

# Rapid, Accurate and In-Situ Quantification of Lead for Drinking Water Applications



National Environmental  
Monitoring Conference 2018

**Merwan Benhabib, PhD**  
VP Engineering





# Sources of **LEAD** in Drinking Water



**Copper Pipe with Lead Solder:** Solder made or installed before 1986 contained high lead levels.



**Faucets:** Fixtures inside your home may contain lead.



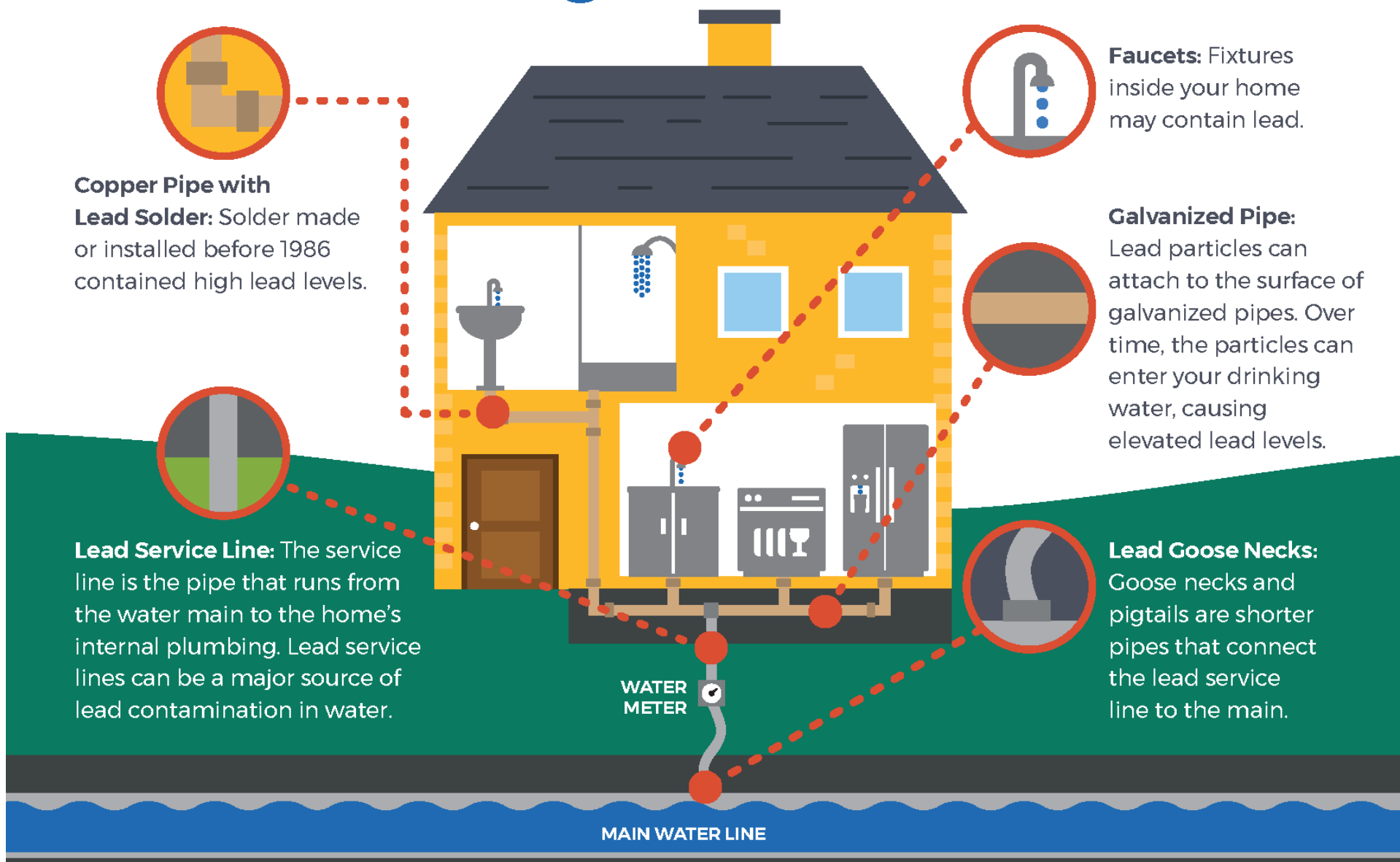
**Galvanized Pipe:** Lead particles can attach to the surface of galvanized pipes. Over time, the particles can enter your drinking water, causing elevated lead levels.



**Lead Service Line:** The service line is the pipe that runs from the water main to the home's internal plumbing. Lead service lines can be a major source of lead contamination in water.



**Lead Goose Necks:** Goose necks and pigtails are shorter pipes that connect the lead service line to the main.



WATER  
METER

MAIN WATER LINE

# Current monitoring procedure

## GC/MS :

- Expensive
- Skilled labor required
- Dedicated system and team
- Time consuming
- Not deployable



## Colorimetric assay:

- Not quantitative
- No speciation
- Interferences



# Raman spectroscopy

1920s



1990s



2000s



Optical telecommunications  
drove technology needed for  
portable, in-line, and compact  
Raman spectroscopy

But Raman is weak

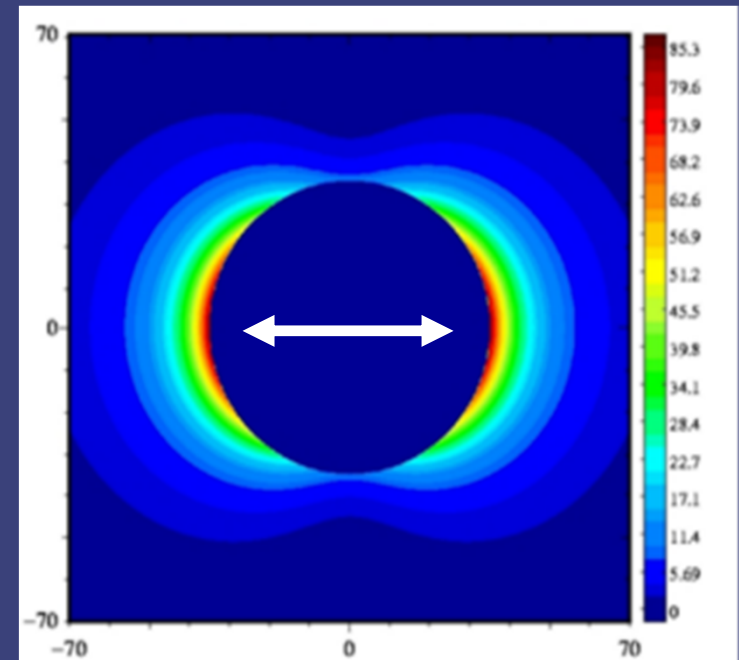
- $\sigma_{NR} \sim 10^{-30} \text{ cm}^2/\text{molecule}$
- **1 in 10 million photons**





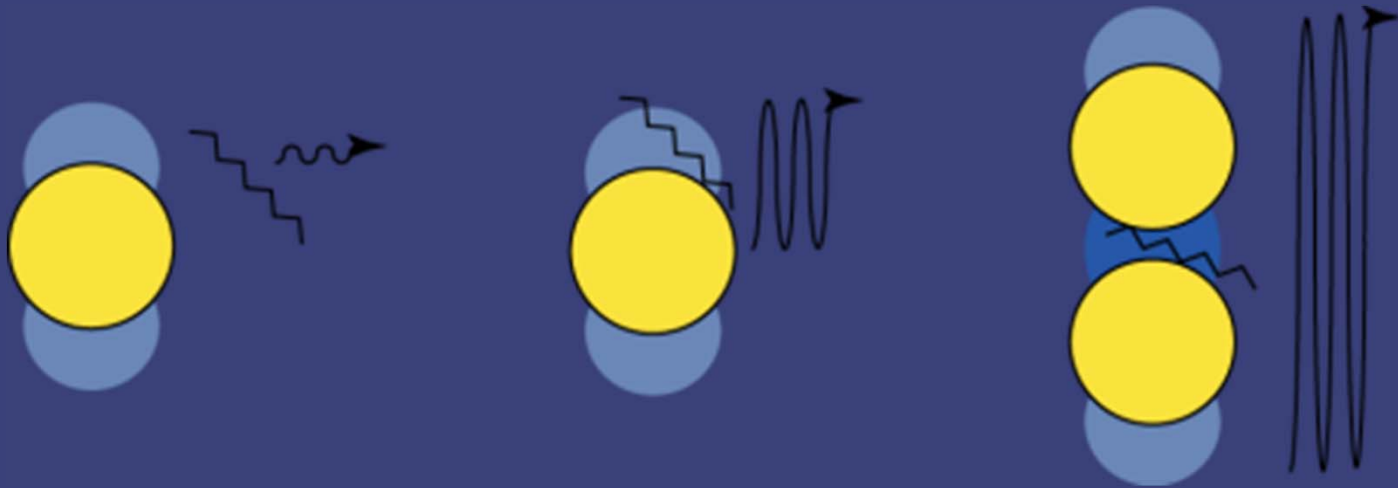
# Surface-Enhanced Raman Scattering (SERS)

- At a rough metal surface
  - Increased field intensity
  - Which means increased Raman signal
- SERS activity quantified by Enhancement Factor
  - EF range: 1 -  $10^{10}$



# Multi-particle effects

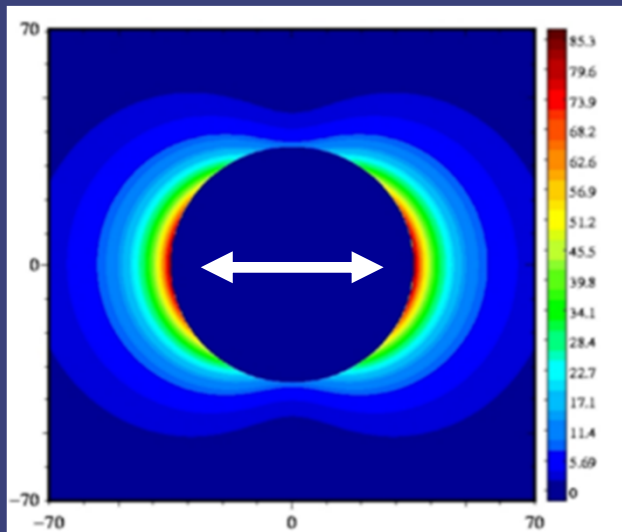
Nanoparticles (gold/silver) enable **ppb-level** detection



“Hot Spots”

# Analyte / substrate interaction

- The SERS effect requires an interaction at the surface – within a couple nanometers
- Analyte must
  - Interact with the substrate
  - Interact with a linker molecule
  - Change the properties of another SERS-active molecule
- Gold nanoparticles: many options to control surface properties





# Raman spectrometer equipment

## Instrumentation

- 785-nm, 60-mW at substrate
- Cooled ( $-20^{\circ}\text{C}$ ) CCD detector
- $200\text{--}2000\text{-cm}^{-1}$ ,  $4\text{-cm}^{-1}$  resolution



# Why not widely used?

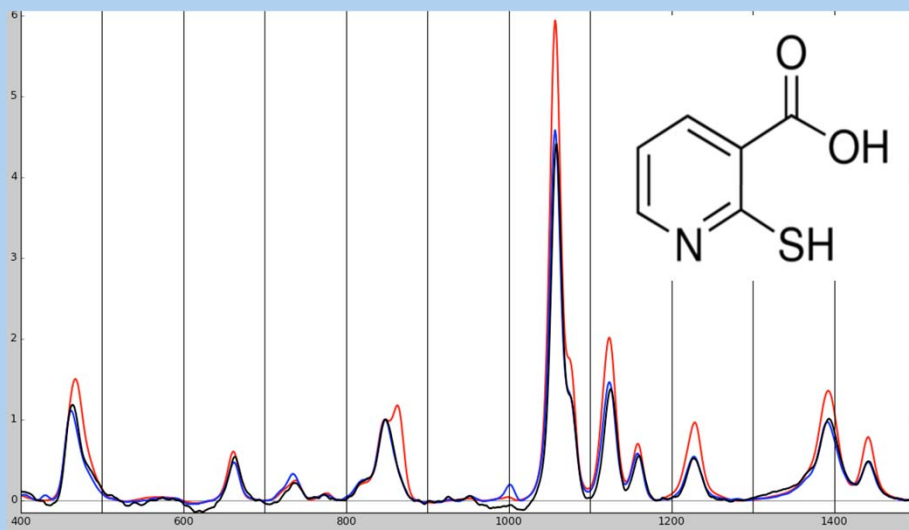
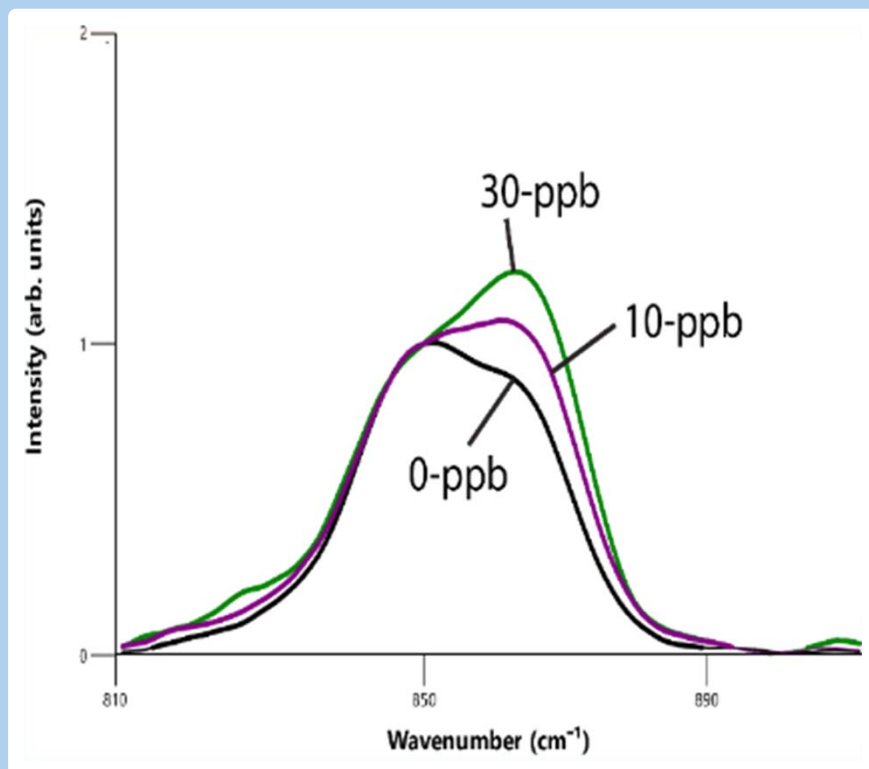
- Achilles' heel: reproducibility
  - Variations in substrate properties
  - Stochastic nanoparticle alignments
- One reviewer: "SERS doesn't work"

**OndaVia has made SERS a quantitative, repeatable method using:**

- Internal standards
- Nanoparticle structure
- Surface modifications
- Intelligent software

# Surface modification using 2-mercaptoisonicotinic acid \*

\* Tan E., Yin P., Lang X., Zhang H., L. Guo. A novel surface-enhanced Raman scattering nanosensor for detecting multiple heavy metal ions based on 2-mercaptoisonicotinic acid functionalized gold nanoparticles (2012), *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, Vol 97, p1007-1012.

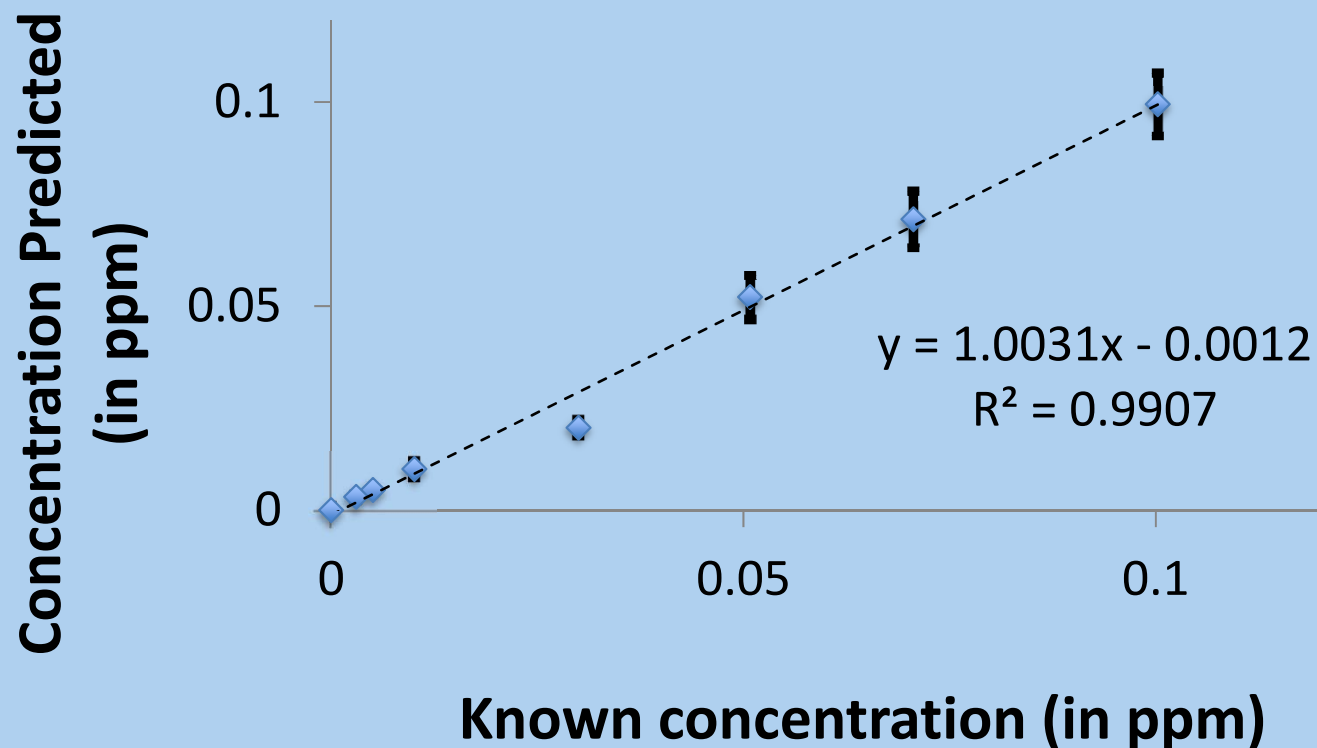


# Quantitative SERS

- Calibration and interferences:
  - Standard Addition (lead and sodium sulfide)
  - Accuracies of better than 10% reported with presence of competing ions
- Simple Standard Operating Procedure allows for quick analyses:
  1. Prepare 6 vials of 0.9 ml of sample
  2. Add 0.1 ml of DI, 25, 50, 75, 100 ppm of Lead and 1 ppm of sulfide in
  3. Add to each vials 40 ul of a 2-MNA with NaCl solution
  4. Wait 10 min
  5. Mix with Nanoparticles
  6. Measure each vial

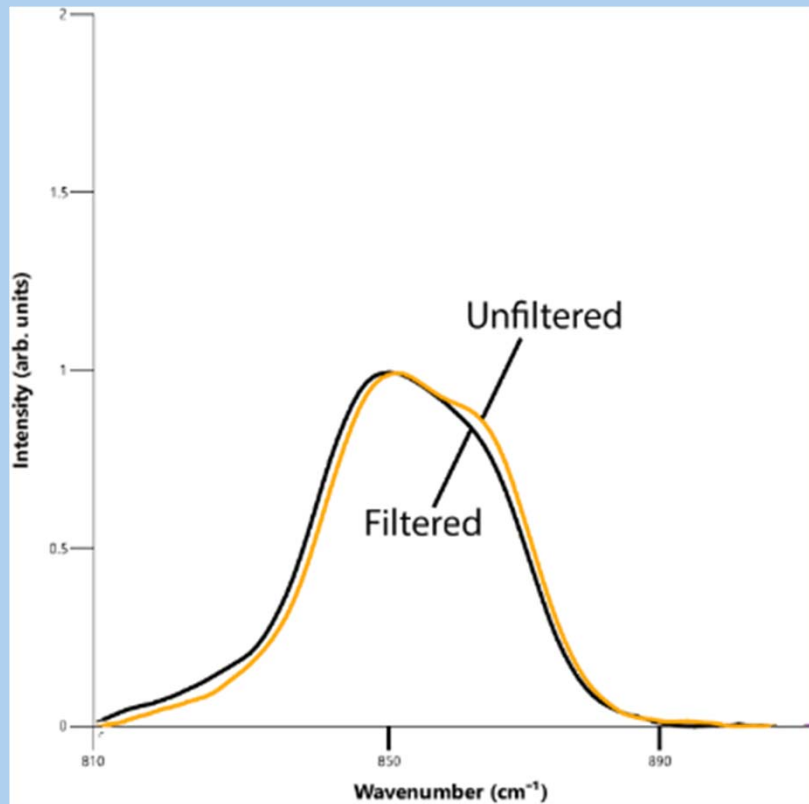
# Lab Standard Calibration Curve

- Wide dynamic range of interest 0 - 100ppb
- Accurate quantification better than 10%.



# Quantitative analysis in real world samples

- Filtered vs. Unfiltered



Tap water expected	Averages measured
8.3	8.8
4.5	4.2
2.2	2.3
1.1	1.3
0.00	0.06



# Ultra compact solid state spectrometer

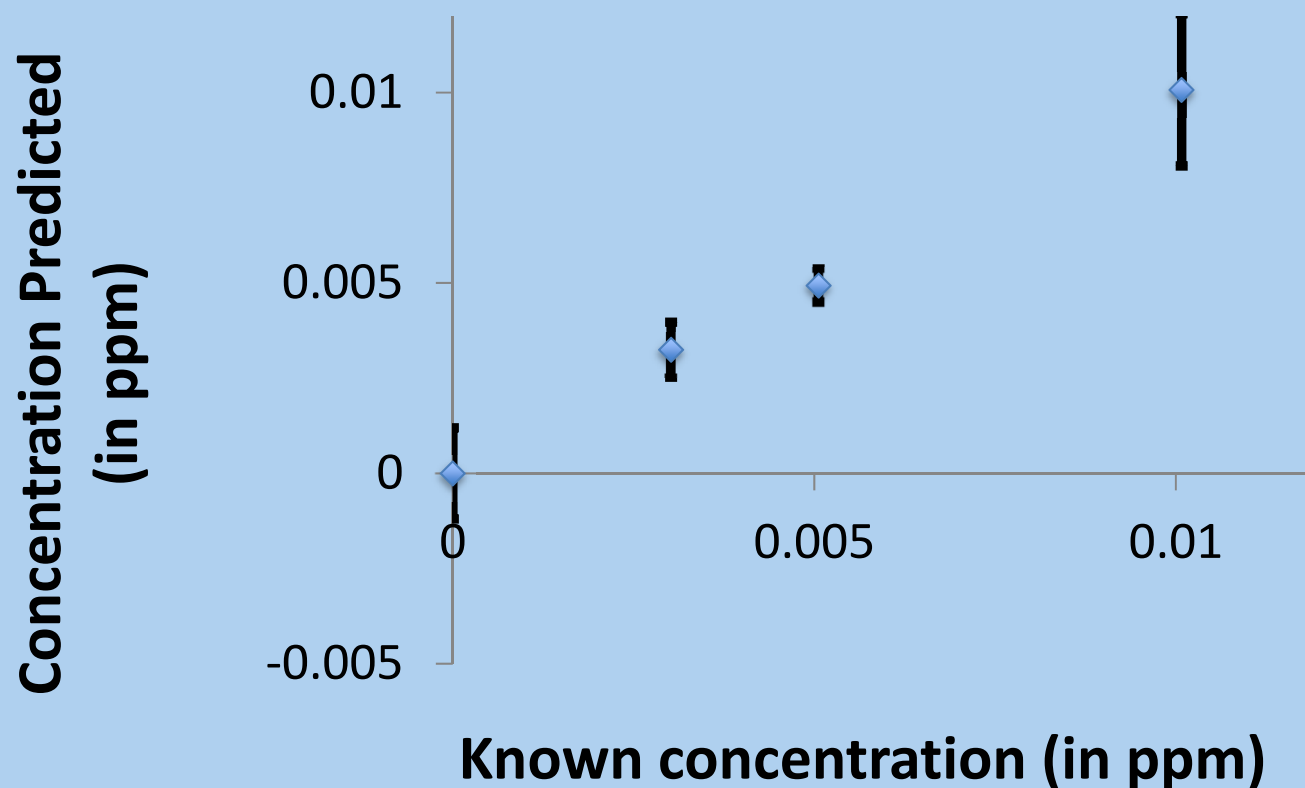
## Instrumentation

- 785-nm, 50-mW, 10  $\mu\text{m}$  slit, No temp control
- APS-CMOS Detector
- 200-2000- $\text{cm}^{-1}$ , 5- $\text{cm}^{-1}$  resolution



# Lab Standard Calibration Curve

- Accurate quantification better than 10%.





OndaVia, Inc.

# Special thanks to

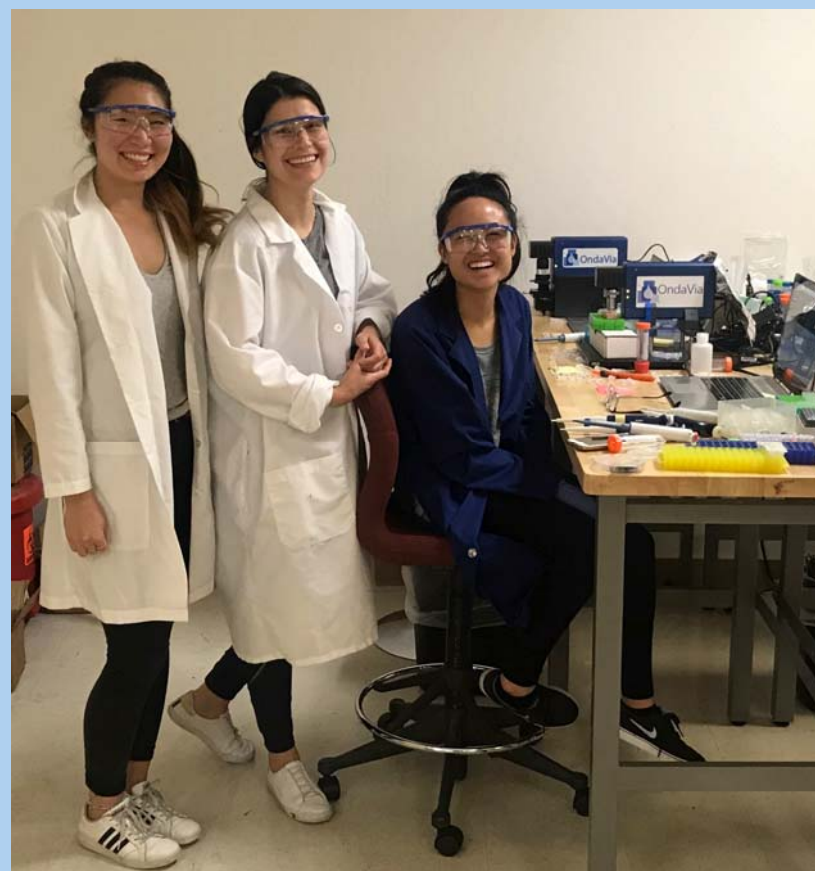


Mark Peterman, PhD

Mayra Zaragoza

Melissa Yao

Kristle Cruz Garcia



And to...

