Particle Size Distribution Measurements with the Novel 1 nm-SMPS

Fast Scanning at few Nanometers

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UNDERSTANDING, ACCELERATED

Motivation

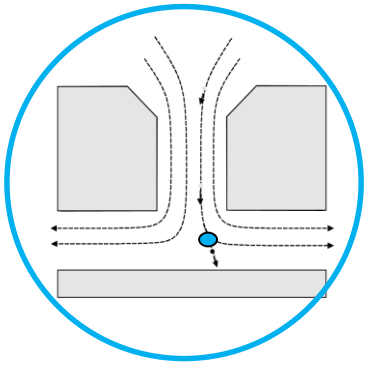
Enable research towards understanding where particles come from – formation and growth

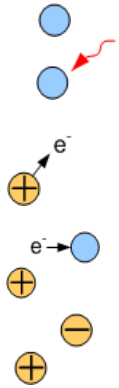
+ Basic research applications
  + Atmospheric research, health effect studies, combustion emissions research and filtration research
+ Industrial applications
  + Material Science: Particle source characterization (e.g. flame synthesis, spark generation), NP functionalization, coating of nanoparticles, reaction kinetics
  + Products: catalysts, cancer drugs, OLEDs, storage, pigments, …

SMPS – Theory of Operation

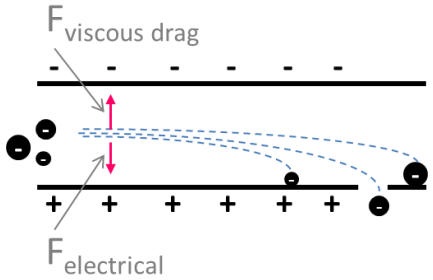
Uses 1st principle „Electrical Mobility“ method for particles sizing and counting → no calibration needed

Impactor | Neutralizer | DMA | CPC





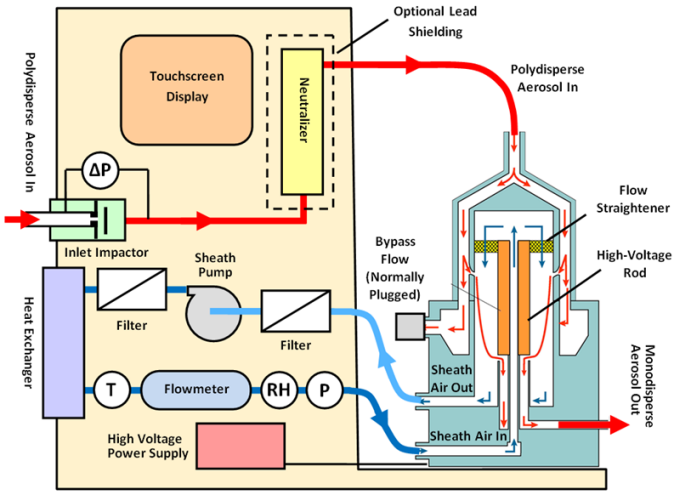
Differential
Mobility **A**nalyzer



Condensation
Particle **C**ounter

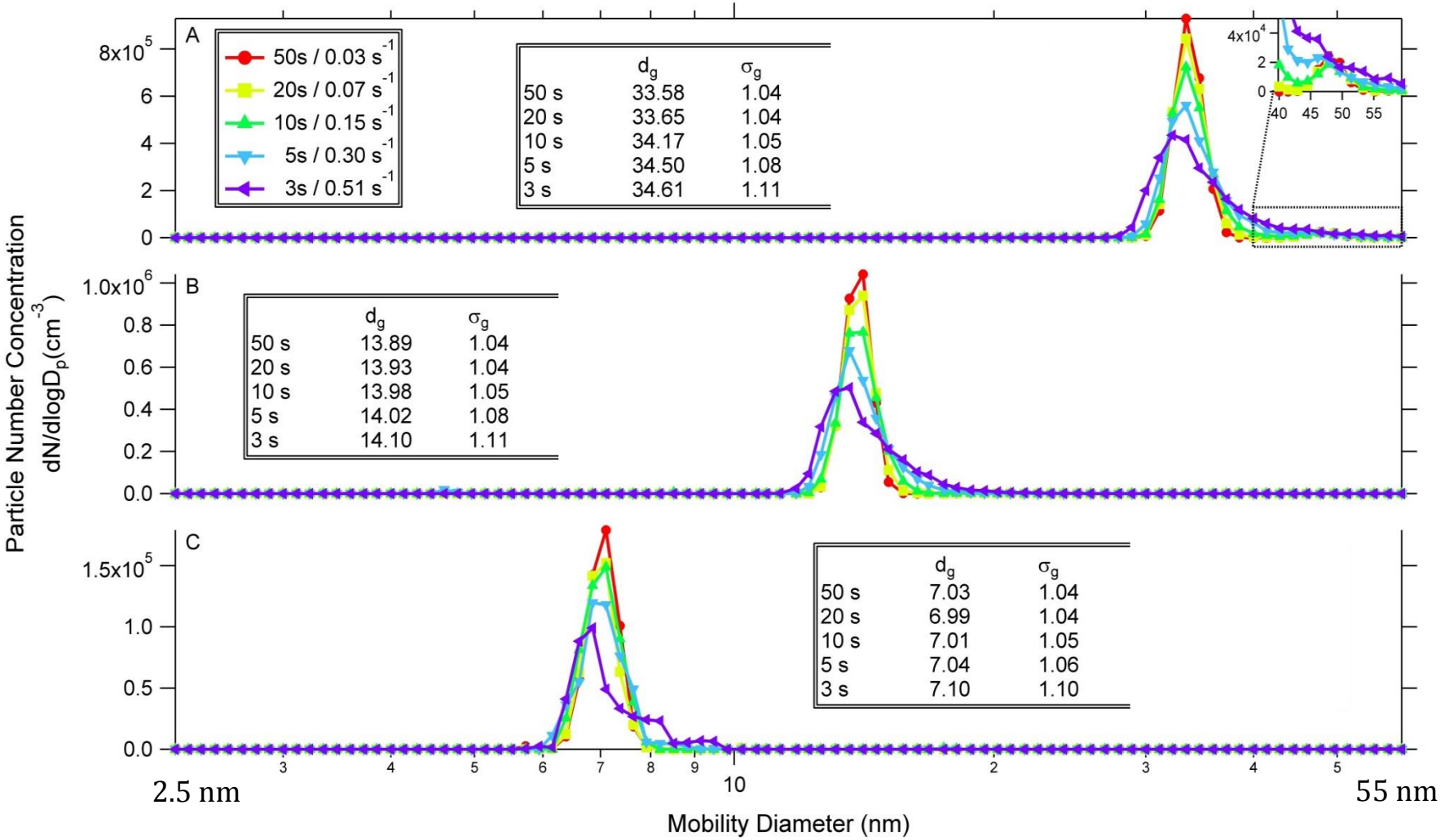
Instrument Characterization

Nano-SMPS



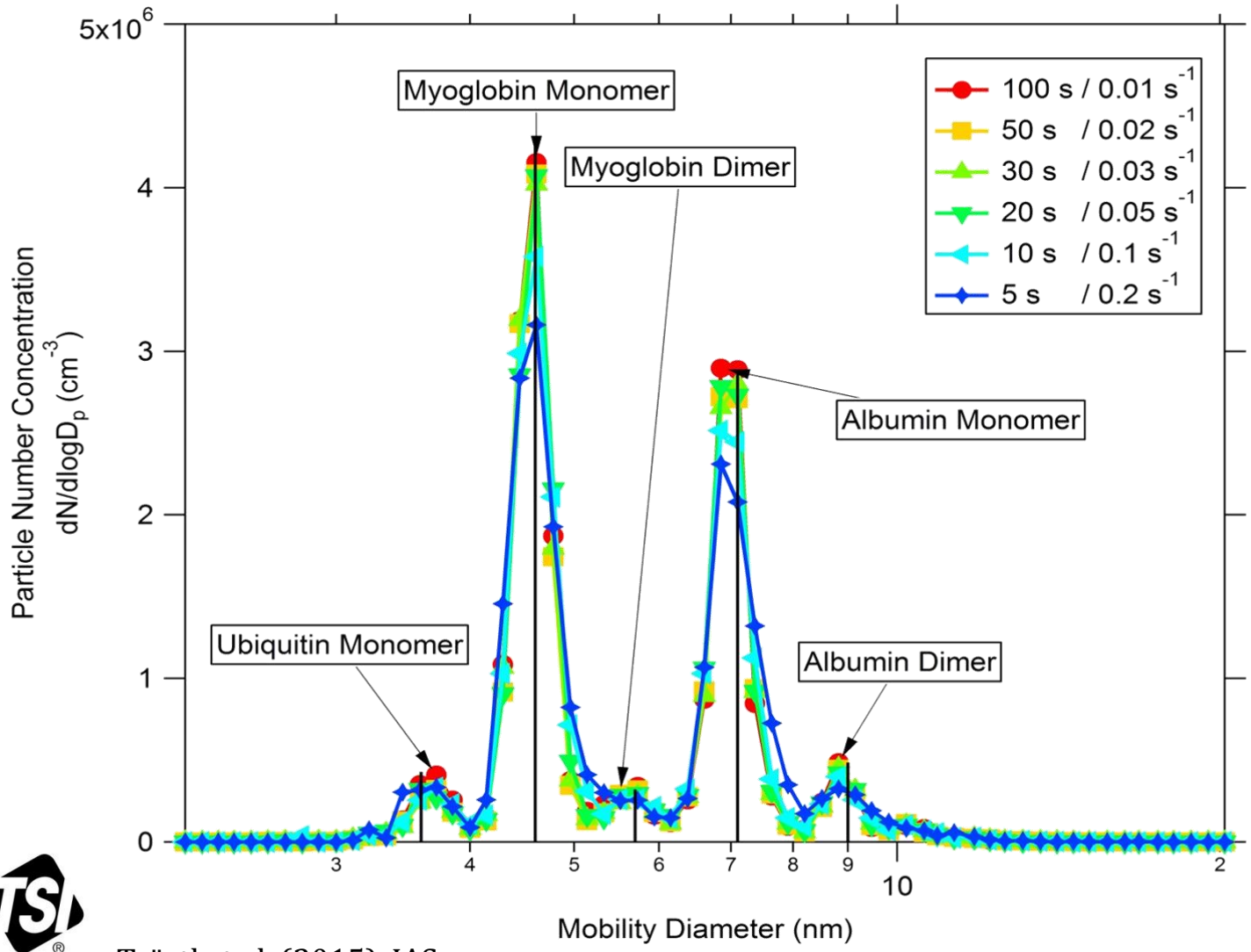
Fast and Precise Measurement in the sub-20 nm Size Range using a Scanning Mobility Particle Sizer (Tröstl et al. 2015, JAS)

Monodisperse Sucrose solutions with different scan times



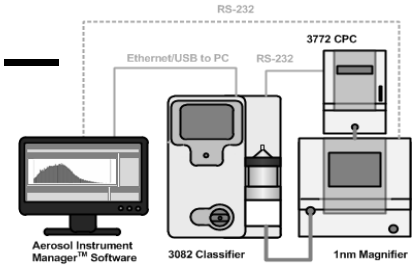
Tröstl et al. (2015), JAS

Fast Sizing & High Accuracy
Protein Mix



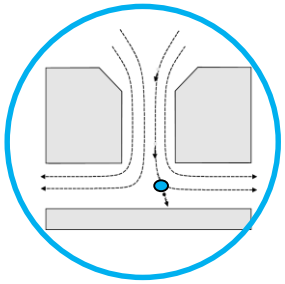
Tröstl et al. (2015), JAS

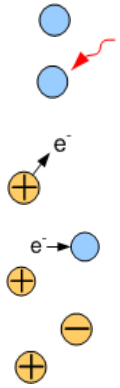
1 nm SMPS with DEG CPC – Theory of Operation



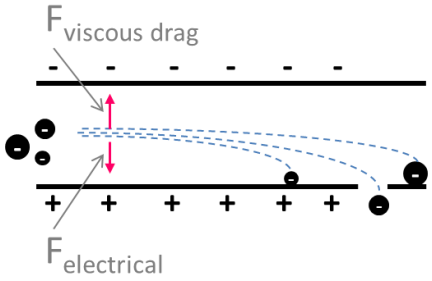
1 nm DMA **1 nm CPC**

Impactor → Neutralizer → DMA → Nano Enhancer → CPC



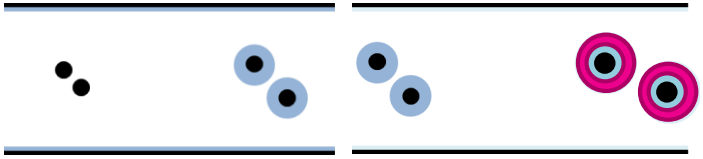


Differential
Mobility **A**nalyzer



Working fluid DEG (Diethylene glycol) Grows ~100 nm droplets

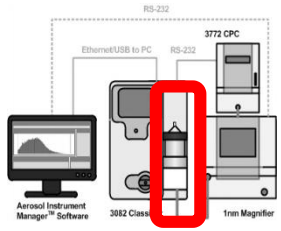
Working fluid Butanol. Grows µm particles and counts them

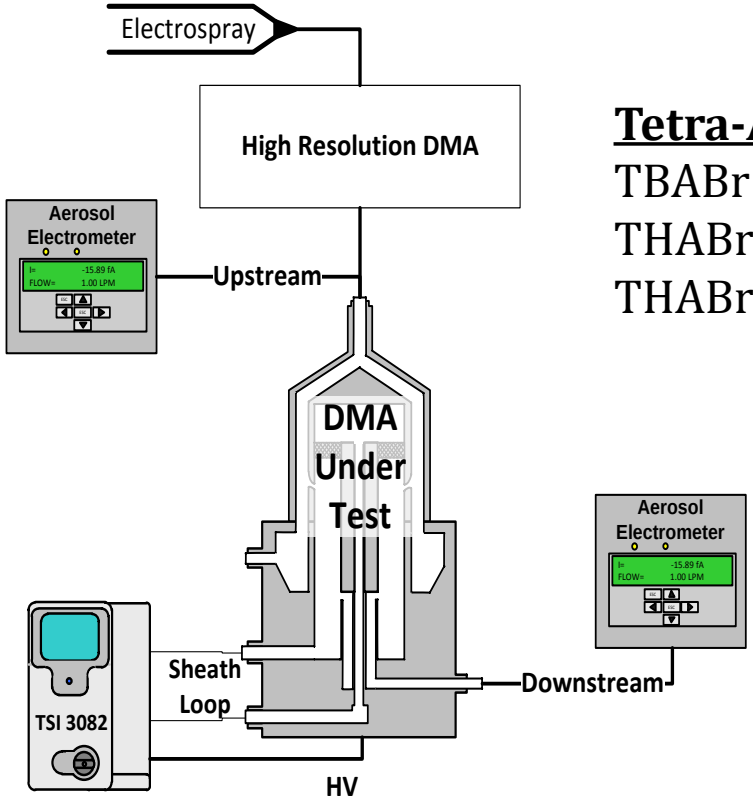


Jiang, Kuang, Attoui & McMurry (2011) AS&T 45 (4)
Iida, Stolzenburg & McMurry (2009) AS&T 43 (1)
Dahlkötter et al., Extending Particle Size Distribution Measurements down to 1nm, NOSA 2016

 11/22/2016

Characterization 1 nm DMA Resolution Setup





Tetra-Alkyl Ammonium Ions

TBABr monomer: 1.24 nm
THABr monomer: 1.47 nm
THABr dimer: 1.74 nm

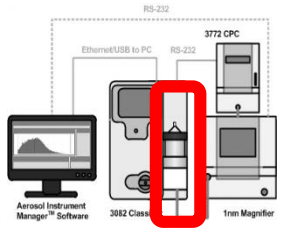
Setup and methologies based on Jiang et al (2011), AS&T 45:4, 480-492

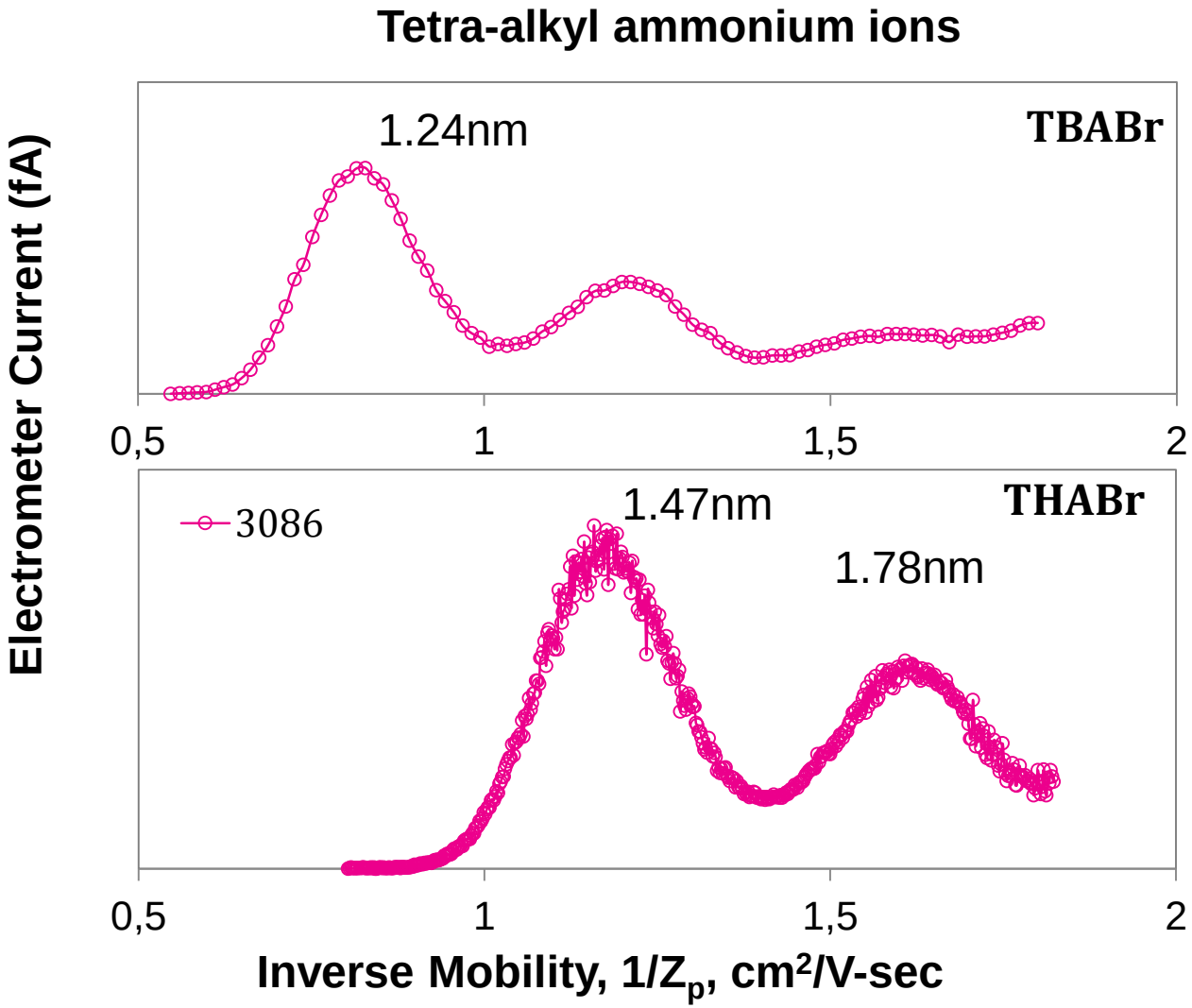
Stolzenburg, Attoui, Han, Spielvogel and Scheckman (2016), J. Aero. Sci, In Preparation

 11/22/2016

1 nm DMA Characterization

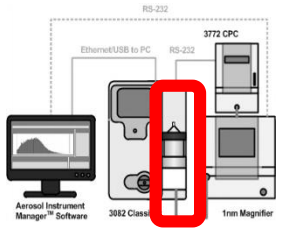
Sizing Scans of Ions



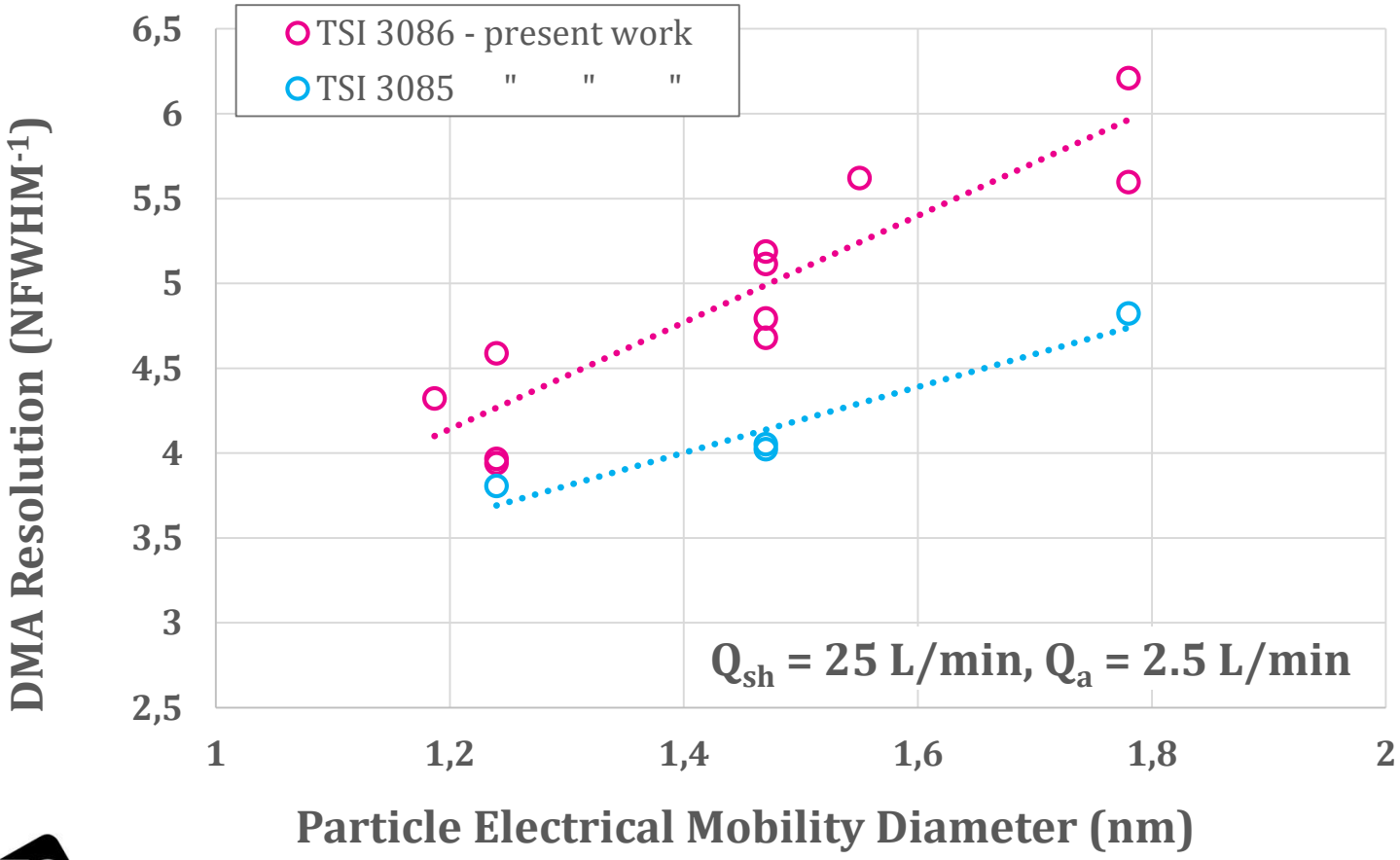


Stolzenburg, Attoui, Han, Spielvogel and Scheckman (2016), J. Aero. Sci, In Preparation

1 nm DMA Resolution



Average 20% improvement in DMA resolution



Stolzenburg, Attoui, Han, Spielvogel and Scheckman (2016), J. Aero. Sci, In Preparation

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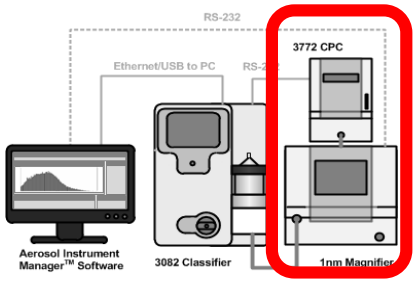
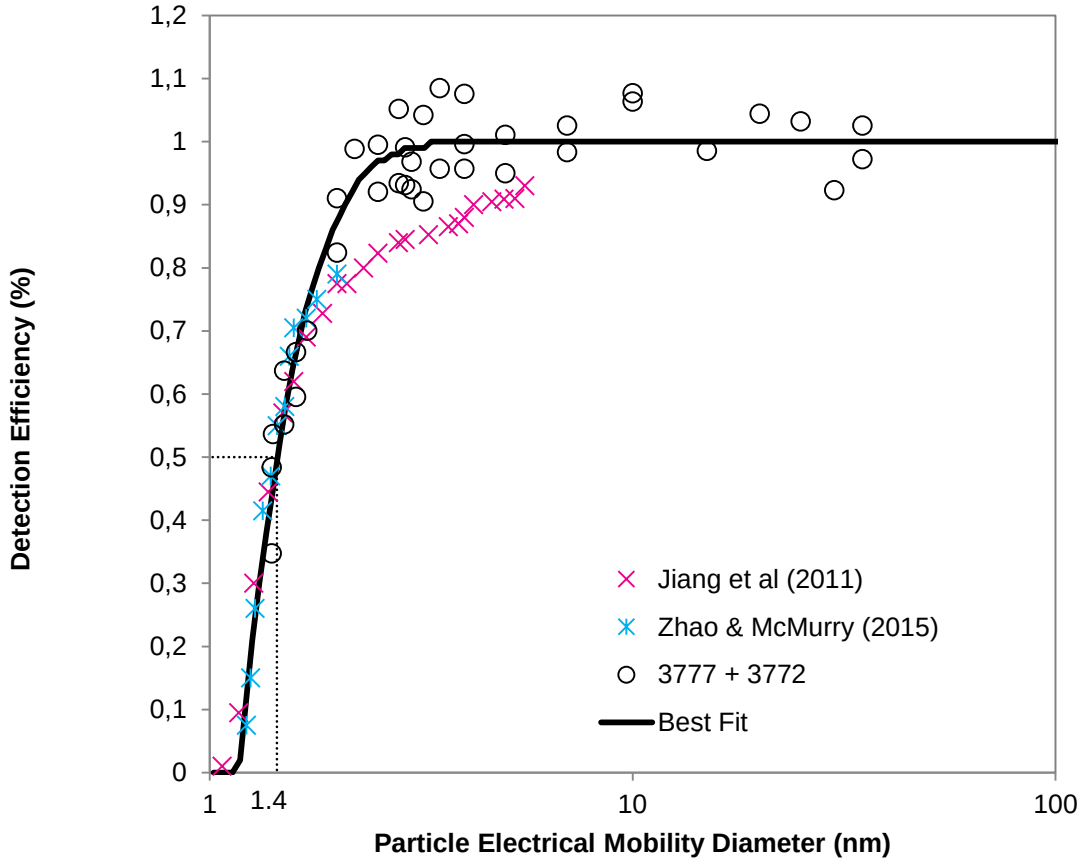
CPC Characterization - Efficiency



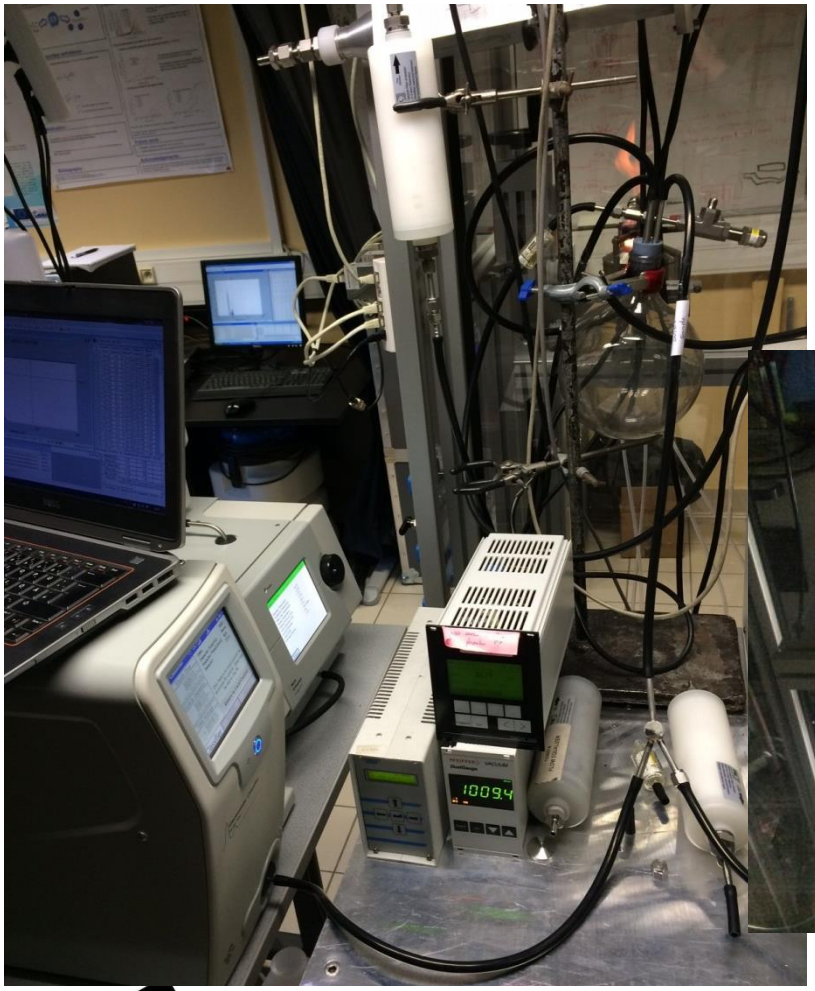


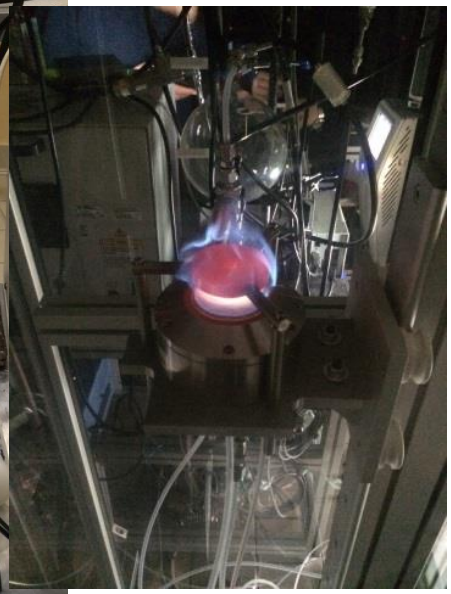
+ D50 of 1.4 nm mobility diameter (geom. 1.1 nm)
+ False count rate <0.01 #/cm^3 (12 hour average)
+ Unattended operation for 1 week minimum
+ 300 000 particles/cm^3 (with CPC 3772)

Aerosol: negatively charged NaCl particles

Zhao et al (2015) AAAR Annual Conference, Minneapolis
Jiang et al (2011) AS&T, 45(4)

Applications: Flame Aerosol
First 1 nm SMPS Test Uni. Lille, France

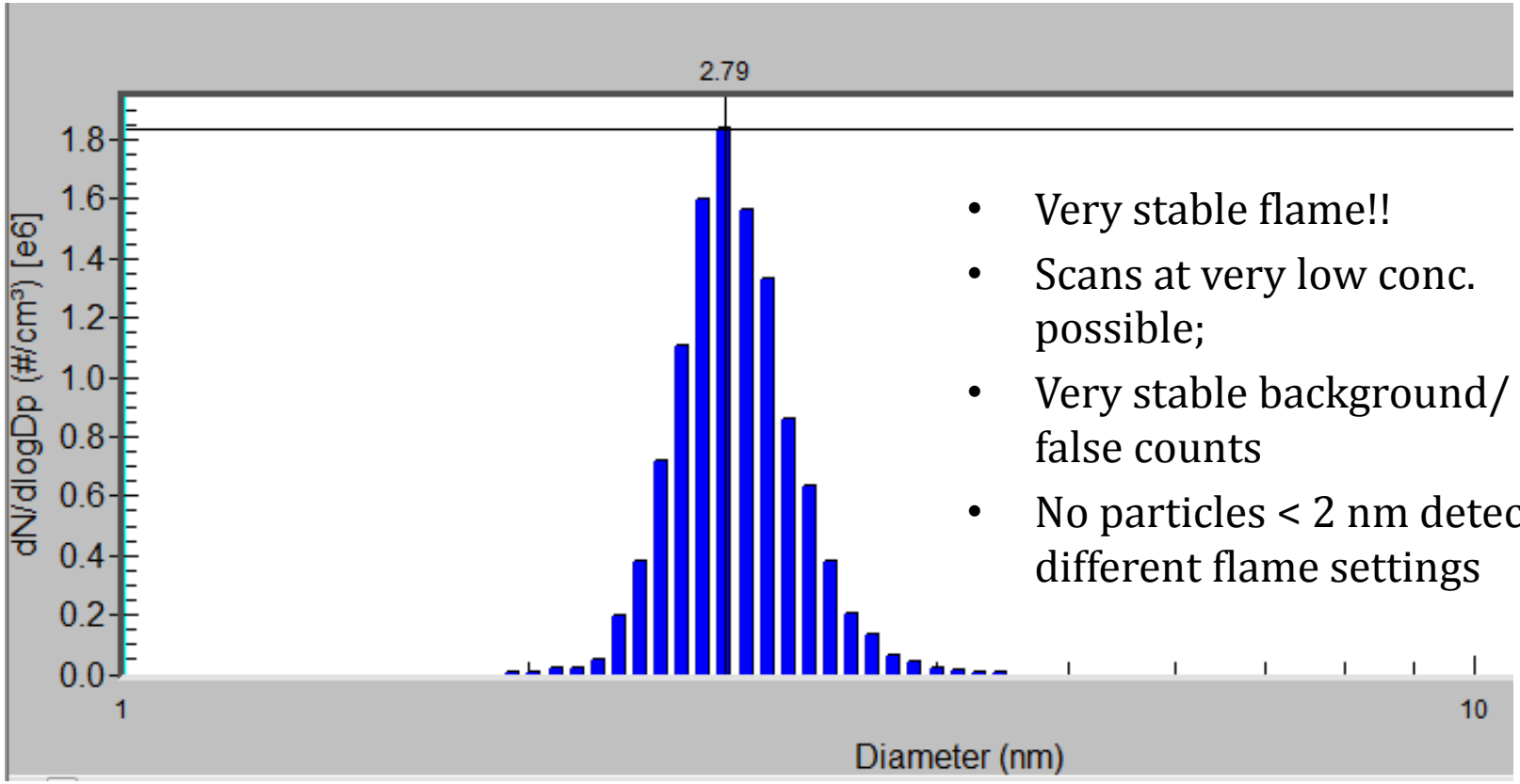




- TSI demo setup: 308202, 3088, 3085, 3777 with 3772
- Sample Aerosol from Ethylene-Air or Butane-Air premixed flame at Cerla

In cooperation with University Lille, Cerla and PC2A France

1 nm SMPS – Butane-Air flame

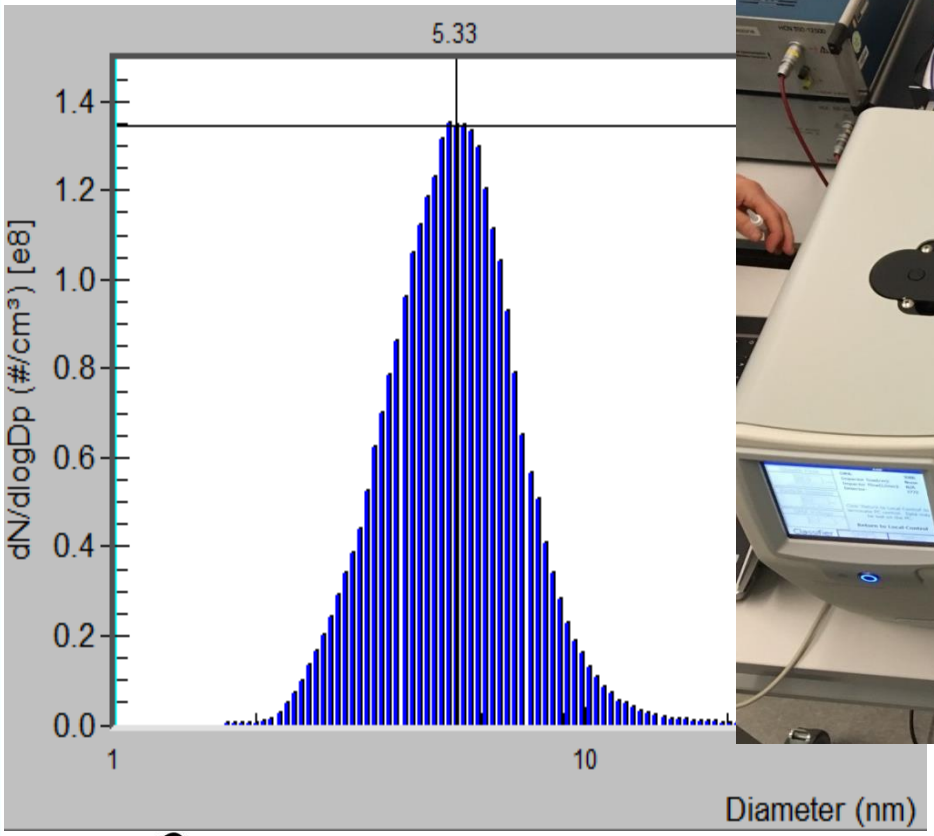


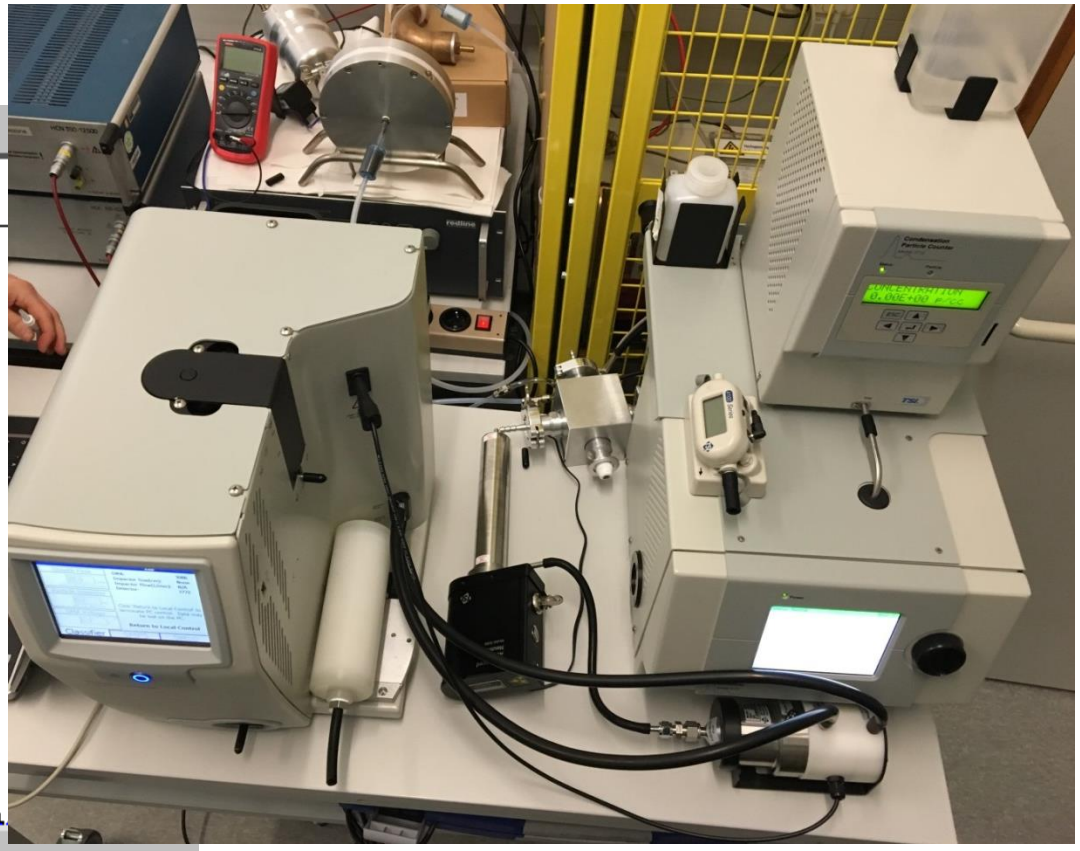
- Very stable flame!!
- Scans at very low conc. possible;
- Very stable background/ low false counts
- No particles < 2 nm detected in different flame settings

In cooperation with University Lille, Cerla and PC2A France

Applications: Metal-Nanoparticle
TU-Clausthal, Germany

Silver spark generator Size Distr.





In cooperation with TU Clausthal, Germany

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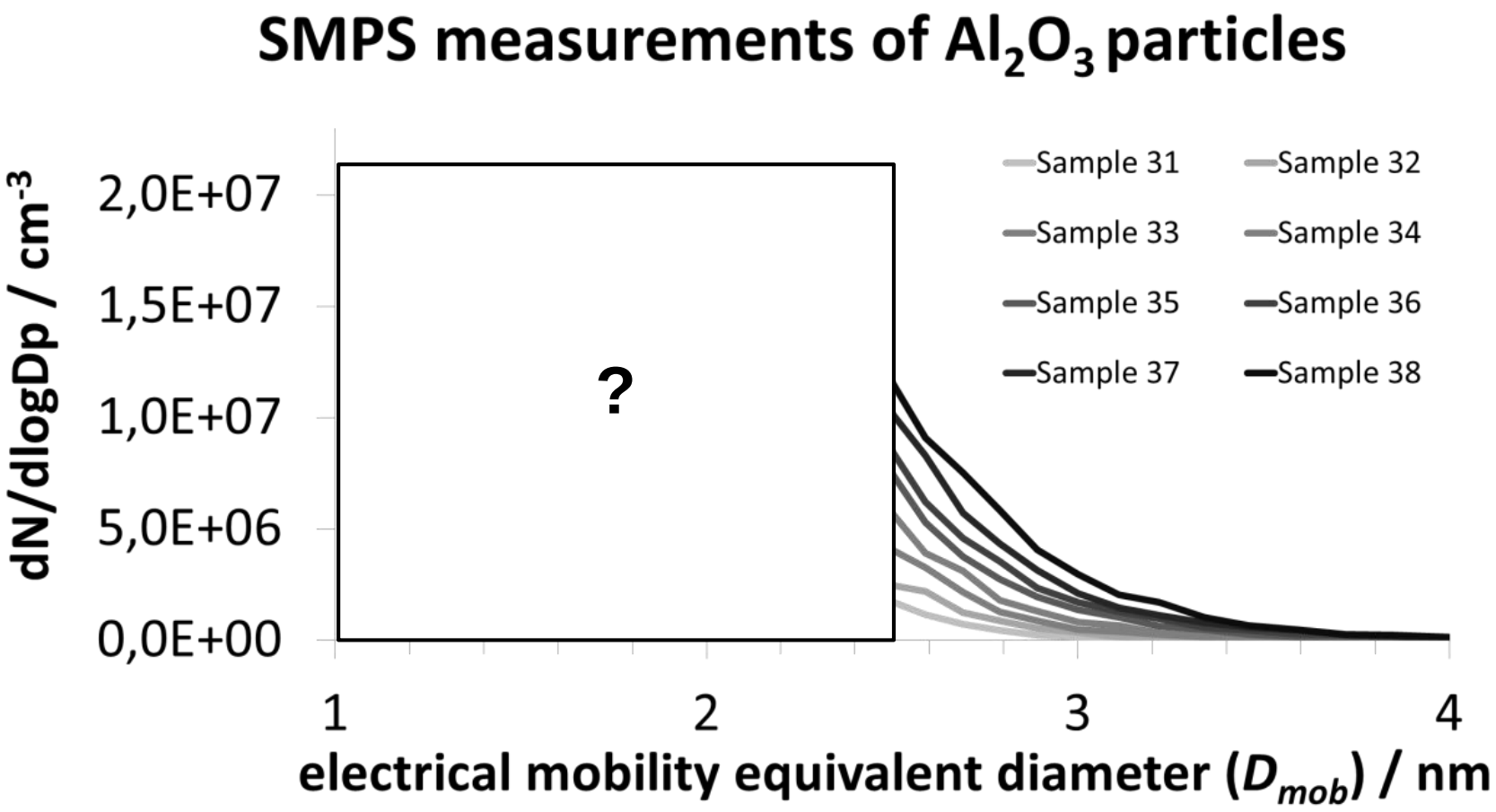
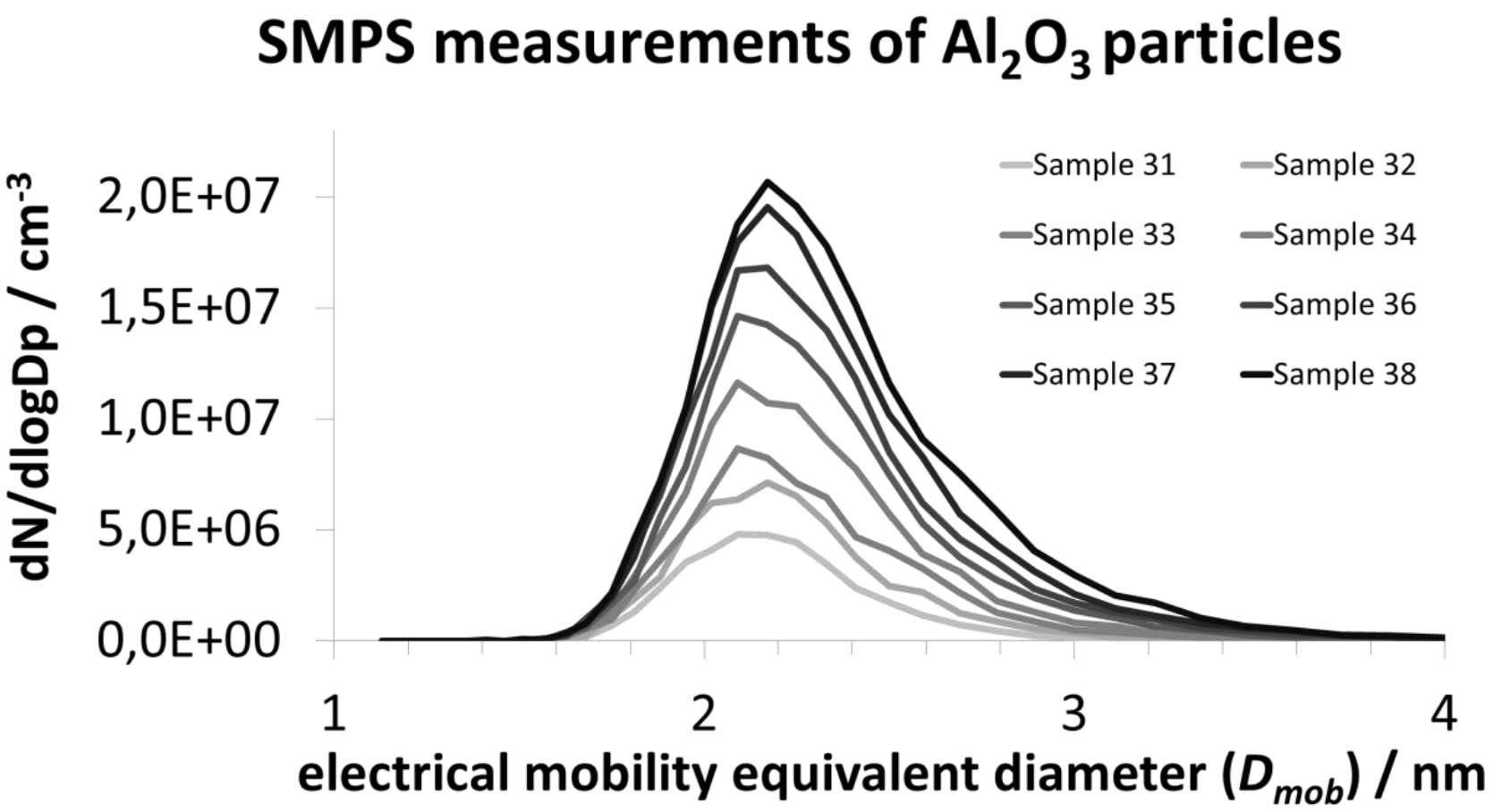
Applications: Metal-Oxide Nanoparticles



Particles generated by Dielectric Barrier Discharge (DBD)

In cooperation with TU Clausthal, Germany

Applications: Metal-Oxide Nanoparticles



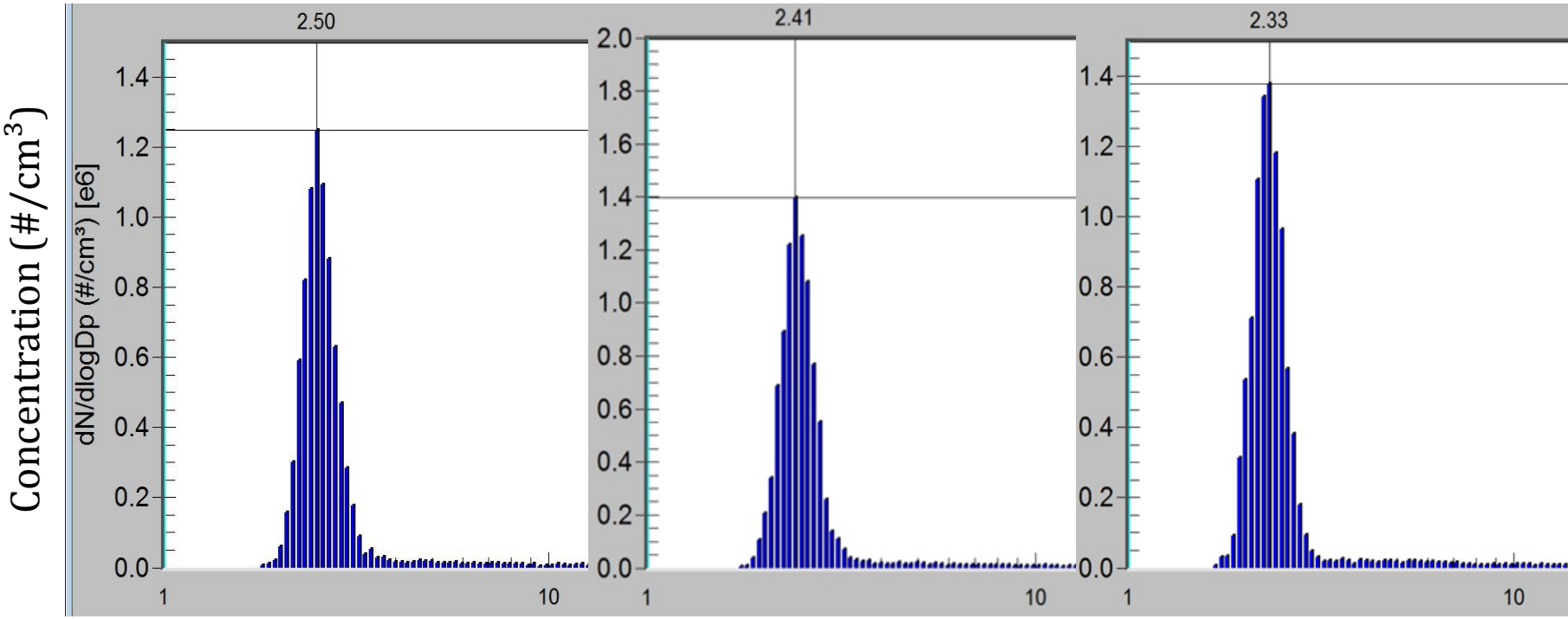
Particles generated by Dielectric Barrier Discharge (DBD)

In cooperation with TU Clausthal, Germany

Influence of Aerosol Flow

Silver Nanoparticles at different Bypass Flows

Bypass Flow: 0 L/min → 2.5 L/min → 7.5 L/min

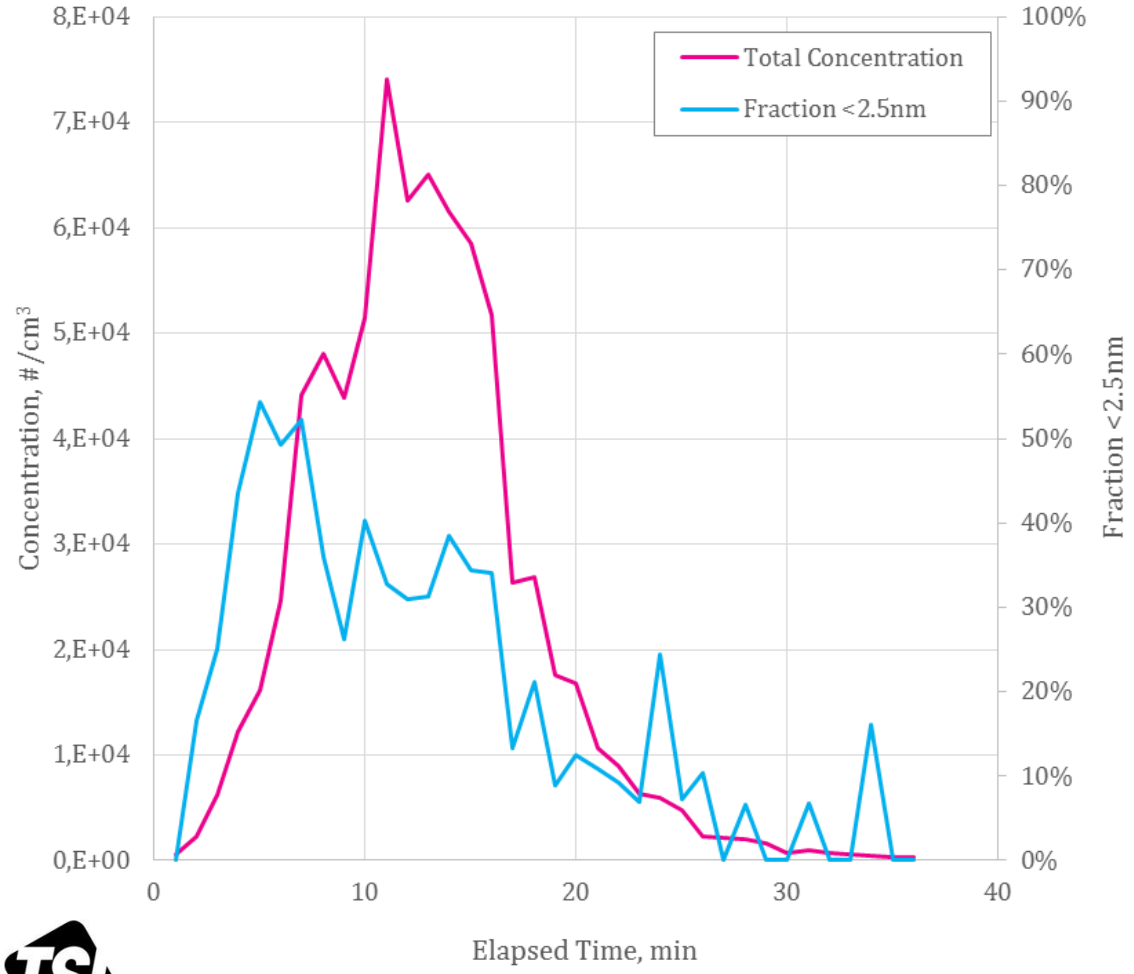


Smallest size bin found in these experiments: 1.68 nm
Particles generated by Dielectric Barrier Discharge (DBD)

In cooperation with TU Clausthal, Germany

Applications: Indoor Air Quality

Emissions from household cooking appliance



Rise to concentration peak from heating DeLonghi toaster oven (oven door open) set on Bake at 400 °F.

Note the number of particles below 2.5 nm.

Thanks to Lance Wallace for this data

Summary

+ Nano-SMPS characterization
  - Fast scanning advantages, 3-5 second scans in the 3-20 nm range possible
  - High accuracy of size scans

1nm-CPC, Model 3777-72

+ NEW 1 nm SMPS (3938E77) → SMPS Size distribution measurements starting at 1 nm
  - New Nano Enhancer (Model 3777)
  - New 1 nm DMA (Model 3086)

1nm-SMPS, Model 3938E77

+ First field applications confirm improved performance compared to Nano-SMPS
  - Enables research towards understanding of nanoparticle formation and growth

Thank you! Any Questions?

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Key References

Iida, K., Stolzenburg, M. R., and McMurry, P. H., 2009, "Effect of Working Fluid on Sub-2 nm Particle Detection with a Laminar Flow Ultrafine Condensation Particle Counter," Aerosol Science and Technology, 43(1): 81–96

Jiang, J., Attoui, M., Heim, M., Brunelli, N. a., McMurry, P. H., Kasper, G., Flagan, R. C., Giapis, K., and Mouret, G., 2011, "Transfer Functions and Penetrations of Five Differential Mobility Analyzers for Sub-2 nm Particle Classification," Aerosol Science and Technology, 45(4): 480–492

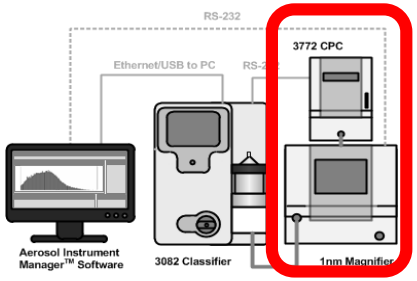
Jiang, J., Chen, M., Kuang, C., Attoui, M., and McMurry, P. H., 2011, "Electrical Mobility Spectrometer Using a Diethylene Glycol Condensation Particle Counter for Measurement of Aerosol Size Distributions Down to 1 nm," Aerosol Science and Technology, 45(4): 510–521

Jiang, J., Zhao, J., Chen, M., Eisele, F. L., Scheckman, J., Williams, B. J., Kuang, C., and McMurry, P. H., 2011, "First Measurements of Neutral Atmospheric Cluster and 1–2 nm Particle Number Size Distributions During Nucleation Events," Aerosol Science and Technology, 45(4): ii–v

Tröstl, J., Tritscher, T., Bischof, O.F., Horn, H.-G., Krinke, T., Baltensperger, U., and Gysel, M., 2015. Fast and precise measurement in the sub-20nm size range using a Scanning Mobility Particle Sizer. Journal of Aerosol Science 87, 75-87

Wimmer, D., Lehtipalo, K., Franchin, A., Kangasluoma, J., Kreissl, F., Kürten, A., Kupc, A., Metzger, A., Mikkilä, J., Petäjä, T., Riccobono, F., Vanhanen, J., Kulmala, M., and Curtius, J.(2013): Performance of diethylene glycol-based particle counters in the sub-3 nm size range, Atmospheric Measurement Techniques, 6, 1793-1804, doi:10.5194/amt-6-1793-2013

CPC Characterization - Linearity



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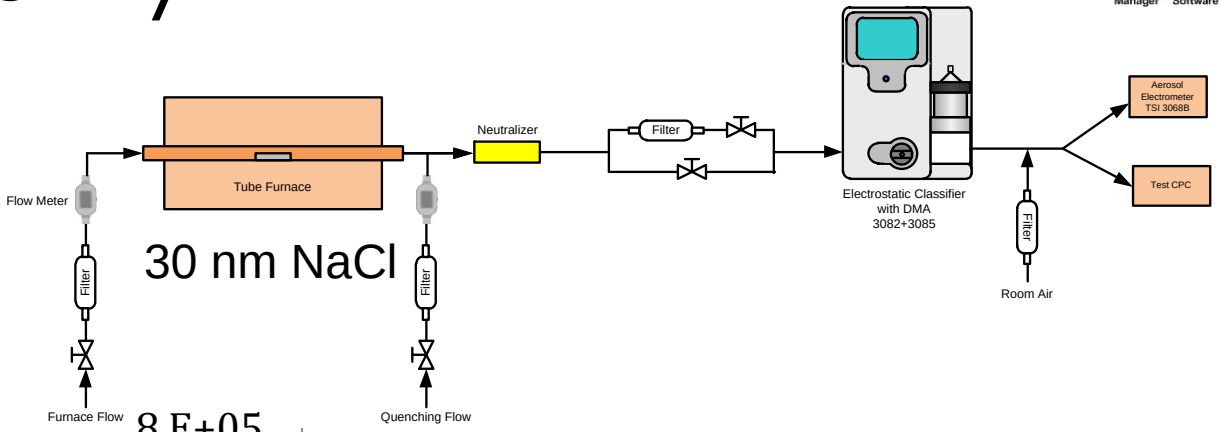

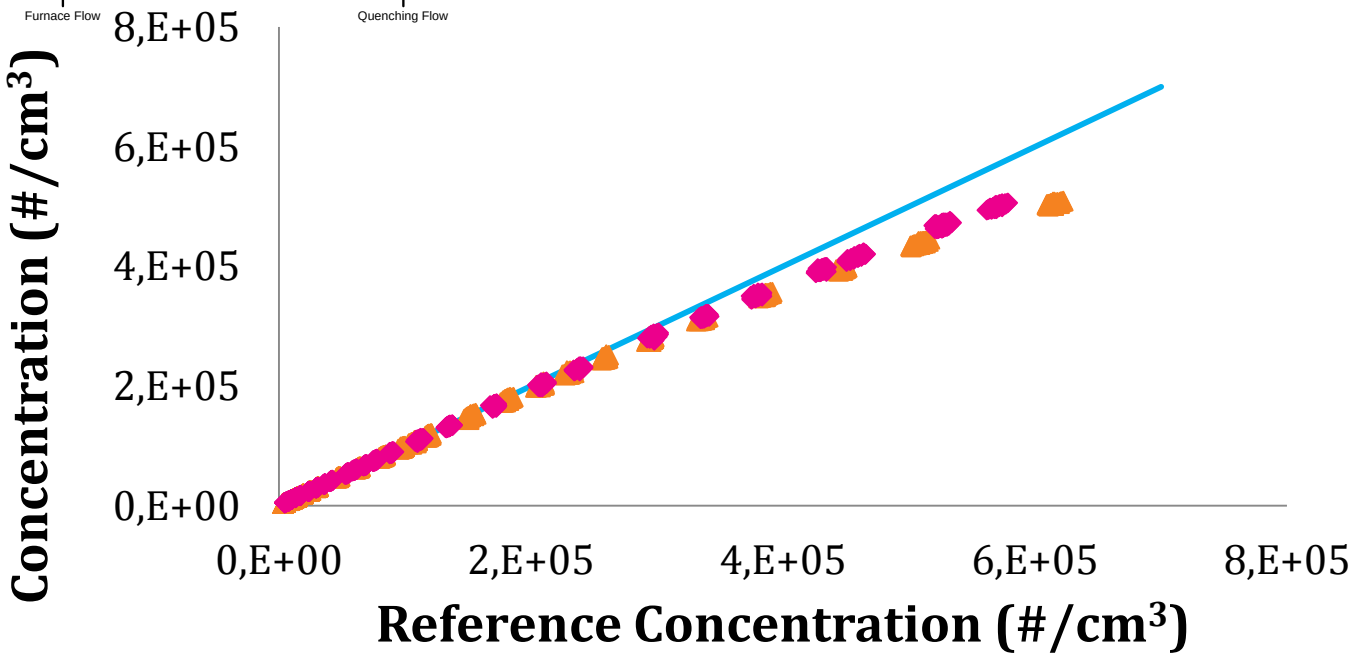

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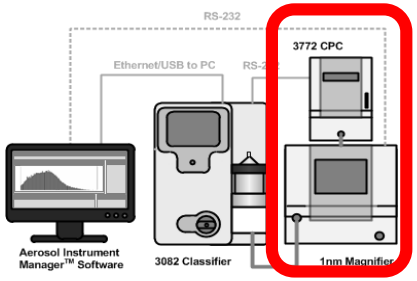
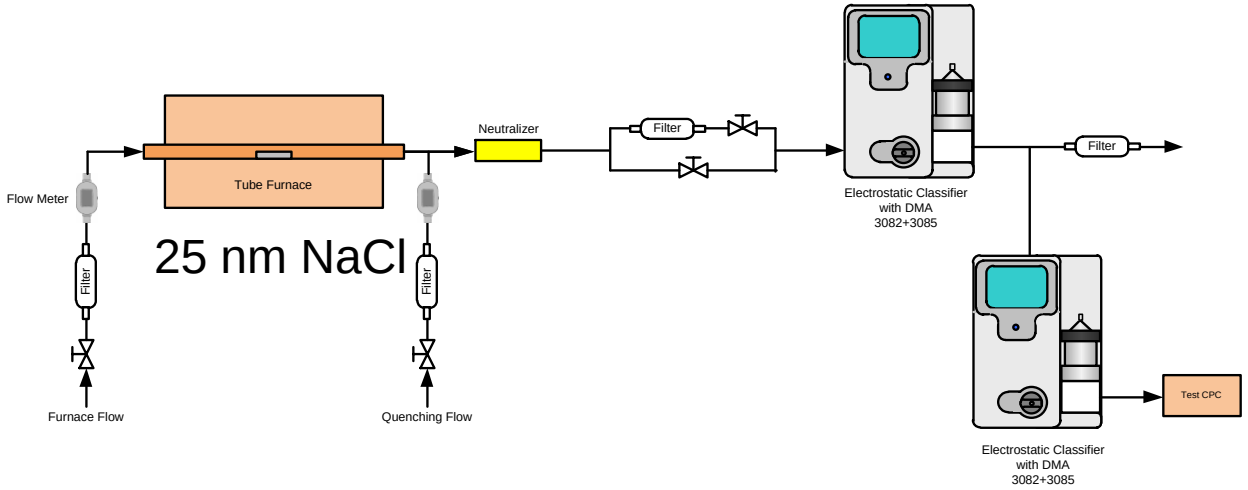
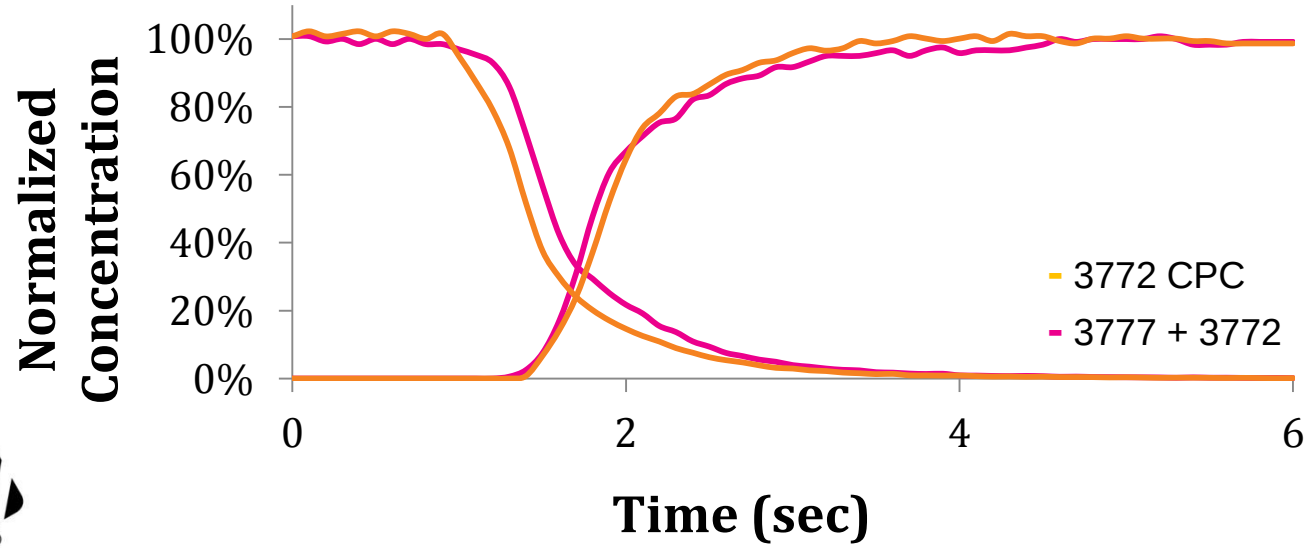
CPC Characterization – Response Time







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MODEL 3777 NANO ENHANCER

Particle Size Range
Min. Detectable Particle (D_{50}): 1.4 nm (electrical mobility diameter, 1.1 nm geometric diameter), verified with NaCl particles

Flow

Aerosol Flow Rate	2.5 L/min
Aerosol Outlet Flow Rate	1.0 L/min
Transport Flow Rate	1.5 L/min
Flow source	External vacuum
Flow Control	Volumetric flow control of transport flow internal critical orifices. Aerosol flow rate controlled by 3772 CPC.

Aerosol Medium
Recommended for use with air; safe for use with inert gases such as nitrogen, argon, and helium (performance specifications are for air)

Condensing Liquid

Working Fluid	Diethylene Glycol (DEG, ≥99%)
Filling System	Electronic liquid-level sensor initiates automatic filling as needed, requires connection to fill bottle
Water Removal	Sheath air is dried using a water separator and refillable desiccant dryer

Communications

Protocol	Command set based on ASCII characters
Interfaces	RS-232 9-pin, D-sub connector

Further details on www.tsi.com

1nm CPC SYSTEM
(MODEL 3777 NANO ENHANCE AND MODEL 3772 CPC)

Particle Size Range
Min. Detectable Particle (D_{50}): 1.4 nm (electrical mobility diameter, 1.1 nm geometric diameter), verified with NaCl particles

Particle Concentration Range
0 to $3x10^5$ particles/cm^3, single particle counting with continuous, live-time coincidence correction

Particle Concentration Accuracy
±10% at <$1.65x10^5$ particles/cm^3
±15% at $3x10^5$ particles/cm^3

Response time
<4s to 95% in response to concentration step change

False Background Counts
<0.01 particle/cm^3, based on 12-hr average

SPECIFICATIONS

Size range
1 to 50 nm

Resolution
R=4.7 at 1.47 nm

Flow rate range
(flows provided by Model 3082 Classifier and/or external source)

Aerosol flow rate	0.1 to 2.5 L/min
Sheath flow rate	2 to 25 L/min
Bypass flow rate	0 to 12 L/min