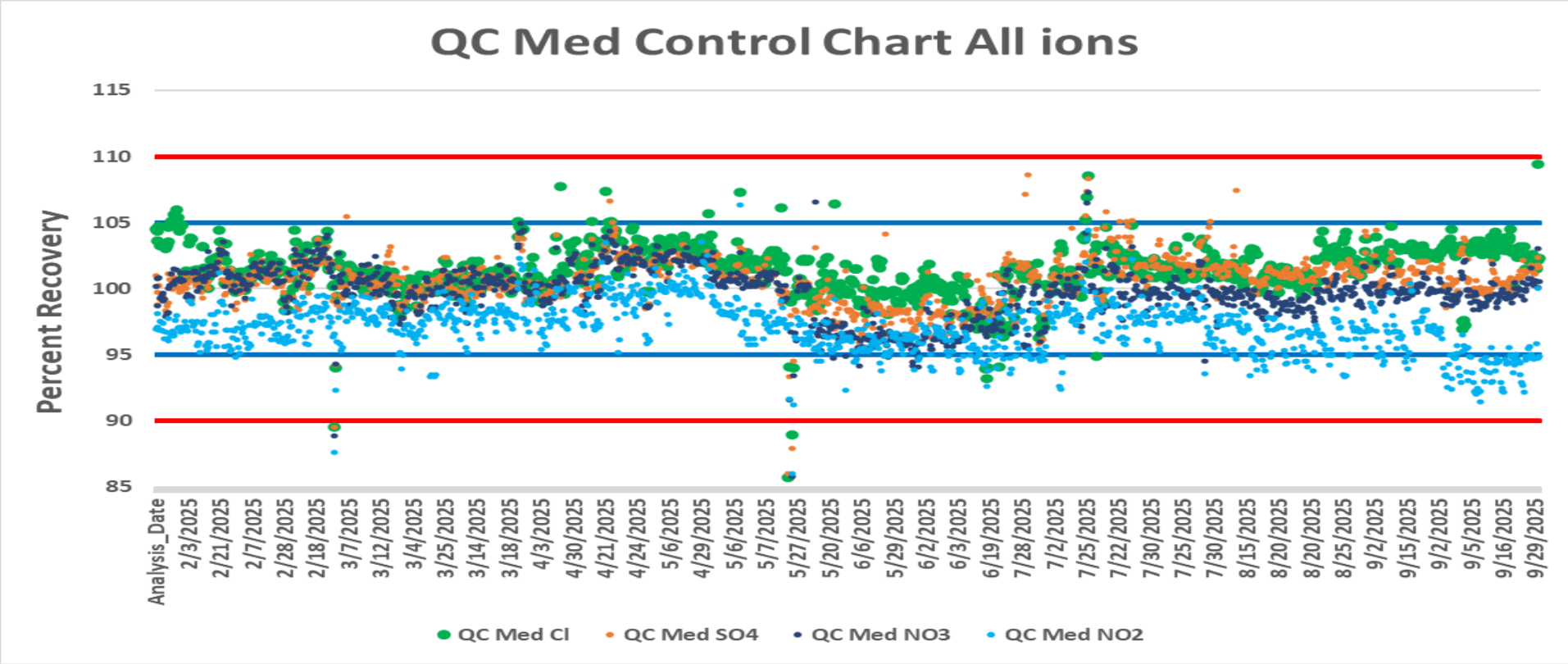
	Chloride	Nitrite	Nitrate	Sulfate
2023	0.006 ppm	0.010 ppm	0.008 ppm	0.011 ppm
2024	0.006 ppm	0.010 ppm	0.014 ppm	0.012 ppm
2025	0.006ppm	0.010 ppm	0.011 ppm	0.012 ppm

Control Charts



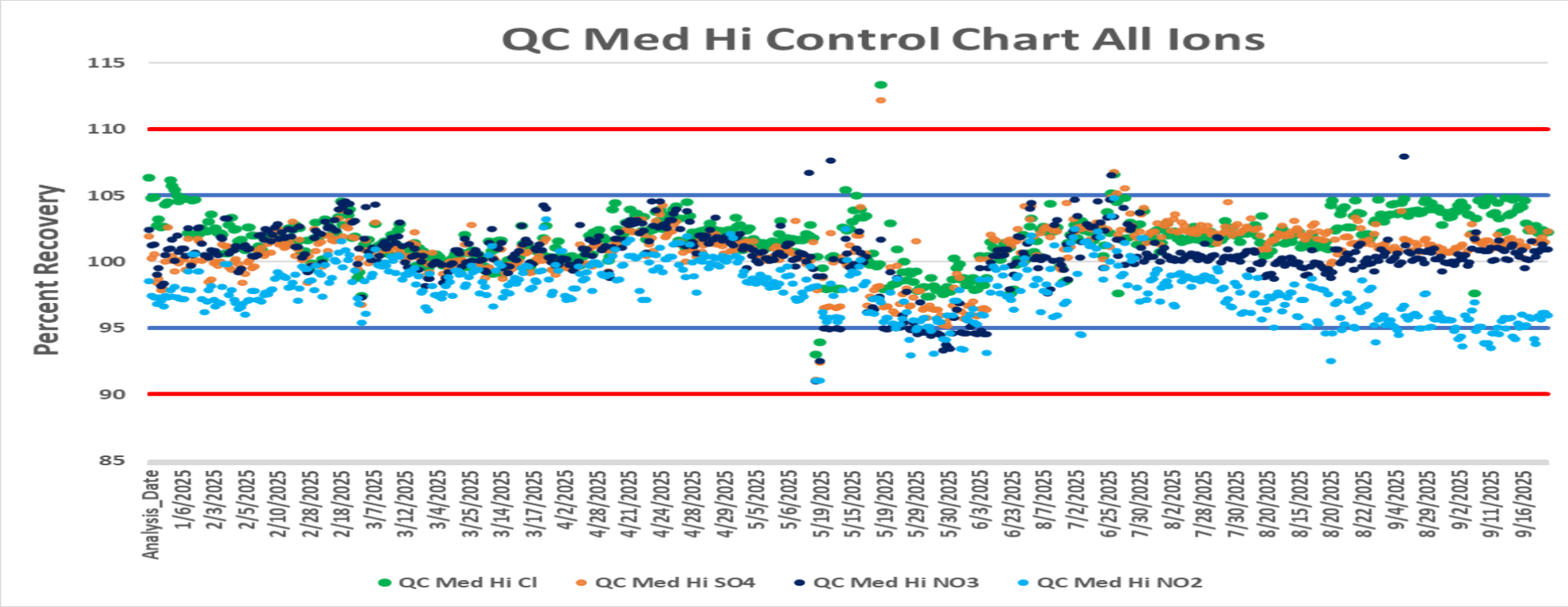
Ion	Median Percent Recovery	Average Percent Recovery	Count	% Failures
Chloride	101%	101%	675	0.44%
Sulfate	101%	101%	675	0.30%
Nitrate	99.7%	99.7%	675	0.30%
Nitrite	96.0%	96.1%	675	0.74%

Control Charts



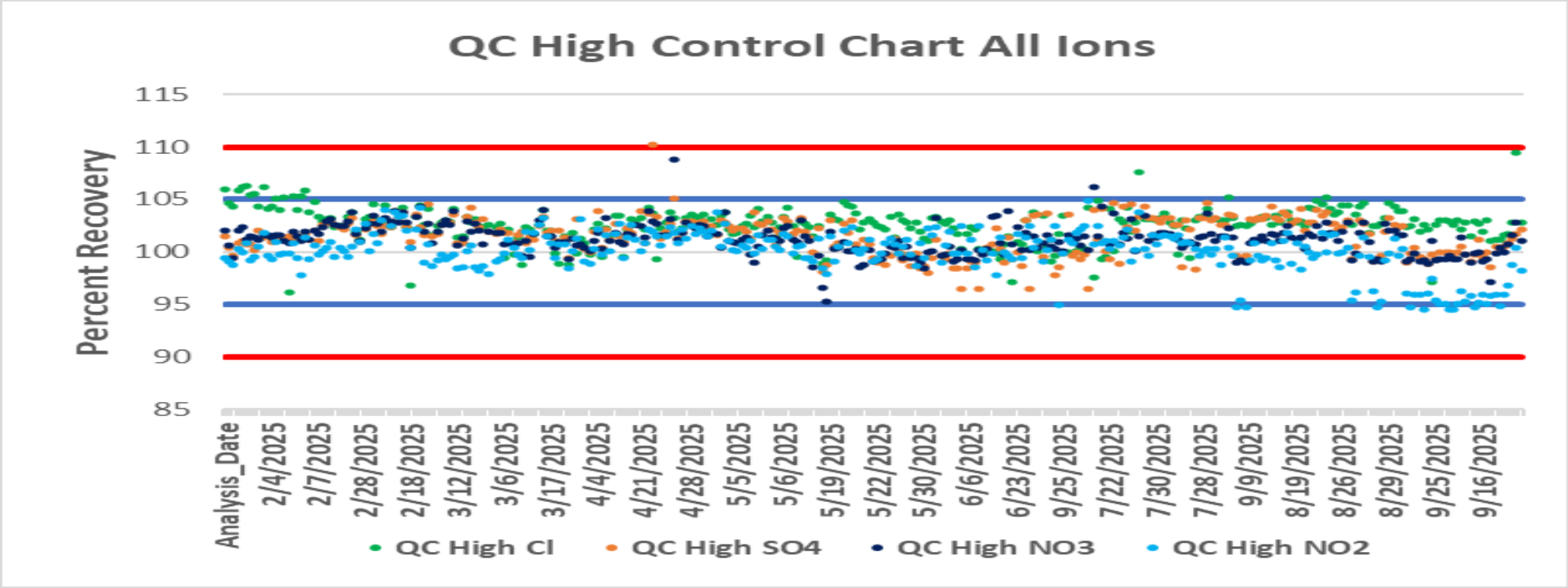
Ion	Median Percent Recovery	Average Percent Recovery	Count	% Failures
Chloride	101%	101%	1252	0.40%
Sulfate	101%	101%	1252	0.40%
Nitrate	99.8%	99.6%	1252	0.40%
Nitrite	97.2%	97.0%	1252	0.40%

Control Charts



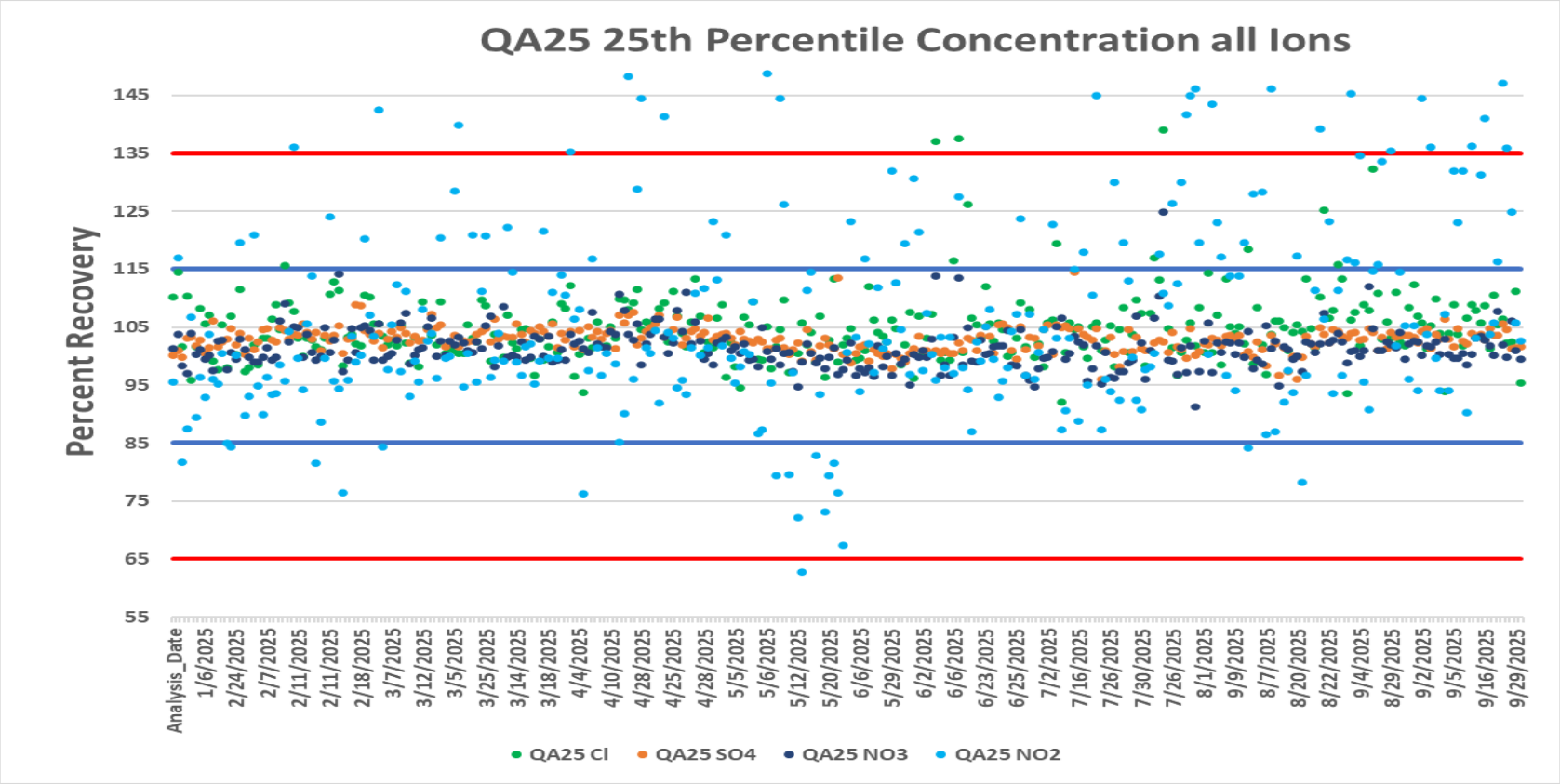
Ion	Median Percent Recovery	Average Percent Recovery	Count	% Failures
Chloride	102%	102%	614	0.16%
Sulfate	101%	101%	614	0.16%
Nitrate	101%	100%	614	0%
Nitrite	98.0%	97.9%	614	0%

Control Charts



Ion	Median Percent Recovery	Average Percent Recovery	Count	% Failures
Chloride	103%	103%	307	0.33%
Sulfate	102%	102%	307	0.65%
Nitrate	101%	101%	307	0.33%
Nitrite	100%	100%	307	0.33%

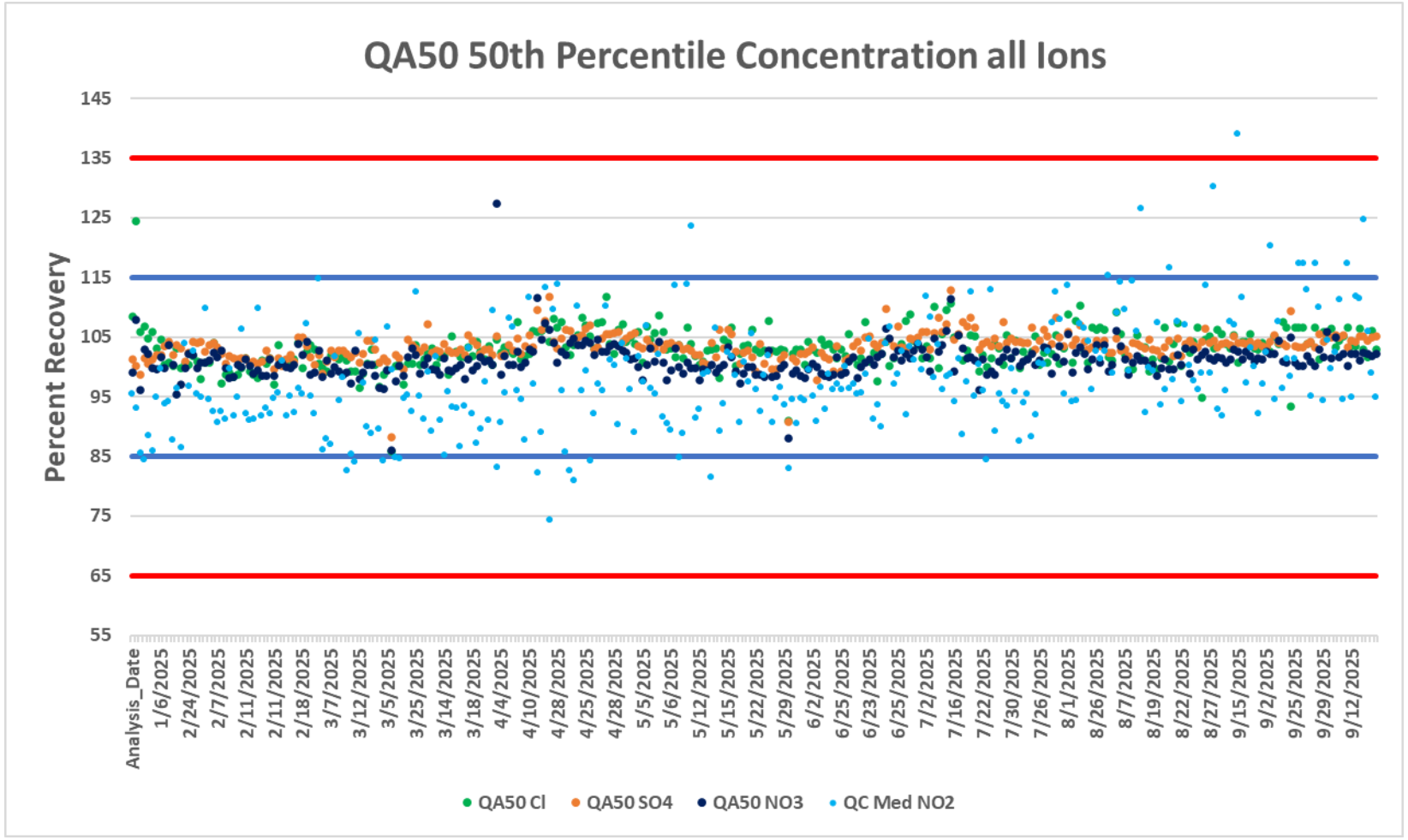
Control Charts



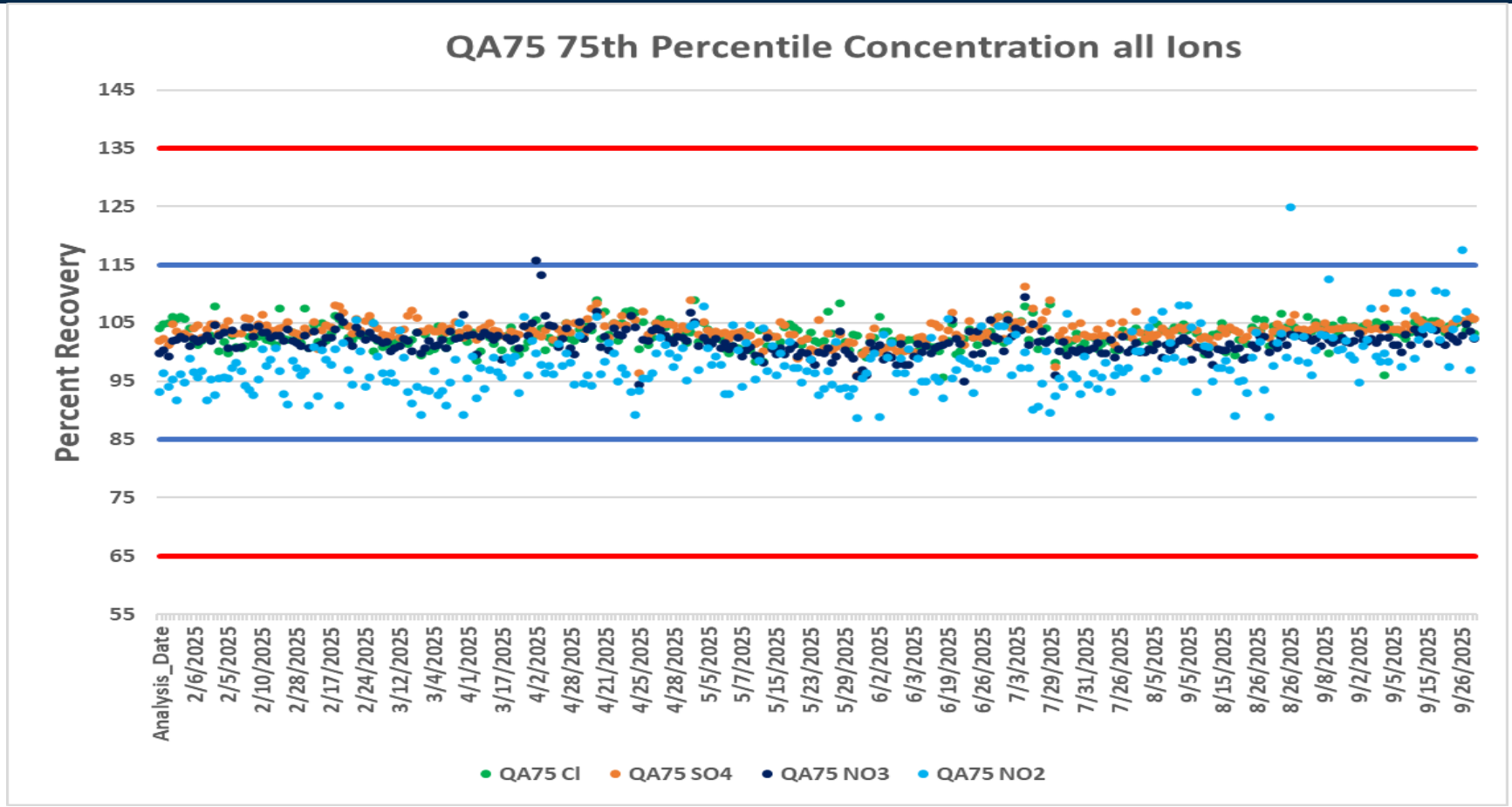
Approximate Air Concentration

Chloride - 0.016 µg/m^3
Sulfate - 0.300 µg/m^3
Nitrate - 0.092 µg/m^3
Nitrite - 0.007 µg/m^3

Control Charts

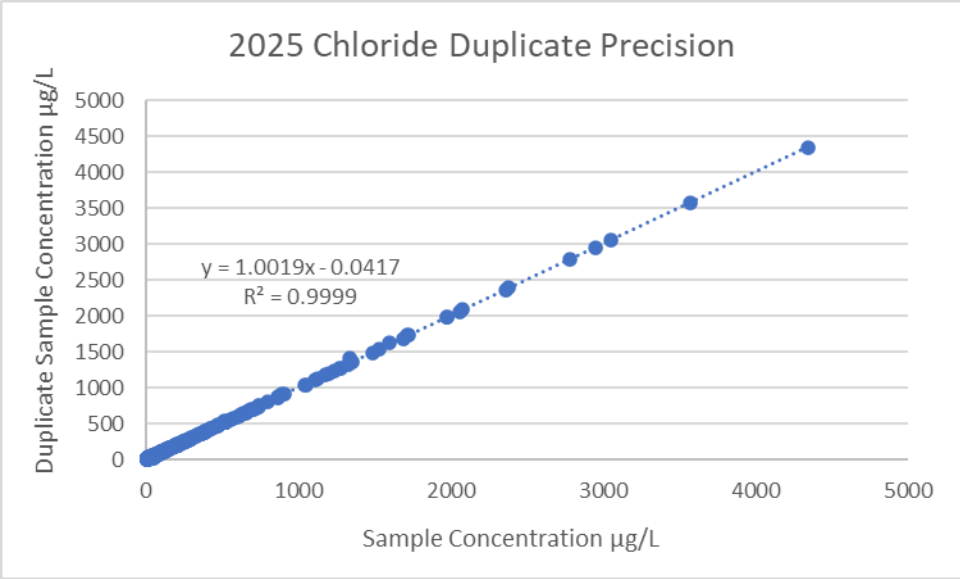


Chloride - 0.031 µg/m^3
Sulfate - 0.60 µg/m^3
Nitrate - 0.180 µg/m^3
Nitrite - 0.013 µg/m^3

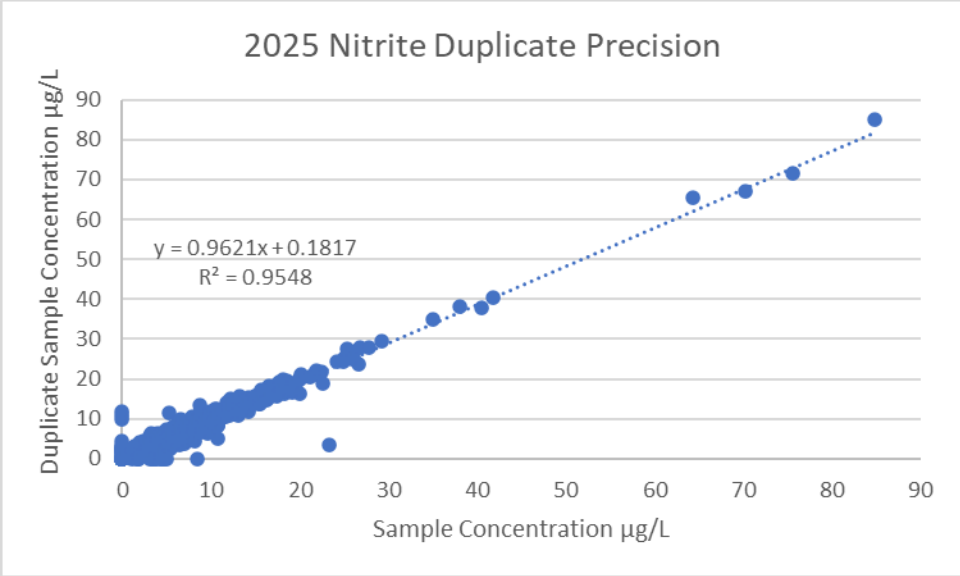


Chloride - 0.061 µg/m^3
Sulfate - 1.2 µg/m^3
Nitrate - 0.36 µg/m^3
Nitrite - 0.026 µg/m^3

Duplicate Precision

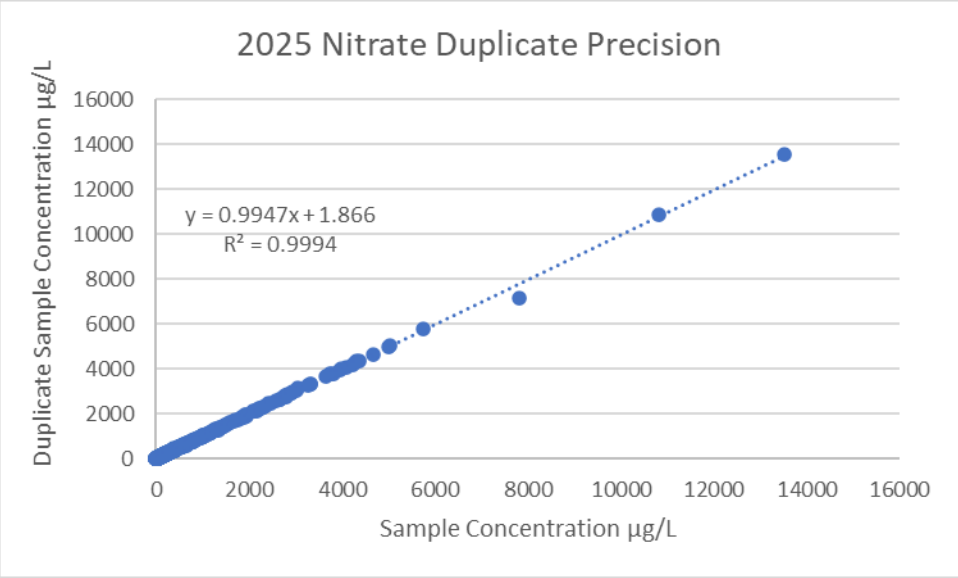


Chloride Percent Differences
Average = -0.04%
Median = -0.01%
Maximum = 82.8%
Minimum = -109%
Count = 840
Failures 0.36%

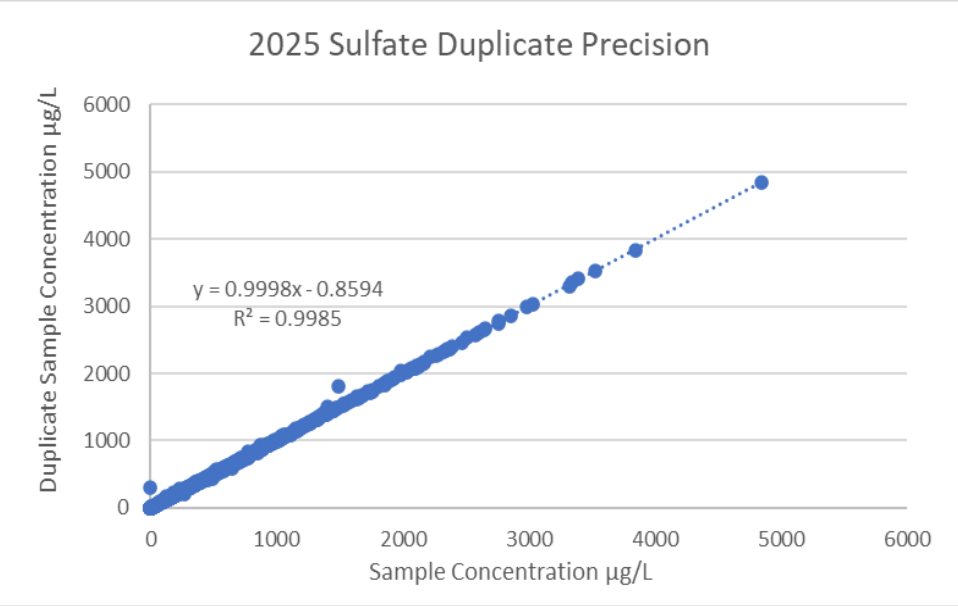


Nitrite Percent Differences
Average = -1.77%
Median = -0.0%
Maximum = 200%
Minimum = -200 %
Count = 840
Failures 3.69%

Duplicate Precision



Nitrate Percent Differences
Average = 0.67%
Median = 0.01%
Maximum = 200%
Minimum = -200 %
Count = 840
Failures 1.20%



Sulfate Percent Differences
Average = 0.24%
Median = 0.00%
Maximum = 200%
Minimum = -200%
Count = 840
Failures 1.31%

Reanalysis

	Chloride	Nitrite	Nitrate	Sulfate
2025 median RPD	0.71%	2.17%	0.23%	-0.33%
2025 Average RPD	0.71%	-3.71%	-0.35%	1.44%

- Roughly 5% of each batch of 400 NPS samples are reanalyzed after the original analysis.
- The Relative Percent Differences are calculated and verified against the DQO requirements.
- Any samples failing to meet DQO's are reanalyzed a third time to check.

Routine Filter Re-Extraction

- Extraction efficiencies were evaluated on 373 samples.

- Efficiency is 100% if the ion concentration measured in the re-extraction is 0, if the ion concentration is higher than the MDL, the efficiency is calculated by the difference divided by the sum. For NO_2, 100% if ion concentration is below 20 ppb.

Chloride	Nitrite	Nitrate	Sulfate
99.3%	93.2	98.2	100

RTI International

Comparability Environment and Climate Change Canada Proficiency Testing Program

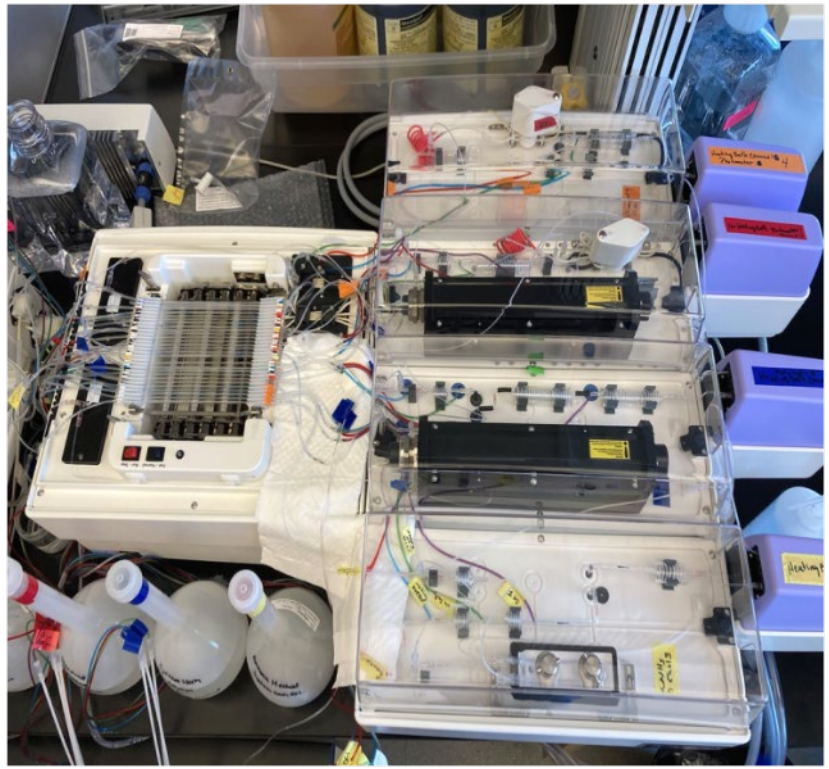
Summer 2023	Winter 2023	Winter 2024	Summer 2025
Very Good	Good	Good	Very Good

- Twenty-six participating laboratories
- Measure and report results for chloride, nitrate, sulfate, sodium, potassium and ammonium from ten samples
- Rain water matrix
- Laboratories receive an overall performance rating calculated using statistics and visually show in a plot of laboratory accuracy vs. precision

Total Nitrogen Research and OrthoPhosphate

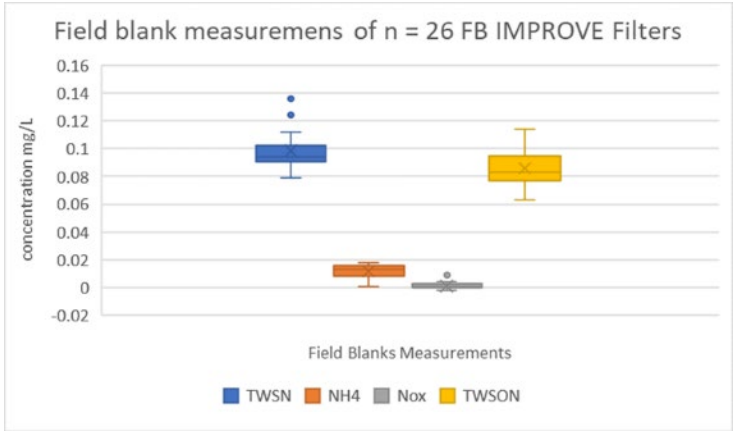
Segmented Flow
Colorimetric measurement

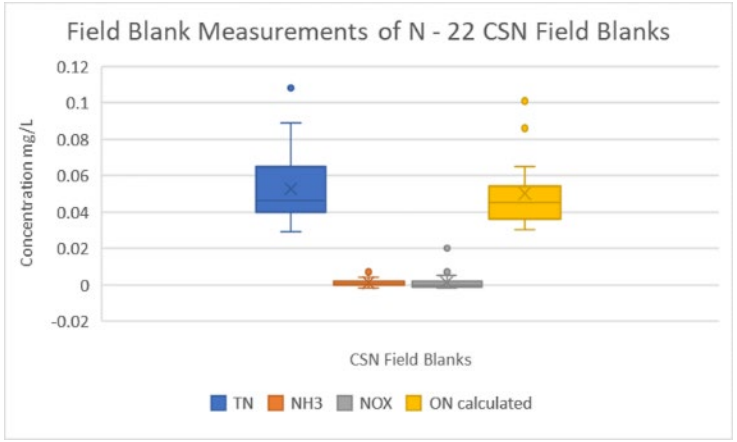
- NH_4^+
- $NO_x = NO_2^- + NO_3^-$
- Total Nitrogen inline persulfate/UV digestion)
- PO_4^{3-}



Total Water Soluble Nitrogen (TWSN)
Total Water Soluble Organic Nitrogen (TWSON)
TWSON = TWSN – (NH_4 + NO_x)
Total Water Soluble Phosphorus (TWSP)
Total Water Soluble Ortho Phosphate (TWSPO)

Method Development TN





Minimum Detection Limits

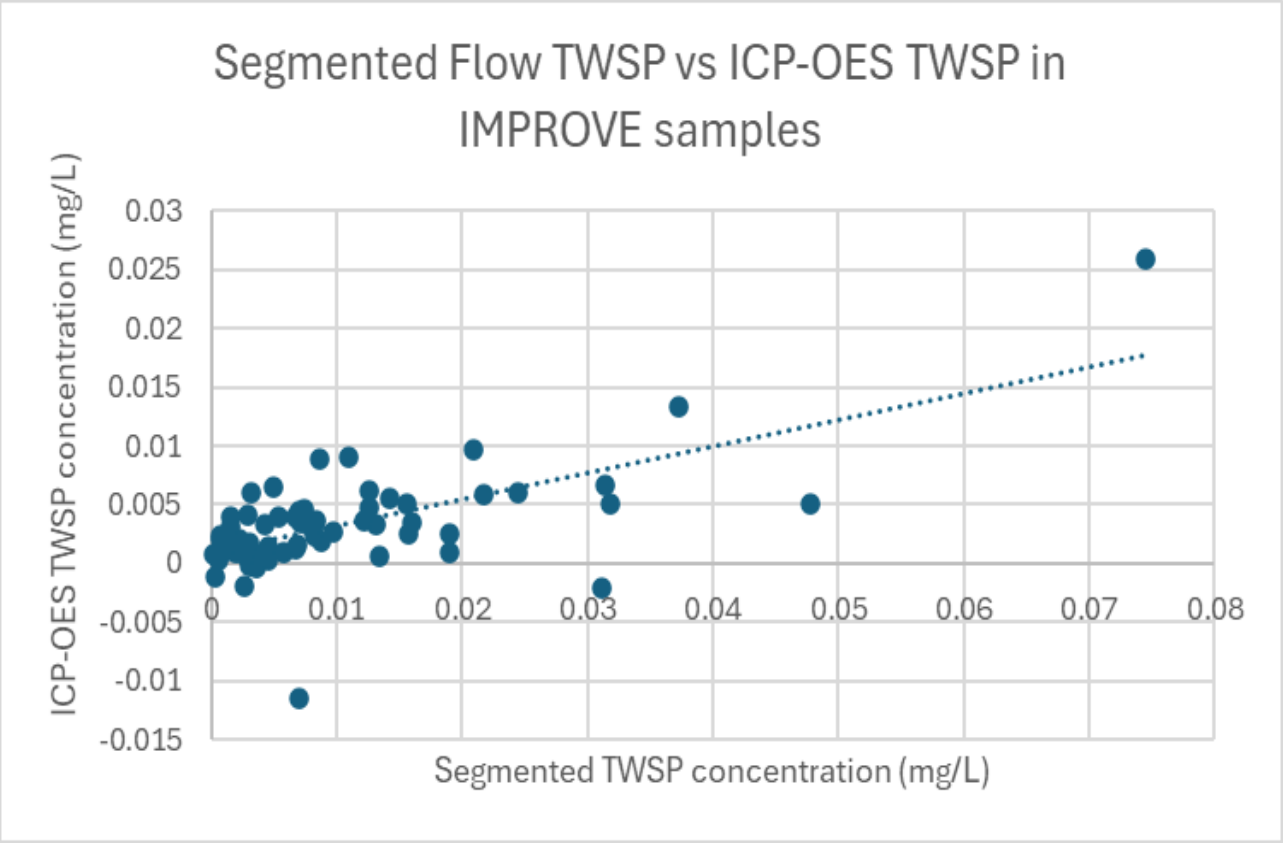
TWSN	NH4	NO3	TWSON
0.025 mg/L	0.017 mg/L	0.003 mg/L	0.045 mg/L
0.012 mg/L	0.013 mg/L	0.002 mg/L	0.020 mg/L

ECCC Average Recovery f

TN	NH4 by Seg	NH4 by IC	NO3 by Seg	NO3 by IC
95.5%	84.3%	99.1%	98.3%	98.9%

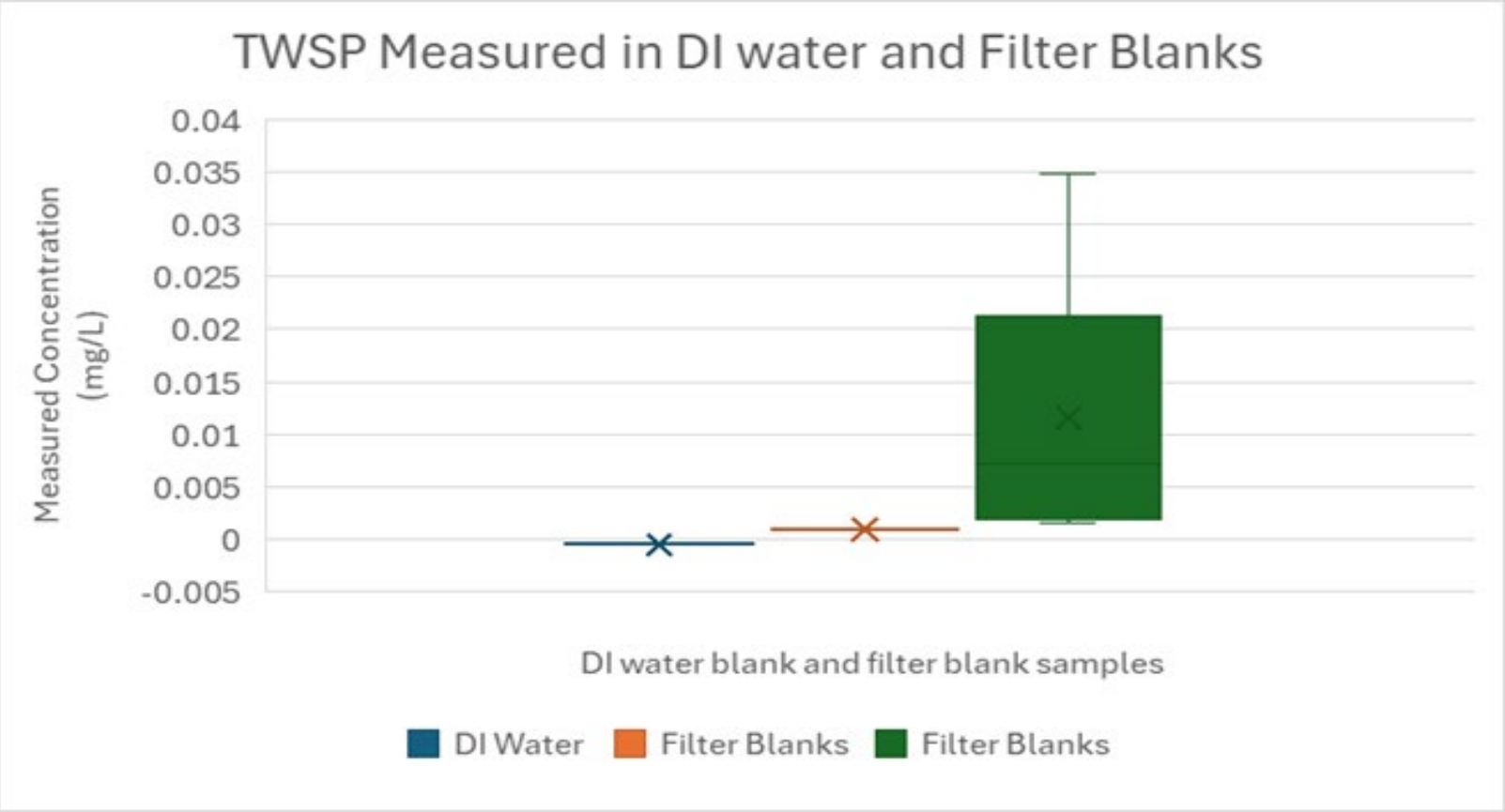
Validation Study for TWSP

MDL = 0.0005 mg/L as P by Segmented Flow Analysis
Analytical range 0.0005 – 0.250 mg/L



Identified NPS samples with peaks that are consistent with Orthophosphate peak in IC data. Measured these samples by ICP-OES and Segmented Flow analysis. Segmented Flow samples biased high compared to ICP-OES

Interferences?



Measurements of Deionized water, freshly prepared filter blanks, and older filter blanks. Suspected degradation of filters and particles in samples impacts measured results by Segmented Flow analysis for Nylon filter samples

Acknowledgements

- RTI IC team Kat Lindskog, Adam Conway, Amelia Smith, Evan Thorp, Laurie Stella, Lauren Johann, Katie Olive, Zahkura Eastman

- UCD team for validating the data

- RTI for continued research funding

delivering **the promise of science**
for global good

RTI INTERNATIONAL

Name Tracy Dombek
Email tdombek@rti.org
Phone Number 919-541-5934