Electronic Supplementary Material (ESI) for Environmental Science: Nano.

Supporting Information

Improvements in the detection and characterization of engineered nanoparticles using spICP-MS at microsecond dwell times

Manuel D. Montaño[‡,], Hamid R. Badiei[§], Samad Bazargan[§], James F. Ranville[‡]*

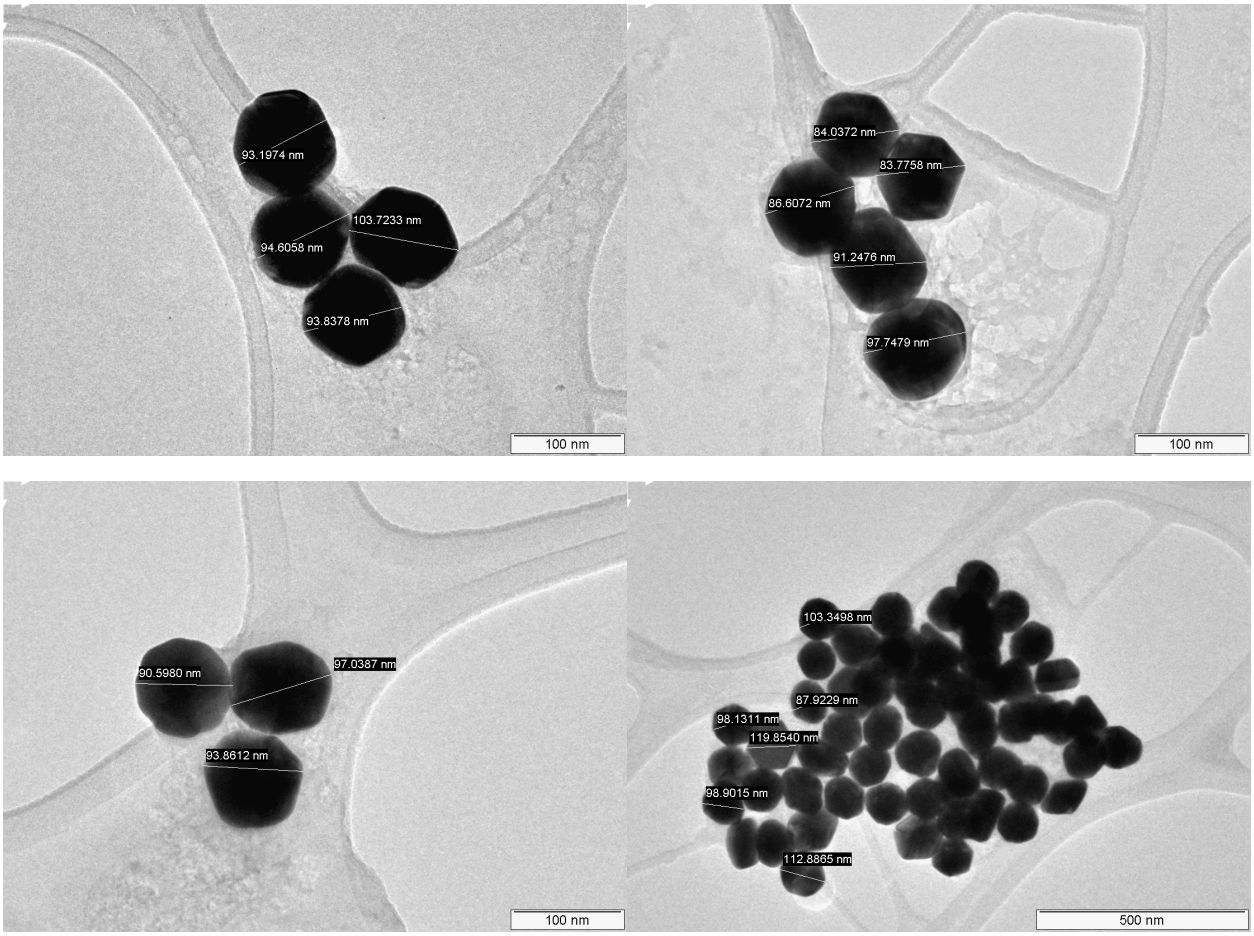
[‡]Colorado School of Mines, Chemistry and Geochemistry, Golden, Colorado, 80401, United States

[§]Perkin Elmer, Inc., Woodbridge, Ontario, L4L 8H1, Canada

Table S1. Typical running instrument conditions for Perkin Elmer NexION 300Q ICP-MS

Instrument Parameter	Value
Nebulizer Gas Flow	0.85 – 1 ml/min
Auxiliary Gas Flow	1.02 ml/min
Plasma Gas Flow	16L/min
ICP RF Power	1600 W
Analog Stage Voltage	-1900 V
Pulse Stage Voltage	1600 V
Discriminator Threshold	12
Deflector Voltage	-9.5 V
Sample Flow Rate	0.975 ml/min

Figure S1. Transmission electron microscopy images of 100nm gold nanoparticle (BBI Solutions) with EDX spectrum. Images collected using FEI-TEM CM200 with a LaB_6 filament operated at 200 kV. Manufacture TEM of this specific batch gives a particle sizes of 99.6 ± 8.0nm



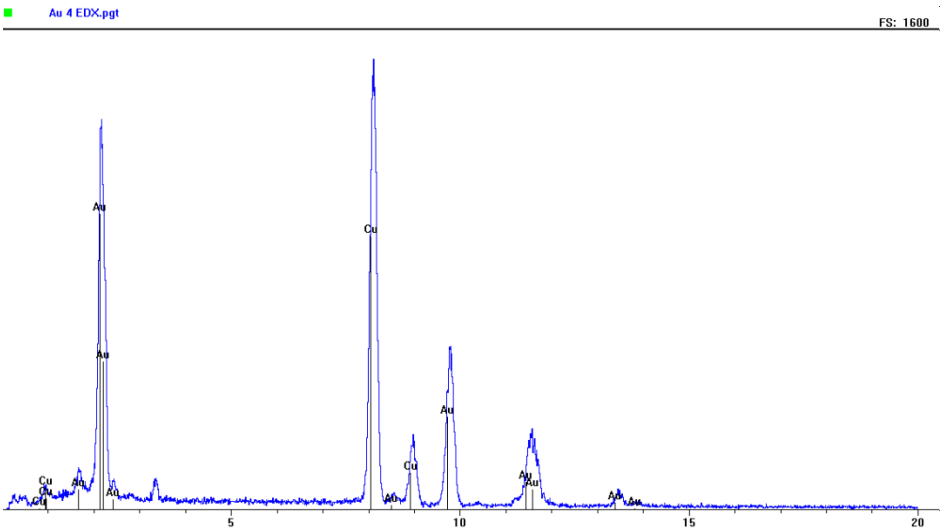
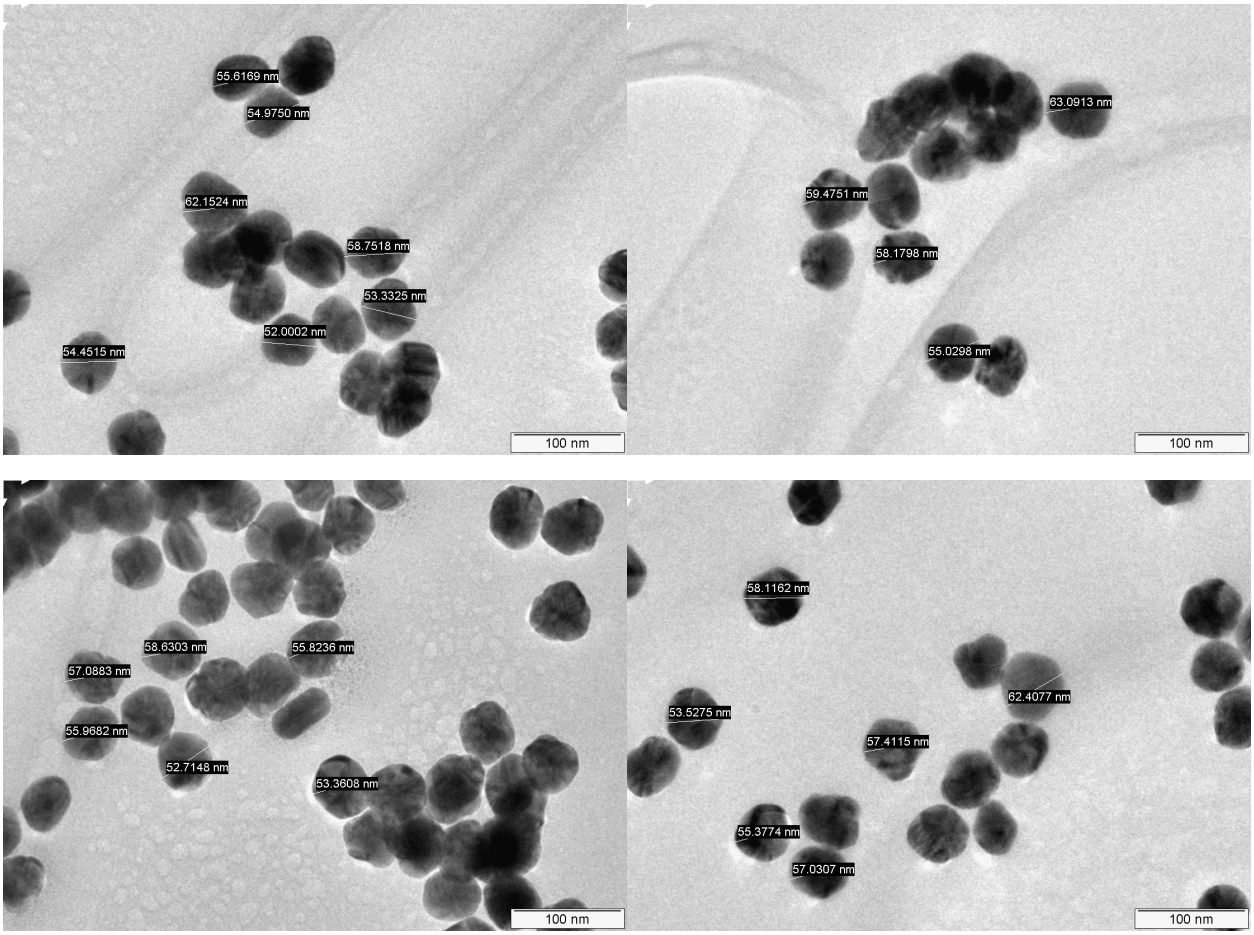


Figure S2. Transmission electron microscopy images of 60nm silver nanoparticle (NanoComposix) with EDX spectrum. Images collected using FEI-TEM CM200 with a LaB_6 filament operated at 200 kV. Manufacturer TEM gives a particles size of 59.6 ± 6.3nm



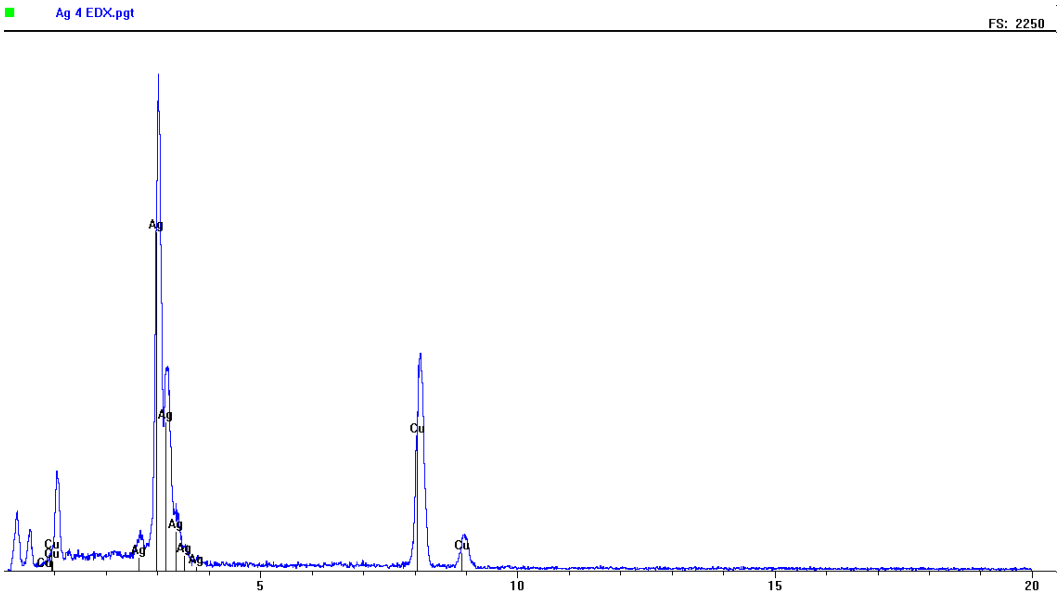


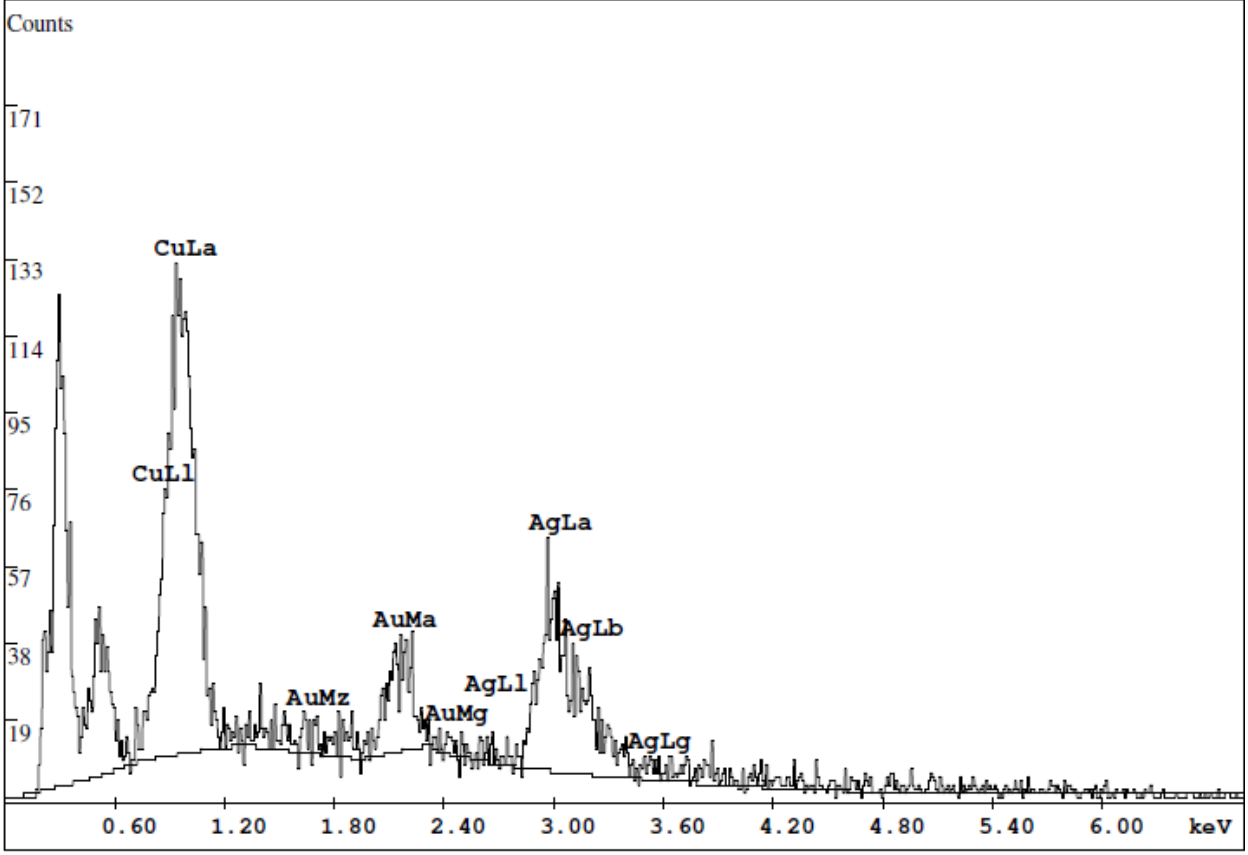
Figure S3. EDX of gold core / silver shell nanoparticle obtained from FESEM (JEOL USA, Ltd; JSM7000F)

C:\EDS\USR\mmontano\20130906\AuAg_3.spc

Label:AuAg_3

kV:7.0 Tilt:2.0 Take-off:36.5 Det: SUTW Sapphire

Res:167 Amp.T:6.40 FS:194 Lsec:51 6-Sep-2013 13:41:36



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
CuL	30.21	45.30	0.2476	1.1265	0.7276	1.0001
AuM	17.43	8.43	0.1406	0.8202	0.9836	1.0002
AgL	52.37	46.26	0.5016	0.9811	0.9763	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
CuL	24.10	2.62	3.12	9.20
AuM	5.28	3.00	8.83	1.76
AgL	9.94	1.87	5.16	5.32

Figure S4. Transmission electron microscopy image of gold core / silver shell nanoparticle. Images taken with FEI Tecnai T-12, 120 kV working voltage at 26,000x magnification.

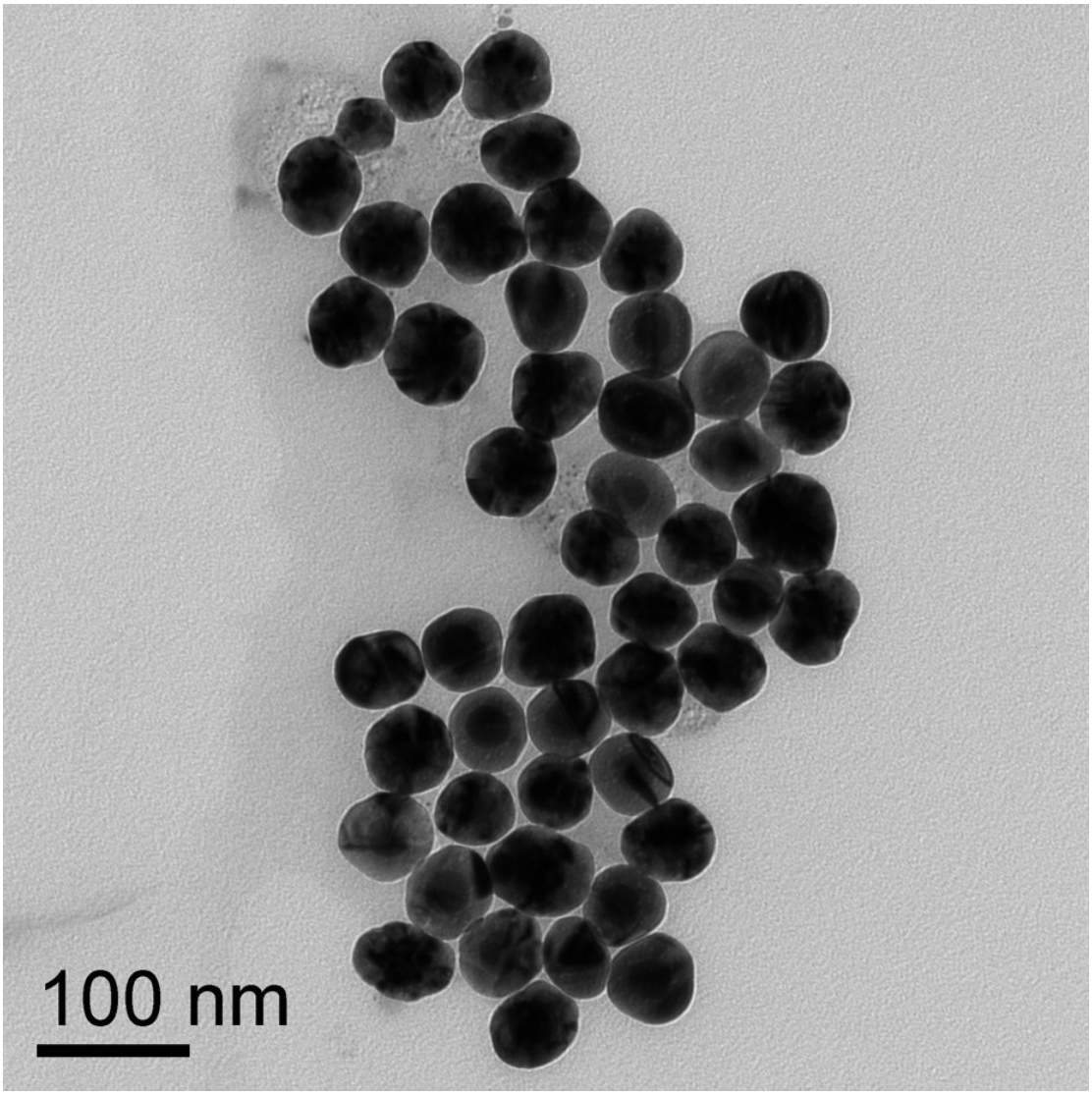


Figure S5. (Left) Centrifugal FFF coupled to ICP-MS fractogram showing elution of particles of a singular mass comprised of both gold and silver. (Right) Asymmetrical FFF chromatogram shows elution of particles of a singular hydrodynamic diameter comprised of both gold and silver. This is evidence of one particle type containing both elements, rather than two separate particle types.

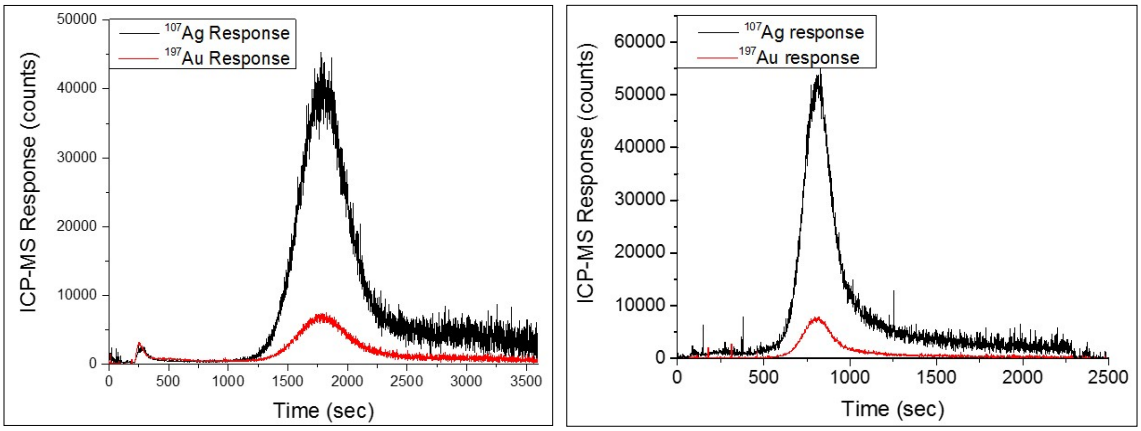


Figure S6. Size distributions of 100nm gold nanoparticles at three different dwell times (0.1, 3 and 10ms) and five different mass concentrations (0.5, 1, 2, 5, and 10 µg Au/L). Percentages of particles representing 1, 2, and 3-or-more particles detected within a single dwell time event are listed. Distributions are a combination of triplicate measurements.

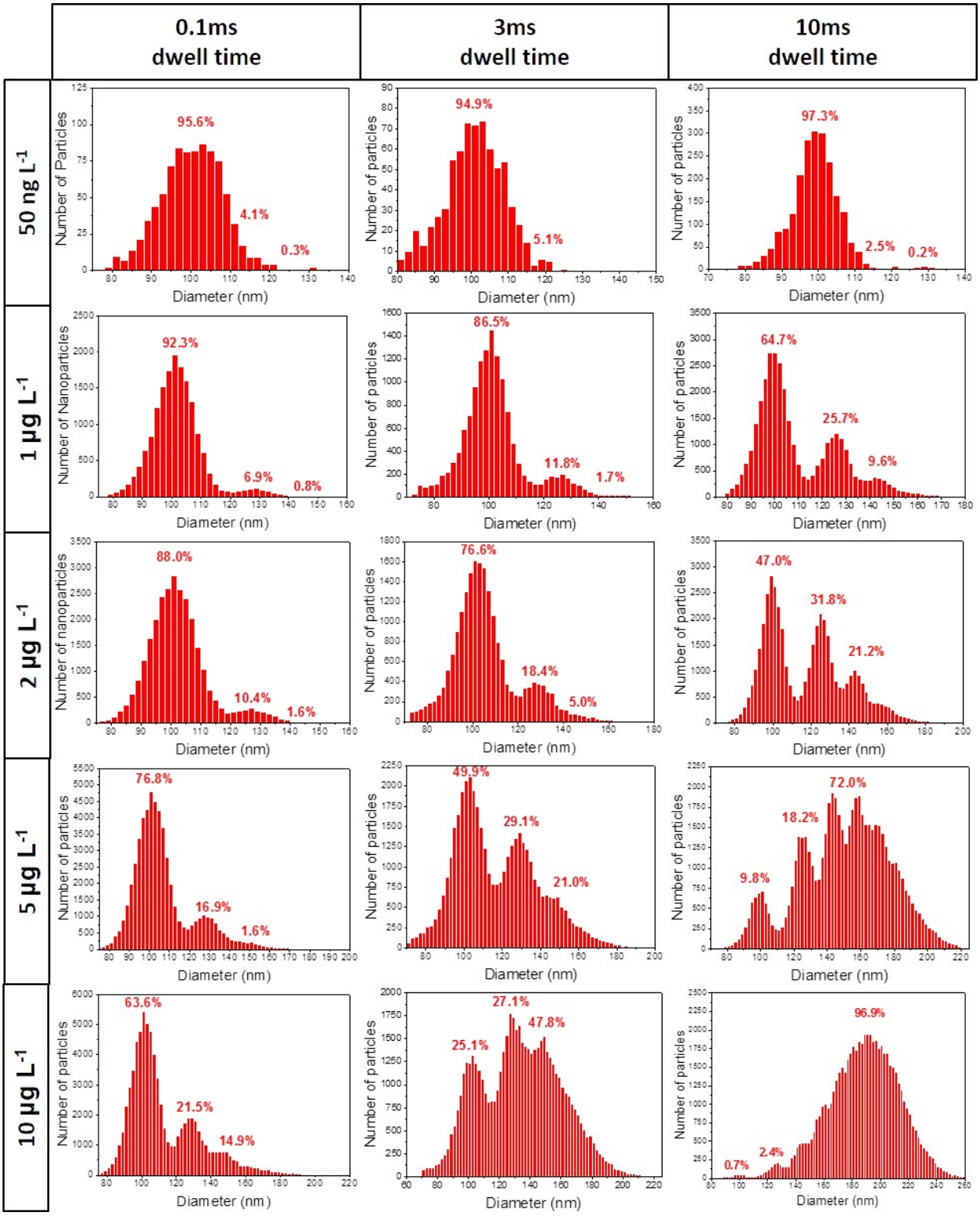


Figure S7. Additional count distribution plots of 60nm citrate-capped silver nanoparticles (50ppt by Ag mass) at increasing concentrations of dissolved silver (50, 200, 500ppt dissolved Ag^{+}). Analysis performed at three different dwell times (0.1, 3, and 10ms) to demonstrate increase in resolution from dissolved background with shorter dwell times. Computed sizes are shown when sufficient resolution from the background is present.

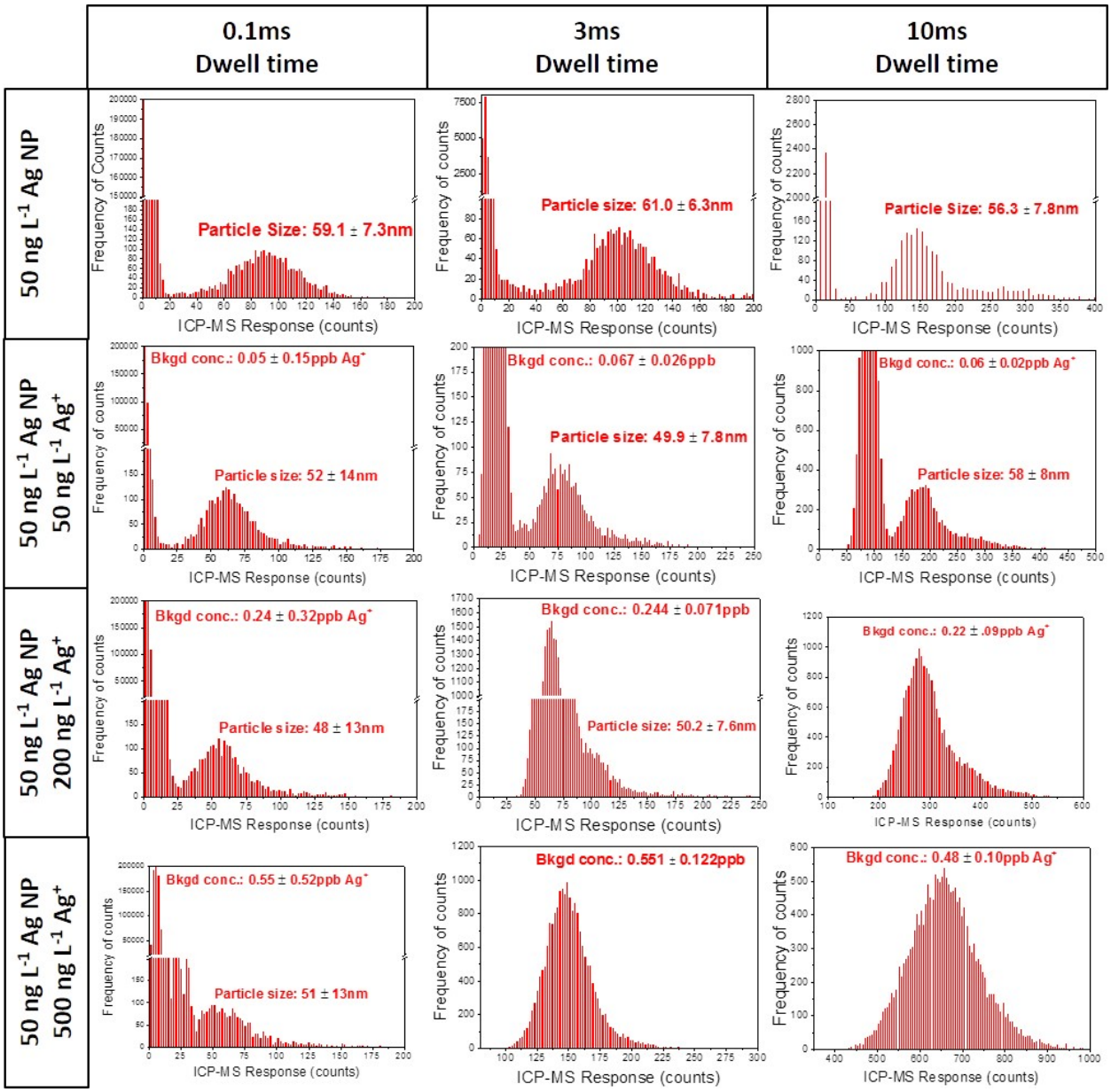


Figure S8. Screen capture of analysis tab from nano software package (Perkin Elmer)

Workflow: Contains the batch file for sample information such as autosampler location, sample name, sample type, analysis method, transport efficiency, and sample flow rate.

Method: Loaded method detailing dwell time used, sample time, analyte of interest with pertinent information (i.e. mass fraction, density, ionization efficiency), dissolved calibration standards, and particle size standards (if needed).

Calibration: Provides the dissolved and particle calibration curves for the analysis method being used.

Realtime Signal: Window showing data collection of signal at dwell time specified over a user-specified time span.

Realtime Histogram: A generated histogram showing the distribution of count intensities (peak area) as analysis is proceeding.

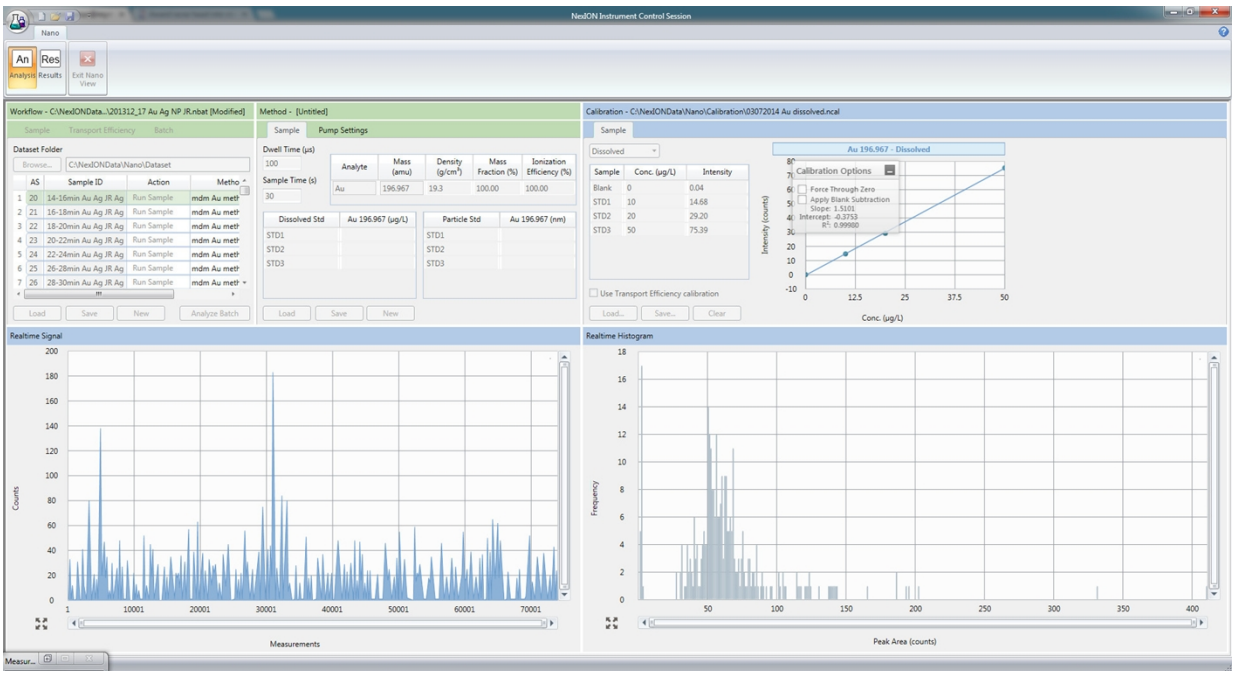


Figure S9. Screen capture of results tab from Nano software package.

Method: Describes the analyte of interest, density of particle, and the mass fraction of the analyte of interest contained in the particle. In addition, the transport efficiency (determined from analysis of a gold NP standard) is also shown and used in size calculations.

Calibrations: Shows the calibration plot for the dissolved and/or particle standards used in determining the mass flux of analyte in the analysis.

Results Grid: Tabulated information of analyzed samples. Details particle size as calculated by dissolved standards calibration curve and/or particle standard calibration curve. The number of peaks (related to particle number concentration) is also shown. Other information related to the average count intensity generated from the particle and generated from the dissolved background is also shown.

Histogram: Shows the size distribution of the analyzed particle. The window below the histogram can be repositioned to exclude background or other particle counts. Particle size can be fitted to a LogNormal, Gaussian, or Max Intensity fit.