An Exploration of Some Capabilities and Limitations of spICP-MS

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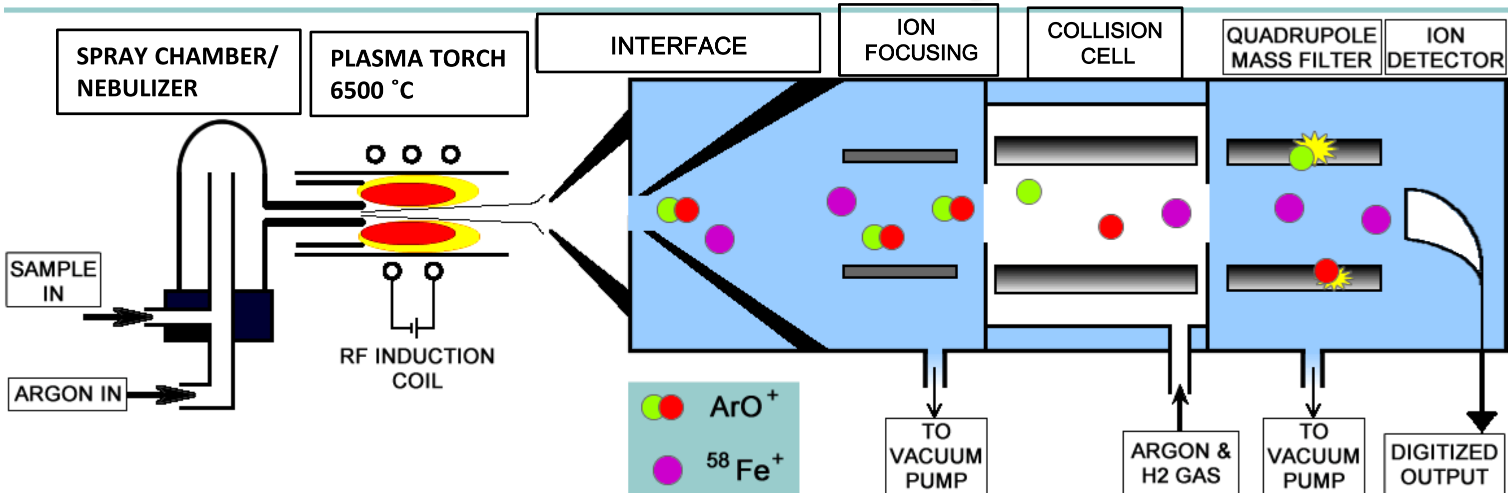
July 7, 2015
Workshop on Quantifying Exposure to Engineered Nanomaterials (QEEN) from Manufactured Products
Arlington, Virginia

National Institute of Standards and Technology
U.S. Department of Commerce

Inductively Coupled Plasma Mass Spectrometry

Powerful tool for elemental analysis

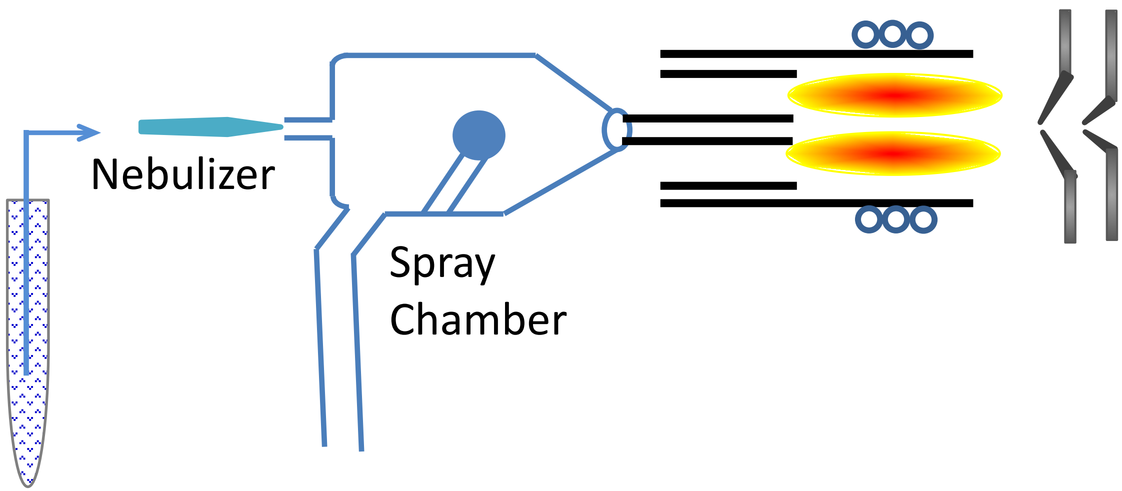


- ✓ Extremely high sensitivity and elemental specificity
- ✓ Multi-elemental capabilities
- ✓ Direct isotopic information
- ✓ Easy coupling with different separation techniques

and when operated in single particle mode…

- ✓ Particle Size and Size Distribution
- ✓ Particle Number Concentration
- ✓ Ionic Content

Single Particle (sp)ICP-MS



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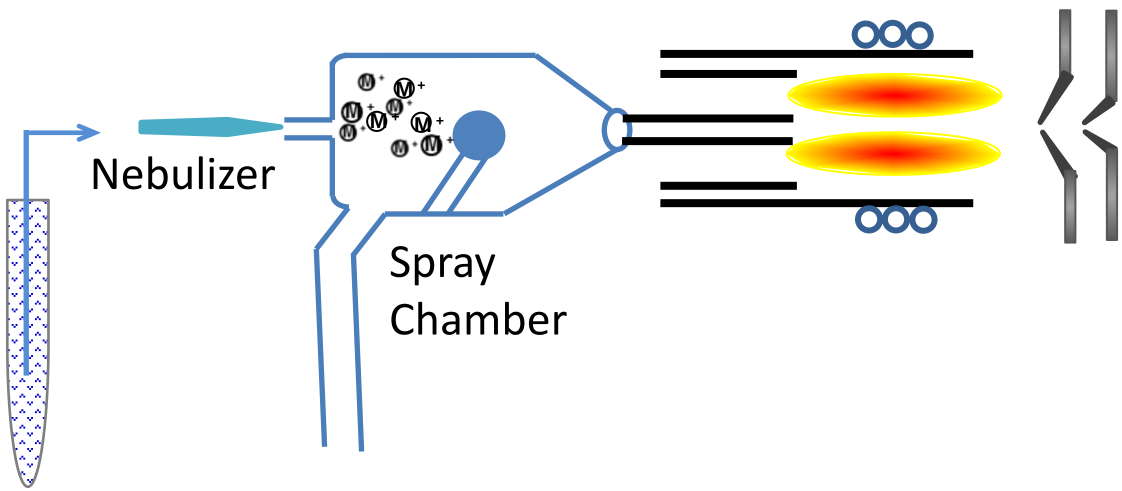

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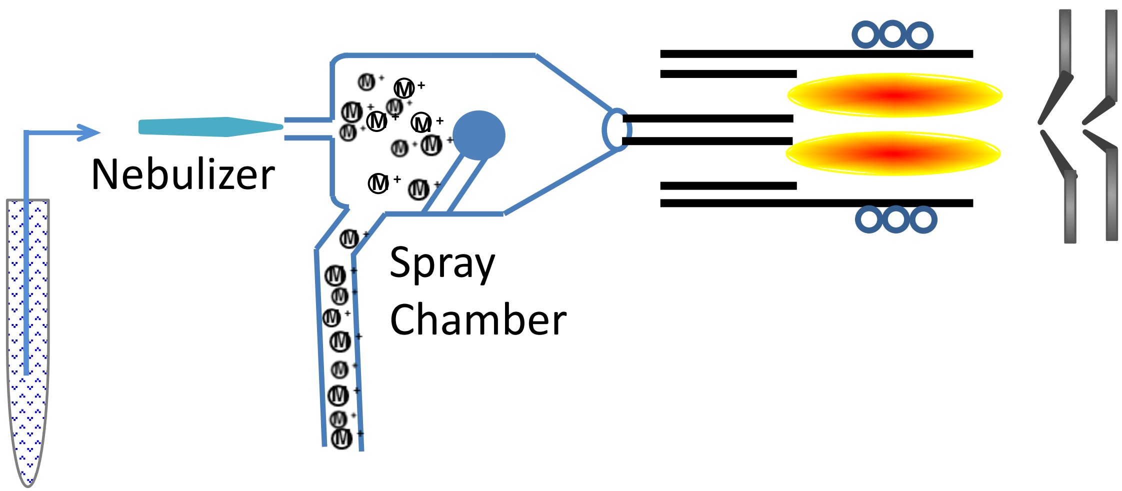

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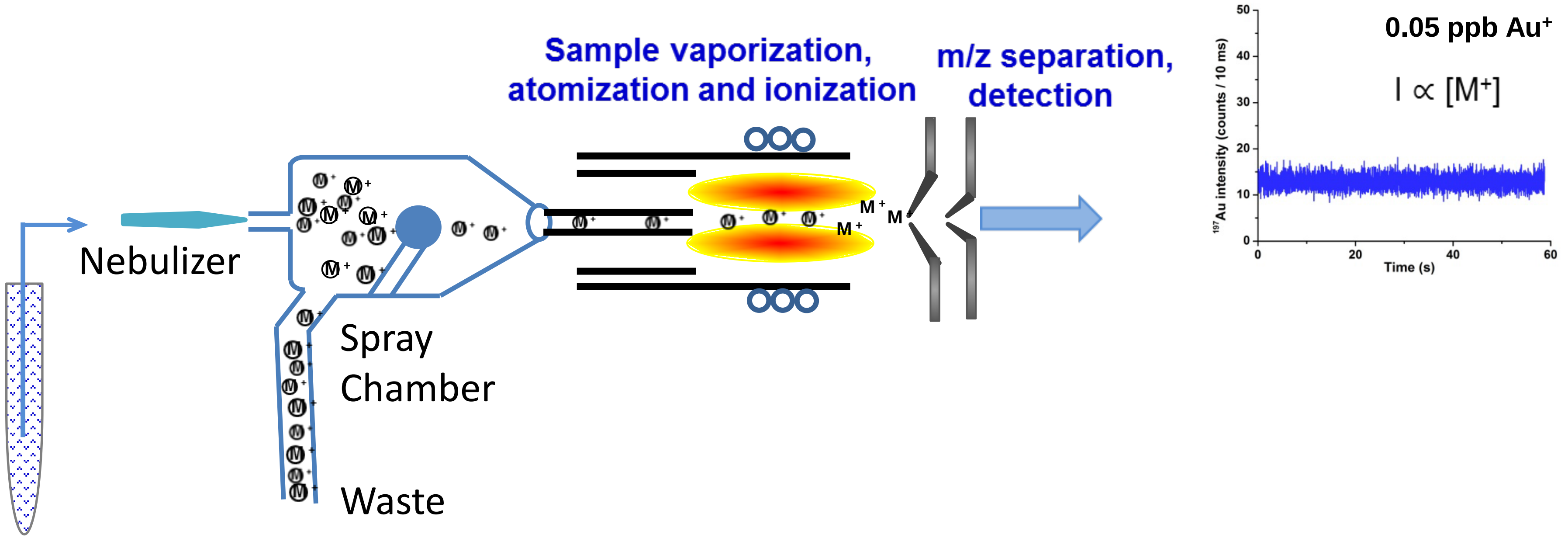

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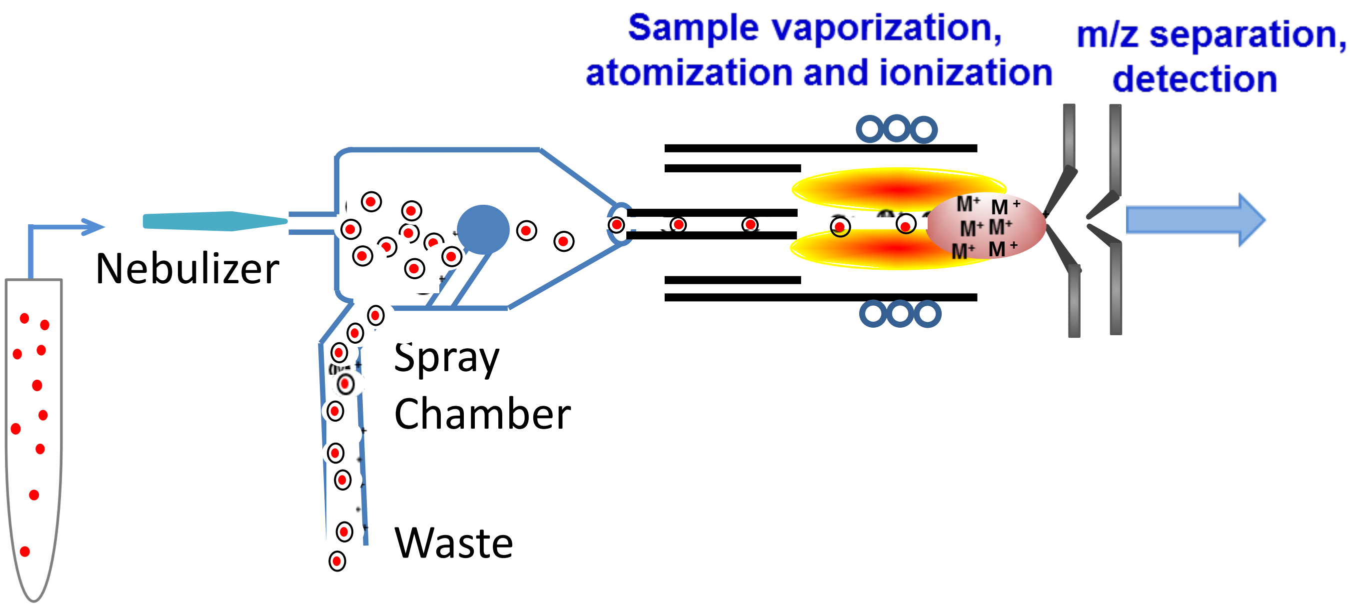
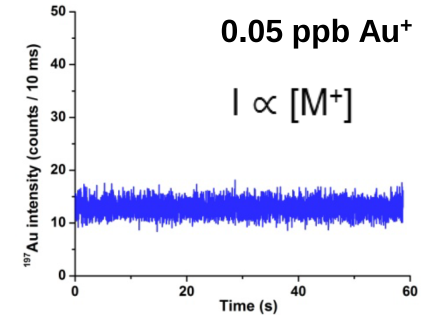
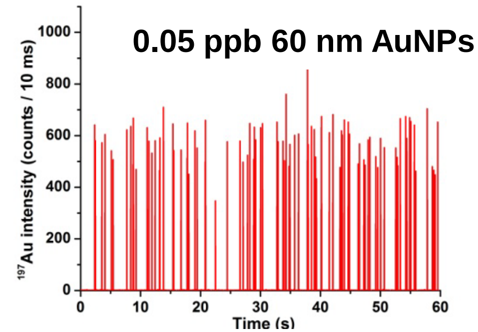






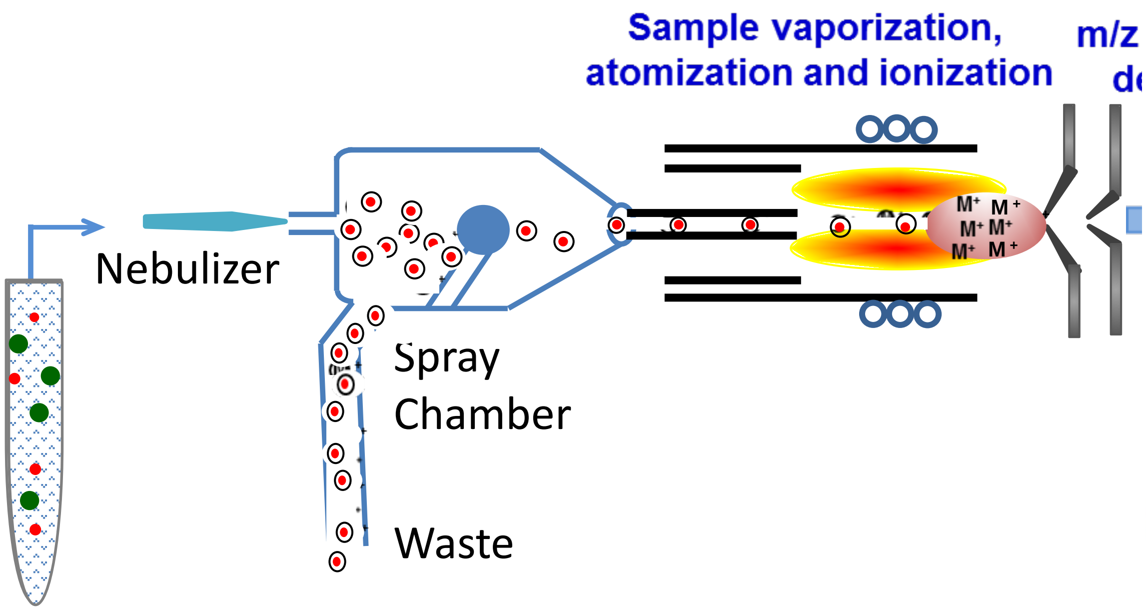
N of spikes ∝ particle N concentration

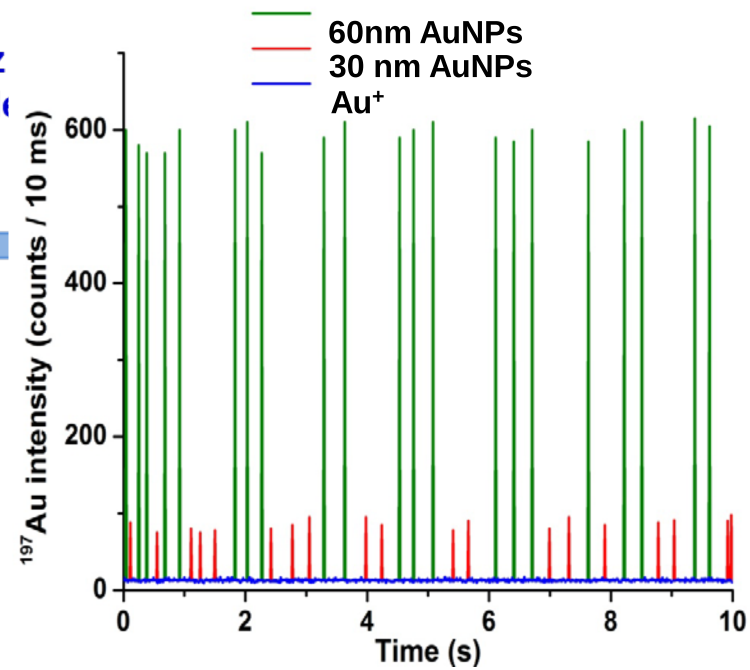
$I \propto m_p$

$m_p = \pi d^3 \rho / 6$

$d_p = \sqrt[3]{(6 m_p / \pi \rho)}$

Single Particle (sp)ICP-MS





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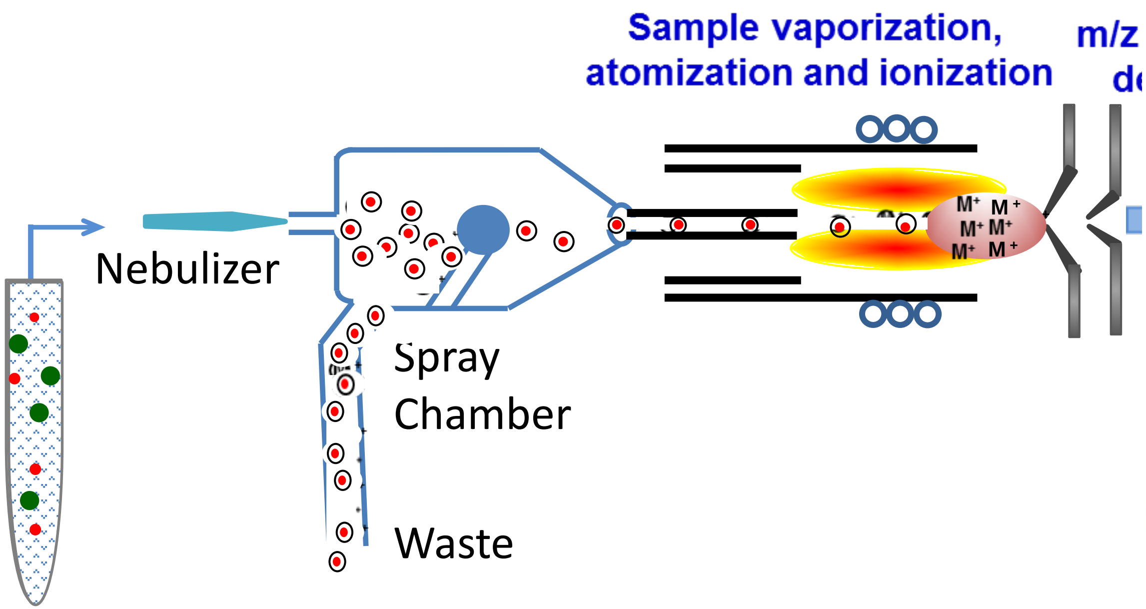
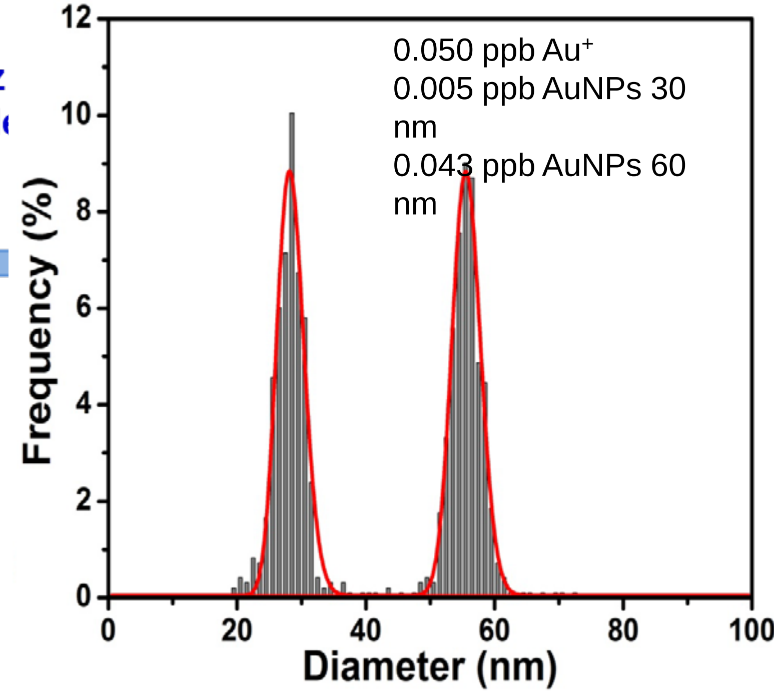




- Dwell time: 10 ms
- Flux, 100 – 135 particles per min; 3 to 6 min
  - 18, 0000 /mL
- Particle pulses distinguished from the background using a five times standard deviation (5σ) criterion (Tuoriniemi, J., Cornelis, G., Hassellov, M., *Anal. Chem.* (2012) 84:3965-3972.)

N of spikes ∝ particle N concentration

$I \propto m_p$

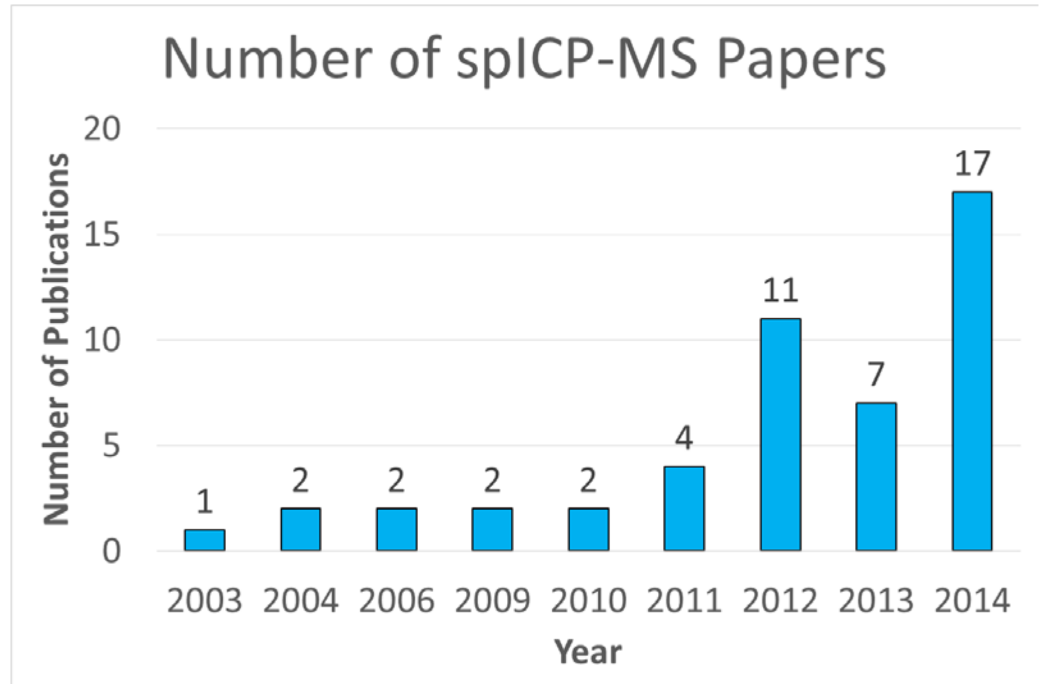
$m_p = \pi d^3 \rho / 6$

$d_p = \sqrt[3]{(6 m_p / \pi \rho)}$

Outline

Method validation is required

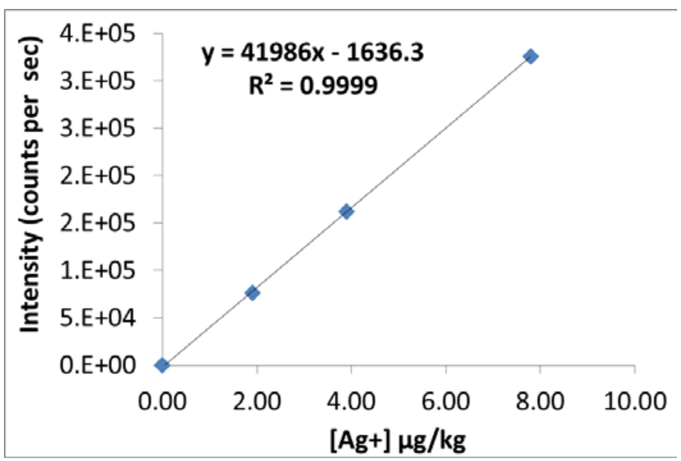
- Reproducibility and accuracy of particle size, particle size distribution and dissolved silver (Ag) content of NIST RM 8017 PVP stabilized AgNPs
    - Kragten Spreadsheet approach to examine and combine uncertainty components
- Size distribution of gold (Au) NPs in several commercially available suspensions
    - Validation with high-resolution scanning electron microscopy (HR-SEM)
- Detection of nanosilver in textiles



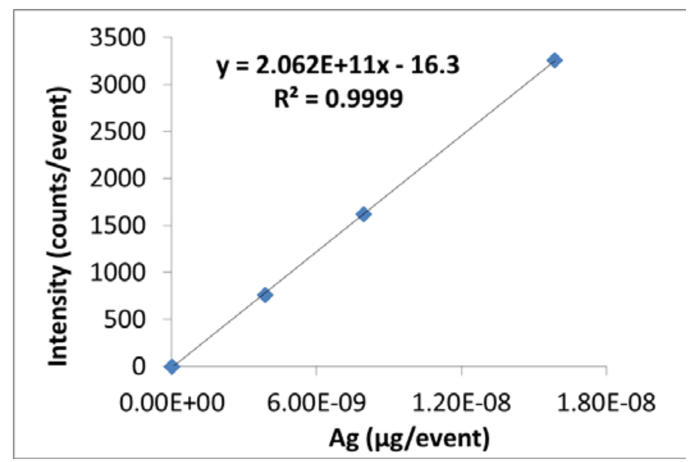
Calibration of mass/size by spICP-MS

- Nanoparticle reference materials
  - Few available
- Micro droplet calibration
  - Requires specialized instrumentation
- Standard solution calibration
  - Relatively easy to implement…

Pace H. E., Rogers N. J., Jarolimek C., Coleman V. A., Higgins C. P., Ranville J. F., *Anal. Chem.* (2011) 83:9361–9369



$$C * q_{liq} * t_{dwell} * \eta_n$$



- Requires accurate transport efficiency

Impact of Transport Efficiency

Kragten spreadsheet – a tool to calculate the uncertainty of a measurement

Quantity Description	Quantity Name, Symbol	Quantity Value	Standard Uncertainty, u_i	Type of Uncertainty Assessment	Relative u_i (%)	Degrees of Freedom, ν_i
Meas.Transport Efficiency	η_n	5.00	0.10	B	2.00%	999999999
C Ag+ solution (µg/kg)	$C\ Ag^+$	7.953	0.040	A	0.50%	999999999
Uptake rate (g/min)	q_{liq}	0.1743	0.00048	A	0.28%	3
Measured signal of Ag+ Stnd soln (cour	$I\ Ag^+$	2,226.9	1.46	A	0.07%	17958
Bkg counts per dwell, water	$I_{bkg}\ Ag^+$	0.86	0.34	A	39.43%	17978
Measured signal of Ag NP in AgNP Unk	I_p AgNP *unkn*	376.67	11.47	A	3.05%	361
Bkg counts per dwell, AgNP unknown	I_{bkg} Ag	2.39	0.12	A	5.04%	17600
Density Ag (g/cm3)	ρ Ag	10.49	0.0064	B	0.06%	999999999

		u(ηn)	u(C Ag+)	u(qliq)	u(I Ag+)	u(Ibkg Ag+)	(Ip AgNP unkn	u(Ibkg Ag)	u(ρ Ag)	
		0.10	0.040	0.00048	1.46	0.34	11.47	0.12	0.0064	
	ν_i	999999999	999999999	3	17958	17978	361	17600	999999999	
Quantity Name, Symbol	**Quantity Value**	**Quantity Value + u_i**								
ηn	5.00037	5.10037	5.00037	5.00037	5.00037	5.00037	5.00037	5.00037	5.00037	
C Ag+	7.95254	7.95254	7.99224	7.95254	7.95254	7.95254	7.95254	7.95254	7.95254	
qliq	0.17434	0.17434	0.17434	0.17483	0.17434	0.17434	0.17434	0.17434	0.17434	
I Ag+	2226.88364	2226.88364	2226.88364	2226.88364	2228.34000	2226.88364	2226.88364	2226.88364	2226.88364	
Ibkg Ag+	0.86462	0.86462	0.86462	0.86462	0.86462	1.20558	0.86462	0.86462	0.86462	
Ip AgNP unkn	376.67129	376.67129	376.67129	376.67129	376.67129	376.67129	388.14200	376.67129	376.67129	
Ibkg Ag	2.38676	2.38676	2.38676	2.38676	2.38676	2.38676	2.38676	2.50707	2.38676	
ρ Ag	10.49000	10.49000	10.49000	10.49000	10.49000	10.49000	10.49000	10.49000	10.49635	
MF Value, Y	70.72	71.19	70.84	70.79	70.71	70.73	71.44	70.71	70.71	
		0.46833	0.11749	0.06528	-0.01542	0.00361	0.71521	-0.00758	-0.01427	$c_i u_i$
		0.21934	0.01380	0.00426	0.00024	0.00001	0.51153	0.00006	0.00020	$(c_i u_i)^2$
Standard Uncertainty, u_c	0.87	29.3%	1.8%	0.6%	0.03%	0.002%	68.3%	0.01%	0.03%	rel $(c_i u_i)^2$
Degrees of Freedom, ν	768									
Coverage Factor, k	1.96									
Expanded Uncertainty, U	1.70	2.40%								

Impact of Transport Efficiency

Kragten spreadsheet – a tool to calculate the uncertainty of a measurement

Quantity Description	Quantity Name, Symbol	Quantity Value	Standard Uncertainty, u_i	Type of Uncertainty Assessment	Relative u_i (%)	Degrees of Freedom, ν_i
Meas.Transport Efficiency	η_n	5.00	0.50	B	10.00%	999999999
C Ag+ solution (µg/kg)	$C\ Ag^+$	7.953	0.040	A	0.50%	999999999
Uptake rate (g/min)	q_{liq}	0.1743	0.00048	A	0.28%	3
Measured signal of Ag+ Stn	$I\ Ag^+$	2,226.9	1.46	A	0.07%	17958
Bkg counts per dwell, wate	$I_{bkg}\ Ag^+$	0.86	0.34	A	39.43%	17978
Measured signal of Ag NP i	I_p AgNP *unkn*	376.67	11.47	A	3.05%	361
Bkg counts per dwell, AgNF	I_{bkg} Ag	2.39	0.12	A	5.04%	17600
Density Ag (g/cm3)	ρ Ag	10.49	0.0064	B	0.06%	999999999

		u(ηn)	u(C Ag+)	u(qliq)	u(I Ag+)	u(Ibkg Ag+)	u(Ip AgNP unkn	u(Ibkg Ag)	u(ρ Ag)	
		0.10	0.040	0.00048	1.46	0.34	11.47	0.12	0.0064	
	ν_i	999999999	999999999	3	17958	17978	361	17600	999999999	
Quantity Name, Symbol	**Quantity Value**	**Quantity Value + u_i**								
ηn	5.00037	5.10037	5.00037	5.00037	5.00037	5.00037	5.00037	5.00037	5.00037	
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Ibkg Ag+	0.86462	0.86462	0.86462	0.86462	0.86462	1.20558	0.86462	0.86462	0.86462	
Ip AgNP unkn	376.67129	376.67129	376.67129	376.67129	376.67129	376.67129	388.14200	376.67129	376.67129	
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Standard Uncertainty, u_c	0.87	29.3%	1.8%	0.6%	0.03%	0.002%	68.3%	0.01%	0.03%	rel $(c_i u_i)^2$
Degrees of Freedom, ν	768									
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Expanded Uncertainty, U	1.70	2.40%								

Impact of Transport Efficiency

Kragten spreadsheet – a tool to calculate the uncertainty of a measurement

Quantity Description	Quantity Name, Symbol	Quantity Value	Standard Uncertainty, u_i	Type of Uncertainty Assessment	Relative u_i (%)	Degrees of Freedom, ν_i
Meas.Transport Efficiency	η_n	5.00	0.50	B	10.00%	999999999
C Ag+ solution (µg/kg)	$C\ Ag^+$	7.953	0.040	A	0.50%	999999999
Uptake rate (g/min)	q_{liq}	0.1743	0.00048	A	0.28%	3
Measured signal of Ag+ Stn	$I\ Ag^+$	2,226.9	1.46	A	0.07%	17958
Bkg counts per dwell, wate	$I_{bkg}\ Ag^+$	0.86	0.34	A	39.43%	17978
Measured signal of Ag NP i	I_p AgNP *unkn*	376.67	11.47	A	3.05%	361
Bkg counts per dwell, AgNF	I_{bkg} Ag	2.39	0.12	A	5.04%	17600
Density Ag (g/cm3)	ρ Ag	10.49	0.0064	B	0.06%	999999999

		u(ηn)	*u(C Ag+)*	*u(qliq)*	*u(I Ag+)*	*u(Ibkg Ag+)*	*(Ip AgNP unkn*	*u(Ibkg Ag)*	*u(ρ Ag)*
		0.50	0.040	0.00048	1.46	0.34	11.47	0.12	0.0064
	ν_i	999999999	999999999	3	17958	17978	361	17600	999999999
Quantity Name, Symbol	**Quantity Value**	**Quantity Value + u_i**							
ηn	5.00000	5.50000	5.00000	5.00000	5.00000	5.00000	5.00000	5.00000	5.00000
C Ag+	7.95254	7.95254	7.99224	7.95254	7.95254	7.95254	7.95254	7.95254	7.95254
qliq	0.17434	0.17434	0.17434	0.17483	0.17434	0.17434	0.17434	0.17434	0.17434
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MF Value, Y	70.72	73.00	70.84	70.79	70.70	70.72	71.44	70.71	70.71
		2.28285	0.11749	0.06528	-0.01542	0.00361	0.71519	-0.00758	-0.01427
		5.21139	0.01380	0.00426	0.00024	0.00001	0.51150	0.00006	0.00020
Standard Uncertainty, u_c	2.40	90.8%	0.2%	0.1%	0.00%	0.000%	8.9%	0.00%	0.00%
Degrees of Freedom, ν	45105								
Coverage Factor, k	1.96								
Expanded Uncertainty, U	4.70	6.64%							

Ways to Measure Transport Efficiency

Particle Size Method   Particle Frequency Method

Pace *et al.*, *Anal. Chem.* (2011) 83:9361

- Require monodispersed NP standard RM size and PNC or analyte concentration

$$\eta_n = \frac{counts\ per\ mass\ Dissolved}{counts\ per\ mass\ NP\ RM} \qquad \eta_n = \frac{f(Ip)}{qliq\ * PNC\ NPRM}$$

- spICP-MS no longer an independent method
- Accuracy dependent on accuracy of assigned particle size and concentration

Size Method:
(56 ± 2.9)nm (5 % rel.)

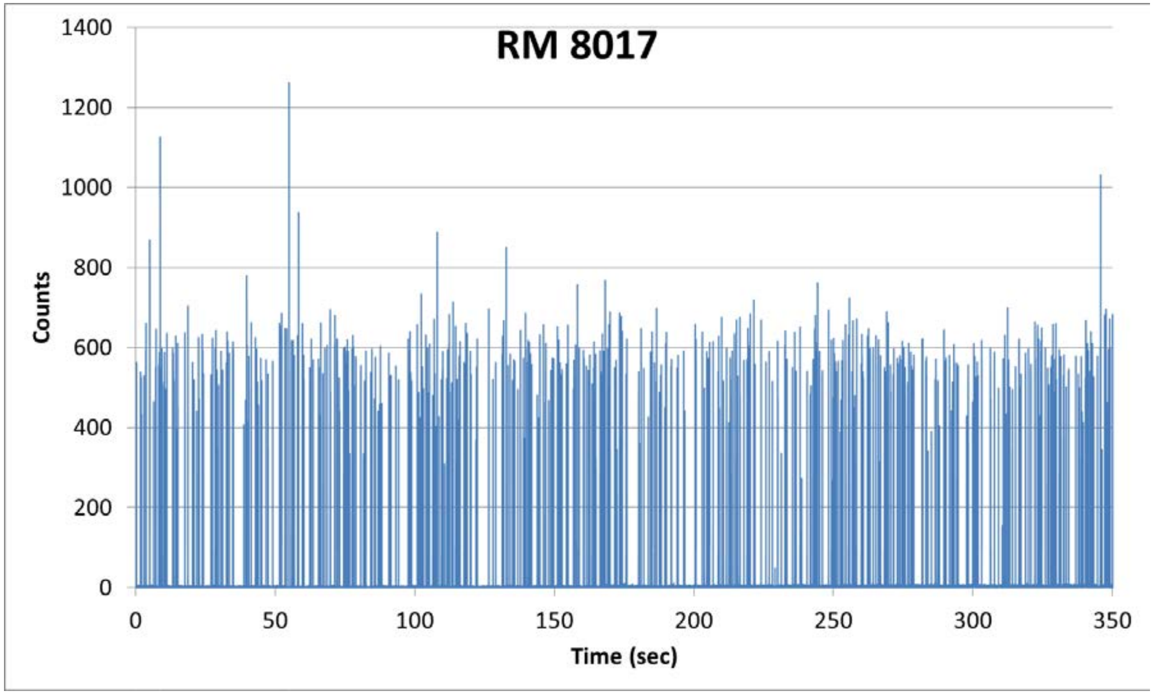
± 16 % rel. u_c
in T.E.

± 10 % rel. U
in spICP-MS diameter
for RM 8017

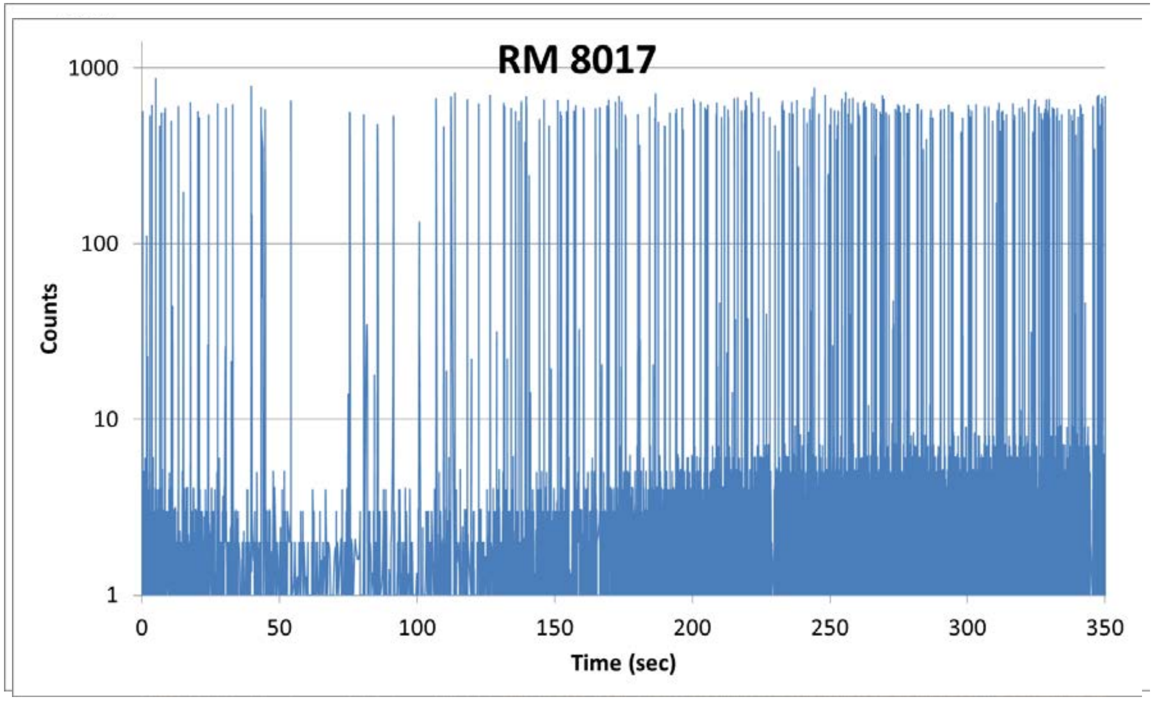
Freq Method:
(56 ± 2.9)nm (5 % rel.)
(51.86 ± 0.32) µg/g
(0.6 % rel.)

RM8013 Technique	Particle Size,nm	Uncertainty (u_c)
AFM	55.4	0.15
SEM	54.9	0.20
TEM	56.0	0.25
DMA	56.3	0.75
DLS (BS 173°)	56.6	0.70
SAXS	53.2	2.7
Combined u_c : 2.9		

Reproducibility of spICP-MS Measurements of Size and Dissolved Ag Content

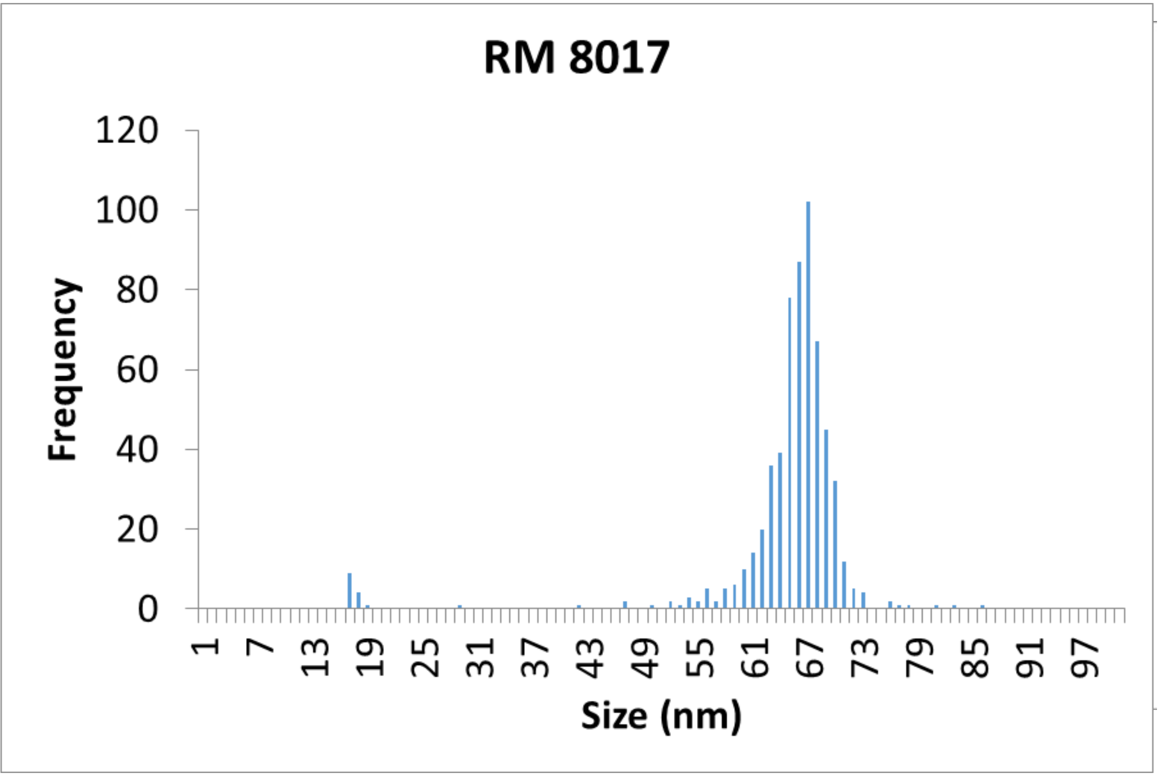


Reproducibility of spICP-MS Measurements of Size and Dissolved Ag Content



- 2.5E07 fold dilution of RM 8017 for spICP-MS analysis
  - dilutes ionic signal

Reproducibility of spICP-MS Measurements of Size and Dissolved Ag Content

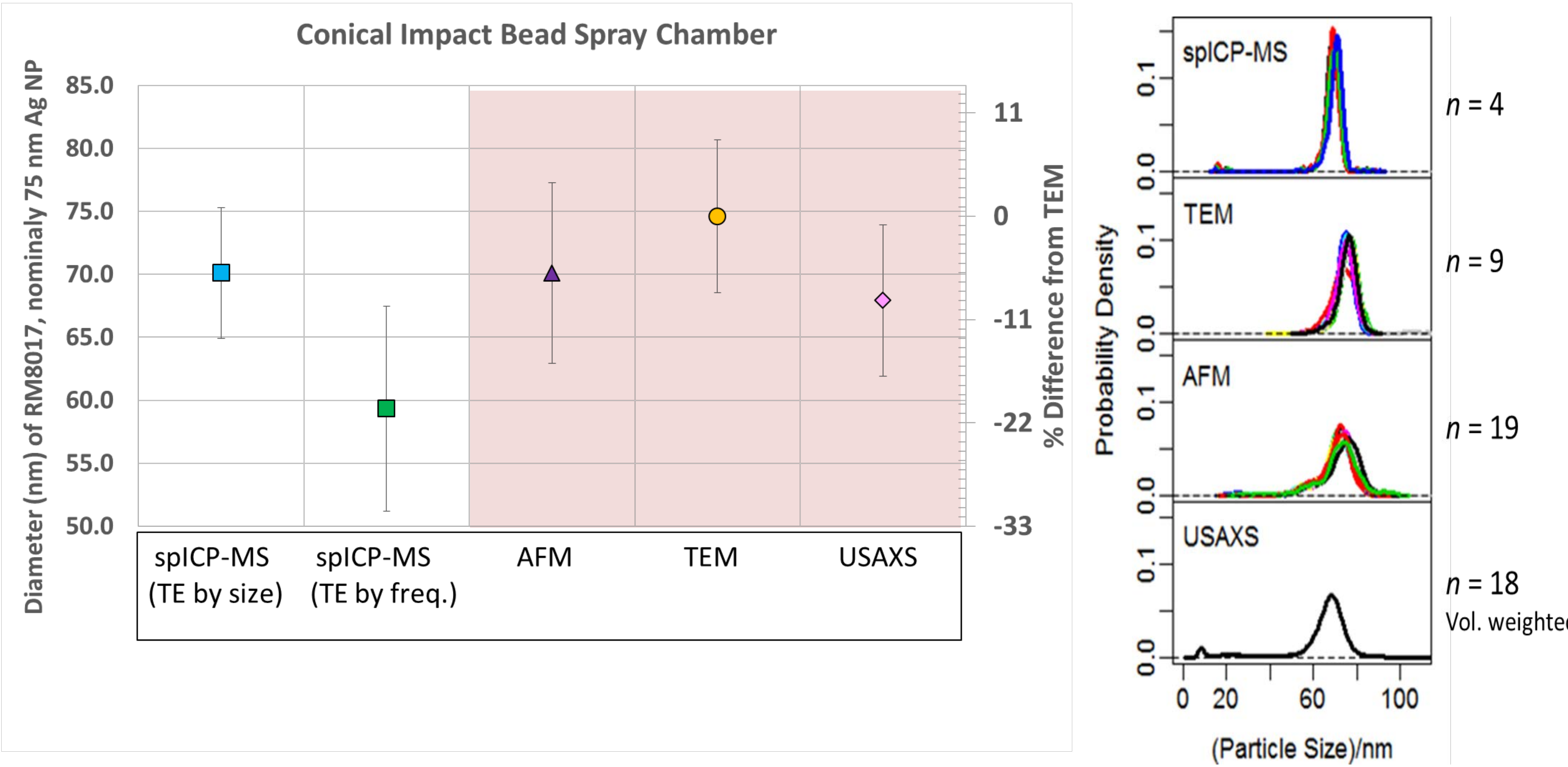


Vial	Size[a], nm (N)	Ag^+ Fraction (%) (spICP-MS)	Ag^+ Fraction (%) (IDA)[b]
1	68.65 (297)	5.6	--
2	68.55 (275)	7.0	2.9
3	69.18 (362)	2.9	2.8
4	70.42 (256)	1.2	3.3
Mean	69.20	4.2	3.0
SD	0.86	2.6	0.3
U	7.0		

[a]Trimmed Mean; transport efficiency by particle size method

- 2.5E07 fold dilution of RM 8017 for spICP-MS analysis
  - dilutes ionic signal

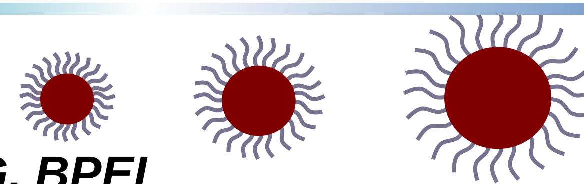
spICP-MS results for RM 8017 and Comparison to other sizing methods



- Generally, good agreement with AFM and USAXS
- Differences between TEM and spICP-MS for solution based calibration of size using frequency method to measure transport efficiency

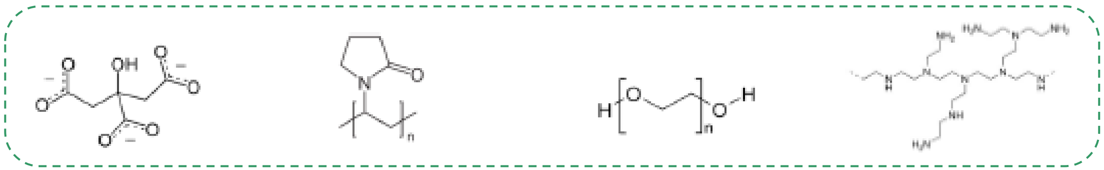
Materials: Commercial AuNPs Suspensions

- ***3 different Au NP sizes: 30, 60, 100 nm***



- ***4 different Au NP coatings: citrate, PVP, PEG, BPEI***

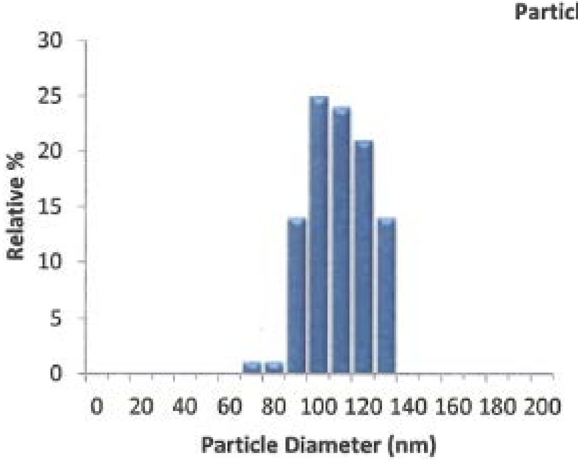
Citrate **PVP** **PEG** **BPEI**



Highly Negative **Negative** **Neutral** **Highly Positive** → Zeta Potential at pH 7

- **Citrate 100 nm**

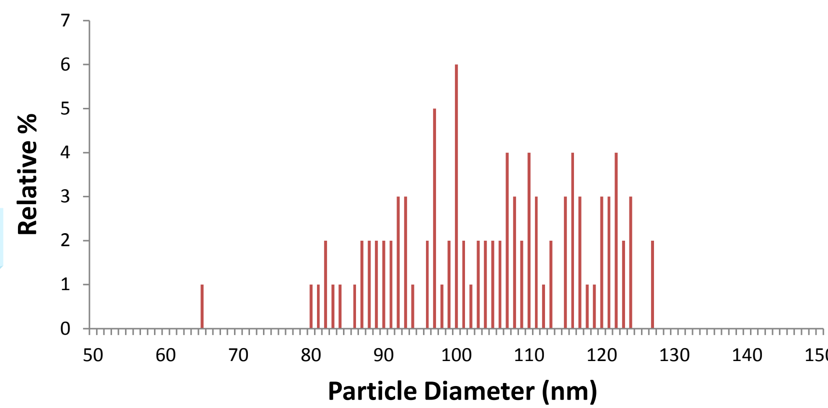
Diameter (TEM):	104.0 ± 13.1 nm	Hydrodynamic Diameter:	108.0 nm
Coefficient of Variation:	12.6 %	Zeta Potential:	-51.4 mV
Surface Area (TEM):	2.9 m^2/g	pH of Solution:	8.1
Mass Concentration (Au):	0.052 mg/mL	Particle Surface:	Sodium Citrate
Particle Concentration:	4.6E+09 particles/mL	Solvent:	Aqueous 2mM Citrate



Supplier TEM size distribution

104.0 ± 13.1 nm

100 NPs analyzed



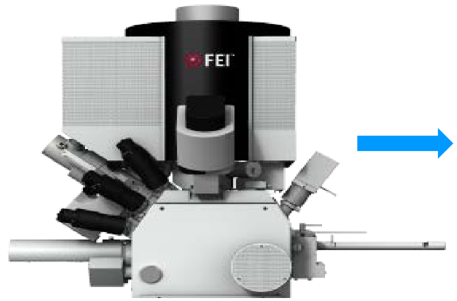
More rigorous commercial NP **size and size distribution characterization** is required

Validation of spICP-MS for Size/Size Distribution Measurements

High Resolution Scanning Electron Microscopy (HR-SEM)

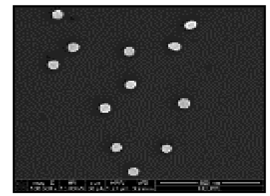
✓ **Sub-nanometer size resolution (≈ 0.8 nm)**

Si chips 5x5 mm



FEI Helios Dual-Beam Microscope

ImageJ software



NP size distribution

- ❑ Key instrument parameters: 15 kV, 86 pA, 30 s, 3.5 mm. Magnification 15k - 100k
- ❑ 500-1000 NPs analyzed per sample, n=6
- ❑ Citrate and PVP coated AuNPs (negative): http://ncl.cancer.gov/NCL_Method_PCC-15.pdf
    - ❑ Oxygen Plasma Cleaning + Aminopropyldimethylethoxysilane for Si chips functionalization
- ❑ PEG and BPEI coated: direct deposition on Si chip

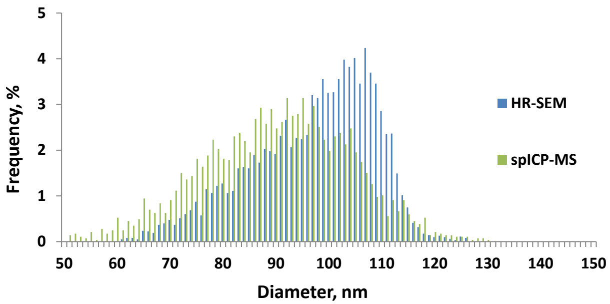
spICP-MS:

- ➢ ThermoFisher X-7 and X-II quadrupole ICP-MS
- ➢ 10 ms dwell time, 360 s acq. Time, 0.5 mL min^{-1} flow rate
- ➢ Particle number conc.: 15 x $10E^{7}$ particles L^{-1}, 1000 NPs per sample, n = 3 to 6
- ➢ Transport efficiency calculated by particle size method

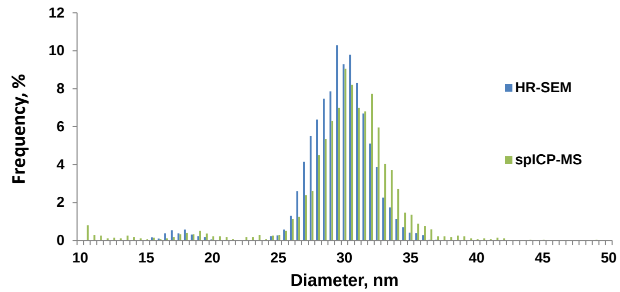
Results: Negatively Charged Commercial AuNP Suspensions

➢ **Citrate 100 nm and PVP coated AuNPs (negative): Same SEM Sample Preparation : Oxygen Plasma Cleaning + Aminopropyldimethylethoxysilane for Si chips functionalization, n=6**

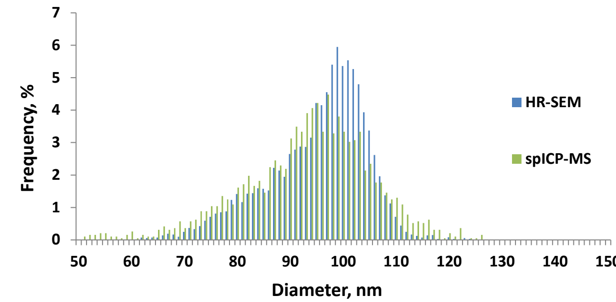
➢ **Citrate 100 nm**



➢ **PVP 30 nm**



➢ **PVP 100 nm**

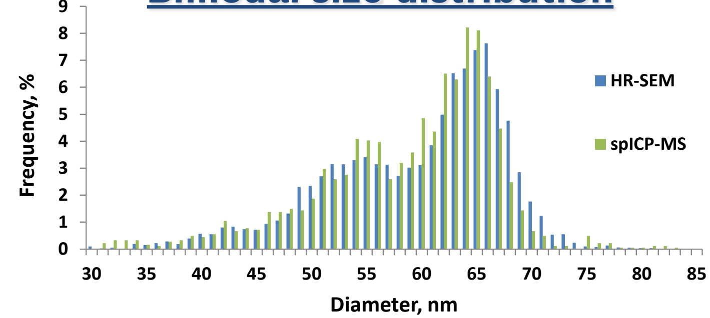


	Mean Size (nm)**		
AuNPs	**HR-SEM**	**spICP-MS**	**TEM supplier**
Citrate 100 nm	96.7 ± 0.3	101.7 ± 0.7	104.0 ± 2.6
PVP 30 nm	29.3 ± 0.1	29.7 ± 0.2	29.7 ± 0.5
PVP 100 nm	94.8 ± 0.2	92.7 ± 0.6	100.0 ± 1.5
PVP 60 nm	59.1 ± 0.1	58.3 ± 0.2	55.9 ± 1.6

** Expanded uncertainty of the mean for 95% coverage; only measurement repeatability.

➢ **PVP 60 nm**

Bimodal size distribution

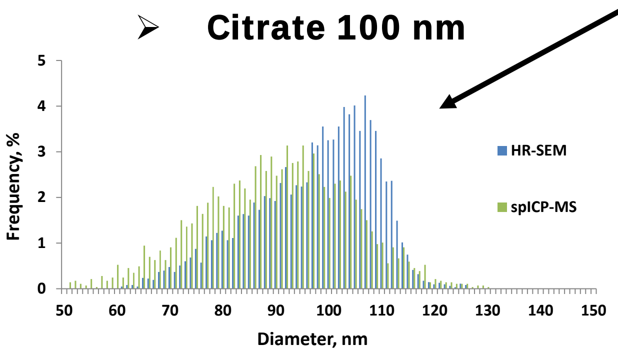


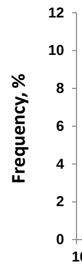
"Successful agreement between spICP-MS and HR-SEM for negatively charged coating AuNP size distribution"

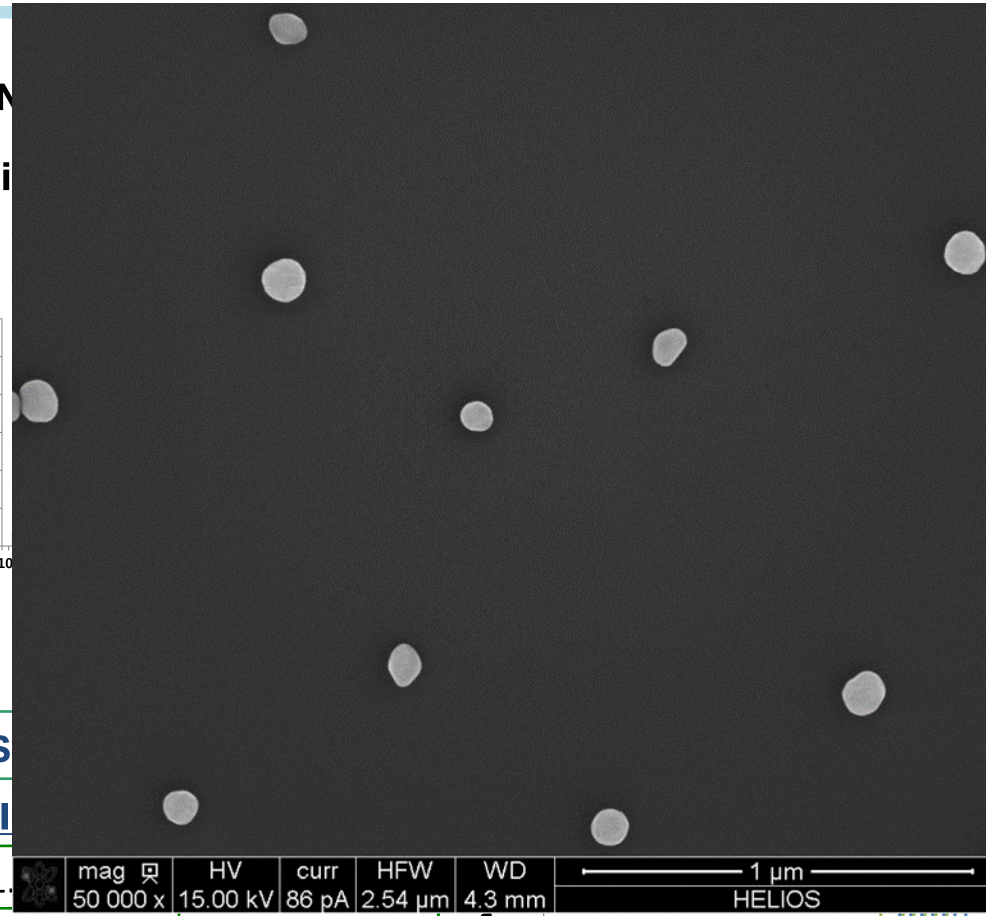
Results: Negatively Charged Commercial AuNP Suspensions

- Citrate 100 nm and PVP coated AuN ... gen

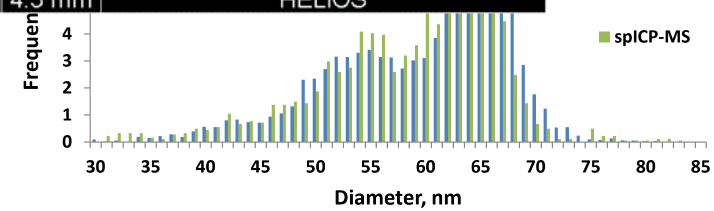
  Plasma Cleaning + Aminopropyldi ... =6







	Mean S...		...tion
AuNPs	**HR-SEM**	**spI...**	
Citrate 100 nm	96.7 ± 0.3	101.	
PVP 30 nm	29.3 ± 0.1	29.7 ± 0.2	29.7 ± 0.5
PVP 100 nm	94.8 ± 0.2	92.7 ± 0.6	100.0 ± 1.5
PVP 60 nm	59.1 ± 0.1	58.3 ± 0.2	55.9 ± 1.6

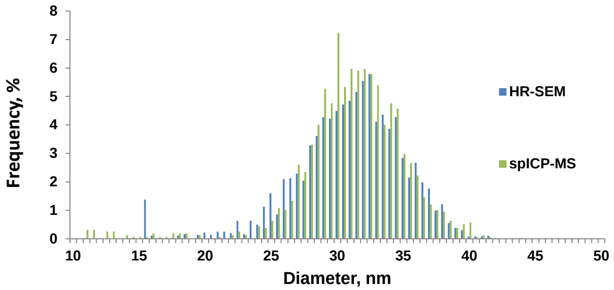


** Expanded uncertainty of the mean for 95% coverage; only measurement repeatability.

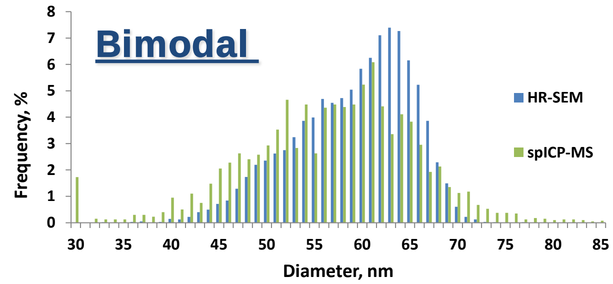
"Successful agreement between spICP-MS and HR-SEM for negatively charged coating AuNP size distribution"

Results: Positively Charged and Neutral Commercial AuNP Suspensions

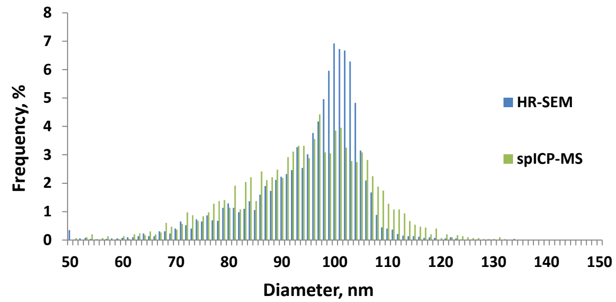
➢ **BPEI 30 nm**



➢ **BPEI 60 nm**



➢ **BPEI 100 nm**

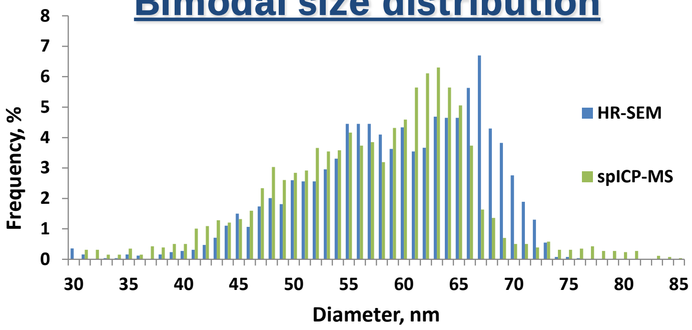


	Mean Size (nm)**		
AuNPs	**HR-SEM**	**spICP-MS**	**TEM supplier**
BPEI 30 nm	31.2 ± 0.1	30.9 ± 0.1	30.9 ± 0.6
BPEI 60 nm	60.1 ± 0.2	59.8 ± 0.3	63.7 ± 1.5
BPEI 100 nm	94.6 ± 0.3	94.1 ± 0.3	98.0 ± 2.0
PEG 60 nm	58.7 ± 0.3	57.0 ± 0.4	65.3 ± 1.6

** Expanded uncertainty of the mean for 95% coverage; only measurement repeatability.

➢ **PEG 60 nm**

Bimodal size distribution



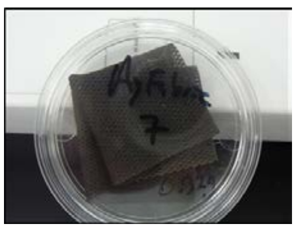
"Successful agreement between spICP-MS and HR-SEM for positively and neutrally charged coating AuNP size distribution"

Elemental and Chemical Analysis of Commercial Textiles Claiming a nAg component

- **Seven Textiles**

Sock fabric (S#2, 4)
"nano silver technology"

Conductive fabric (S#5, 6)
Silver-plated



Medical fabric (S#7, 8)
"nanocrystaline coating"

Sport fabric (S#9)
"silver nano ions"

Sample	XPS Wt % Ag	XPS Metallic?	EDS Ag Detected ?	ICP-MS Wt % Ag
2	ND	UKN	No	1.2E-6
4	ND	UKN	No	0.0024
5-side1	53.6	Yes	Yes	10.8
5-side-2	48.4	Yes		
6-side-1	42.5	Yes	Yes	9.2
6-side-2	38.2	Yes		
7-side-1	71.8	No	Yes	10.9
7-side-2	79.3	No		
8-side-1	72.9	No	Yes	12.3
8-side-2	79.6	No		
9-stiching	<0.1	UKN	No	0.0021
9-front	<0.1	UKN		

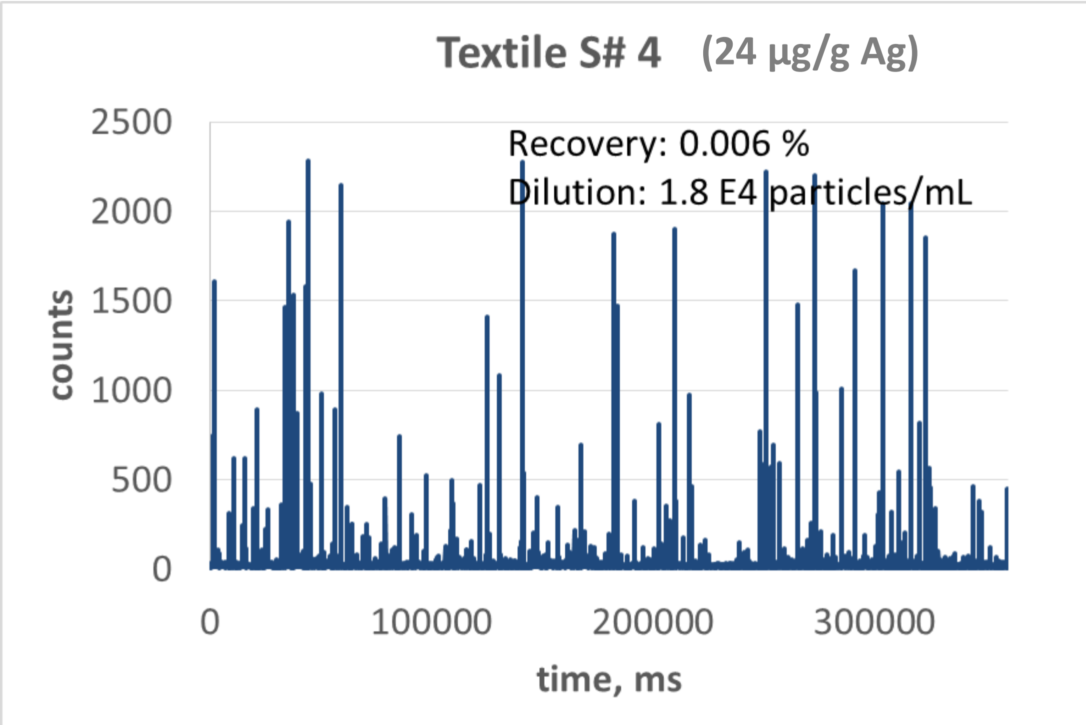
XPS by J. Gorham *EDS by D. Holbrook* *ICPMS by K. Murphy and J Liu*

- **XPS**
  - **Detected Ag in 5/7 samples**
  - **Measureable in 4/7**
  - **Two samples composed of Ag^0**
- ***EDS***
  - ***Detected Ag in 4/7 samples***
- **ICP-MS**
  - **Measurable Ag in 7/7 samples**
  - **ppb to % levels**

spICP-MS Analysis of Textiles

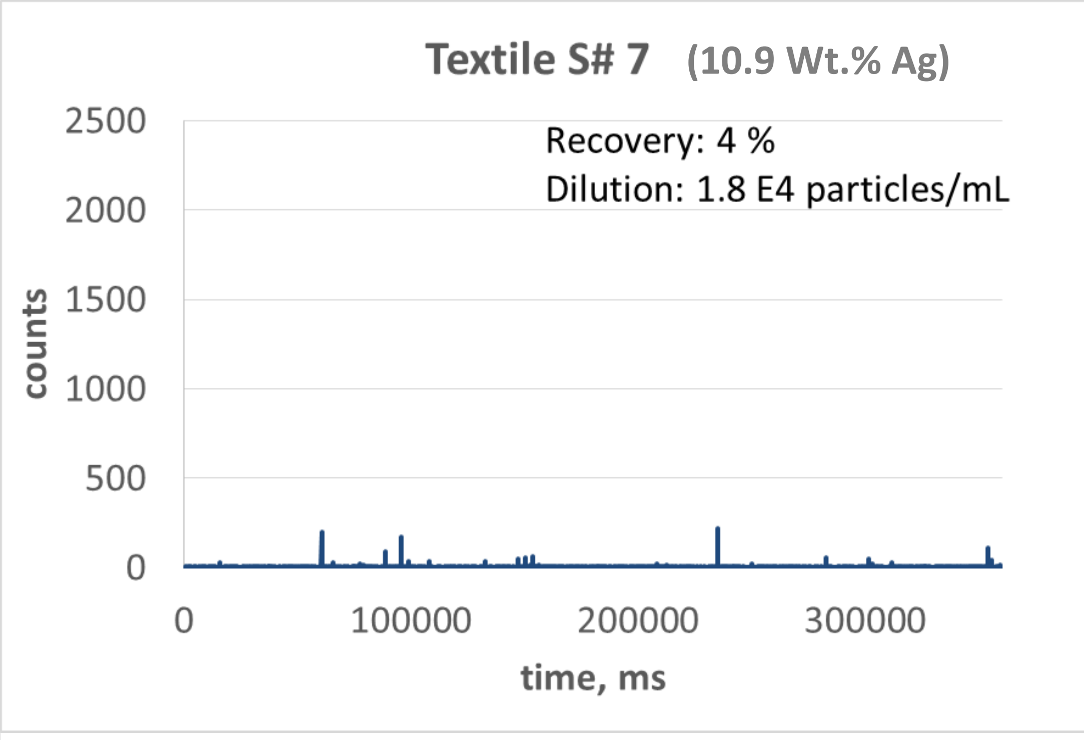
- **Place in test tube in 10 mL water (ph 5.5)**
- **sonicate for 30 min. in ice bath**
- **Measure recovery**
- **spICP-MS analysis:**
  - **0.45 µm PVDF filter**
  - **dilute to ≈1.8 E4 particles/mL**
    - **≈12 ppt**
  - **Particle pulses distinguished from the background using a 5σ criterion**

spICP-MS Analysis of Textiles



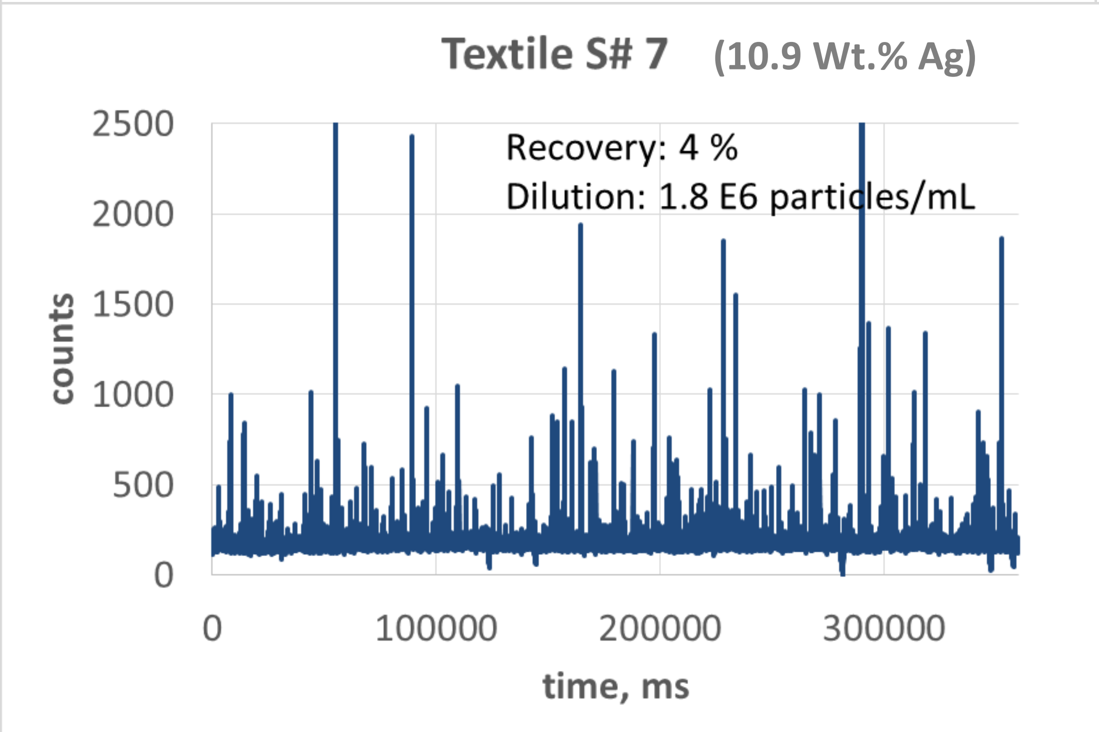
criterion

spICP-MS Analysis of Textiles

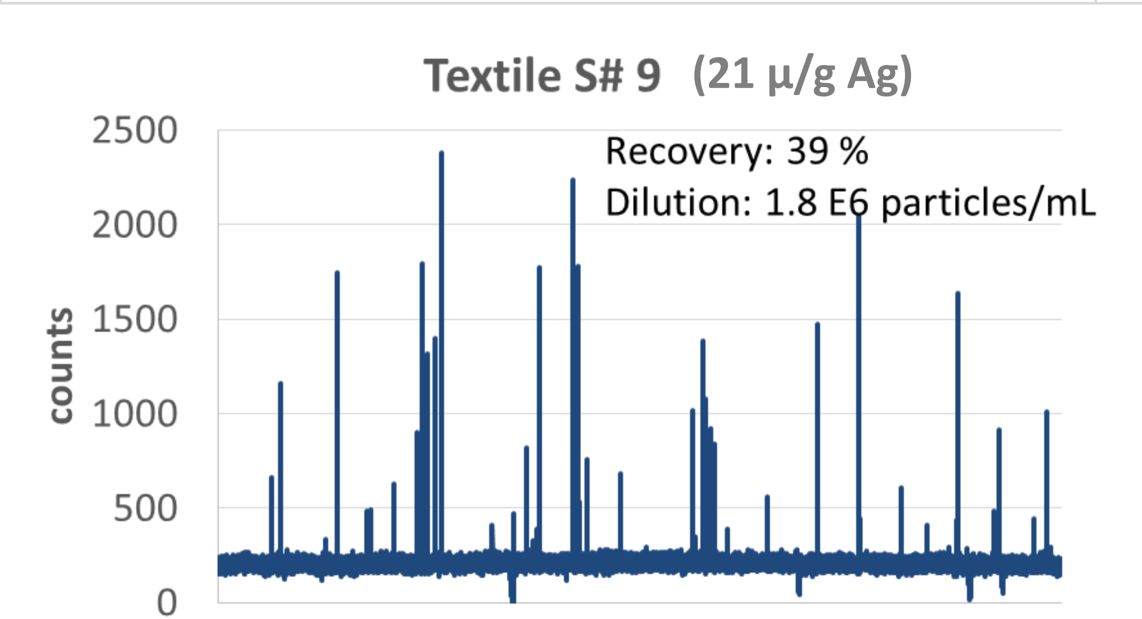


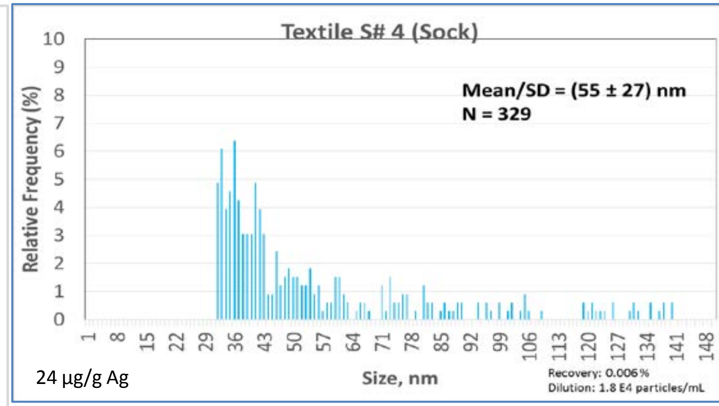
criterion

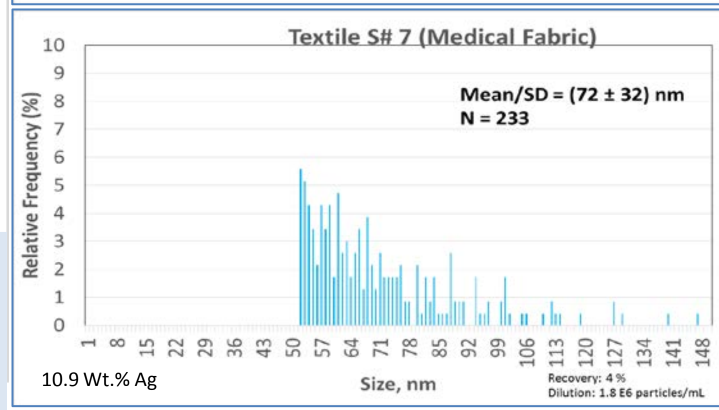
spICP-MS Analysis of Textiles

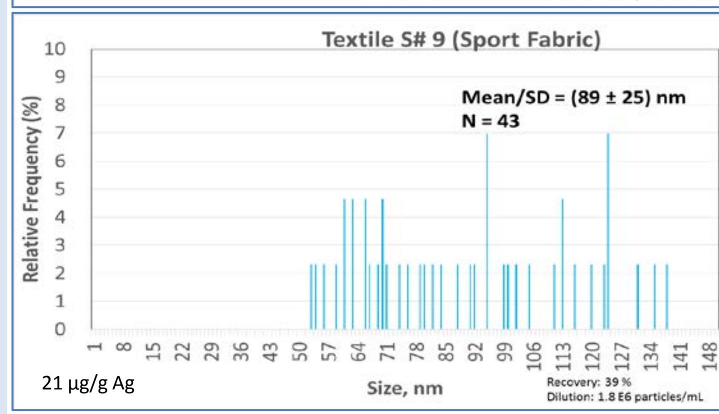


spICP-MS Analysis of Textiles









A Note of Caution!

- Variable size detection limit
  - 15 nm for Ag without Ag^+
- Proper dilution is critical to avoid coincidence
  - Unknown AgNP/Ag^+ ratio
  - Unknown target particle size
- Silver is reactive
  - Ag^0 forms Ag^+
  - Ag^+ forms Ag^0
- Distribution in solvent may not match distribution on textile

Imagining Analysis of Textiles: 'The Hunt for Nano'

- SEM images of commercial textiles
  - No real evidence of AgNPs

Imaging by R.D. Holbrook and G. Stan

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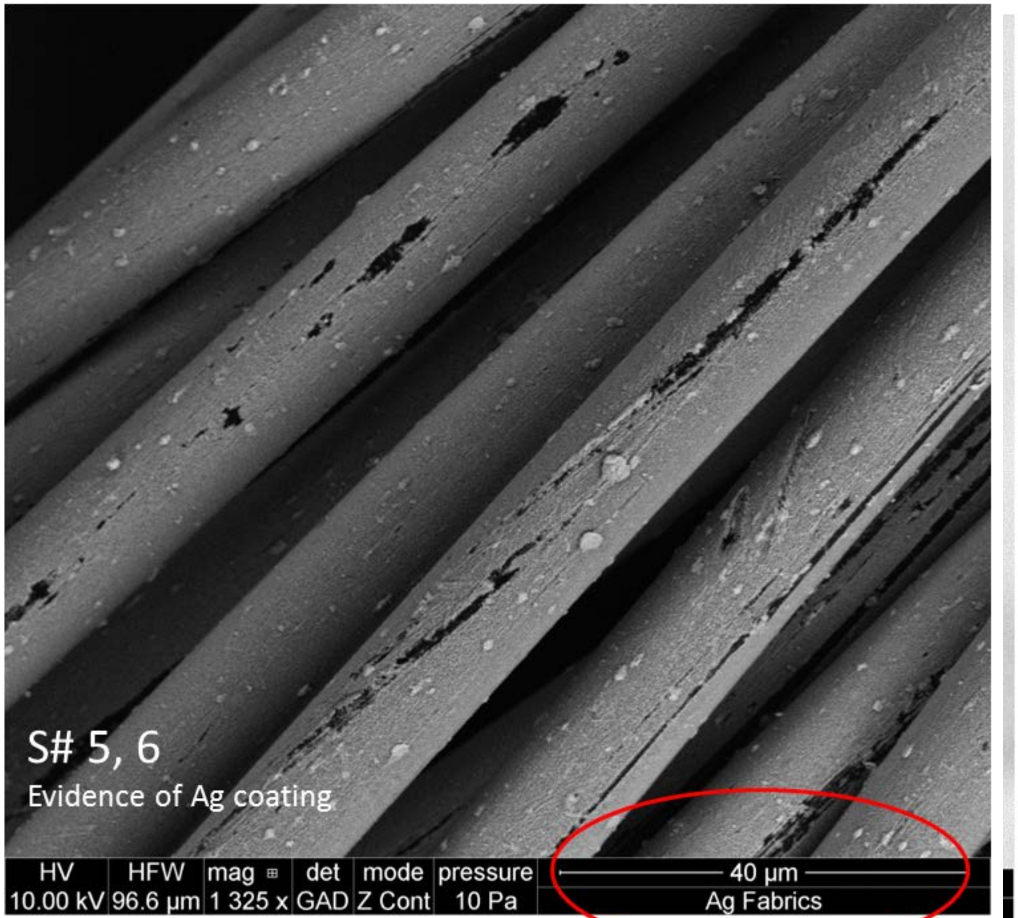

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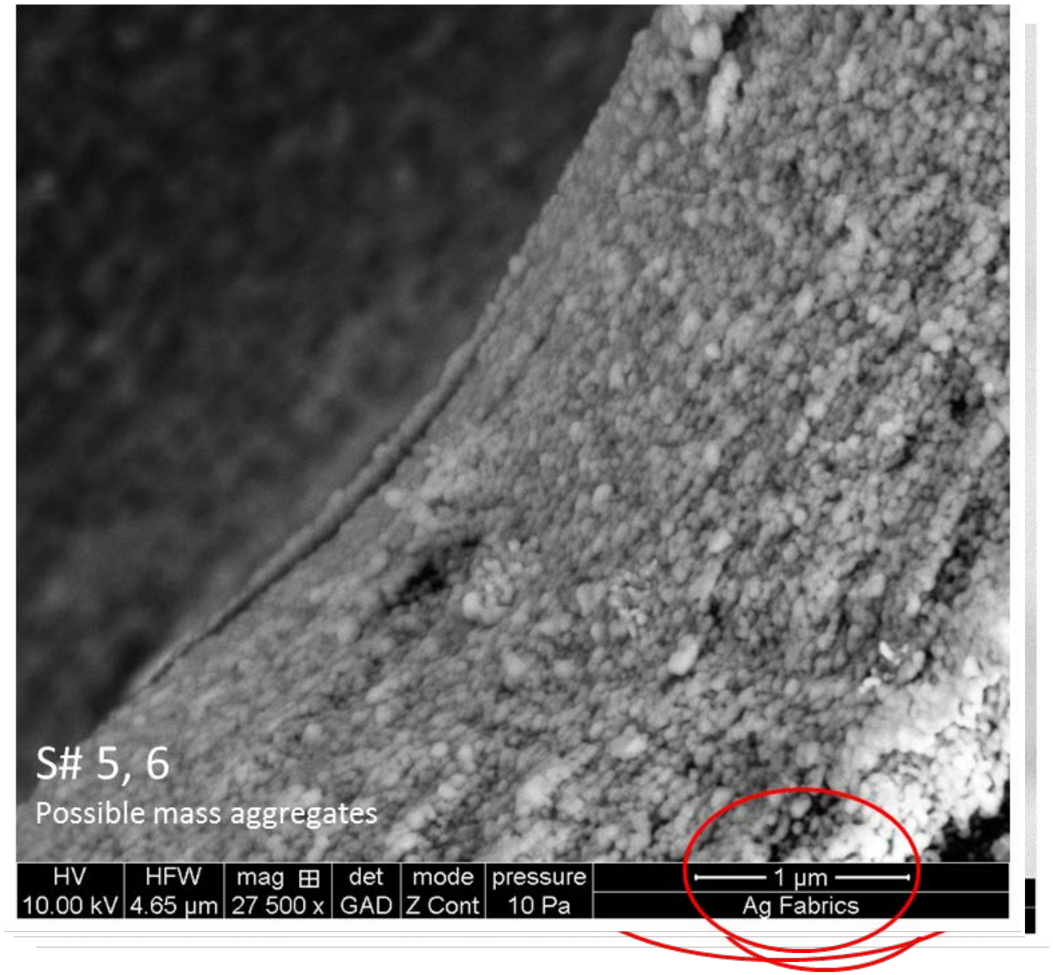

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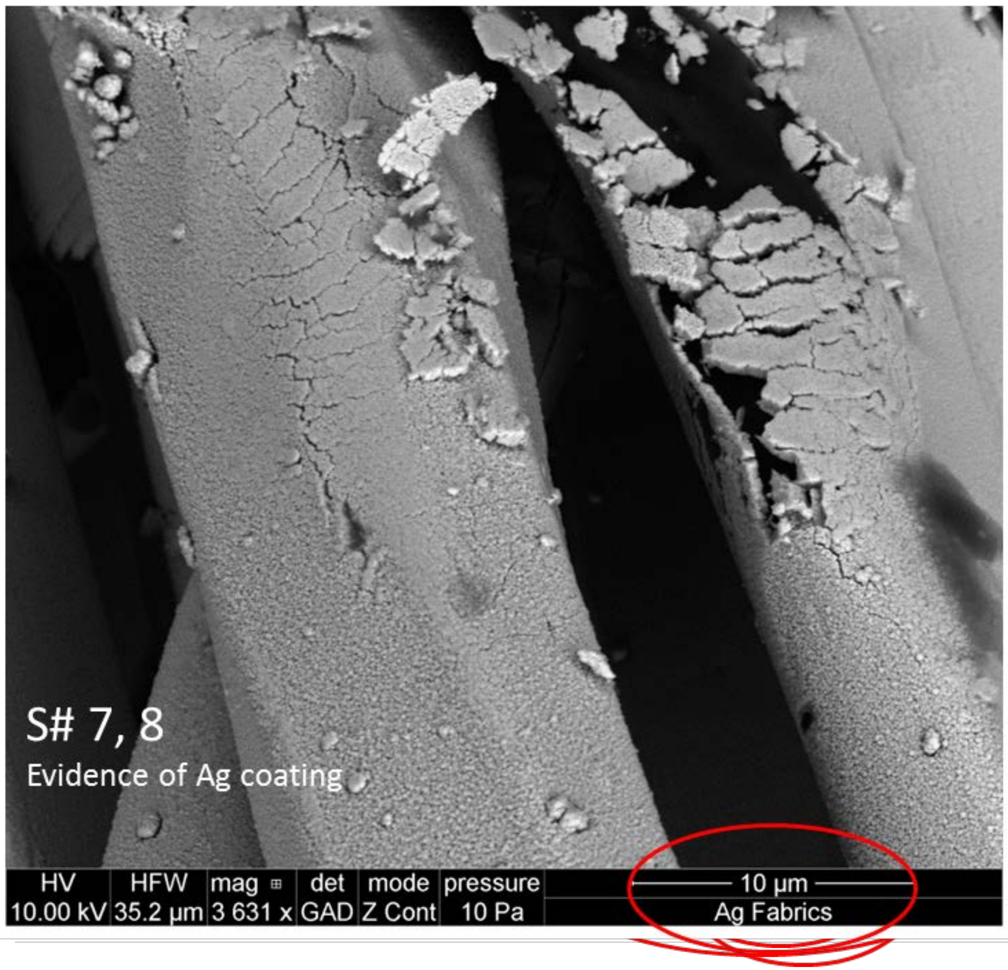

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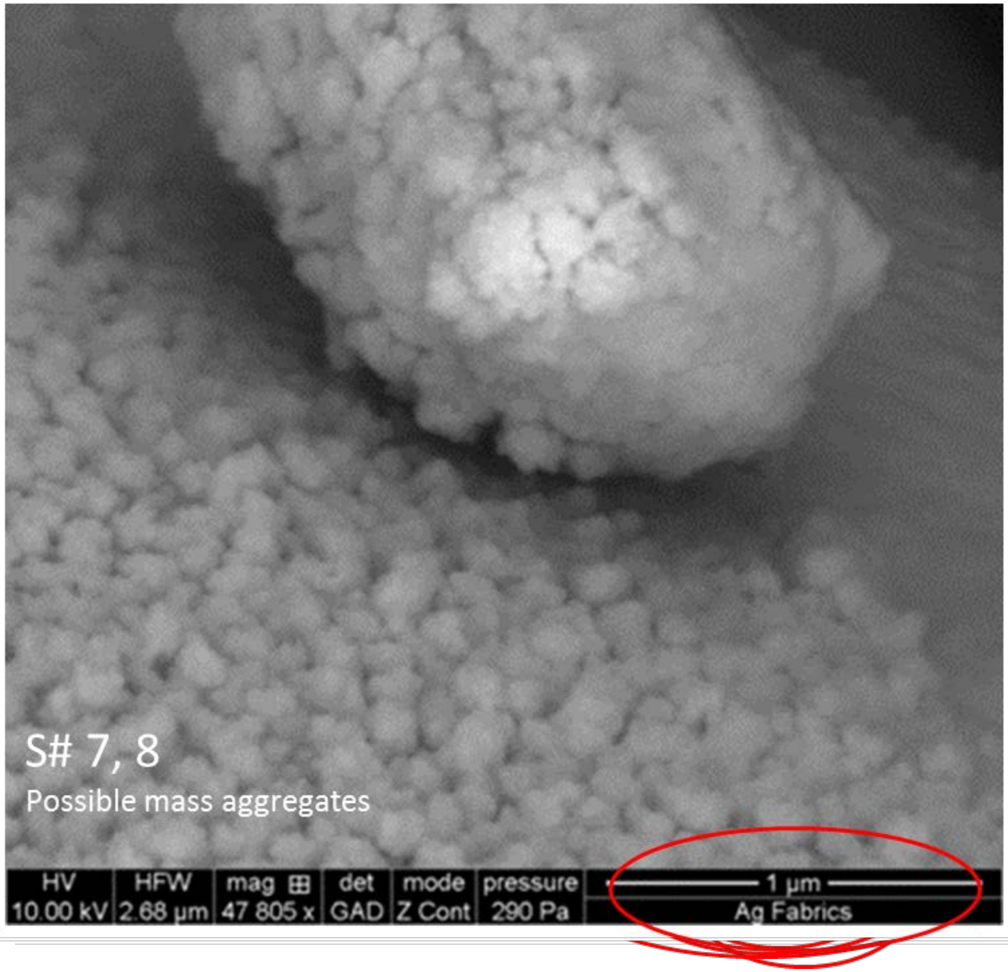
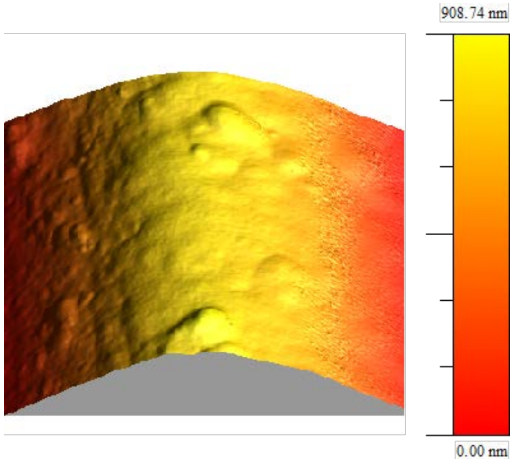
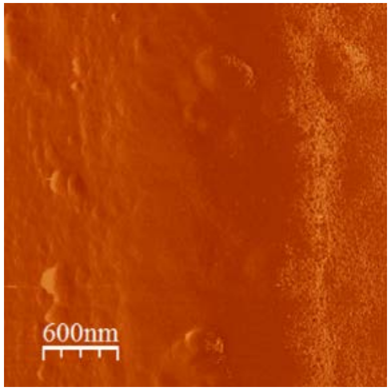


AFM images of commercial textiles
- Surface devoid of AgNP contribution





Imaging by R.D. Holbrook and G. Stan

Development of a Test Material

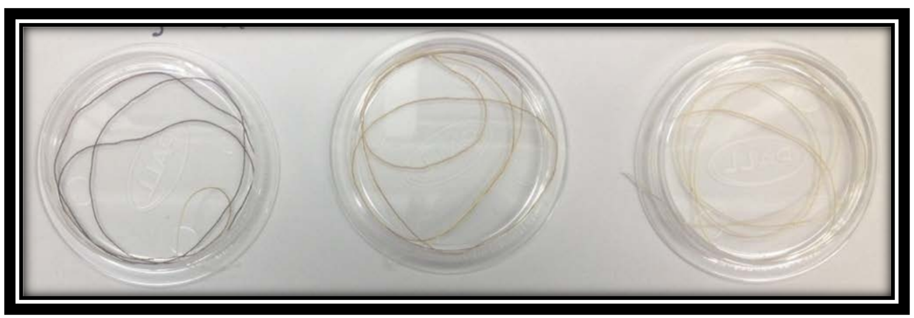
- Use to measure limits of quantitation for each method and apply lessons learned to better approach the characterization of ‘unknowns’
- Approach:
  - Employ citrate reduction technique designed for 20 nm AgNP suspensions
  - Develop multiple loadings plus a blank control
- Results
  - All 5 test threads were
  - loaded with varying silver
  - concentrations.
  - A distinct color change to light
  - yellow was observed. The control
  - remained a white color

+

$$n_i\,(x[Ag^+] \;+\; y[Citrate] \;+\; z[NaBH_4]) \xrightarrow{\text{Water}\ \Delta}$$

High Starting $[Ag^+]$ 8E-04 mol/L

Low Starting $[Ag^+]$ 1.9E-05 mol/L



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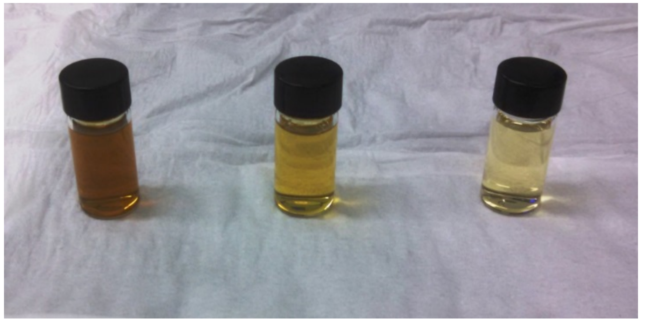
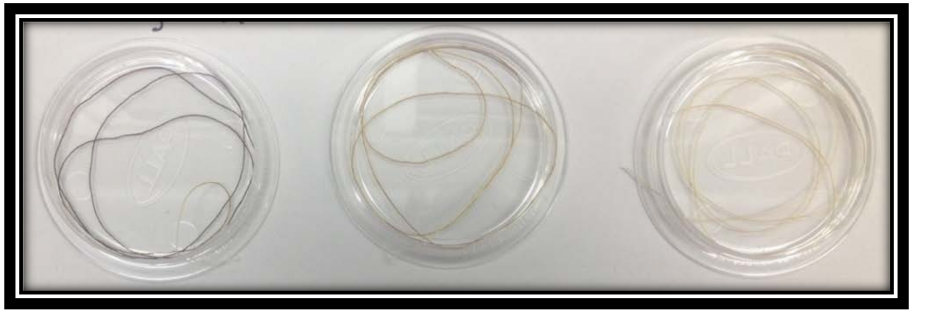
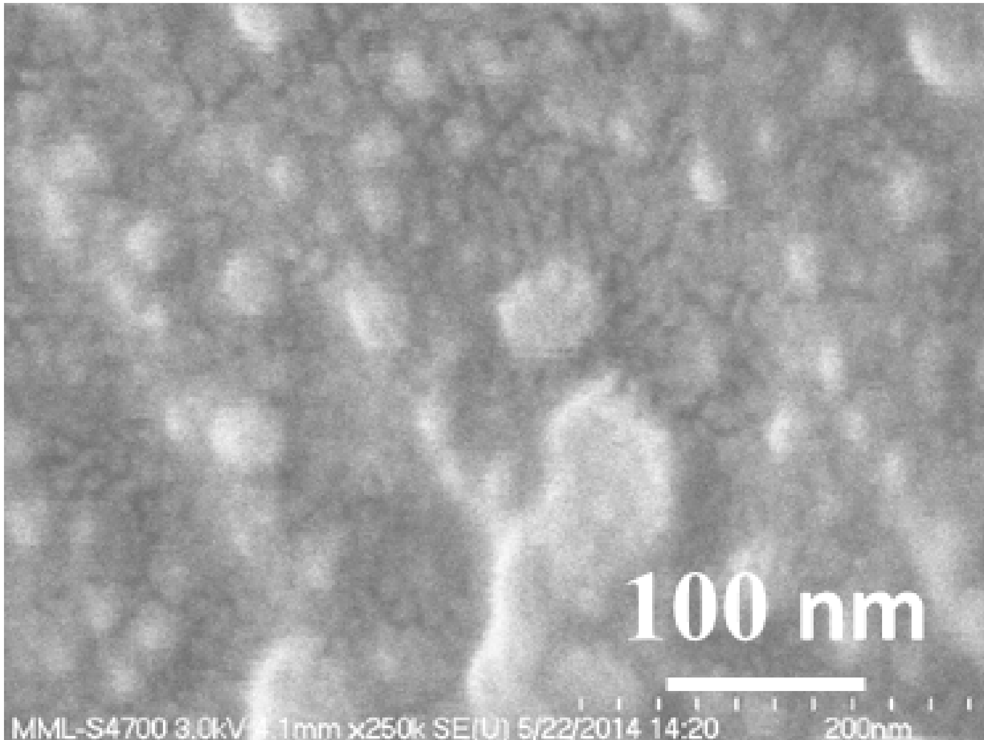


High Starting [Ag^+]
8E-04 mol/L

Low Starting [Ag^+]
1.9E-05 mol/L

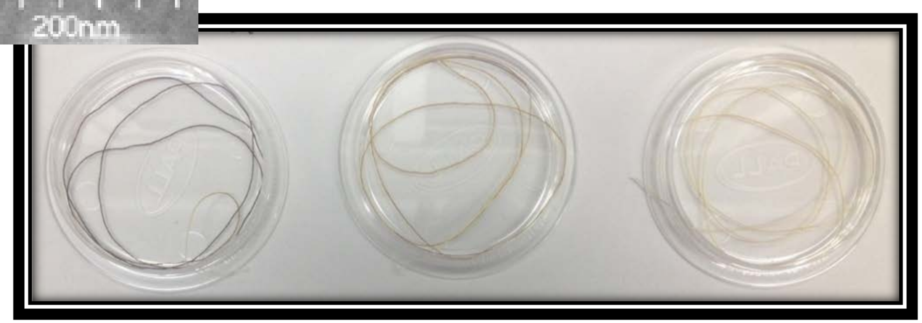


Development of a Test Material



ng [Ag^+]

Low Starting [Ag^+]
1.9E-05 mol/L



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Imaging by TM Nguyen

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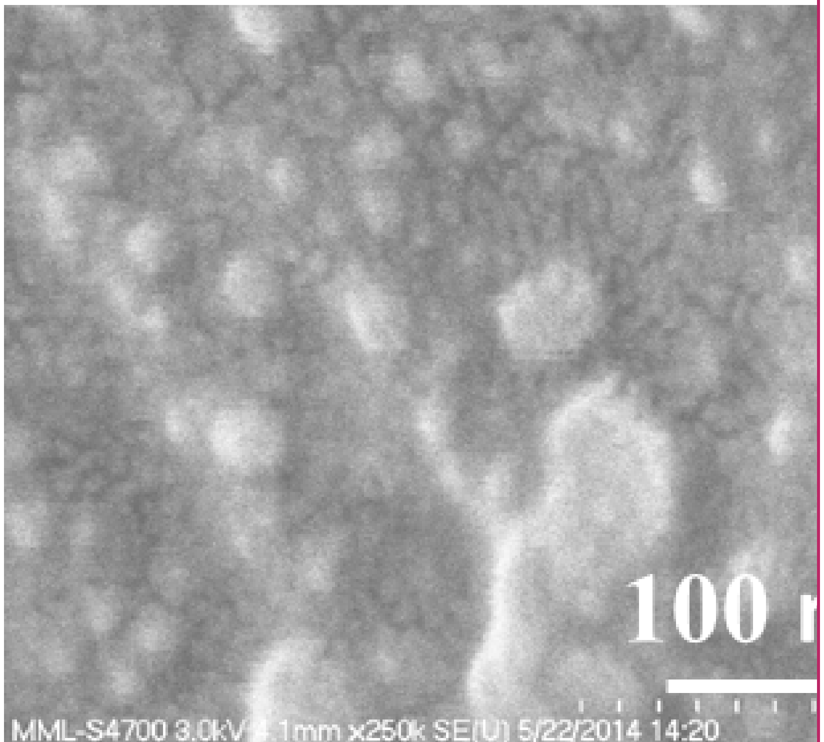

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Imaging

NIST Special Publication 1200-8

Preparation of silver nanoparticle loaded cotton threads to facilitate measurement development for textile applications

Version 1.0

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D. Tselenchuk
G. Stan
T. M. Nguyen
R.D. Holbrook
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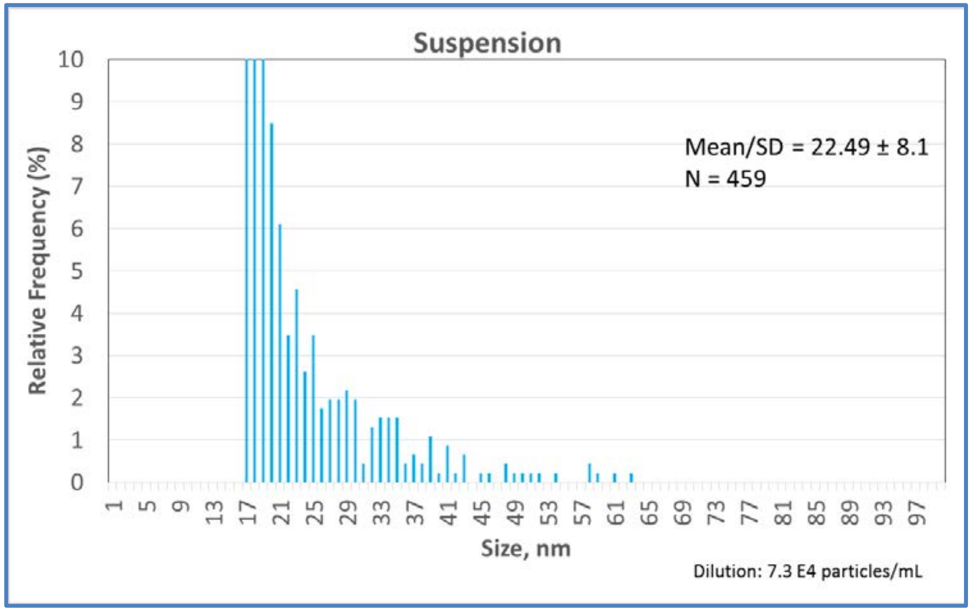
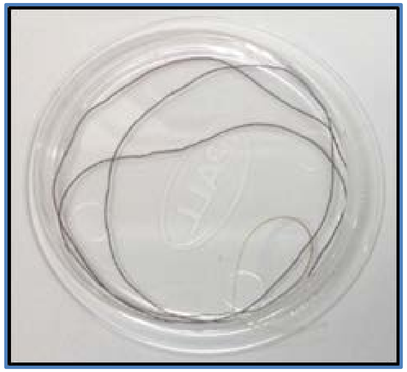
R.F. Cook
V.A. Hackley

NIST
National Institute of Standards and Technology
U.S. Department of Commerce

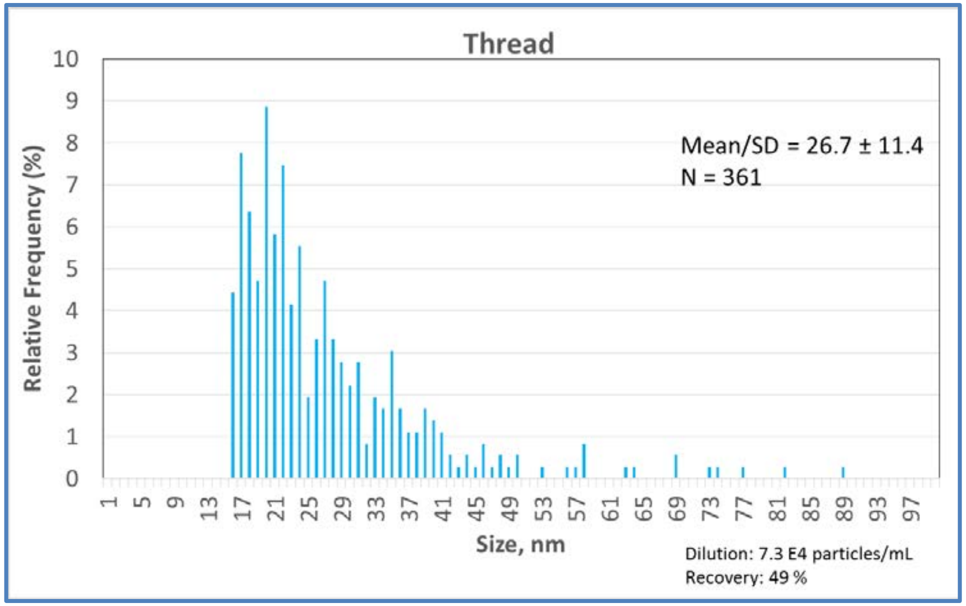
spICP-MS Analysis of Test Threads

0.018 Wt % Ag





0.3 Wt % Ag



Conclusions

- spICP-MS is a promising method for the characterization of particle size/size distribution of test materials and RM's
  - Applicable to concurrent measurement of number concentration and ionic content, as well, but capabilities have not yet been rigorously examined at NIST
- For solution based calibration, uncertainty analysis reveals that variance in the transport efficiency provides the largest contribution to the total uncertainty of the spICP-MS measurement
  - Differences between the size method and frequency method for determining the transport efficiency were observed; size method yielded lower bias
- Solution based method – application to solid samples requires development of extraction methods that do not alter the particle size distribution
- Method requires a priori knowledge about particle composition, shape and size
- At present, size dynamic range is limited: best case is AuNPs, 10 nm to 80 nm (standard conditions); 200 nm (reduced sensitivity conditions)

Acknowledgements and Information

- <u>RM 8017</u>

  Project coordination: Rob MacCuspie, Vince Hackley, and Justin Gorham
  AFM: Rob MacCuspie
  TEM: John Bonevich
  USAXS: Andrew Allen
  spICP-MS: Karen Murphy and Jingyu Liu
- <u>Commercial AuNP Suspensions</u>

  HR-SEM: Antonio Montoro Bustos, Kavuri P. Purushotham, András E. Vladár
  spICP-MS: Antonio Montoro Bustos
- <u>Textiles</u>

  Project coordination: Justin Gorham, Vince Hackley, Robert Cook, Debra Kaiser
  XPS: Justin Gorham
  SEM: Dave Holbrook and TM Nguyen;   AFM: G. Stan
  spICP-MS: Karen Murphy and Jingyu Liu

NIST “Protocols for Nano-EHS” website:
http://www.nist.gov/mml/nanoehs-protocols.cfm

THANK YOU