



# EPA ORD Lead Particulate Research and Related Support

Research Overview Presented to NSF DWTU Task Group on Higher Lead Influent

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# Field Work



## Site 1

- Gradual change in pH over years linked to destabilization of lead scale on pipes
- Release of lead has put city over the LCR lead action level
- Lead service lines are major source of lead
- City sampling brought into the question the effectiveness of city-provided certified POU devices
- EPA field testing to examine the nature (soluble versus particulate) of lead release was performed



# Water Sampling Approach

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- Record stagnation time, color of POU filter light, when the filter was last replaced, and made other notes.
- Run water around filter device for 6500 mL, then place POU device in line for next 500 mL of water. Record flowrate.
- Collect next 1000 mL (treated water sample).
- Put device back in by-pass mode and collect next 1000 mL (tap water sample). Record flowrate.
- Run POU device in by-pass mode for 7 minutes, then collect a series of background tap water quality samples (to be analyzed by ORD).
- POU filters were replaced with new filter. Filters were kept for analysis.
- Water samples were acidified and analyzed by EPA R2 for lead by ICP-MS.



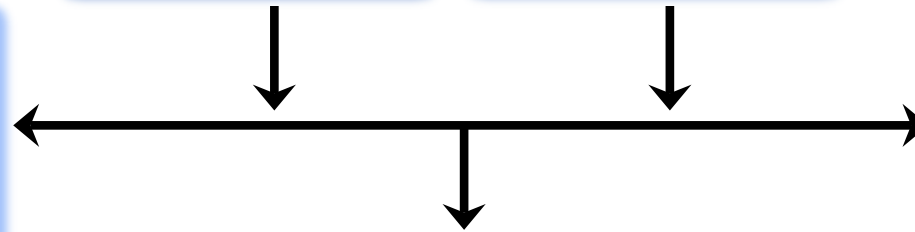
1. Tap water  
(untreated, 7<sup>th</sup> L sample)



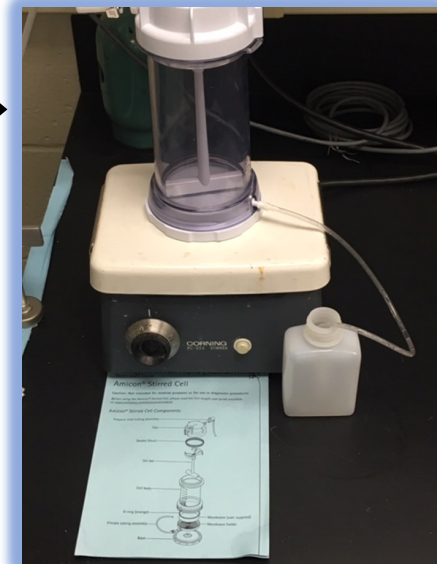
2. Treated water  
(filtered, 8<sup>th</sup> L sample)



No filter (total lead)



0.2  $\mu\text{m}$  syringe filter

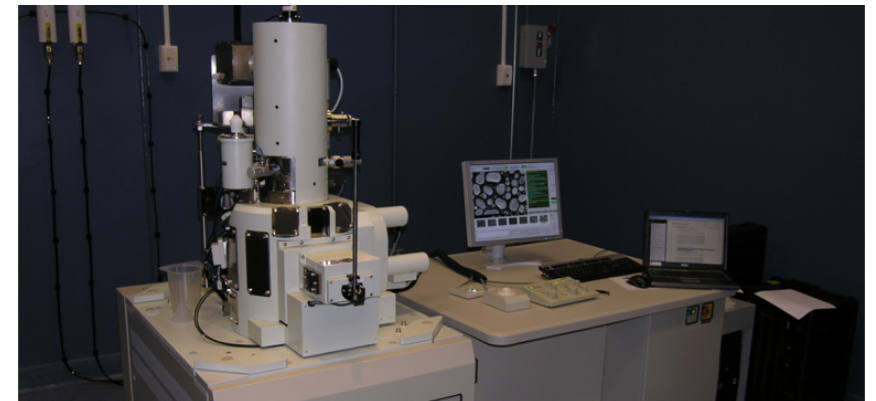
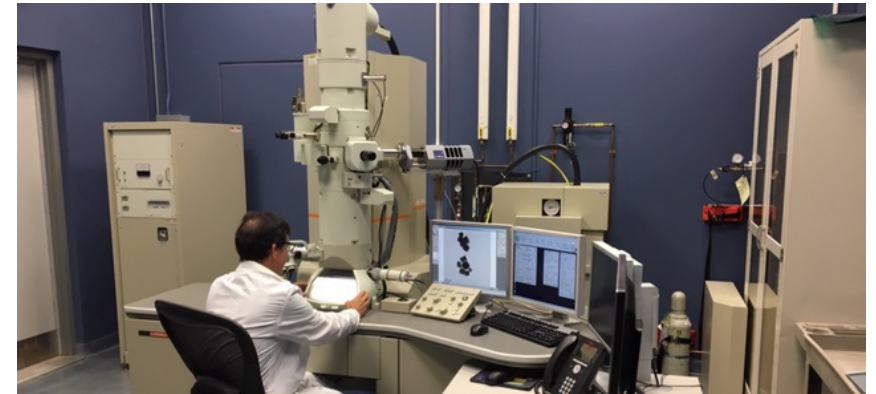
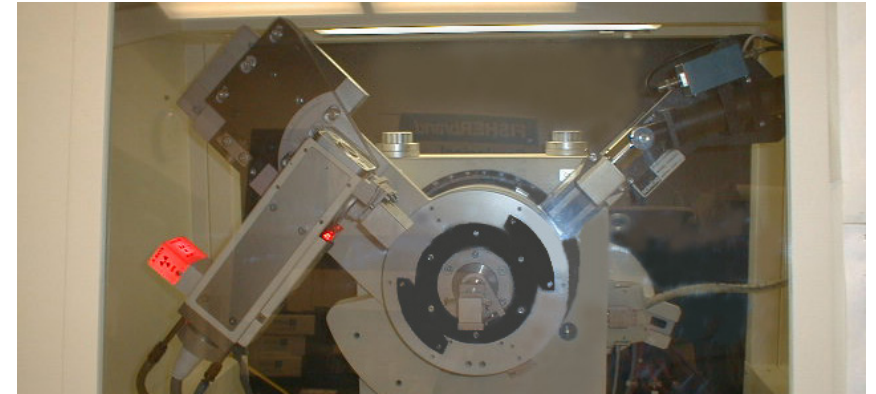


Ultrafiltration  
("soluble" lead)

A total of six samples per home were collected.

# Solids Analysis

- EPA ORD Advanced Materials Solids Analysis Research Core (AMSARC)
- Examine particles trapped on ultrafilters
- Scanning Electron Microscopy (SEM)
- Transmission Electron Microscope (TEM)
- Energy Dispersive Spectroscopy (EDS)
- X-ray diffraction (XRD)



# Results

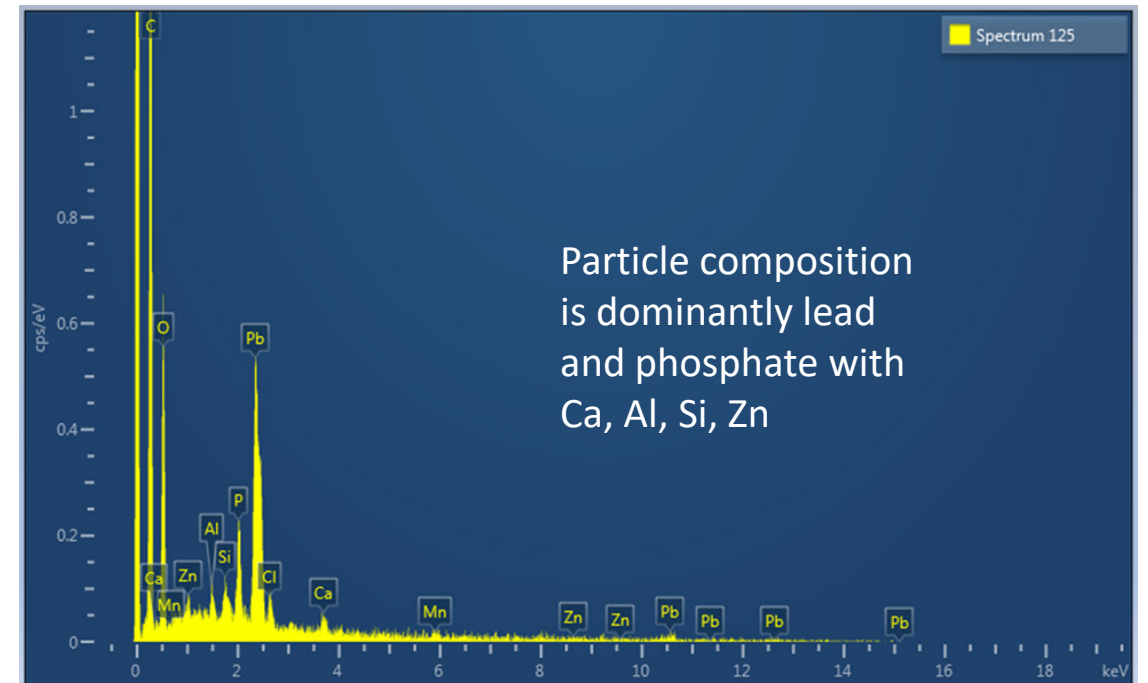
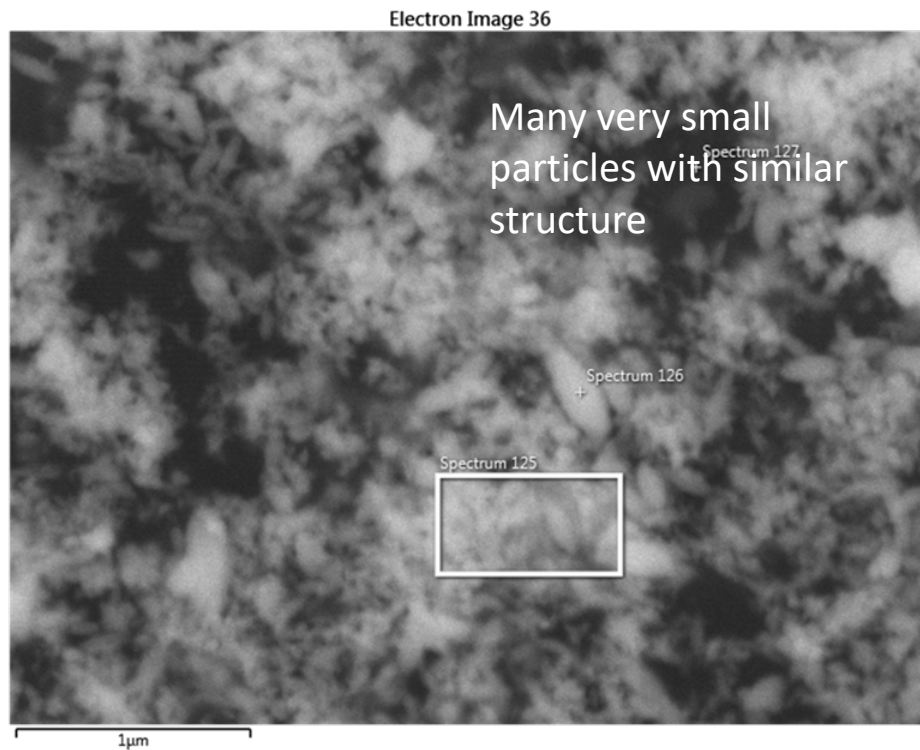
## Analysis of Lead Species in Tap and POU or Pitcher Treated Water

Home	Total lead in tap water, $\mu\text{g/L}$	Total lead in POU or pitcher filter treated water, $\mu\text{g/L}$	Particle lead fraction in tap water	Particle lead fraction in POU or pitcher filter treated water
Home 1	1300	45	98%	87%
Home 2	75	28	59%	82%
Home 3	727	22	96%	82%
Home 4	33	1	70%	3%

# House 1

## Tap Water; SEM/EDS Result 2

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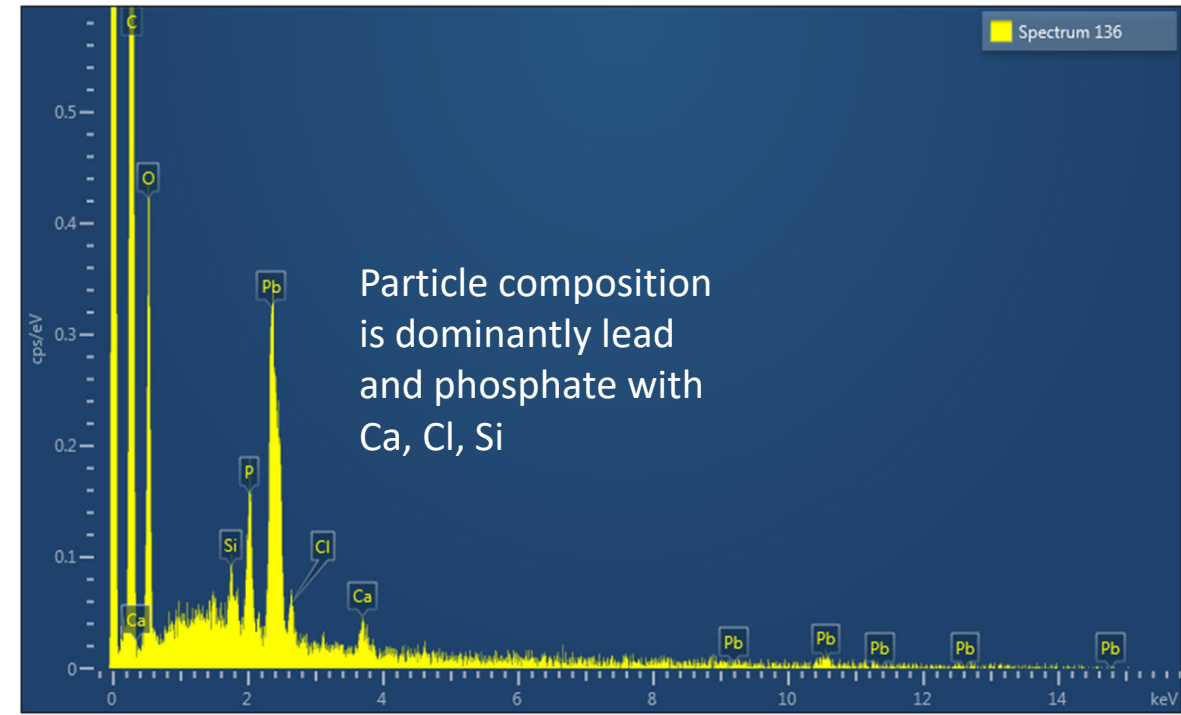
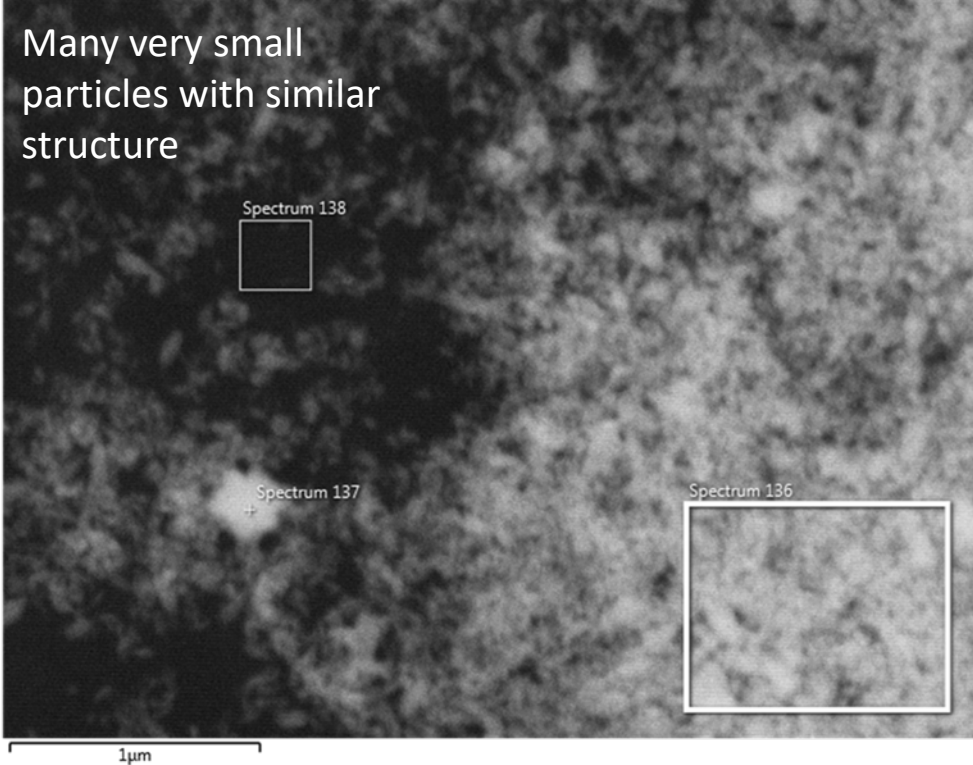




# House 1

## *POU Treated Water*; SEM/EDS Result

Electron Image 39





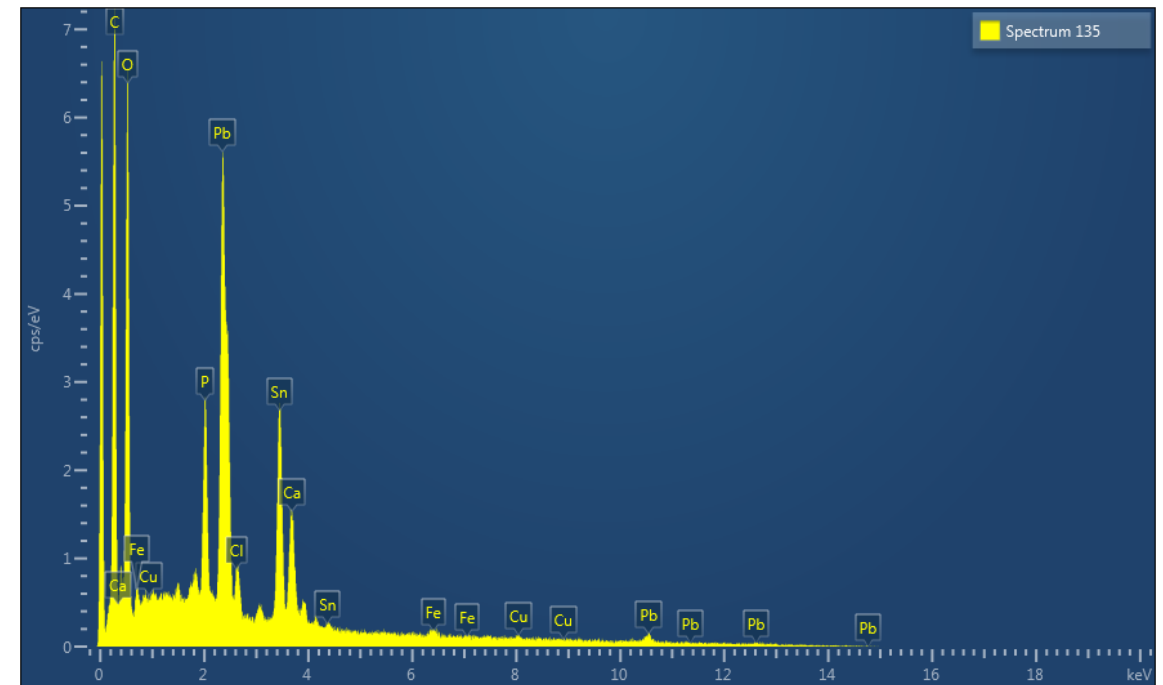
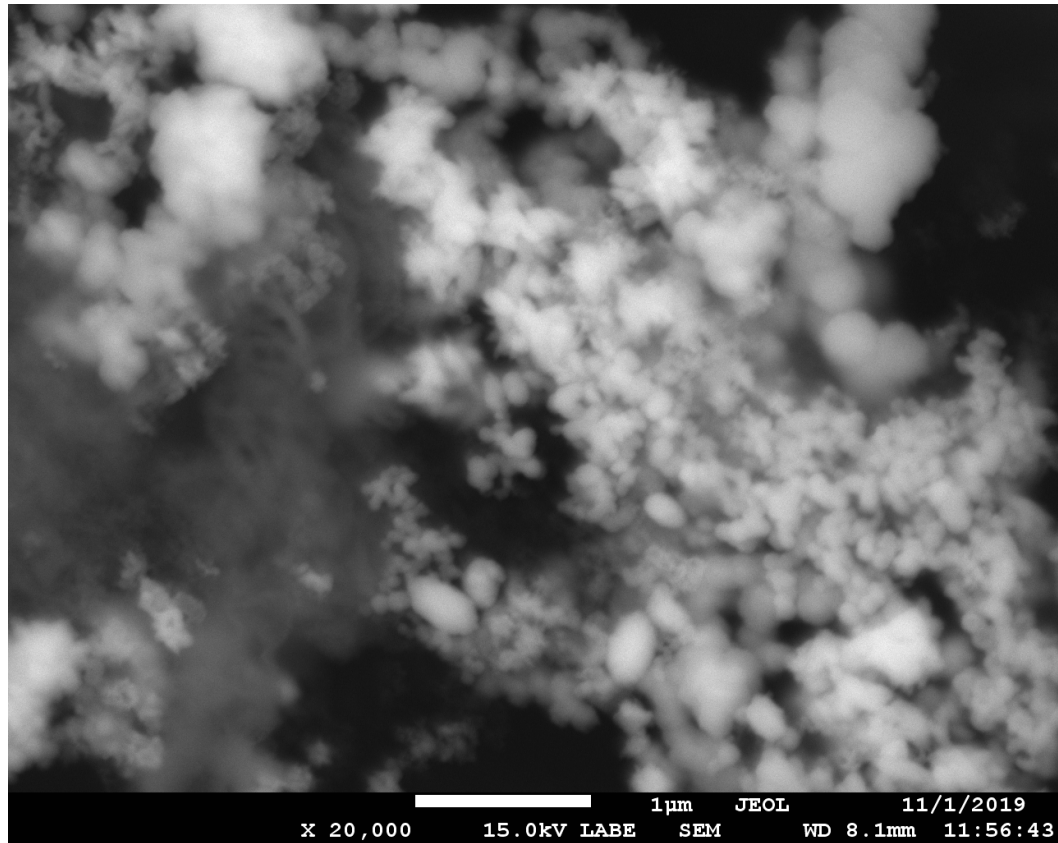
## Site 2

- Source water change was made
- Release of lead has put the city over the LCR lead action level
- Lead levels have remained elevated for months and have levelled-off
- No lead service lines- leaded solder primary source of lead
- EPA field testing to examine the nature (soluble versus particulate) of lead release was performed

# Sampling Location 5 – First Draw Sample

## SEM/EDS analysis

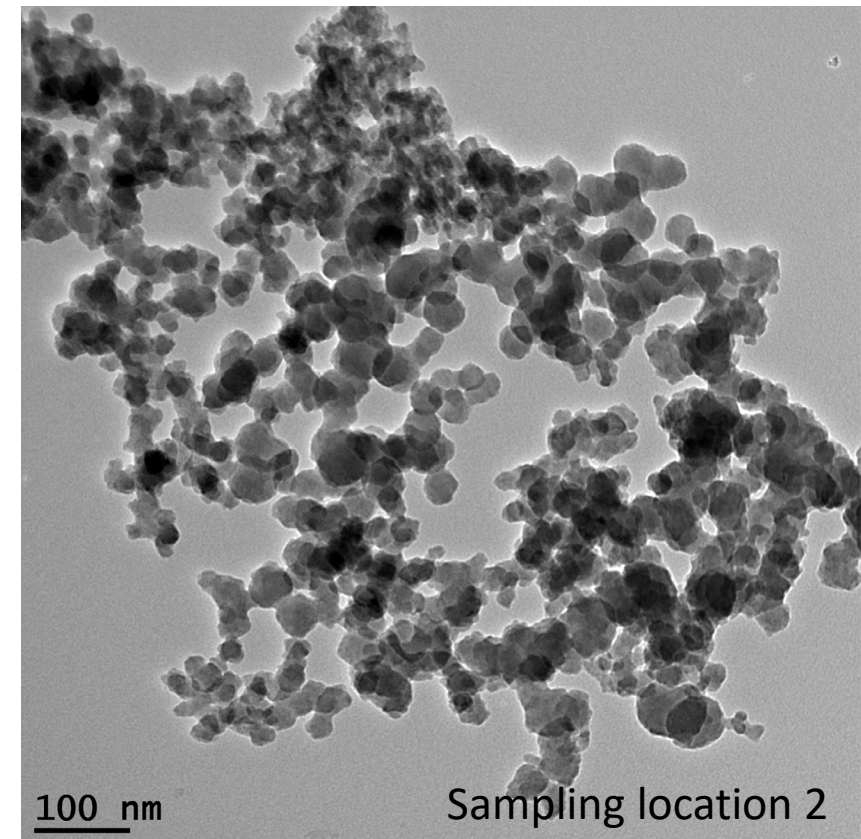
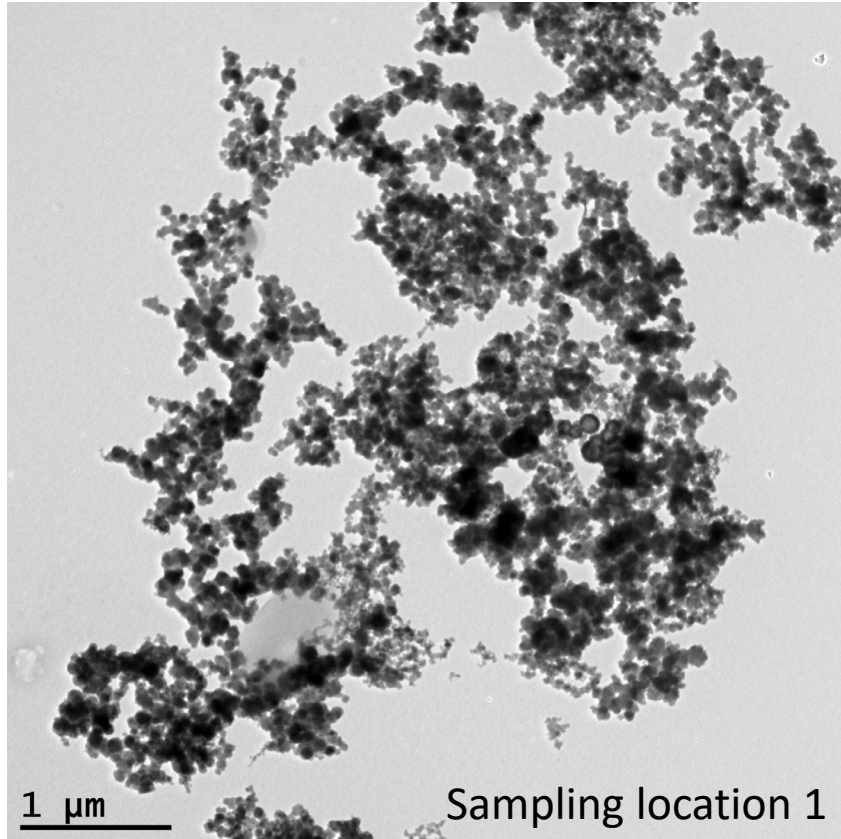
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# First Draw Samples

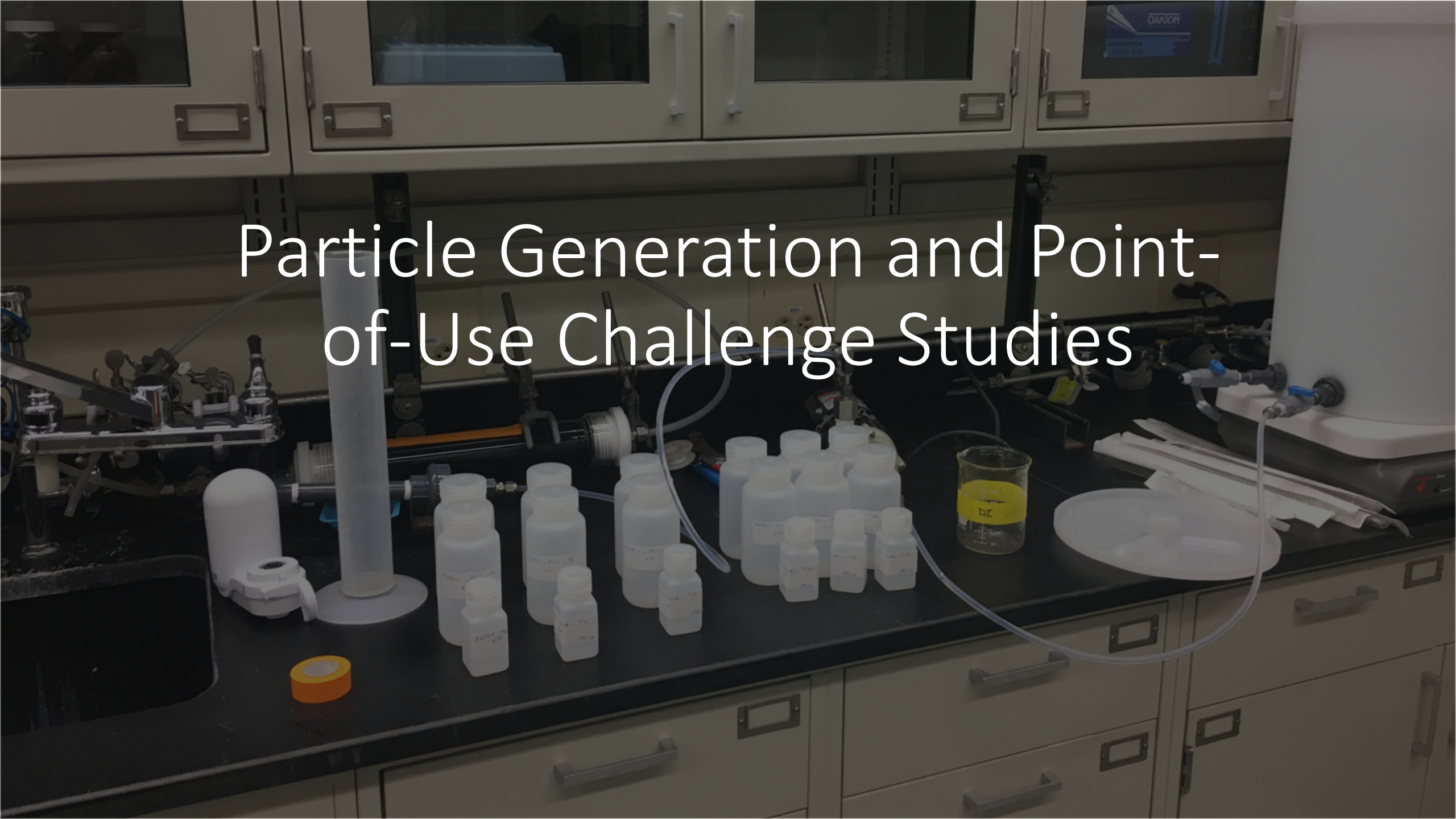
## TEM/EDS analysis

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# Particle Generation and Point-of-Use Challenge Studies



# Experimental conditions

## Experimental conditions

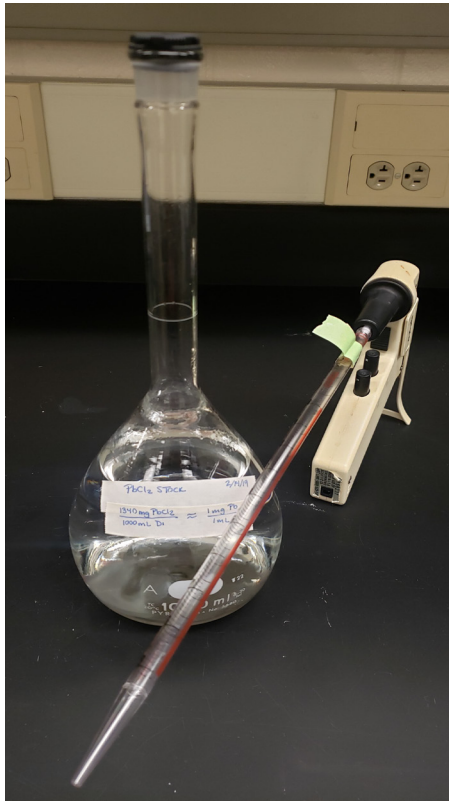
Lead Phosphate Particles:  
pH 7.5, DIC 7 mg/L,  
Orthophosphate 3 mg/L,  
Lead 0.100 mg/L

- Lead was prepared from lead chloride,  $\text{PbCl}_2$
- Orthophosphate was prepared from sodium orthophosphate,  $\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$
- Dissolved inorganic carbon (DIC) was prepared from sodium bicarbonate,  $\text{NaHCO}_3$
- pH was adjusted with 0.6 N hydrochloric acid (HCl) and 0.6 N sodium hydroxide, NaOH

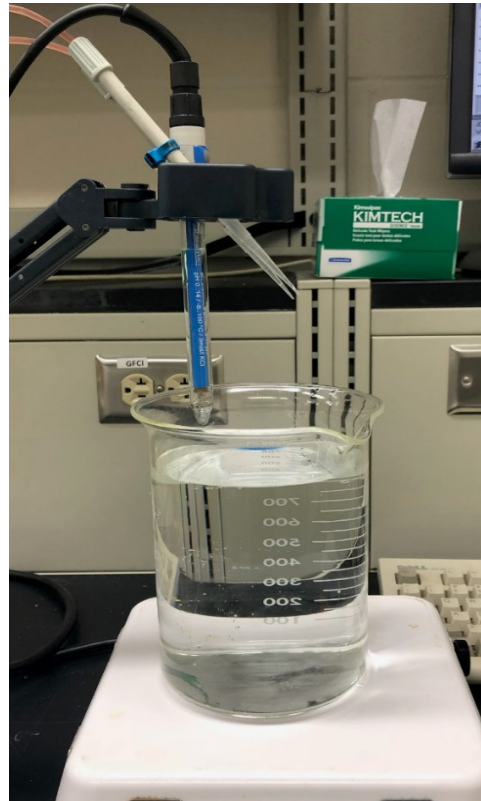


# Nanoparticle Generation Approach

10 mL

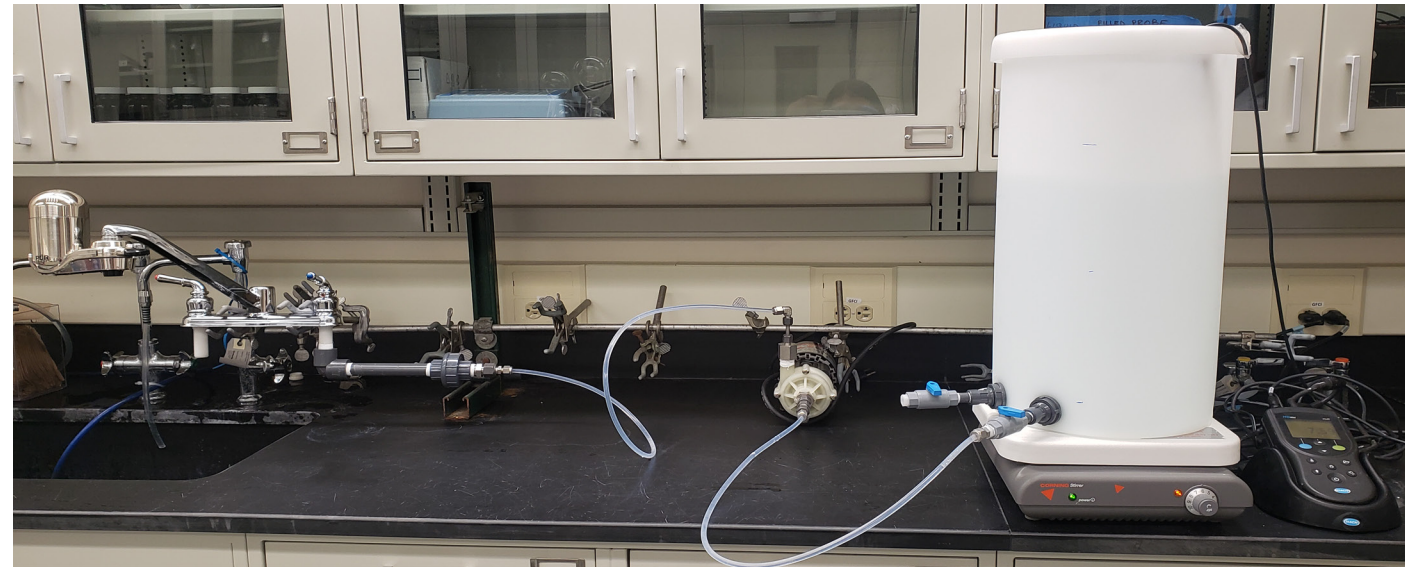


Primary Soluble  
Lead Stock



Secondary Lead Particle  
Stock Suspension (5 mg  
Pb/L)

400 mL



Final Lead Particle  
Suspension (0.10 mg  
Pb/L)

# Operation of the Faucet-Mounted and Pitchers POU Devices

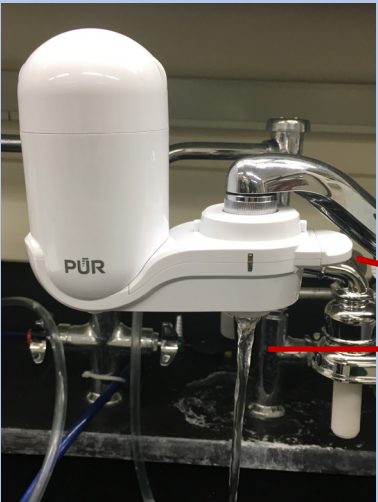
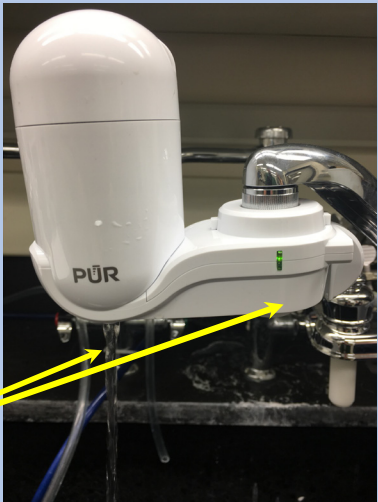
- Following manufacturer's instruction: pre-flushing or soaking of the POU
- Faucet-mounted: Running prepared challenge water (57 L) at a minimum of 20 psi
  - If filter equipped with a meter/lights, ensure it is working properly
- Pitchers: Rinse 3X with DI water before 1<sup>st</sup> use, follow manufacturer's instructions
- Sampling: before and after the POU
- Parameters analyzed:

Time of sample	Total Pb	0.2 µm – Pb	Ultrafiltration – Pb	Total Pb – Palintest	TIC	Inorganics	Hach - orthoP	pH and temperature
Beginning	X	X	X	X			X	X
Middle	X	X	X	X	X	X	X	X
End	X	X	X	X			X	X



# Filtration Flow

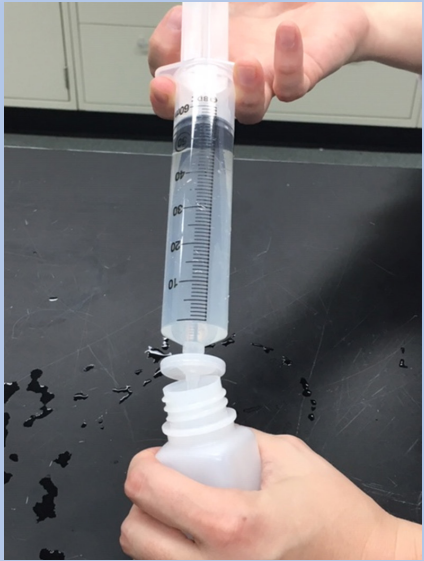
Filter mode



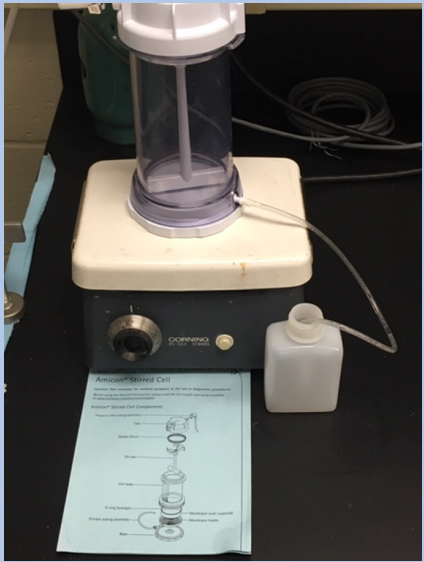
By-pass mode



No filter (total lead)



0.2  $\mu\text{m}$  syringe filter



Ultrafiltration  
("soluble" lead)

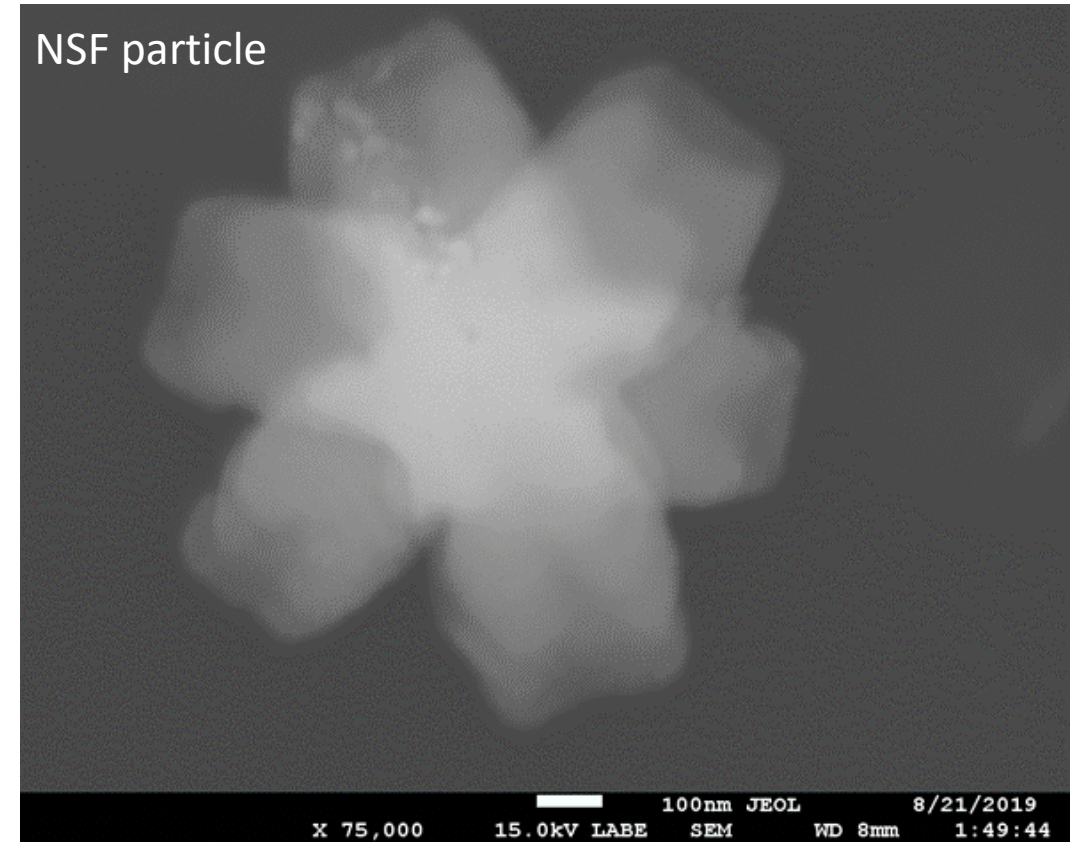
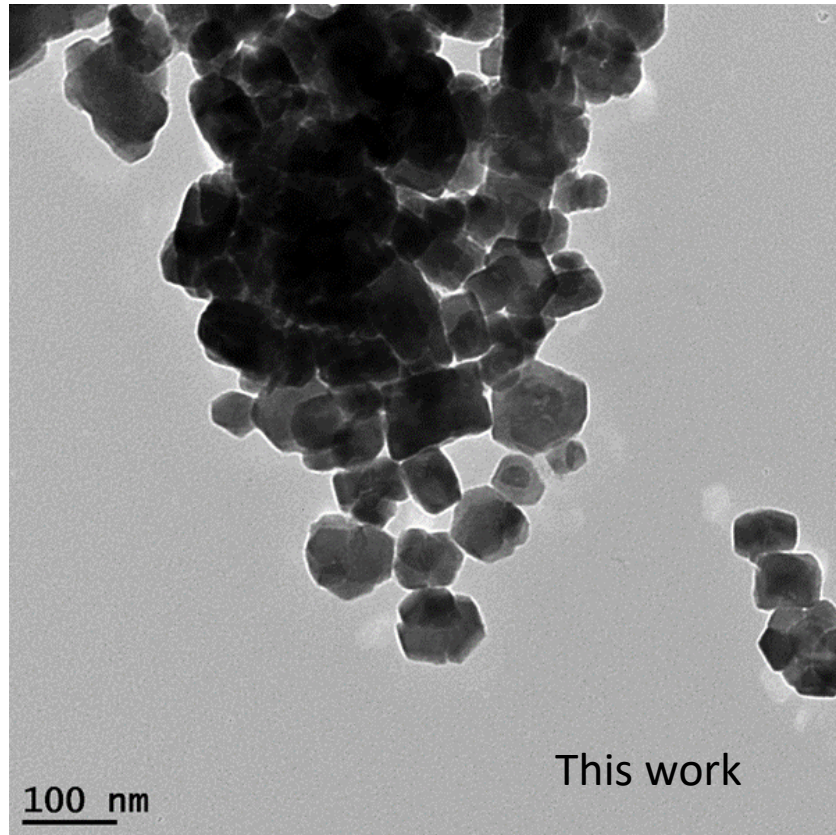
# Preliminary Results





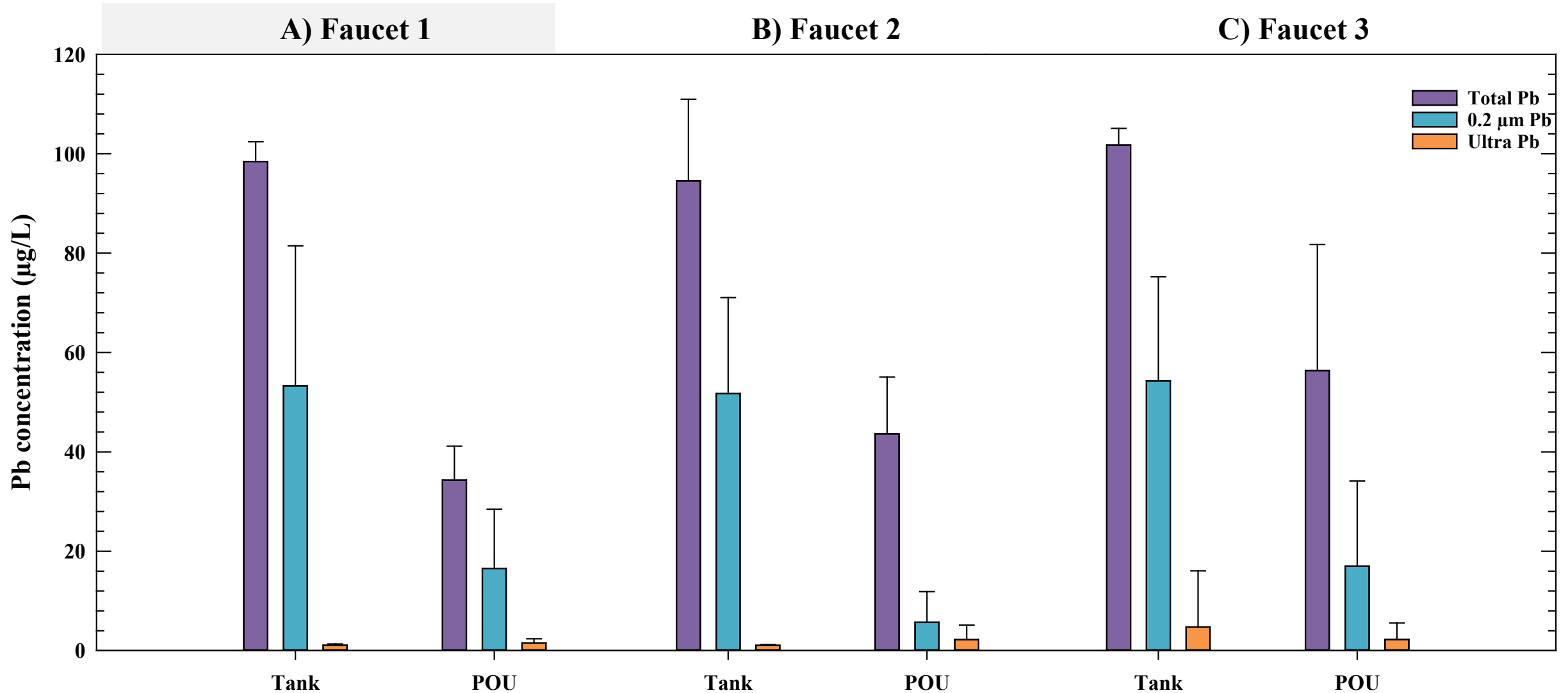
# Lead Nanoparticles versus NSF Challenge Lead Particle

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# Filtration Results – Faucet-mounted POUs

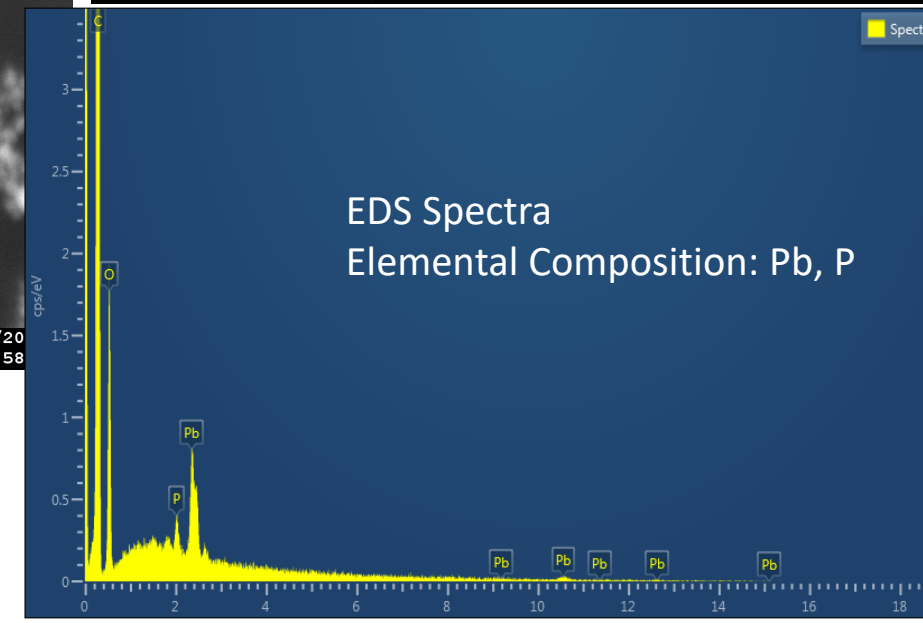
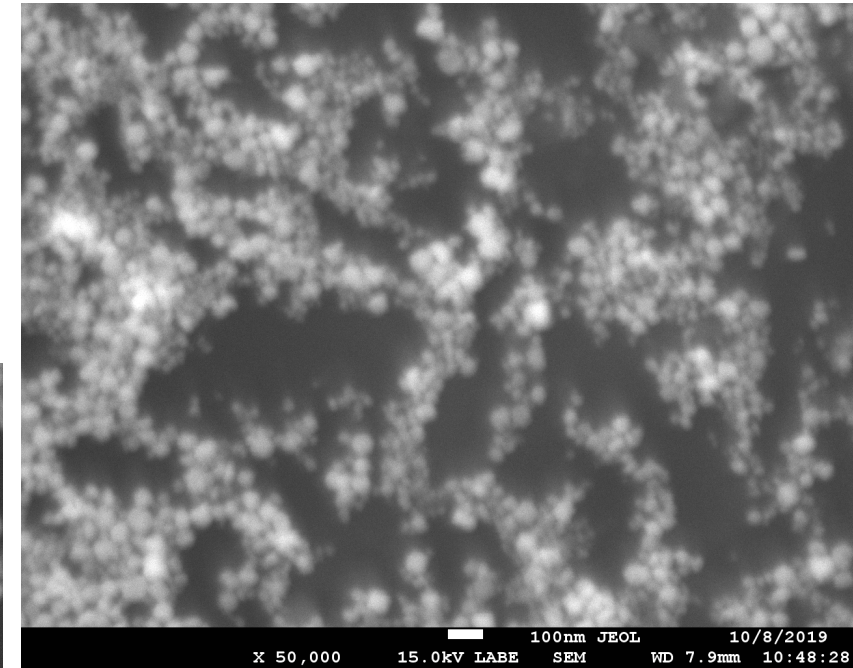
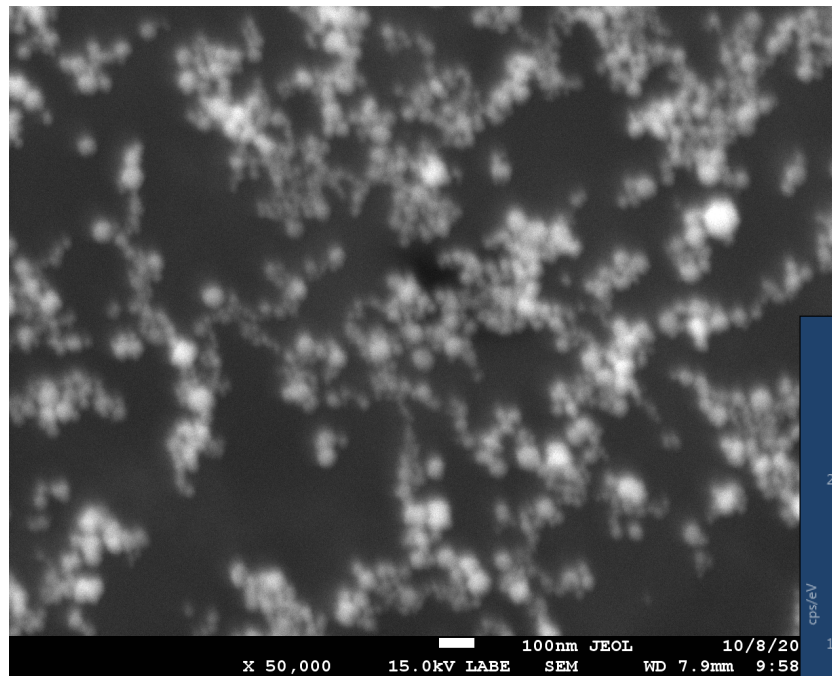
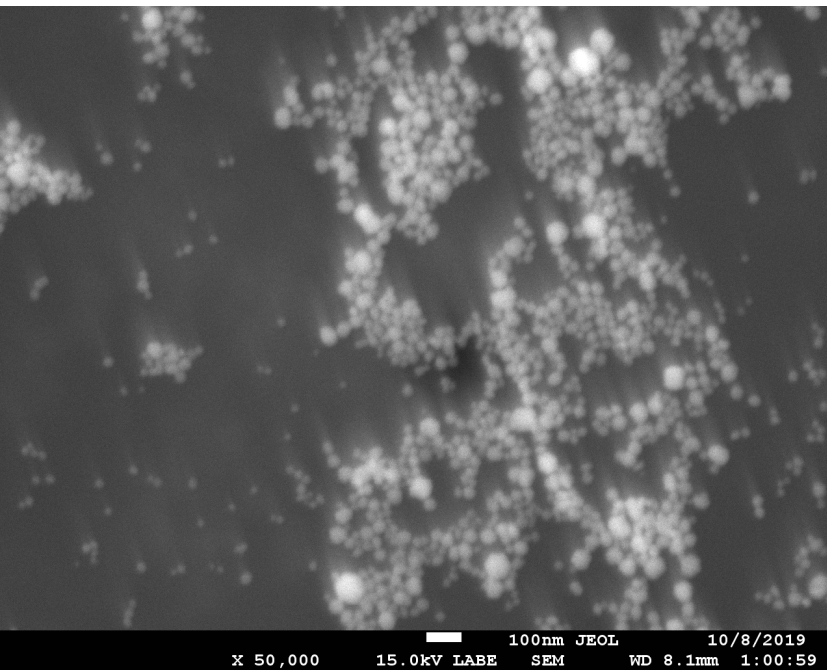


# Test POU16: Pitcher Filter Lead Nanoparticles

POU

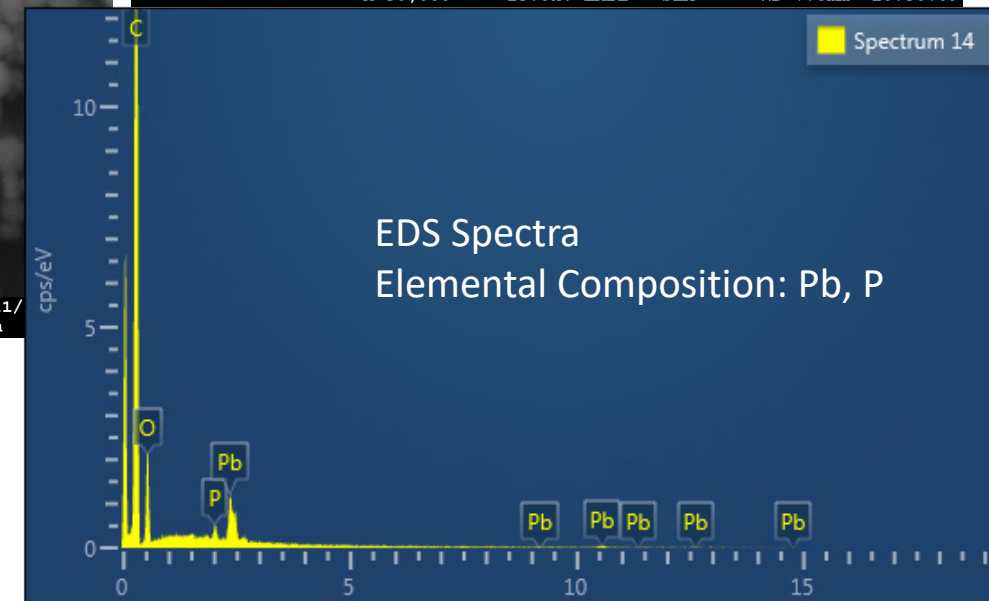
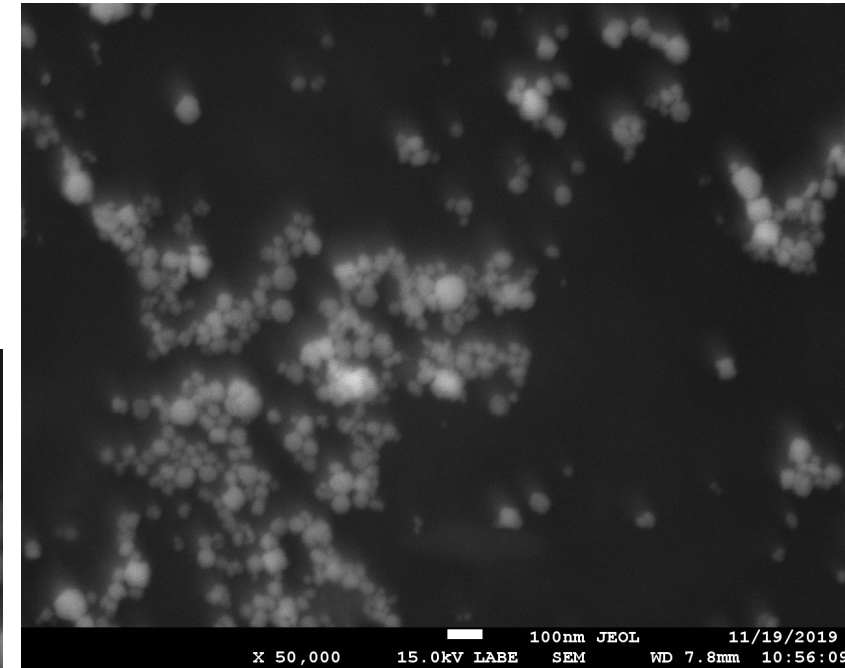
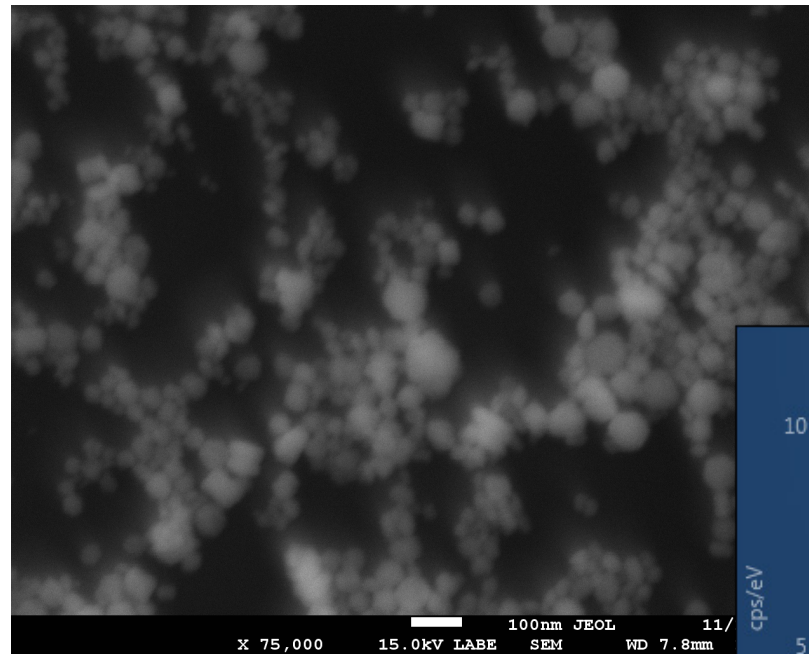
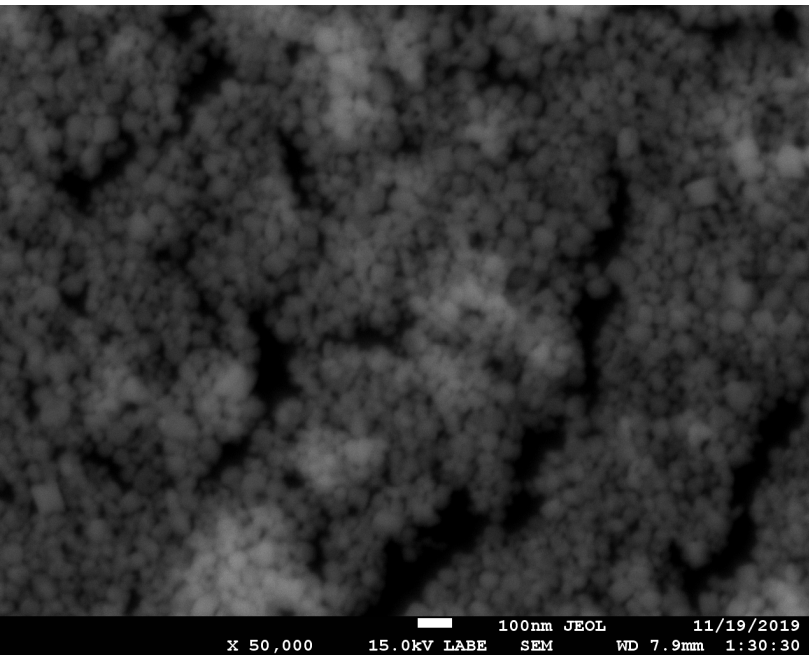
Stock

Tank



# Test POU23: Faucet Filter

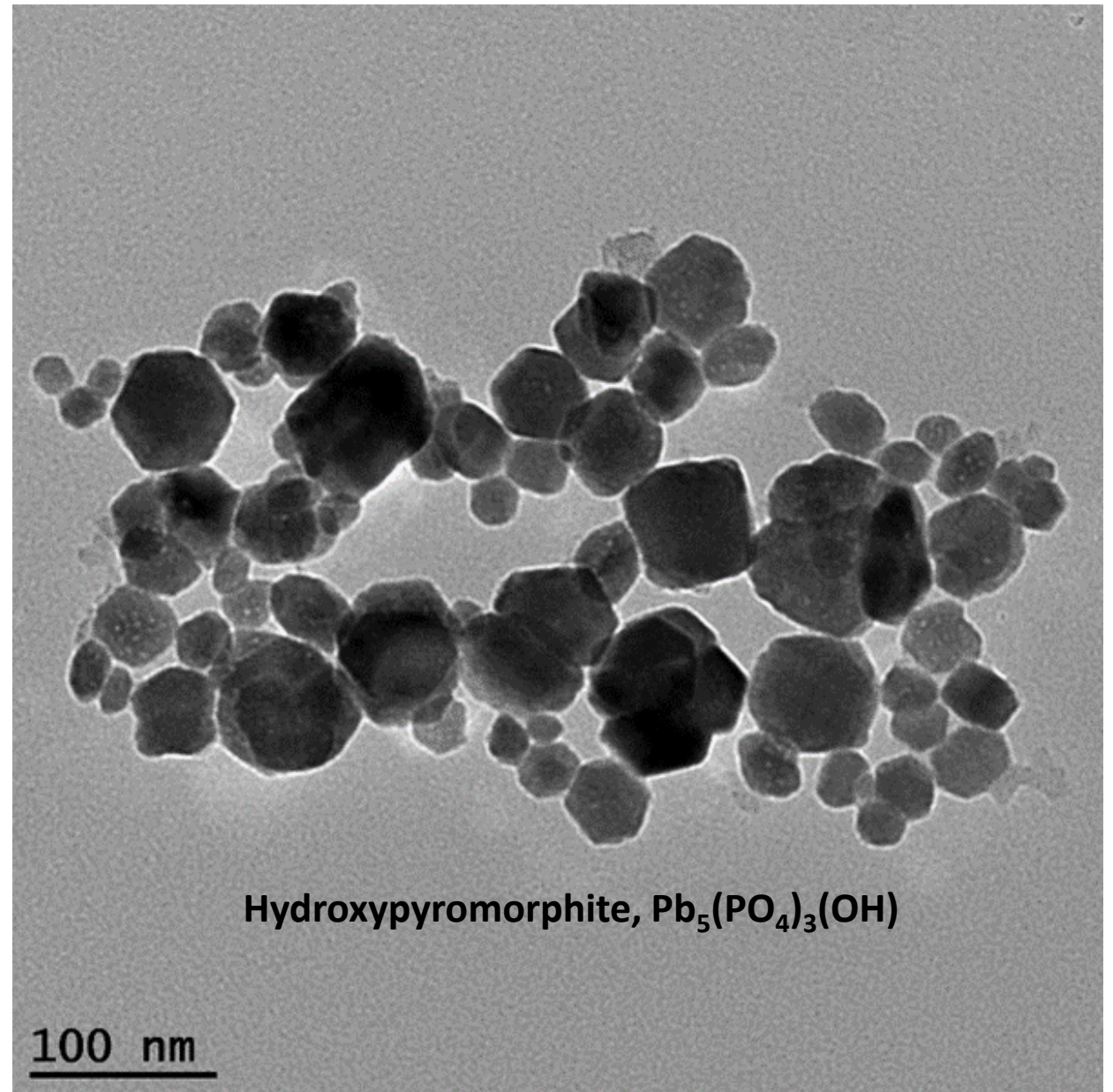
## Lead Nanoparticles





# TEM Image of Lead Phosphate Nanoparticles

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# Conclusions to Date

- Very small lead-phosphate nanoparticles have been identified in drinking water at two field test communities and were the major contributor of total lead to the tap water.
- Stable lead nanoparticle suspension were created in the laboratory: diameter < 100 nm, hydroxypyromorphite.
- Laboratory generated particles very similar to field observed particles.
- POU and pitcher filters that were certified to remove soluble and particulate lead performed poorly at removed lead phosphate nanoparticles with the exception of one pitcher filter.





### **Notice**

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