SYNTHÈSE DE SPHÈRES D'ARGENT DE 2 À 100 NM À HAUTE CONCENTRATION POUR LA CALIBRATION DES CPC ET DES DMA : UNE SOLUTION INSTRUMENTALE PRÊTE À L'EMPLOI

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TITLE

Generating 2 to 100 nm Silver Spheres in High Concentrations for CPC and DMA Calibration: A Turnkey Instrumental Setup

RESUME

Un étalonnage précis des instruments de mesure des aérosols requiert une source d'aérosols stable et bien définie produisant des particules sphériques solides, inertes, insolubles et thermiquement stables. Les particules d'argent peuvent satisfaire à l'ensemble de ces critères lorsque l'aérosol est généré et traité de manière appropriée. Nous présentons ici une approche de haute qualité, fondée sur une configuration remarquablement simple, permettant de produire des particules sphériques d'argent compactes et thermiquement stables jusqu'à 100 nm pour l'étalonnage des compteurs de particules par condensation (CPC), des analyseurs de mobilité différentielle (DMA) et des facteurs de réduction de concentration des particules (PCRF).

ABSTRACT

Accurate calibration of aerosol instruments requires a stable, well-defined aerosol source that produces solid, inert, insoluble, and thermally stable spherical particles. Silver particles can fulfil all those properties if the aerosol is produced and treated in a certain way. Here we demonstrate a high-quality approach built on an elegantly simple setup to produce thermally stable and compact spherical silver particles up to 100 nm for Condensation Particle Counter (CPC), Differential Mobility Analyser (DMA) and Particle Concentration Reduction Factor (PCRF) calibration.

MOTS-CLES : nanoparticules d'argent, thermiquement stable, sphérique, étalonnage / **KEYWORDS:** silver nanoparticles, thermally stable, spherical, calibration.

1. INTRODUCTION

As measurements of ultrafine particles (UFPs) become more crucial and regulated, especially for ambient and transport-related emissions (e.g., EU directive 2019/1242, UNECE Regulation No. 83 (Euro 7)), ensuring good agreement and comparability between different measurement techniques is becoming increasingly important (Lahde *et al*, 2023). To ensure this, various standards for UFP instrumentation were created, e.g., ISO27891:2015 and EN16976:2024 for Condensation Particle Counters (CPCs) or the Particle Concentration Reduction Factor (PCRF) for Volatile Particle Remover (VPR) systems (PMP UNECE Regulation No. 83 (Euro 7), Mamakos, 2012). A stable and defined aerosol source is essential for these standards and calibrations, ideally producing solid, unreactive, insoluble, and thermally stable spherical particles. Salts, emery oil or soot are often used, but they do not satisfy all the mentioned properties (Wlasits *et al*, 2020; Giechaskiel and Melas, 2022). However, silver particles can fulfil all of those properties if the aerosol is produced and treated in a certain way. Here we demonstrate a high-quality approach built on an elegantly simple setup to produce thermally stable and compact spherical silver particles up to 100 nm for CPC and PCRF calibration.

2. SIMPLE SPG SETUP FOR GENERATING 100 NM PARTICLES

Spherical silver particles are commonly created by sintering agglomerated silver particles (Ku and Maynard, 2006; Tuch *et al*, 2016). However, the biggest challenge is to make spherical particles in high enough concentrations for the application without secondary coagulation after the sintering process, which would compromise the sphericity of the particles. Normally this is done by a complicated tandem Differential Mobility Analyser (DMA) setup. Figure 1 presents an elegantly simple experimental setup that successfully overcomes this challenge producing thermally stable and compact spherical silver particles in high concentrations.

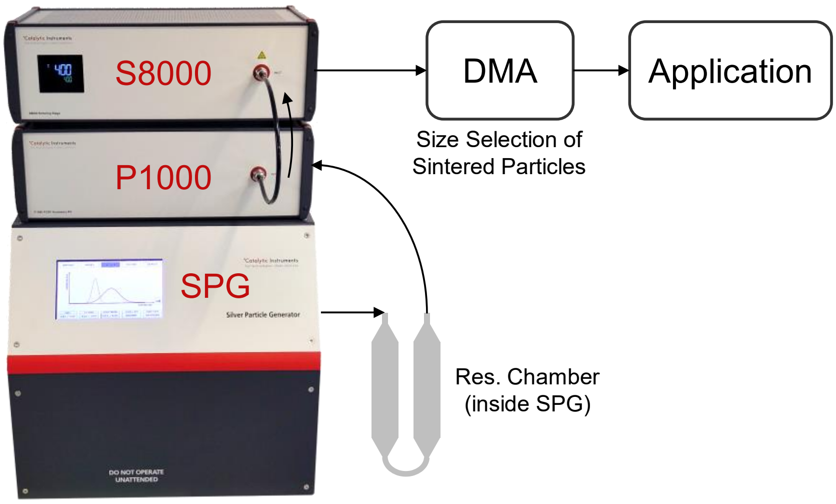

Figure 1. Experimental Setup for generating spherical and thermally stable silver particles with the SPG.

The silver particles are produced with the Silver Particle Generator (SPG) from Catalytic Instruments (Hammer et al, 2022). The SPG's ability to control the heater temperature in precise 1°C steps not only allows for stable production of narrow size distributions of spherical silver particles but also enables the ability to shift these distributions by tuning the temperature. The resulting raw output size distributions for different temperatures are shown in Figure 2.

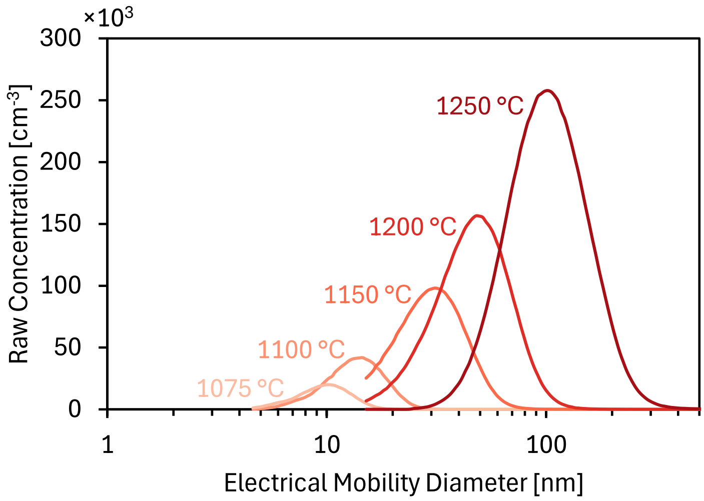

Figure 2. Raw output size distributions of the SPG for different temperatures.

After the SPG, coagulation processes in the SPG-integrated residence chamber and the externally connected Catalytic Instruments PCRF Accessory Kit (P1000) form large agglomerates and concurrently remove the remaining small silver particles by diffusion. The large agglomerates enter the Particle Sintering Device (S8000 by Catalytic Instruments) where they sinter to thermally stable and spherical particles. The resulting silver particles cover diameters up to 100 nm at concentrations between 10^3 and 10^5 cm^{-3}. These high concentrations are useful for CPC linearity calibration (EN16976 requires concentrations from 10^3 to $5*10^4$ cm^{-3}). Smaller concentrations and sizes down to 2 nm can easily be achieved by lowering the SPG temperature and optimizing flow rate and path. Sizes below 30 nm should be generated without Residence Chamber and without P1000, to reduce agglomeration and diffusion losses. Changes in SPG settings are very quick (< 5 min). This offers the possibility to perform a CPC linearity check not via dilution of generated aerosol but rather generating the aerosol as needed. This eliminates uncertainties from dilution stages and reduces the number of multiply charged particles to a point, where multiple charge correction is not necessary anymore (< 1%). This is achieved by moving the particle size distribution to smaller sizes and therefore always size selecting from the right flank of the PSD at the lowest concentration necessary, minimizing the number of particles that are larger than the selected size. Future work will look into creating a fully automated CPC calibration test bench, bringing down costs and instrument downtimes for CPC users.

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3. CONCLUSION

This study presents an easy, flexible, and stable method for generating solid, unreactive, insoluble, thermally stable, and spherical silver particles with diameters up to 100 nm at concentrations up to 10^5 cm^{-3}. These particles can be used for calibration of CPC, DMA or PCRF or for any other UFP application requiring a highly defined, stable aerosol source.

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