Transformation of Silver Agglomerates via Sintering:

A Study using the SPG and the °Particle Sintering Device

APPLICATION NOTE 030-24

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Introduction

The controlled sintering of silver nanoparticles plays an important role in aerosol science, particularly in the calibration of particle measurement instruments and in environmental monitoring applications. Precise control over the morphology and size of nanoparticles is essential for ensuring the accuracy, stability, and reproducibility of measurement systems. In applications such as the calibration of **Condensation Particle Counters (CPCs)** and **Particulate Number Counting Systems (PNCS)**, well-defined, monodisperse aerosols are required to comply with international standards, such as **ISO 27891:2015 and EN 16976:2024**.

Silver nanoparticles, known for their **thermal stability and chemical innert**, are widely used in such applications (Duman et. al., (2024)). However, their initial formation often results in **irregularly shaped agglomerates**, which can impact measurement accuracy and are required by ISO 27891:2015 and EN 16976:2024 standards. **Controlled sintering** allows the transformation of these agglomerates into compact, spherical particles, thereby enhancing their uniformity and ensuring consistent interaction with measurement systems.

This study investigates the **sintering process of monodisperse silver nanoparticles**, employing a **tandem Differential Mobility Analyzer (DMA) setup** to evaluate size, morphology, and stability. The **°Silver Particle Generator (SPG)** is used to produce silver aerosols with high size precision, while the **°Particle Sintering Device (S8000)** enables controlled reshaping of agglomerates under defined thermal conditions. As demonstrated by **Ku et al. (2006)**, we also observe significant morphological transformations at **400 °C**, where silver agglomerates transition into compact, thermally stable particles. Our findings confirm these previously reported result, highlighting the consistency of sintering effects at this temperature.

These findings are particularly relevant for **emissions testing**, where accurate **particulate matter measurement** is critical (**Ku et al., 2006; Berger et al., 2024**). **Sintered silver particles** improve **thermal stability** and minimize **particle loss** in **Volatile Particle Removers (VPRs)** and related **automotive applications** (**Tuch et al., 2016**). Beyond **calibration**, controlled sintering also benefits **toxicological studies** and **nanomaterial characterization** in **environmental and industrial research** (**Zihlmann et al., 2014; Ku et al., 2006**).

The **SPG** reliably produces **monodisperse silver particles** with a **geometric mean diameter (GMD) deviation of ±1%**, covering a size range from **2 to 300 nm**, ensuring high stability and reproducibility in aerosol generation. When combined with the **S8000**, the system generates **spherical silver particles** with a **mobility diameter of up to 100 nm**, which are comparable to aerosols produced by a **classical double-tube furnace setup**, while offering significant advantages in terms of efficiency and practicality. The **SPG** and **S8000 setup** requires only **one-tenth of the space** of a traditional furnace and reduces the **setup time by 90%**, allowing operation with less skilled personnel and leading to significantly lower overall operational costs, while the cost of consumables remains similar to that of a furnace. Experimental results confirm that **particle reshaping begins at 100 °C and is fully achieved at 400 °C**, demonstrating that **sintered silver aerosols** provide stable and reproducible measurements, reinforcing the **SPG's suitability for precise aerosol calibration** in compliance with **ISO 27891:2015**, while further expanding its applications in **aerosol metrology**.

2. Experimental Setup

2.1 Silver Particle Generation

The **°Silver Particle Generator (SPG)** operates at **1150 °C (Mode 2)**, using a controlled **airflow of 1.55 L/min** to generate silver aerosols. The particles initially form as **irregularly shaped agglomerates**, with mobility diameters typically in the range of **200 to 300 nm**.

The generated particles are heated further in the **S8000**, with temperatures ranging from 20 °C to 700 °C and residence times between 2.6 to 9 seconds. A Tandem Differential Mobility Analyzer setup is used to classify particles before and after sintering, assessing changes in size and shape.

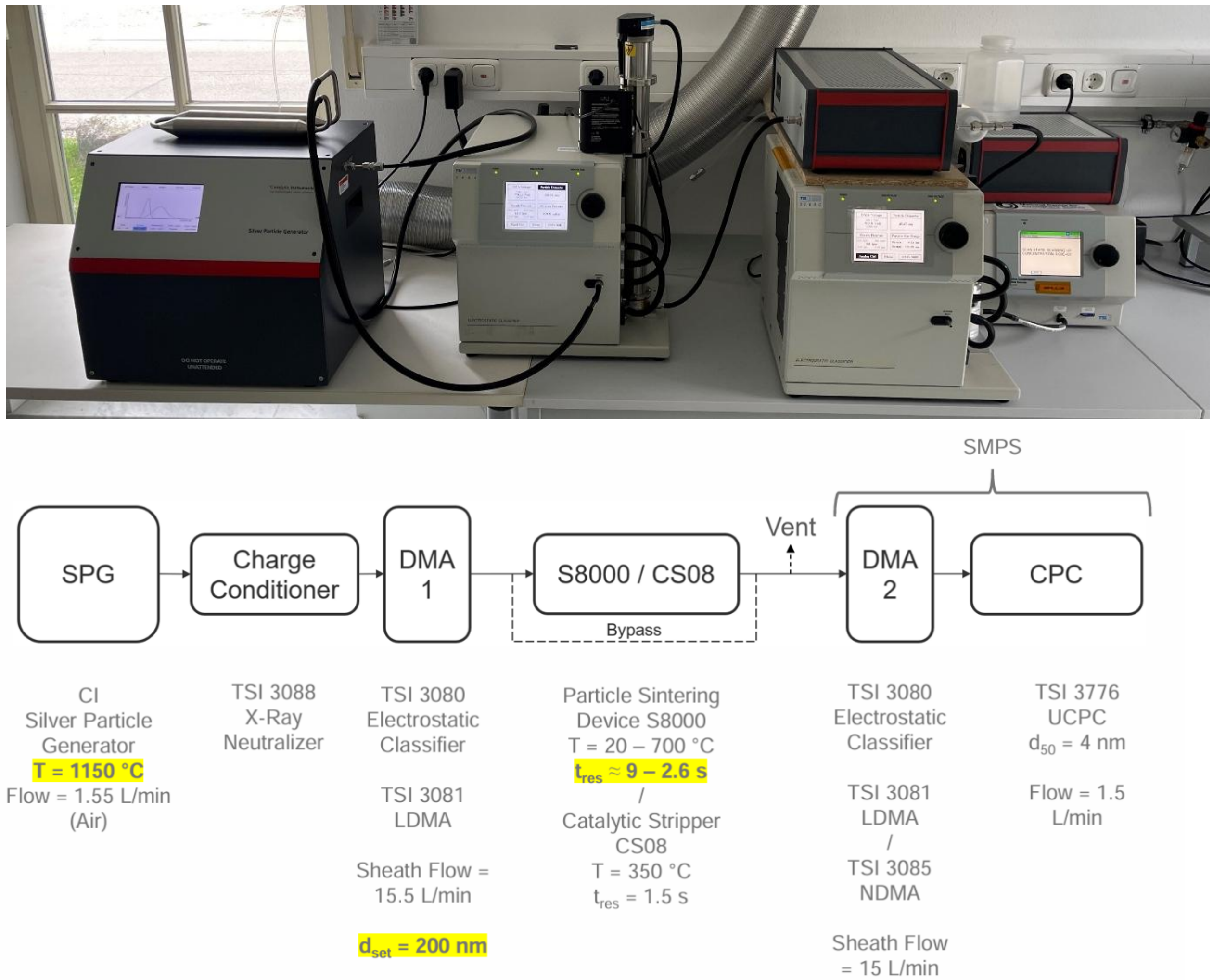


Figure 1 This figure illustrates the complete experimental setup, highlighting the SPG, S8000 sintering device and associated measurement instruments (Berger et al., 2024).

3. Results and Discussion

3.1 Initial Particle Morphology

Prior to sintering, **silver aerosols exist as fractal-like agglomerates**. These agglomerates exhibit high **surface roughness** and variable particle shapes (see Fig. 3).

3.2 Effect of Sintering Temperature

Experimental observations confirm that the **sintering process significantly alters the morphology of silver agglomerates (see Fig. 2)**.

- **100 °C:** Initial restructuring and compaction of agglomerates begin.
- **200 – 300 °C:** Particles exhibit neck formation and partial coalescence.
- **400 °C:** Transformation into compact **spherical particles** occurs.
- **500 – 700 °C:** No further reduction in GMD is observed, confirming that restructuring is complete (see Fig. 3). However smaller particles melt together hence decrease the total number concentration.

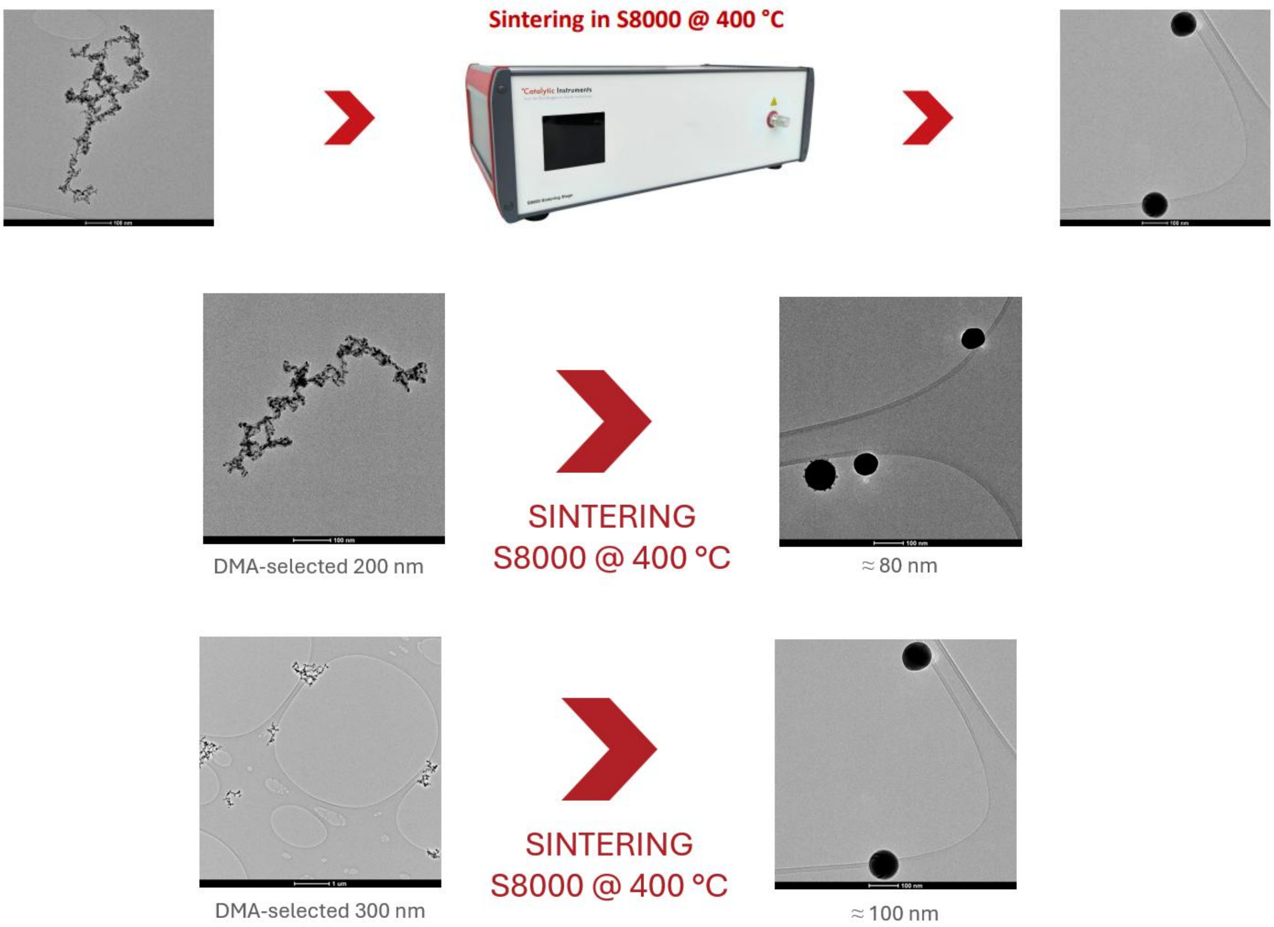


Figure 2 This figure illustrates the complete experimental setup, highlighting the SPG, S8000 sintering device and associated measurement instruments (Berger et al., 2024).

These findings align with previous research by **Ku et al. (2006)**, which indicate that **complete sintering of silver occurs at 400 °C** without further structural changes at higher temperatures.

Particle Morphology: TEM images illustrate the transformation of silver agglomerates into spherical particles at 400 °C. This temperature effectively reshapes 200 nm particles into 80 nm spheres and 300 nm agglomerates into approximately 100 nm spheres.

3.3 Comparison with Other Studies

Research by **Ku et al. (2006)** confirms that **sintering effects begin at 100 °C**, with no significant changes in **GMD** observed beyond **400 °C to 700 °C**. Our results align with these findings, demonstrating that **400 °C is sufficient** to achieve **stable, compact particle morphology** (**see Fig. 2**). The potential influence of **higher temperatures on particle restructuring and long-term stability** remains an open question and will be further investigated in **future studies**.

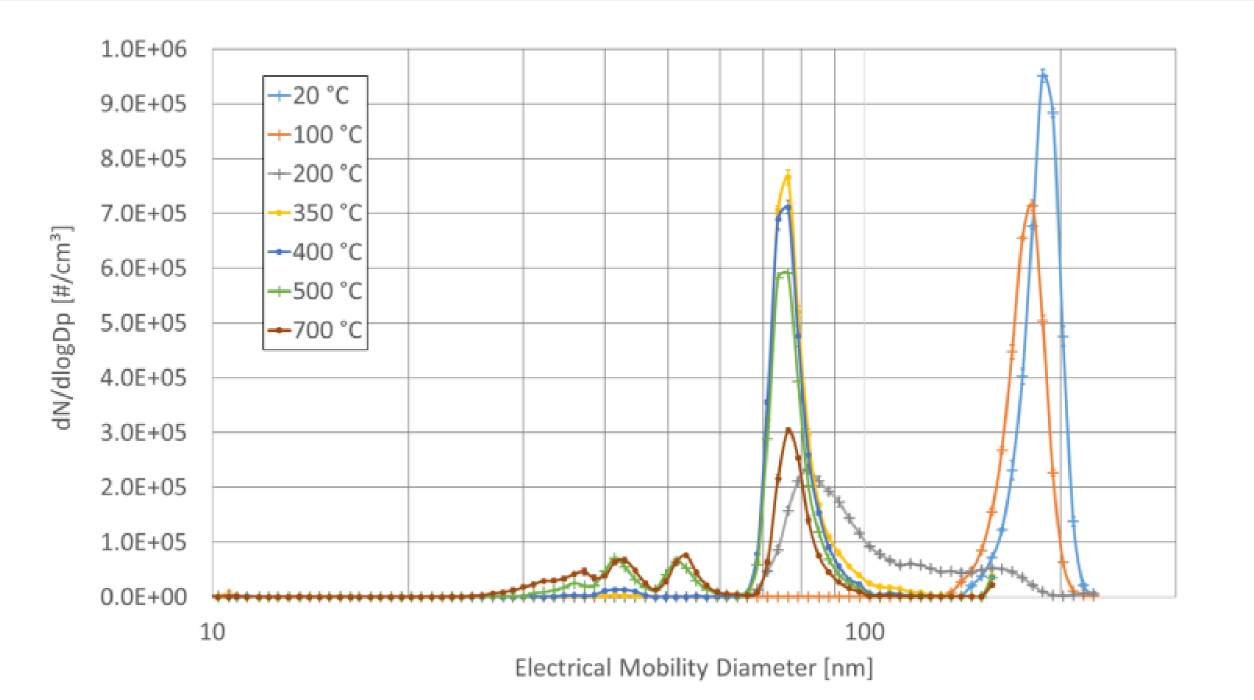


Figure 3 Size distribution graphs illustrating the reduction in particle size and improvement in uniformity post. Before sintering: The particles appear as irregularly shaped agglomerates with larger sizes (e.g., around 200 nm or 300 nm depending on the selected diameter in the DMA setup).After sintering: The agglomerates are reshaped into smaller, spherical particles (e.g., 200 nm agglomerates become roughly 75-80 nm spheres, and 300 nm agglomerates are transformed into 100 nm spheres) (Berger et al., 2024).

3.4 Sintering Temperature – Effect on CPC counting efficiency

In order to evaluate whether higher sintering temperatures create further compaction, we calibrated the Grimm 5412 CPC **(d_{50} = 10 nm),** using silver particles sintered between 400 °C and 700 °C. The results indicate that **sintering temperatures between 400 °C and 700 °C** have **no measurable influence** on CPC calibration performance, confirming the **high reproducibility** of the calibration setup (see Fig. 4). This aligns with previous findings, demonstrating that sintered silver nanoparticles provide **consistent and stable calibration performance** across different thermal conditions.

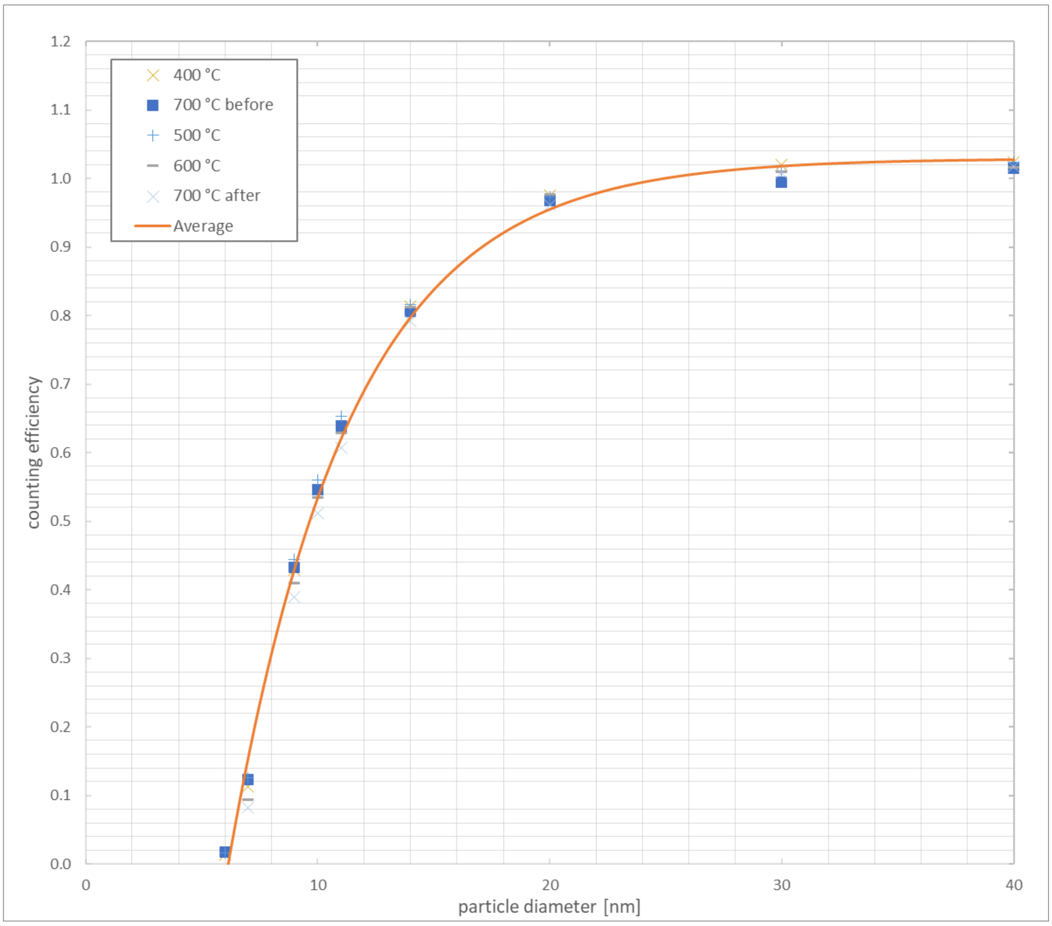


Figure 4 Counting efficiency as a function of particle diameter for different sintering temperatures (400 °C – 700 °C). The data was obtained using the Grimm 5412 CPC (d_{50} = 10 nm), showing no significant impact of sintering temperature on the counting efficiency before and after the first measurement at 700 °C and the final measurement again at 700 °C (Berger et. al. (2024).

These results further validate the suitability of **sintered silver particles for CEN-compliant CPC calibration**[1], ensuring no relevant variations in **measurement accuracy** regardless of the sintering temperature.

[1] EN 16976:2024 – Ambient Air – Determination of the Number Concentration of Atmospheric Aerosols. European Committee for Standardization (CEN). Available at: https://standards.iteh.ai/catalog/standards/cen/ab8b1143-a1d3-481b-b268-38a3b1da18b7/en-16976-2024.

Based on these observations, **400 °C is recommended** as the **optimal sintering temperature** when using the **S8000 sintering device**, as it provides **reliable particle transformation** while maintaining **measurement stability**. Future studies will investigate the **long-term structural effects** of higher sintering temperatures and the **behavior of sub-23 nm particles**, which are critical for **ultrafine particle calibrations**.

Conclusion

The combined use of the **°Silver Particle Generator** and the **°Particle Sintering Device** has proven highly effective in generating thermally stable, compact, and spherical silver particles, making them ideal for calibration purposes. This study has demonstrated that 400 °C is the optimal sintering temperature, enabling efficient reshaping of agglomerates while preserving particle integrity. The findings highlight the robustness and precision of the SPG and S8000 setup in applications requiring high accuracy, such as emissions testing and environmental monitoring.

The study also underscores the versatility of the **SPG** and **S8000** in creating monodisperse particles with minimal size deviations, paving the way for enhanced particle calibration methods. By transforming irregular agglomerates into uniform spheres, the system ensures consistent performance in particle measurement instruments such as Condensation Particle Counters and Particulate Number Counting Systems. These advancements contribute significantly to compliance with international standards such as ISO 27891:2015, supporting the growing need for reliable and reproducible nanoparticle calibration.

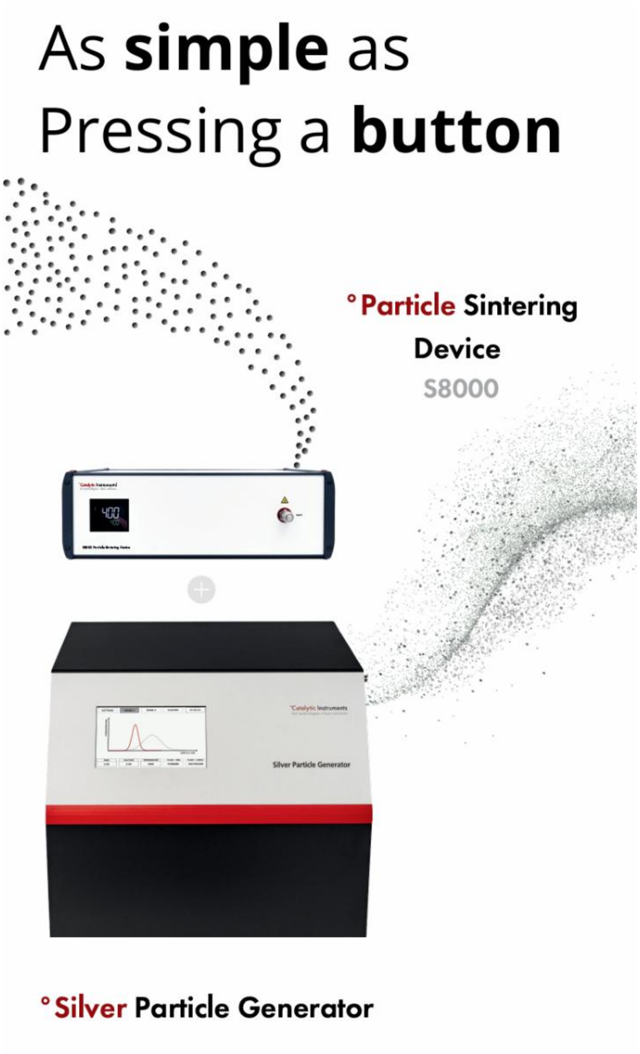
We like to emphasize the ease of use of this particular setup, including the compact design. The space requirement is approximately 550 × 400 × 480 mm compared to a classical double furnace setup with 2 × 800 × 500 × 400 mm (L × W × H). The setup time for the **SPG** and **S8000** is approximately 15 minutes and can be done by any technician without aerosol knowledge. In comparison, the dual furnace setup is estimated to take approximately ½ day of a skilled aerosol scientist.

Outlook

Future research will explore several aspects to further enhance the **SPG + S8000** setup. A key focus will be on the sintering behavior of smaller nanoparticles, particularly those below 23 nm, which are critical for CEN-compliant CPC calibrations. As shown in previous studies, precise control over the sintering temperature and residence time is essential for ensuring stable, monodisperse particles in this size range. Understanding how these ultrafine particles behave under varying thermal conditions will improve the accuracy of nanoparticle characterization and support the development of more reliable calibration standards

Additionally, efforts will explore the generation of larger sintered particles to broaden the range of particle sizes available for calibration in particular for DMA calibration but also PTI compliant calibration. This could provide valuable insights for applications in industries such as pharmaceuticals, materials science, and toxicological research. A key aspect of this work will involve optimizing the setup to reduce thermophoretic losses and minimize agglomeration post-sintering, ensuring that the particles maintain their uniformity and stability throughout the calibration process.

Further refinement of calibration techniques, including the integration of advanced measurement technologies, will aim to improve the accuracy and efficiency of particle measurement systems. Expanding the utility of the **SPG + S8000** setup into new domains, such as industrial nanoparticle applications and advanced materials testing, remains a promising avenue for future exploration.



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