FIA Lab

Fluidics Intelligently Automated

Ammonia & TKN Measurements by the OPA Method

Lessons from Experience

Contents

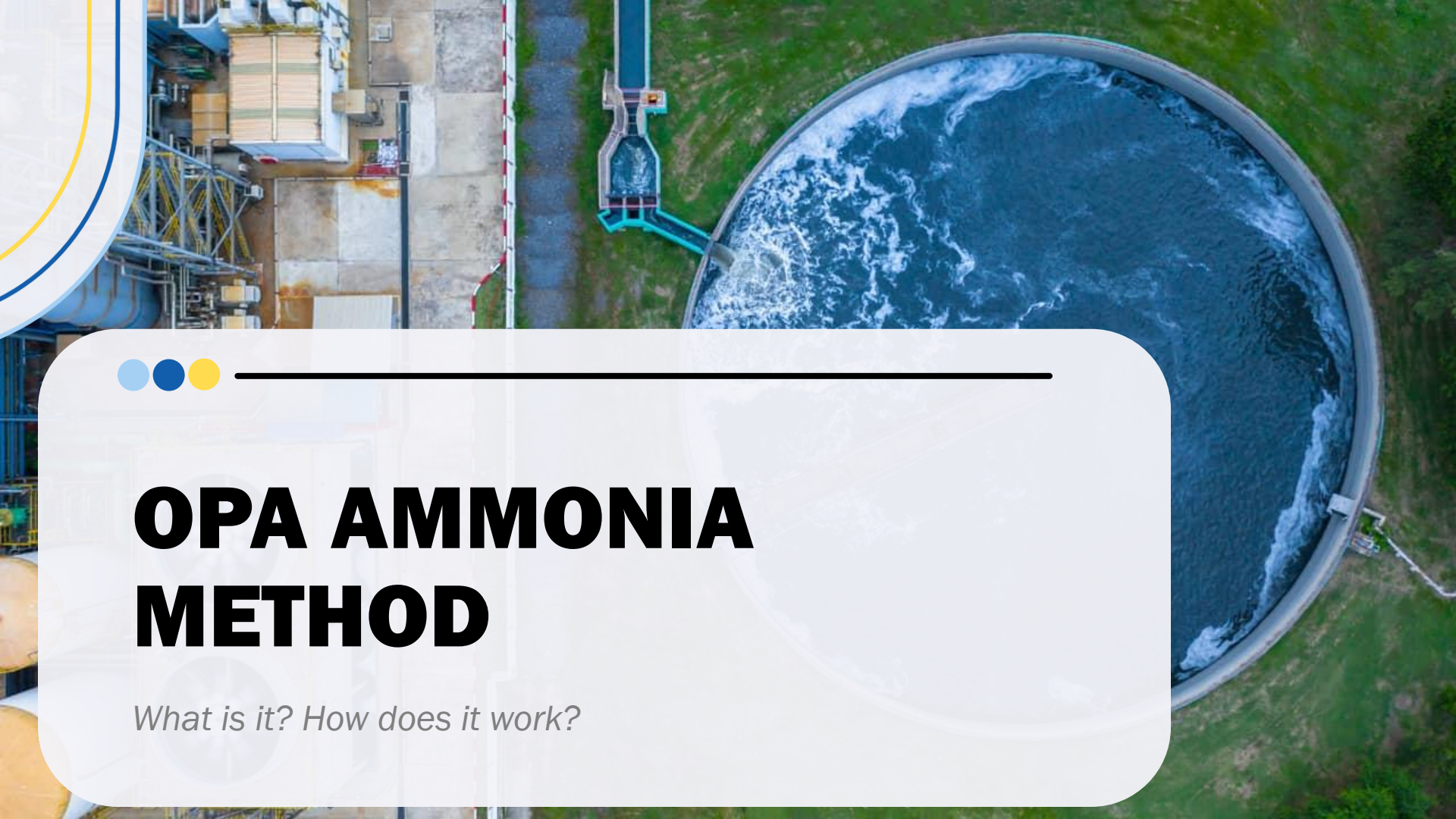
FIA Lab

HELLO!

My name is Dr. Ilkka Lähdesmäki

Chief Scientist

You can find me at ilkka@flowinjection.com

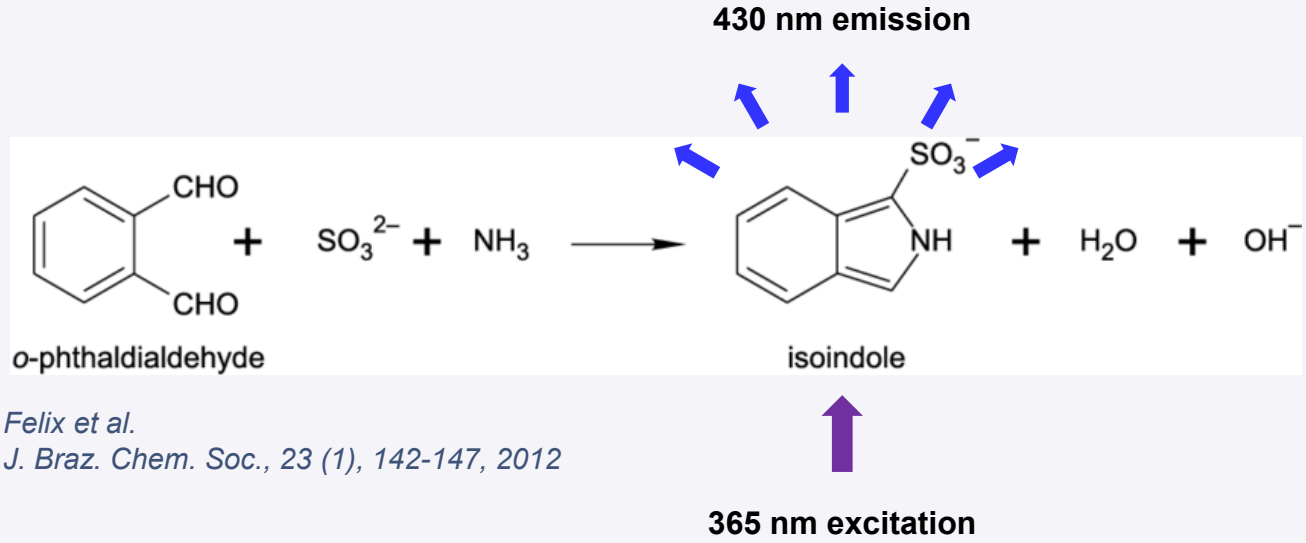


OPA AMMONIA METHOD

What is it? How does it work?

OPA CHEMISTRY

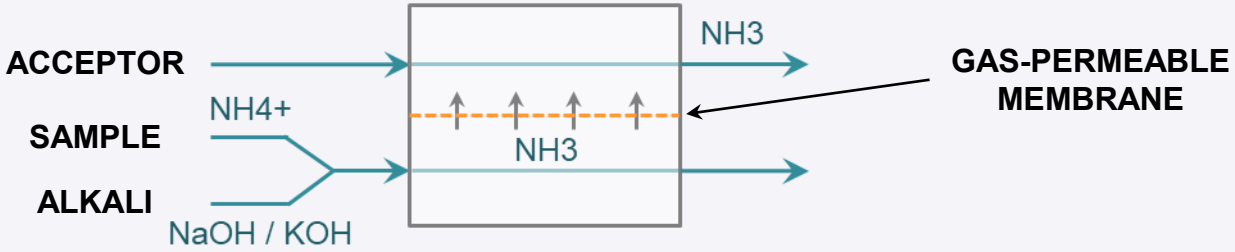
- o-phthalaldehyde reacts with NH3, forming a fluorescent product



Felix et al.
J. Braz. Chem. Soc., 23 (1), 142-147, 2012

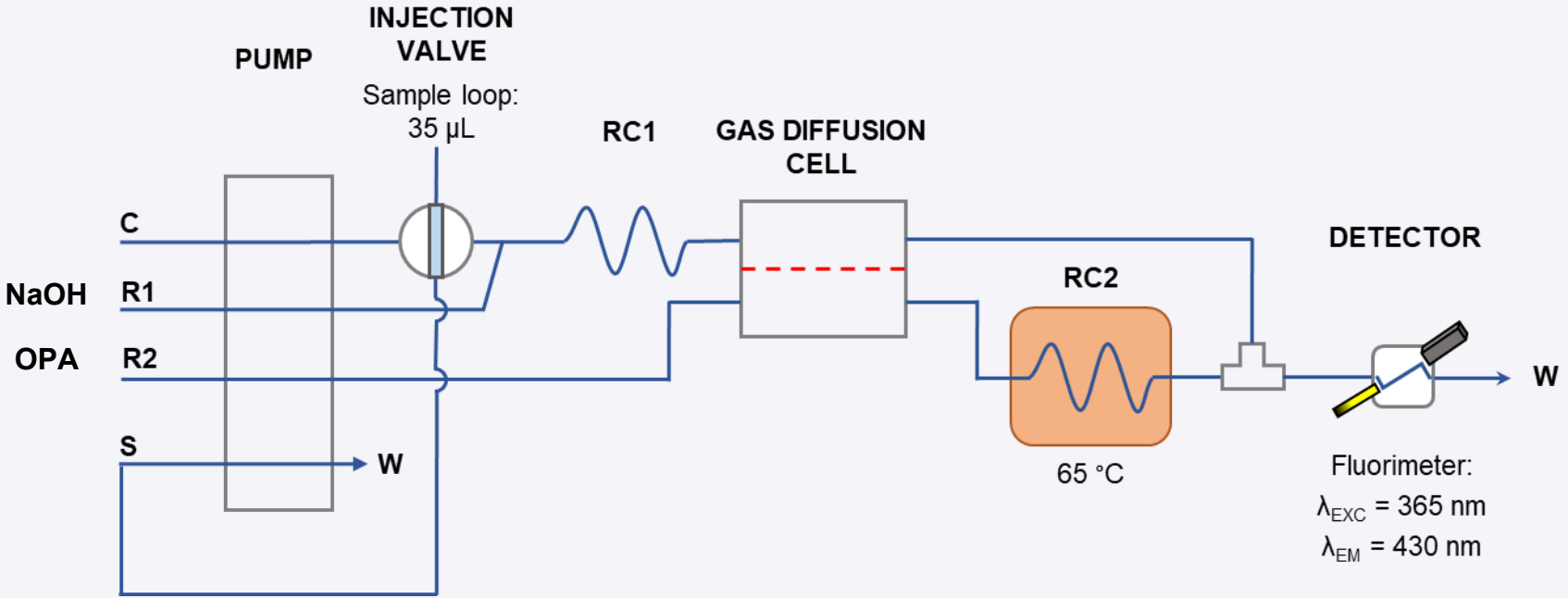
GAS DIFFUSION CONCEPT

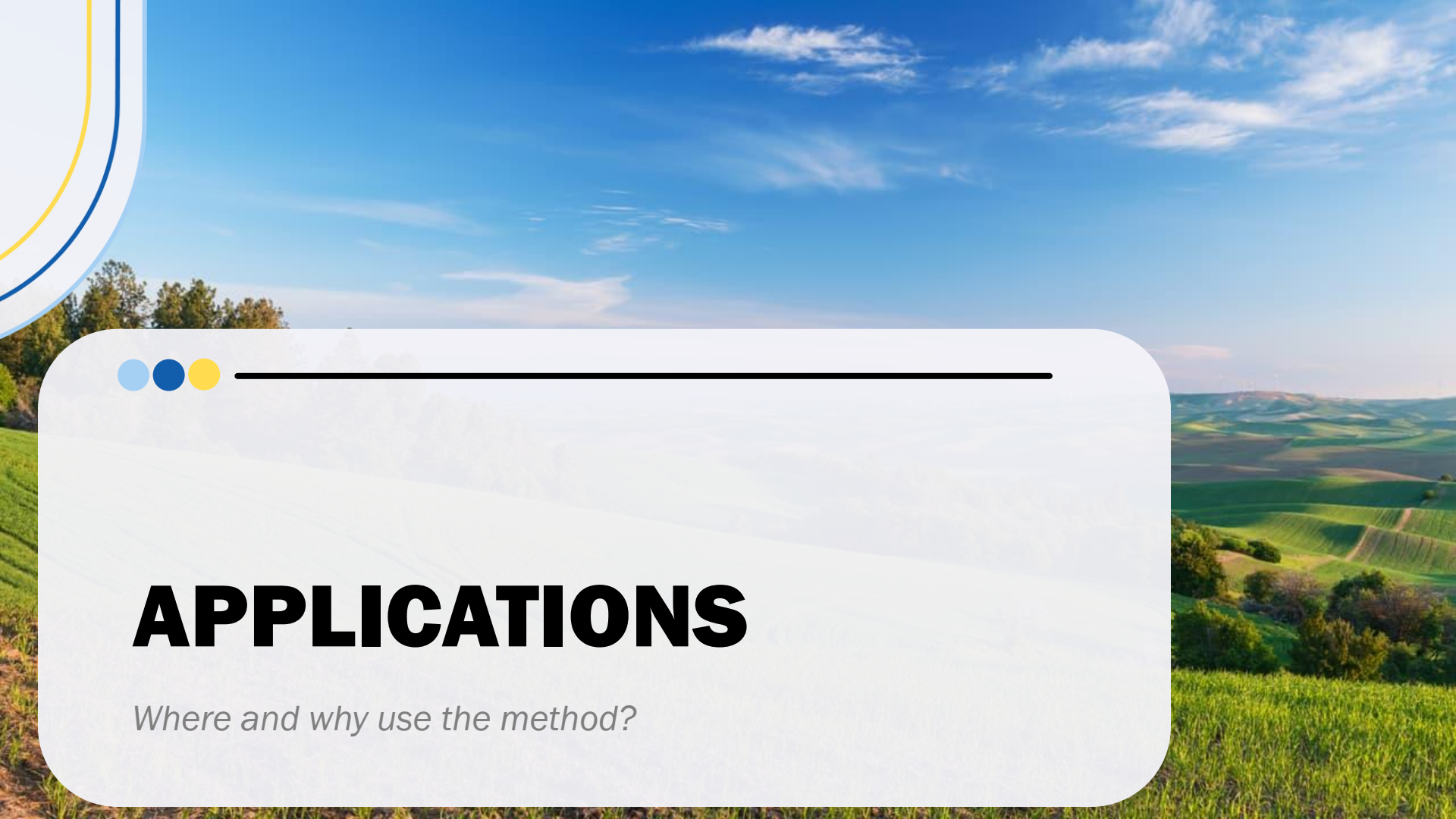
- Sandwich cell consisting of donor and acceptor compartments
- Gas-permeable membrane in between compartments
- Replaces distillation



- GD methods: FIAlab-100 (NH3), Timberline-Ammonia-001 (NH3), OIA-1677 (CN)

FIA SETUP FOR OPA METHOD





APPLICATIONS

Where and why use the method?

USES FOR OPA METHOD

- Formally, uses specified in 40 CFR Part 136
  - Ammonia
  - TKN (following manual digestion)
- Original method specifications:
  - Range 0.05 – 10 mg N/L
  - MDL 0.012 mg N/L

WHEN/WHY TO USE?

- No phenol
- Sensitive detection – wide dynamic range
- Fewer reagents compared to traditional phenate method
- Color reagent much more stable compared to traditional phenate method

PRACTICAL CONSIDERATIONS

Improvements from everyday use

COLOR REAGENT STABILITY

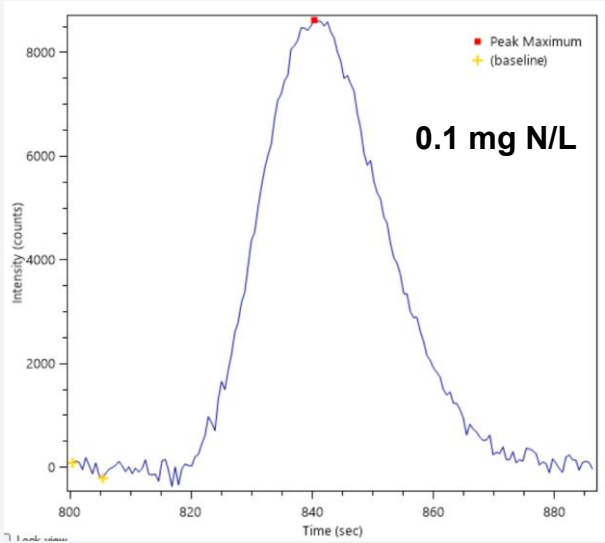
- The official FIAlab-100 method does not specify stability for the OPA reagent
- Initially, working instructions made for a stability of 4 weeks
- In practice, have noticed that even after 12 weeks the reagent is working very well
- No need to store in refrigerator

OPTIMIZED DETECTOR

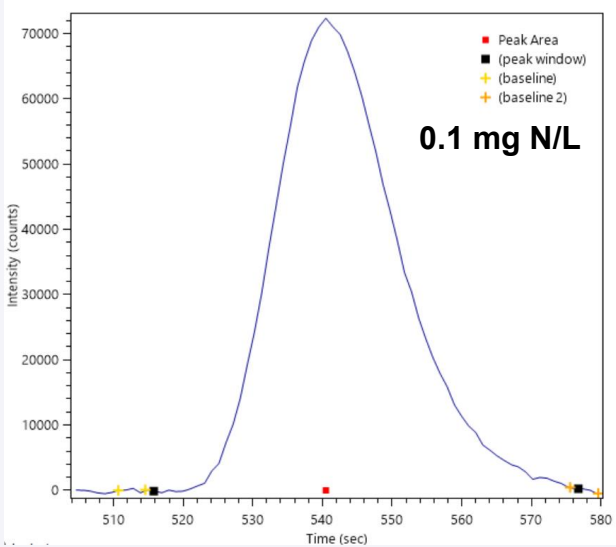
- The optical arrangement in the fluorescence detector was optimized for better performance
- Data collection parameters were set for optimal signal-to-noise

OPTIMIZED DETECTOR

- Optimized detector → lower detection capability



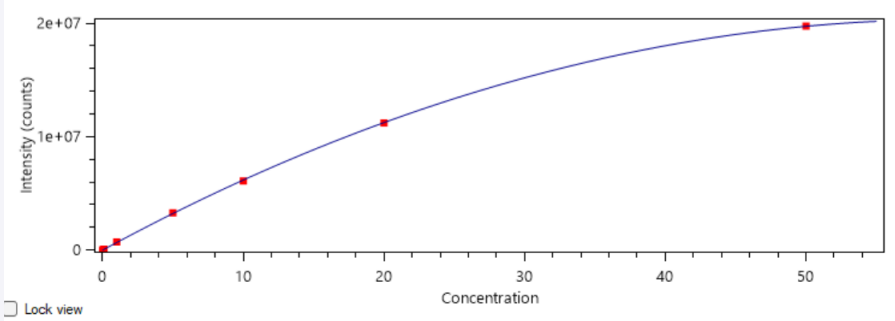
Original detector setup



Optimized detector setup

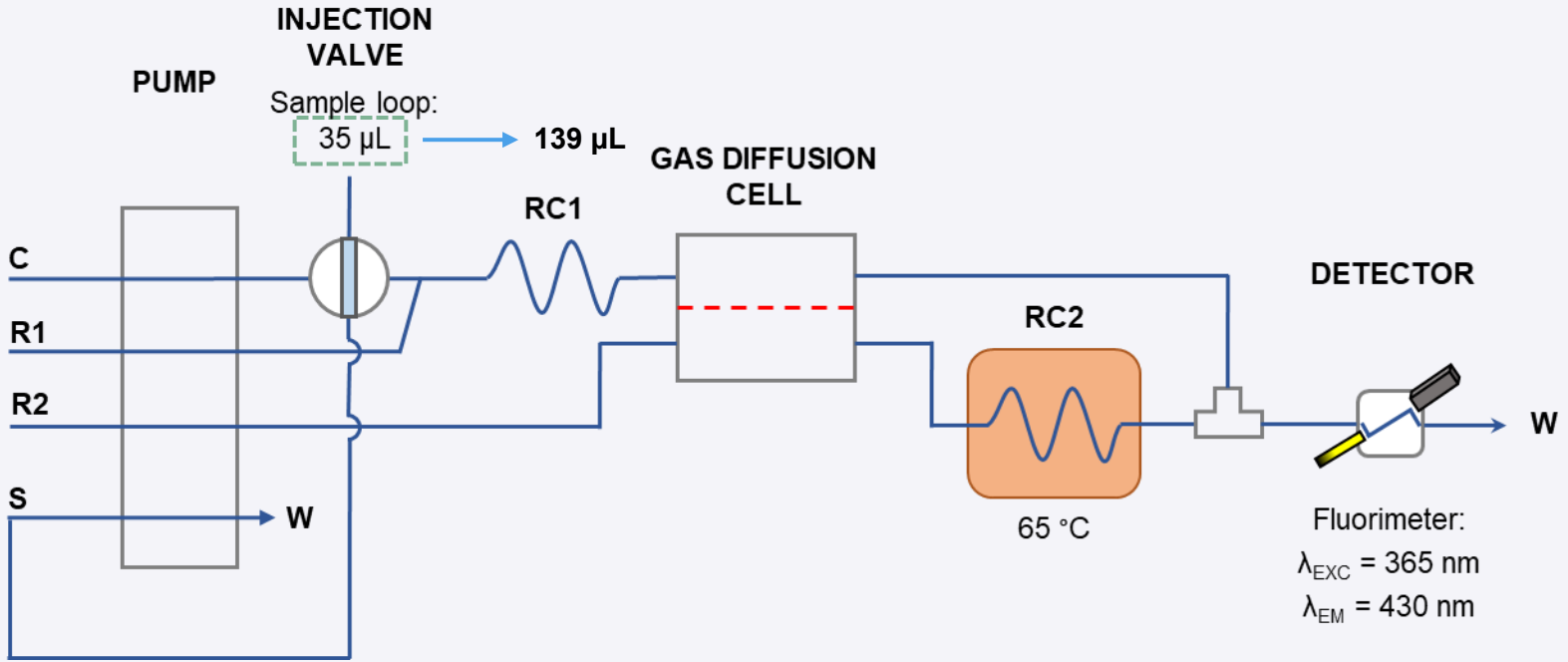
OPTIMIZED DETECTOR

- Optimized detector → **wide range 0.01-50 mg N/L (3.5 decades!)**

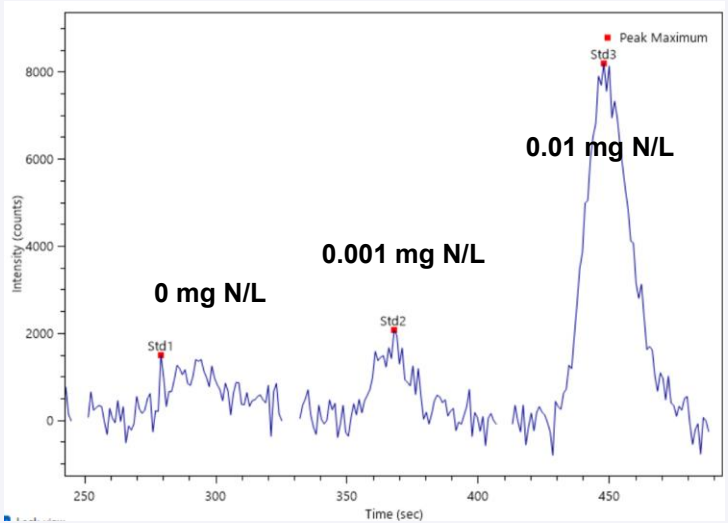


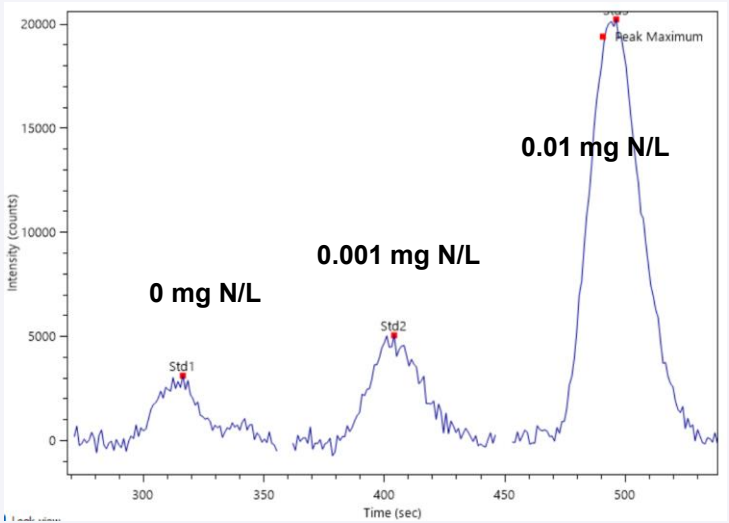
	Name	Peak Response	Known Concentratic	Calculated Concentratic	% Error	Peak Time	Enabled
▶	Std1	2183	0	0.0000	NA	2025-06-2...	☑
	Std2	3569	0.001	0.0021	105.54	2025-06-2...	☐
	Std3	8823	0.01	0.0099	1.48	2025-06-2...	☑
	Std4	72368	0.1	0.1042	4.21	2025-06-2...	☑
	Std5	707555	1	1.0557	5.57	2025-06-2...	☑
	Std6	3299526	5	5.1080	2.16	2025-06-2...	☑
	Std7	6095713	10	9.8430	1.57	2025-06-2...	☑
	Std8	11221211	20	19.9382	0.31	2025-06-2...	☑
	Std9	19756585	50	50.1299	0.26	2025-06-2...	☑

LOW LEVEL DETECTION



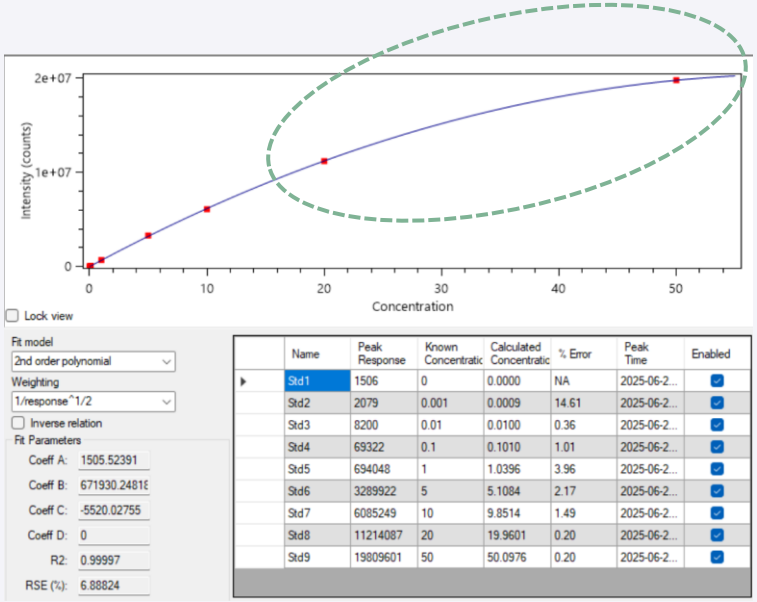
LOW LEVEL DETECTION





- Larger sample loop improves the low-end signals …

LOW LEVEL DETECTION



	Name	Peak Response	Known Concentratic	Calculated Concentratic	% Error	Peak Time	Enabled
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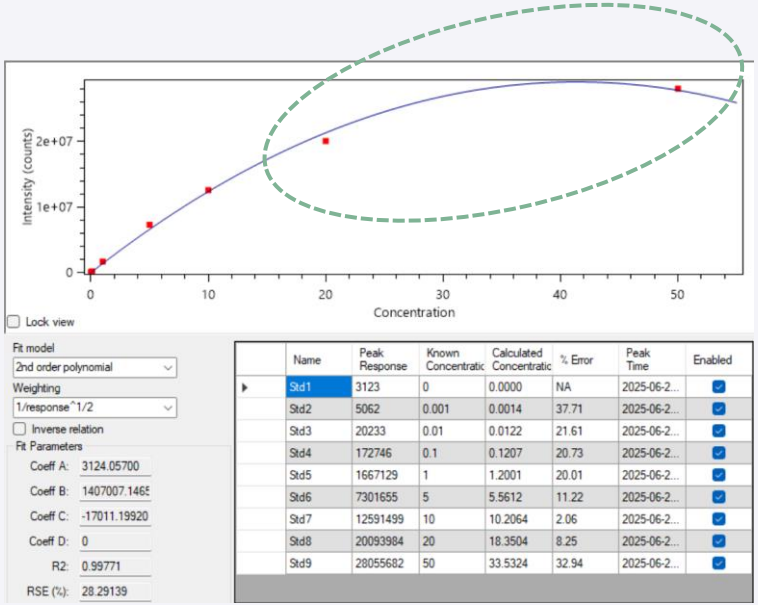
	Std2	2079	0.001	0.0009	14.61	2025-06-2...	☑
	Std3	8200	0.01	0.0100	0.36	2025-06-2...	☑
	Std4	69322	0.1	0.1010	1.01	2025-06-2...	☑
	Std5	694048	1	1.0396	3.96	2025-06-2...	☑
	Std6	3289922	5	5.1084	2.17	2025-06-2...	☑
	Std7	6085249	10	9.8514	1.49	2025-06-2...	☑
	Std8	11214087	20	19.9601	0.20	2025-06-2...	☑
	Std9	19809601	50	50.0976	0.20	2025-06-2...	☑



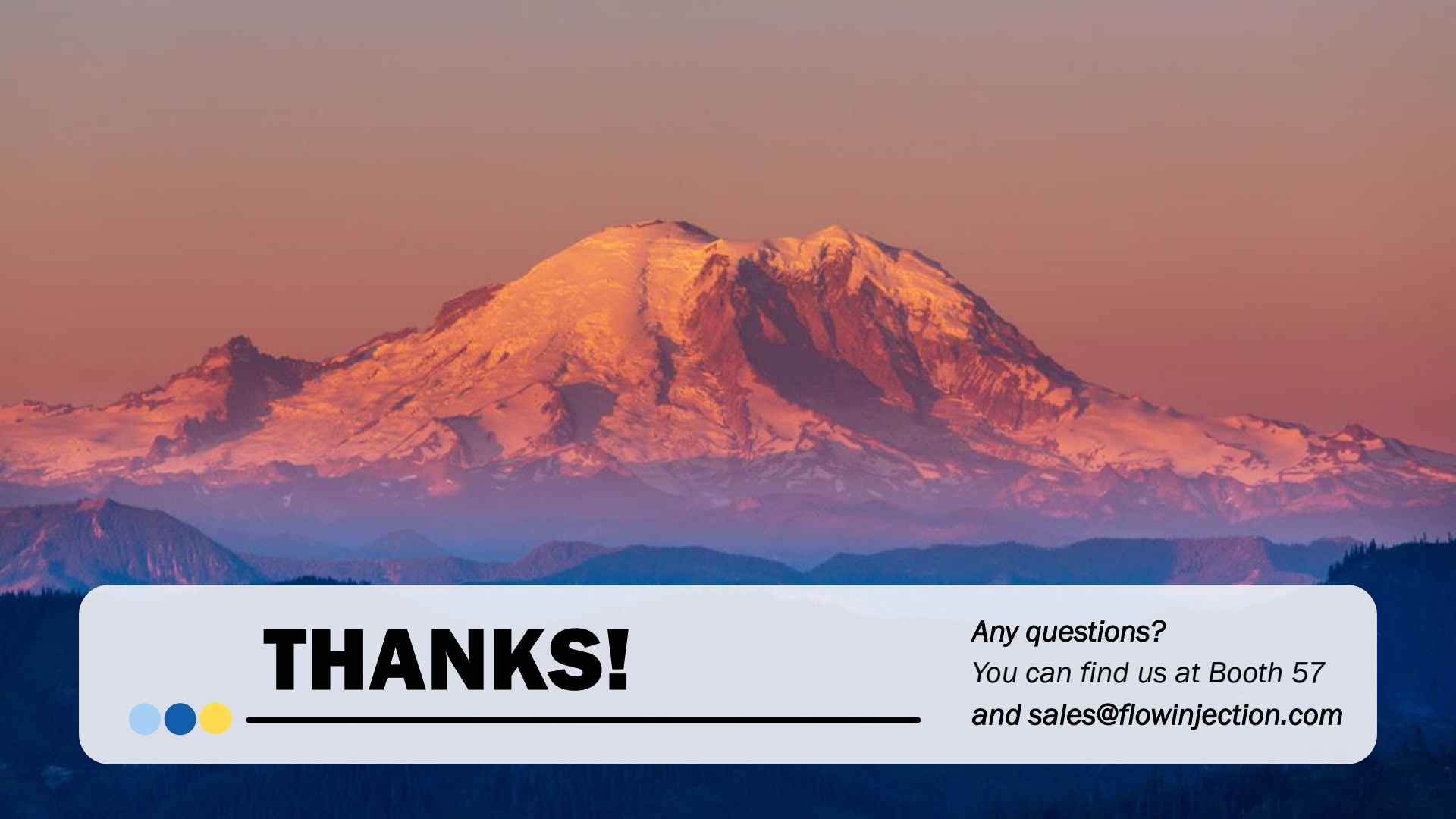
	Name	Peak Response	Known Concentratic	Calculated Concentratic	% Error	Peak Time	Enabled
▸	Std1	3123	0	0.0000	NA	2025-06-2...	☑
	Std2	5062	0.001	0.0014	37.71	2025-06-2...	☑
	Std3	20233	0.01	0.0122	21.61	2025-06-2...	☑
	Std4	172746	0.1	0.1207	20.73	2025-06-2...	☑
	Std5	1667129	1	1.2001	20.01	2025-06-2...	☑
	Std6	7301655	5	5.5612	11.22	2025-06-2...	☑
	Std7	12591499	10	10.2064	2.06	2025-06-2...	☑
	Std8	20093984	20	18.3504	8.25	2025-06-2...	☑
	Std9	28055682	50	33.5324	32.94	2025-06-2...	☑

- ... but degrades the high end of the calibration curve

CONCLUSIONS

CONCLUSIONS

- The OPA method can be used for ammonia and TKN
- The OPA color reagent has exceptional stability
- With optimized detector + data collection setup, can cover a range of 3.5 decades



THANKS!

Any questions?

You can find us at Booth 57

and sales@flowinjection.com