Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
National Metrology Institute

Development of metrological standards for traceable particle number measurements of automotive exhaust emissions

WG 3.23: Aerosol and Particle Diagnostics

Andreas Nowak, Arne Kuntze, Johannes Rosahl, Margit Hildebrandt, Volker Ebert, Egbert Buhr

Overview

- About PTB: Role of legal metrology
- Primary PN standard: Needs and procedures
- PN Results: Inter comparison and PTB setup
- Conclusion and outlook

PTB: To measure is to know

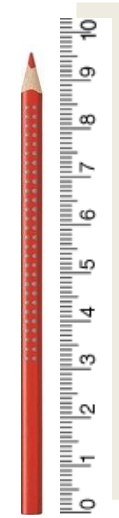
About PTB:

- Federal Ministry of Economics and Technology (BMWi)
- 170 Mio. € budget, plus third party funding
- Approx. 1300 permanent staff and 550 non-permanent staff including 110 PhD students

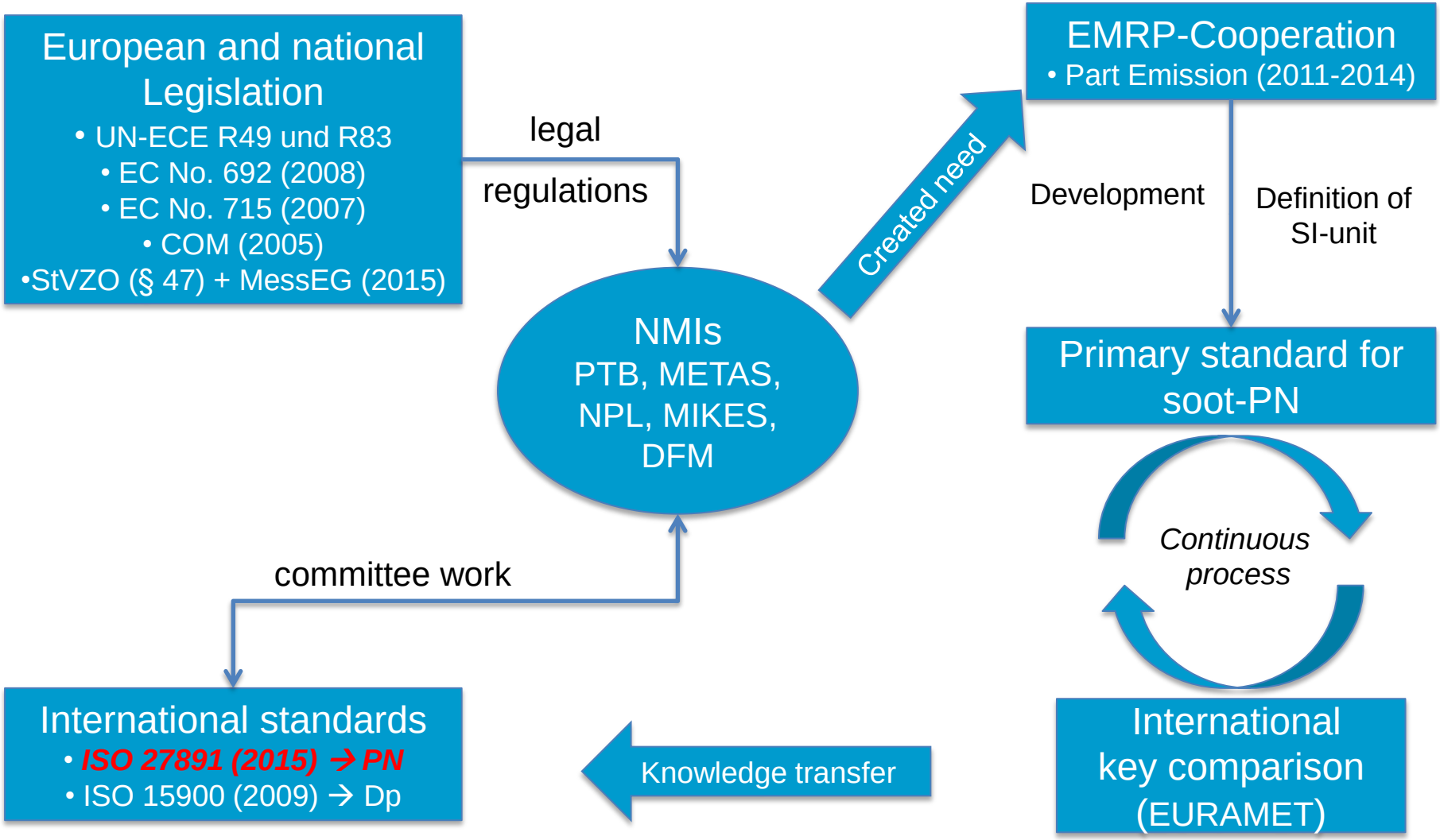
125 Jahre genau
1887 - 2012

Role of PTB in legal metrology:

- Provides traceability to national standards by calibrating reference standards of verification authorities and notified bodies
- Type approval and certification according to 23 German laws and regulations (e. g. verification act, law on civilian firearms)
- Guidance of federal ministries, the verification authorities and notified bodies
- Guidance of manufacturers in understanding the verification act and the MID
- Collaboration in respective to international organisations – OIML, WELMEC

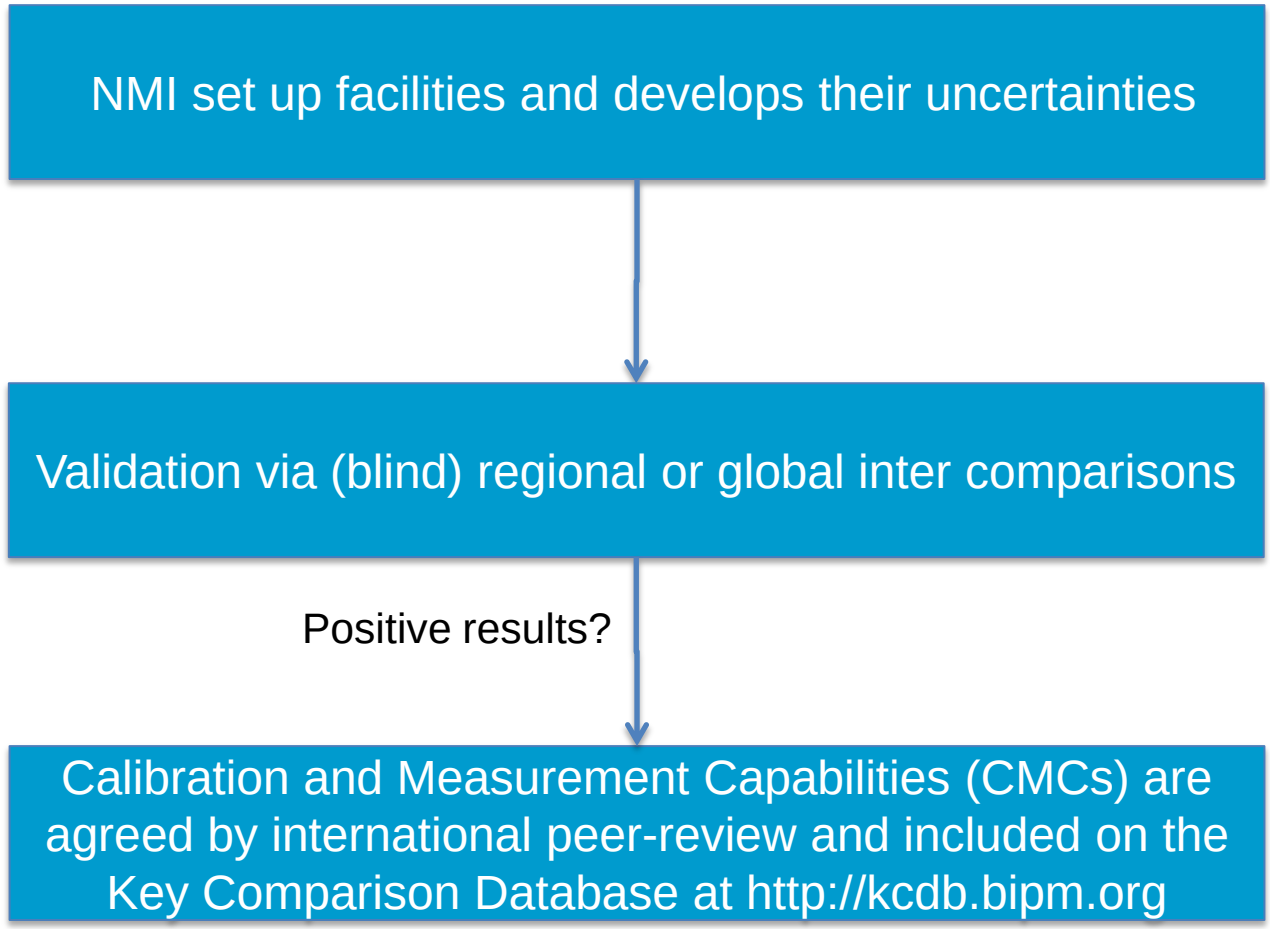


Need for PN-primary standard



Procedure of PN inter comparison

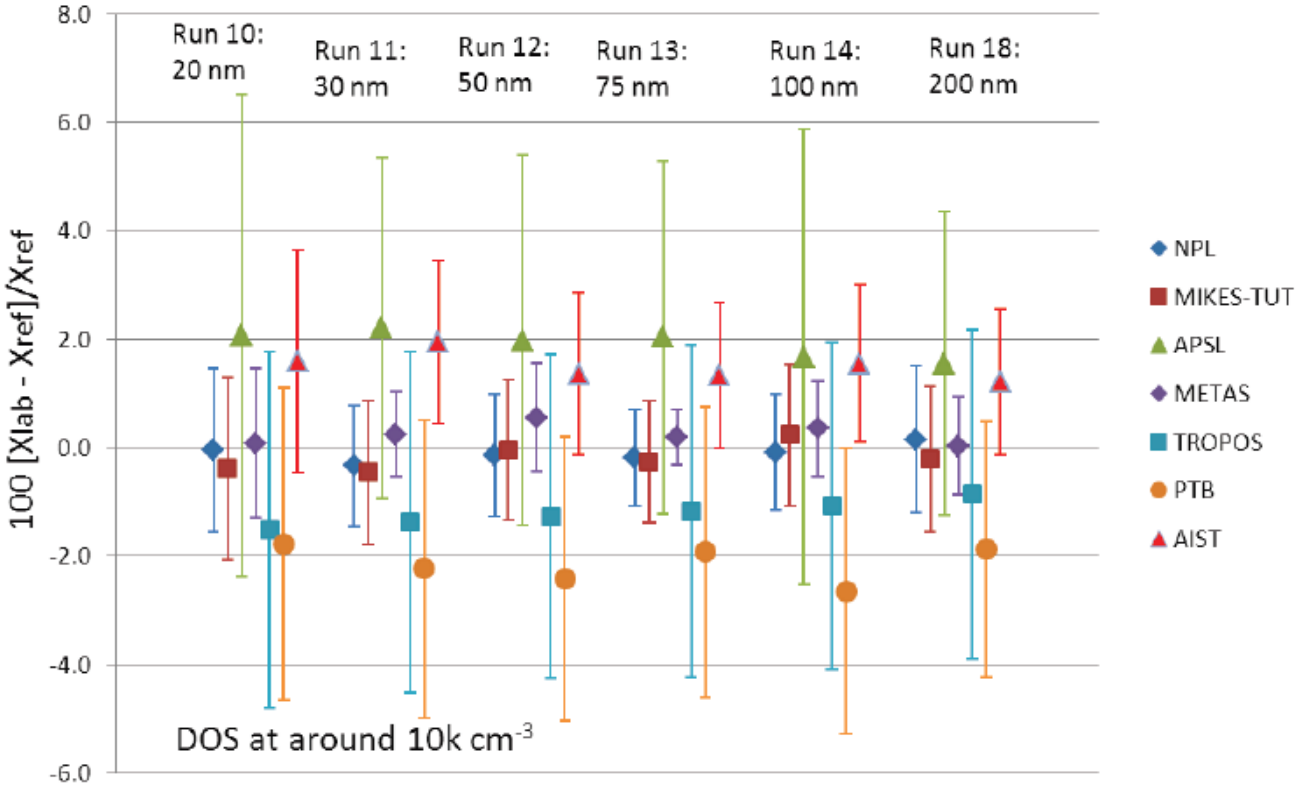
→Establishing NMI capabilities and measurement uncertainties



PN-comparison: Results of primary method

First comparison of its kind for Faraday Cup Aerosol Electrometer (FCAE) in EURAMET 1244 (Mar. 2014):

- Different types of aerosols: soot; DOS; NaCl
- DOS-particle with sizes from 20 to 200 nm and conc. at 10.000 cm^{-3}



more information see:

- NPL Report AS 85
- R. Högström et al Metrologia 51 (2014) 293–303

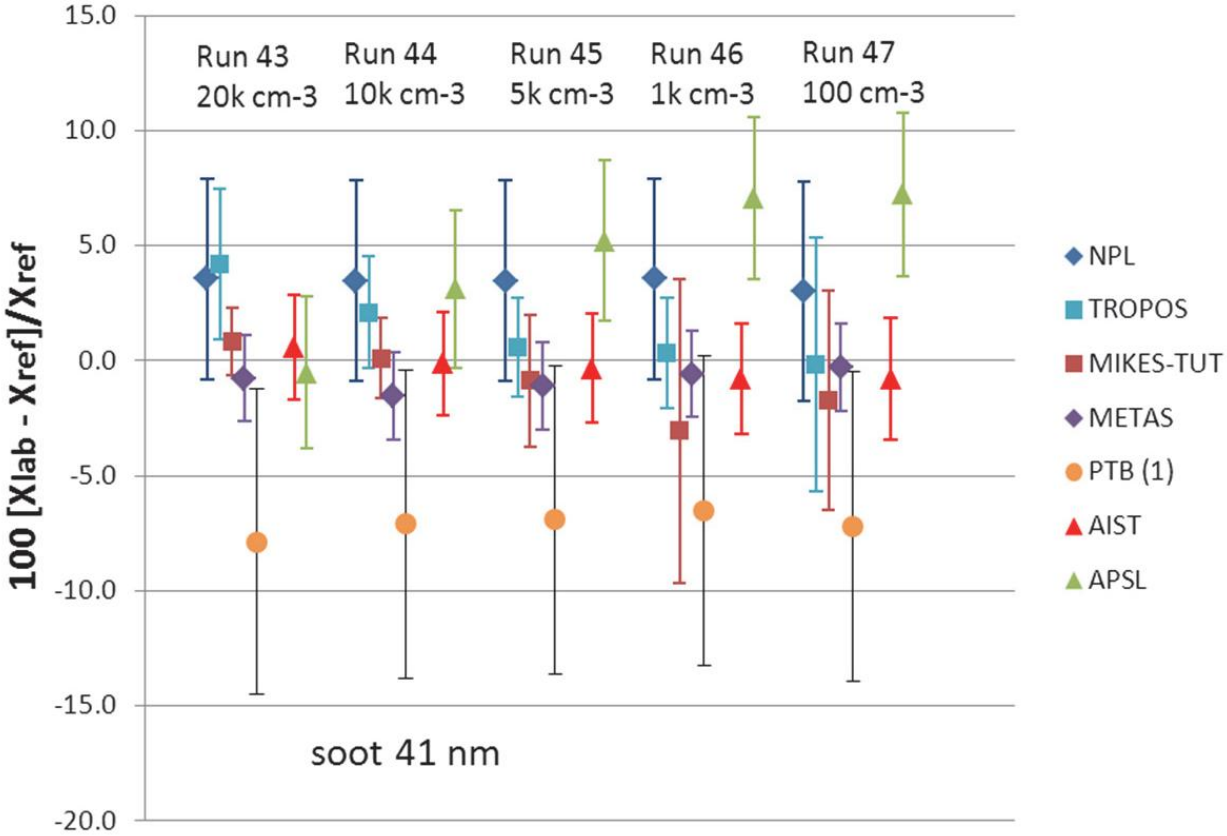
→ For all participants: Uncertainty of ± 2% for DOS particle regime

→ PTB: Full uncertainty budget of ± 7 % for soot particle size 15 to 150 nm, conc. at 650 to 12.000 cm^{-3}

PN-comparison: Results of secondary method

Also first comparison of its kind for condensation particle counter (CPC) in EURAMET 1282 (Nov. 2014):

- Different types of aerosols: soot; silver sintered (more spherical) and unsintered
- Soot particle size range 23 to 100 nm, conc. 100 to 20.000 cm³



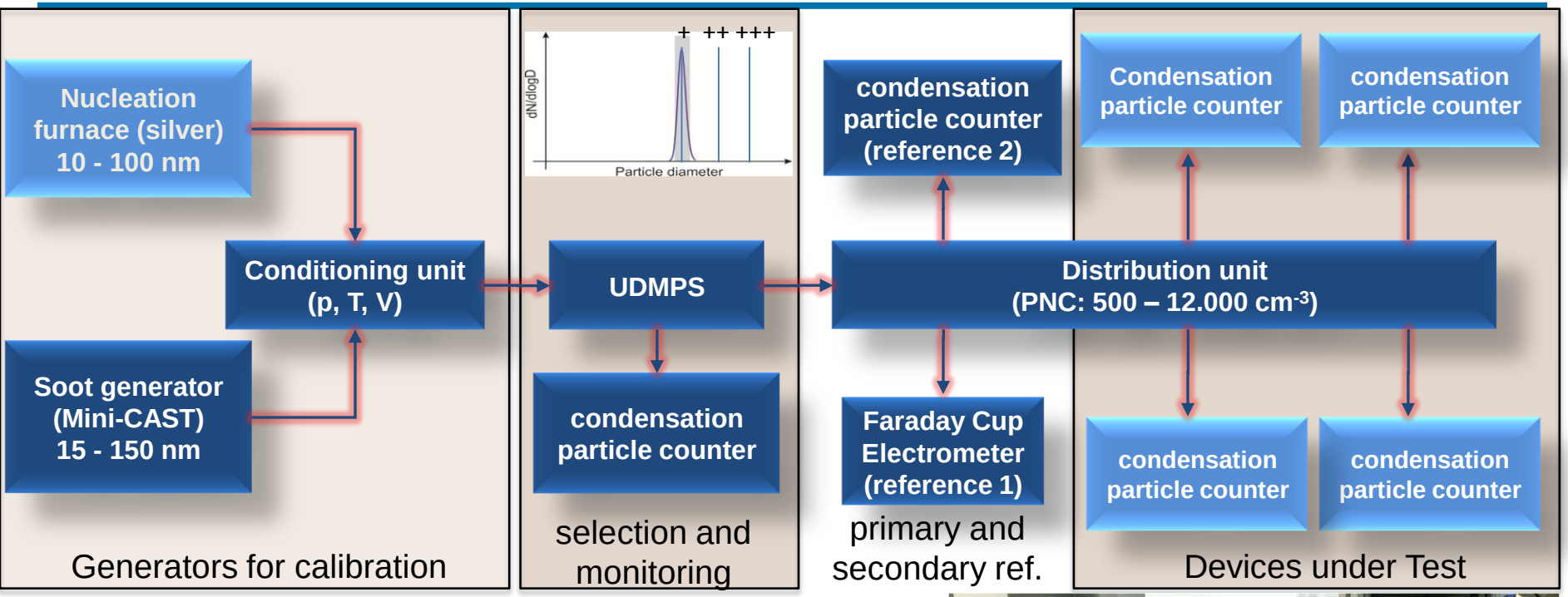
more information see:

- NPL Report AS 94

→ For all participants: Uncertainty budget of ± 7 % for soot regime

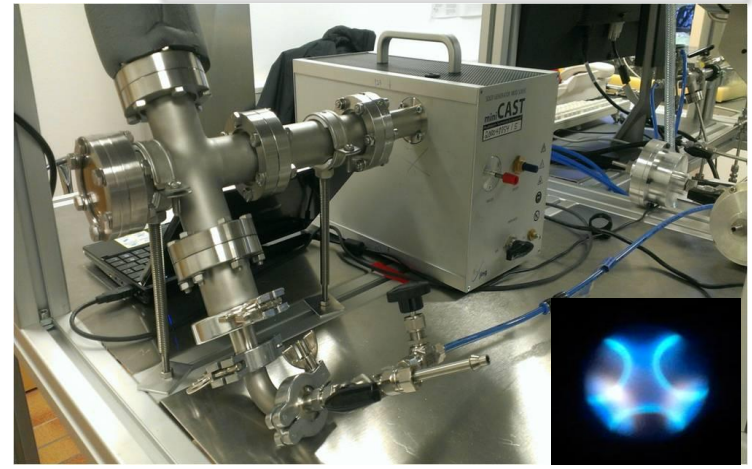
→ PTB: Full uncertainty budget of ± 10 % (soot, silver) at this time

PTB-primary standard for PN



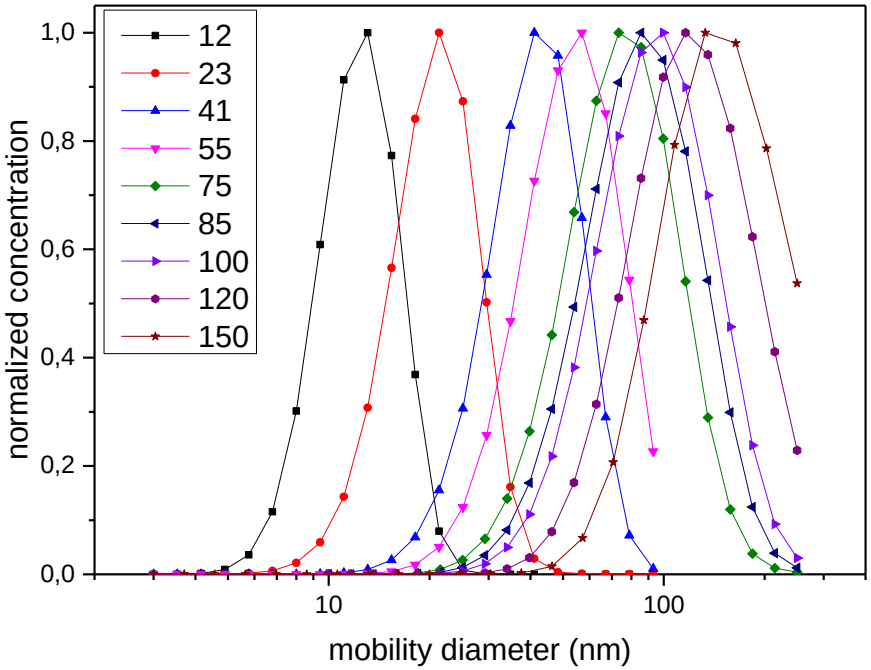
Goal: Suitable calibration aerosols for PMP conform EECPC

- → High purity substance for calibration (propane and silver)
- → High monodispersity (Minimization of multiple charges)
- → Sufficient particle number concentration **(PNC)**
- → Tunable particle diameter between 10 to 100 nm **(Dp)**
- → Validate morphological properties
- → High thermal stability, up to 350 °C

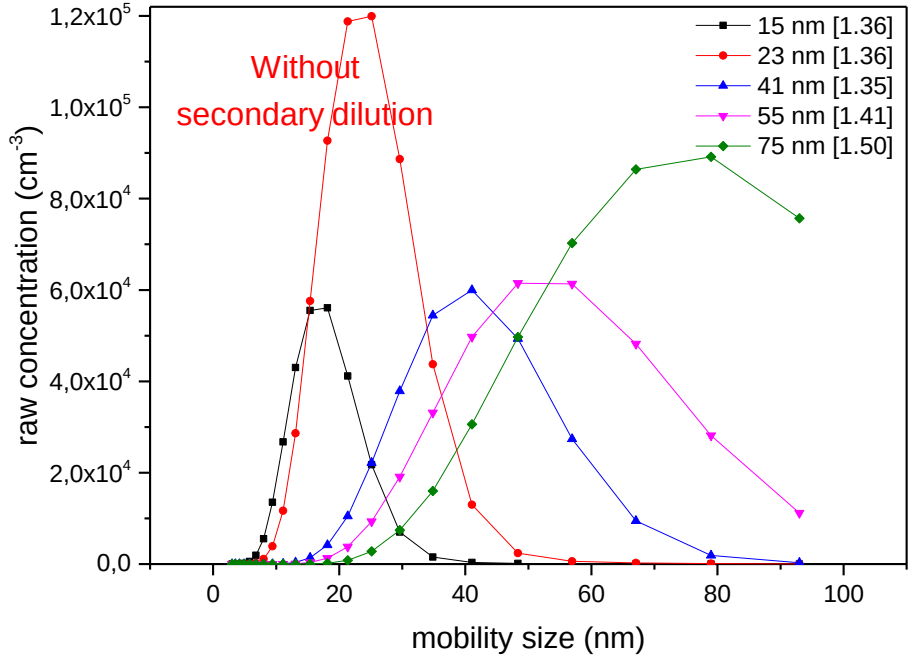


Results of soot generator: operation points

Full range of soot generator



PMP range for calibration of EECPC

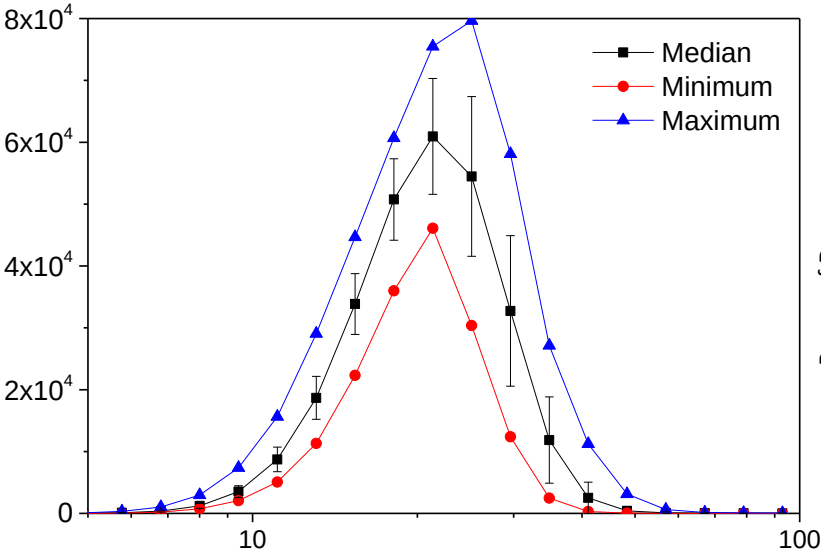


- only PN spectra below 75 nm relevant for calibration
- narrow PNSD could be observed

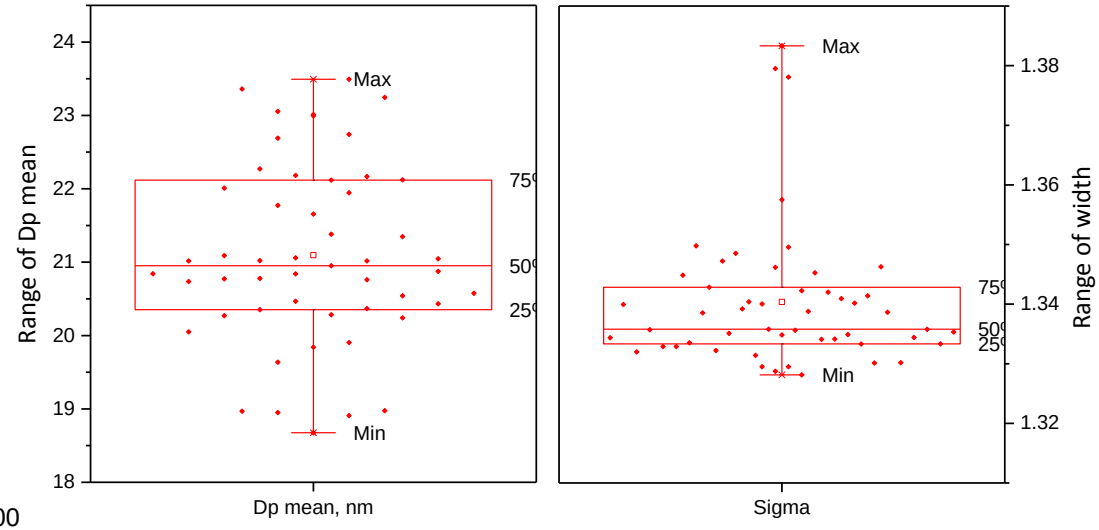
Results of soot generator: long term stability

- Point of interest 23 nm (counting efficiency of 50%) about 1 year of operation

Stability of PNSD



fit parameter for Dp mean and width of PNSD

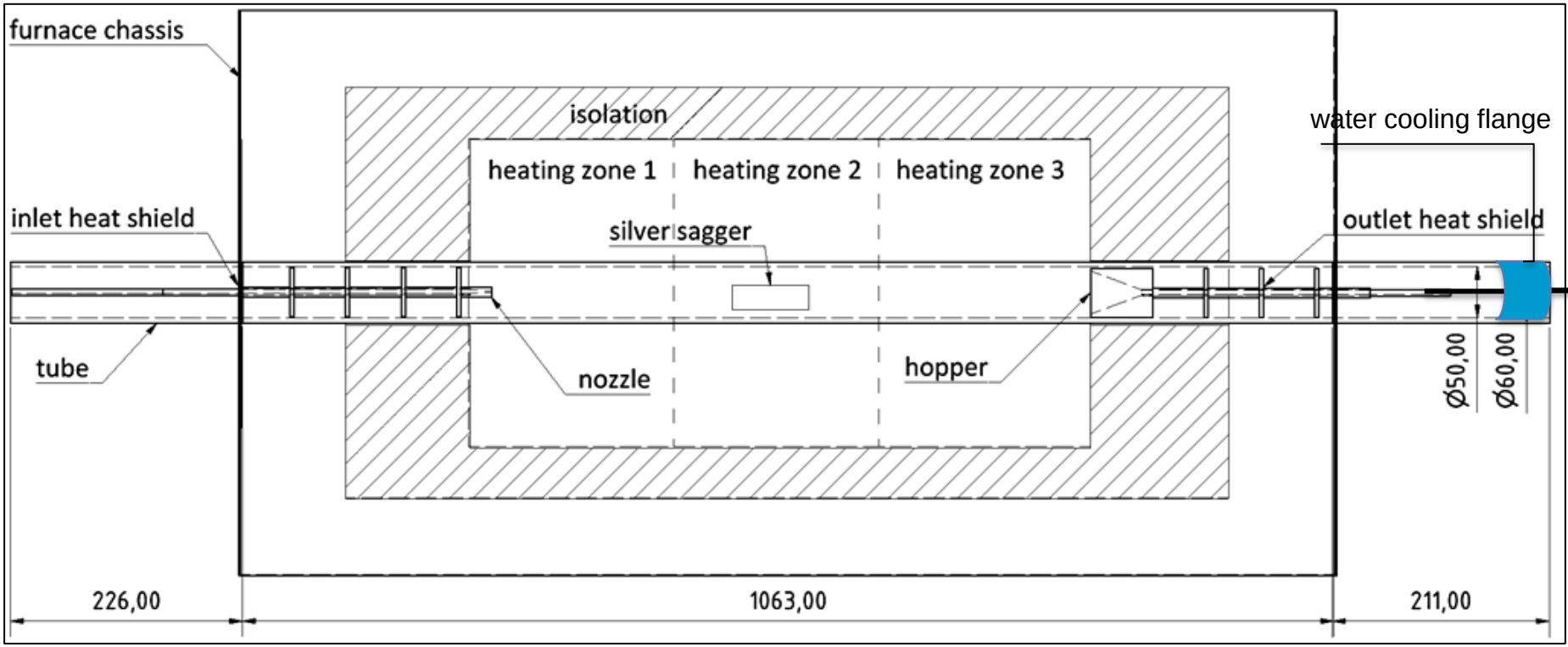


→ Increased stability by thermoconditioning of soot particles, but still not suitable for high thermal stability

→ NMIs decided to use Ag nanoparticles as suitable candidate for these criteria:

→ Advantage: Full calibration of entire PMP-system (VPR, DF and CPC)

Ag-Nucleation furnace: PTB Optimization

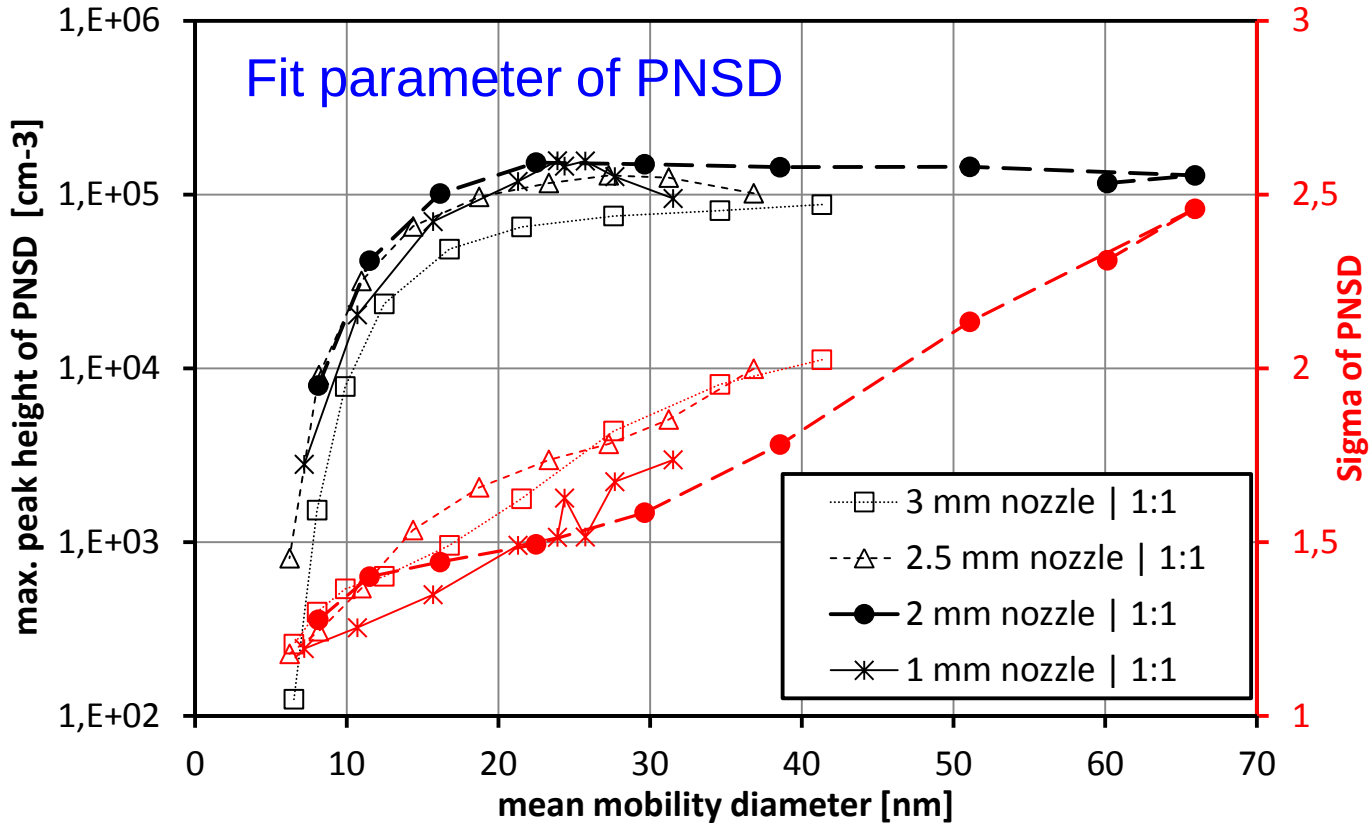


larger furnace with 3 heating zones → larger particles → increase of residence time

- optimization flow scheme and T gradient → implementation of heating shields:
  - inlet shield with nozzle (different sizes)
  - outlet shield with hoper
- minimization of Ag-agglomerates → water cooling flange at end of tube → shock cooling
- Goal to fulfill PMP size range from 23 to 55 nm

Results for new nucleation furnace

- For different nozzle sizes at one flow ratio (1:1) from 960 °C up to 1300 °C



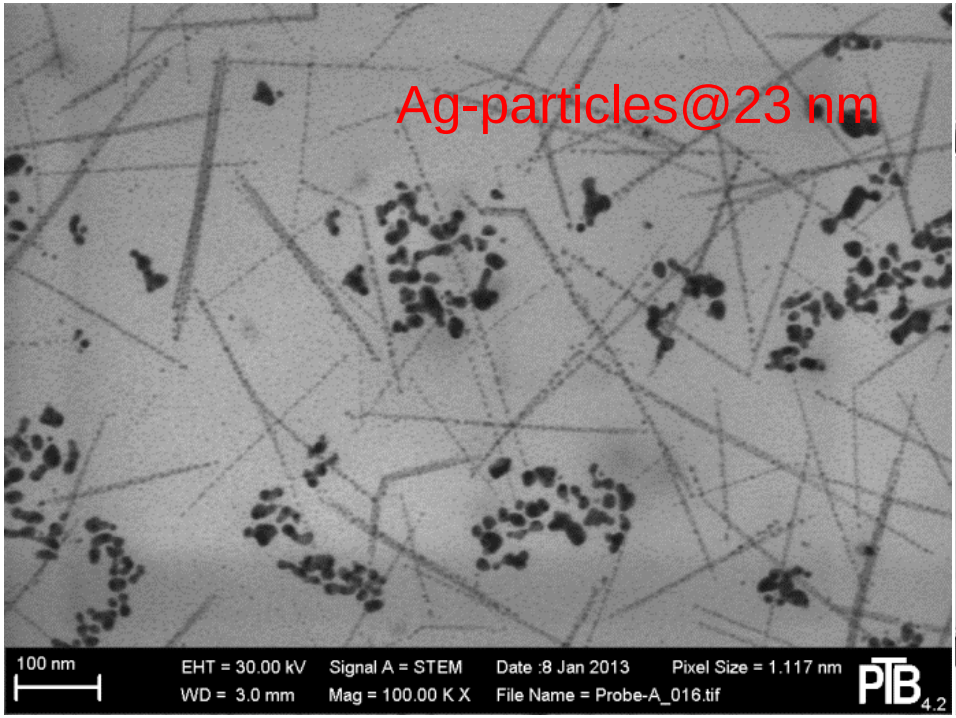
- High PNC (> 10^5 cm^{-3}) above 15 nm
- 2 mm nozzle: mean mobility diameter up to 65 nm, relatively broad size distribution

  → 2 mm nozzle well suited for calibration setup

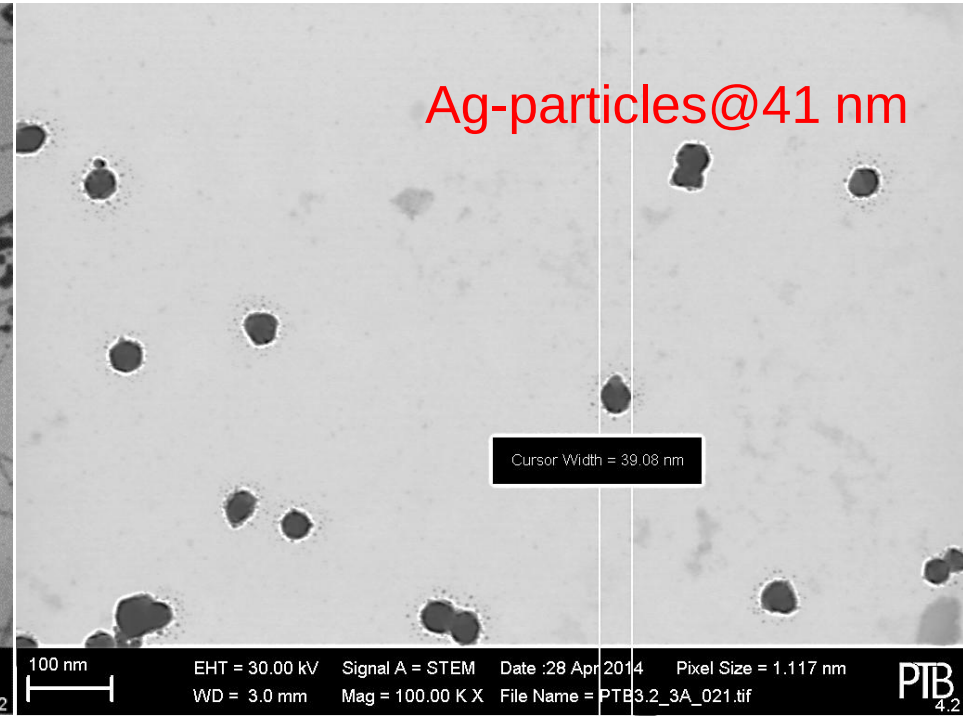
Results of nucleation furnace: Particle Shape

- STEM pictures for different types of tube furnaces

Old furnace construction comparable to Scheibel and Porstendörfer



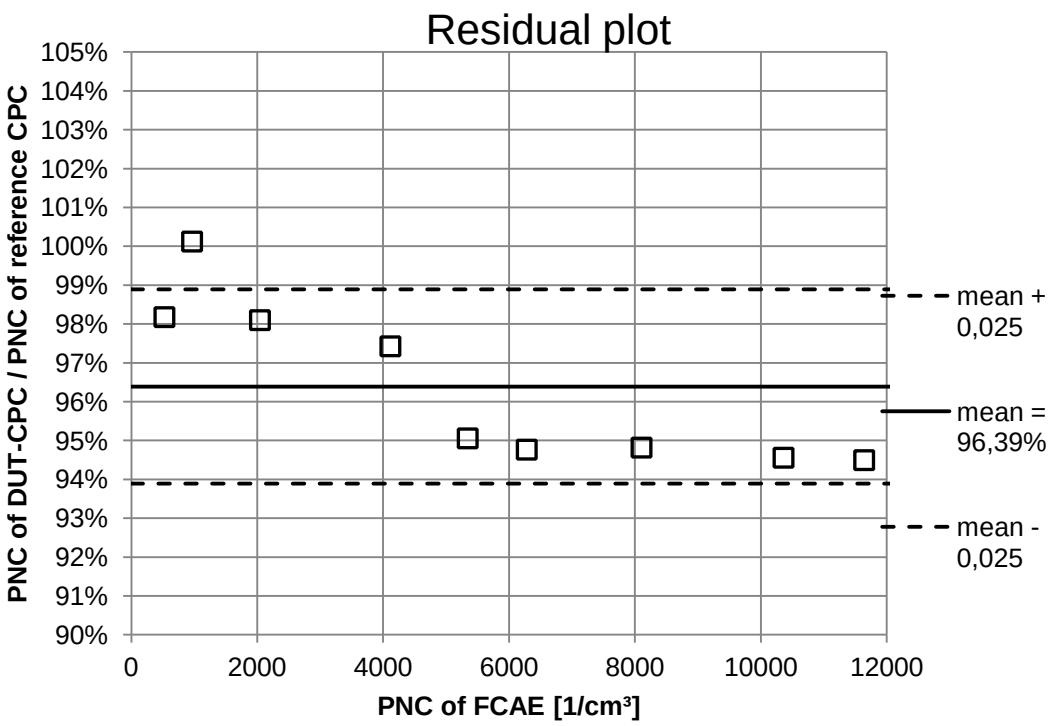
New tube furnace at PTB

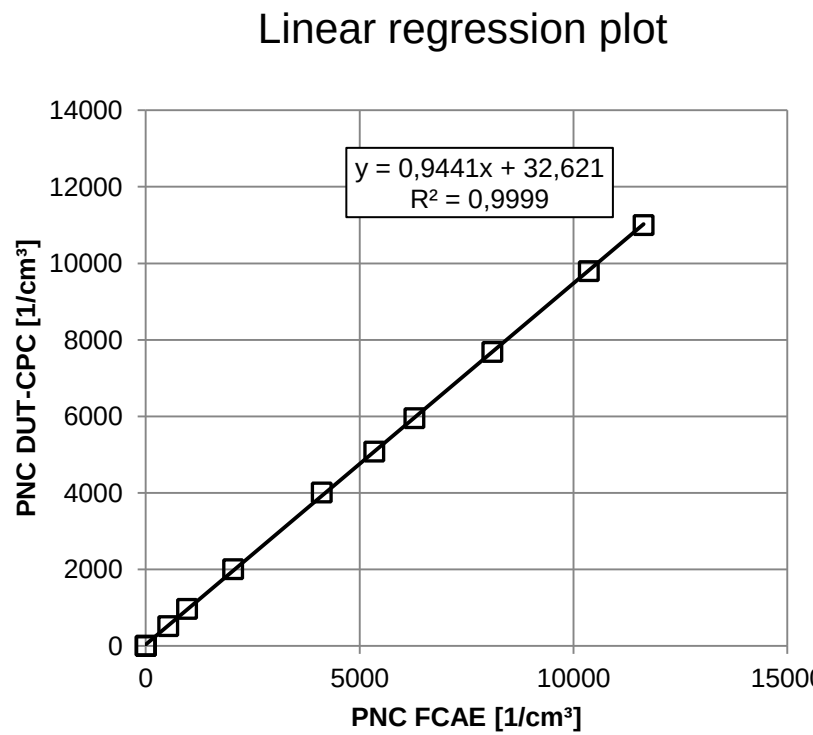


➢ Without sintering mostly spherical particles

First calibration: Linearity test of EECPC

- For PTB-EECPC against Ref. FCAE and Ref. CPC



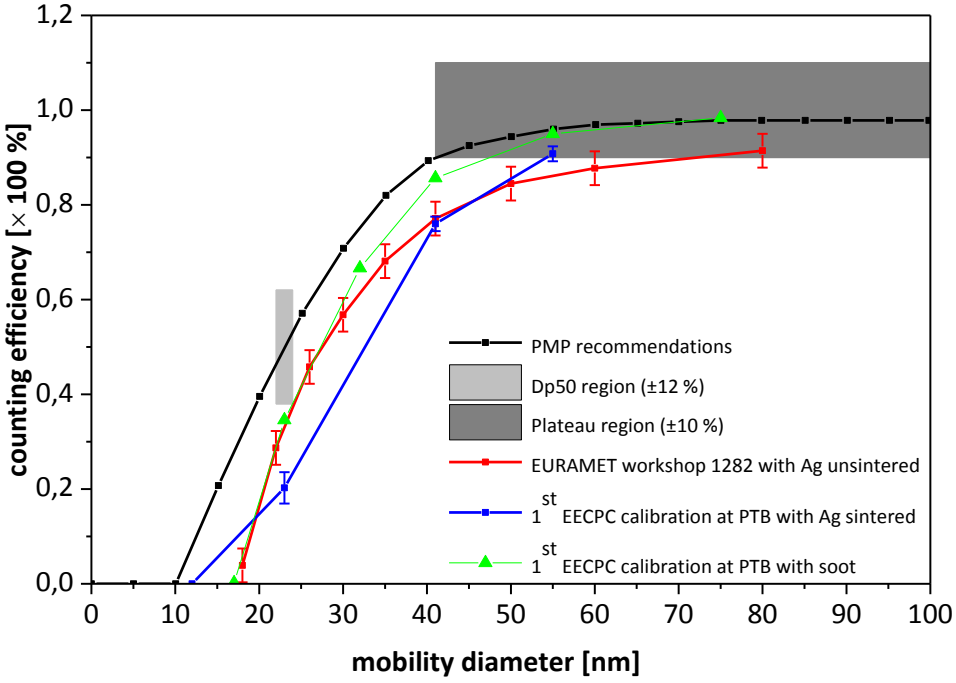


→ analysis fulfills ISO 27891 (Mar. 2015) based on thermoconditioned soot particles

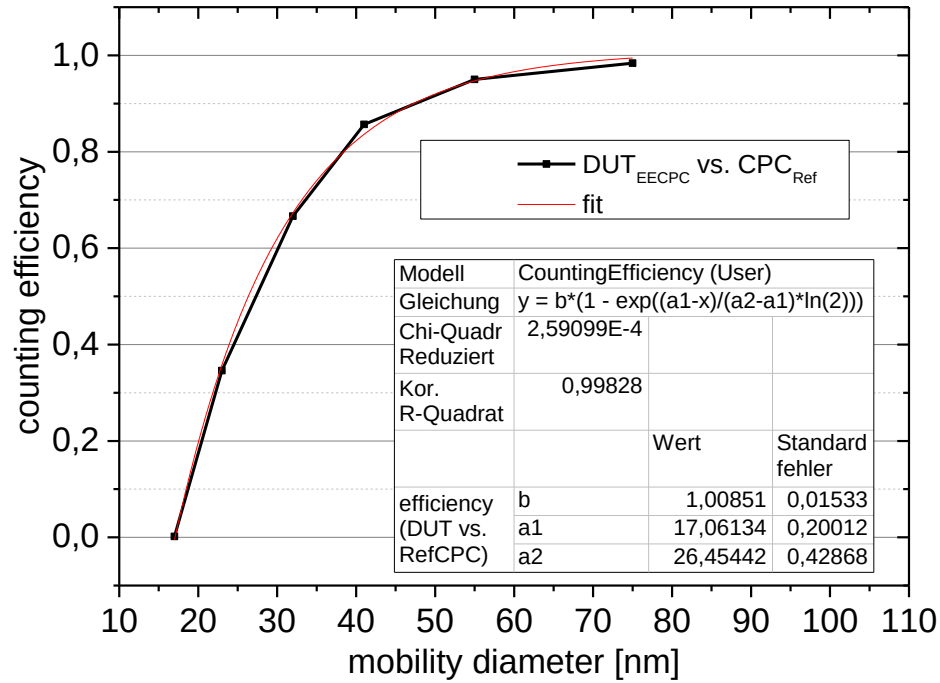
First calibration: Counting efficiency of EECPC

- PTB results compared to EURAMET workshop in Leipzig

For soot and silver particles



Only soot particles (thermoconditioning)



→ Analysis for fit function based on formula given by ISO 27891 (Mar. 2015)

→ Slight underestimation of Dp50 efficiency for all calibration aerosols

Conclusion

- Primary and secondary PN reference method validated via international inter comparison workshops

- PMP-EECPC calibration setup was build for soot as well as for Ag particles in size range from 15 to 75 nm with suitable PNC up to 12.000 cm^{-3}

- Evaluation of PTB calibration procedures is comparable to ISO 27891

Outlook

- Further optimization of Ag size range to smaller sizes below 10 nm via secondary nucleation furnace

- Minimization of charge correction factors above 60 nm

  →2nd UDMPS with unipolar charging

  →Increasing of PNC for monodisperse fraction

- Internal Audit is needed to provide external calibration certificates for EECPC end user

  ➔ Establish PTB-EECPC calibration service in middle 2016

Thank you very much for your attention!
→ Questions?

Physikalisch-Technische Bundesanstalt
Braunschweig and Berlin
Bundesallee 100
38116 Braunschweig

Dr. Andreas Nowak
Email: andreas.nowak@ptb.de
www.ptb.de