

SUPPLEMENT: RECOMMENDATION

Additional research was performed to address the question of deviations between various sizing methods for Au-particles below 100 nm and the consequences on the DMA calibration.

Similar deviations in sizing nanoparticles were also observed in various institutes and are also the subject of investigations in other on-going projects (iMERA-Plus T3.J1.1, APMP). Systematic differences were observed in the sizes obtained by AFM, TSEM, SAXS, DLS and DMA (among others) techniques for various particle types. The consistent differences observed in various studies suggest that the different techniques measured different equivalent-diameters (i.e. different measurands). Full agreement within the stated uncertainty can only be obtained for ideal cases.

Potential contributors to the observed deviations include:

- Shape effects: For irregular-shaped particles the surface-equivalent diameter (determined by imaging techniques) may not be representative of particle mobility or other equivalent diameter determined by on-line techniques.
- Size effects: As the size of the particles decreases, their movement deviates from that expected in the continuum regime and some "slip" corrections for DMA measurements are necessary. The traceability of this correction factors is not established yet.
- Polydispersity of the samples: All samples are associated with a finite distribution width in which case the pooling of the mean diameter may depend on the property employed to weigh the results.

Particle preparation procedures: The differences in the preparation methods may have an effect on the particle size (e.g. contamination with impurities, loss of volatiles). A common observation in most studies was that the relative deviations become less important for particle sizes above 100 nm. While geometric diameters can be traceable this does not apply for the mobility particle size. However, for automotive applications it is the mobility diameter that is standardized (ISO 15900) and not the geometrical diameter. The scientific base to directly correlate the mobility diameter to alternative geometric-based measurands could not be established in the framework of this project. Therefore the consortium came up with the following recommendation to calibrate mobility particle size:

- The certified size standards shall consist of monodisperse spherical particles normally in a suspension with a certification as a reference material according to ISO Guide 30:1992. The material for the particles shall allow perfect spheres.
- The certified size standards shall have particle diameters above 80 nm and the standard deviation of size distribution shall be below 5 % of the diameter.
- The DMA shall be adjusted with at least one certified size standard. The initial calibration of particle sizing is performed by applying the relevant equations of ISO 15900. The adjustment of the particle selection by the DMA is performed by changing the high voltage or the sheath air flow. The aerosol flow shall be kept constant during adjustment and subsequent measurement.
- The particle mobility diameter for particles below 80 nm is calculated using the equations from ISO 15900 and is assumed to be correct by convention.