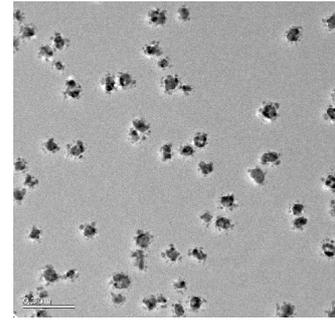


Aim of the study

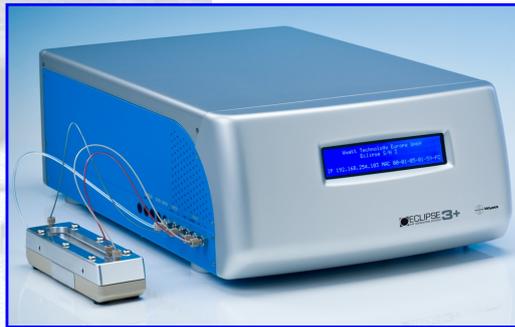
Engineered nanoparticles (NPs) have already found their way from science to various applications in everyday life and in consumer products. Especially Ag-, TiO₂ and ZnO NPs are commonly used in this area. However, NPs used in spray products may exert critical effects on exposed users. To understand the effects of release, exposure and transport of NPs and to evaluate possible risks for humans, selective and sensitive analytical methods are necessary in order to detect these materials in aerosols or liquid solutions. A new and promising technique for the size separation of nanoparticles for subsequent characterisation is the asymmetric flow field flow fractionation (AF4). To achieve this, the combination of AF4 with inductively coupled plasma mass spectrometry (ICPMS) and other detectors, e.g. UV/VIS or light scattering, represents a powerful analytical method. In this poster we present the characterisation of Ag-nanoparticles using this approach. We show that this technique yields highly reliable data that are comparable to results derived from electron microscopy imaging.



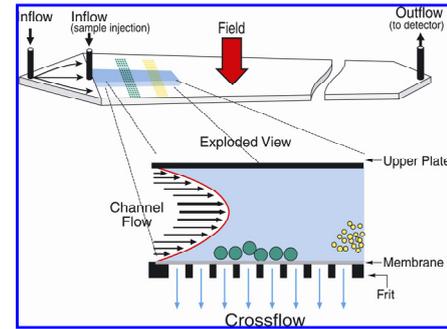
Methods and Instrumentation

For the separation and detection an asymmetric flow field flow fractionation (AF4) system coupled to ICPMS was used. Size calibration was performed with NIST Au standards (SRM 8011, 8012 & 8013) with a diameter of 10, 30 and 60 nm. The resulting particle distribution was compared with data from electron microscopy imaging. For specification and parameters of instruments see table.

Wyatt Eclipse3™ AF4		Thermo Finnigan Element2 ICPMS	
Carrier	18 ml/cm D water	RF - Power	1400 W
Channel Flow (Vc)	0.5 mL/min	Reflected Power	< 4 W
Cross Flow (Vx)	1 mL/min	Plasma Gas	16 L/min
Membrane Type	GE PVDF 30 kD	Nebulizer	ESI PFA-ST @ 1 L/min
Channel/Spacer	30 cm channel, small spacer, 350 µm height	Spray Chamber	Scott double pass, quartz, cooled to 5 °C
Injection Volume	10 µL	Integration Time	300 ms in E-Scan Mode

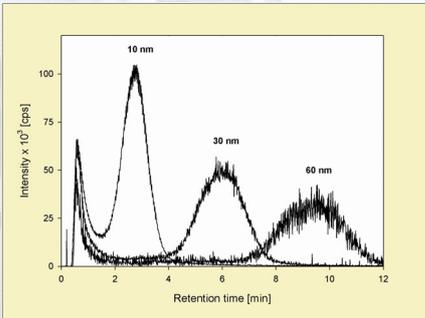


High-end equipment for particle separation: Wyatt's Eclipse AF4 instrument

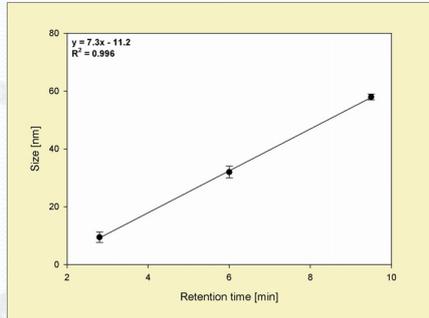


The principle of particle separation by AF4

System Calibration

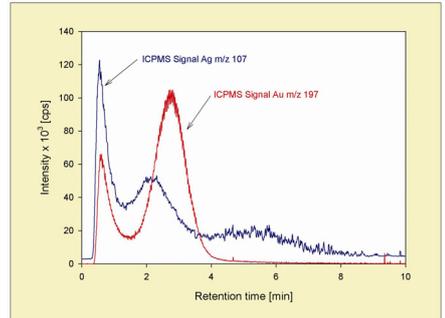


Separation of NIST Au particle standards (Diameter [Ø] 10, 30 and 60 nm) of each 50 g Au/L with AF4 and ICPMS detection (m/z 197).



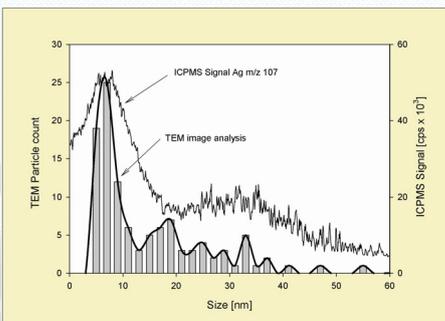
Calibration with NIST Au particle standards (Ø 10, 30 and 60 nm) - plotting size vs. retention time allows calculation of particle distribution.

Measurement of Silver Nanoparticles in Consumer Spray Product



AF4 separation of a nanoscale Ag spray product (dilution 1:2000) and spike of 50 g Au/L with Ø 10 nm and ICPMS detection. The particle size of this polydisperse sample varies between 6 and 30 nm.

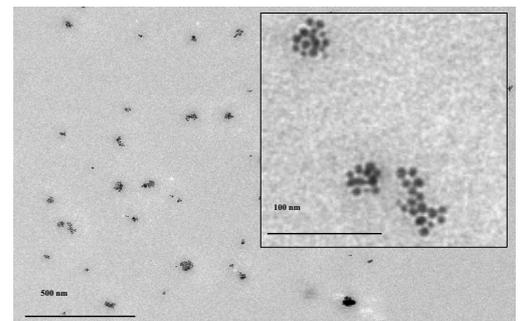
Evaluation of Data: Comparison with Electron Microscopy Results



Size distribution of a nanoscale Ag spray product: AF4 separation with ICPMS detection vs. image analysis of TEM pictures (n=160 particles) shows strong correlation of data.

Summary

Coupling of nanoparticle separation by AF4 to ICPMS detection results in a very sensitive method for nanoparticle analysis which has a large range of detection. The data obtained with this approach are comparable to those achieved by electron microscopy, the latter of which is much more expensive and troublesome. In this study, the analysis of a spray sample shows a polydisperse distribution of particles from approx. 6 to 30 nm in size. During the spraying process, this pattern changes due to the formation of larger nanoparticle aggregates.



Particle distribution changes during a spray event: larger particles are formed by agglomeration or aggregation processes (electron micrograph).

