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Swiss Confederation

Swiss Federal Office of Metrology METAS

Towards international harmonisation of nanoparticle size and number – EURAMET 1027

Teddington, 8th June 2010

Jürg Schlatter, METAS, Switzerland



Overview

- Traceability
- Measures for particles in aerosols
- Comparison experiment
- Results of comparison experiment
- Conclusions
- Future challenges

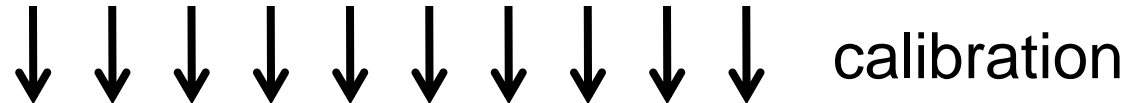


Traceability 1/2: User level

National standards at
National Metrology institutes (NMI)



Transfer-Standards of
(accredited) calibration laboratories
(e.g. instrument manufacturers)

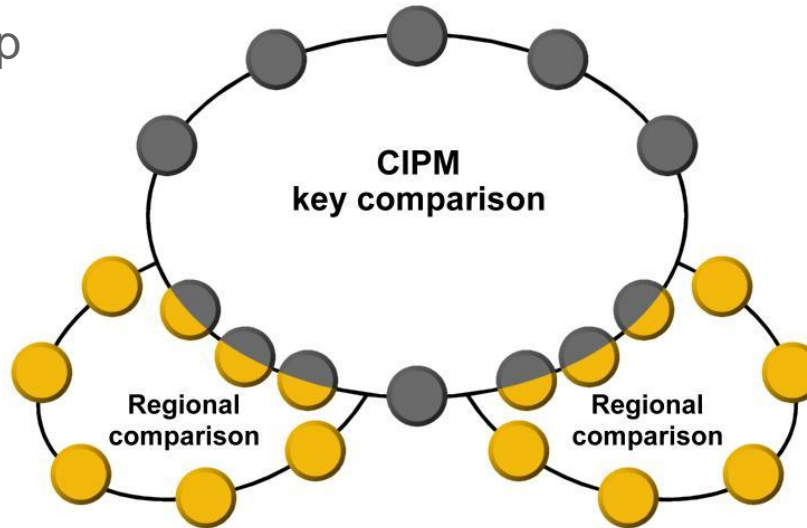


Measuring instruments of user
(e.g. industry, authorities, accredited laboratories)



Traceability 2/2: international level

Gas working group
(GAWG) in
CCQM of CIPM
www.bipm.org



SC gases of TC-MC
www.euramet.org

BIPM-database for the Calibration and Measurement Capabilities CMC lists the internationally accepted services or reference materials

Calibration and Measurement Capabilities

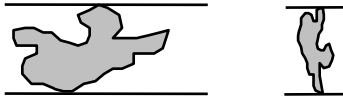
Amount of substance, gases, Switzerland, METAS (Federal Office of Metrology)

Note: No ranges of certified values in reference materials declared by Switzerland.
The notation $Q[a, b]$ stands for the root-sum-square of the terms between brackets: $Q[a, b] = [a^2 + b^2]^{1/2}$

NMI Service Identifier	Measurement Service Sub-Category	Matrix	Measurand		Dissemination Range of Measurement Capability			Range of Expanded Uncertainties as Disseminated					Mechanism(s) for Measurement Service Delivery	
			Analyte or Component	Quantity	From	To	Unit	From	To	Unit	Coverage factor	Level of confidence		Is the expanded uncertainty a relative one?
232-1	Environmental	nitrogen	carbon monoxide	Amount-of-substance fraction	40	200	$\mu\text{mol/mol}$	0.8	0.8	%	2	95%	Yes	Calibration of gases
232-2	Environmental	nitrogen	carbon monoxide	Amount-of-substance fraction	1	50	mmol/mol	0.4	0.4	%	2	95%	Yes	Calibration of gases
232-3	Environmental	nitrogen	carbon	Amount-of-substance	50	150	mmol/	0.4	0.4	%	2	95%	Yes	Calibration of

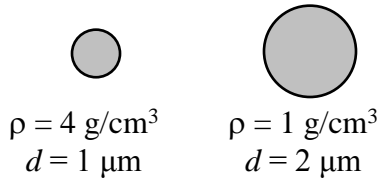


Measures 1/2: Single particle



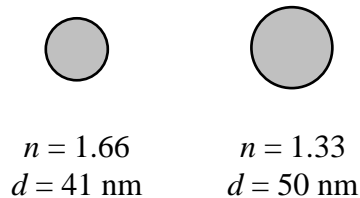
Geometric diameter:

Considering their size, the two particles are approximately the same.



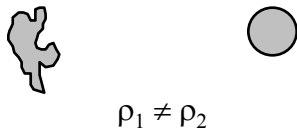
Aerodynamic diameter:

Considering the sedimentation velocity, the two particles are approximately the same.



Optical diameter (as a function of λ , φ):

Considering the light scattering properties, the two particles are approximately the same.



Mobility diameter:

Considering the diffusion behavior, the two particles are approximately the same.



Measures 2/2: Set of particles

Diameter for a set of particles in aerosols:

- Geometrical mean \hat{d}_g
- Median d_{50}
- Mode d
- Mean \bar{d}

Amount of particles in aerosols

- Number concentration
- Concentration of ingredients (e.g. coulometry for carbon)
- Surface concentration
- Mass concentration



Experiment 1/5: Instruments

Aerosol manifold*

Particle counters:

METAS: CPCs, 2.3 L/min
UBA: CPC, 1.5 L/min
NPL: CPC, 0.6 L/min
AIST: CPC, 1.0 L/min



Particle sizers:

METAS: SMPS, 0.3 L/min
FORCE: ELPI, 10 L/min
UBA: SMPS, 1.0 L/min
NPL: SMPS, 0.6 L/min



Structure analyser:

DFM: AFM sampler, 0.5 L/min



*Tube length are adapted to flow rate to compensate diffusion losses. ⁷



Experiment 2/5: Measuring parameters

- **Particle number concentration:**

Number of particles per volume with all diameters at actual ambient condition

Range: 10^3 cm^{-3} to 10^6 cm^{-3}

- **Particle size:**

Mode and **geometrical mean** of the size distribution with the particle diameter as the equivalent electrical mobility

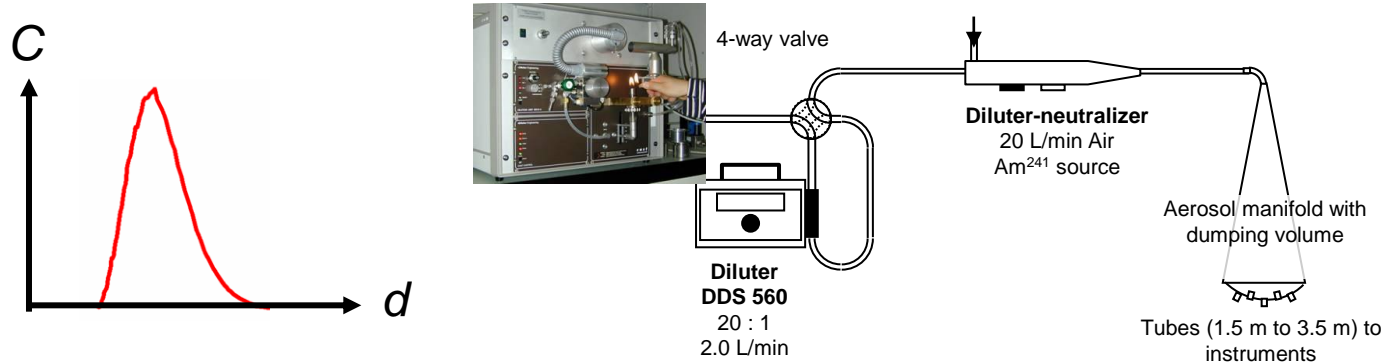
Range: 50 nm to 180 nm



Experiment 3/5: Particle Generation

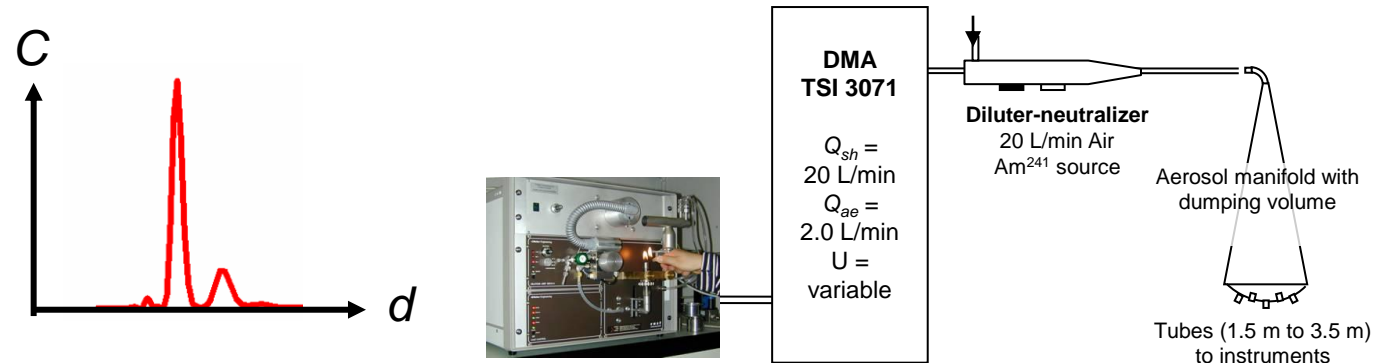
"natural" size distribution

70 nm, 100 nm, 140 nm and 170 nm at 10^3 cm^{-3} to 10^6 cm^{-3}



monodisperse size distribution

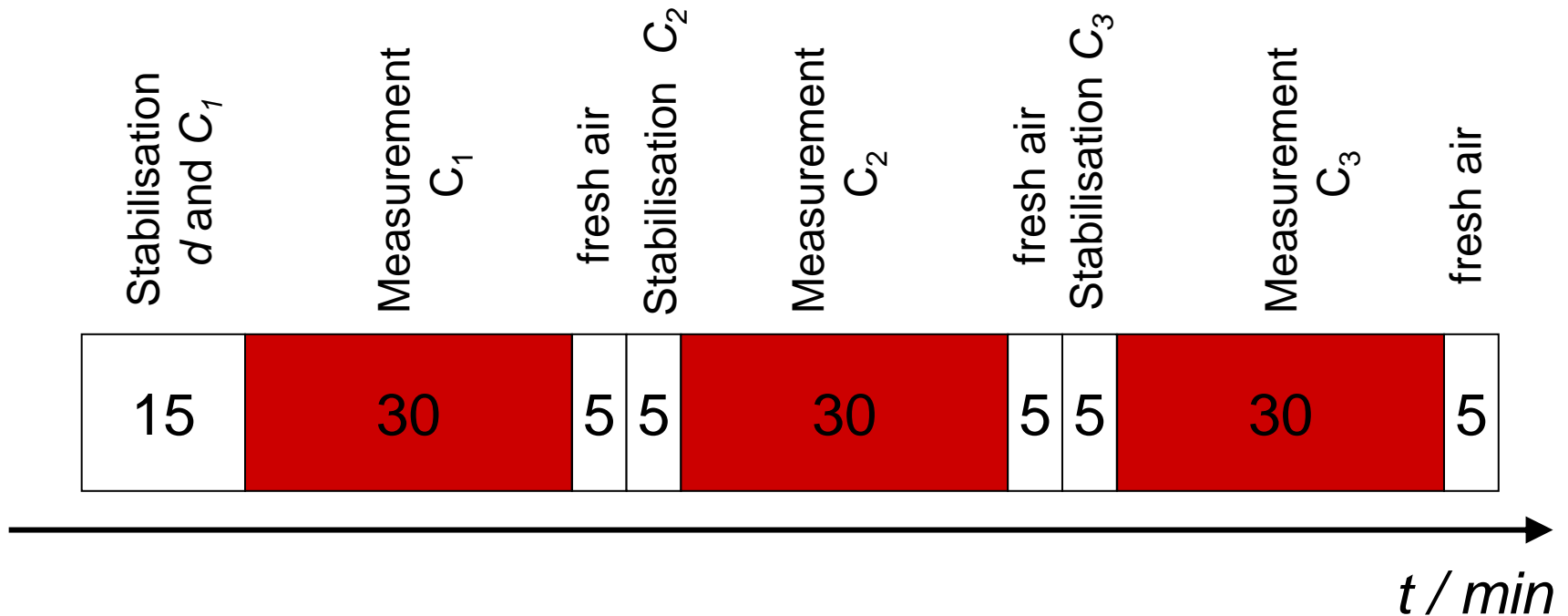
50 nm, 70 nm, and 180 nm at 10^3 cm^{-3} to 10^4 cm^{-3}





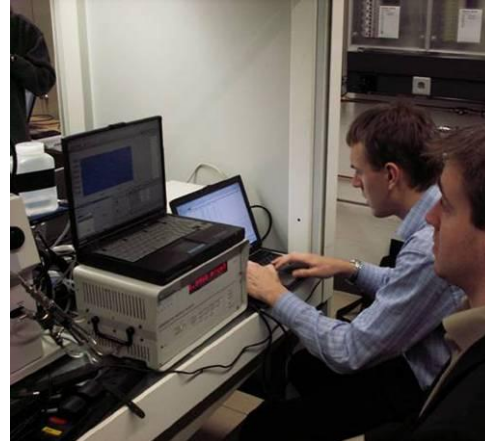
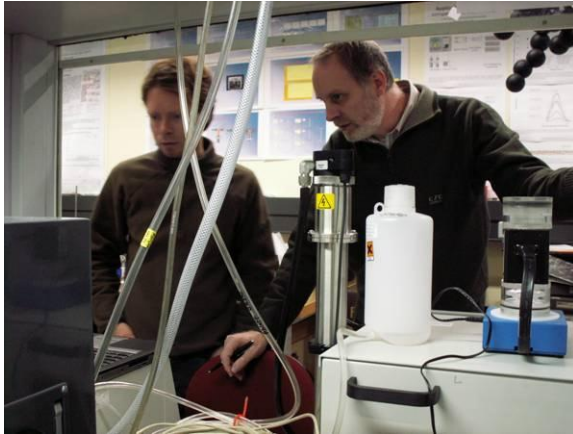
Experiment 4/5: Measurement cycles

Cycle per particle size:





Experiment 5/5: Discussions

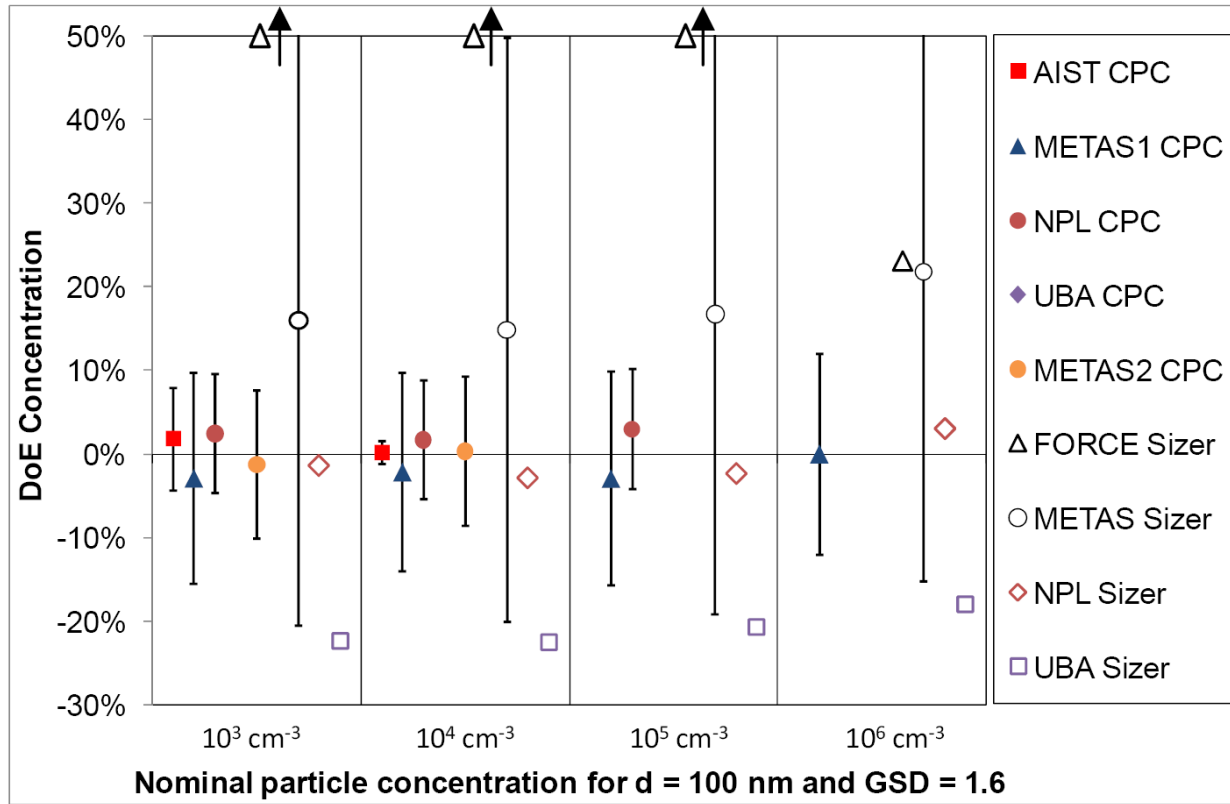


MANSA, 8th-9th June 2010
Jürg Schlatter



Results: Number concentration 1/2

"natural" size distribution: one size and various concentrations

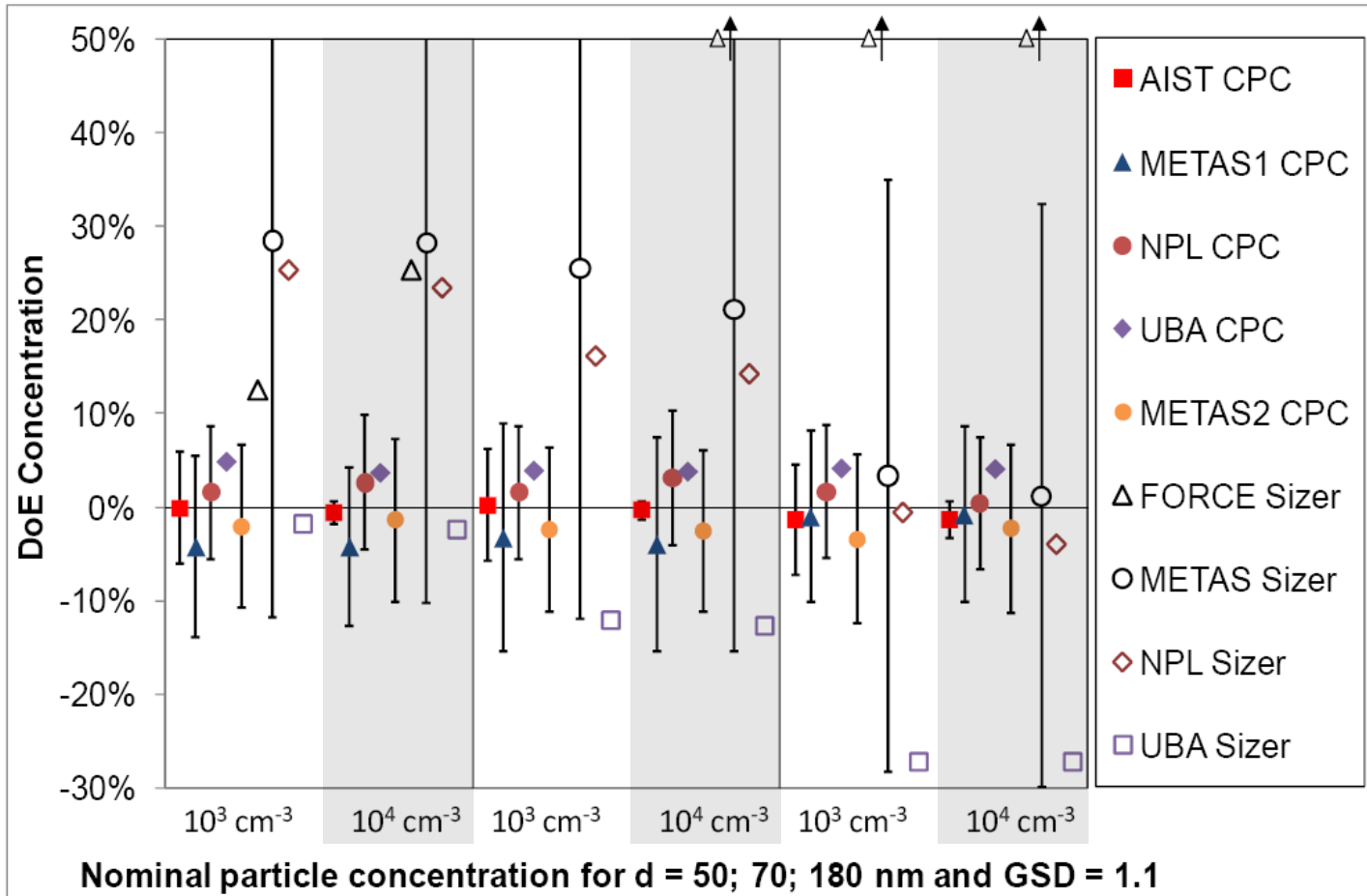


- Counters agree within 5 % (■ ▲ ● ●)
- Deviations for *mobility sizers* (SMPS by TSI) are larger (○ ◇ □)
- *aerodynamic sizer* (ELPI by DEKATI) not optimized for nanoparticles (△)



Results: Number concentration 2/2

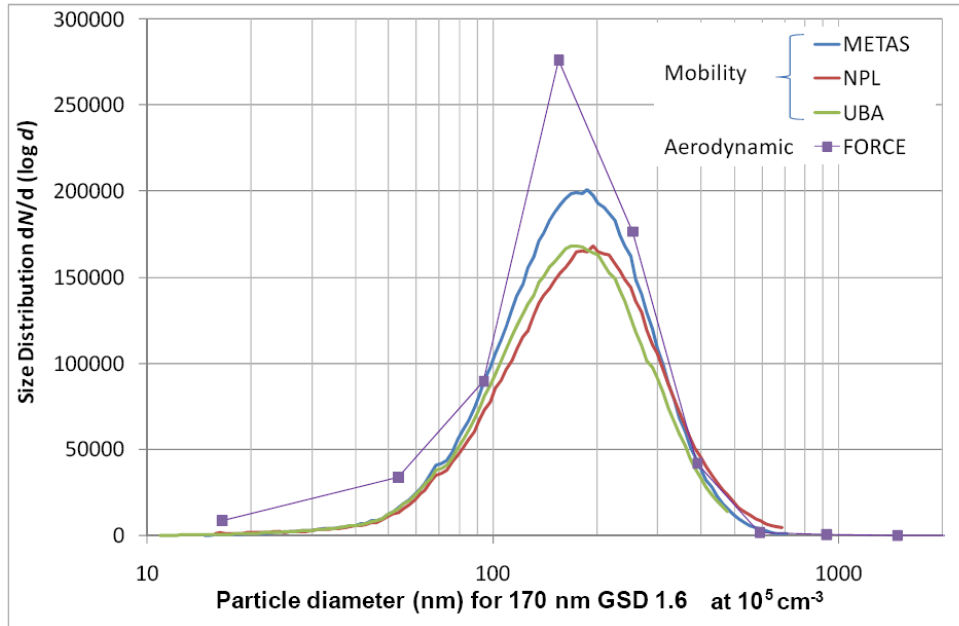
“monodisperse” size distribution: various sizes and concentrations



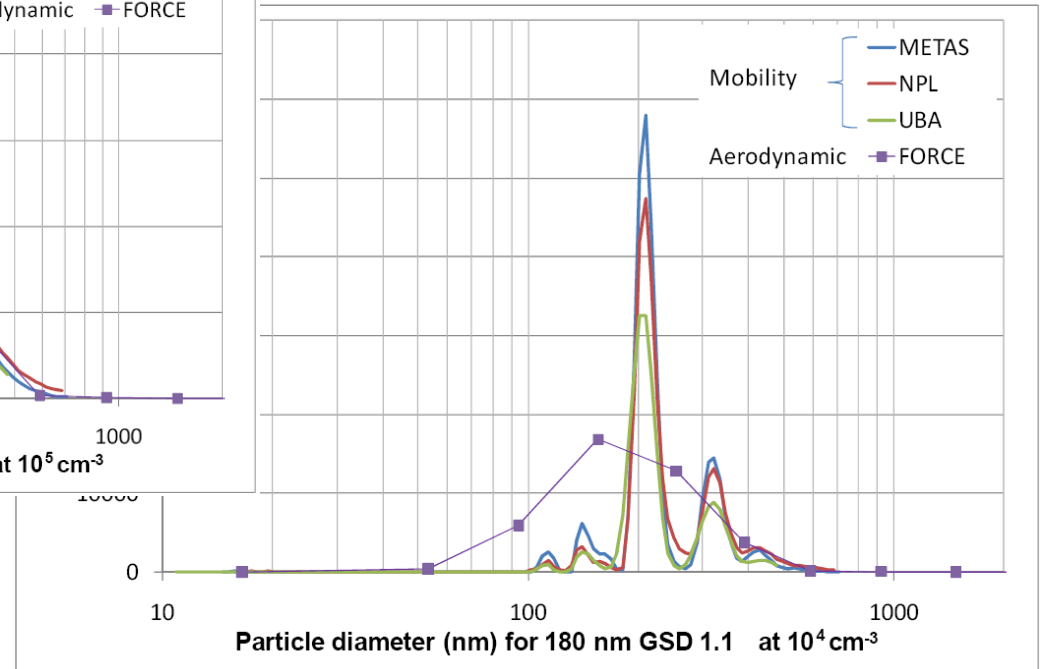


Results: Size distribution 1/3

“natural” size distribution at highest concentration



“monodisperse” size distribution at highest concentration

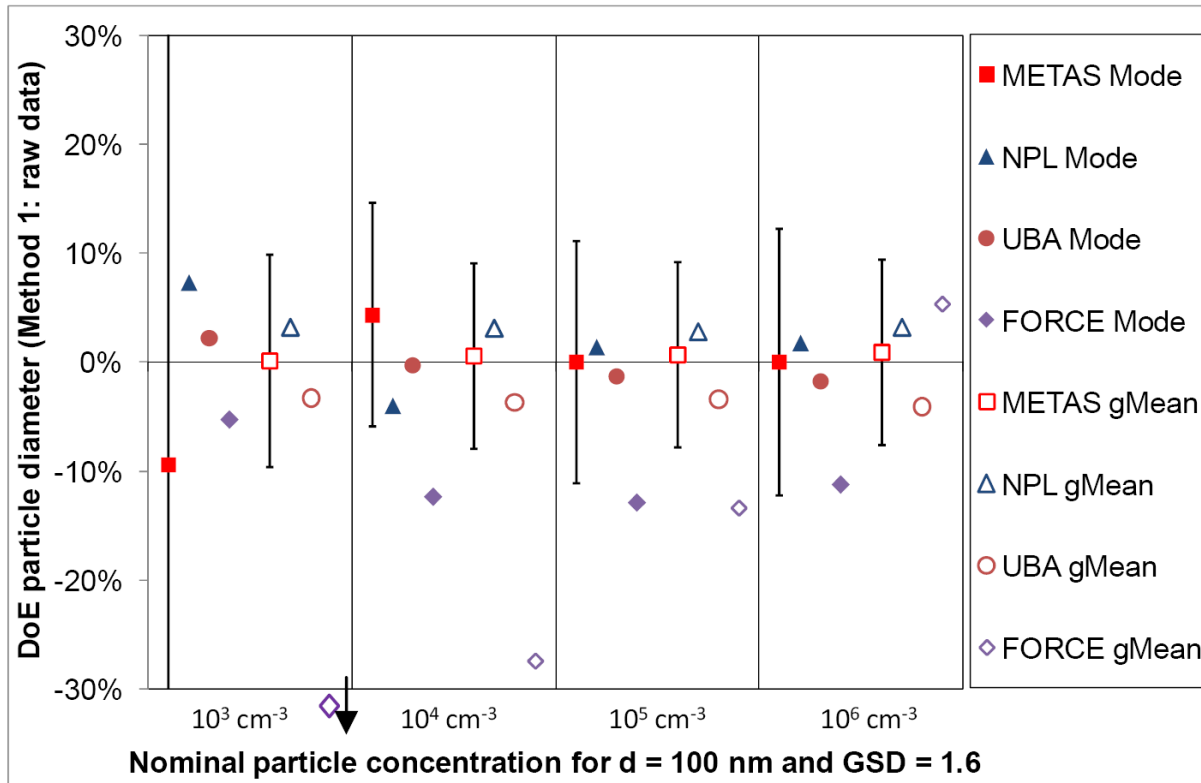


- Measuring principle defines the size resolution
- Higher concentrations give smoother size distributions



Results: Size geometric mean 2/3

“natural” size distribution: one size and various concentrations

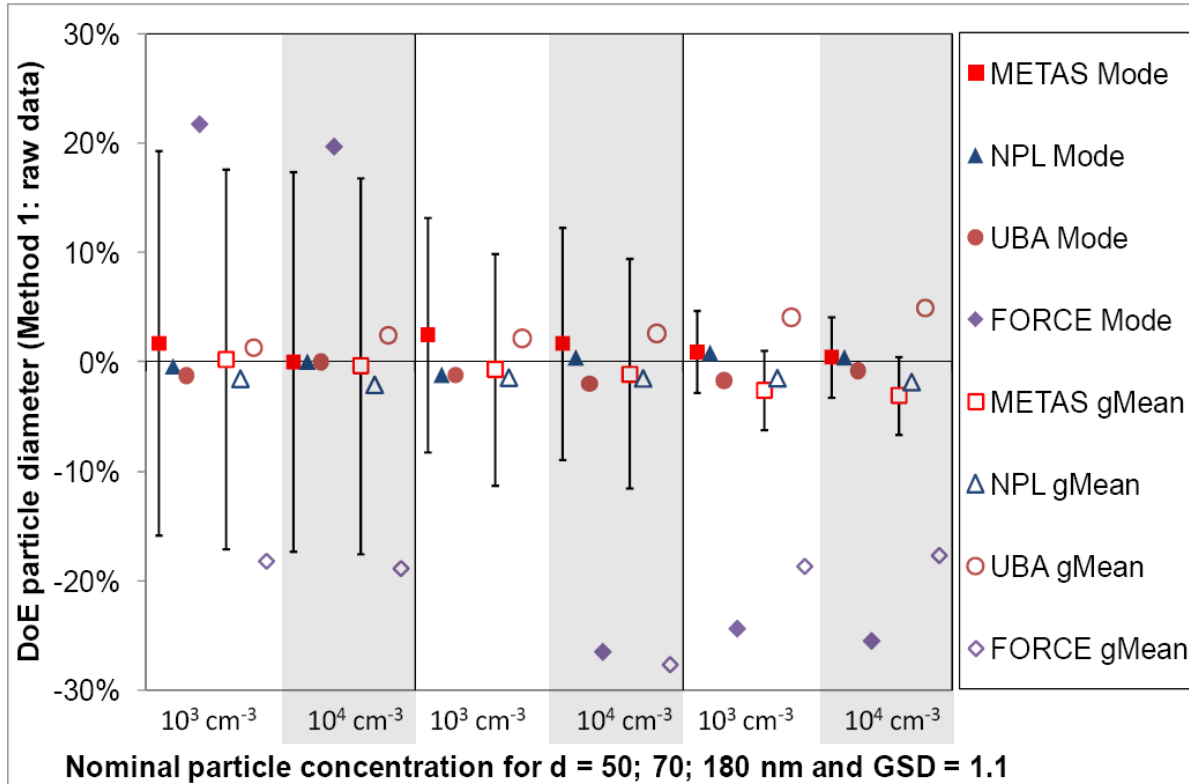


- Deviation of SMPS smaller than 5 %
- Diffusion loss correction proprietary by instrument manufacturer
- ELPI deviation caused by measuring principle (◇ ◆)



Results: Size geometric mean 3/3

“monodisperse” size distribution: various sizes and concentrations



- Deviation of SMPS smaller than 3 %
- Only a few uncertainty calculations
- ELPI deviation caused by measuring principle (◇ ◆)



Results: Other measures

Shape visualisation (AFM)



Other size information

- Geometric mean, mode, mean
- Geometric standard deviation
- Curve fitting



Conclusions

Number concentration for sizes 50 nm ... 180 nm:

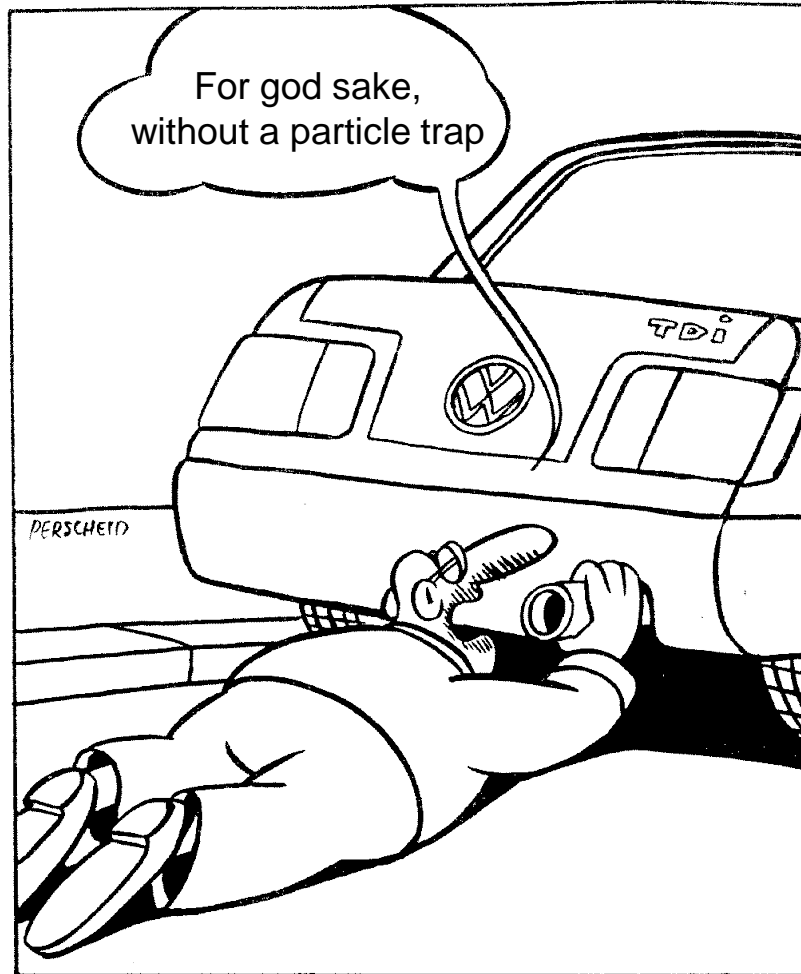
- Condensation particle counter better than particle sizers
- Deviation for CPCs smaller than 5 %

Particle size (geometric mean) for sizes 50 nm ... 180 nm:

- Identical instrument manufacturer = identical results?
- Deviation for SMPS smaller than 5 %
- Slightly lower deviations at higher concentrations
- Size correction due to diffusion losses of small particles shifts results by 10 % to smaller particles. This correction is proprietary to instrument manufacturer.



Future challenges 1/3



Martin quickly found an alternative to harmful cigarettes smoke



Future challenges 2/3

Uncertainty calculation

- Number concentration (especially for small diameters)
- Parameter of the size distribution: d_g , \hat{d} , σ_g
- Diffusion loss calculation
- Curve fitting (and extrapolation)

Expand measurement range

- Automotive regulation: 23 nm, 41 nm
- Aviation regulation: 20 to 30 nm
- Ambient air: selection of a (few) size classes (www.ufipolnet.eu)

Generation of a stable aerosol

- Real monodisperse aerosol (with known electrical charges)
- Thermally stable aerosol (automotive application of thermodiluter)
- Other particles: Oil droplets, PSL, NaCl, NH_4SO_4 , TiO_2
- “Ambient aerosol in the laboratory”



Future challenges 3/3

Requirements for future comparisons:

- Separate campaigns for number concentration and size distribution
- Various particle types
- Limited size range
- Comparison of mode of size distribution

Limitations:

- SMPS-back-transformation
- Integration of SMPS size distribution to number concentration



Thank you!

Report from EURAMET 1027 as pdf
on www.euramet.org >Projects >Project Database

Euramet 1027 interests:

