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EURAMET
European Association of National Metrology Institutes

Measuring Soot Particles from Engines: Recommendations from EMRP-ENV02 Project in WP1

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ENV02- consortium

JRC EUROPEAN COMMISSION

ie Institute for Energy

DFM

PTB

METAS

NPL National Physical Laboratory

MIKES

TROPOS Leibniz Institute for Tropospheric Research

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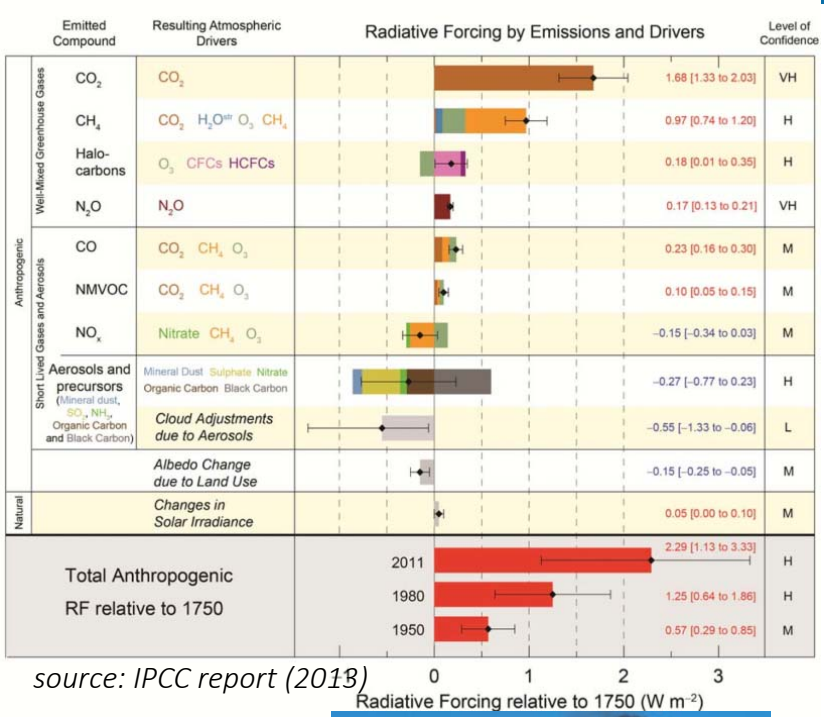
    - particle number standards

- Summary

Introduction
"soot" in PartEmission

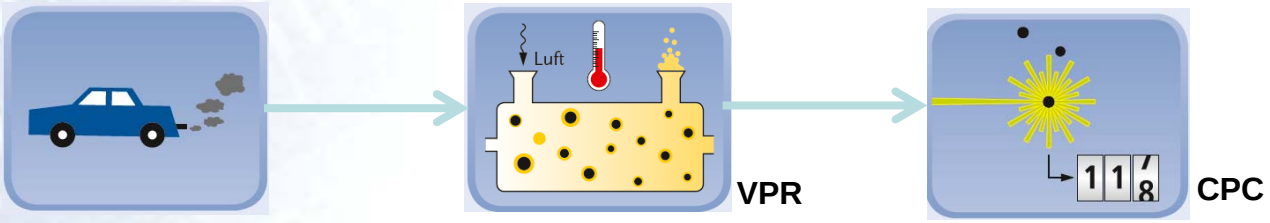
- "soot" is defined as the impure carbon particles resulting from an incomplete hydrocarbon combustion;
- "soot" is carcinogenic (WHO 2012)
- "soot" is the 2nd largest cause for global warming
- Two work package in PartEmisison address "soot":
  - *automotive particle emission metrics*
  - methods for periodic emissions control

  (See Poster Session 3: 29 and 31)



source: IPCC report (2013)

Introduction
UN-ECE R49 and R83 requirements



- Calibration of the entire measurement system at certified particle mobility diameters of 23 nm to 100 nm and certified number concentrations between 10^1 cm^{-3} and 10^4 cm^{-3}

- Calibration of the particle concentration reduction factor of the volatile particle remover (VPR) with 30 nm, 50 nm and 100 nm particles
  → temperature stable calibration aerosol up to 400 °C

- Calibration of the counting efficiency of the engine exhaust condensation particle counter (EECPC) with 23 ,41 and 55 nm particles → “soot-like”-aerosol

Automotive particle emission metrics work programme and deliverables

- A particle number concentration standard for soot particles shall be established with the aim of providing a independent calibration services for end users and industry, in particular for the calibration of measuring instruments for the type approval of Euro 5b / 6 diesel vehicles.

- The mentioned goal is achieved by six deliverables:

1.	Particle size standards
2.	Temperature resistant aerosol
3.	«soot-like» aerosol
4.	Particle number concentration calibrations services
5.	National particle number concentration standards
6.	“Round robin” test initiated

Automotive particle emission metrics particle size standards – SI traceability

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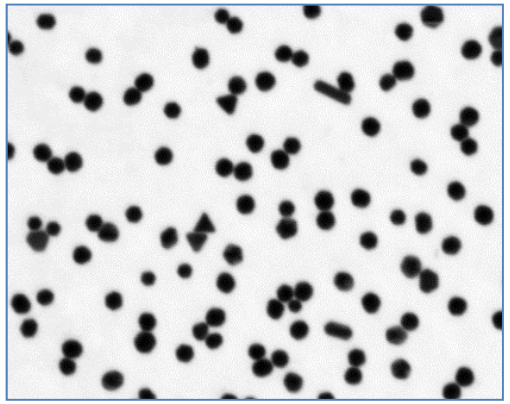
- Spherical reference gold and silver particles studied for the calibration of differential mobility analyser (DMA)
- Successful aerosolization (electrospray generator), but systematic deviations between measured mobility diameter (DMA) and geometric diameter (TSEM)



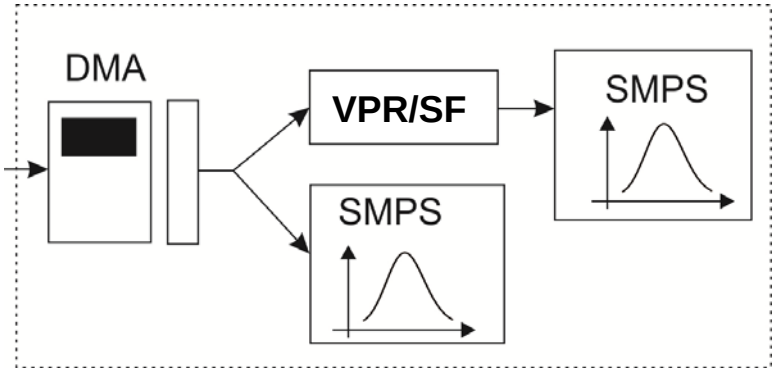
TSEM image, Au 30 nm particles

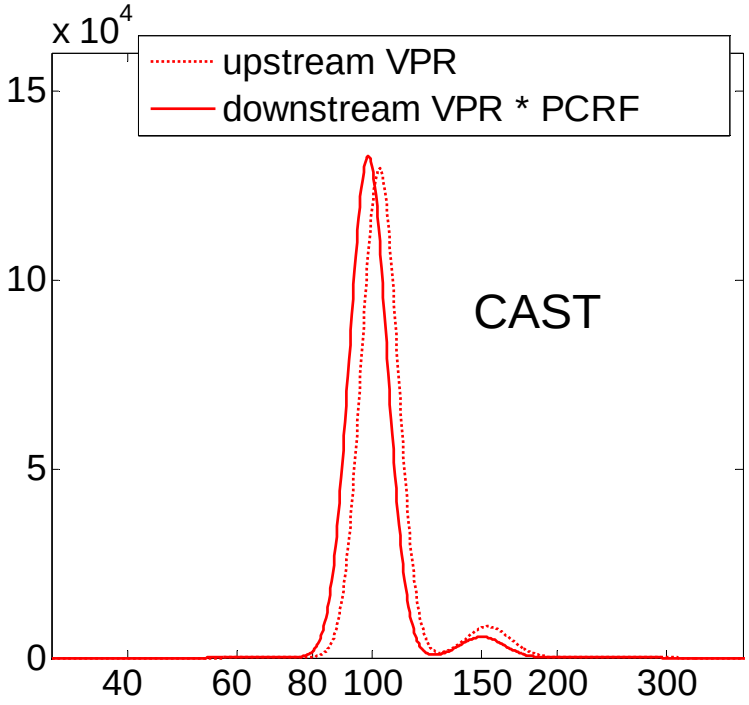
Nominal diameter	20 nm	30 nm	40 nm
TSEM mean diam	21.0 ± 1.9 nm	29.8 ± 2.0 nm	44.5 ± 2.2 nm
TSEM mode diam	20.4 nm	29.3 nm	42.9 nm
DMA mode diam	24.0 ± 0.4 nm	33.8 ± 0.6 nm	44.6 ± 1.0 nm
Factor DMA/TSEM	**1.18**	**1.15**	**1.04**

- Imperfect sphericity or slip correction factor could explain the increasing differences with decreasing particle sizes.
- The consortium discussed the need for further investigations and issued a recommendation (please see web site of ENV02 “PartEmission”, supplement)

Automotive particle emission metrics temperature resistance – results

- Thermal stability assessed via comparing size distribution upstream and downstream of an VPR or sintering furnace (SF)
- Graphite spark aerosol does not need thermal treatment; large size distributions
- CAST combustion aerosols need thermal treatment to be thermally stable;
- Silver particle aerosols need sintering at 400°C for thermal stability; upper size limited to 50 nm





Automotive particle emission metrics "soot-like" aerosols – counting efficiency

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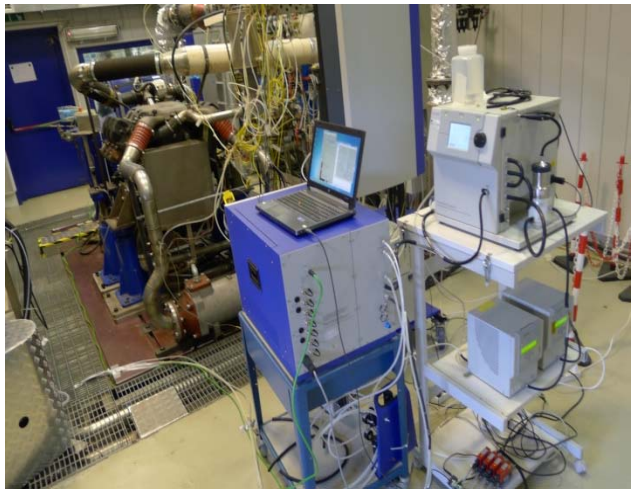
- Counting efficiencies at 23 nm & 41 nm of a PMP compliant CPC were measured with miniCAST aerosol, graphite spark aerosol and and selected "real-soot" (Daimler V6 HD engine; DAF Paccar engine, moped engine) according to ISO/FIS 27891.

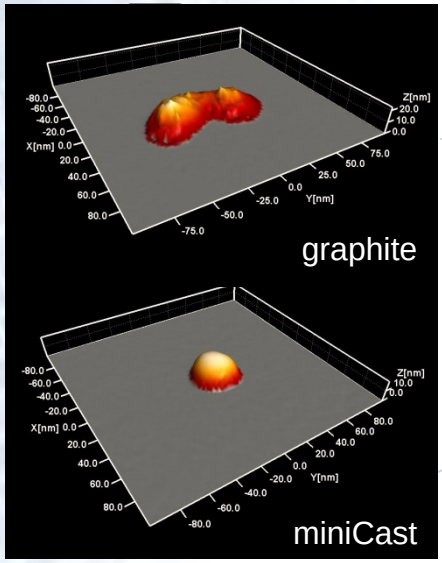


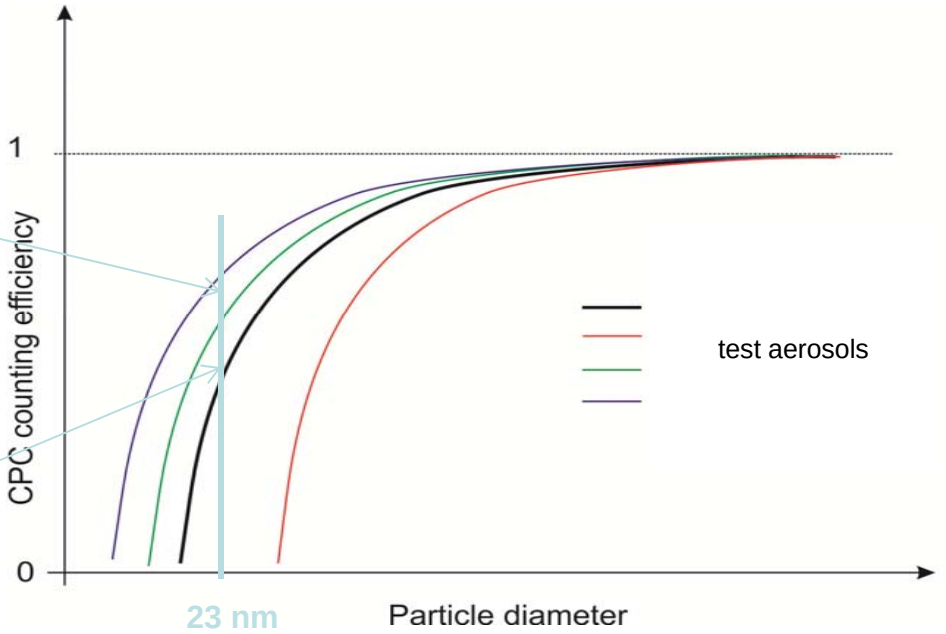
- Engine -> DPF (-> CVS) -> PMP -> SMPS ->EECPC (Ref CPC)
  miniCAST/graphite spark-> PMP or CS -> SMPS ->EECPC (Ref CPC)

Aerosol	Efficiency @ 23 nm	Efficiency @ 41 nm
miniCAST	26.5 % - 48.5 %	77.5 % - 79.9 %
graphite spark	41.4 % - 48.5 %	79.9 % - 93.3 %
«real soot»	42.5 % - 56.6 %	83.5 % - 89.2 %

Automotive particle emission metrics "soot-like" aerosols - morphology

- Material dependent counting efficiencies found; calibration aerosols as well as different engine conditions for "real-soot" vary by ~20 % @ 23 nm.



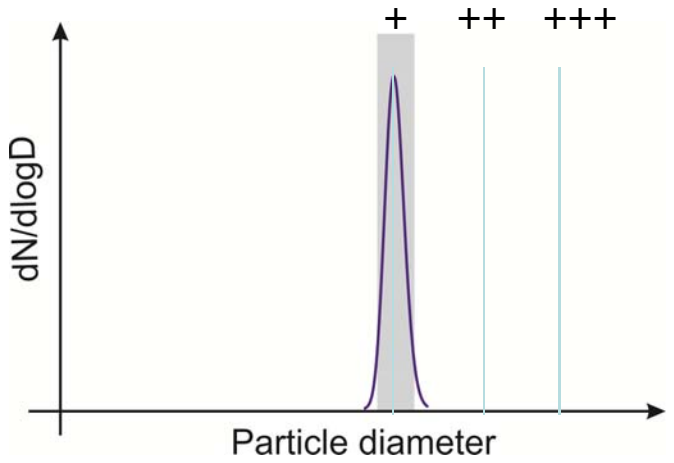


- "soot-likeness" is not the best criterion for a calibration aerosols considering the intrinsic variability of real soot and tested aerosols.

Automotive particle emission metrics suitable calibration aerosols – NMI's criteria

- Better criteria for a suitable calibration aerosols:

  Monodispersity
  Single charge
  Tunable size
  Sufficient number concentration
  Controlled morphology (spherical)
  Thermal stability
  …



- Calibration of the particle number concentration of a test CPC by comparison with an aerosols electrometer according to ISO/FIS 27891.

→ SI-Traceability to units Volume, Time and Ampere.

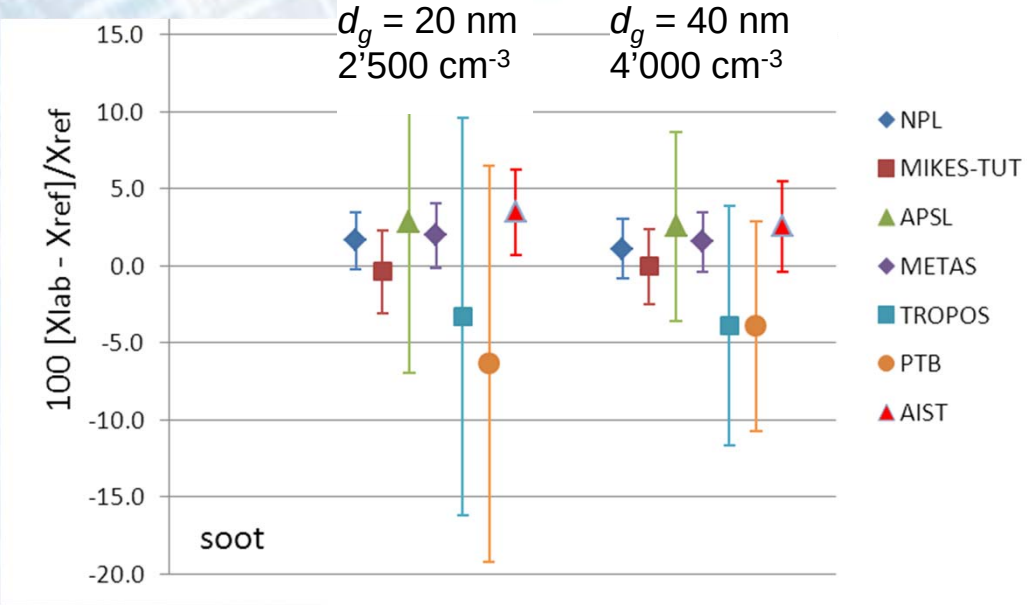
→ Linear interpolation for small number concentrations.

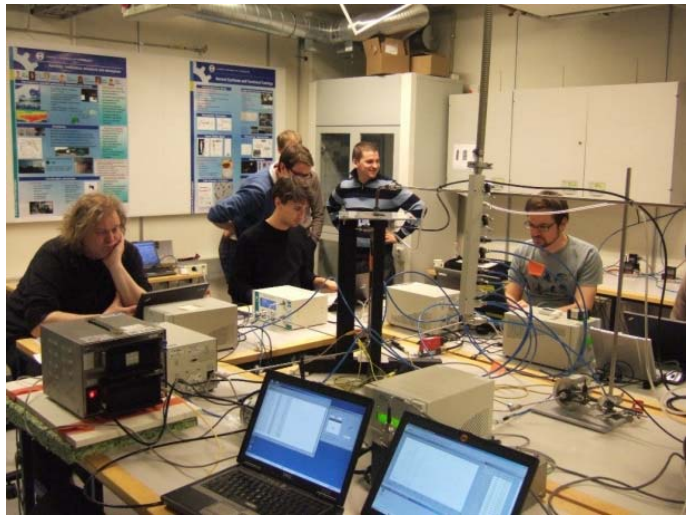
Automotive particle emission metrics particle number standards – FCE comparison

- EURAMET 1244 compared measurements of airborne charge concentration (in $fC \cdot cm^{-3}$). First comparison of this kind!
- The comparison was based on measurements of a common aerosol source and was hosted by the Tampere University of Technology (TUT) in Finland on 18-22 March 2013.
- aerosol sources:
  SCAR (single charge aerosol reference)
  Multiple charge soot generator
- Particle sizes from 6 nm to 200 nm.
- Concentration range from 0.15 $fC \cdot cm^{-3}$ - 3 $fC \cdot cm^{-3}$ (equivalent to around 1,000 particles cm^{-3} to 20,000 particles cm^{-3})

Automotive particle emission metrics particle number standards – FCE comparison

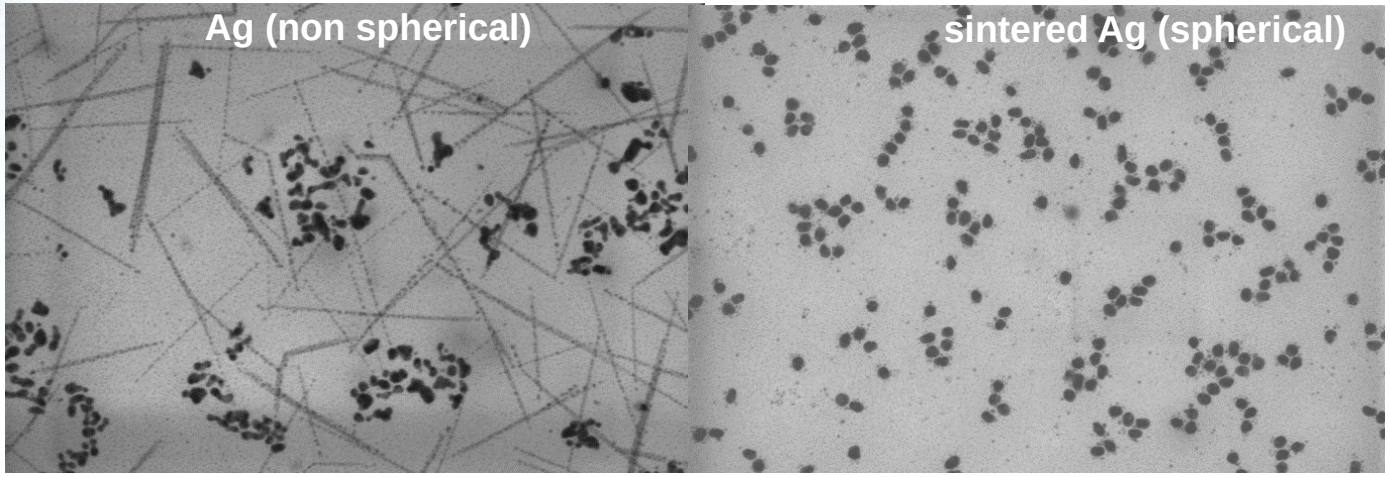
- Participants generally agree within 2 % in the size range 20 nm to 100 nm and number concentrations above 5000 cm^{-3} for singly charged particles.
- JRC results were 10 % to 30 % lower than the other participants' results; most probably due to commercial instrument design.
- Larger deviations result at lower particle sizes and particle concentrations and soot particles (see Högström et all at Metrologica 51, 293)





Automotive particle emission metrics particle number standards – CPC comparison

- EURAMET 1282 compared measurements of particle number concentration (in cm^{-3}).
- The comparison was based on measurements of a common aerosol source and was hosted at TROPOS in Leipzig on 14-18 October 2013.
- aerosol sources:
  sintered silver
  silver
  soot of MINI-CAST

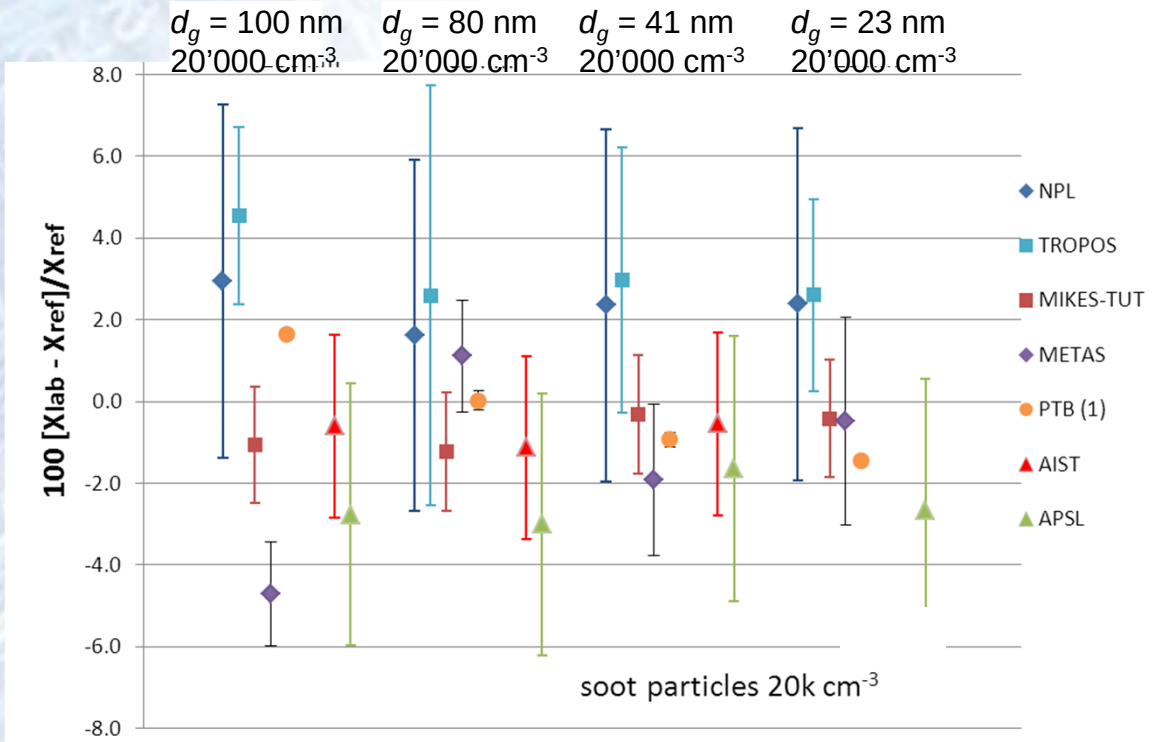


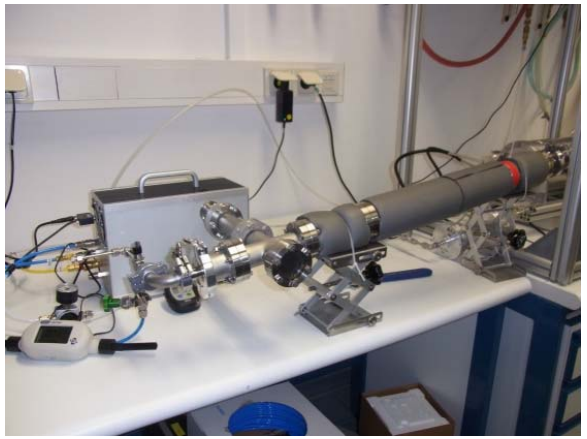
- Particle sizes from 6 nm to 100 nm.
- Concentration range 100 particles cm^{-3} to 20’000 particles cm^{-3}

Automotive particle emission metrics particle number standards – CPC comparison II

- Preliminary results suggest that the agreement of participants is less good than in the FCE comparison.

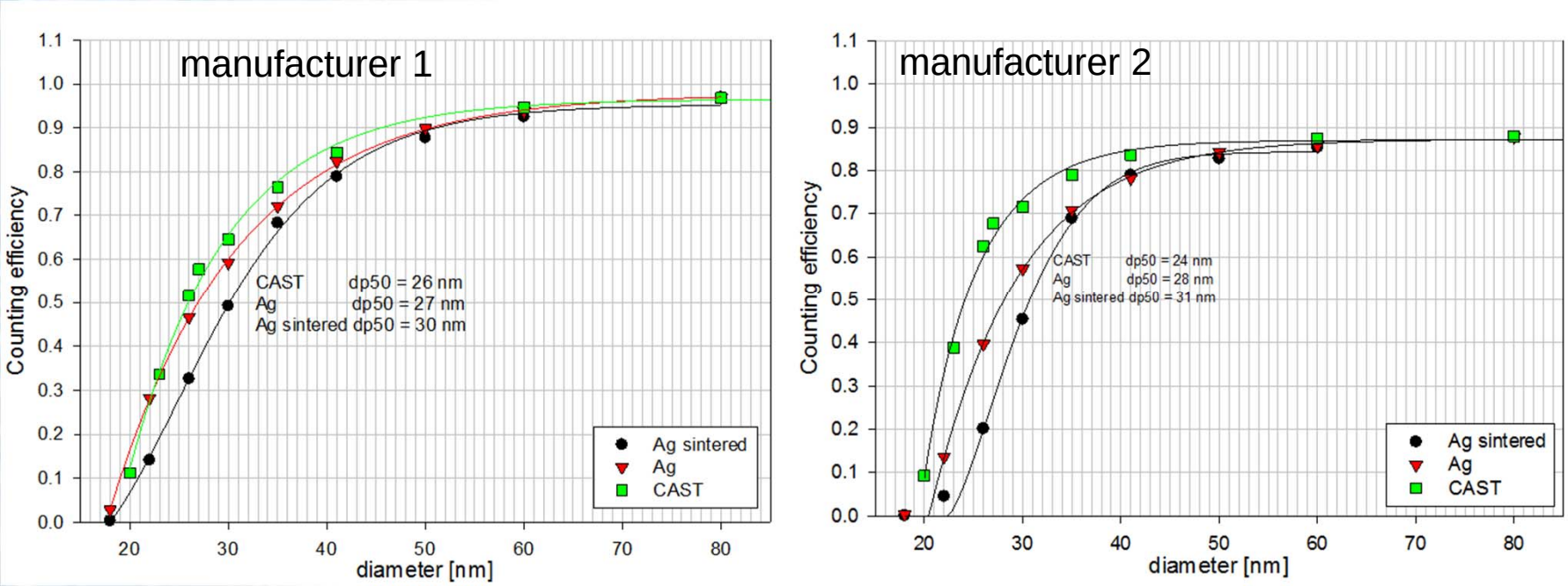
- Increased uncertainties down the traceability chain from primary FCE → reference CPC (ISO/FIS 27891).





Automotive particle emission metrics particle number standards – “round robin”

- The “round robin” took place in conjunction with the CPC comparison at TROPOS in Leipzig on 14-18 October 2013.
- Two instrument manufacturer attended the “round robin” with their “PMP-compatible” CPCs:



- Results will be discussed with instrument manufacturer.

Automotive particle emission metrics
Summary of achieved results

- Mobility and geometric diameter differ below 40 nm.
  → Recommendation to calibrate DMA size measurements.
- Thermally treated combustion, sintered silver and graphite aerosol are partially suitable to calibrate PMP-compatible VPRs.
- There is not a single “soot-like” aerosol suitable to calibrate counting efficiencies of PMP-compatible CPCs.

  →Heterogeneous nucleated silver particles are promising “primary” calibration aerosols

- Worldwide comparability of particle charge and number measurements demonstrated.

- EMRP PartEmission allowed to improve and built up the infrastructure in Europe

Thank you very much

Questions?

Automotive particle emission metrics particle size standards – recommendation

- The certified size standards shall consist of monodisperse spherical particles normally in a suspension (ISO Guide 30:1992 certification). The material for the particles shall allow perfect spheres.

- The certified size standards shall have particle diameters above 80 nm and a maximal standard deviation of 5 % of the diameter.

- The DMA shall be adjusted with at least one certified size standard. 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 kept constant during adjustment and subsequent measurement.

- The particle mobility diameter for particles below 80 nm is calculated using the equations from ISO 15900 (e.g. slip correction) and is assumed to be correct by convention.

Automotive particle emission metrics particle number calibration – ISO/DIS 27891

PTB

- ISO/DIS 27891 describes a calibration procedure for condensation particle counters (CPCs)

  CPCs measure particle number concentration in the size range from a few nm to a few µm.

  The DIS refers to the role of NMIs in providing certification for reference CPCs, and aerosol electrometers, which can be used to calibrate CPCs

- METAS, NPL and PTB have extended or newly developed calibration capabilities for FCE (aerosol electrometers) and reference CPCs.

  Relevant particle size range for engine exhaust is 5 nm to 200 nm.

  Services to be provided after successful validation by two comparisons.

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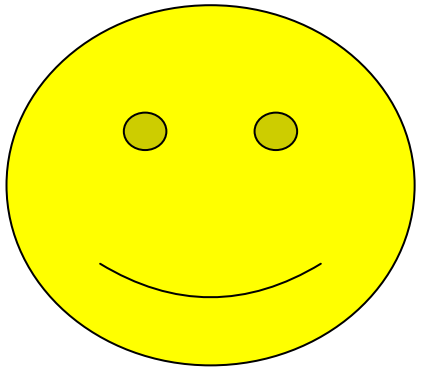

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