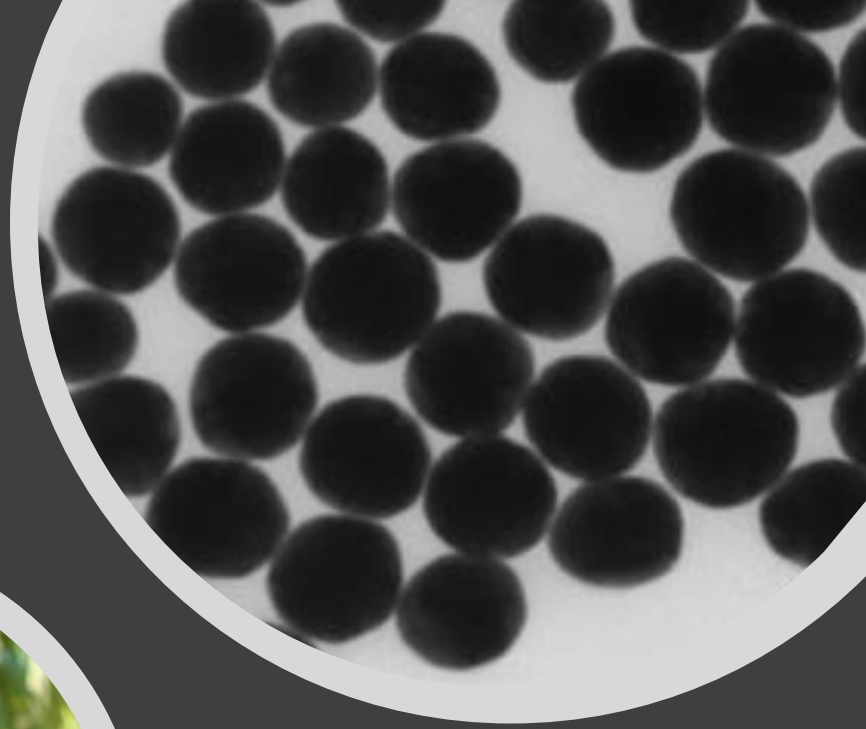
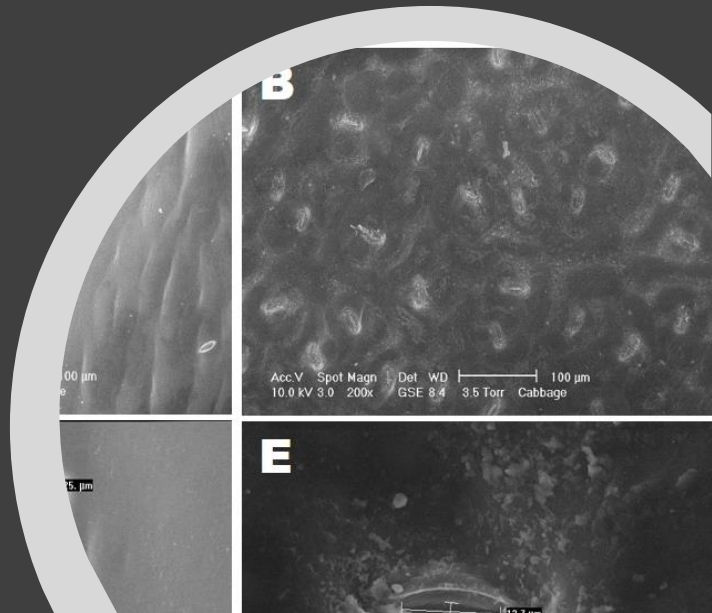
Quantification of Multi-Element Nanoparticles in Environmental Samples Using Single Particle ICP-MS, with Arturo Keller at UCSB





Jenny Nelson

Studies with Arturo Keller

J Nanopart Res (2018) 20: 101
https://doi.org/10.1007/s11051-018-4192-8

RESEARCH PAPER

Detection of nanoparticles in edible plant tissues exposed to nano-copper using single-particle ICP-MS

Arturo A. Keller · Yuxiong Huang · Jenny Nelson

Received: 17 January 2018 / Accepted: 14 March 2018 / Published online: 9 April 2018

Arturo Keller

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Environmental Pollution

Research Paper | ENVPOL_2019_5954

Fast Multi-Element Quantification of Nanoparticles in Wastewater and Sludge Using Single Particle ICP-MS

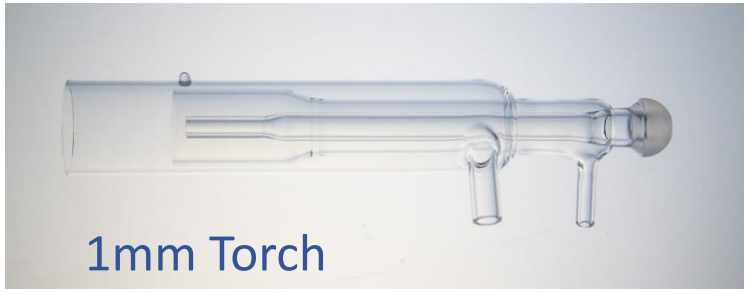
Yuxiong Huang, Arturo Keller, Pabel Cervantes, Jenny Nelson
Submitted 22 Oct 2019

Under Review 24 Oct 2019

Alert: keep me informed about the submission status for this manuscript

Mass Spectrometer used in our studies

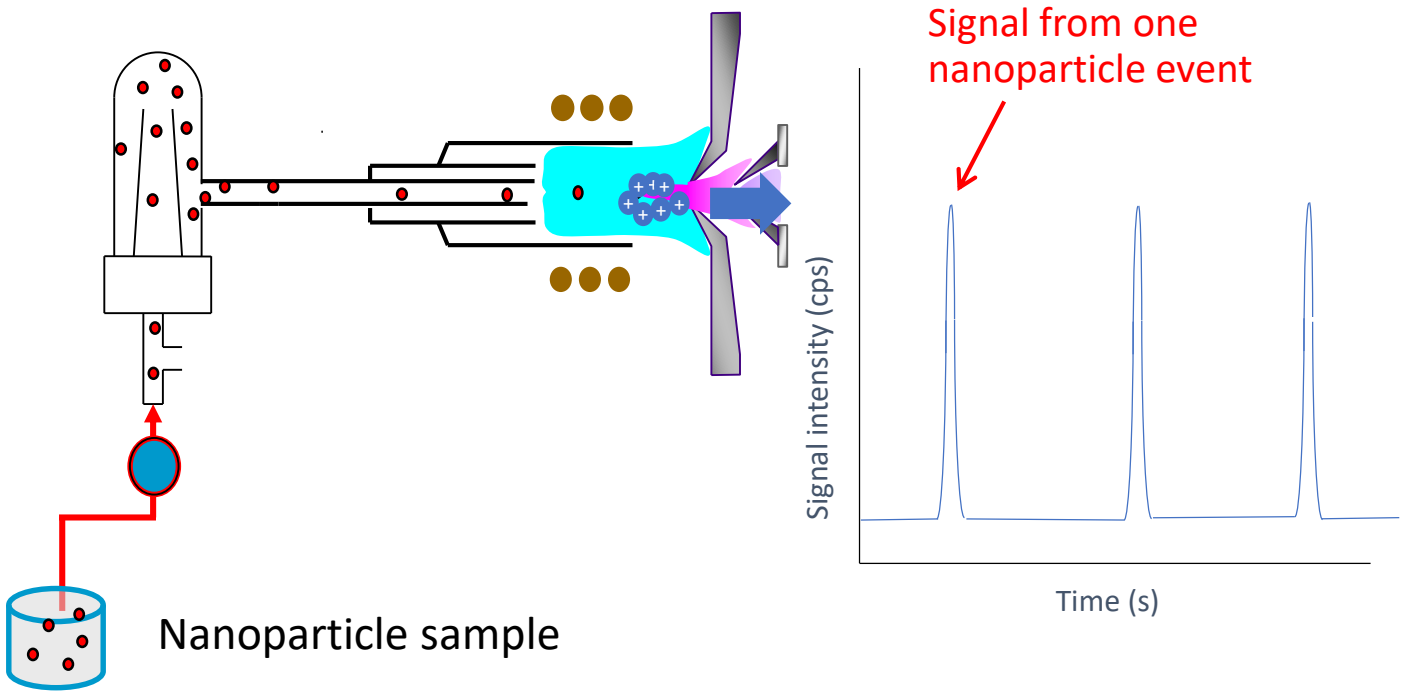
7900 quadrupole ICP-MS – ultra-sensitive, ultra-fast 0.1 ms dwell



NP characterization

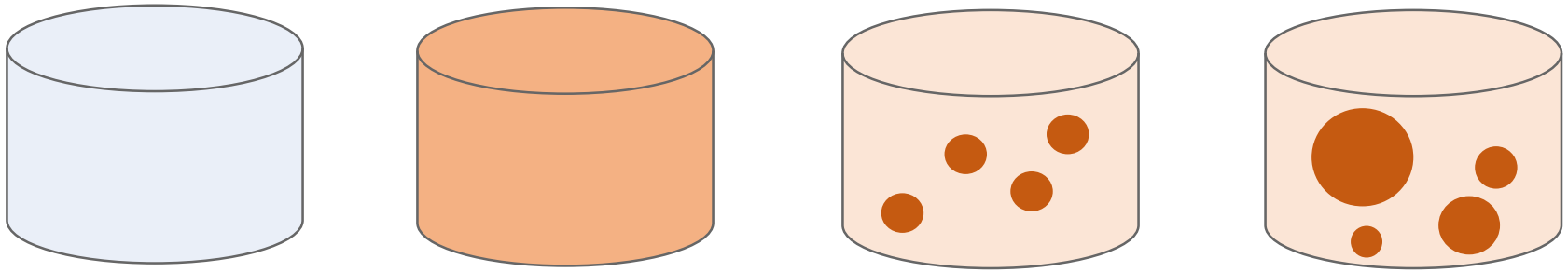
Single Particle ICP-MS (spICP-MS)

- Each nanoparticle gives a transient signal (a plume of ions generated from the particle)
- Use time resolved data acquisition and analysis
- Measure particle concentration, particle effective diameter and composition



What is sNP analysis?

➢ Sample Types for single nanoparticle analysis



Ionic Blank

- Blank sample

Ionic Standard

- Dissolved metal solution

- Used to calibrate response factor(s).

Reference Material

- Metal suspension liquid*2.
- Known, either known mean particle size or known conc is needed.
- Known conc.
- Used to calibrate nebulizer efficiency (η_n).

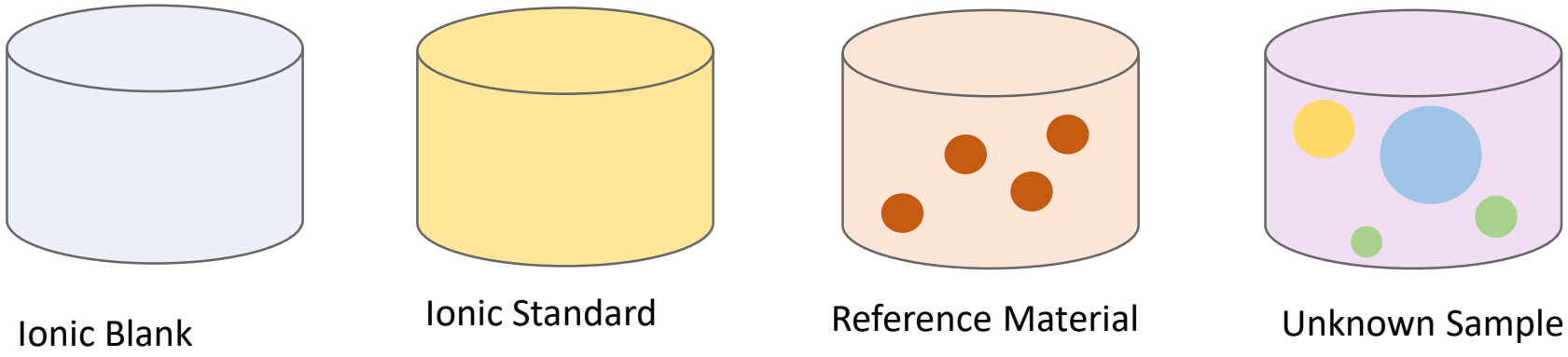
Unknown Sample

- Metal suspension liquid*2.
- To be analyzed
- Unknown particle size and conc.

*1 New sample types dedicated for sNP analsys.
*2 Usually with small amount of dissolved metal as an impurity.

Advanced operation – Multi-Element Screening

- Sample prep: In case number of elements is 2+

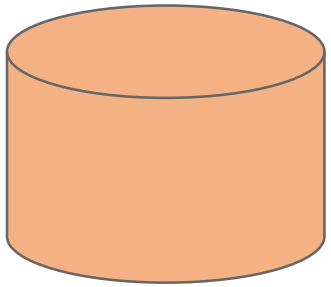


Ionic Blank

Ionic Standard

Reference Material

Unknown Sample



Ionic Standard for
Reference Material

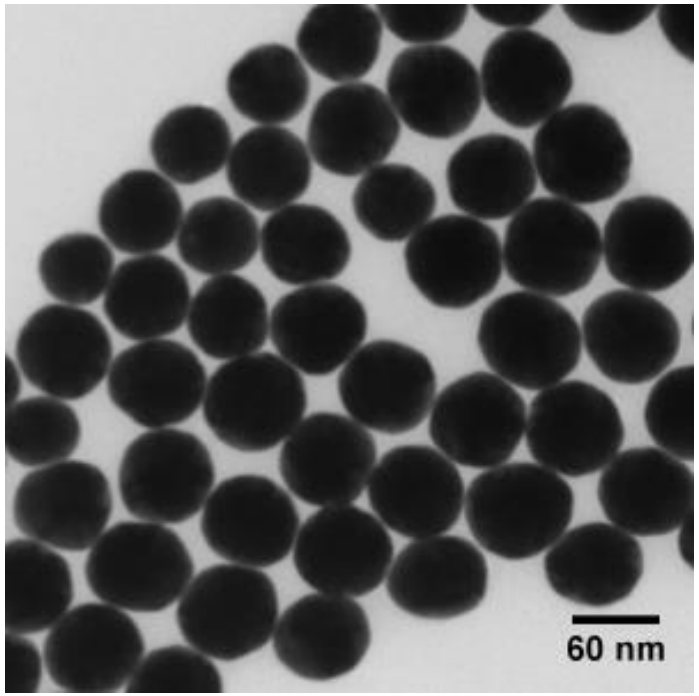
Contains both of
A and B
Such as:
1ppb A + 1ppb B

Element A

Element B

Mixture
of A and B

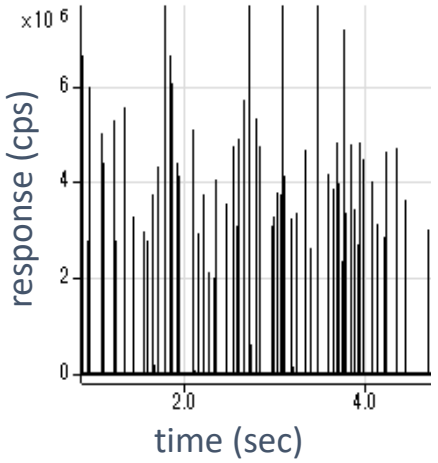
Reference Material



nanoComposix

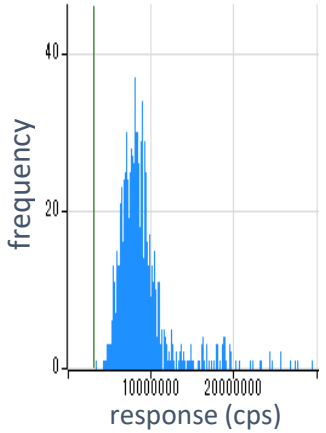
Workflow for NP characterization

ICP-MS TRA data



convert

Response vs frequency

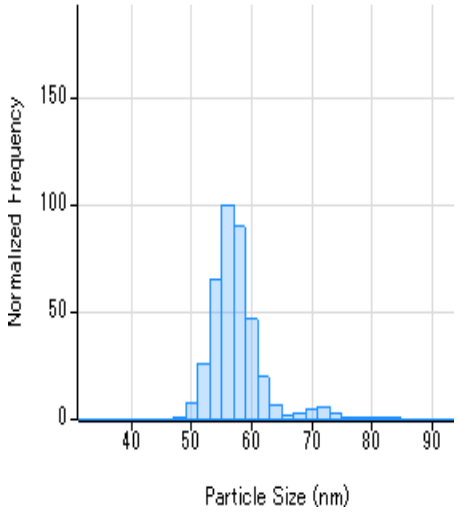


input

- Analyte response factor > Mass of analyte in particle
- Nebulization efficiency (calculated from reference material)
- Analyte density
- Analyte mass fraction in sample particle

calculate

Size distribution



Tabulate and Report

Sample				197 Au						
Data File	Acq. Date-Time	Type	Sample Name	Nebulization Efficiency	# of Particles	Conc. (particles/l)	Conc. (ng/l)	Ionic Conc. (ppb)	BED (nm)	Particle Size (nm)
001IONB.d	5/13/2015 3:10:19 AM	IonicBlk	blank							
002IONS.d	5/13/2015 3:13:07 AM	IonicStd	1ppb							
003_RM.d	5/13/2015 3:15:21 AM	RM	30nm 5ppt	0.071	619	2.5E+7	5.0	0.0178	4.21	27
004SMPL.d	5/13/2015 3:18:20 AM	Sample	60nm 50ppt	0.071	617	2.5E+7	48.6	0.0260	4.73	55
005SMPL.d	5/13/2015 3:20:27 AM	Sample	60nm 50ppt	0.071	681	2.8E+7	51.8	0.0266	4.76	55
006SMPL.d	5/13/2015 3:22:35 AM	Sample	60nm 50ppt	0.071	635	2.6E+7	48.8	0.0258	4.72	55
007SMPL.d	5/13/2015 3:24:26 AM	Sample	60nm 50ppt	0.071	656	2.7E+7	50.4	0.0272	4.80	55
008SMPL.d	5/13/2015 3:28:22 AM	Sample	60nm 50ppt	0.071	649	2.6E+7	48.9	0.0240	4.60	55
009SMPL.d	5/13/2015 3:30:54 AM	Sample	30nm 5ppt	0.071	579	2.3E+7	4.5	0.0083	3.23	26
010SMPL.d	5/13/2015 3:33:29 AM	Sample	30nm 5ppt	0.071	589	2.4E+7	4.8	0.0078	3.16	26
011SMPL.d	5/13/2015 3:37:10 AM	Sample	30nm 5ppt	0.071	568	2.3E+7	4.5	0.0078	3.16	26
012SMPL.d	5/13/2015 3:39:03 AM	Sample	30nm 5ppt	0.071	558	2.3E+7	4.5	0.0076	3.13	26
013SMPL.d	5/13/2015 3:43:05 AM	Sample	30nm 5ppt	0.071	600	2.4E+7	4.7	0.0075	3.12	26

Study 1

J Nanopart Res (2018) 20: 101
https://doi.org/10.1007/s11051-018-4192-8

RESEARCH PAPER

Detection of nanoparticles in edible plant tissues exposed to nano-copper using single-particle ICP-MS

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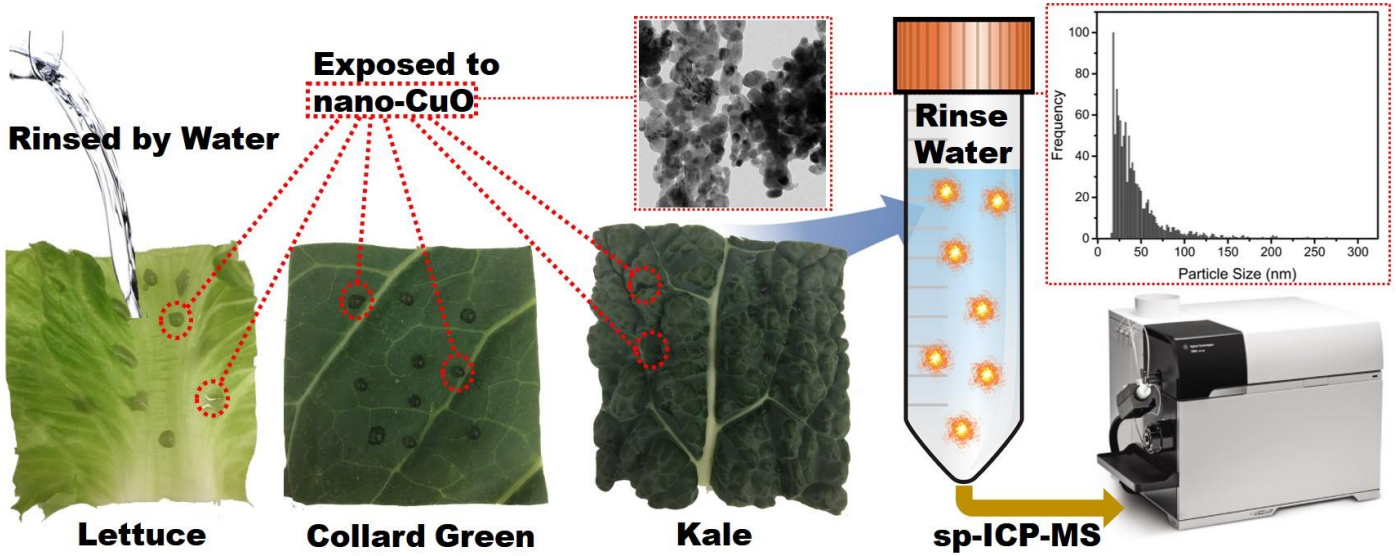
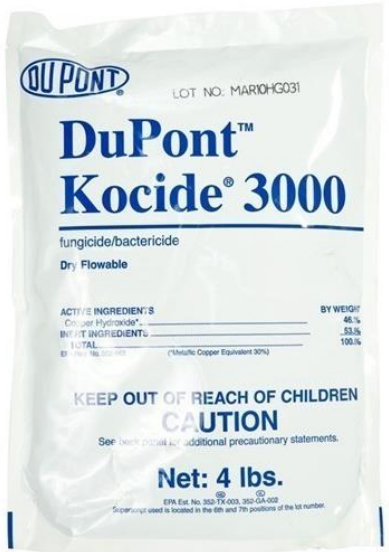



Arturo Keller


Yuxiong Huang

Application of ENMs in Agriculture

- Nano-pesticide/fungicide/bactericide
- Nano-fertilizer



Detect ENMs in leaf tissues via spICP-MS

Organic Vegetables

Lettuce
(Lactuca sativa var. green leaf cultivar)

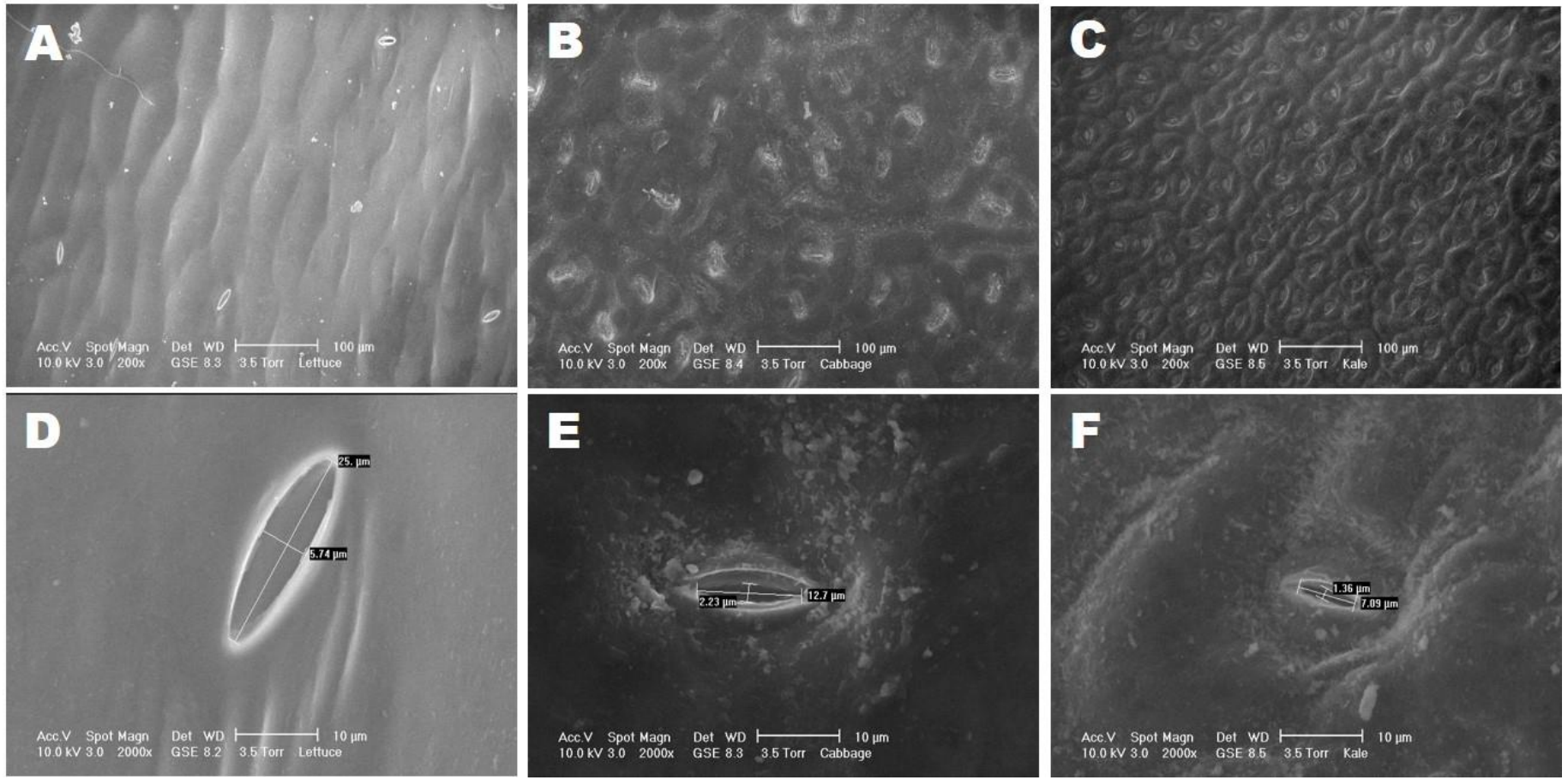
Collard Green
(Lactuca sativa var. green leaf cultivar)

Kale
(*Brassica oleracea, var. Acephala Lacinato)*

Cal-Organic FARMS
Healthy by Choice!
Since 1984

Leaf surface roughness

ESEM

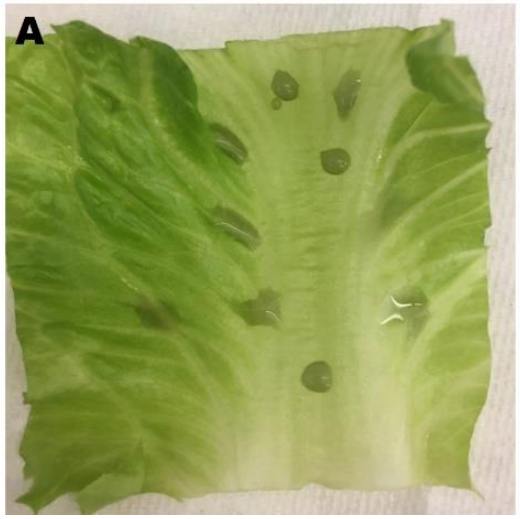


Stomatal opening

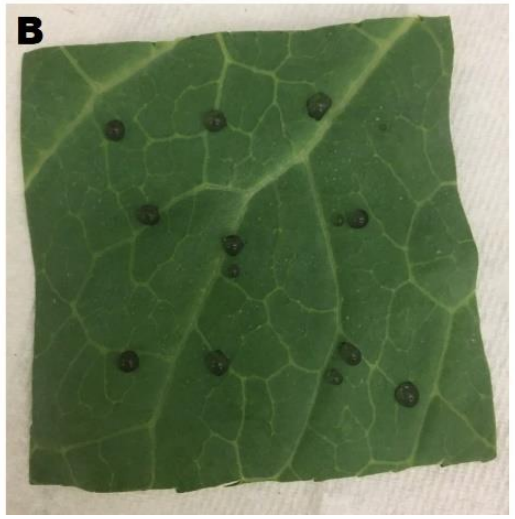
Lettuce **Collard Green** **Kale**

A. A. Keller, Y. Huang, J. Nelson; *J. Nanoparticle Res.* **2018,** *20* (4), 101.

Expose Leaf to nano-CuO



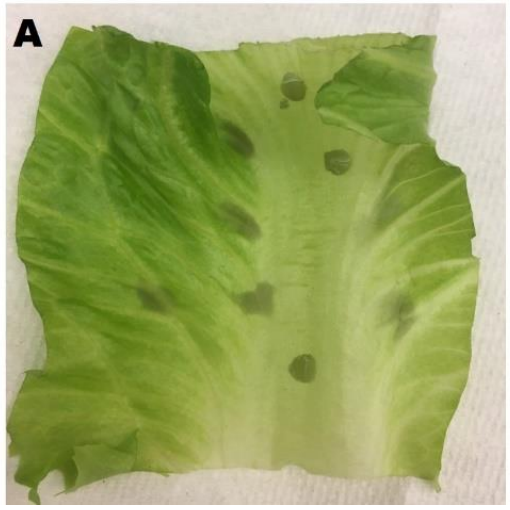
Lettuce

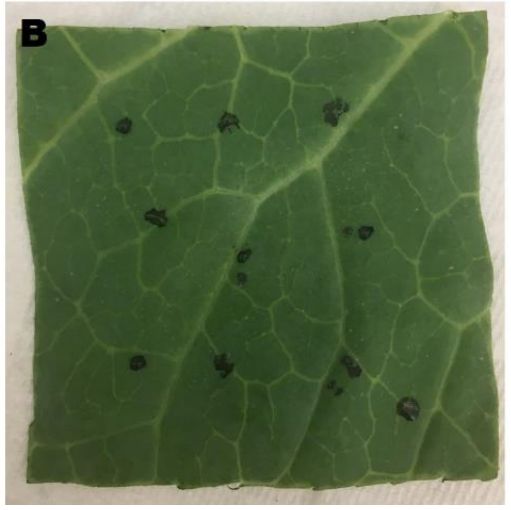


Collard Green

Kale

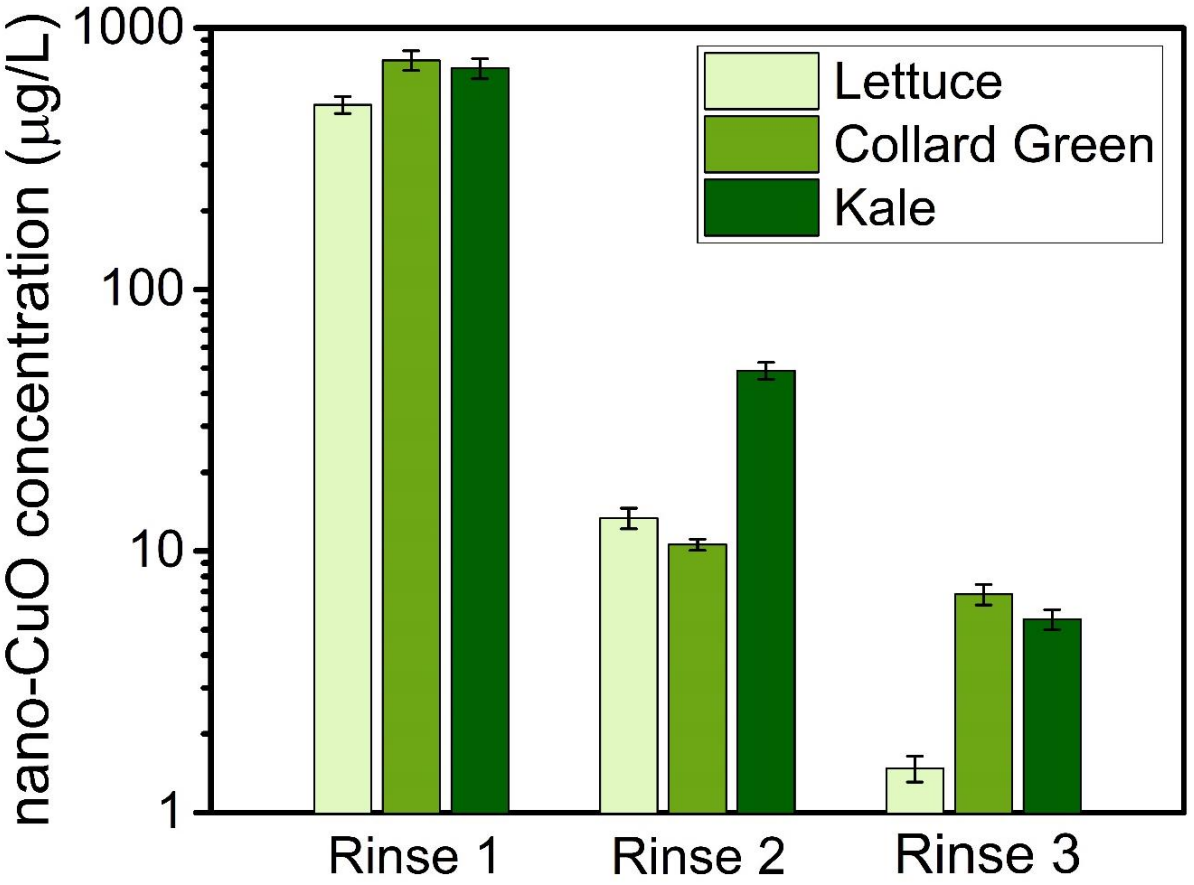
2-hour air dry





Rinse Leaf with DI after nano-CuO exposure

Detect with spICP-MS



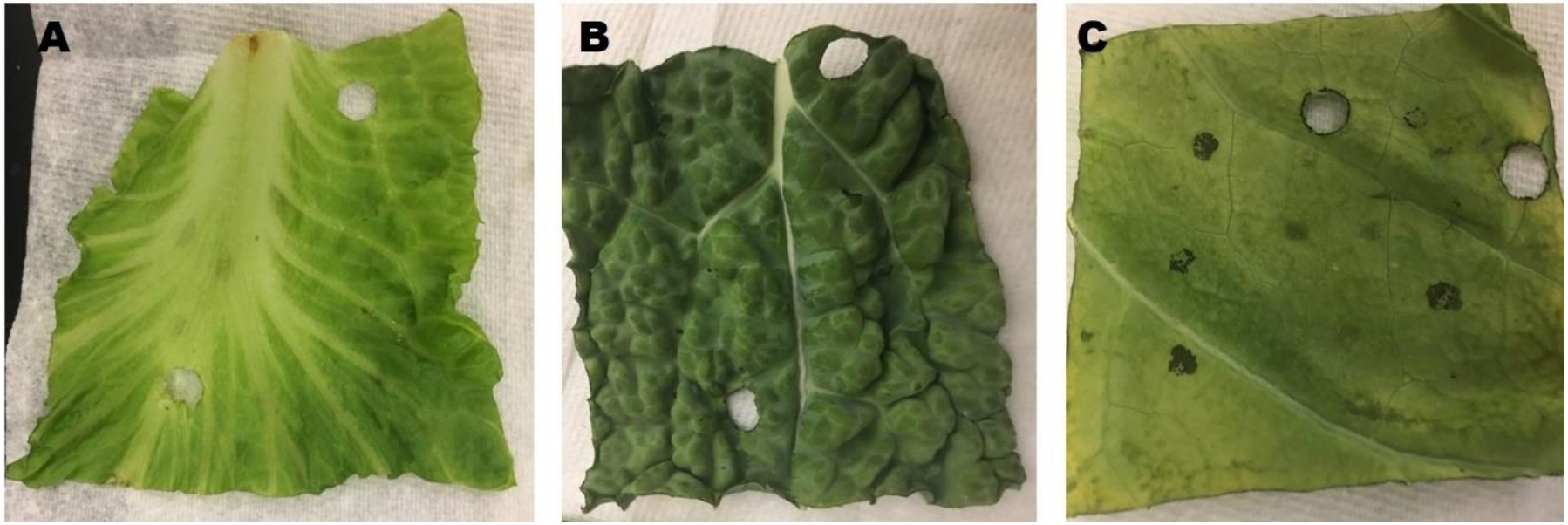
- Concentrations in first rinse around 500-750 µg/L
- Residual washable concentration after 2 rinses is less than 10 µg/L
- Leaf surface roughness may influence residual

Any nano-CuO within the leaf tissues?

Enzymatic digestion

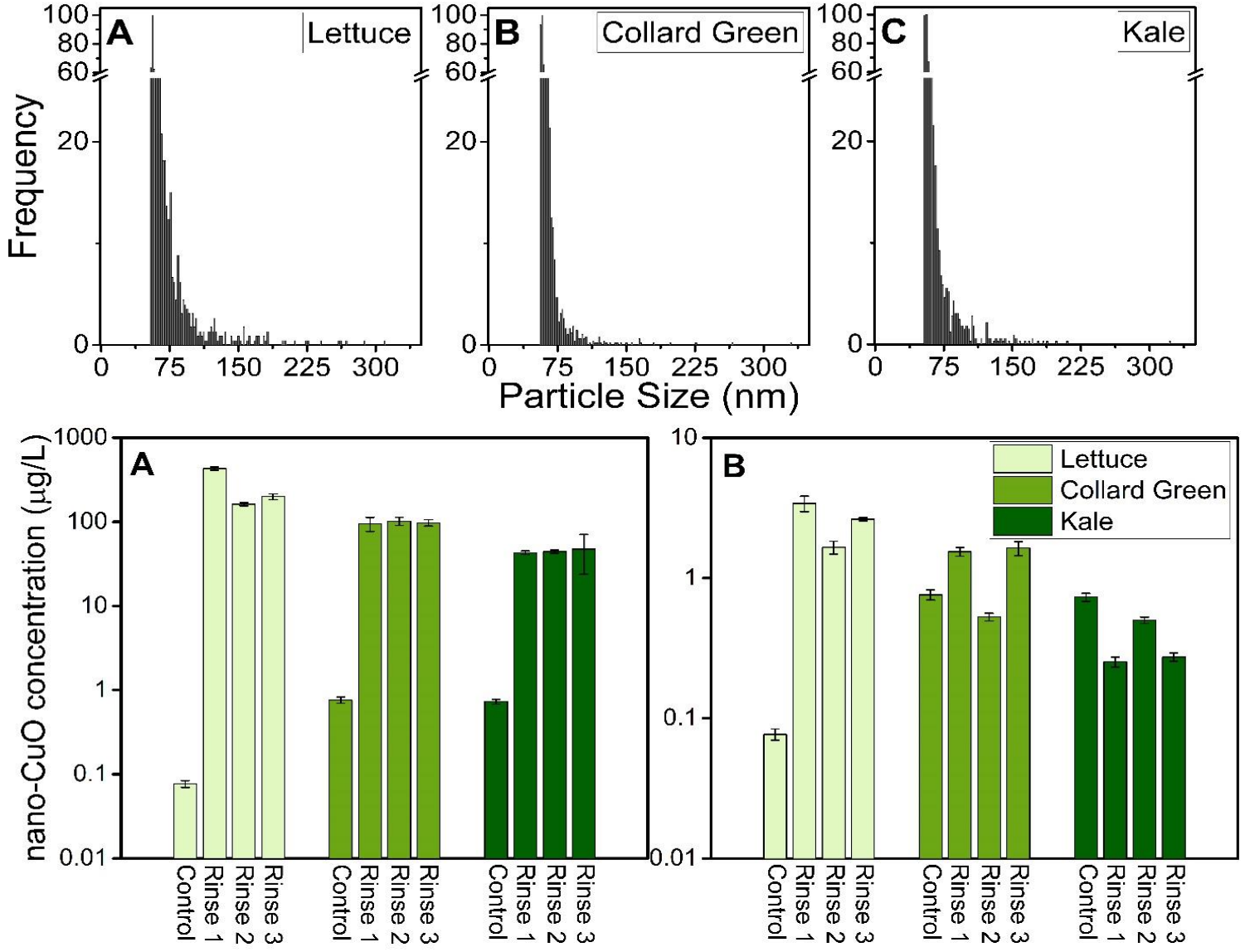
Macerozyme R-10 enzyme

- Mixed with plant tissue samples to digest tissues and release nano-CuO
- 24 hr digestion
- Neutral pH to avoid digesting nano-CuO
- Filtration
- Analyze with spICP-MS



Lettuce Collard Green Kale

Yes! Leaf tissues retain ENMs



Study 2 with Arturo Keller

Environmental Pollution

Research Paper | ENVPOL_2019_5954

Fast Multi-Element Quantification of Nanoparticles in Wastewater and Sludge Using Single Particle ICP-MS

Yuxiong Huang, Arturo Keller, Pabel Cervantes, Jenny Nelson

Submitted 22 Oct 2019

Under Review 24 Oct 2019

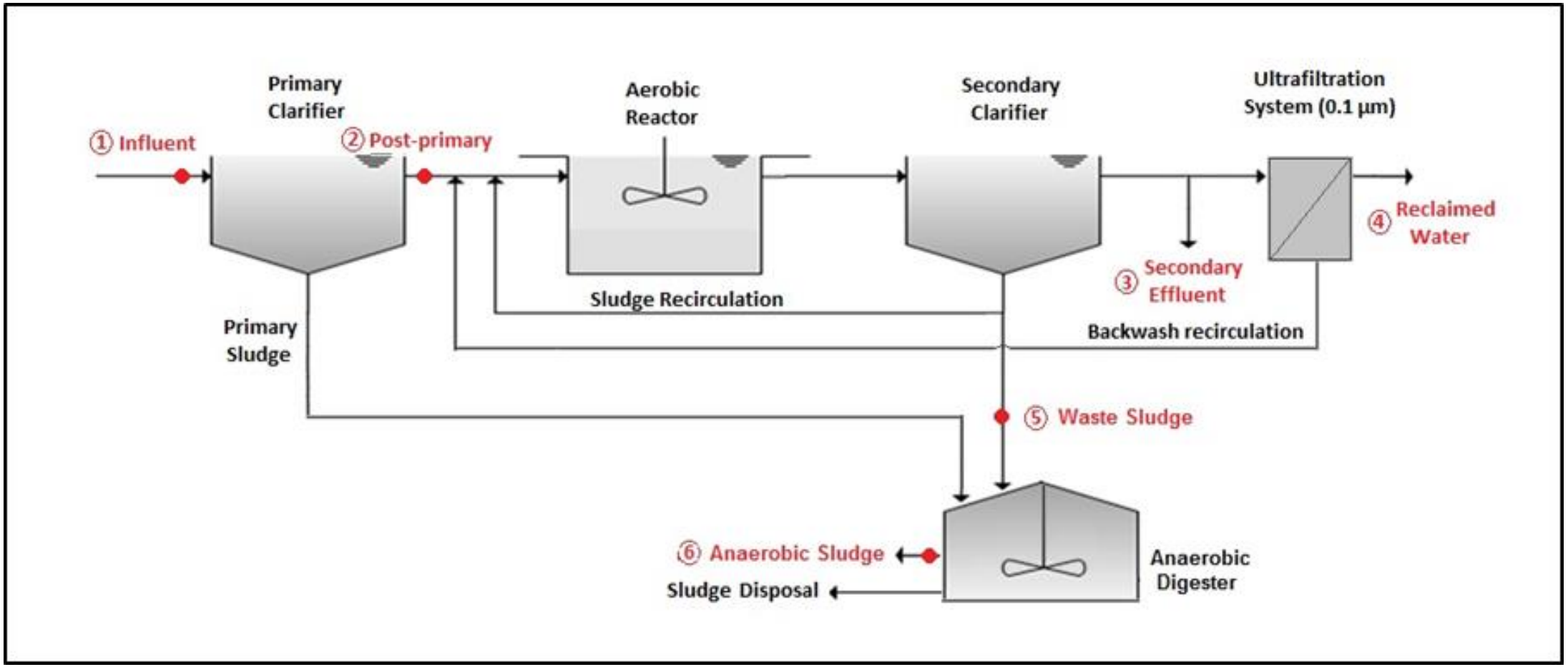
Alert: keep me informed about the submission status for this manuscript

Arturo Keller

Yuxiong Huang

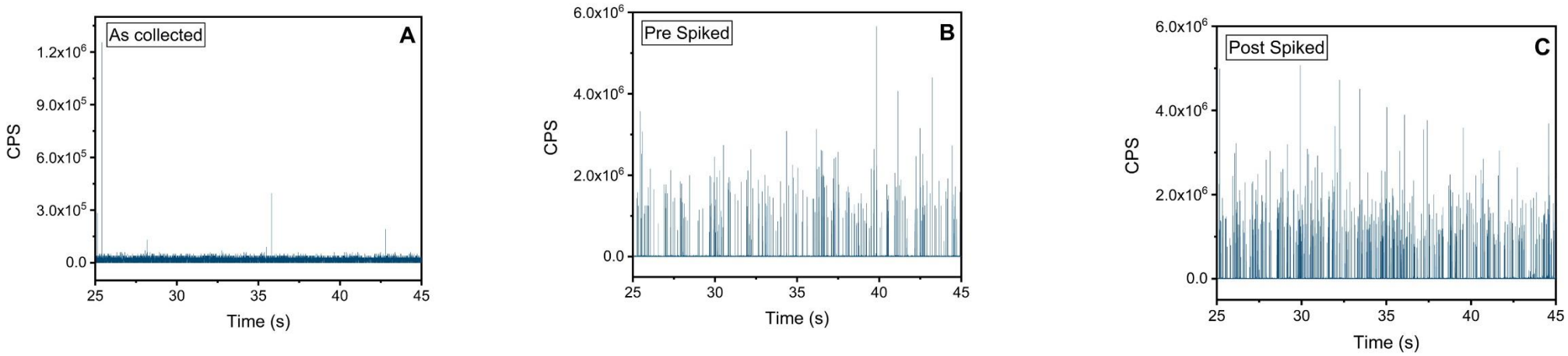
Pabel Cervantes-Avilés

Collecting NP samples in WWTP process

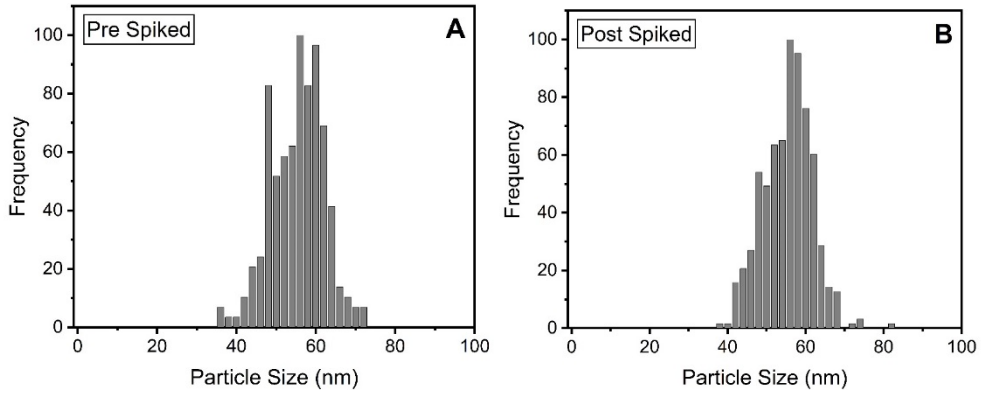


spICP-MS was used to monitor up to 16 elements simultaneously in water and wastewater samples

spICP-MS Measurement

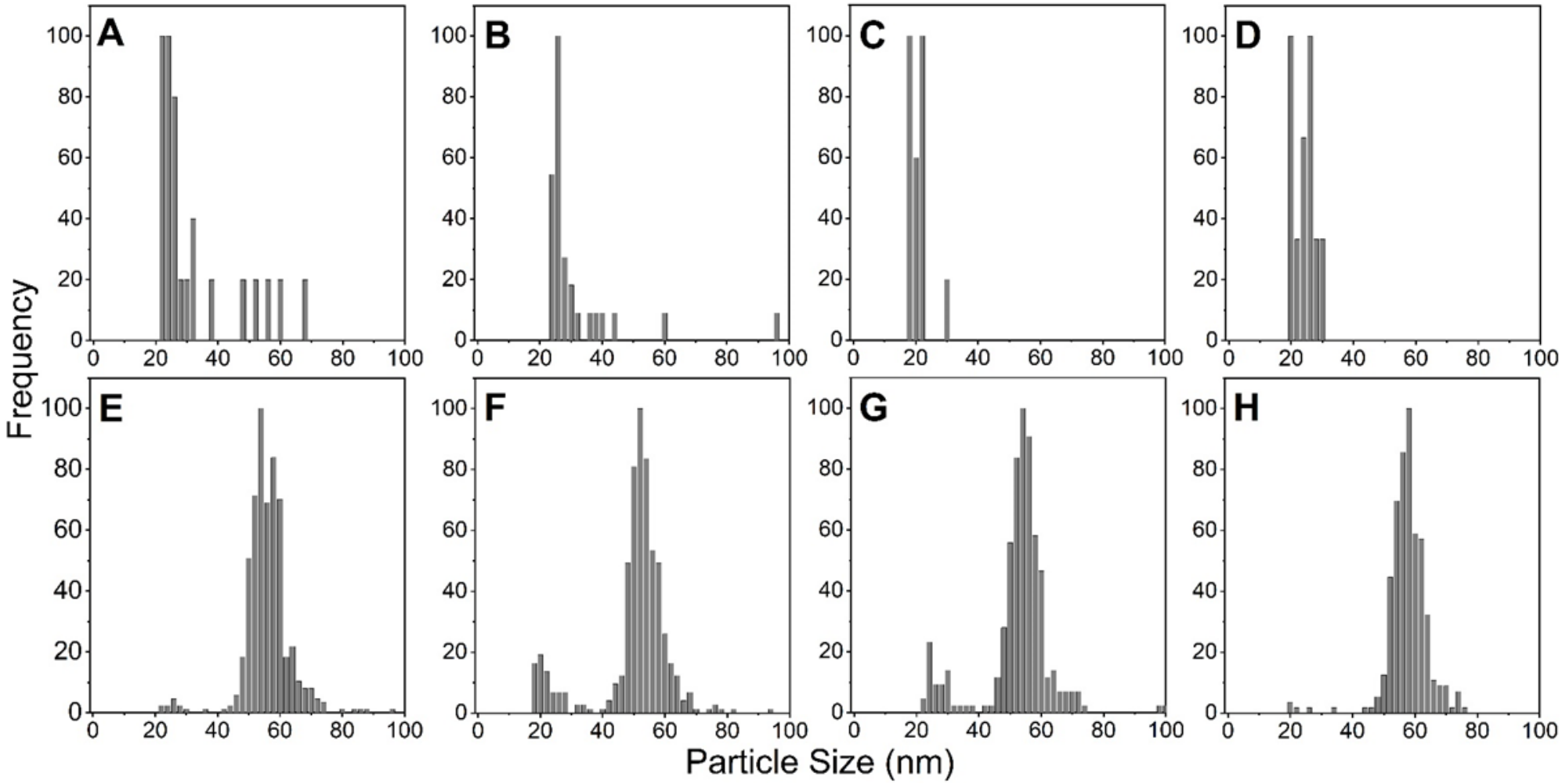


Time resolved data for Au-containing NPs in sludge samples (A) as collected; (B) pre-centrifugation spiked with 60 nm Au NPs; and (C) post-centrifugation spiked with 60 nm Au NPs.



Size distribution of 60 nm Au NPs reference materials spiked at 100 ng/L detected in the retentate of activated waste sludge

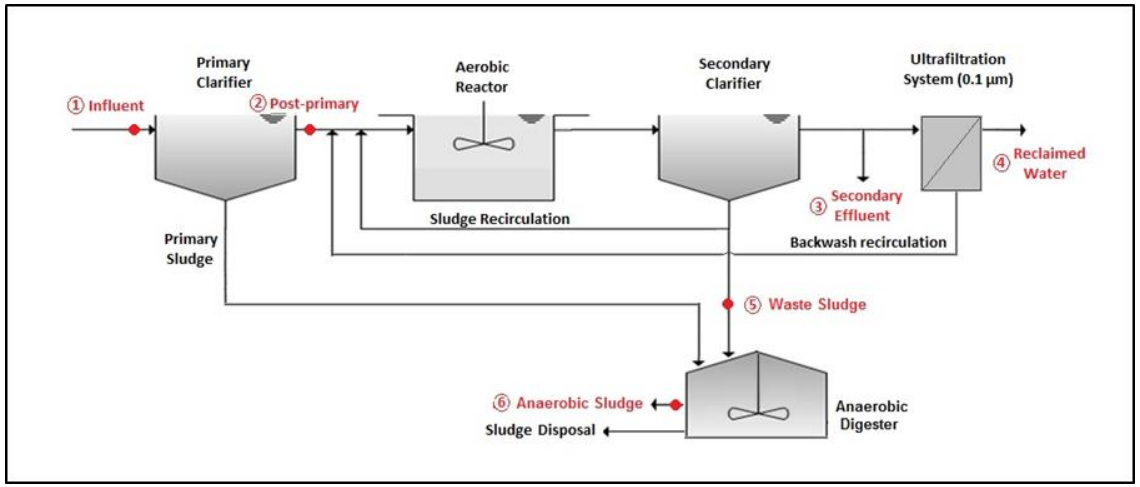
spICP-MS Measurement

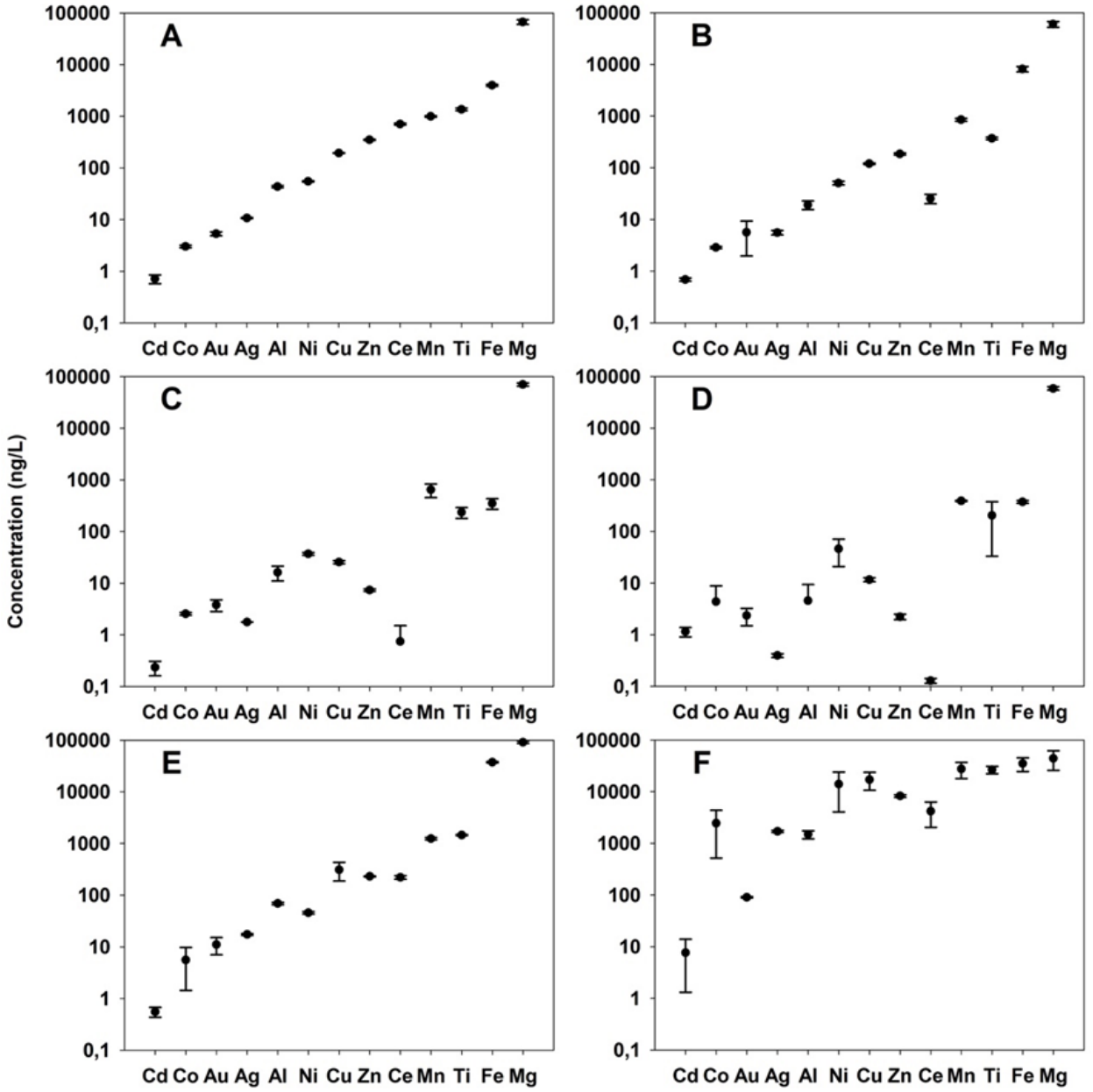


Ag NP size distribution in unspiked (A) influent; (B) post-primary; (C) waste sludge; (D) anaerobic sludge; and in spiked (E) influent; (F) post-primary; (G) waste sludge; (H) anaerobic sludge.

Mass concentration of NPs at different stages of wastewater treatment

(A) influent; (B) post-primary; (C) secondary effluent; (D) reclaimed water; and in spiked (E) waste sludge; (F) anaerobic sludge.



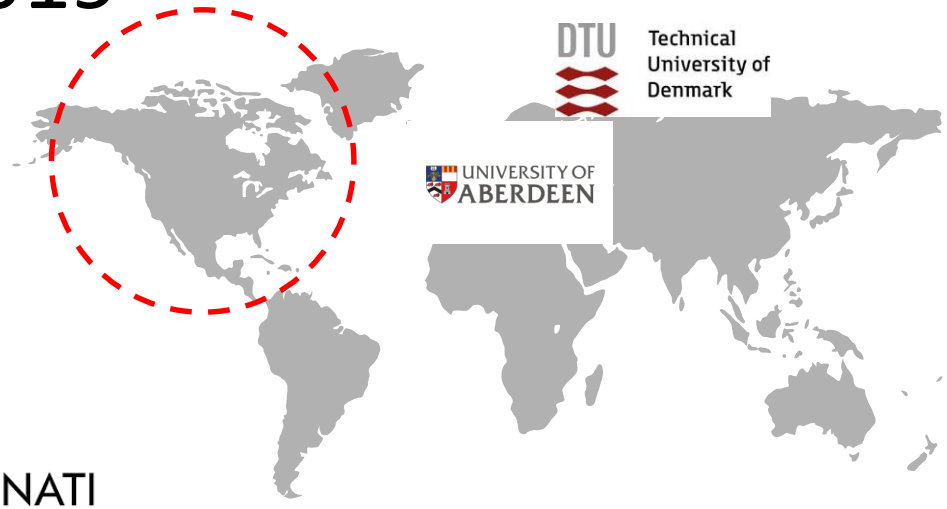


Conclusions

- You need to read the papers to get the entire spICPMS NPs story for both studies
- spICP-MS offers an excellent approach for quantitative analysis of nanoparticles
- spICPMS provides concentration, size distribution, composition, dissolved ion concentration
- Can be applied to water and wastewater samples

- Challenges remain with regards to samples that contain high levels of natural NPs

My Collaboration Partners from 2012-2019



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UNIVERSITY OF ABERDEEN

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NEW BELGIUM BREWING

Chevron

University of CINCINNATI

Abbott Nutrition

NREL
NATIONAL RENEWABLE ENERGY LABORATORY

IFSH
INSTITUTE FOR FOOD SAFETY AND HEALTH

FDA U.S. FOOD & DRUG ADMINISTRATION

CEM

DARTMOUTH

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Agrolab
Laboratorio
Inocuidad · Food safety

Acknowledgments

ARTURO KELLER

Yuxiong Huang, now Prof. at TBSI @ Shenzen

Pabel Cervantes, postdoc at UCSB

Adeyemi S Adeleye, now Prof. at UCI

Tarun Anumol, Agilent Technologies

Chevron

Agilent Technologies

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