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Quantification and size characterisation of silver nanoparticles in environmental aqueous samples and consumer products by single particle-ICPMS

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Abstract

Single particle-inductively coupled plasma mass spectrometry (SP-ICPMS) is a promising technique able to generate the number based-particle size distribution (PSD) of nanoparticles (NPs) in aqueous suspensions. However, SP-ICPMS analysis is not consolidated as routine-technique yet and is not typically applied to real test samples with unknown composition. This work presents a methodology to detect, quantify and characterise the number-based PSD of Ag-NPs in different environmental aqueous samples (drinking and lake waters), aqueous samples derived from migration tests and consumer products using SP-ICPMS. The procedure is built from a pragmatic view and involves the analysis of serial dilutions of the original sample until no variation in the measured size values is observed while keeping particle counts proportional to the dilution applied. After evaluation of the analytical figures of merit, the SP-ICPMS method exhibited excellent linearity ($r^2 > 0.999$) in the range $(1 - 25) \times 10^4$ particles mL^{-1} for 30, 50 and 80 nm nominal size Ag-NPs standards. The precision in terms of repeatability was studied according to the RSDs of the measured size and particle number concentration values and a t-test ($p=95\%$) at the two intermediate concentration levels was applied to determine the bias of SP-ICPMS size values compared to reference values. The method showed good repeatability and an overall acceptable bias in the studied concentration range. The experimental minimum detectable size for Ag-NPs ranged between 12-15 nm. Additionally, results derived from direct SP-ICPMS analysis were compared to the results conducted for fractions collected by asymmetric flow-field flow fractionation and supernatant fractions after centrifugal filtration. The method has been successfully applied to determine the presence of Ag-NPs in: lake water; tap water; tap water filtered by a filter jar; seven different liquid silver-based consumer

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