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Unveiling structure

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You can also ask ...

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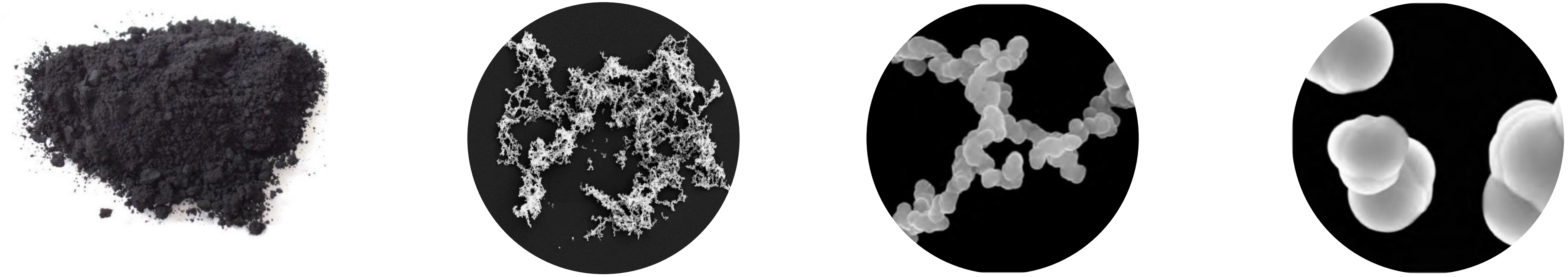
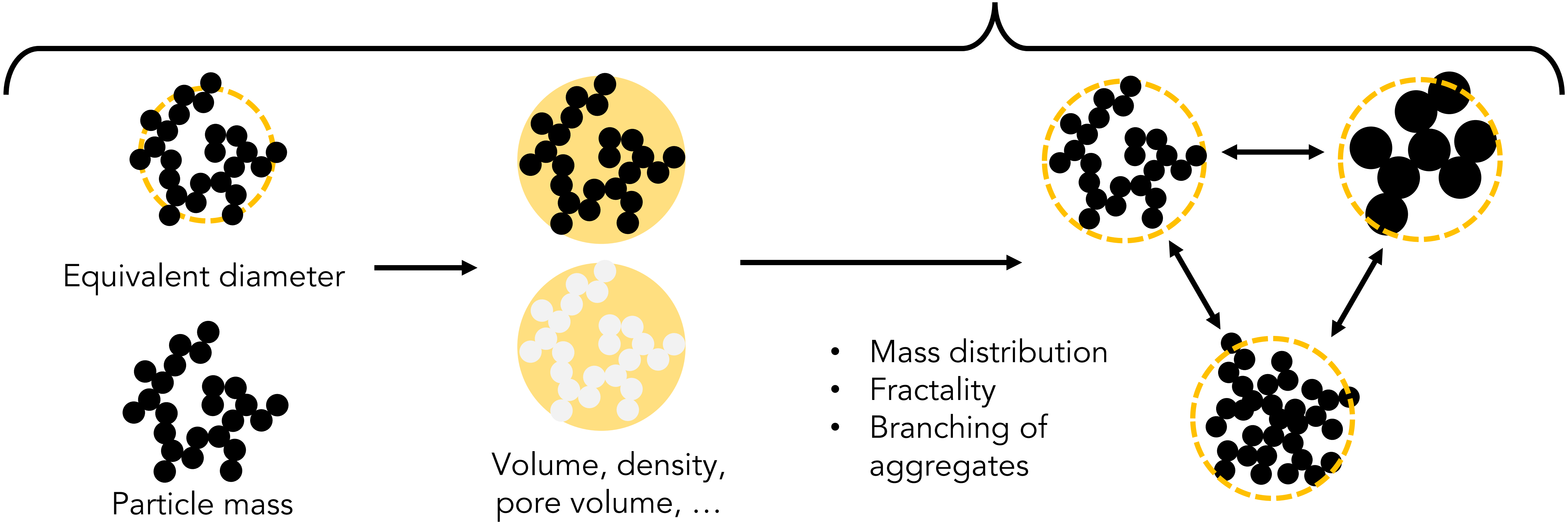


Powder | Agglomerates | Aggregates | Primary particles



Sample preparation

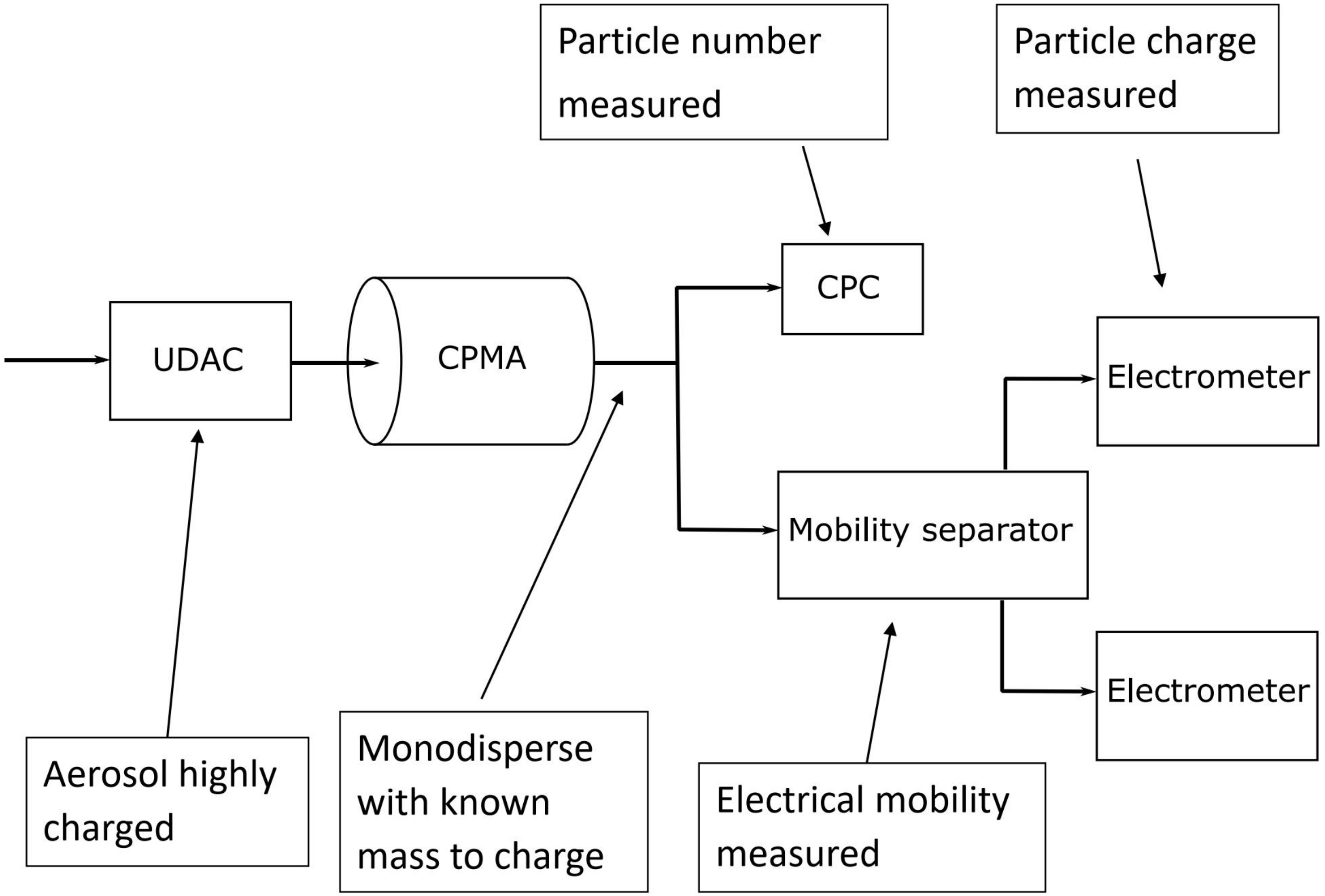
- **Dry dispersion**
  Powder → Aerosolization → Deagglomeration in Venturi nozzles

  Wet dispersion
  Aqueous suspension → 5min sonification 60W → Spray dispersion

  Direct sampling from reactor

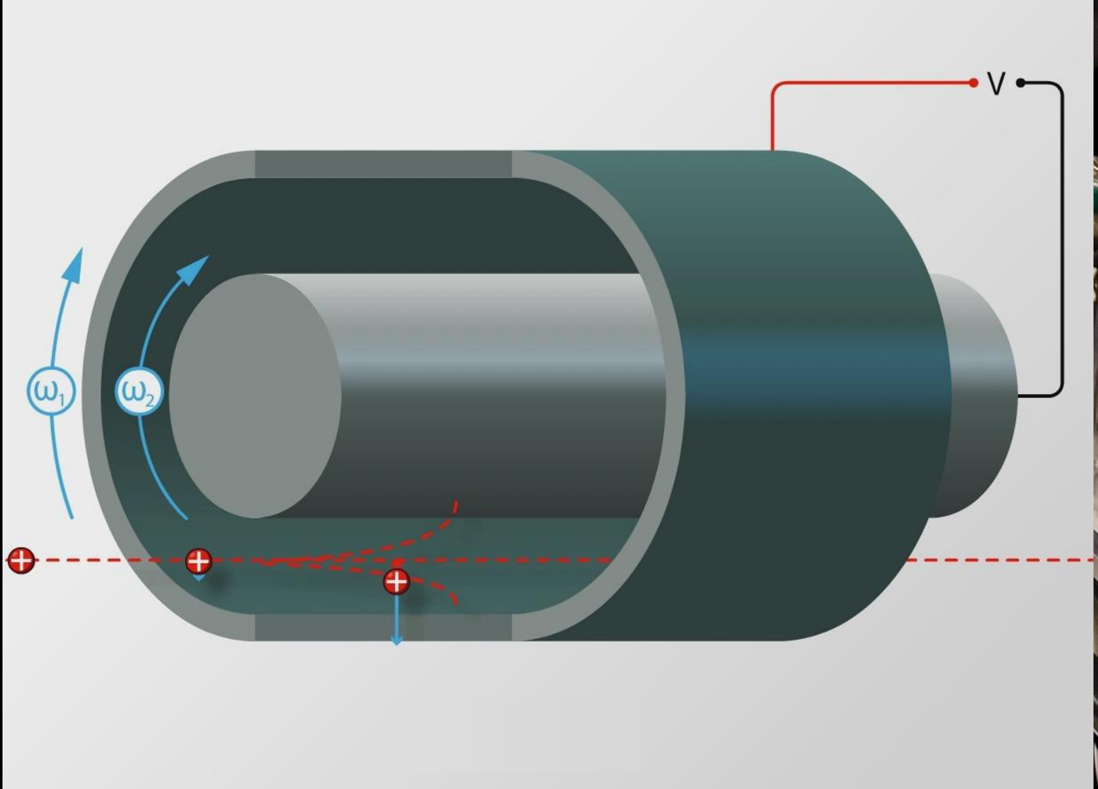
  Coming soon:
  Liquid dispersion in organic solvents

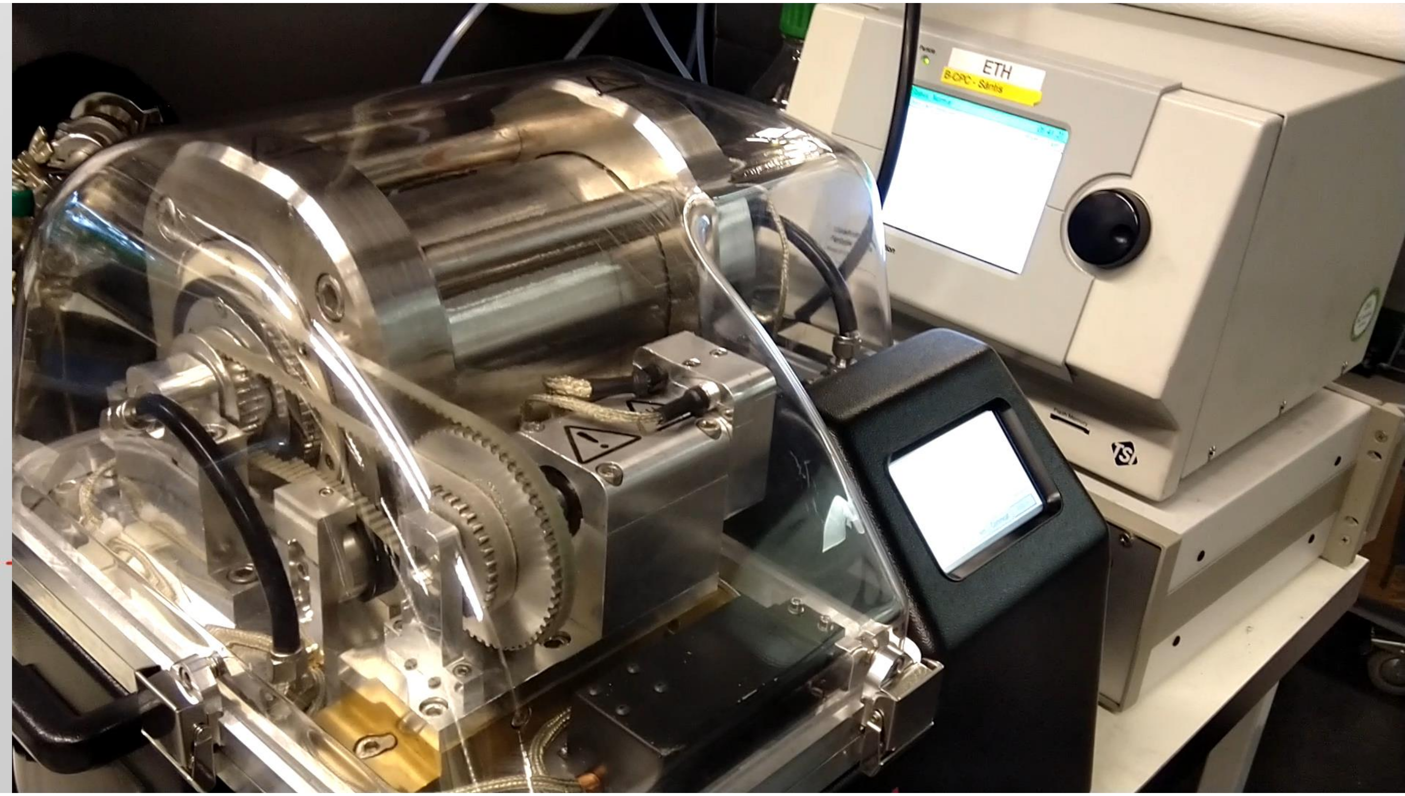
Full schematic: Mass & Mobility Aerosol Spectrometer (M^2AS)



CAMBUSTION

Centrifugal Particle Mass Analyzer



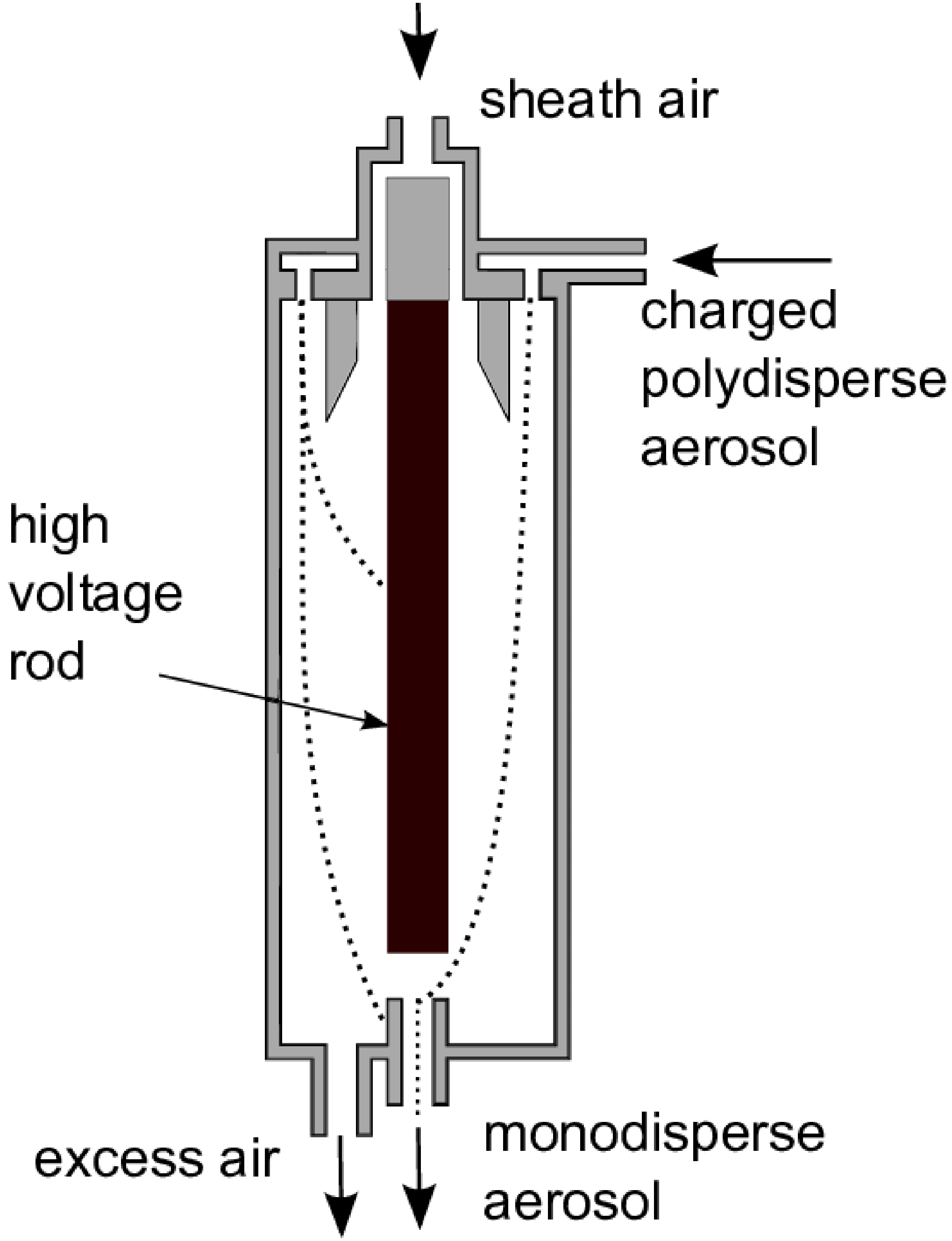


Centrifugal and electrostatic forces
→ selection of particles from 10^{-18} to 10^{-12} gram

Mobility diameter

- Drag-force in air
- Scales with geometric dimensions
- No preferred orientation
  - e.g. chains, needles, platelets
- **Independent of density**
- **Independent of material**

→ Conversion into aerodynamic diameter possible

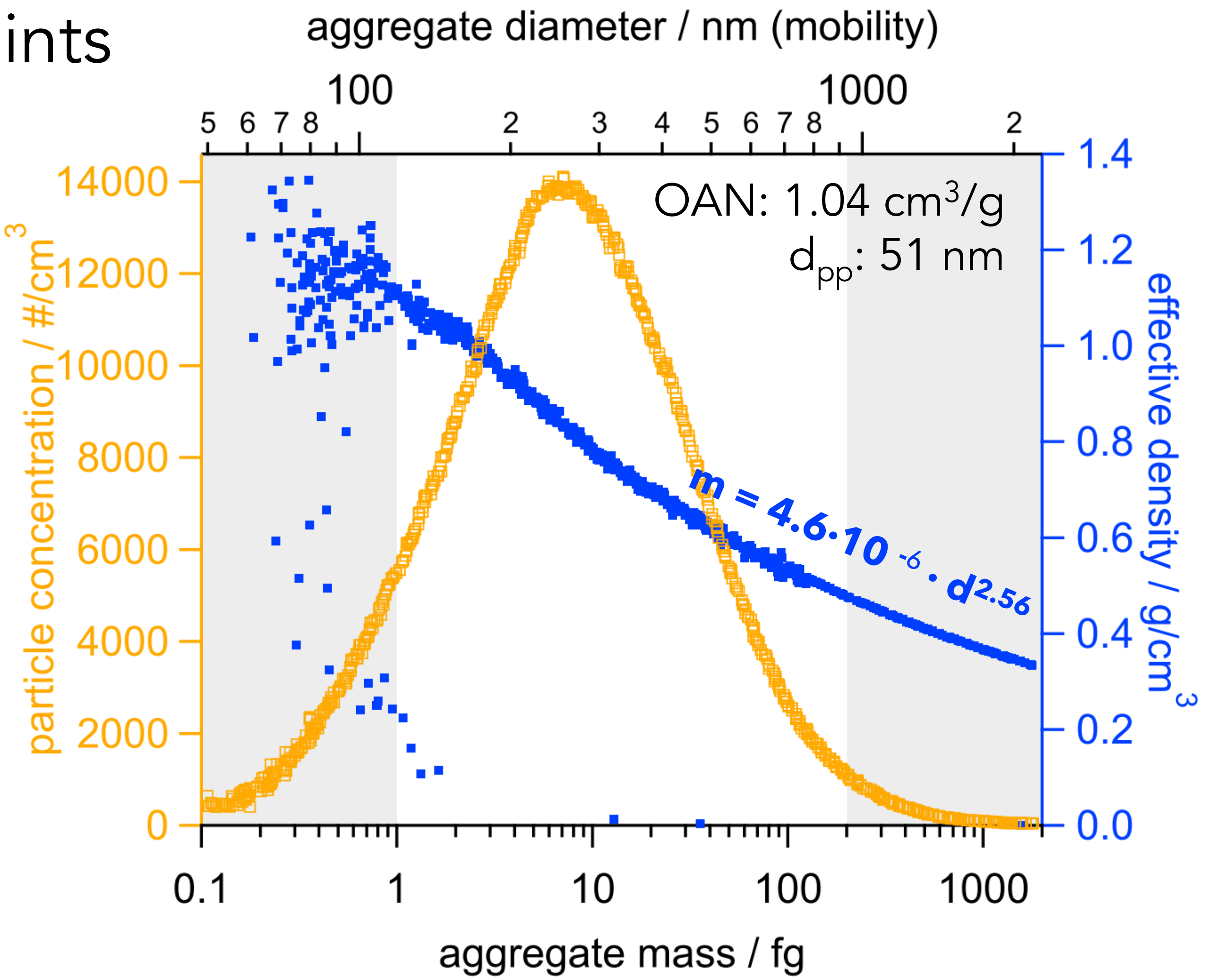


raw data – Low Colour Furnace Black

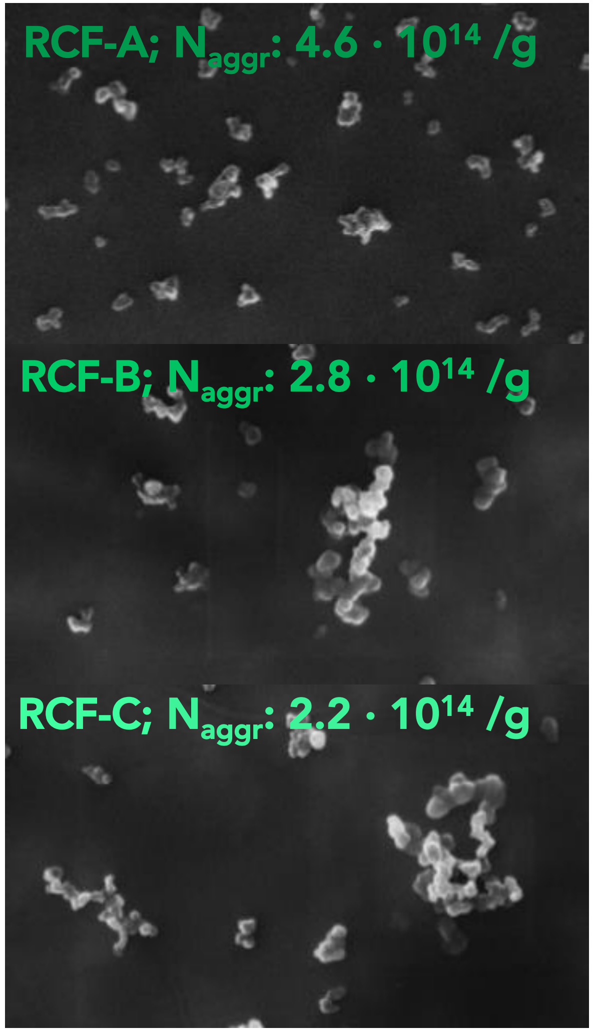
- 12.3 min per scan; 740 data points
  →**21 mio** particles counted

m_{50}: 6.2 fg
d_{50}: 240 nm
ρ_{50}: 0.86 g/cm^3

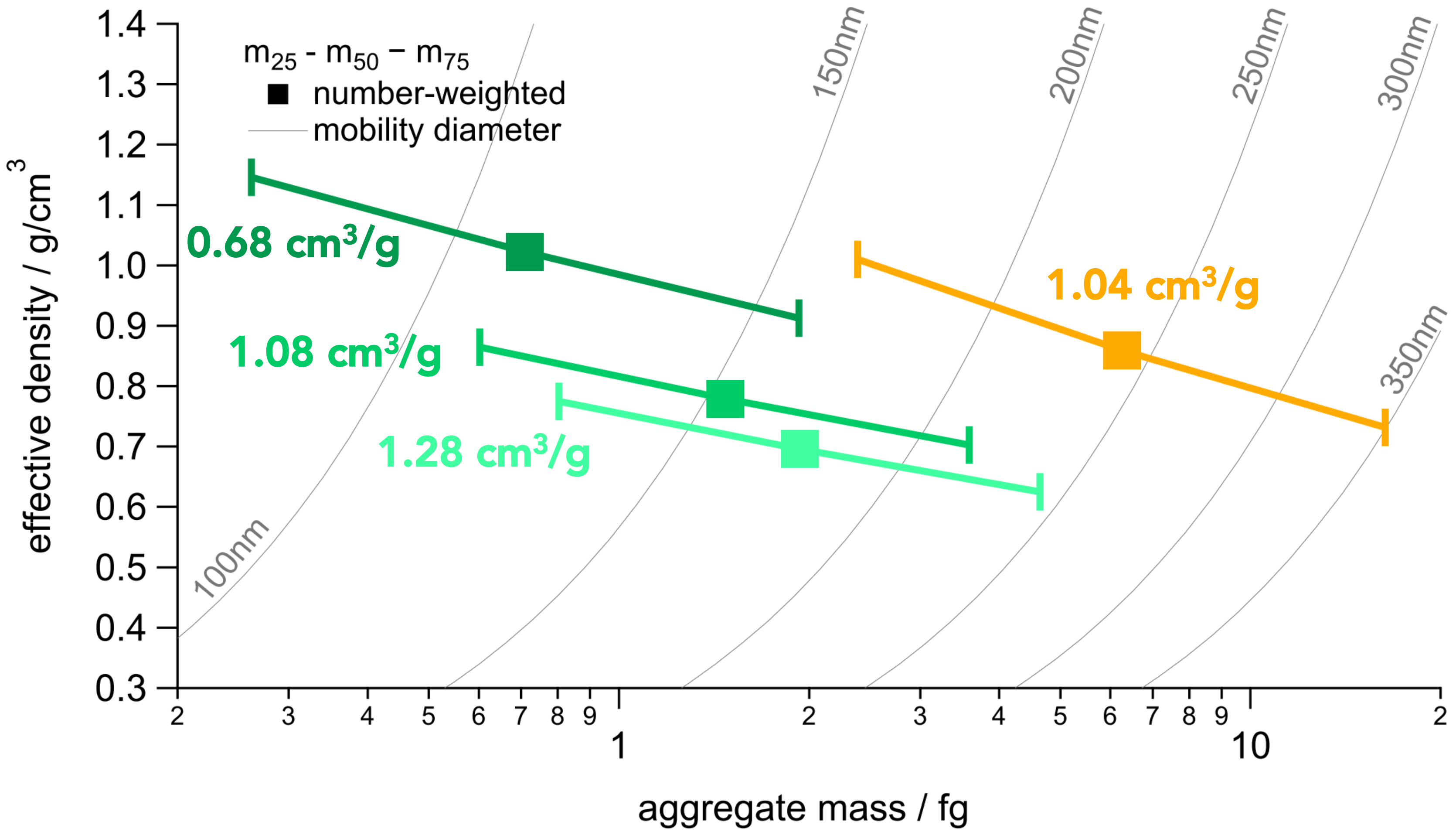
→ 1g of carbon black contains
$5 \cdot 10^{13}$ particles



Aggregation level vs PP-diameter



Regular Colour Furnace Black d_{PP}: 27nm
Low Colour Furnace Black d_{PP}: 51nm



And other materials

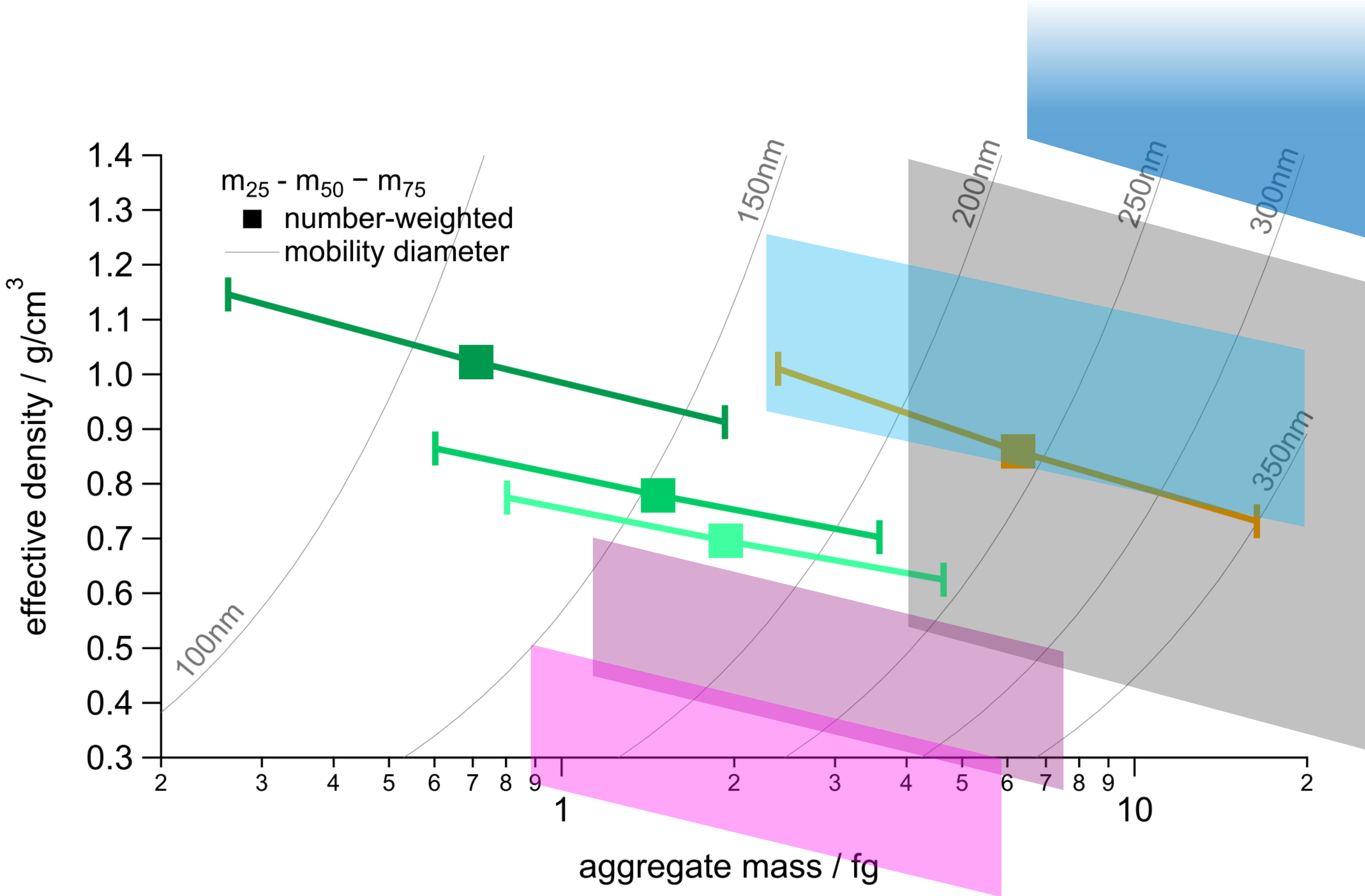
TiO_2 -pigment

TiO_2 -photocatalytic

Methane-pyrolysis

Precipitated silica

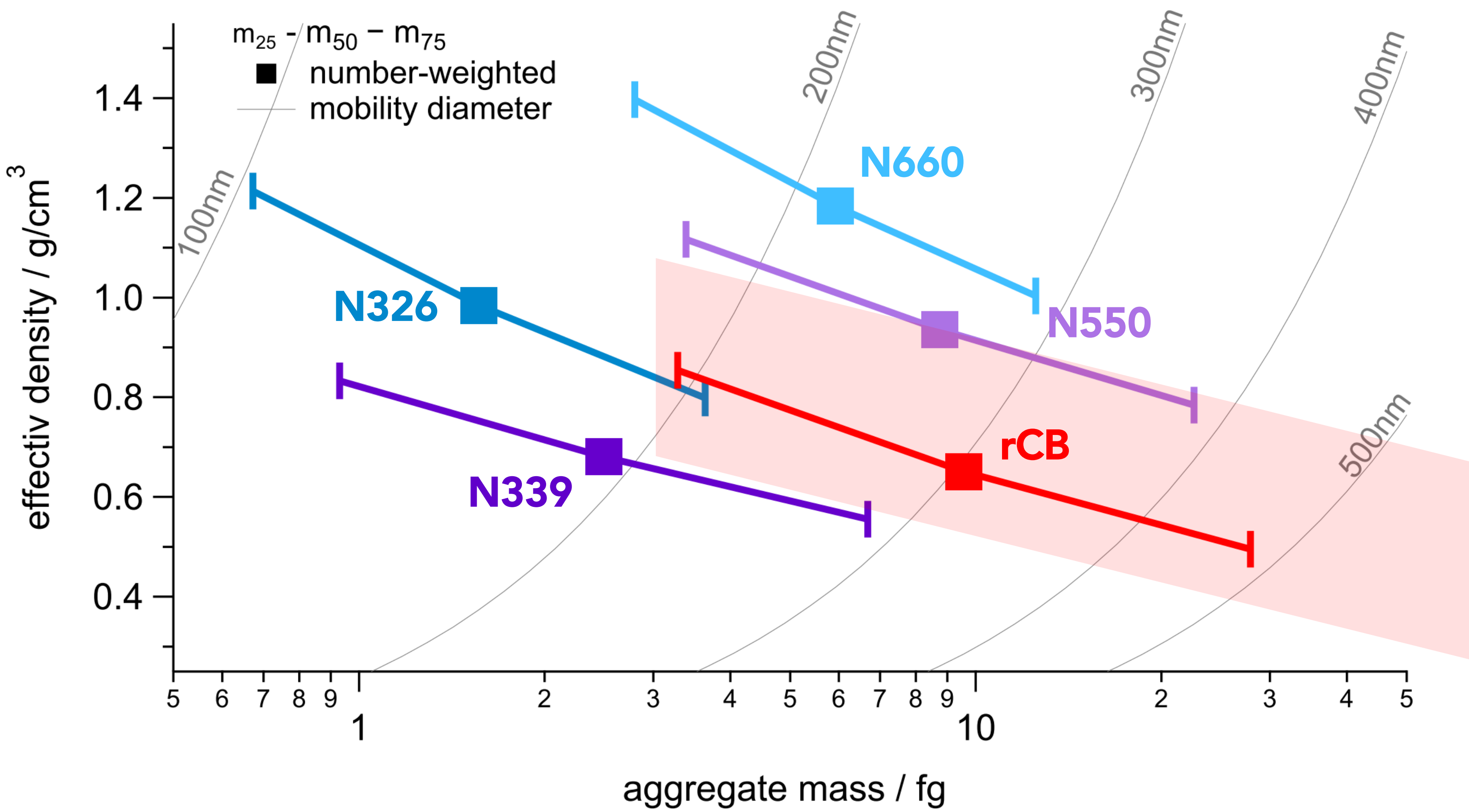
Pyrogenic silica



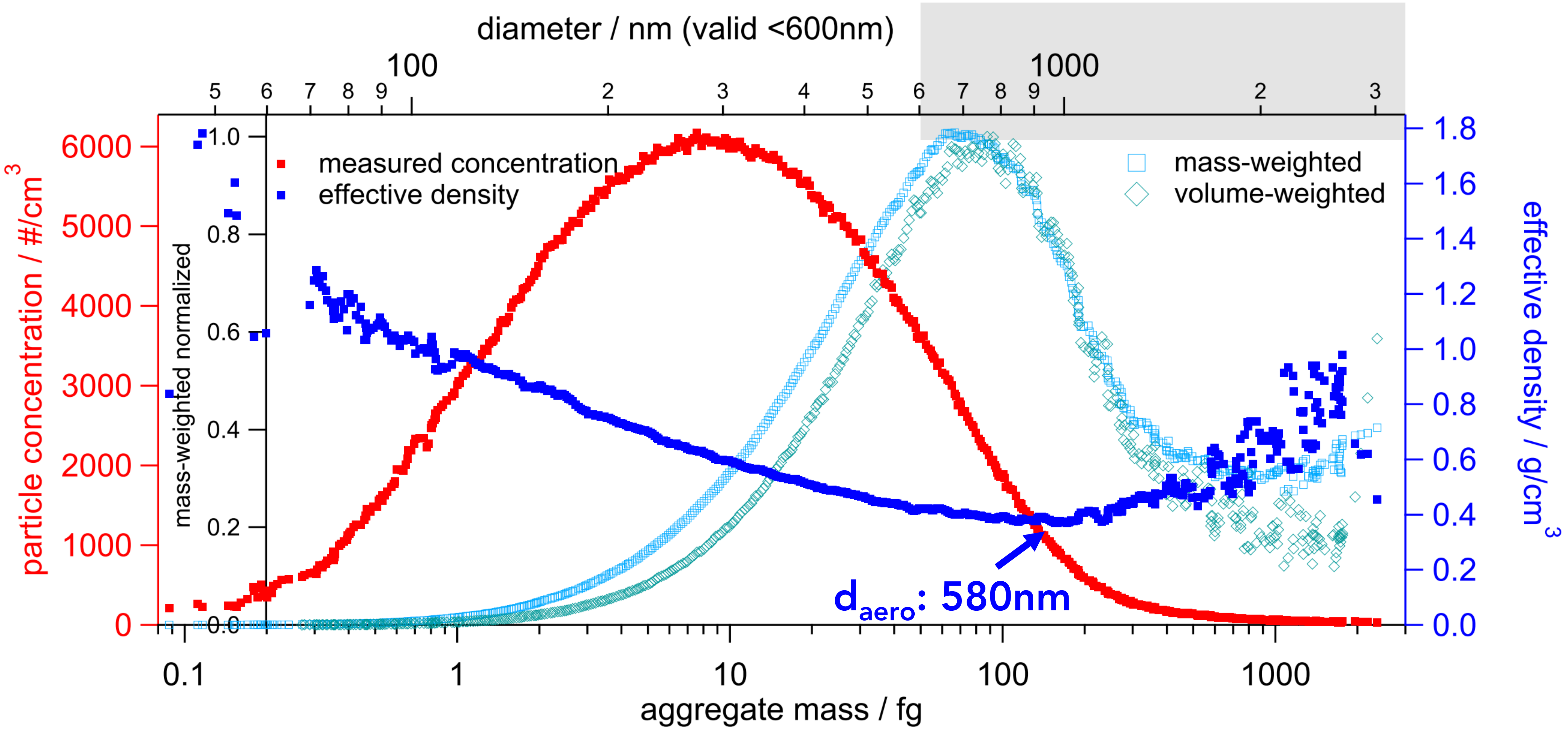
Tire-grade vs rCB

rCB is heavier and larger
than tire-grade
→ fused aggregates
→ dense mineral
components

	OAN cm^3/g	dia_{PP} nm
N660	0.90	67
N550	1.21	56
N339	1.20	26
N326	0.72	27

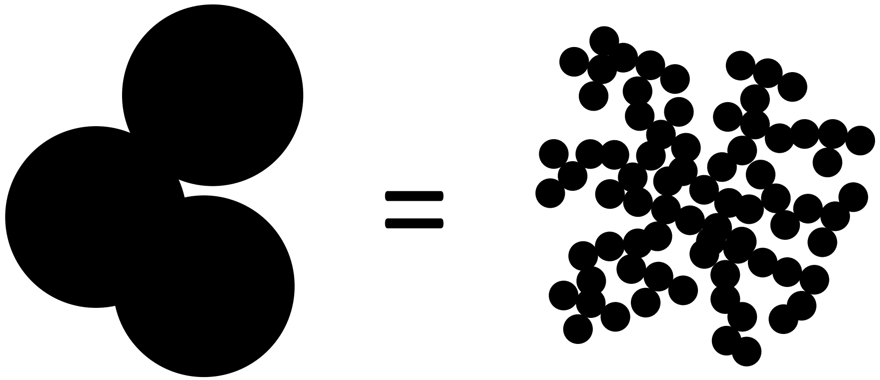
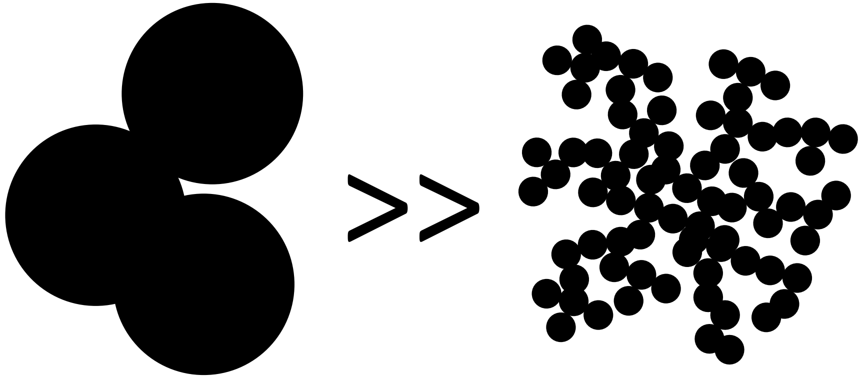


Raw data – rCB



diameter / nm (valid <600nm)
100
1000
5
6
7
8
9
2
3
4
particle concentration / #/cm^3
6000
5000
4000
3000
2000
1000
0
mass-weighted normalized
1.0
0.8
0.6
0.4
0.2
0.0
measured concentration
effective density
mass-weighted
volume-weighted
effective density / g/cm^3
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
d_{aero}: 580nm
0.1
1
10
100
1000
aggregate mass / fg

Diameter estimates

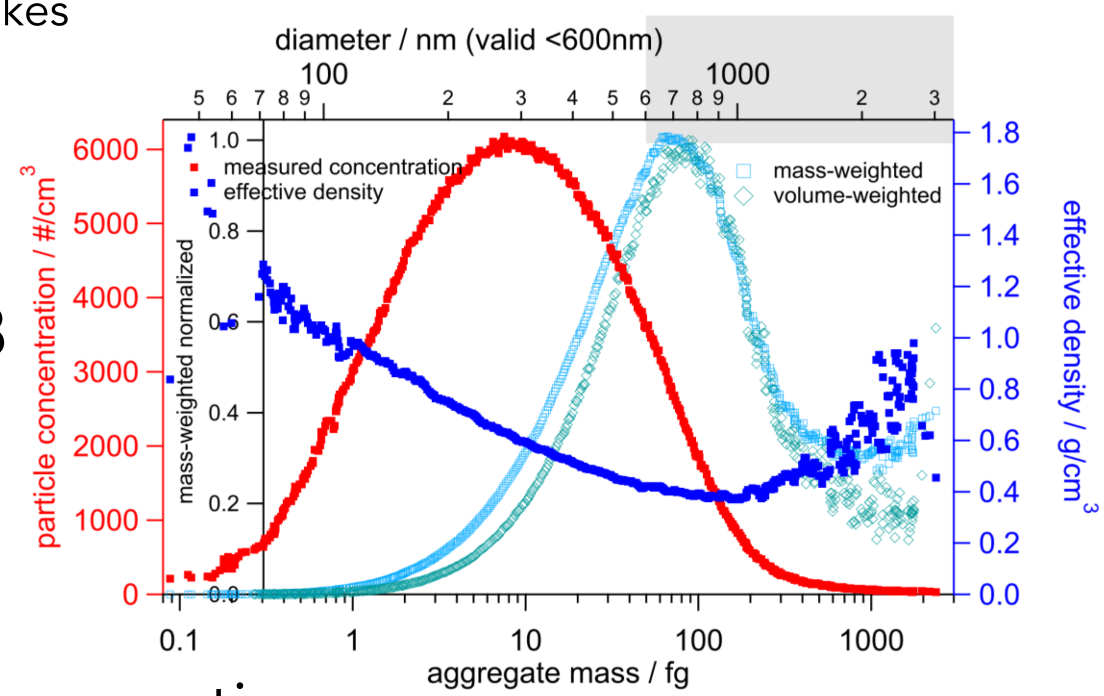
	Drag-force equivalent	Settling velocity equivalent
Air	d_{mob} Mobility diameter (Mass – mobilty aerosol spectrometer)	d_{aero} Aerodynamic diameter (calculated from mass + d_{mob})
Water	d_{hydro} Hydrodynamic diameter (Dynamic Light scattering)	d_{stokes} Stokes diameter (Centrifugal photosedimentation)
	diffusivity and mobility → geometry	geometry and **effective density**
observed diameter		

Diameter conversion
→ Centrifugal Photosedimentation

1. Fitting femtoG number-weighted mass-distribution
2. Extrapolation to mass-weighted distribution
3. Converting the d_{mob} into d_{hydro} and d_{stokes}

- $d_{hydro} \approx 1.1 \cdot d_{mob}$ (±20%)
- $d_{stokes} = \sqrt{\frac{\rho_{eff,hydro}}{\rho_{true}}} \cdot d_{hydro}$

- Correction factor for surfactant bias: 0.88
  - (reference N550, STSA: 39 m^2/g)
  - $\mathbf{d_{stokes} = \sqrt{\frac{\rho_{eff}}{\rho_{true}}} \cdot d_{mob} \cdot 0.953 \cdot 0.88}$



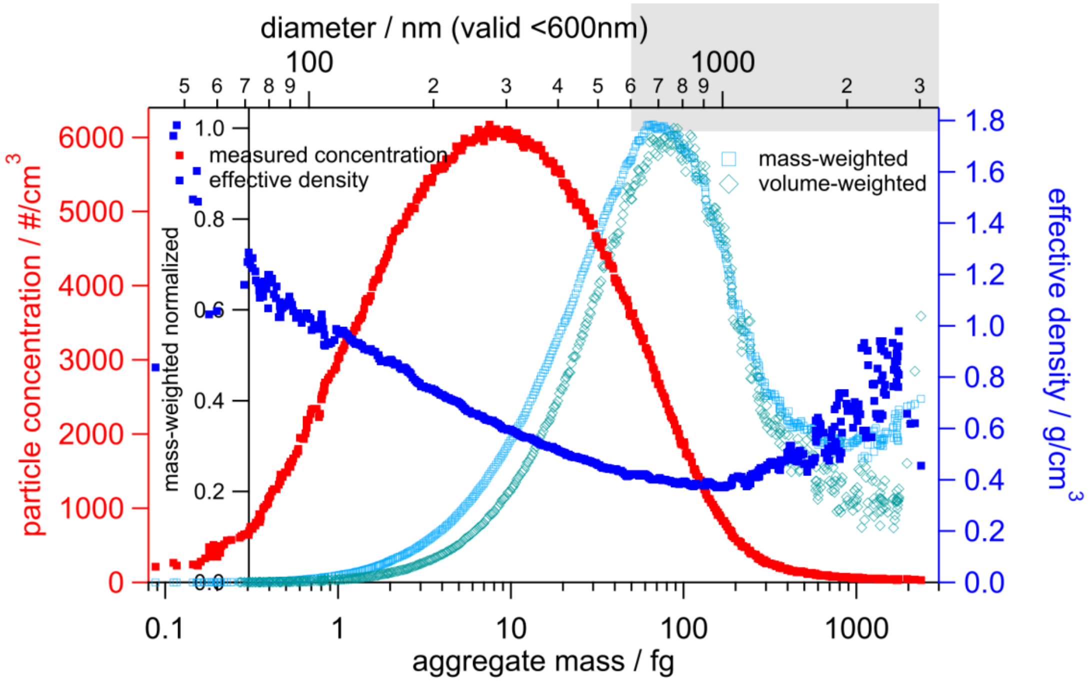
- No correction for optical detection / Mie correction

Diameter conversion
→ Laser diffraction

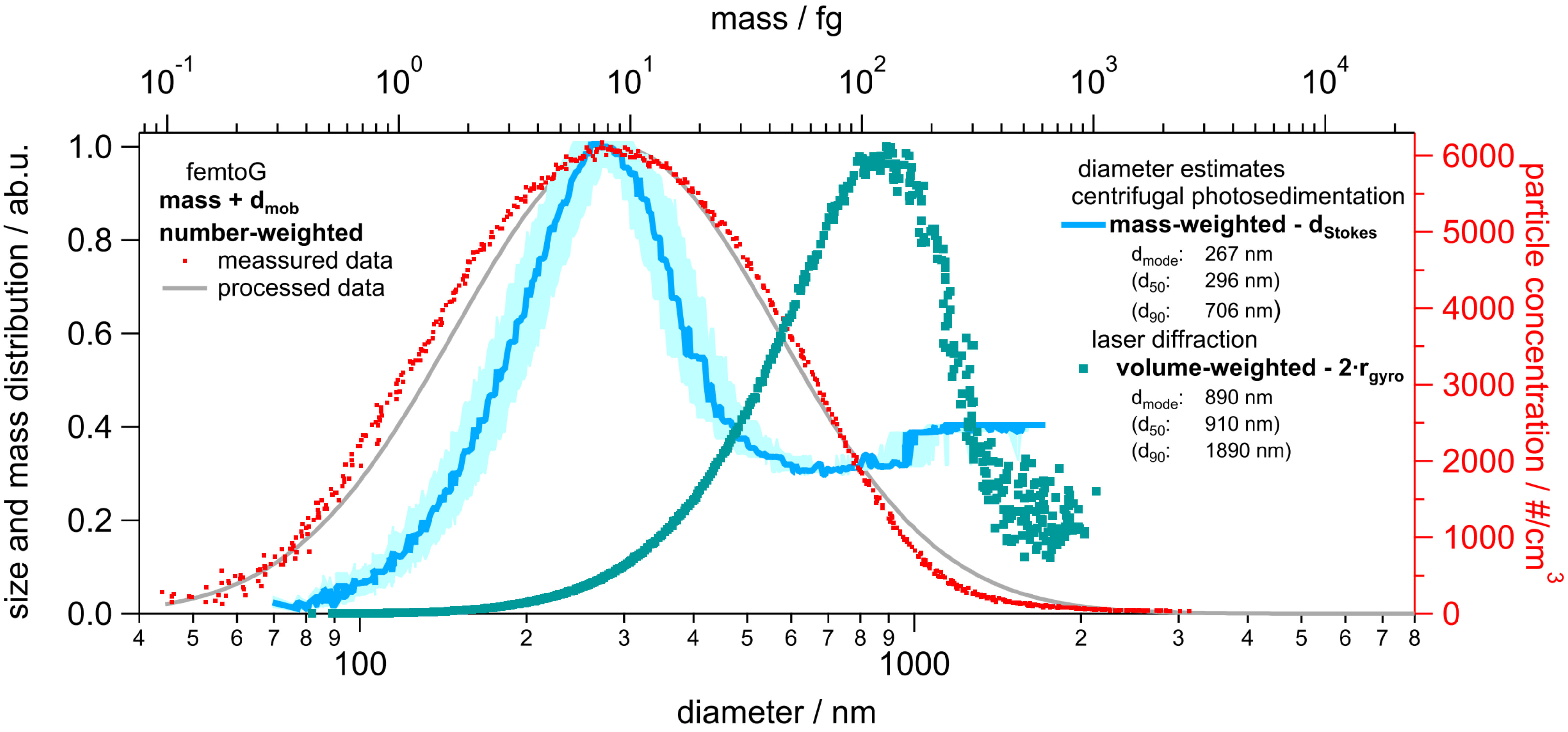
- Forward scattering depends on radius of gyration
- large scattering angles - complex interaction with rCB-aggregates
  → no reference diameter

→ volume-weigthed size distribution

→ $d_{mob} \approx 0.85 \cdot 2r_{gyro}$

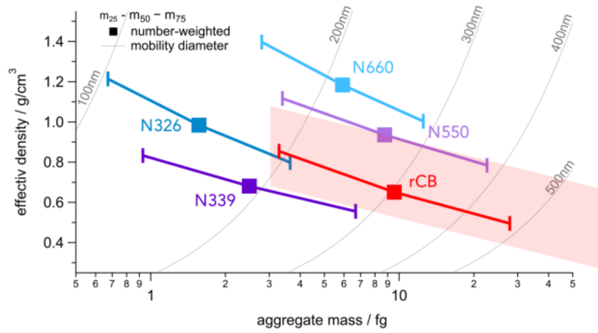


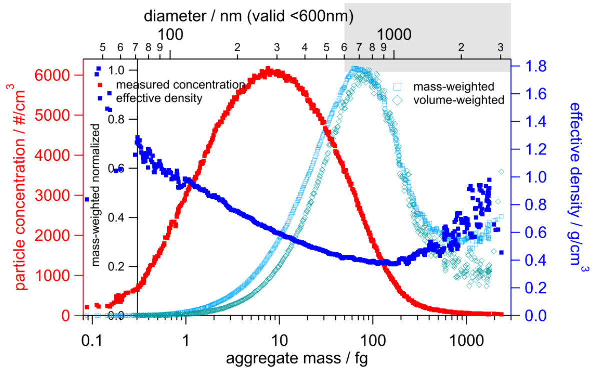
Comparison with other sizing methods



Mass vs. Diameter - A new approach for analysing particle size and structure

- Rapid analysis of vCB and rCB
  - 5-15 min time resolution
  - online product monitoring
- 1 g of vCB contains 10^{13} to 10^{15} aggregates
- rCB consists of two distinct particle populations





get the presentation →

https://femtog.com/assets/CarbonBlackSummit2023.pdf

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Unveiling structure

Fast and comprehensive vCB/rCB characterization

- Lab analysis
- Research projects
- Process monitoring
- Consulting

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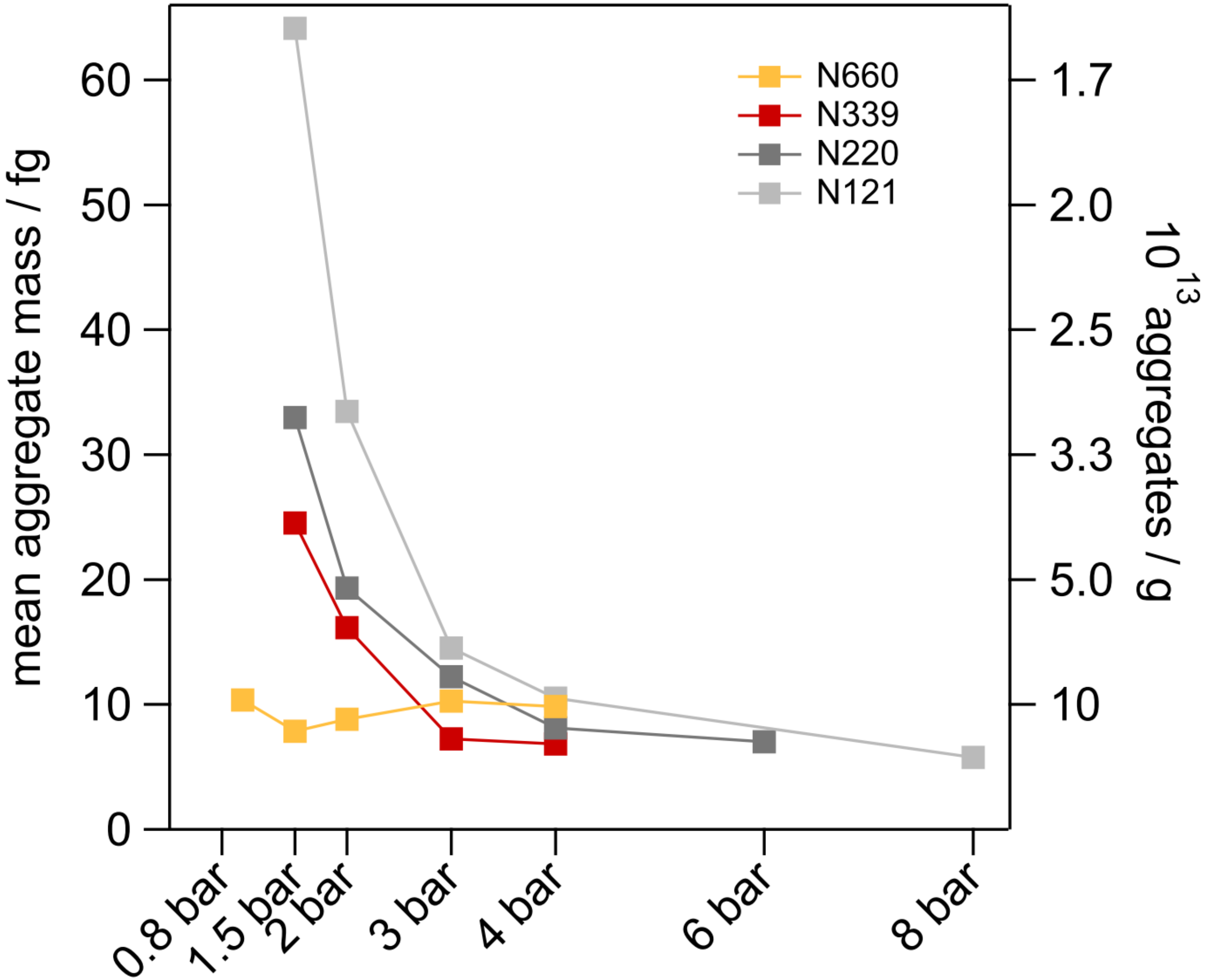
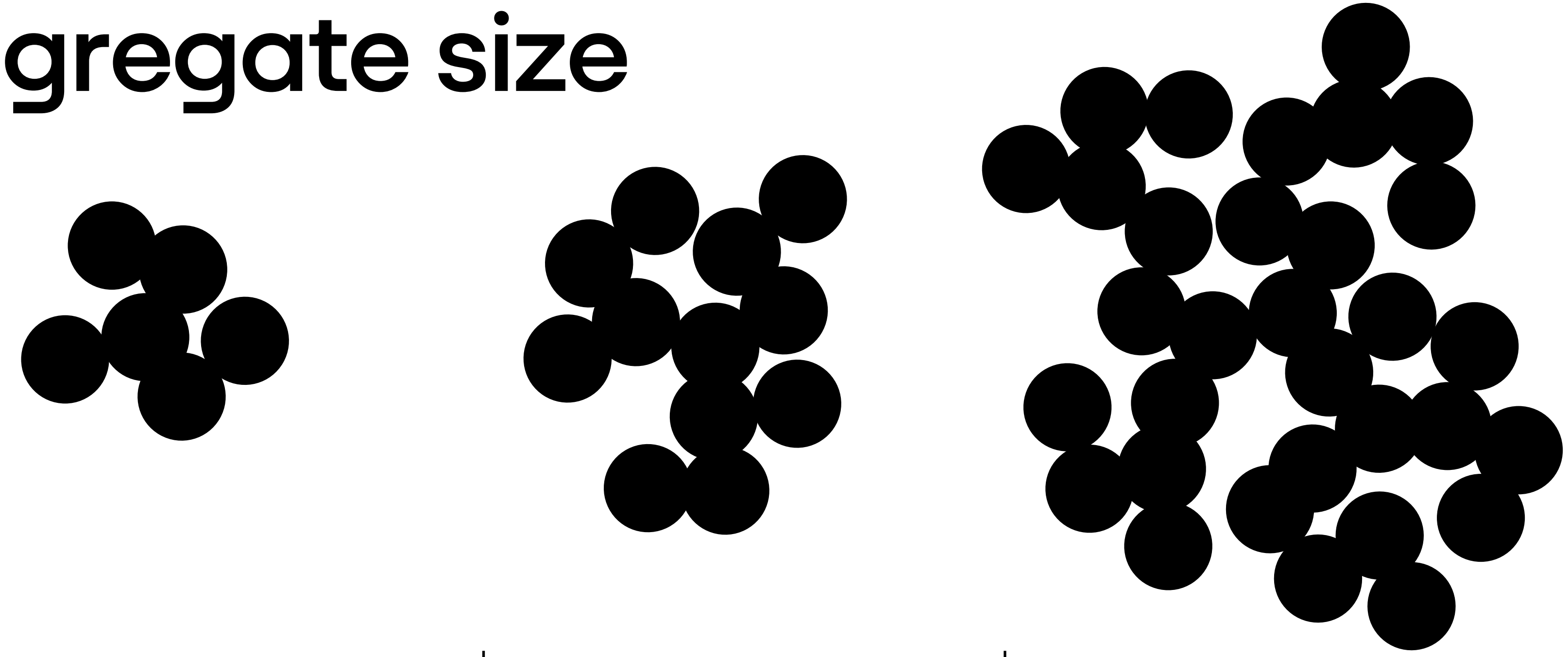
Deagglomeration efficiency

- test for agglomerate stability
  - work in progress
- number of bonds ≈ PP-number
  - change in mass
- pressure → shear



diffusion limited cluster aggregation theoretical aggregate size

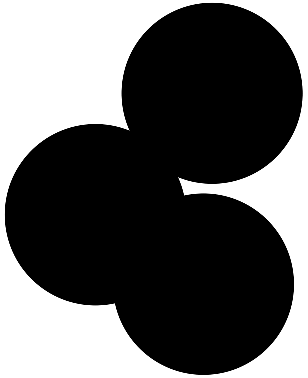
N330
d_{PP} : 30 nm; SSA = 78 m²/g
$d_{TEM,q0}$: 105 nm
$d_{TEM,q3}$: 225 nm
$d_{stokes,q3}$: 133 nm
(Donnet et al. 1993)

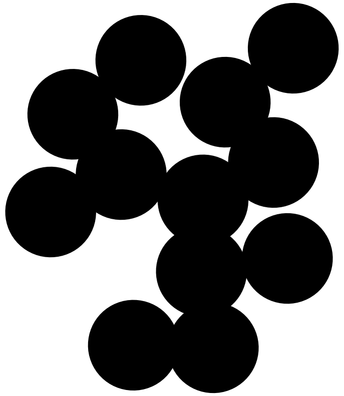


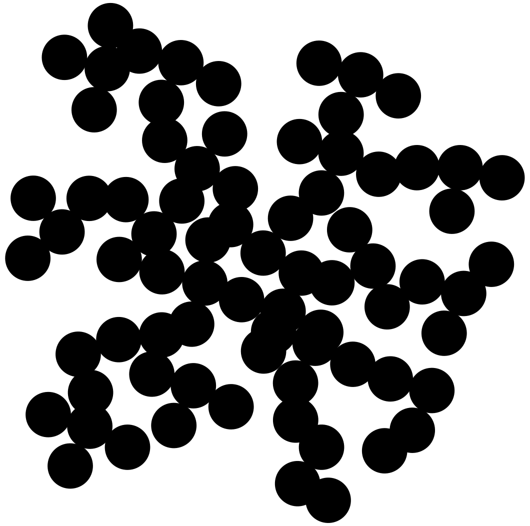
N_{PP}		40	400	4000
mass / fg	$\propto N_{PP}^{1}$	1.05	10.5	104.6
d_{mob} / nm (≈ 0.85·d_{gyro}; ≈ 0.9·d_{hydro})	$\propto N_{PP}^{0.5}$	164	559	2029
d_{stokes} / nm	$\propto N_{PP}^{0.24}$	75	129	222
d_{TEM} / nm (2D cross-section)	$\propto N_{PP}^{0.33}$	103	221	476

Constant aggregate mass / m= 1.04 fg based on DLCA theory

grades:
N6xx
N330
N1xx







d_{PP} / nm	60	30	15
SSA / m^2/g	54	108	216
N_{PP}	5.00	40	320
d_{mob} / nm ($\approx 0.85 \cdot d_{gyro}$; $\approx 0.9 \cdot d_{hydro}$)	**126**	**164**	**247**
d_{stokes} / nm	**92**	**75**	**61**
d_{TEM} / nm (2D cross-section)	103	103	103