attom ES

Attom Note **NA20**

Nanoparticle Analysis in the Presence of an Ionic Background Signal Using Attom ES

- **Nanoparticle analysis at 10^5 Hz**
- **Intelligent peak search and background correction**
- **Reporting particle size with particle and ionic concentration**
- **Powerful data reduction capability**

Introduction

The ICP-MS technique has promised to be a powerful analytical tool for the measurement of nanoparticles but has generally been found to be on the very edge of performance limitations for many applications, especially when measuring samples with ionic metal content. The continuous background signal from the ionic element degrades the analysis due in part to the reduced ability to distinguish background signals from nanoparticle signals and a level of uncertainty introduced by the required background signal subtraction [1].

A more robust method of distinguishing particle signals from background would open up greater possibilities for researchers in the field of nanoparticle analysis.

Data Acquisition

AttoM is able to acquire single ion data with dwell times of 20 µs to 50 µs continuously with no interruptions in the stream of data even in the presence of a continuous background signal. This fast data acquisition allows good discrimination of individual particles [2] but there is still a requirement to consider the ionic background. At 20 µs dwell a single count measured equates to $5e^4$ cps which, depending on the instrument sensitivity and isotope being measured, is approximately 50 ppt to 10 ppb of ionic background. Statistically, the signal from the ionic background will be measured in a generally randomised pattern which means that counts will not be evenly distributed between measurement cycles. This is assuming no cyclical noise from the sample introduction system being used and generally, a free running nebuliser rather than one that is pumped will prevent the worst cyclical noise.

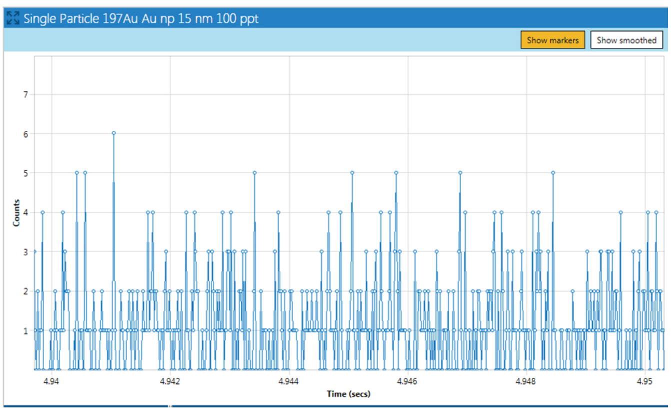


Figure 1: Showing the random and uneven distribution of signals found for the ionic background equivalent to 46,000 cps measured with 20 µs dwell times (i.e. average of 0.92 counts per step).

To distinguish nanoparticle signals from the background, the data processing method needs to be able to sufficiently smooth the background signal and then differentiate peaks using a statistical test based on the noise of the signal.

Data Processing

Nu Quant allows the user to smooth data over multiple iterations and then set criteria for peak detection based on minimum peak width and multiples of the standard deviation of the background noise.

By using a robust peak integration and background determination technique, it is possible to accurately subtract the background from the integrated peaks and report the correct particle sizes. A single calibration of particle size in de-ionised water standards can then be used to determine sizes for samples with variable back ground signals from ionic content.

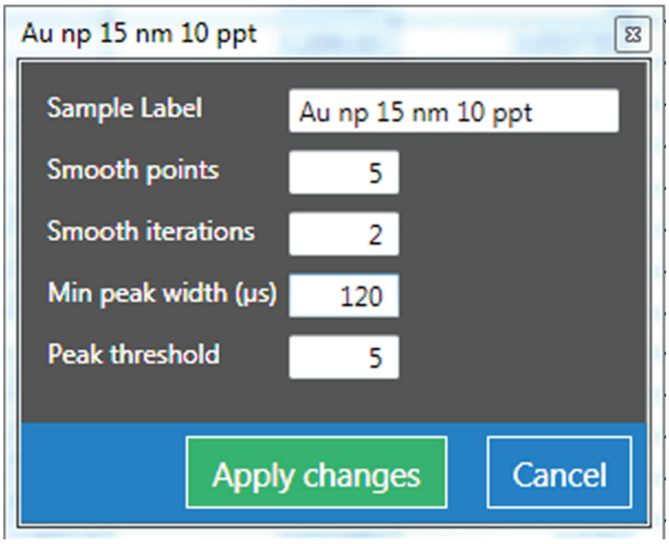
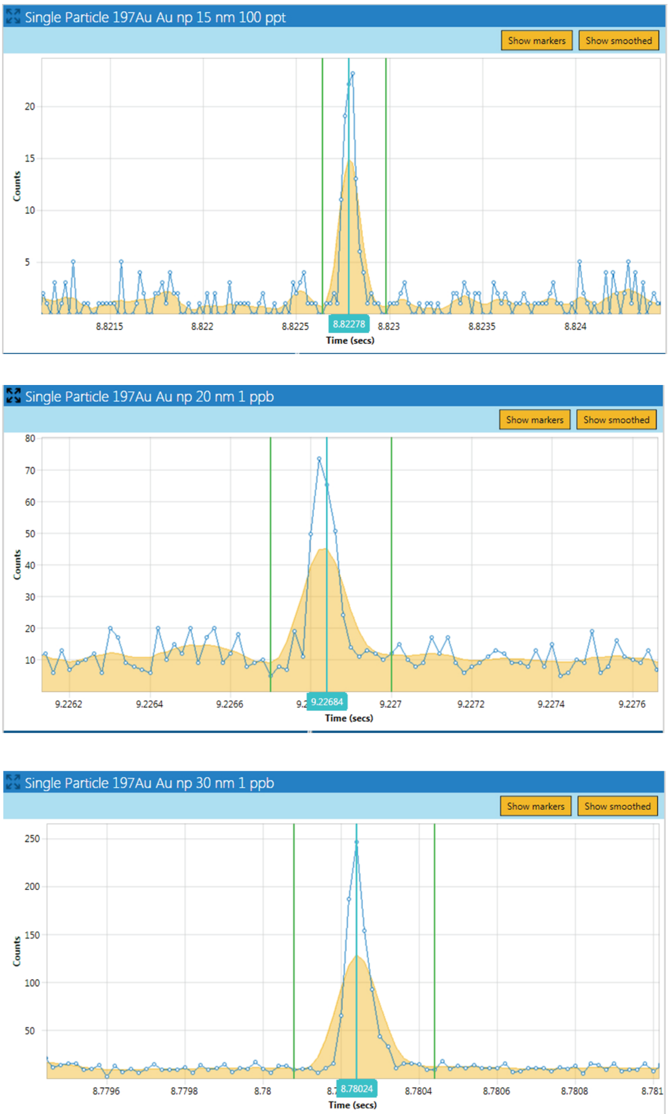
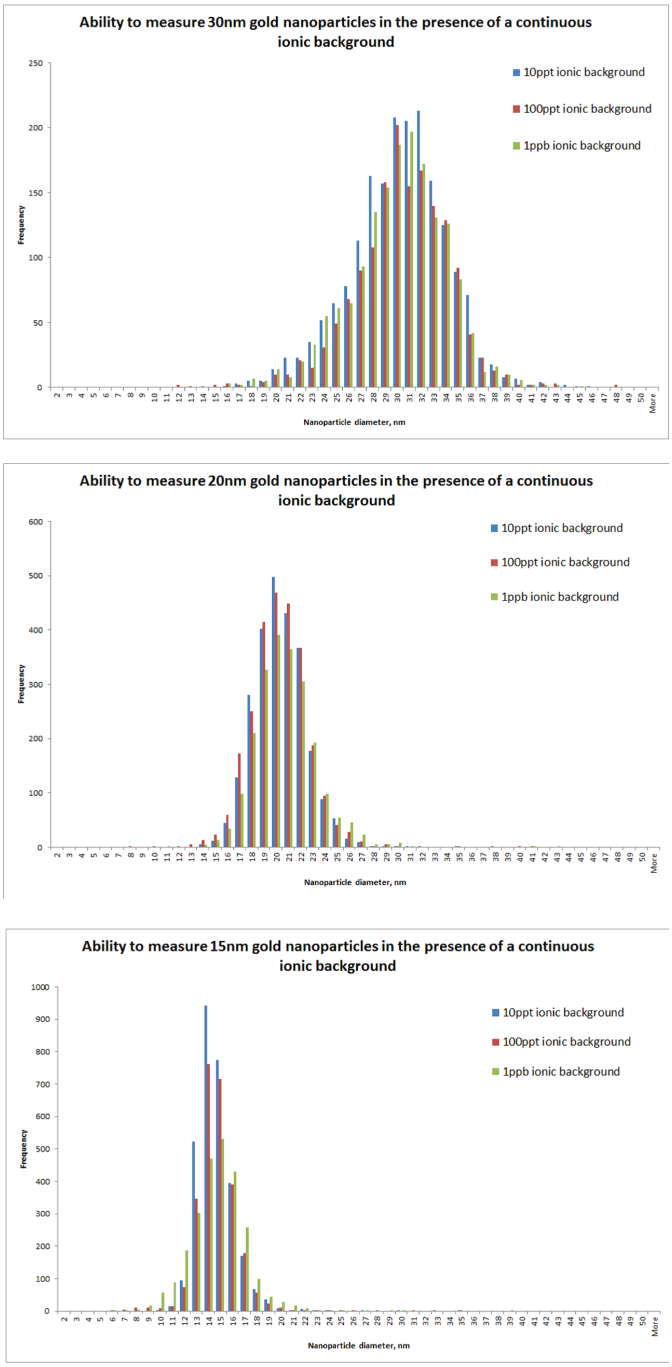


Figure 2: Screenshot showing the available smoothing and peak search criteria which can be tailored for each sample in a batch to optimise peak detection.

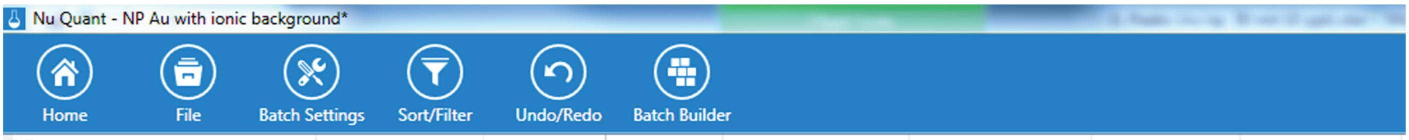


Figures 3a, b, and c: Shows the raw and smoothed data with peak detection for gold nanoparticals in a sample with significant ionic backgrounds. Data acquired using a 20 µs dwell time.



Figures 4a, b, and c: Comparison of the particle size found for 15 nm, 20 nm and 30 nm standards doped with lO ppt, lOO ppt and 1 ppb ionic gold using 20 µs dwell time acquisitions.

Nu Quant automatically calculates the average and standard deviation of signals from all data not found to be part of a nanoparticle peak. This data can then be used to report the ionic background for the sample and the estimated particle size detection limit.



		Method1 197Au (counts)	Particle D, nm	Particle D det. limit, nm	Ionic background, ng/l	% coincident peak	% unresolved peaks
Au np 15 nm	08/12/2015 13:23:54	73.8697			0.245991	0.290416	6.90545
Au np 20 nm	08/12/2015 14:24:20	208.394			0.350984	0	3.67047
Au np 30 nm	08/12/2015 15:18:28	701.22			0.209193	0.053735	1.5583
Au np 15 nm 10 ppt	08/12/2015 13:31:28	78.6071	14.2198	6.3444	5.44438	0.557743	3.34646
Au np 15 nm 100 ppt	08/12/2015 13:42:54	79.5561	14.2524	10.1142	95.3016	0.342988	3.58232
Au np 15 nm 1 ppb	08/12/2015 13:51:38	84.5473	14.3654	15.1749	1044.42	0.934944	7.1679
Au np 20 nm 10 ppt	08/12/2015 14:32:51	215.746	19.9489	6.37519	5.40967	0.198334	1.38834
Au np 20 nm 100 ppt	08/12/2015 14:41:46	214.559	19.8647	10.1197	95.3228	0.153905	2.03925
Au np 20 nm 1 ppb	08/12/2015 14:52:04	231.829	20.3351	15.1896	1050.77	0.411147	1.78164
Au np 30 nm 10 ppt	08/12/2015 15:27:58	731.528	29.8113	6.7458	4.53994	0.106781	1.17459
Au np 30 nm 100 ppt	08/12/2015 15:36:50	745.601	30.0159	10.3046	98.4431	0.128123	0.576553
Au np 30 nm 1 ppb	08/12/2015 15:46:16	723.387	29.7127	15.2884	1087.11	0.06079	0.729483

Figure 5: Screenshot of Nu Quant showing 3 calibration samples in deionised water and the report of particle size, particle size detection limit and ionic gold background for the same standards spiked with different levels of ionic gold standard.

Summary

Nu AttoM ES is proven to be a powerful and flexible ICP-MS for nanoparticle analysis. It is capable of fast data acquisition up to 10^5 Hz with a low dead time detector capable of measuring signals in excess of the equivalent of $1e^7$ cps.

Nu Quant easily and accurately integrates nanoparticle peak data for wide range of sizes and background levels without compromise. The inbuilt NICE scripts are flexible and powerful allowing the calculation of final results for a batch of samples quickly and simply. Reports are available of particle size and concentration as well as estimations of size detection limits and the ionic background content of samples.

References

{1} Peters et al: J. Anal. At. Spectrom., 2015, 30, 1274-1285
{2} Strenge and Engelhard: DOI: 10.1039/c5ja00177c