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Single Particle ICP-MS as a Screening Tool for Nanoparticles

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Introduction

Products based on nanotechnology are already manufactured in the field of electronics, consumer products and pharmaceutical industry, and are beginning to impact the food associated industries. For example, silica is present in nano form in food while dispersions of titanium, iridium, copper, gold and zinc nanoparticles (NP) can be found in nutritional supplements.

Characterisation of NP in food and consumer products is urgently needed to provide customers and risk assessors with reliable information. We therefore studied a time-modulated, inductively coupled plasma mass spectrometry (ICP-MS) method for the determination of NP.

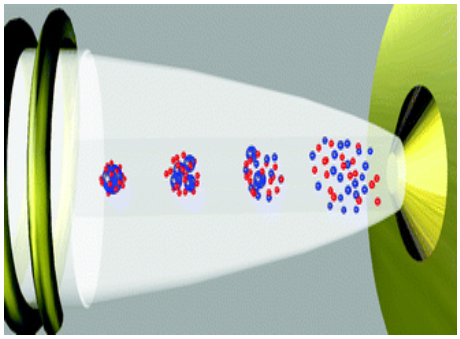

Figure 1: Plume of ions originating from a nanoparticle introduced in the ICP-MS

Principle of Single Particle ICP-MS

When NP are introduced into the ICP-MS they produce a plume of analyte ions. This plume is detected as a signal spike in the mass spectrometer. Using dwell times in the range of 1-10 ms allows the detection of individual NP. Compare figure 2 a and b for the ICP-MS signal of dissolved gold versus gold NP. Using an ionic calibration standard the spikes can be quantified and the mass of the NP determined. From this the size of the particle can be calculated.

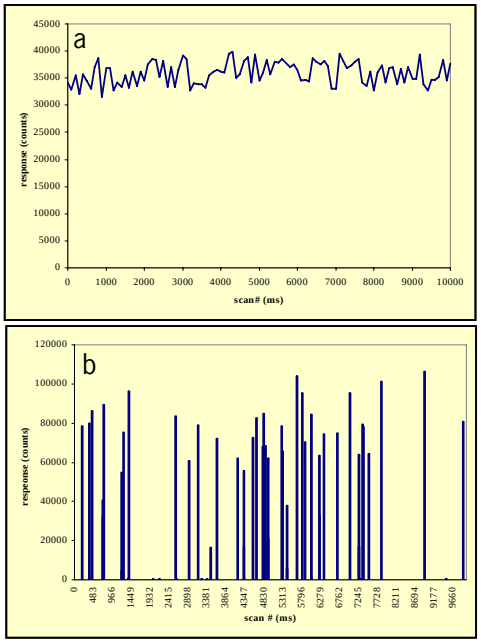

Figure 2: ICP-MS signal of dissolved gold (a) and gold nanoparticles (b)

Results

The method was tested using NIST RM8011, -8012 and -8013 gold NP. The results showed that the method allows the accurate determination of NP concentration and size. Only RM8011 (nominal 10 nm) could not be detected because of its small particle size.

The dwell time is of particular importance since it determines the performance of the method. With a dwell time of 1 ms single NP (with a typical ion plume width of 0.3 ms) may fall into two adjacent scans resulting in an overestimation of the number of NP. The dwell time also limits the dynamic range since multiple NP in one scan should be avoided. A dwell time of 3 ms was found as a good compromise. Measurement times are typically 1 minute.

Advantages of the method are that sample preparation is easy (often only dilution), that NP interactions are minimised, that low detection limits can be obtained, and that the ion-/NP-form ratio of the material in the sample can be determined. The dynamic range of the method is 1-1000 ng/L (60 nm particle) with a detection limit of 0.1 ng/L.

The method was successfully used to detect and measure NP in food and samples from toxicological studies. Food samples are enzymatically digested and the digest diluted. Samples from toxicological studies are generally diluted directly. Figure 3 shows the size distribution of silver NP in a digest sample as determined with single particle ICP-MS. The NP manufacturer states a particle size of 67 ± 17 nm.

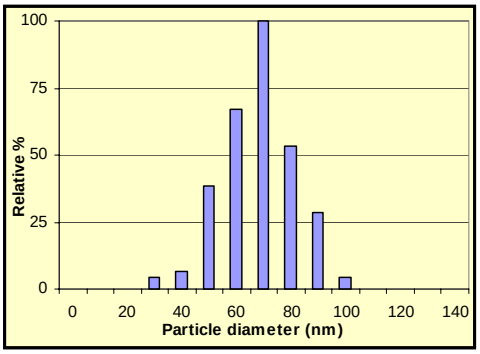

Figure 3: Size distribution of silver nanoparticles in a digestion sample (concentration 25 ng/L).

Conclusion and outlook

Single particle ICP-MS is a fast and easy method to screen samples for the presence of NP with sizes >20 nm. Sample preparation is often limited and NP interactions minimised. Further research should show whether smaller particle sizes can be determined using high resolution ICP-MS.

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