Automation of Inorganic Assays with Flow Injection Analysis (FIA)

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HELLO!

I am Dr. Ilkka Lahdesmaki

Chief Scientist

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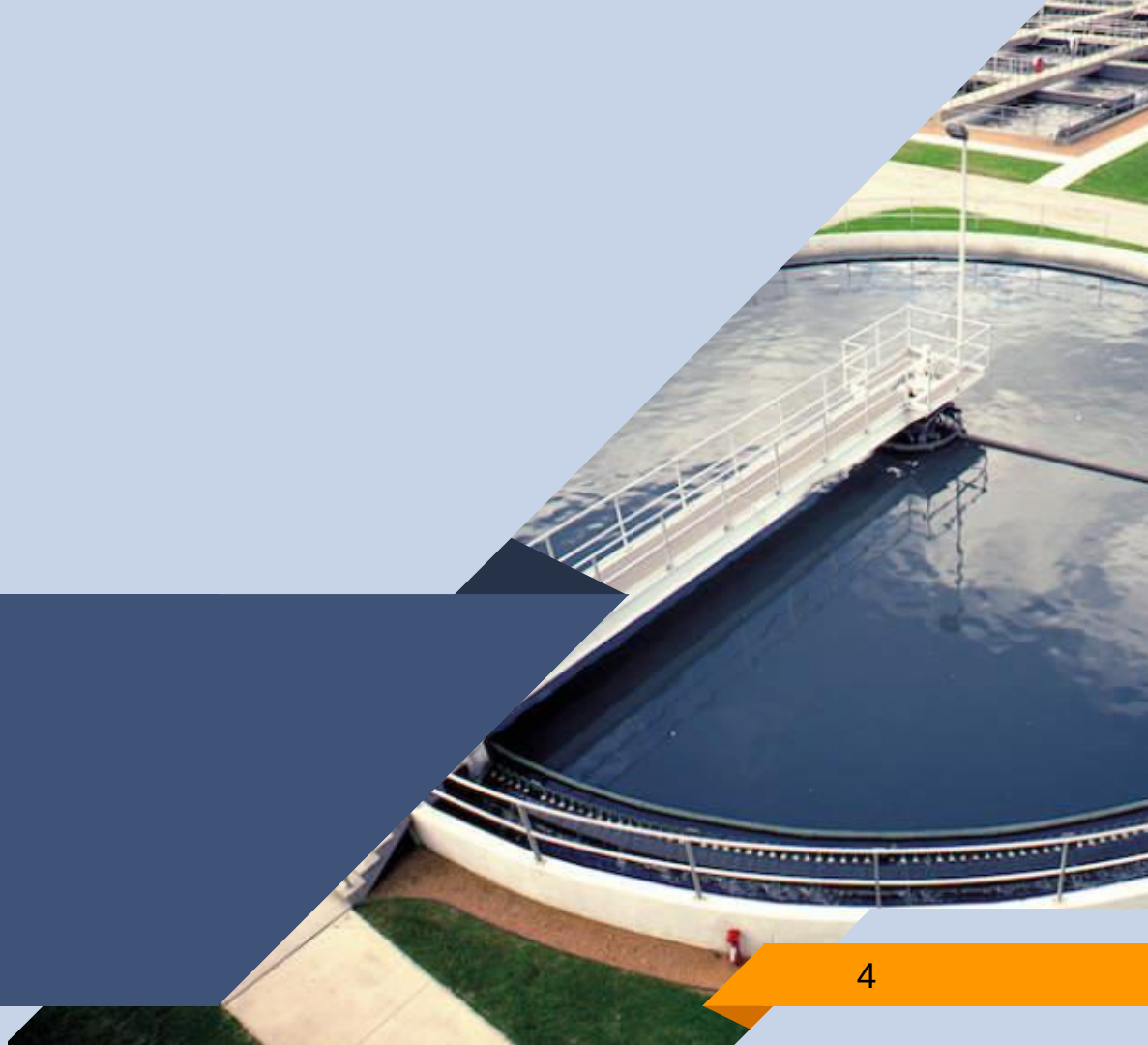
FLOW ANALYSIS

Short History



HISTORY - BENCHTOP

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- Mix sample w/ color-generating reagents
  - Dosing: manual (pipettes)
  - Containers: traditional glassware (beakers, flasks, tubes)
  - Incubation: ambient, water bath
  - Measurement: benchtop spectrometer + cuvette
- Time-consuming, laborious

HISTORY - SEGMENTED FLOW ANALYSIS

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- Automated colorimetric assays
- Invented by Dr. Skeggs in 1957
- Commercialized by Technicon Corp. in the 50's and 60's
- Mix sample w/ color-generating reagents
  - Dosing: peristaltic pump & autosampler
  - Containers: glass coils & tubing, **segmentation w/ air bubbles**
  - Incubation: flow-through heater
  - Measurement: flow-through cuvette/flow cell

HISTORY - SEGMENTED FLOW ANALYSIS

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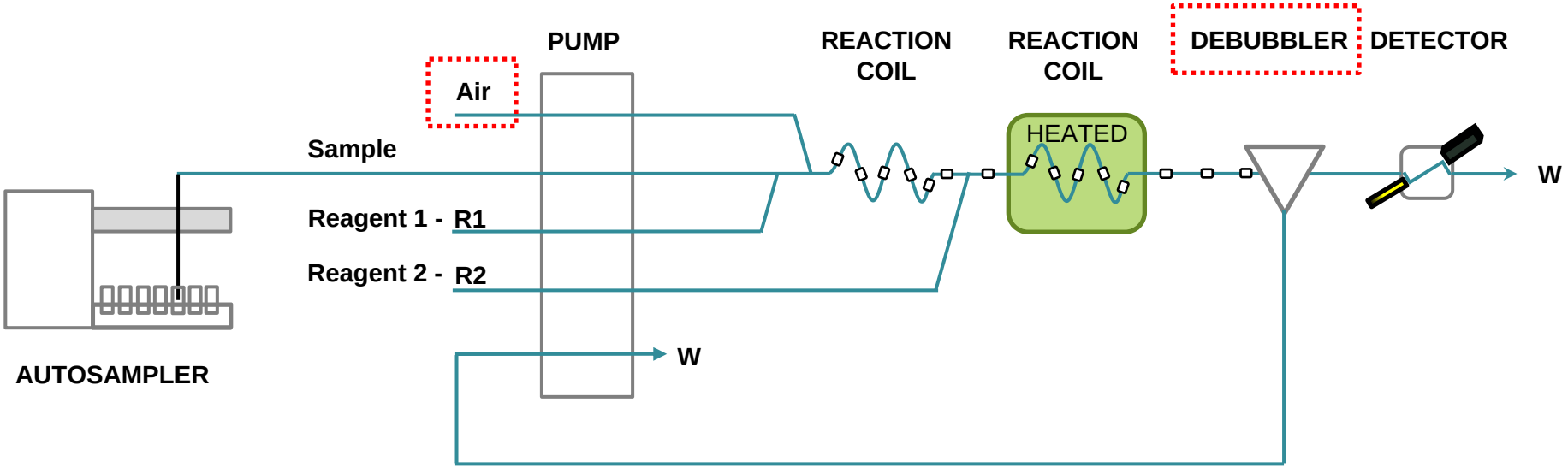

HISTORY - FLOW INJECTION ANALYSIS

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- Invented by Dr. Ruzicka & Dr. Hansen in 1974
- Commercialized by Bifok AB (later Tecator) in the 70's
- Mix sample w/ color-generating reagents
  - Dosing: peristaltic pump, **injection valve**
  - Containers: Teflon coils & tubing, **no air segmentation**
  - Incubation: flow-through heater
  - Measurement: flow-through cuvette/flow cell

HISTORY - FLOW INJECTION ANALYSIS

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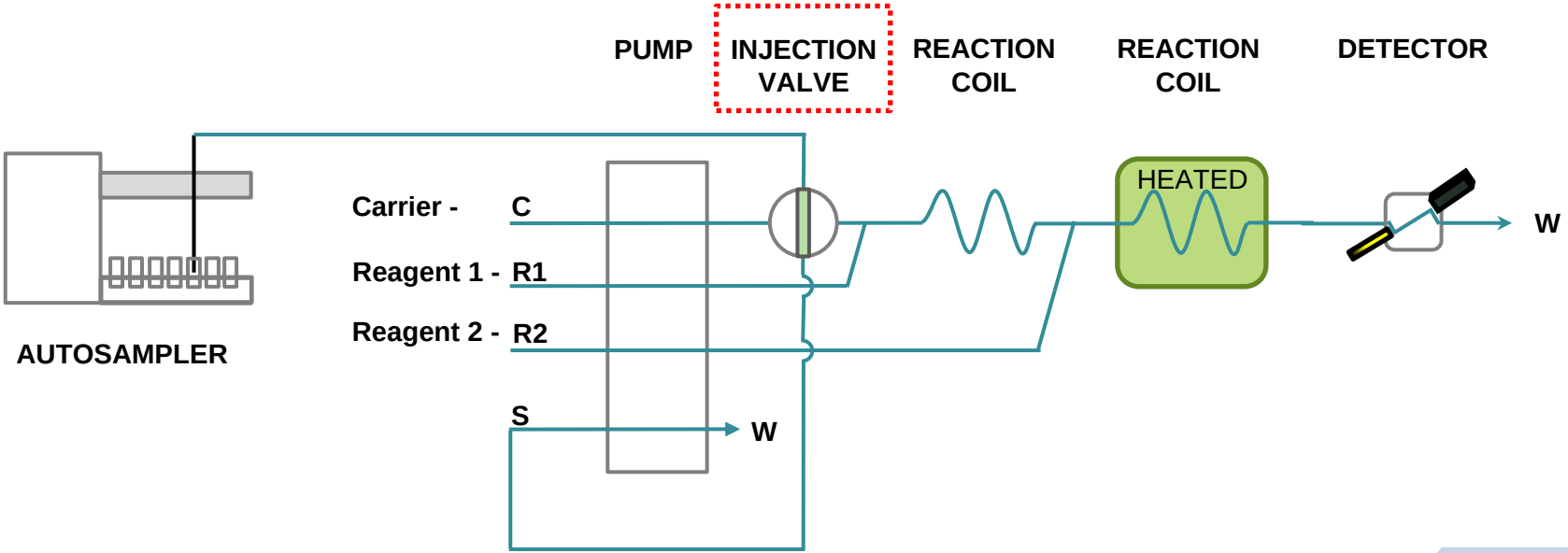

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OPERATION PRINCIPLE

How does FIA work?

FLOW INJECTION ANALYSIS

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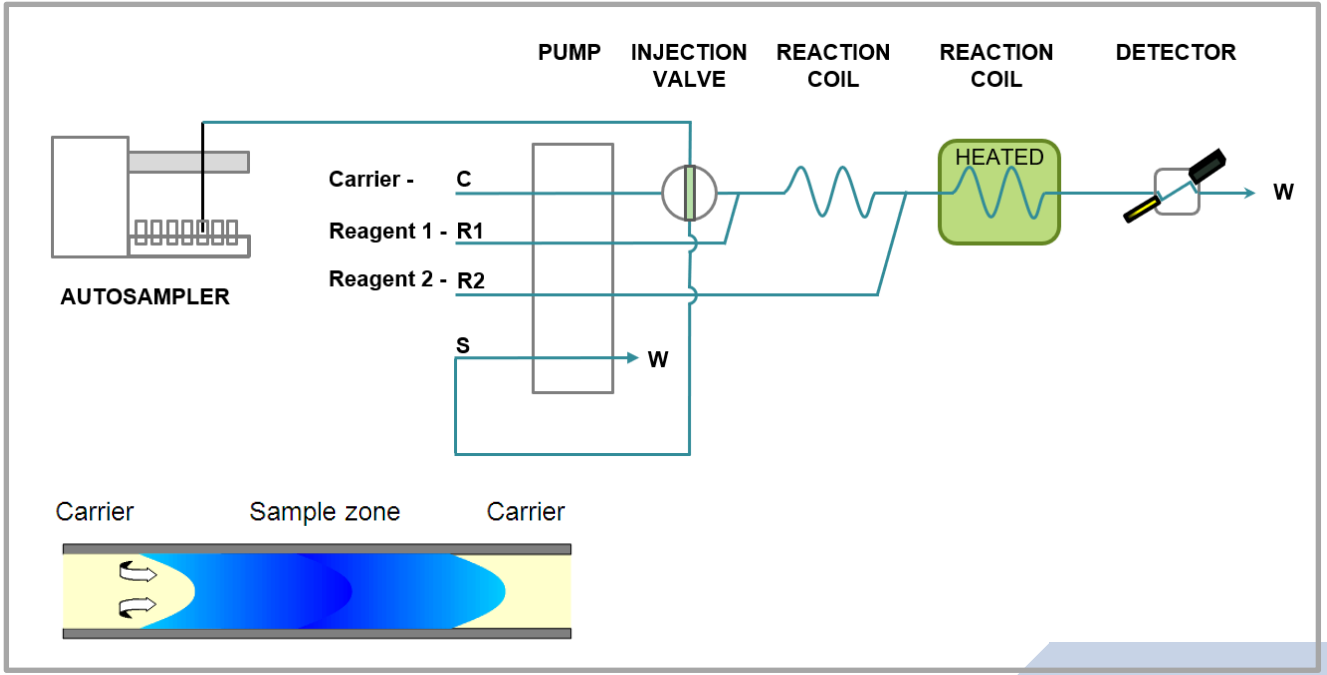
Principles of FIA

- Precise injection
- Controlled transit time
- Controlled dispersion



FLOW INJECTION ANALYSIS

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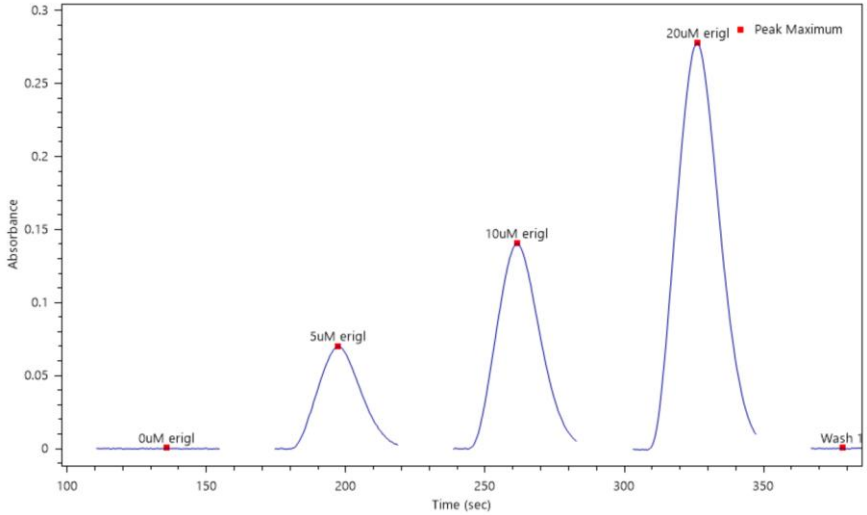
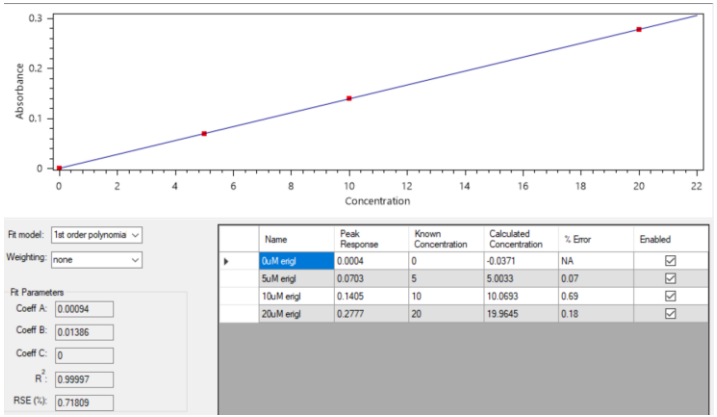
Practical outcome of controlled dispersion

- Signals in shape of a **peak** (like LC, IC)





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WHY DID FIA BECOME ADOPTED?

What differentiates FIA from SFA?

WHY DID FIA BECOME ADOPTED?

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- Dr. Ruzicka & Dr. Hansen offered FIA concept to Technicon
- Technicon's final response, as per Dr. Ruzicka:

"In brief, Technicon's patent attorney, after consulting other patent specialists, pronounced FIA non-patentable. The technical director deemed the technique impractical."*

*Dr. Ruzicka's FIA website (flowinjectiontutorial.com)

WHY DID FIA BECOME ADOPTED?

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- So why did FIA survive?
  - Speed: up to 300 samples / h
  - Speed: calibration done in ~5 min from start
  - Carryover: **complete** return to baseline b/w injections
  - Use of plastic (Teflon) capillary tubing

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PRACTICAL BENEFITS

How does FIA benefit operation and profitability of laboratories?

PRACTICAL BENEFITS

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- Increased speed – sample throughput - “Time is money”
  - High-throughput laboratories – 100s - 1000s of samples / day
  - Every second counts for sample processing time
  - Record ortho-P: 8 sec / sample
- Increased speed – calibration
  - Sample throughput is not the only important speed metric
  - **Calibration speed** greatly matters as well
  - Calibration done in ~5-10 min from run start (method dependent)

PRACTICAL BENEFITS

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- Decreased labor
  - Manual measurements require large staff investment
  - Automation → staff can be assigned to other tasks
- Increased efficiency
  - Automation → staff can multitask while instrument is running
- Handle challenging sample matrices (acid, color)
  - Membrane units (gas diffusion, dialysis)

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PRACTICAL INSTRUMENT & METHOD DESIGN

How to design them so that they work for **practical** applications on **real** samples

CASE STUDY: NH3/TKN BY OPA METHOD

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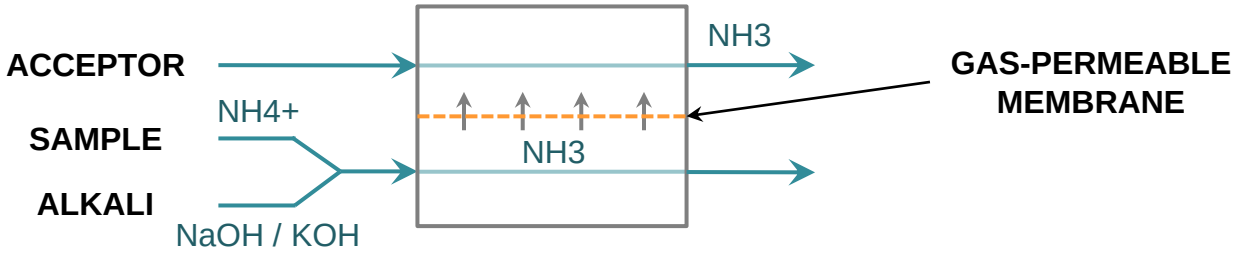
- Use of gas diffusion to replace distillation



CASE STUDY: NH3/TKN BY OPA METHOD

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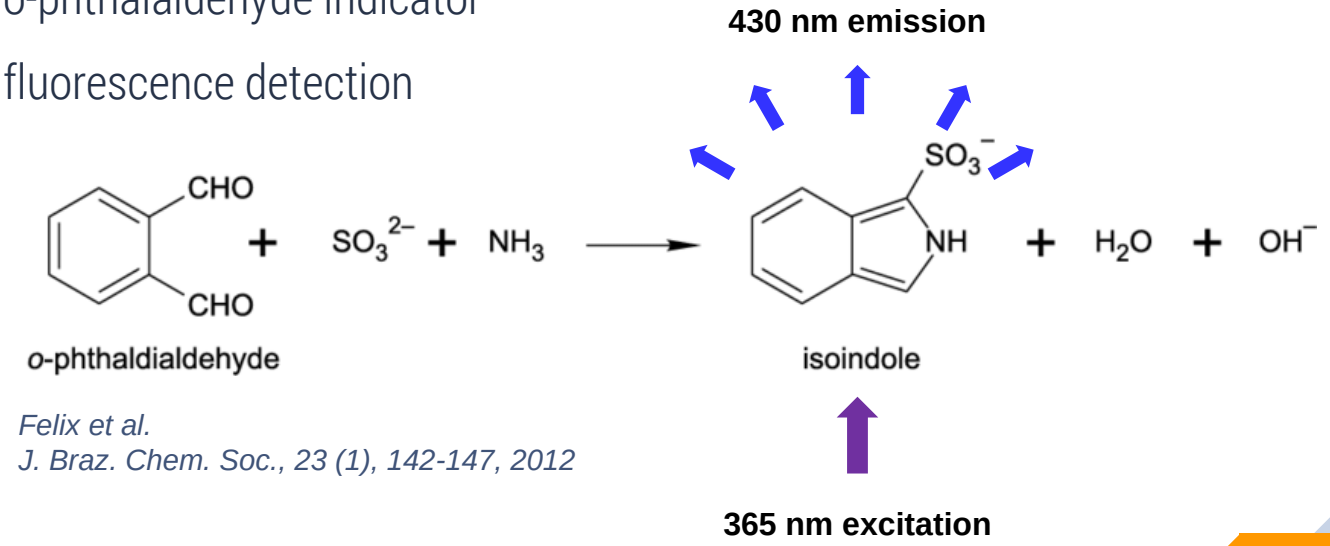
Based on:

- o-phthalaldehyde indicator
- fluorescence detection



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

CASE STUDY: NH3/TKN BY OPA METHOD

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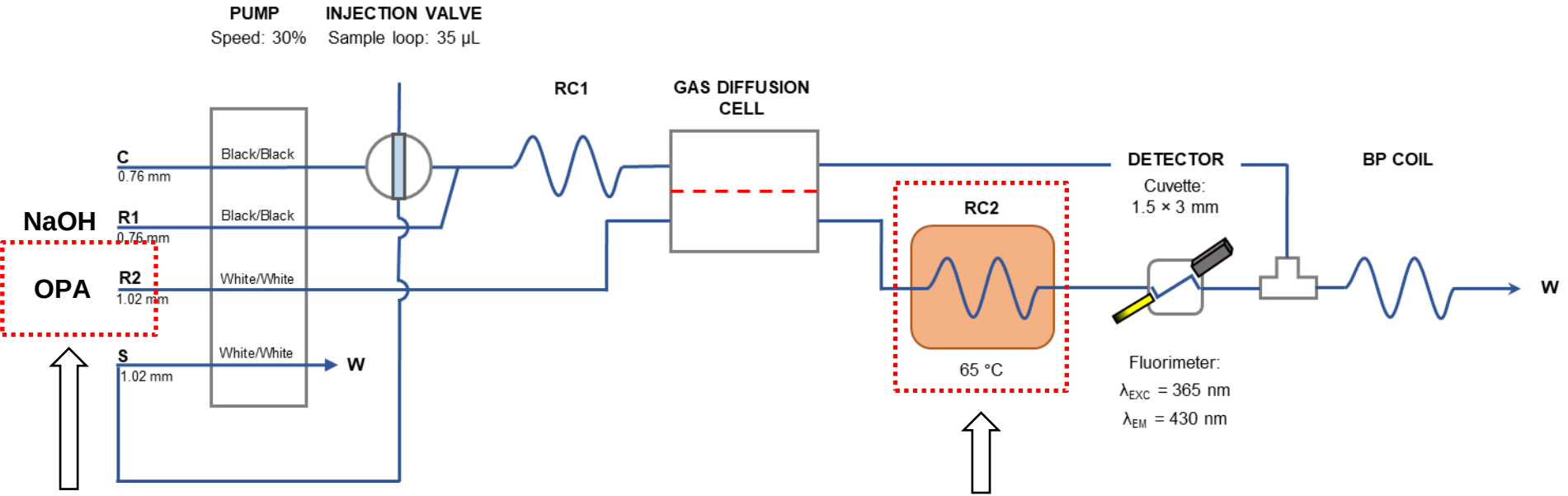

CASE STUDY: NH3/TKN BY OPA METHOD

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Choice of acceptor solution

- Traditionally, NH_3 acceptor is an acid solution
- Here, an alkaline solution (borate buffer) was selected
- Why: **simplification** of manifold, ability to implement on a **compact instrument**

Choice of heater coil length

- OPA reaction is slow ⟶ technically, would require a long heated coil
- Here, a relatively short (~70 in) coil was selected
- Why: **speed of calibration**

CASE STUDY: NH3/TKN BY OPA METHOD

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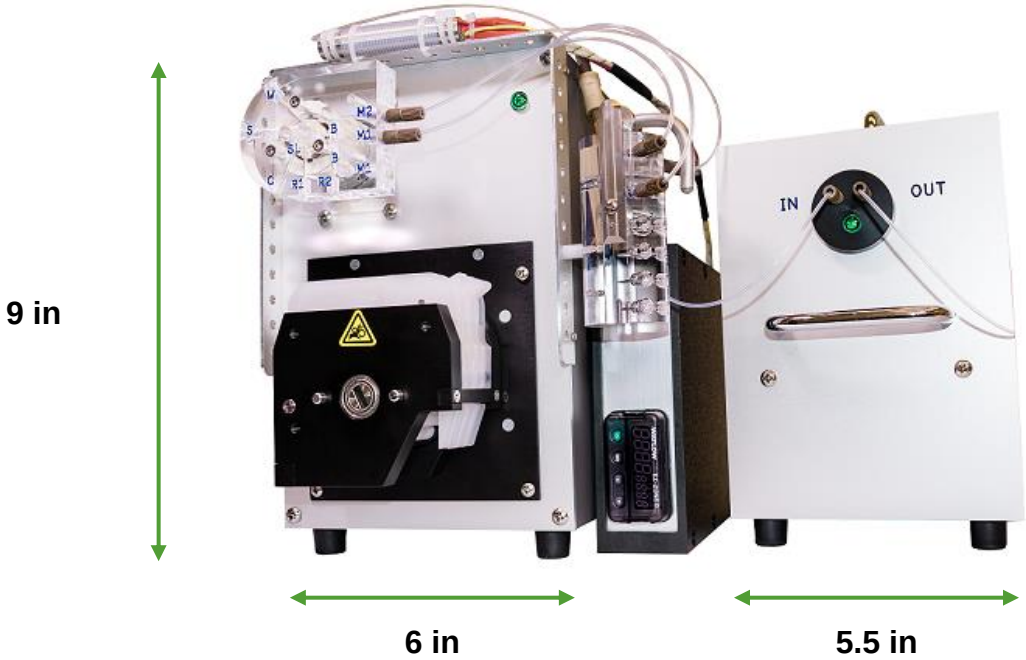

Pictured:

FIAlyzer-1000 with PMT Detector

CASE STUDY: NH3/TKN BY OPA METHOD

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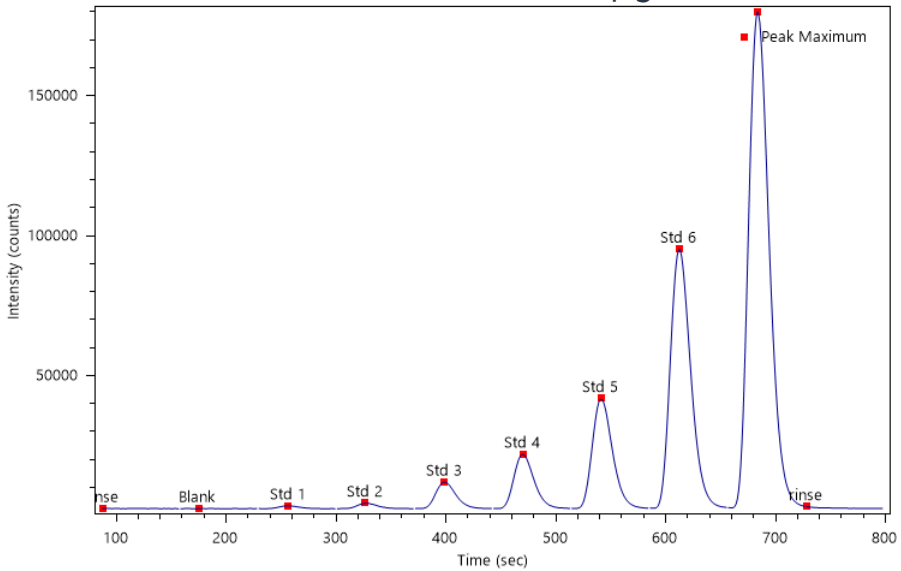
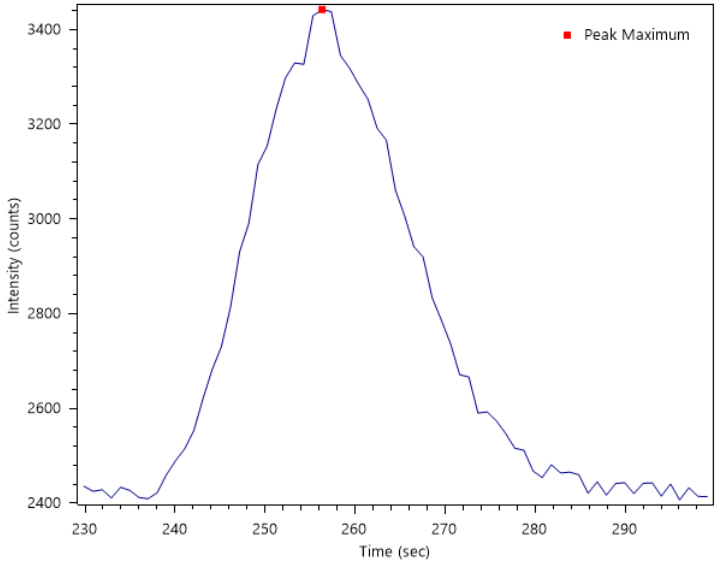
Calibration 50 – 10,000 µg N / L



Single injection 50 µg N / L



CASE STUDY: NH3/TKN BY OPA METHOD

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Detection Limit	12 µg N/L	
Reporting Limit	50 µg N/L	
Range Upper Limit	10 000 µg N/L	
Spike recovery	99.3%	POTW (Anaerobic digester sludge)
	97.3%	Industrial discharge (Food process)
	109%	Industrial discharge (Metal finish)
	96.9%	River water
	102%	POTW (Final effluent, pre-UV)
	105%	POTW (Primary clarifier effluent)
Throughput	50 samples / h	

CONCLUSIONS

"Theory guides, experiment decides."

-- Dr. Izaak Maurits (Piet) Kolthoff

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THANKS!

Any questions?

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