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## New Single Particle Methods for Detection and Characterization of Nanoparticles in Environmental Samples

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Akademisk avhandling för filosofie doktorsexamen i naturvetenskap, inriktning kemi som med tillstånd från Naturvetenskapliga fakulteten kommer att offentligt försvaras tisdagen den 1, 10, 2013 kl. 13:15 i sal KA, Institutionen för kemi och molekylärbiologi, Kemigården 4, Göteborg.

ISBN: 978-91-628-8769-8



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## ABSTRACT

Nanoparticles (NP) are being used in rapidly increasing quantities which has resulted in concerns about possible harmful effects for health and environment. NP are already undergoing similar risk assessment programs as conventional chemicals, and due to their enhanced surface reactivities it has been proposed that the use of NP should be regulated by specific legislation based on particle size.

Number based concentrations and size distributions are thought to be more relevant dose metrics for toxicology than the mass of NP. Because NP are prone to processes such as aggregation, dissolution, or adsorption on surfaces characterization is required during the whole test. To measure the emission of NP and exposure levels in the environment the methods have to be capable of quantifying and sizing particles of interest at parts per billion level concentrations or lower.

Nanoparticle tracking analysis (NTA) was evaluated for measurement of number concentration and size distributions. The technique was considered suitable for monitoring and measuring exposure at relatively high (>  $10^6$  particles mL<sup>-1</sup>) concentrations; however, NTA is relatively unspecific in the sense that it is difficult to distinguish particles of different materials.

To increase sensitivity and specificity single particle inductively coupled plasma mass spectrometry (spICP-MS) was developed for element specific characterization of particles in liquid samples. Validation of both the number concentration and sizing capabilities was carried out at concentrations as low as 10<sup>2</sup> particles mL<sup>-1</sup>. The capabilities of spICP-MS as a fast screening tool for NP was evaluated, and the method was used to quantify trace level contamination of WC particles emitted from wear of winter tire studs and hard coatings.

Variable pressure or environmental scanning electron microscopes (ESEM) can be applied on a waist range of sample types with no or very little sample preparation. Therefore backscattered electron (BSE) imaging in such instrument was chosen as a base for developing a method for quantification of particles in solid samples. The technique was applied for quantifying particles in toxicity tests involving soil biota, and was concluded to be sensitive enough to cover the concentration range that is typically of interest in such tests.

Finally it was concluded that due to the tremendous amount of information obtained on a single particle basis, electron microscopy is a suitable complementing technique for spICP-MS measurements, which otherwise give little information about the structure of the particles.

<u>Keywords</u>: Nanoparticle Metrology Trace particle Characterisation Number concentration