Automation of Inorganic Assays with Flow Injection Analysis (FIA)

FIA Lab

Fluidics Intelligently Automated

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

Table of Contents

- Short history of Flow Analysis
- Operation principle of FIA
- Why did FIA become adopted?
- Practical benefits of FIA
- Practical instrument / method design – USEPA FIAlab 100 method
- Conclusions and questions

FIA Lab

Fluidics Intelligently Automated

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

FIA Lab

Fluidics Intelligently Automated

HELLO!

I am Dr. Ilkka Lahdesmaki

Chief Scientist

You can find me at ilkka@flowinjection.com

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

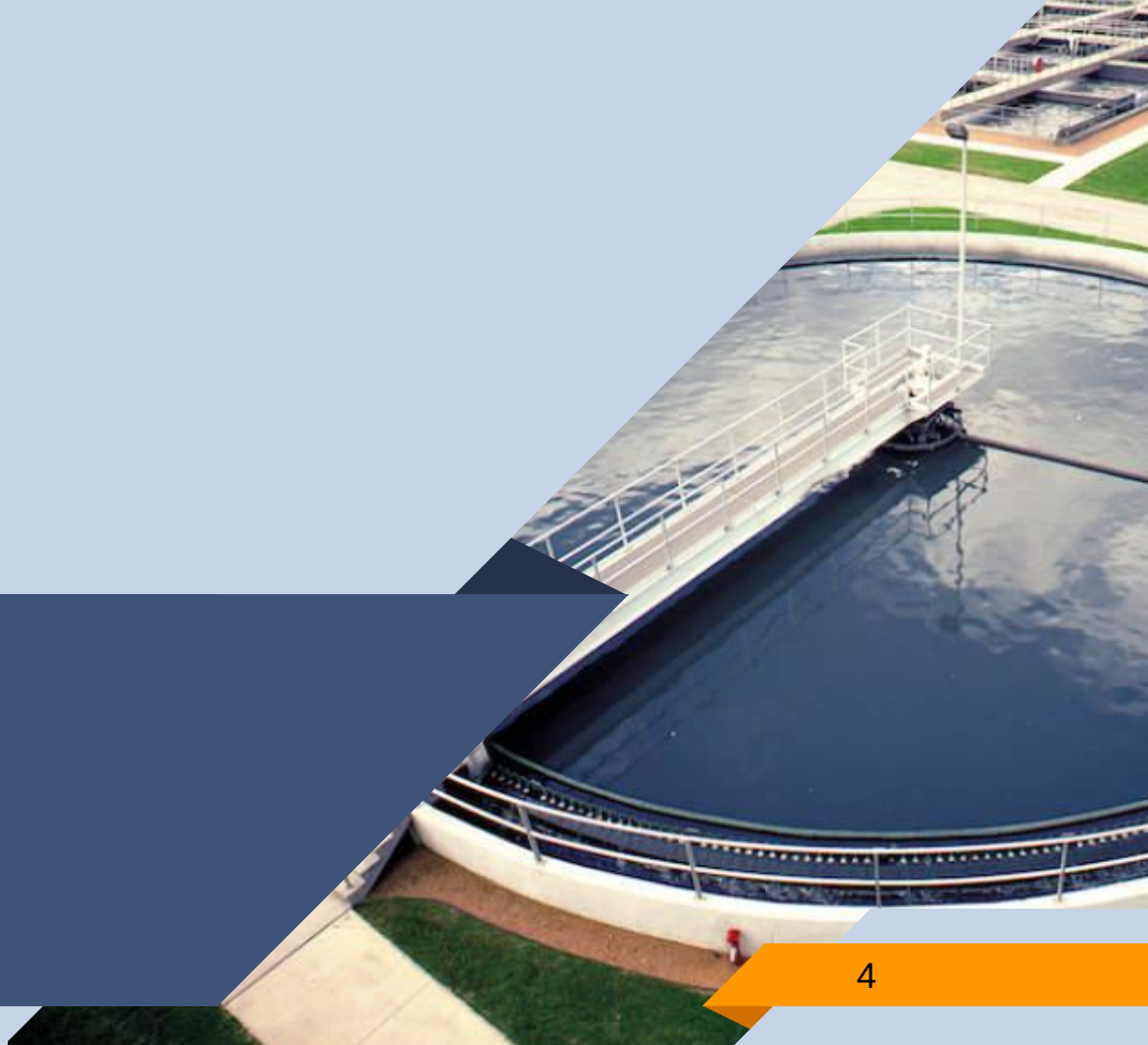
FIA Lab

Fluidics Intelligently Automated

1

FLOW ANALYSIS

Short History



Copyright 2018. FIAlab Instruments, INC. All rights reserved.

HISTORY - BENCHTOP

FIA Lab

Fluidics Intelligently Automated

- 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

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

HISTORY - SEGMENTED FLOW ANALYSIS

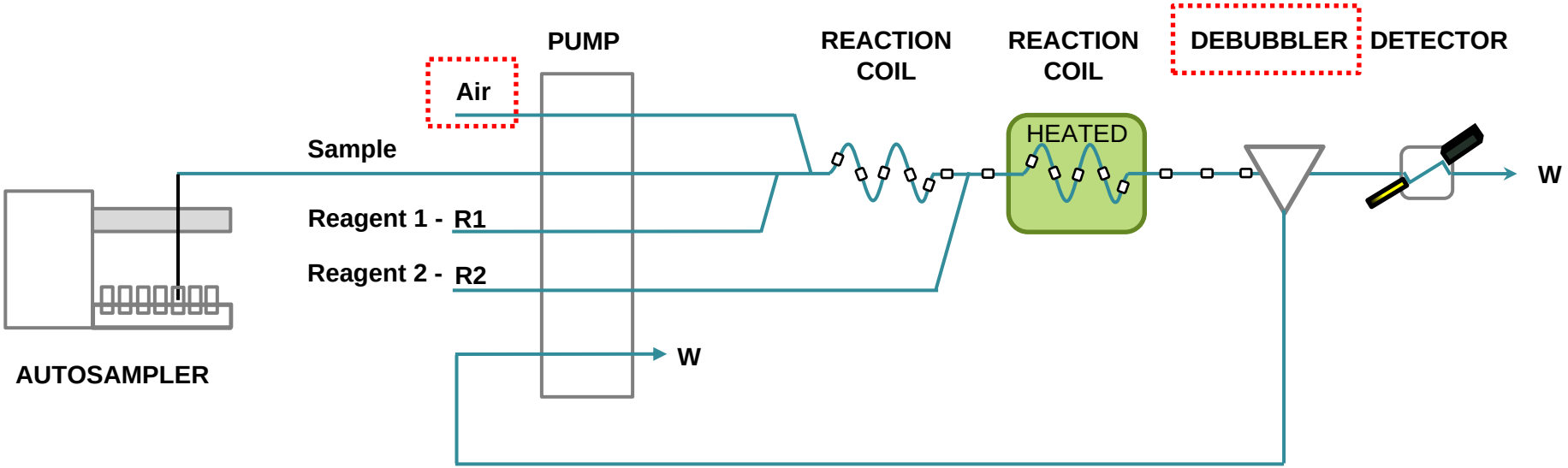
Fluidics Intelligently Automated

- Automated colorimetric assays
- Invented by Dr. Skeggs in 1957
- Commercialized by Technicon Corp. in the 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

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

HISTORY - SEGMENTED FLOW ANALYSIS

FIA Lab
Fluidics Intelligently Automated



Copyright 2018. FIAlab Instruments, INC. All rights reserved.

HISTORY - FLOW INJECTION ANALYSIS

FIA Lab

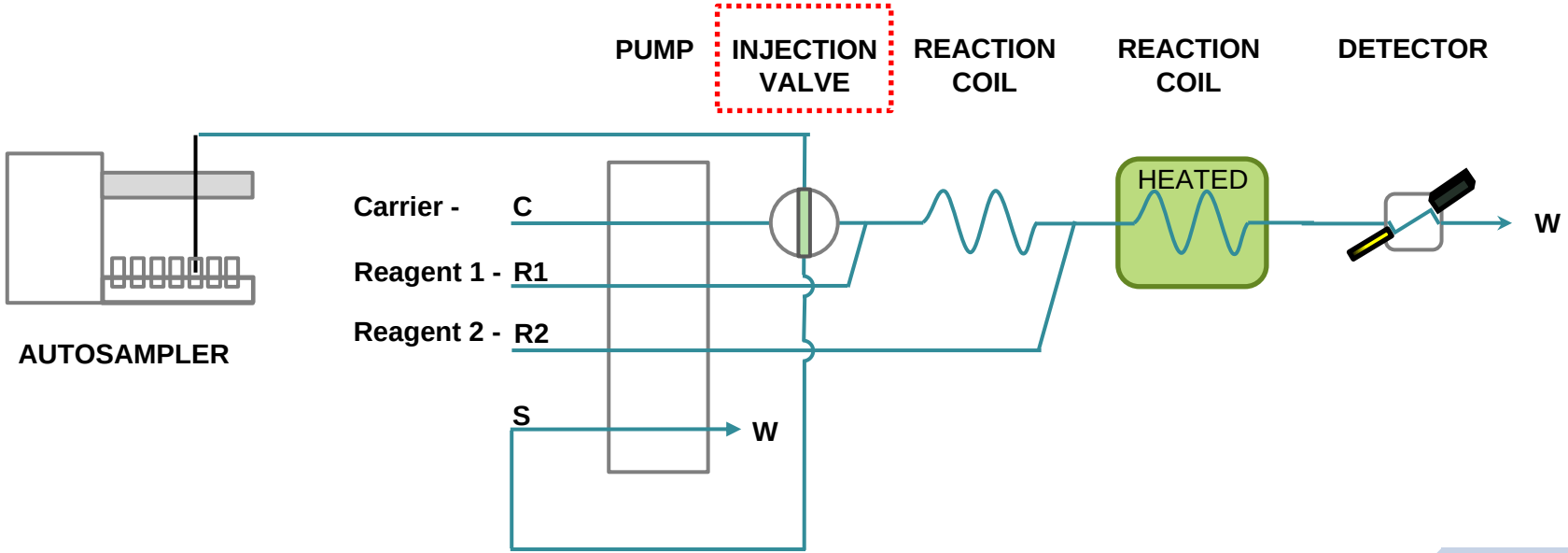
Fluidics Intelligently Automated

- 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

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

HISTORY - FLOW INJECTION ANALYSIS

FIAlab
Fluidics Intelligently Automated



Copyright 2018. FIAlab Instruments, INC. All rights reserved.

FIA Lab

Fluidics Intelligently Automated

2

OPERATION PRINCIPLE

How does FIA work?

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

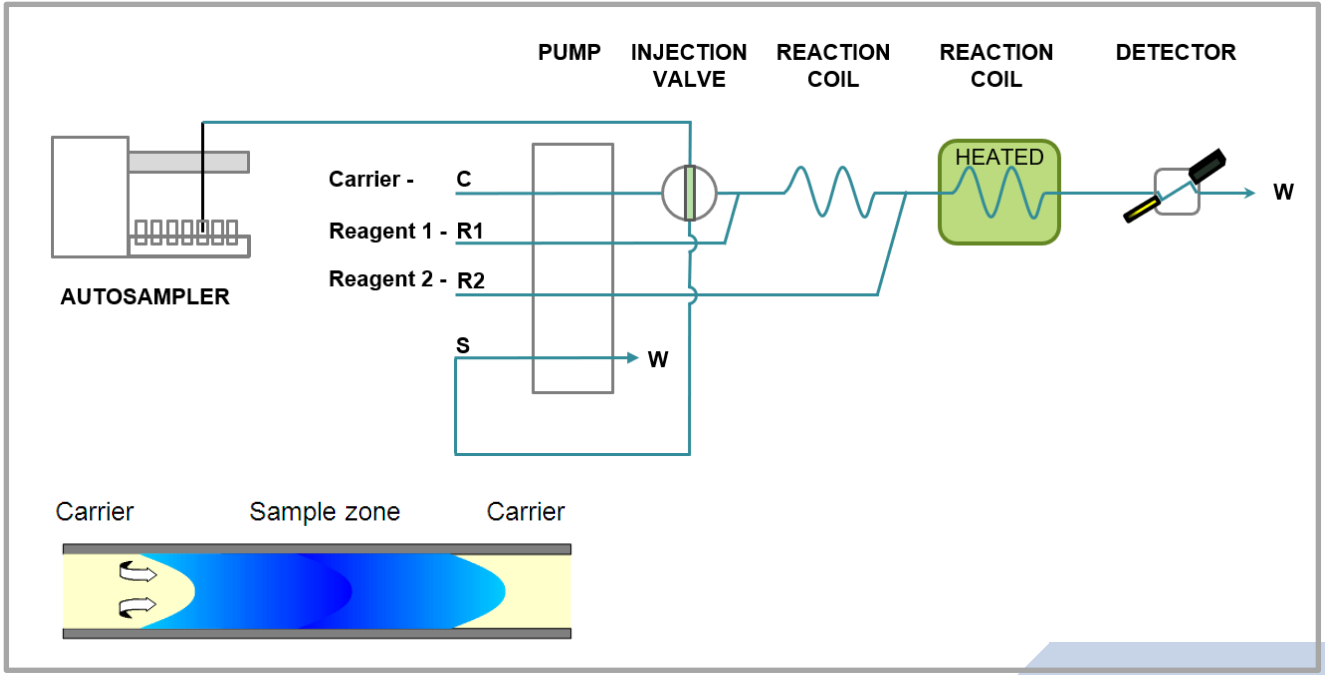
FLOW INJECTION ANALYSIS

FIA Lab

Fluidics Intelligently Automated

Principles of FIA

- Precise injection
- Controlled transit time
- Controlled dispersion



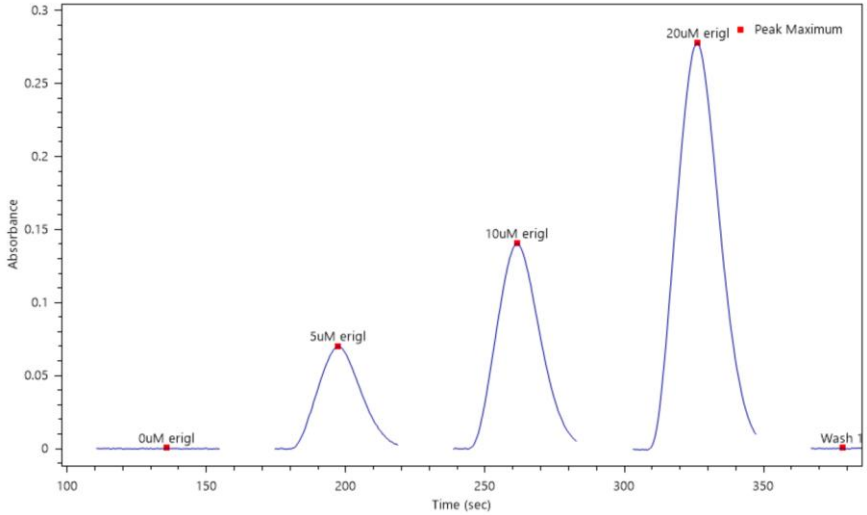
Copyright 2018. FIAlab Instruments, INC. All rights reserved.

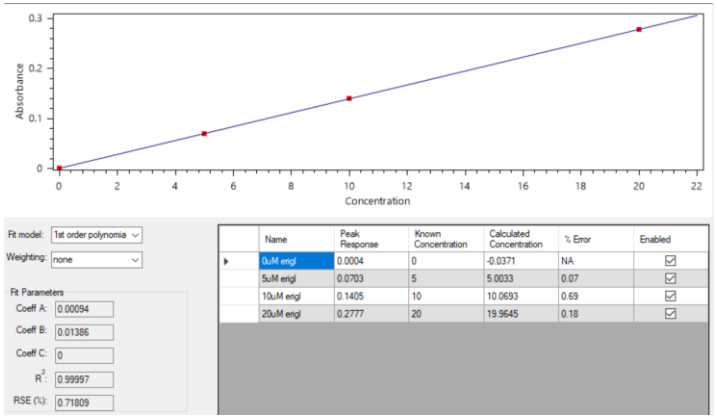
FLOW INJECTION ANALYSIS

FIA Lab
Fluidics Intelligently Automated

Practical outcome of controlled dispersion

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





Copyright 2018. FIAlab Instruments, INC. All rights reserved.

FIA Lab

Fluidics Intelligently Automated

3

WHY DID FIA BECOME ADOPTED?

What differentiates FIA from SFA?

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

WHY DID FIA BECOME ADOPTED?

FIA Lab

Fluidics Intelligently Automated

- 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)

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

WHY DID FIA BECOME ADOPTED?

FIA Lab

Fluidics Intelligently Automated

- 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

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

FIA Lab

Fluidics Intelligently Automated

4

PRACTICAL BENEFITS

How does FIA benefit operation and profitability of laboratories?

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

PRACTICAL BENEFITS

FIA Lab
Fluidics Intelligently Automated

- 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)

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

PRACTICAL BENEFITS

FIA Lab

Fluidics Intelligently Automated

- 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)

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

FIA Lab

Fluidics Intelligently Automated

5

PRACTICAL INSTRUMENT & METHOD DESIGN

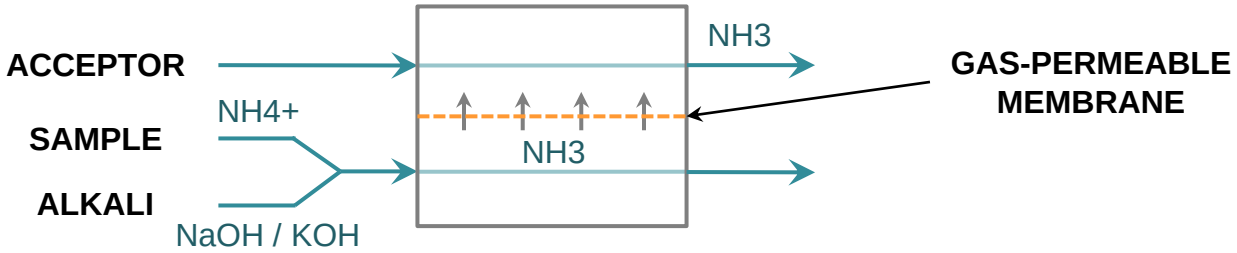
How to design them so that they work for **practical** applications on **real** samples

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CASE STUDY: NH3/TKN BY OPA METHOD

Fluidics Intelligently Automated

- Use of gas diffusion to replace distillation



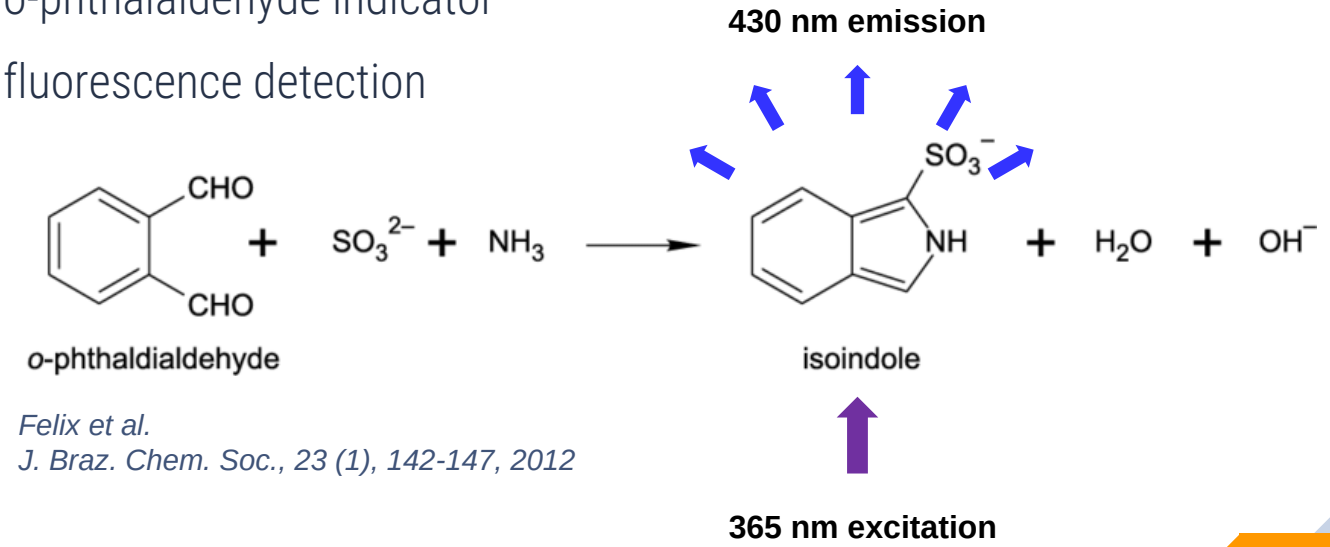
Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CASE STUDY: NH3/TKN BY OPA METHOD

FIA Lab
Fluidics Intelligently Automated

Based on:

- o-phthalaldehyde indicator
- fluorescence detection



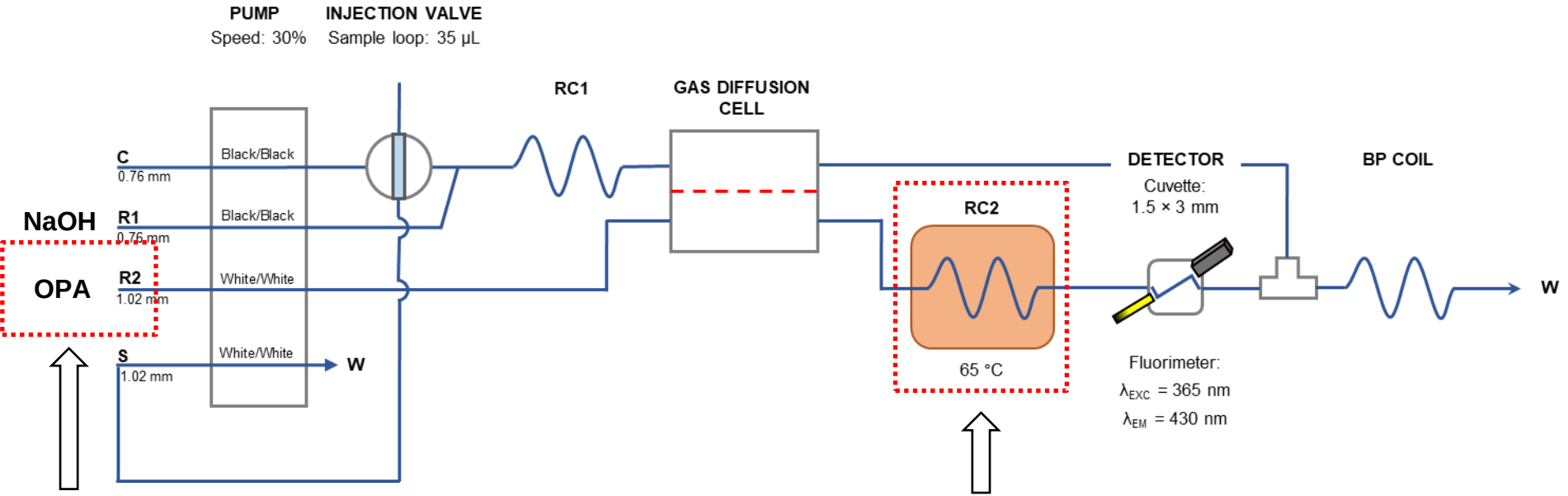
Felix et al.
J. Braz. Chem. Soc., 23 (1), 142-147, 2012

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CASE STUDY: NH3/TKN BY OPA METHOD

FIA Lab

Fluidics Intelligently Automated



Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CASE STUDY: NH3/TKN BY OPA METHOD

FIA Lab
Fluidics Intelligently Automated

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

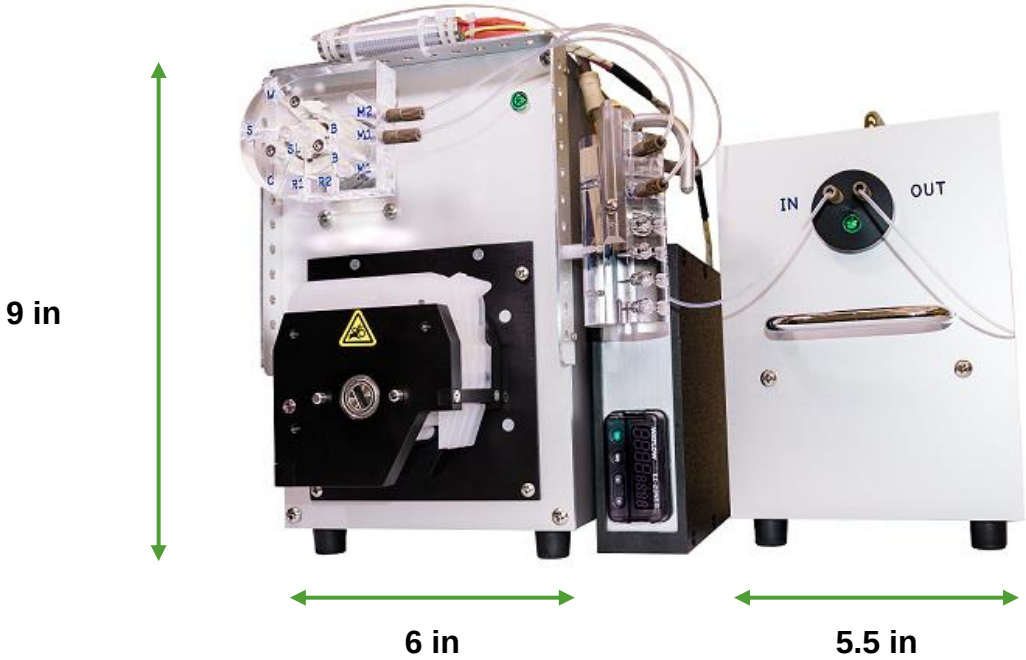
- OPA reaction is slow → technically, would require a long heated coil
- Here, a relatively short (~70 in) coil was selected
- Why: **speed of calibration**

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CASE STUDY: NH3/TKN BY OPA METHOD

FIA Lab

Fluidics Intelligently Automated



Pictured:

FIAlyzer-1000 with PMT Detector

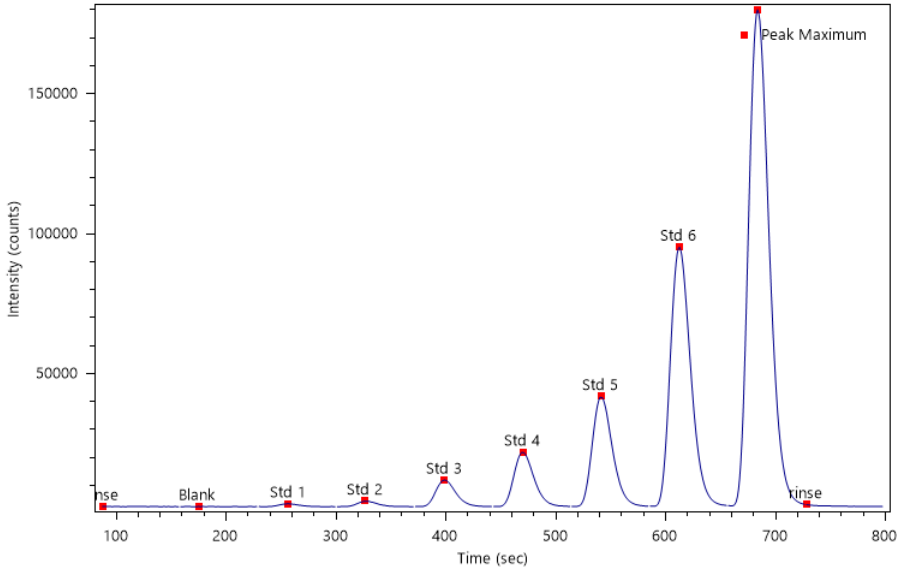
Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CASE STUDY: NH3/TKN BY OPA METHOD

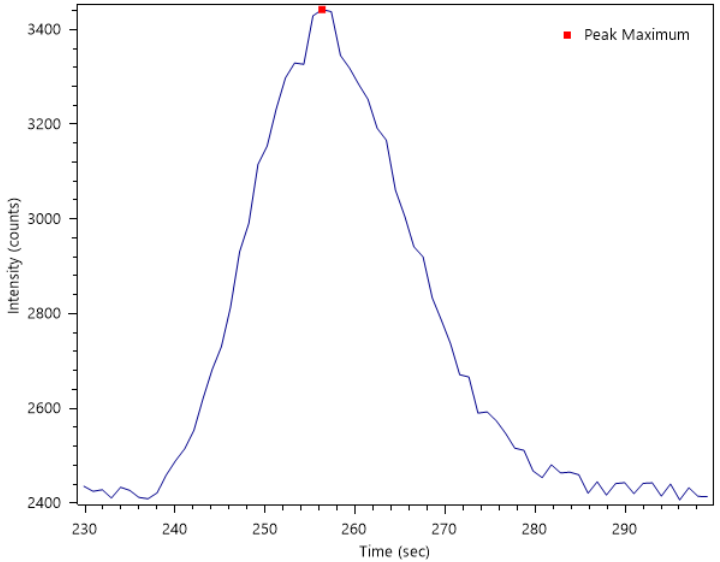
FIA Lab

Fluidics Intelligently Automated

Calibration 50 – 10,000 µg N / L



Single injection 50 µg N / L



Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CASE STUDY: NH3/TKN BY OPA METHOD

Fluidics Intelligently Automated

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	

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

CONCLUSIONS

Fluidics Intelligently Automated

Fluidics Intelligently Automated

"Theory guides, experiment decides."

-- Dr. Izaak Maurits (Piet) Kolthoff

Copyright 2018. FIAlab Instruments, INC. All rights reserved.

FIA Lab

Fluidics Intelligently Automated

THANKS!

Any questions?

You can find me at

sales@flowinjection.com

Copyright 2018. FIAlab Instruments, INC. All rights reserved.