Nutrient Analysis at San Jose RWF Laboratory: Methods and Instrumentation Overview

Buddhima Mahanama
Environmental Laboratory Supervisor
San Jose RWF Laboratory
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CITY OF SAN JOSE
CAPITAL OF SILICON VALLEY

Environmental Services

Delivering world class utility services and programs to improve our health, environment, and economy.

Key Nutrients of Concern in Wastewater Treatment

Nitrogen

- Inorganic Nitrogen (TIN)
  - Ammonia, NH_3
  - Nitrite, NO_2^-
  - Nitrate, NO_3^-
- Total Kjeldahl Nitrogen (TKN)
- Total Nitrogen (TN)

Phosphorus

- Total Phosphorus (TP)
- Orthophosphate (Ortho-PO_4^{3-})

Organic Carbon

- Total Organic Carbon (TOC)
- Dissolved Organic Carbon (DOC)

Importance of Nutrient Monitoring in Wastewater

- Mitigate Eutrophication

- Protect Aquatic Life

- Public Health Concerns

- Meeting Regulatory Compliance
- Support Process Control & Optimization

NPDES and Nutrient Permits

- Effluent Limitations for NH_3, Total:
  - Average Monthly = 3.0 mg/L
  - Maximum Daily = 8.0 mg/L
- Interim Effluent Limitations for TIN:
  - 6400 kg/day

		Parameter				
	Sampling Location	Total NH_3	NO_3^--NO_2^-	TIN	TKN	TP
Sampling Frequency	Effluent	2/Month			NA	1/Month
	Influent	1/Quarter				2/Year

Process & Struvite Monitoring

	Parameter						
Location	NH_3	NO_2^-	NO_3^-	TN	TKN_{Calc}	TP	Ortho-PO_4^{3-}
Raw Sewage	D	M, W	M, W	M, W	M, W	M, W	
Primary Effluent	D	M, W	M, W	M, W	M, W	M, W	M, W
Settled Sewage	D	Th	Th				
Nitrification Influent	D						
Nitrification Effluent	D	D	D				
Secondary Effluent	D	D	D				
Filter Influent	D	D	D			D	D
Filter Effluent		M, W, F	M, W, F				
Final Effluent	D	D	D	2xMn	2xMn		
Recycled Water	Mn	Mn	Mn			Mn	Wk
Digesters	M					M, Th	M, Th
Sludges	T, F					T, F	T, F

D-Daily, Wk-Weekly, Mn-Monthly, M-Monday, T-Tuesday, W-Wednesday, Th-Thursday, F-Friday

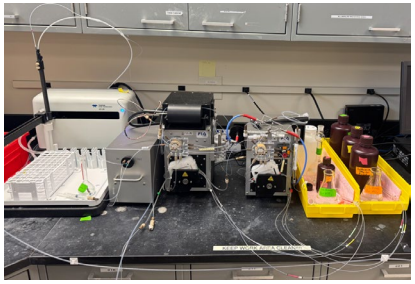
Nutrient Methods used in the San Jose RWF Laboratory

Analyte	Method	Instrument	Technology	Reporting Units
NH_3-N & TKN	EPA FIAlab 100	Flow Injection Analyzer, FIAlyzer-1000	Gas Diffusion & Fluorescence	mg/L
NO_2^--N	EPA 353.2	Flow Injection Analyzer, FIAlyzer-1000	Spectrophotometry	mg/L
NO_3^--N	EPA 300.0	Ion Chromatography	Anion Exchange & Conductivity	mg/L
TP & Ortho PO_4^{3-}	SM 4500 P, F	Discrete Analyzer, Seal AQ2 or 300	Spectrophotometry	mg/L
TOC/DOC	SM 5310 B	TOC-TN Analyzer, Shimadzu TOC L	High-temperature Combustion & NDIR	mg/L
TN	ASTM D8083-16	TOC-TN Analyzer, Shimadzu TOC L	High-temperature Combustion & CLD	mg/L

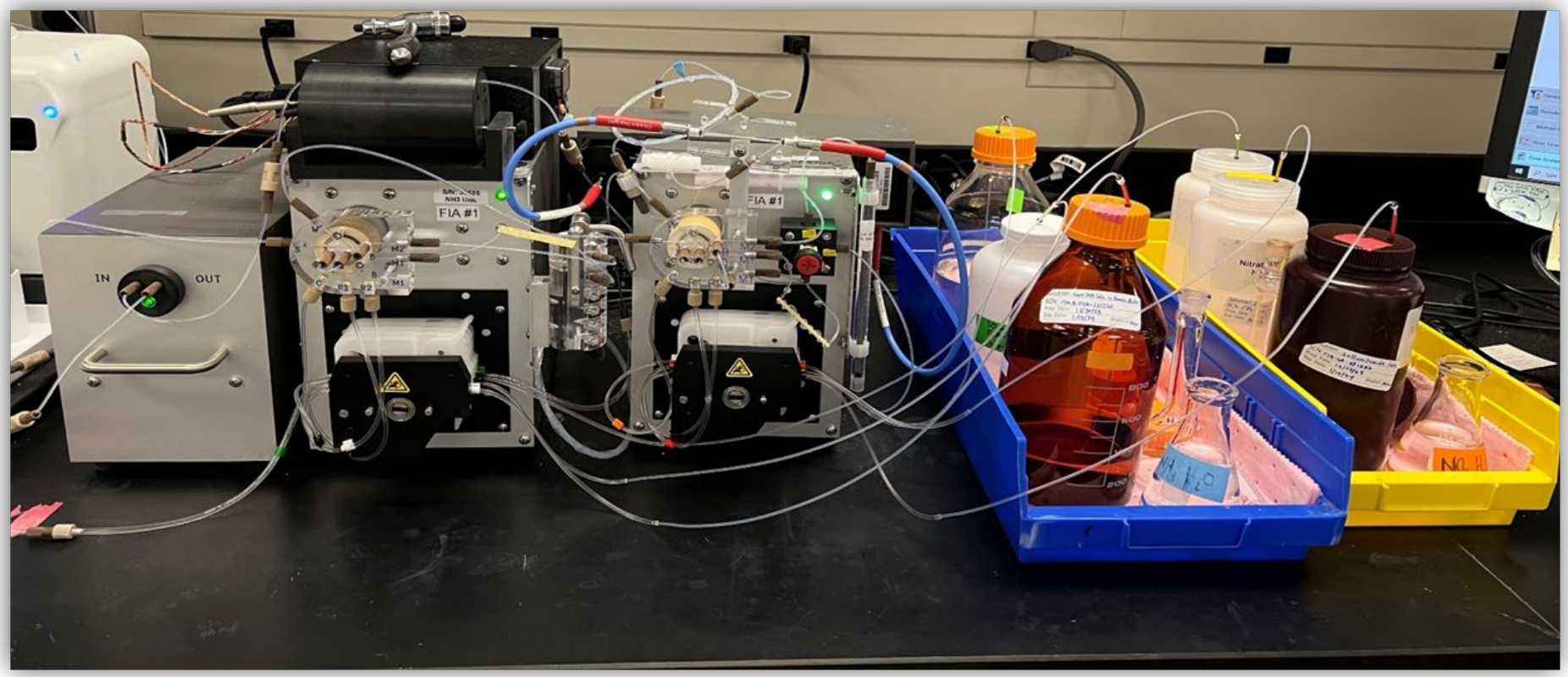
The use of instrument brand names is solely for information only and is not an endorsement by the San Jose RWF Laboratory. Please contact the laboratory directly for more information.

Nutrient Analysis at San Jose RWF Laboratory

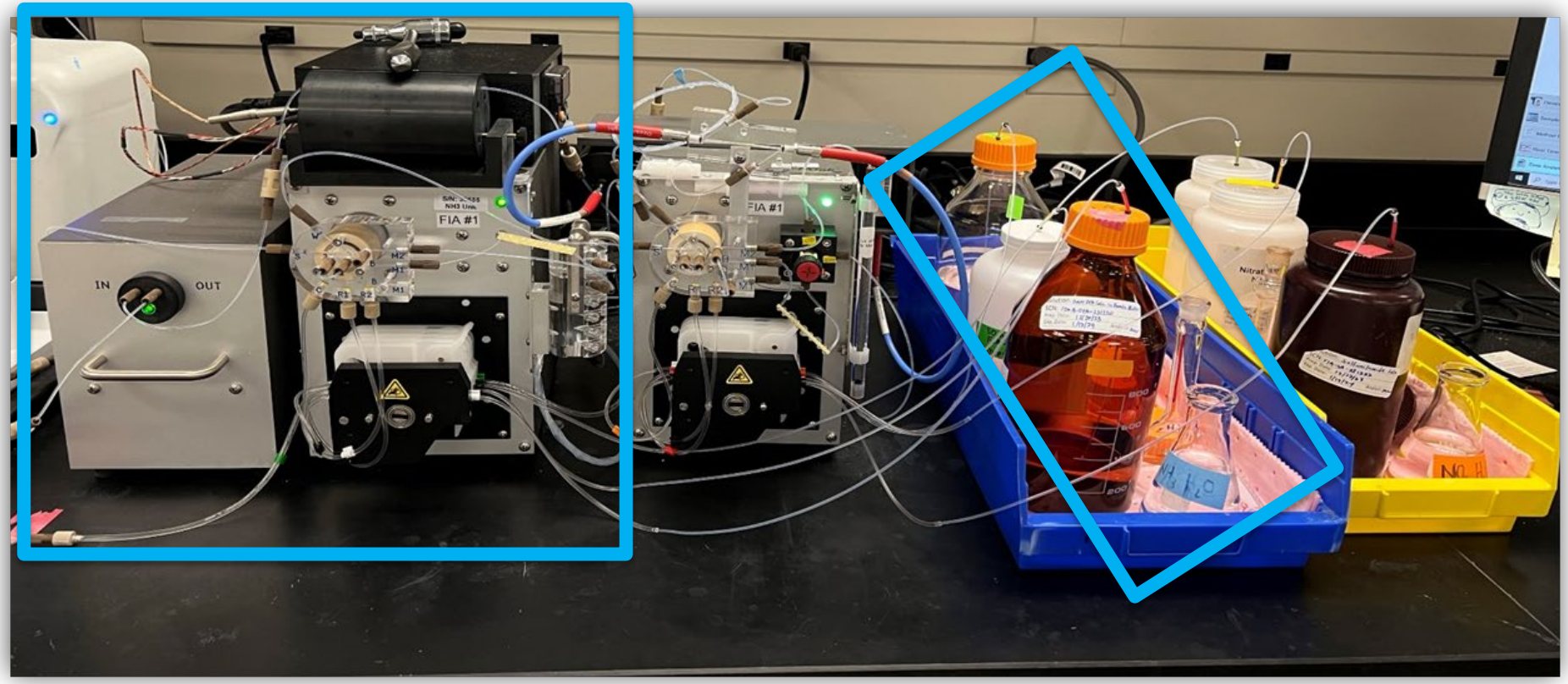
- Flow Injection Analyzer
  - NH_3, TKN, & NO_2^-
- Ion Chromatography System
  - NO_3^-
- Discrete Analyzer
  - TP & Ortho-PO_4^{3-}
- TOC-TN Analyzer
  - TOC/DOC, TN, & $TKN_{calculated}$



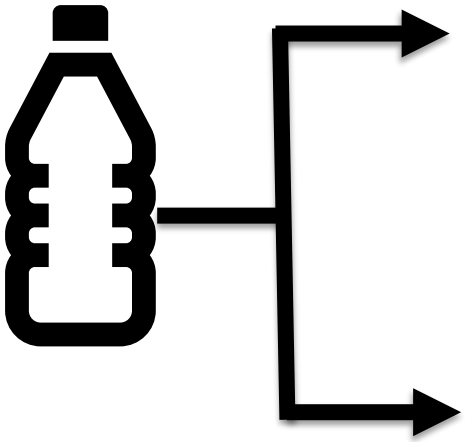
Flow Injection Analysis



Flow Injection Analysis: NH_3 & TKN



Flow Injection Analysis: NH_3 & TKN by EPA FIAlab 100



NH_3 : Centrifuge
(2000 rpm for 20 min)

TKN: Digestion

Free NH_3 & Organic Nitrogen

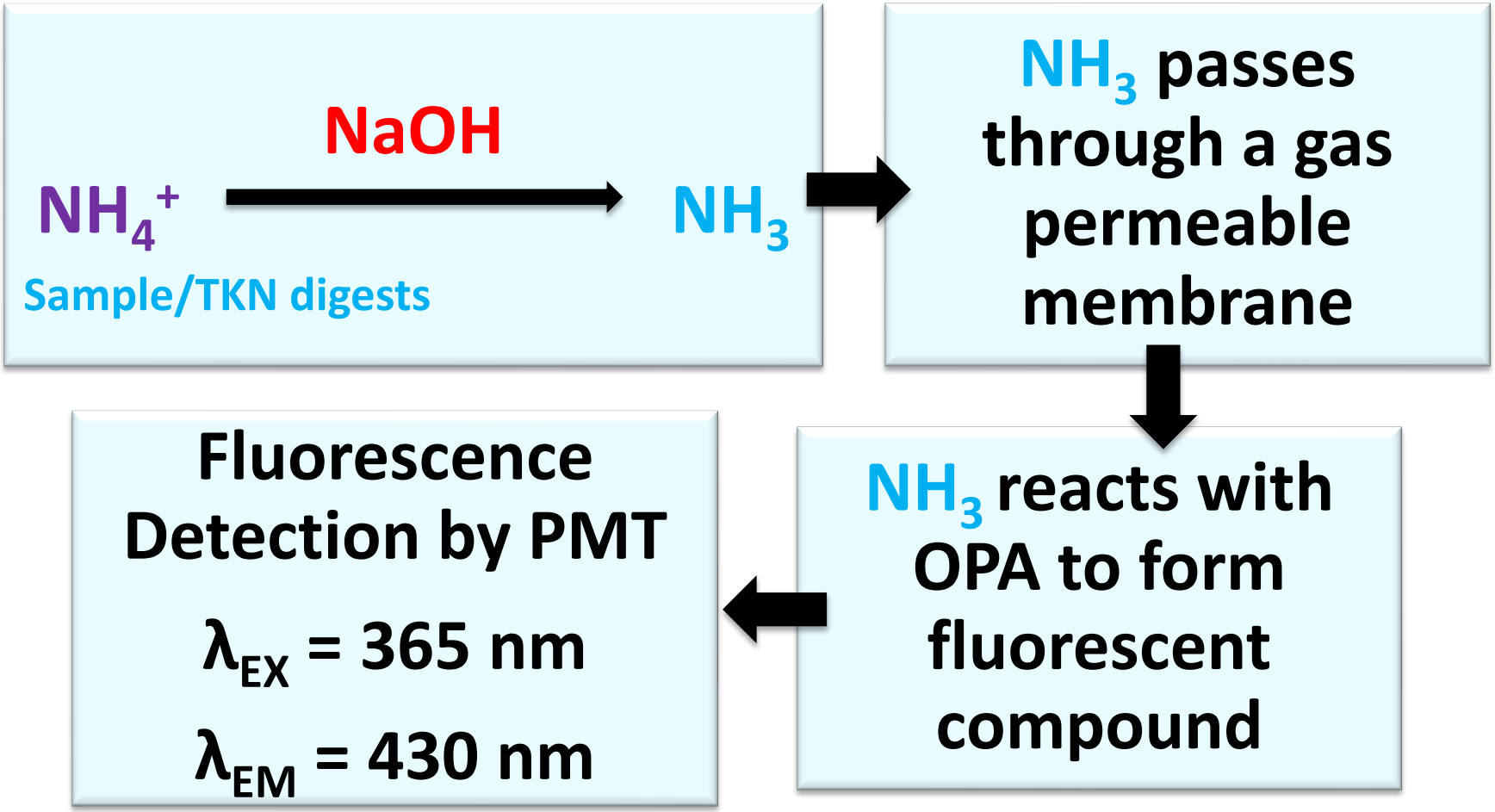
H_2SO_4, K_2SO_4, $CuSO_4$

160 °C, 1 h

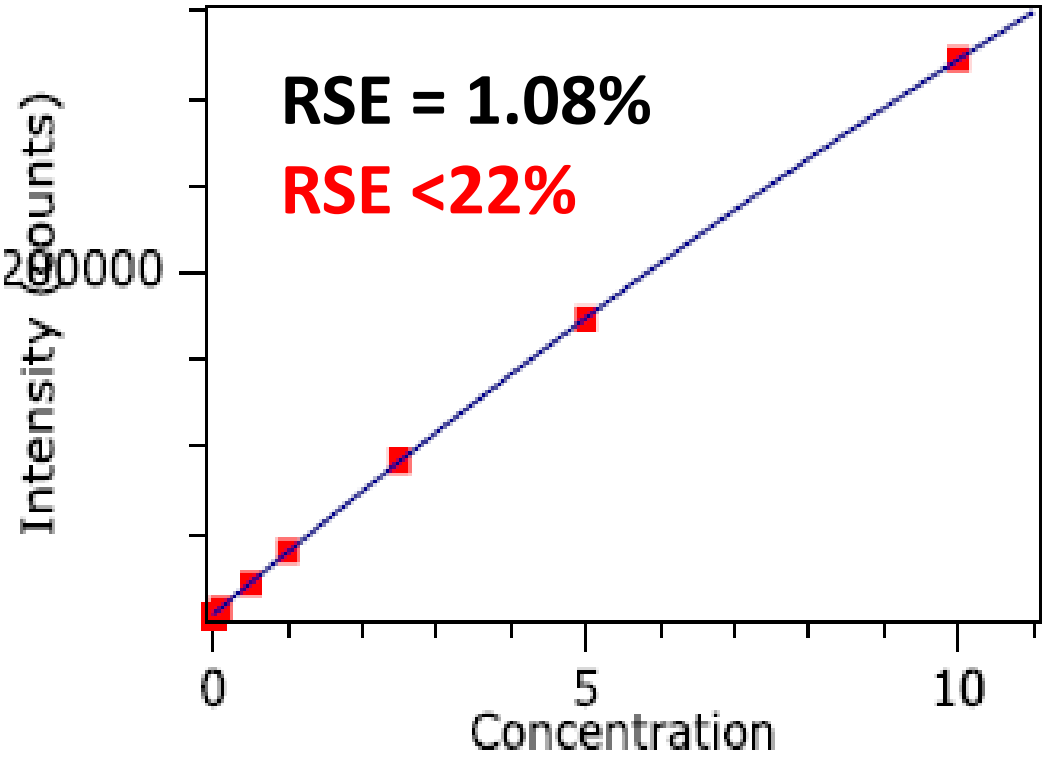
380 °C, 30 min

NH_4^+

Flow Injection Analysis: NH_3 & TKN by EPA FIAlab 100



Flow Injection Analysis: NH_3 by EPA FIAlab 100

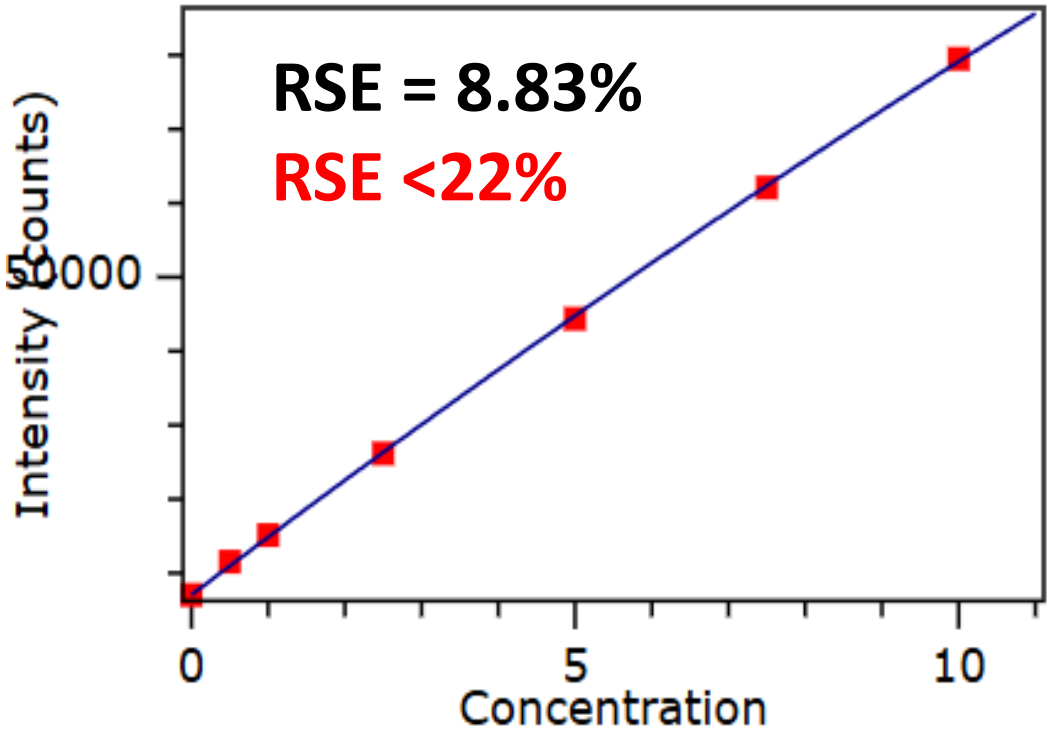


MDL = 0.02 mg/L

RL = 0.1 mg/L

Sample	NH_3 (mg/L)
Final Effluent	0.398
Raw Sewage	30.0
Recycled Water	1.73
Industrial Waste	39.9
South Bay Water	0.301

Flow Injection Analysis: TKN by EPA FIAlab 100

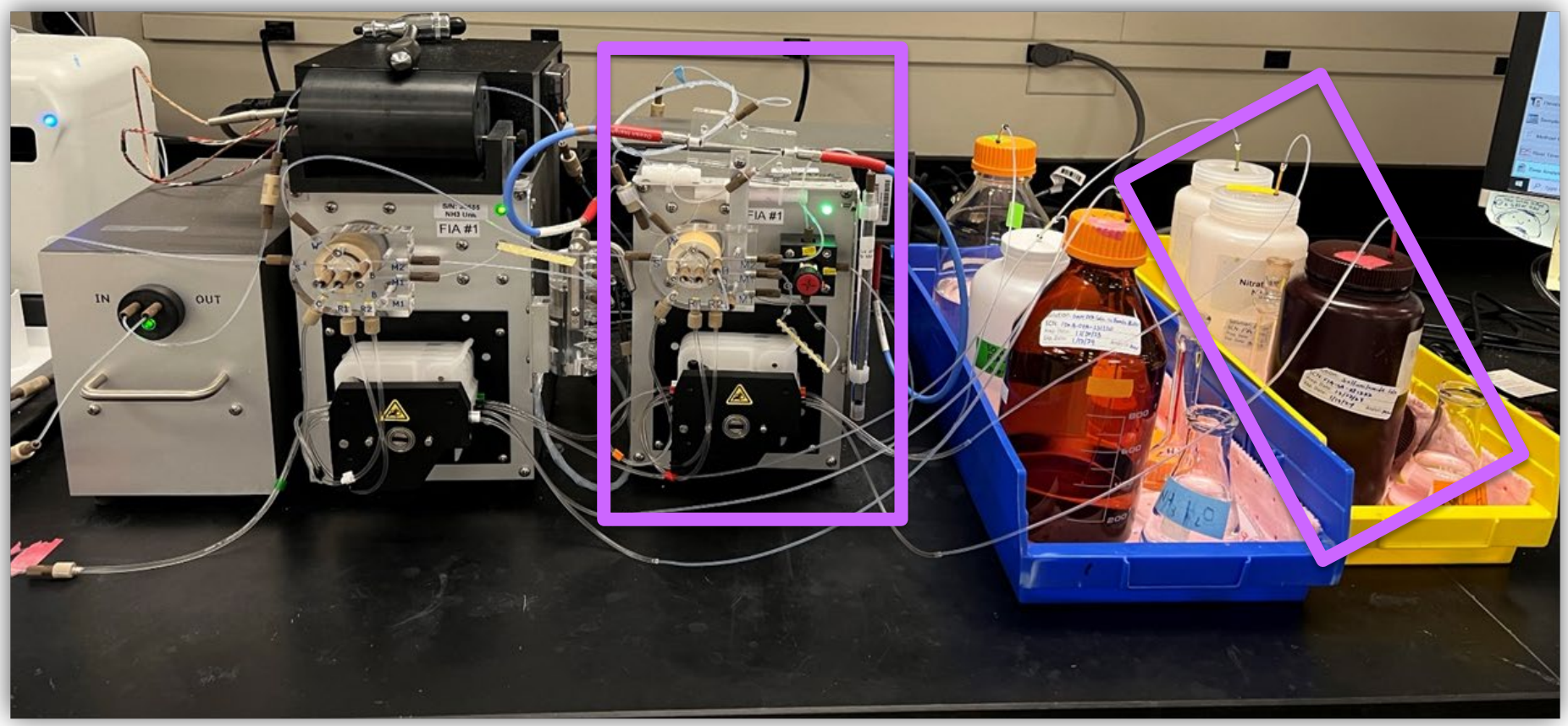


Sample	TKN (mg/L)
Raw Sewage	46.8

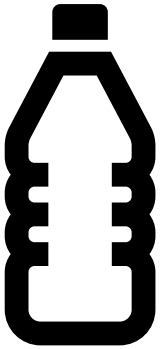
MDL = 0.16 mg/L

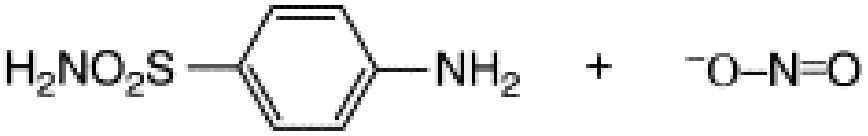
RL = 0.5 mg/L

Flow Injection Analysis: NO_2^-

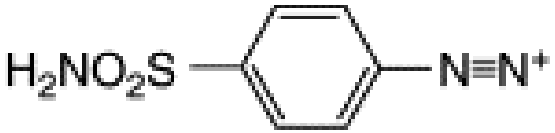


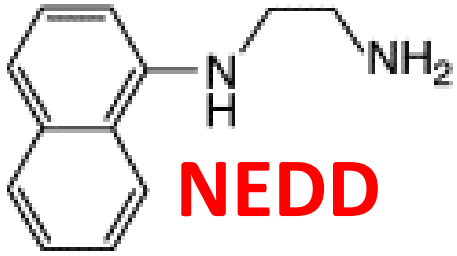
Flow Injection Analysis: NO_2^- by EPA 353.2





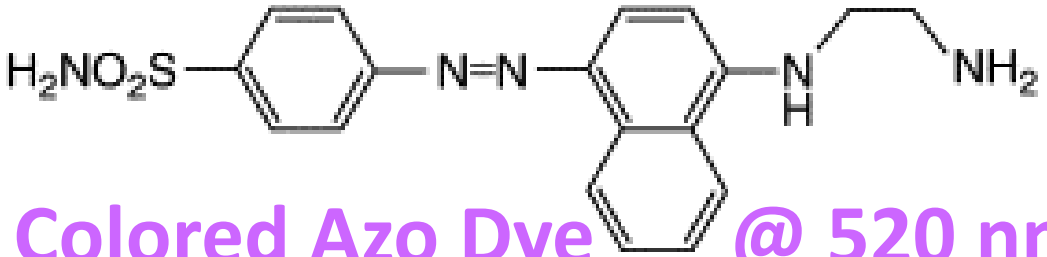
Sulfanilamide





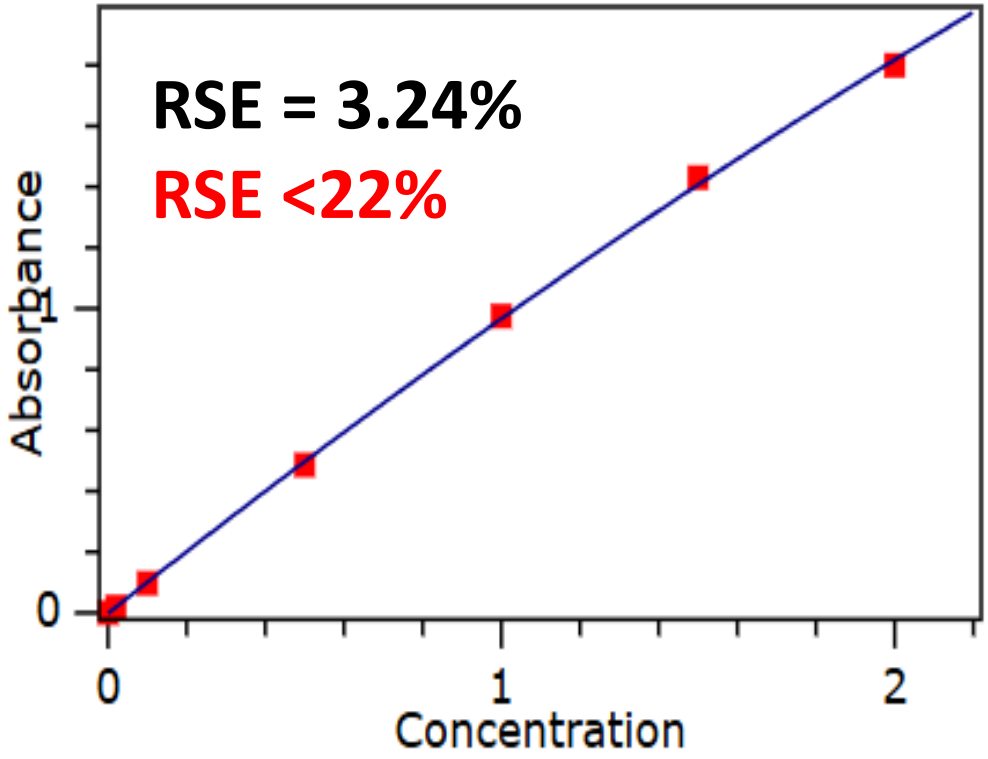
NEDD

Centrifuge (2000 rpm for 20 min)



Magenta Colored Azo Dye @ 520 nm

Flow Injection Analysis: NO_2^- by EPA 353.2



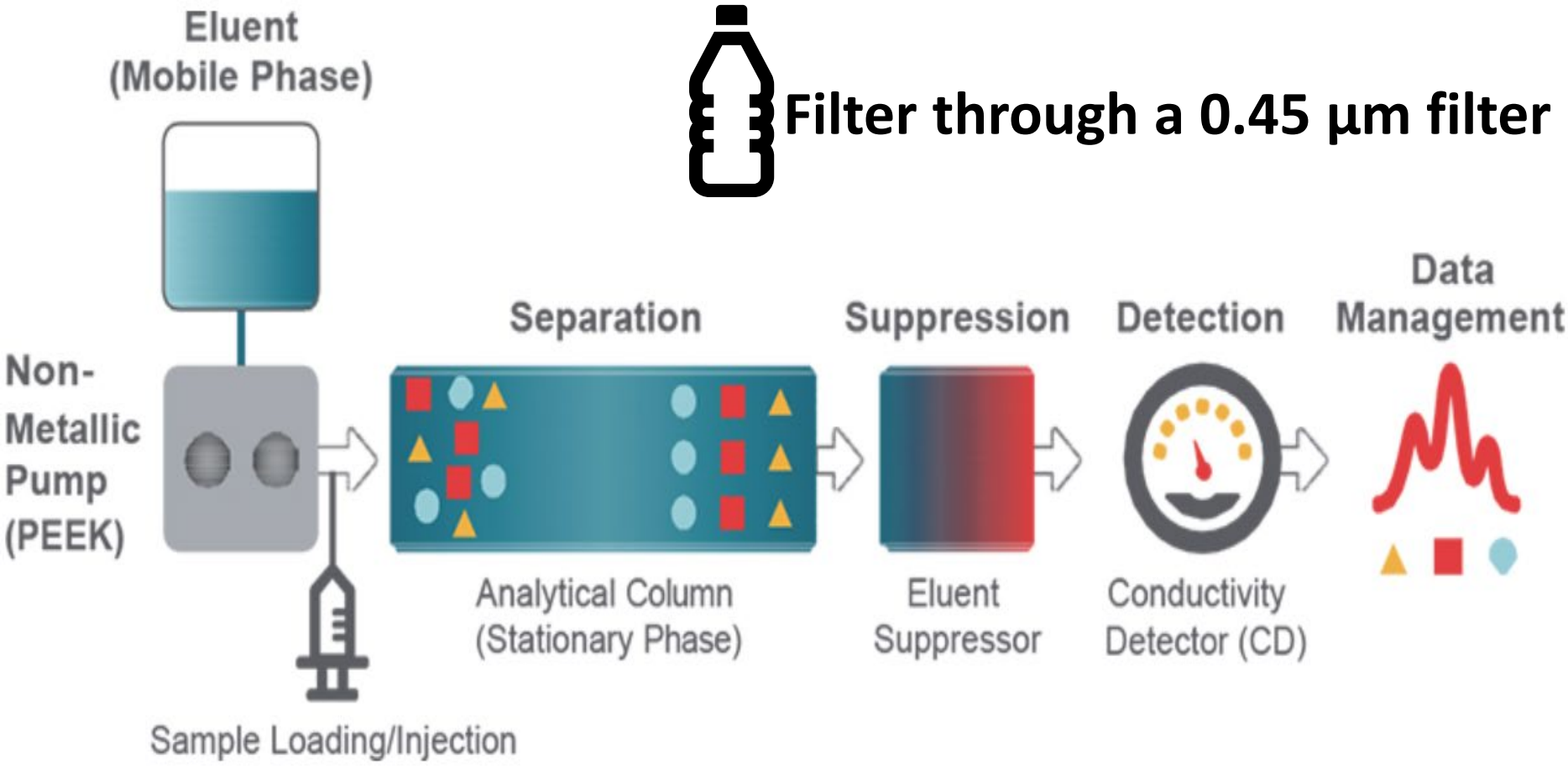
MDL = 0.005 mg/L

RL = 0.02 mg/L

Sample	NO_2^- (mg/L)
Final Effluent	0.166
Raw Sewage	1.15
Recycled Water	0.035

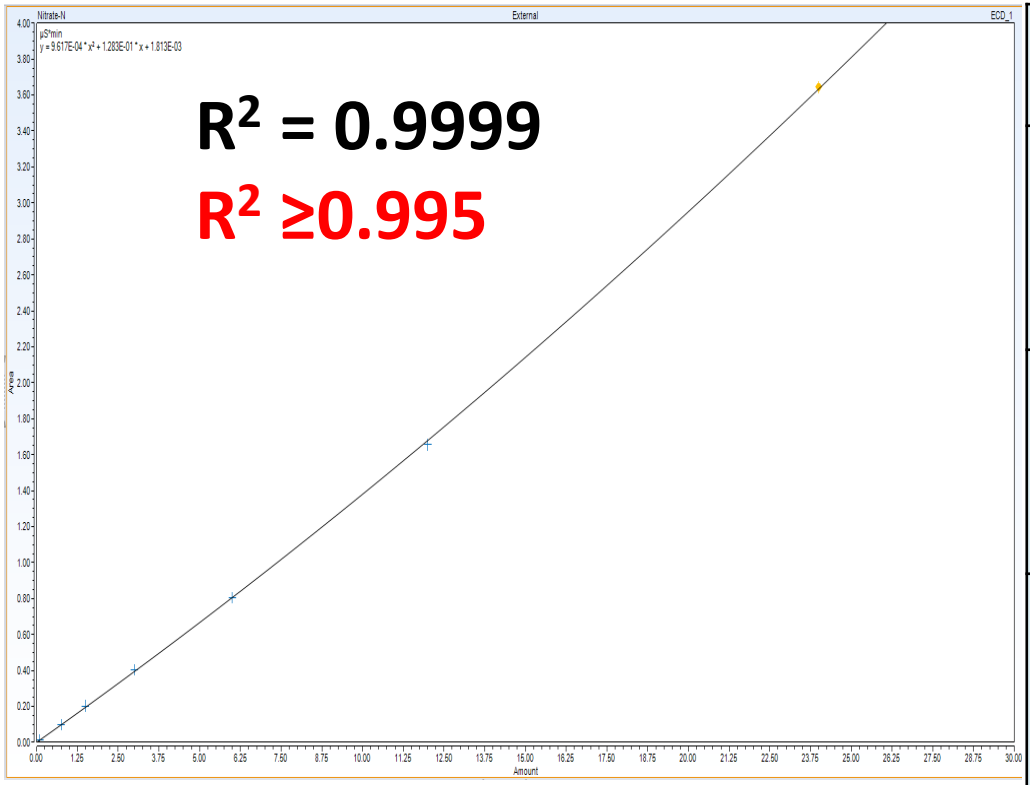
Ion Chromatography: NO_3^-

Ion Chromatography: NO_3^- by EPA 300.0



https://www.spectroscopyeurope.com/article/column-properties-make-impact-ion-chromatography

Ion Chromatography: NO_3^- by EPA 300.0



$R^2 = 0.9999$

$R^2 \geq 0.995$

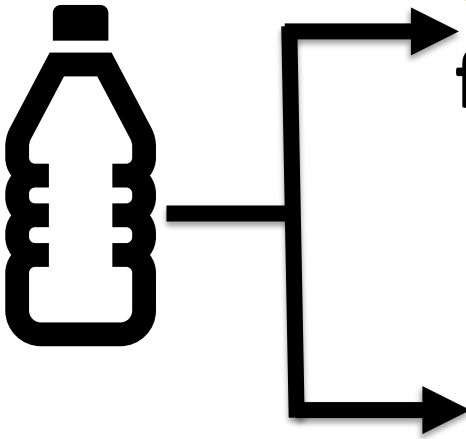
Sample	NO_3^- (mg/L)
Final Effluent	12.7
Raw Sewage	2.98
Recycled Water	7.84

MDL = 0.02 mg/L

RL = 0.1 mg/L

Discrete Analyzer: TP & Ortho-PO_4^{3-}

Discrete Analyzer: TP & Ortho-PO_4^{3-} by SM 4500 P, F



Ortho-PO_4^{3-} : Filter through a 0.45 µm filter within 15 min of collection

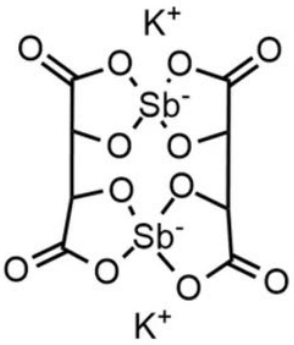
TP: Digestion

Polyphosphates & Organic Phosphates

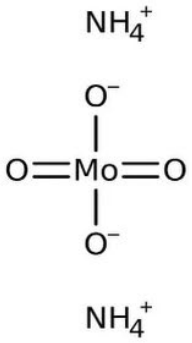
$\xrightarrow[\text{85-95°C, ~3 h}]{\text{11 N } H_2SO_4,\ (NH_4)_2S_2O_8}$

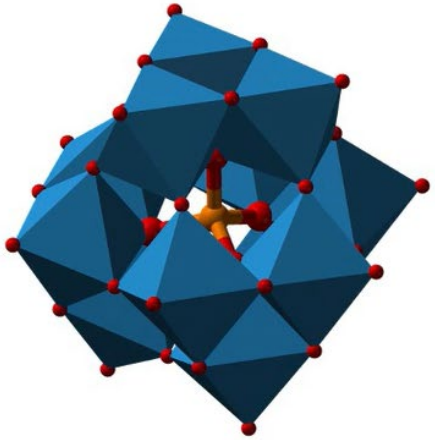
Ortho-PO_4^{3-}

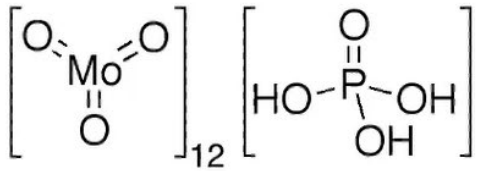
Discrete Analyzer: TP & Ortho-PO_4^{3-} by SM 4500 P, F



Ortho-PO_4^{3-}

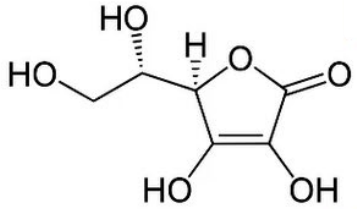






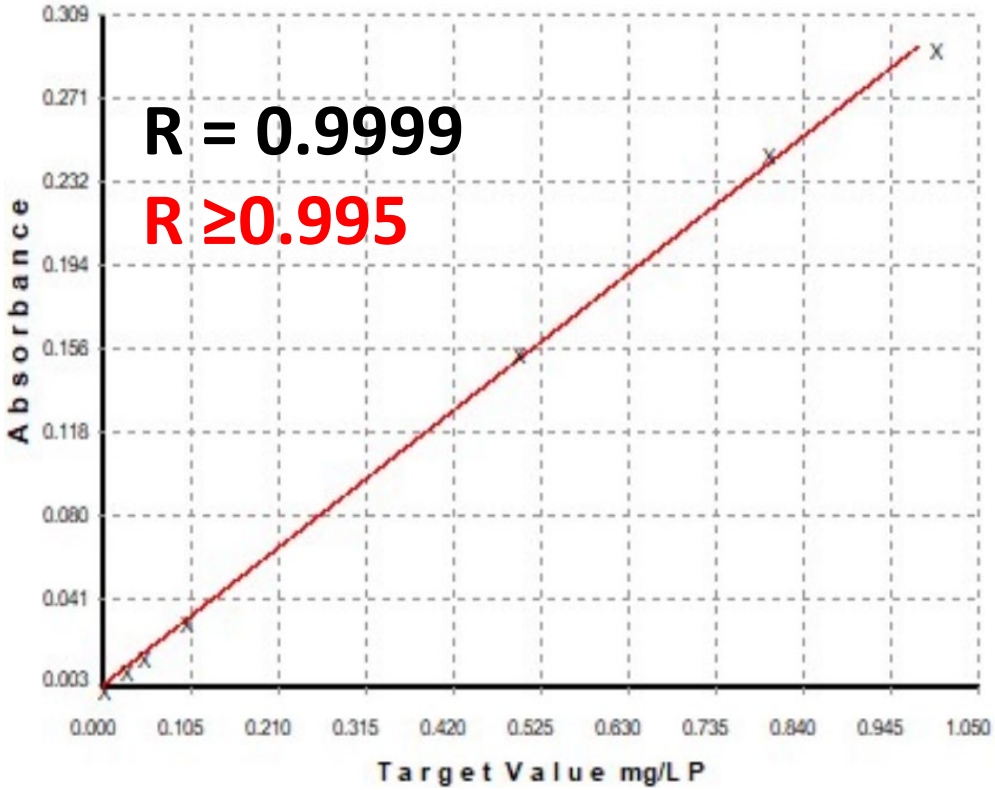
Sb-Phosphomolybdate Complex

Ascorbic Acid



Phospho-molybdenum Blue Complex @880 nm

Discrete Analyzer: TP & Ortho-PO_4^{3-} by SM 4500 P, F



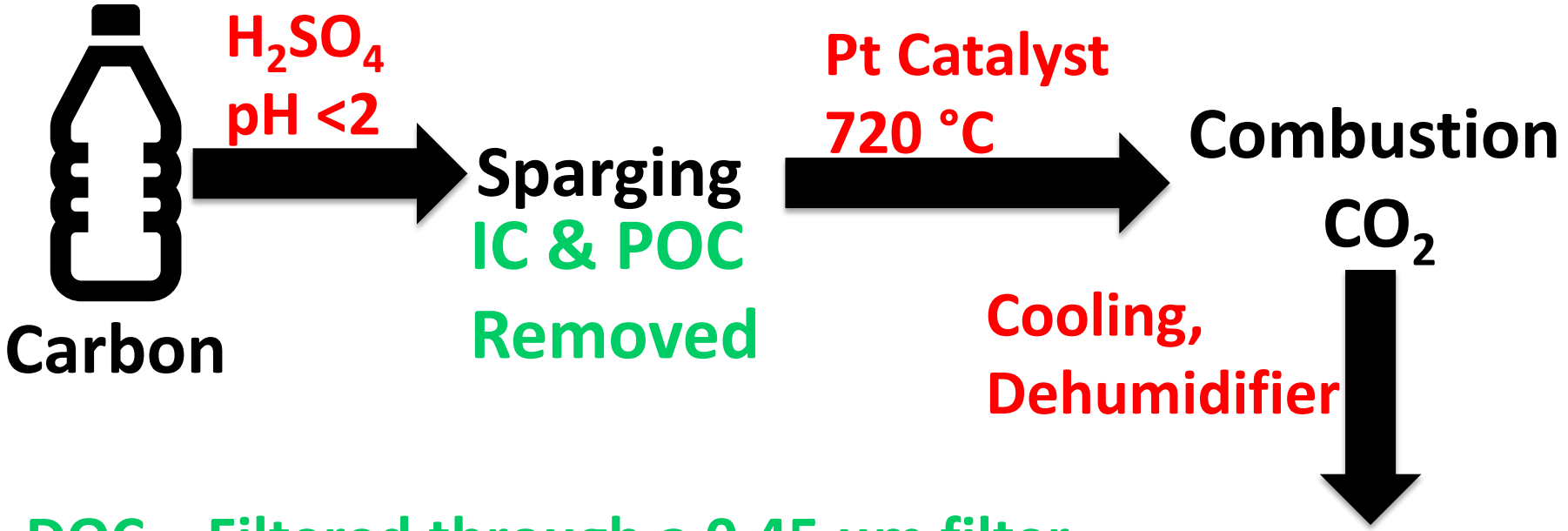
Sample	TP (mg/L P)
Final Effluent	0.367
Raw Sewage	6.63
Recycled Water	0.192

MDL = 0.01 mg/L

RL = 0.03 mg/L

TOC-TN Analyzer: TOC/DOC by SM 5310 B

TOC-TN Analyzer: TOC/DOC by SM 5310 B



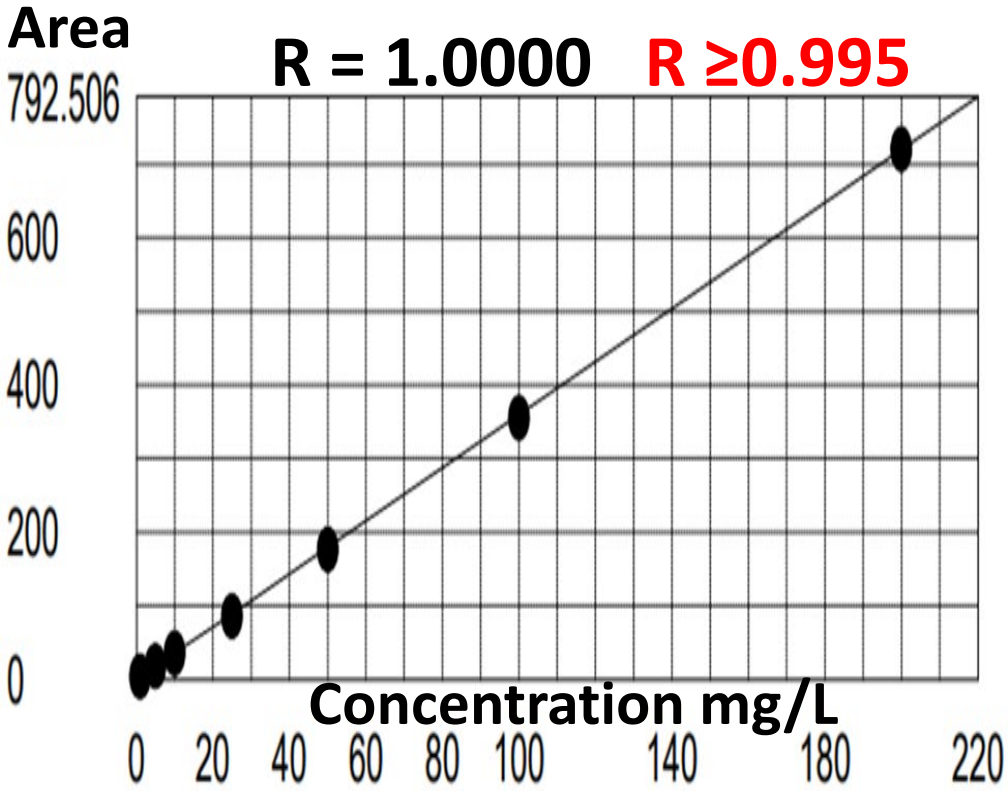
DOC = Filtered through a 0.45 µm filter

IC = Inorganic Carbon

POC = Purgeable Organic Carbon

NPOC = Non-purgeable Organic Carbon

TOC-TN Analyzer: TOC/DOC by SM 5310 B



Sample	TOC (mg/L)
Micro DIW	0.13 (<MDL)

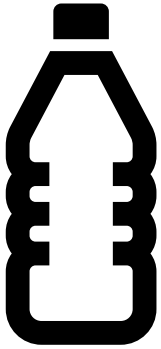
Sample	DOC (mg/L) Filtered
South Bay	2.32

MDL = 0.67 mg/L

RL = 1.0 mg/L

TOC-TN Analyzer: TN by ASTM D8083-16 and $TKN_{calculated}$

TOC-TN Analyzer: TN by ASTM D8083-16 and $TKN_{calculated}$



Nitrogen

Air

Pt Catalyst

720 °C

Combustion

NO

O_3

$NO_2^* + O_2$

NO_2 + Light (hv)

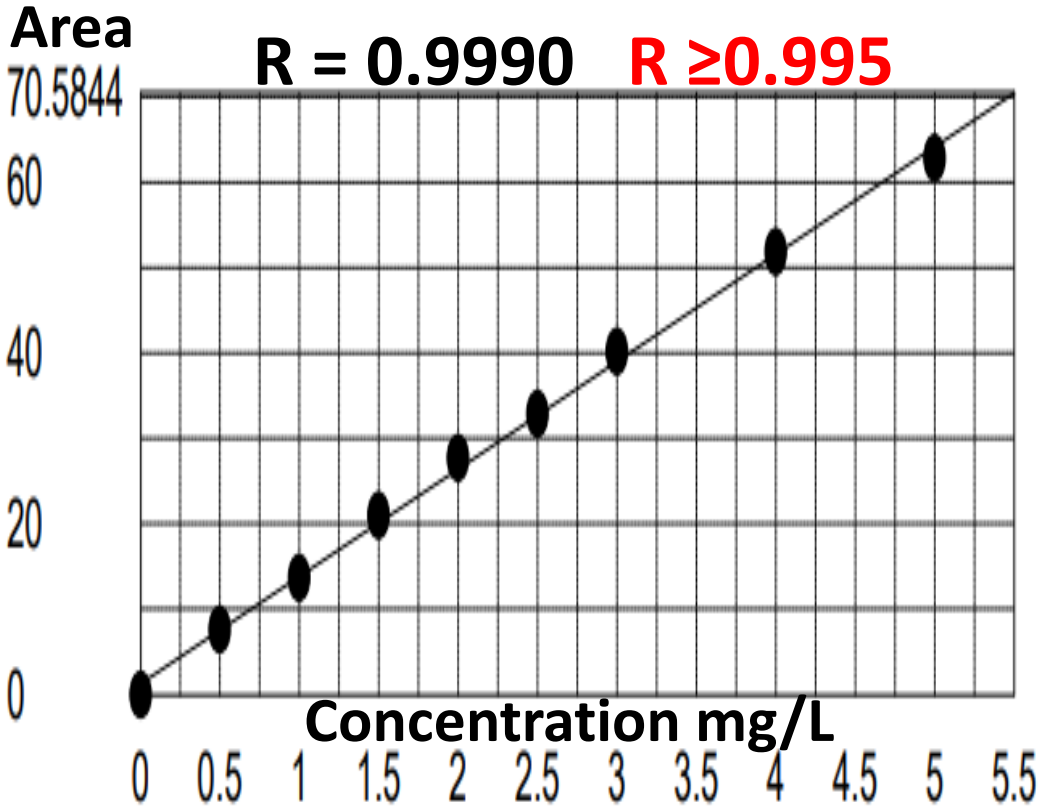
Chemi-luminescence Detection

$$TKN_{calculated} = TN - (NO_2^- + NO_3^-)$$

Flow Injection

Ion Chromatography

TOC-TN Analyzer: TN by ASTM D8083-16 and $TKN_{calculated}$



MDL = 0.09 mg/L

RL = 0.50 mg/L

Sample	TN (mg/L)
Final Effluent	15.8
Raw Sewage	65.2
Primary Effluent	52.8
South Bay	2.66

Method Quality Control

Method \ QC	ICV	ICB/ CCB	MB	LCS	MS/MSD	RPD	CCV
EPA FIAlab 100 (NH_3/TKN)	✘	✓	✓	✓	✓ 20	✓	✓
EPA 353.2 (NO_2^-)	✓	✓	✓	✓	✓ 10	✓	✓
EPA 300.0 (NO_3^-)	✓	✓	✓	✓	✓ 10	✓	✓
SM 4500 P, F (TP, O-PO_4^{3-})	✓	✓	✓	✓	✓ 20	✓	✓
SM 5310 B (TOC/DOC)	✓	✓	✓	✓	✓ 20	✓	✓
ASTM D8083-16 (TN)	✓	✓	✓	✓	✓ 20	✓	✓

Additional Quality Control

SM 5310 B (TOC/DOC):

- **Carry Over Check**

  Highest Calibration standard-blank pair

  Blank <1/2 RL

- **IC Removal Check**

  <1/2 RL

- **Rotating CCV at Low, Mid, and High**

Conclusions

- Types of Nutrients Analyzed at RWF Laboratory
- Sample Preparation
- Overview of Instrumentation & Methods
- Sample Types & Typical Results