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Extending the Concentration Range of Nanoparticle Analysis Using Attom ES

- **Nanoparticle analysis at 10^5 Hz**
- **Flexible peak detection and integration**
- **Measurement of high particle concentration samples**
- **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 including where samples have a range of particle concentrations. Traditionally, ICP-MS signals for nanoparticles are integrated over fixed time periods and any signal measured within the fixed period is deemed to be due to a single particle. This has been highlighted as a large source of error by several researchers and a generally accepted upper limit of 100 measured particles per sec is used to determine sample dilutions [1, 2]. Whilst the use of dwell times significantly lower than the expected width of a nanoparticle peak does reduce the error, when using a fixed integration window there is still a measurable impact which must be accommodated by running samples diluted enough to prevent particle coincidence[3].

The use of longer ICP-MS dwell times also has the possibility of errors due to split particle events where the signal from one particle is measured over two dwell time slots. This gets calculated as two smaller particles instead of the correct single larger one. The use of much shorter dwell times and appropriate peak integration reduces this error to a negligible one [3].

Extending the range of both size and concentration 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 as low as 10 µs continuously with no interruptions in the stream of data. This has the advantage of being able to distinguish split particle events but with the use of a more sophisticated algorithm for peak integration it also has distinct advantages with respect to particle coincidence.

Data Processing

Using the AttoM's Nu Quant software, a peak search method using smoothed data is able to detect the start of a particle peak, the maximum and then the subsequent end of the peak. Counts are then integrated between the start and end points to be used in further calculations for particle size or mass. The criterion for defining a peak start or end is either that the smoothed signal background has reached zero or that an inflexion point has been found between two adjacent peaks.

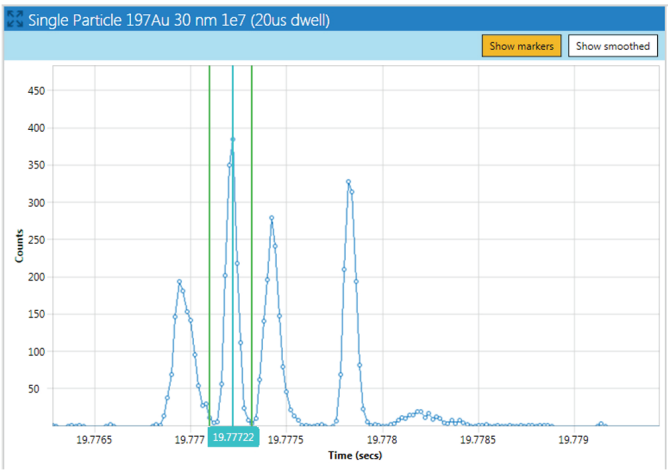

Figure 1: Showing the how Nu Quant can accurately determine the start and end of particle peaks, even when the peaks are clustered together and of varying widths (note the first peak is 280 µs wide at the base and the fourth peak is 180 µs wide so a fixed integration window would not correctly measure all of these peaks).

Calibration

If the particle concentration for one or more standards is known, this can be used to form a calibration using the total number of particle events measured in a given time compared to the known concentration in solution. This calibration can be calculated in Nu Quant along with other parameters and used for subsequent unknown samples. The peak integration method used in Nu Quant allows for linear calibrations to more than 500 particles per second.

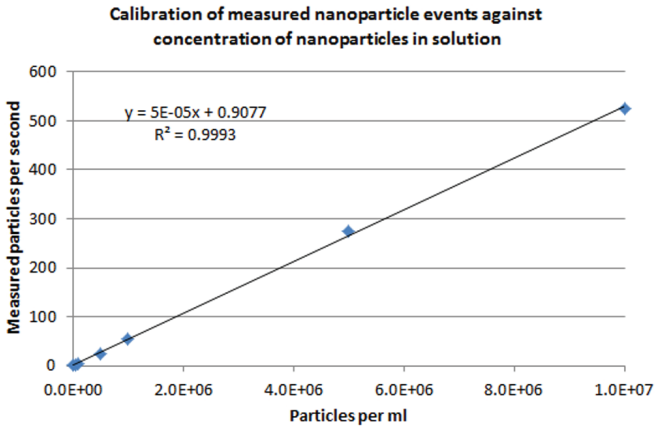

Figure 2: Shows the linear regression fit from a Nu Quant calibration of standards acquired at 40 µS dwell for a range of particle concentrations from $1e^4$ ml^{-1} to $1e^7$ ml^{-1}

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Data Quality Criteria

The Nu Quant peak integration method also calculates the peak width as the full width half maximum (FWHM) which is reported in µs. Where Nu Quant is not able to determine the FWHM due to a noisy signal or unresolved adjacent peak a "null" is reported. Nu Quant is able to report the percentage of found peaks that cannot be fully resolved as a quality criterion. This helps the analyst interpret whether a sample dilution is required to improve data quality.

It is not be possible to determine if two or more particles are fully coincident just by measuring the FWHM. It can be assumed however, that any peak measured which is significantly larger than the mean of all of the peaks could be caused by coincident particles. Nu Quant can report the proportion of measured events where the integrated count exceeds three times the mean of all of the peaks for that sample as a quality criterion.

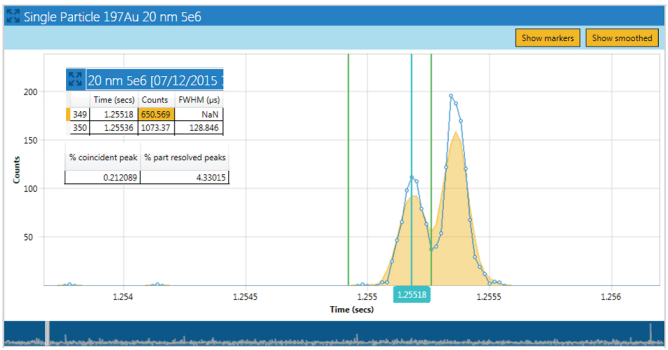

Figure 3: Showing how Nu Quant can report the integrated counts for partially resolved peaks and flag the percentage of partially re solved or coincident peaks.

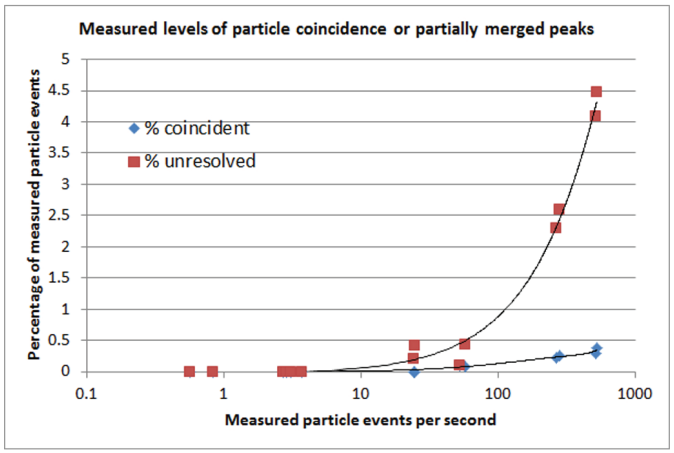

Figure 4: Shows the influence of particle concentration (measured particle events) on the level of partially resolved peaks and possible particle coincidence.

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 can accurately integrate peaks even with several percent of the peaks being only partially resolved. Even at >530 measured particles per second which is over five times greater than that recommended for traditional nanoparticle analysis, the measured level of particle coincidence is <0.5%.

These wide linear ranges for both particle size and particle concentration represent a significant improvement in analytical capability for researchers in the field of nanoparticles.

Nu Quant easily and accurately integrates nanoparticle peak data for wide range of sizes and shapes without compromise. The inbuilt NICE scripts are flexible and powerful allowing the calculation of final results for a batch of samples quickly and simply.

References

{1} 0lesik and Gray: J. Anal. At. Spectrom., 2012, 27, 1143-1155
{2} Peters et al: J. Anal. At. Spectrom., 2015, 30, 1274
{3} Strenge and Engelhard: DOI: 10.1039/c5ja00177c widths, partially merged peaks and continuous background signals to be distinguished.

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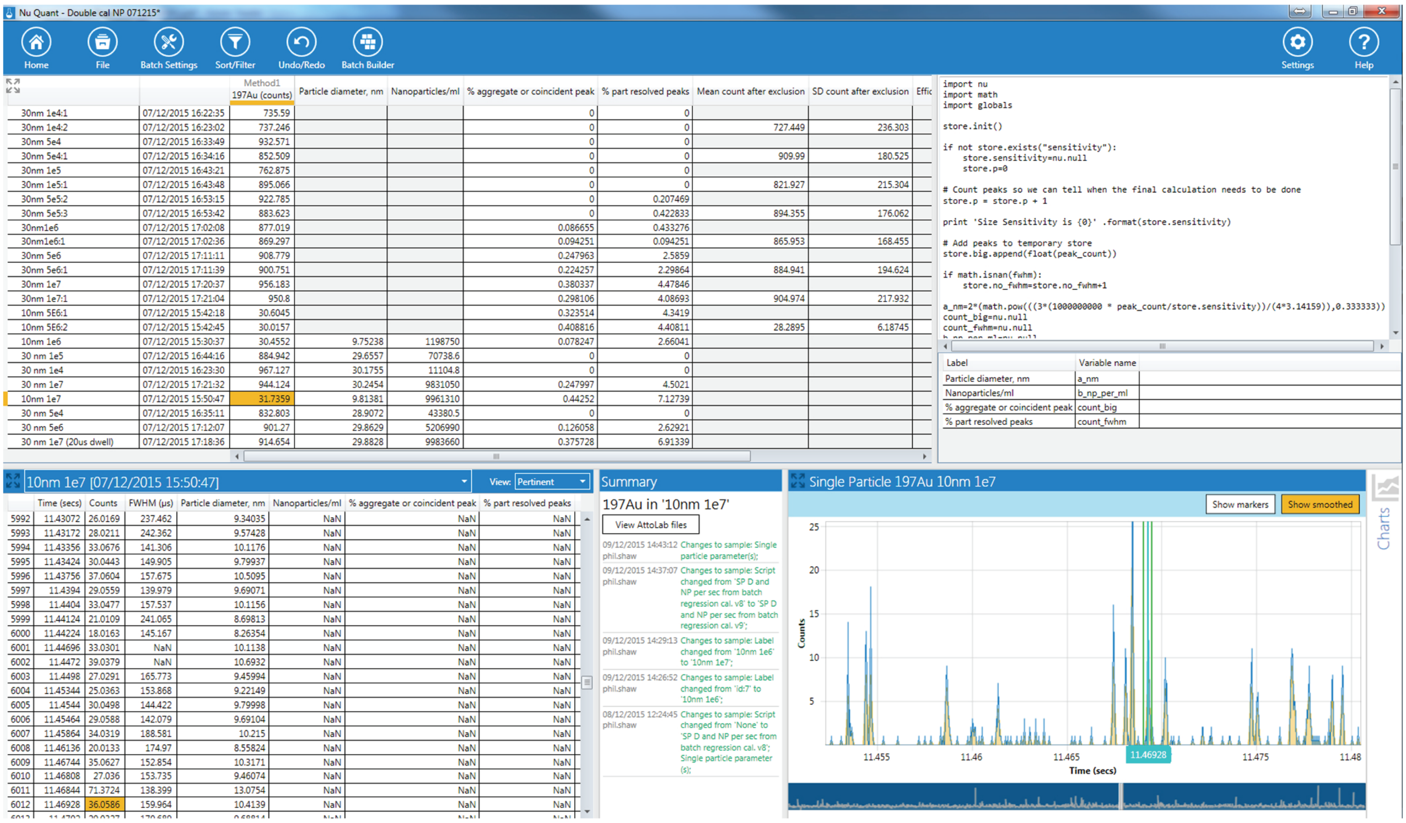

Figure 5: Screenshot showing a Nu Quant batch of calibration standards to calibrate the particle transfer efficiency and then the linear measurement of 10 nm (spectra shown) and 30 nm samples at up to 1e[7] partic/es/ml (measured as >530 particle events/sec).