Mass vs. Diameter
Rapid analysis of the particle size and structure.

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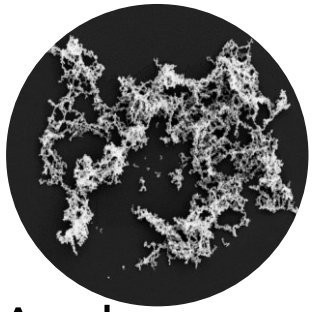
Unveiling structure

What is a particle? → Sample preparation!

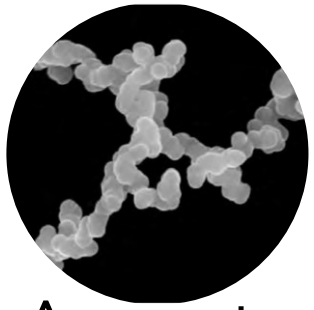
The preparation of a sample material prior to any particle size analysis defines which structural level will be detected.



Agglomerates

Dry dispersion

Powder → Aerosolization → Deagglomeration in Venturi nozzles



Aggregates

Wet dispersion

Suspension → sonification 10 – 300 J/ml → Spray dispersion

working fluids: water, ethanol, acetone, hexane

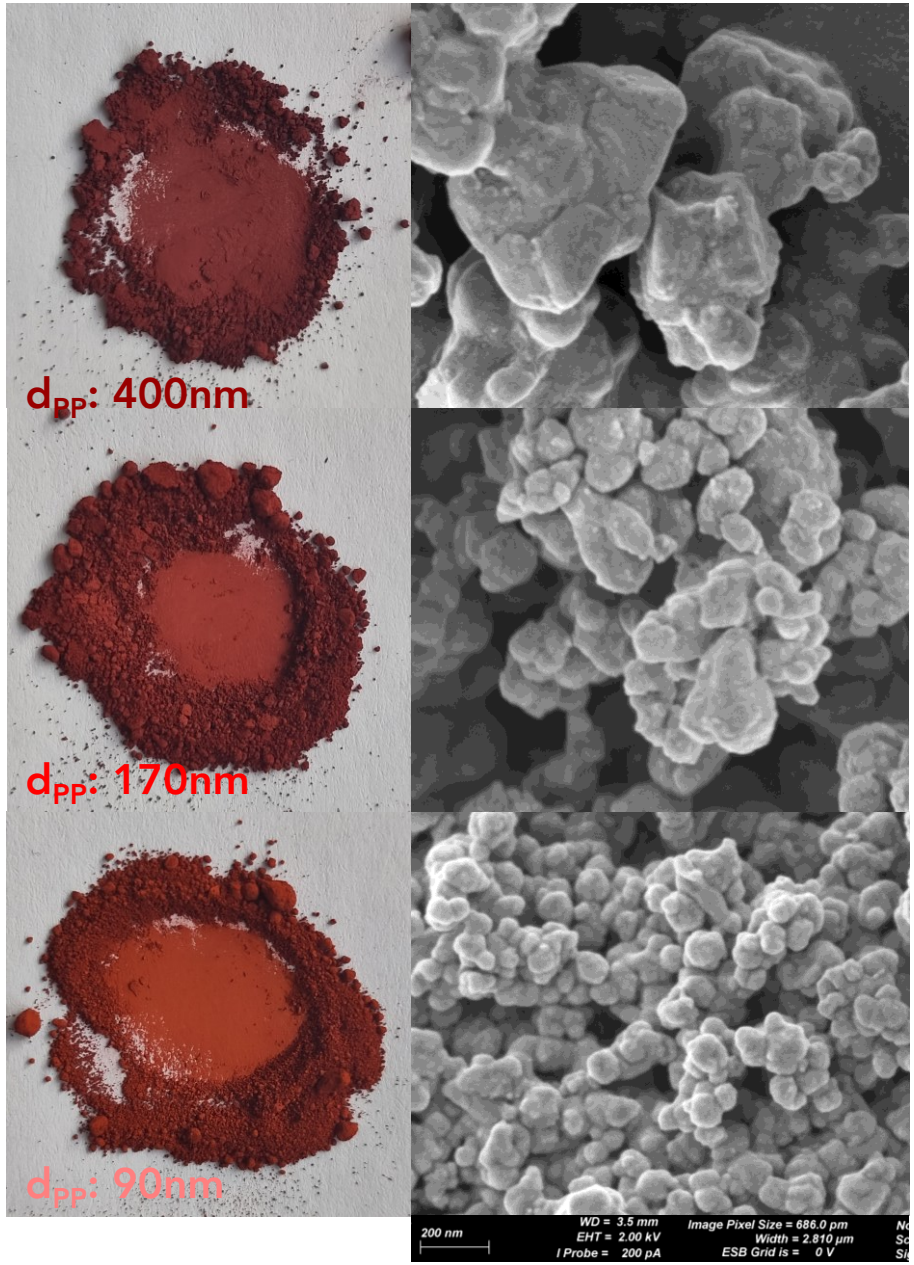
Direct sampling from reactor

Primary particles

What is the size of a particle?

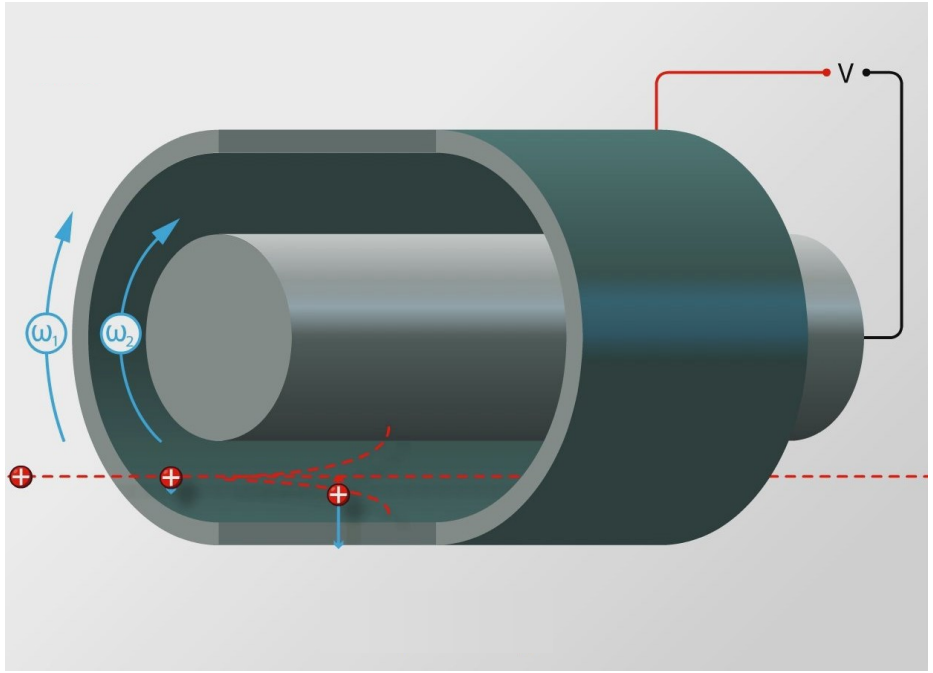
- Commercially available iron red pigment
  - Fe_2O_3 / Magnetite
- „Spheres with different diameters“, according to manufacturer

- Different colour / refractive indices
  → Optical sizing methods diffraction?
- material density vs. effective particle density
  → Sedimentation analysis?



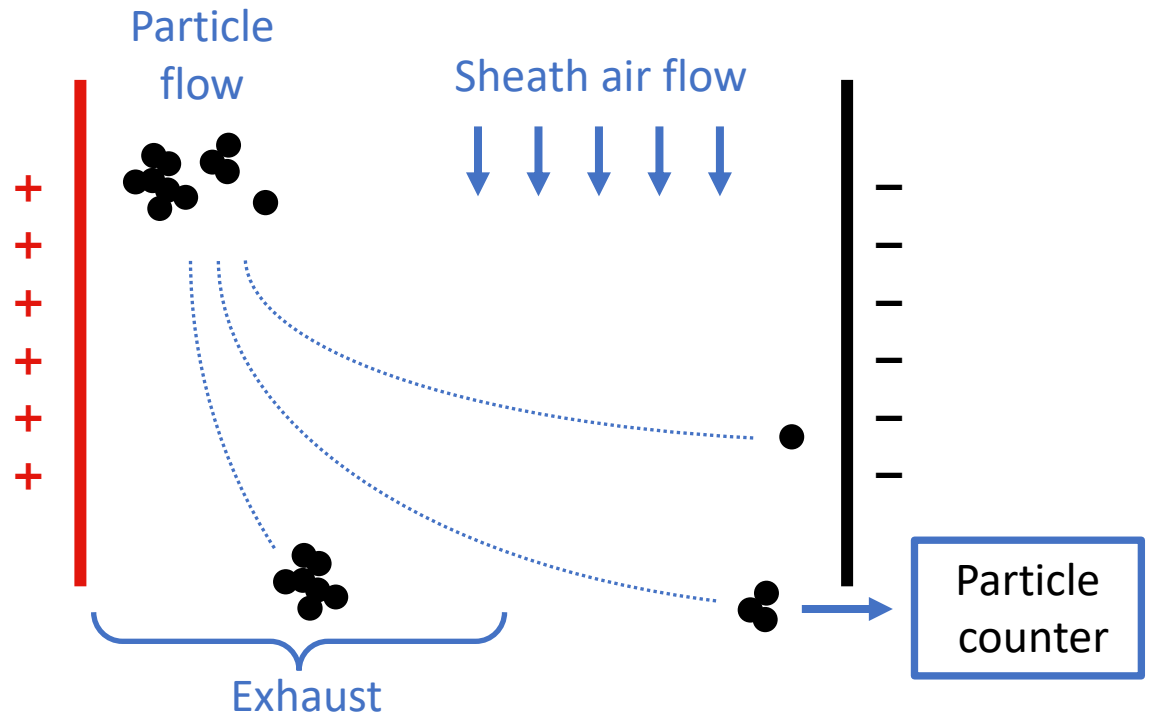
Simultaneous analysis of mass and diameter

Centrifugal Particle Mass Analyzer



Centrifugal force vs electrostatic forces

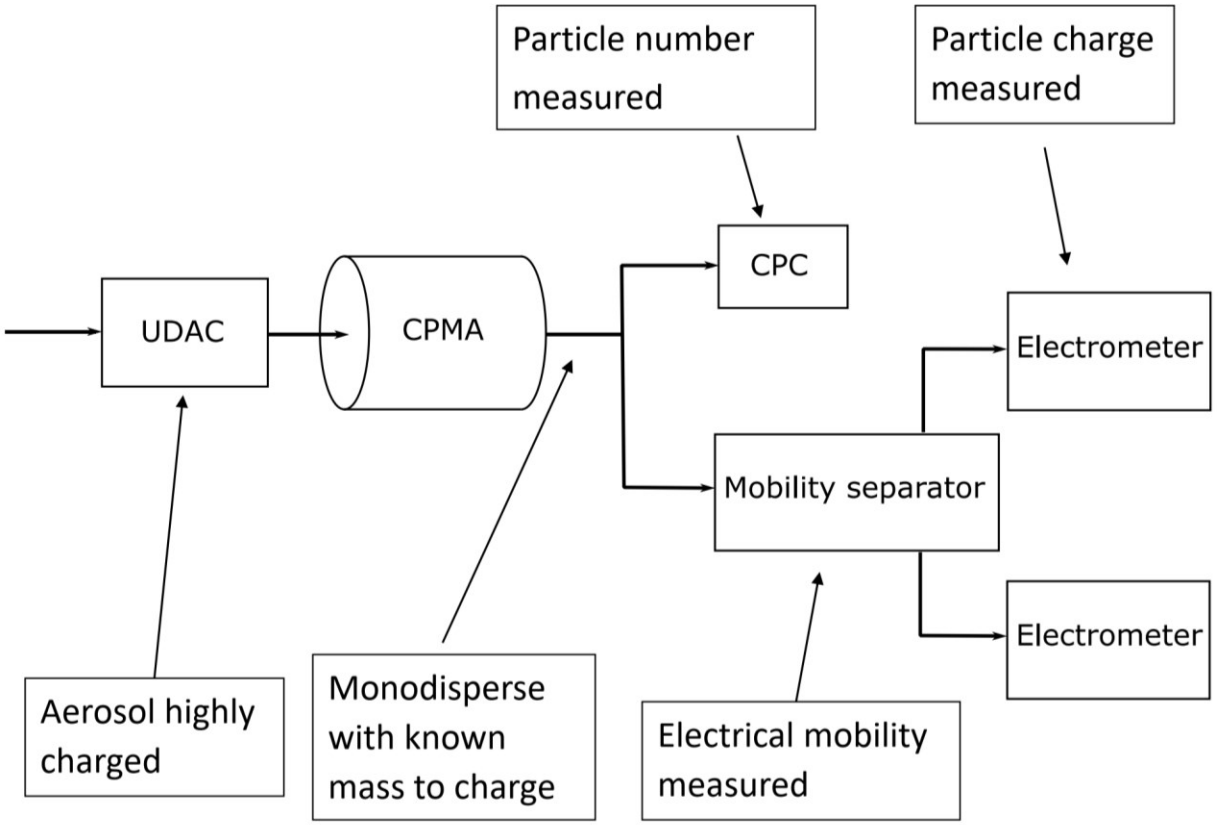
Particle mobility size analyzer



Electrostatic forces vs drag force

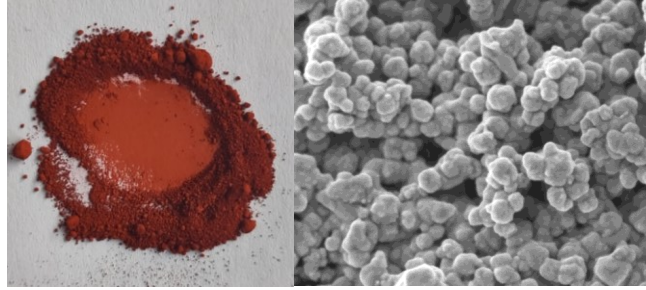
Simultaneous analysis of mass and diameter

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



Slide courtesy of CAMBUSTION

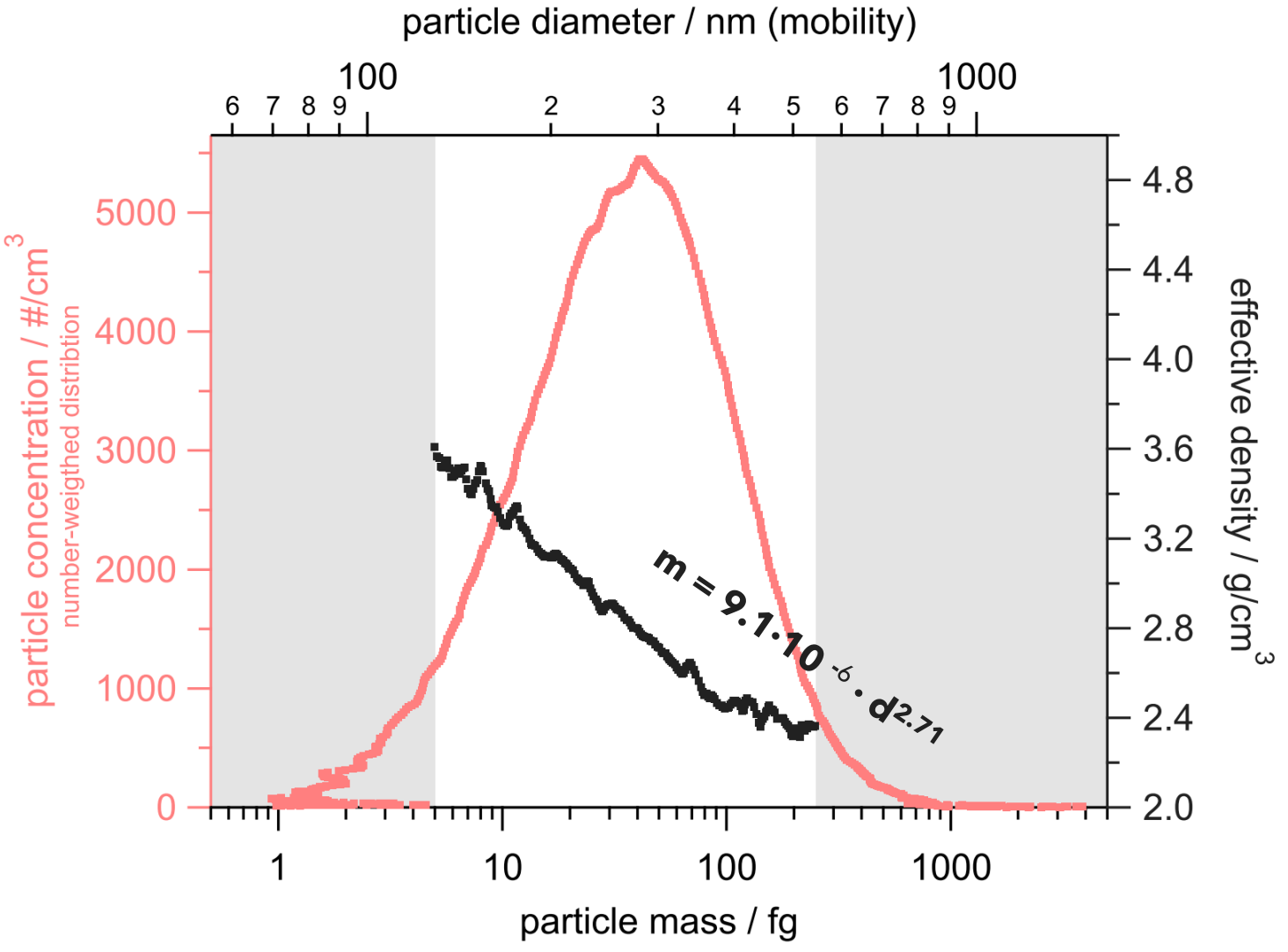
Fe_2O_3 pigment; d_{PP}: 90nm



- 10 min per scan; 600 data points
  → **4.7 mio** particles counted

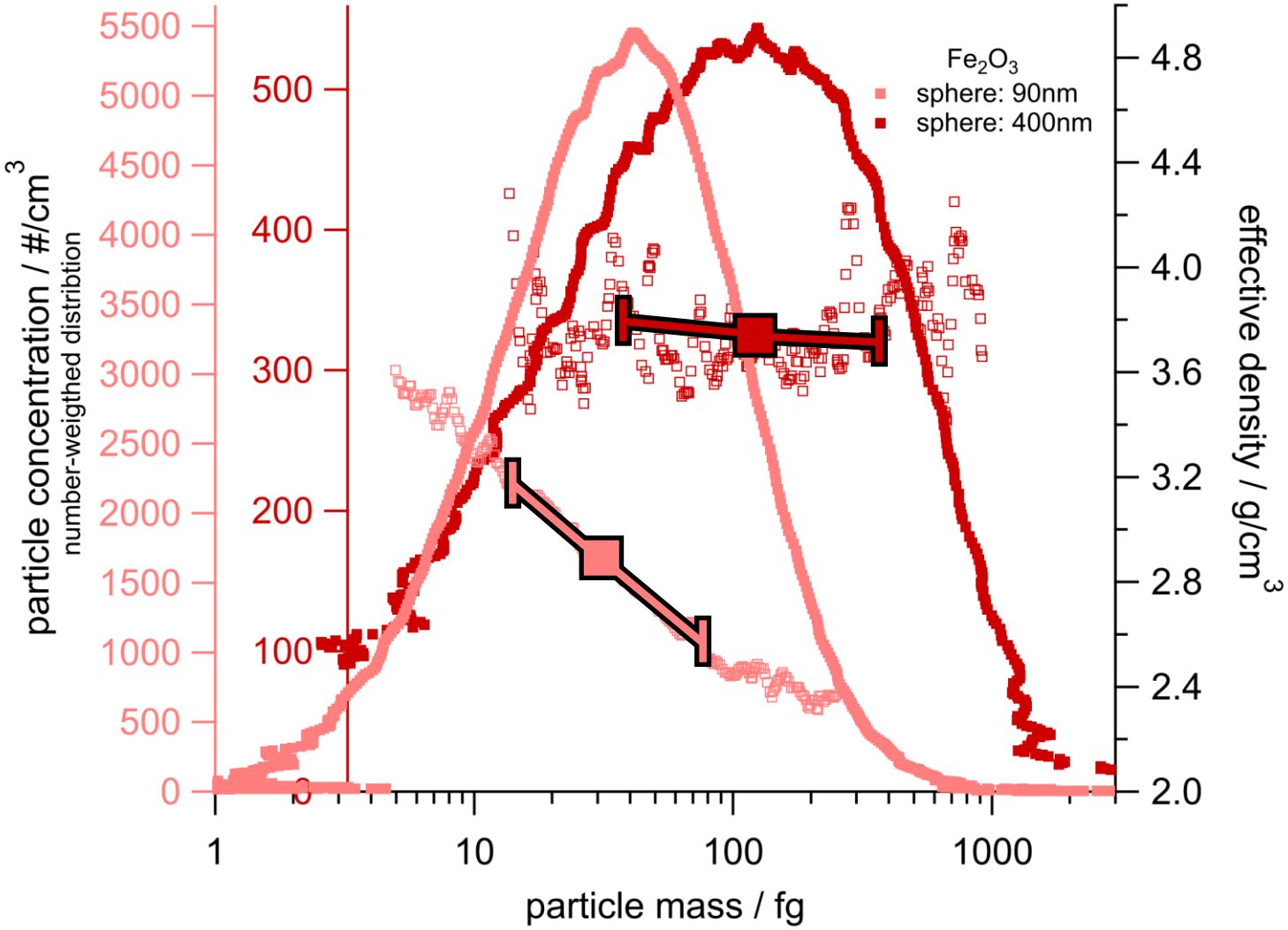
m_{50}: 31 fg
d_{50}: 274 nm
ρ_{50}: 2.9 g/cm^3

→ 1 gram of powder contains $1.8 \cdot 10^{13}$ single particles



Comparison of two Fe_2O_3 pigments

Median mass	Median diameter	Particles per gram	«Fractal»-index
90nm sphere @ 5.0 g/cm³ m_{sphere}: 1.9 fg → **fractal-like aggregate**			
31 fg	274 nm	$18 \cdot 10^{12}$ /g	**2.71**
400nm sphere @ 5.0 g/cm³ m_{sphere}: 167 fg → **isolated «spherical» particles**			
109 fg	380 nm	$2.2 \cdot 10^{12}$ /g	**2.97**



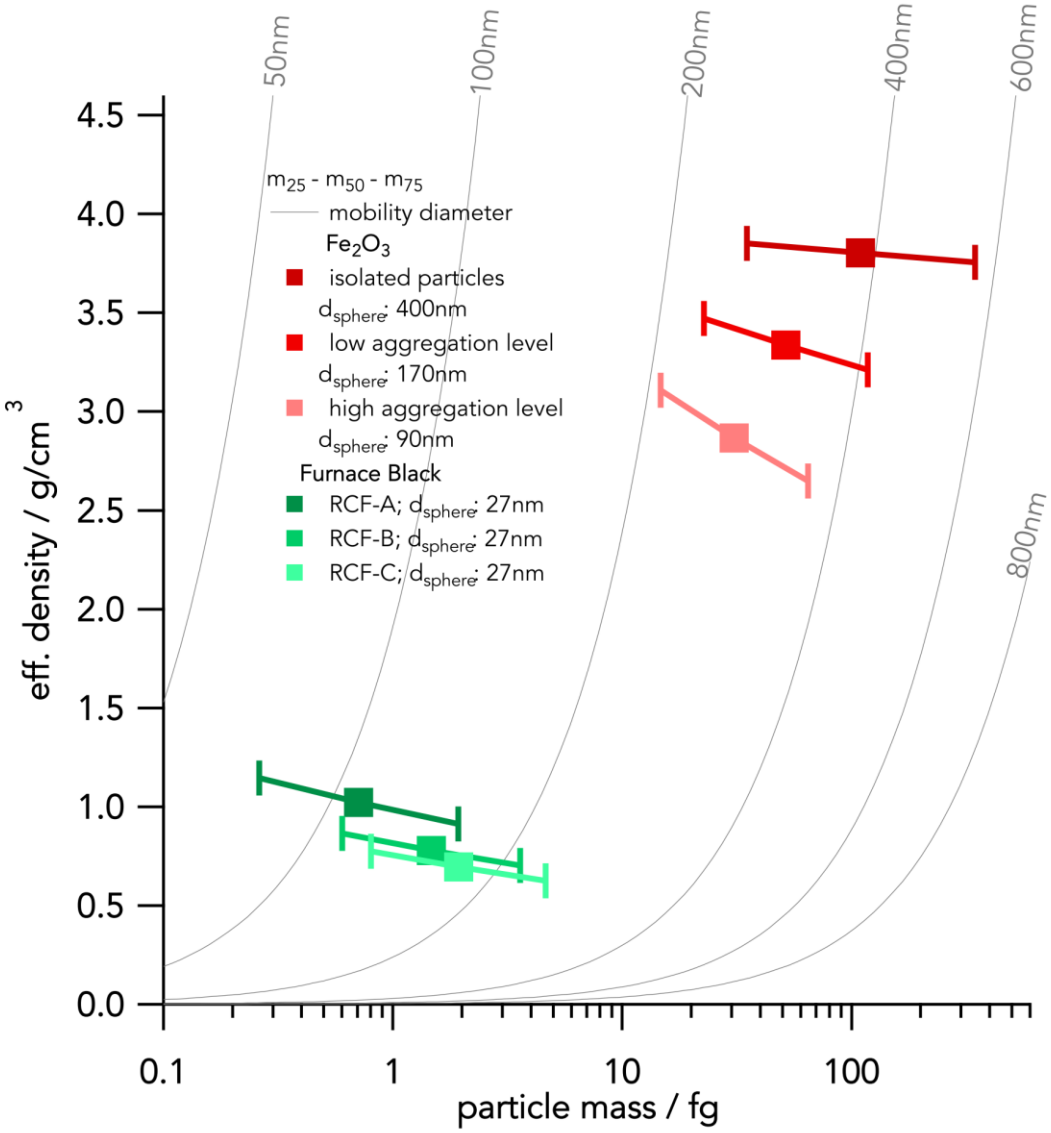
Primary particle size: Fe_2O_3

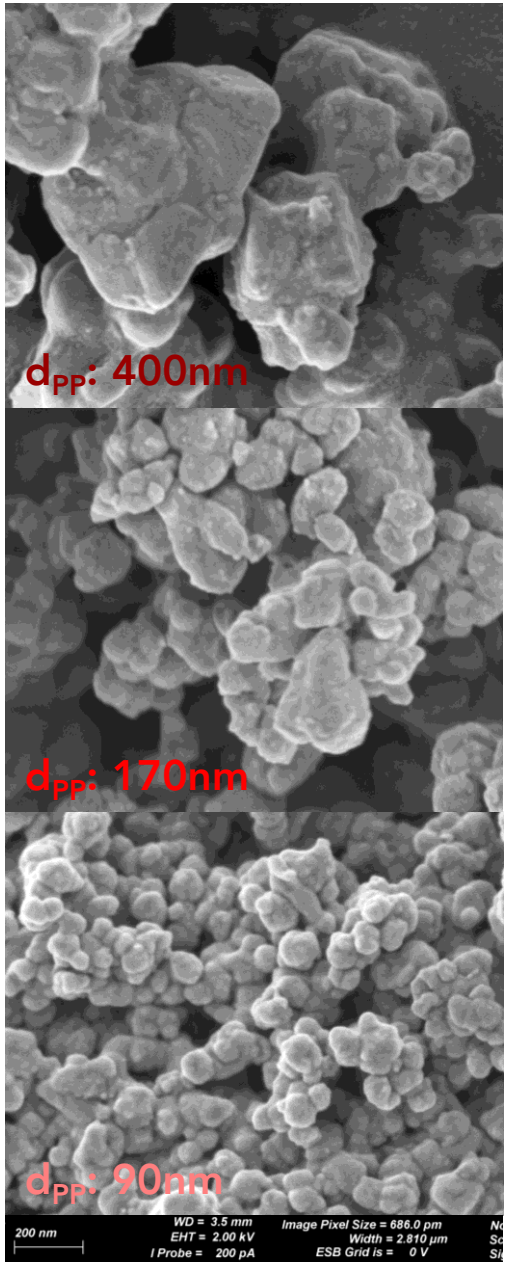
Increase primary particle size from 90 to 400 nm

- higher sphericity
- higher fractal index
- lower aggregation level
- less pore volume

sample preparation:

1. ultrasound deagglomeration
2. nebulizing and drying





Aggregation level: Carbon Black

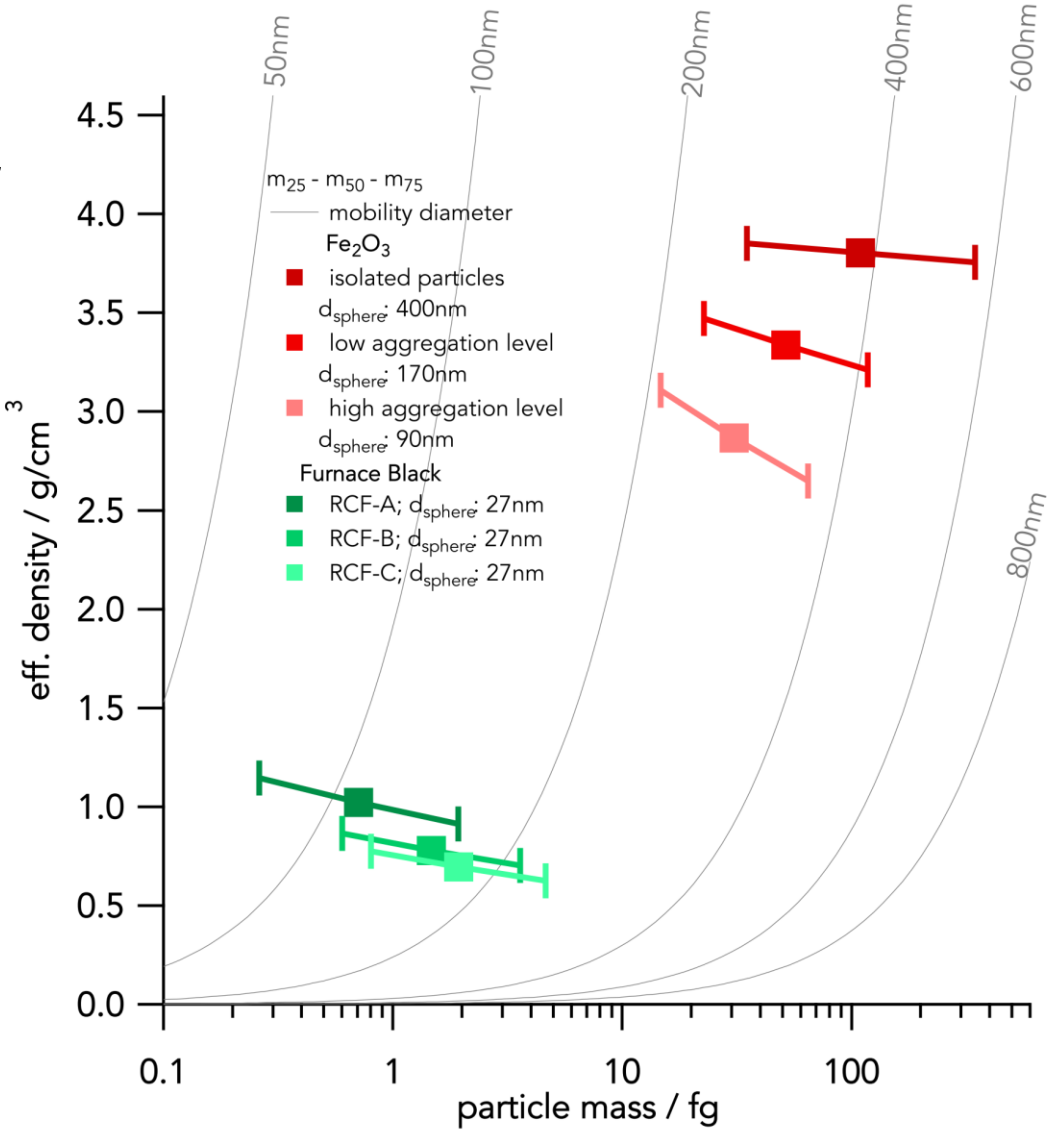
Increasing aggregation level and constant primary particle diameter of 27nm

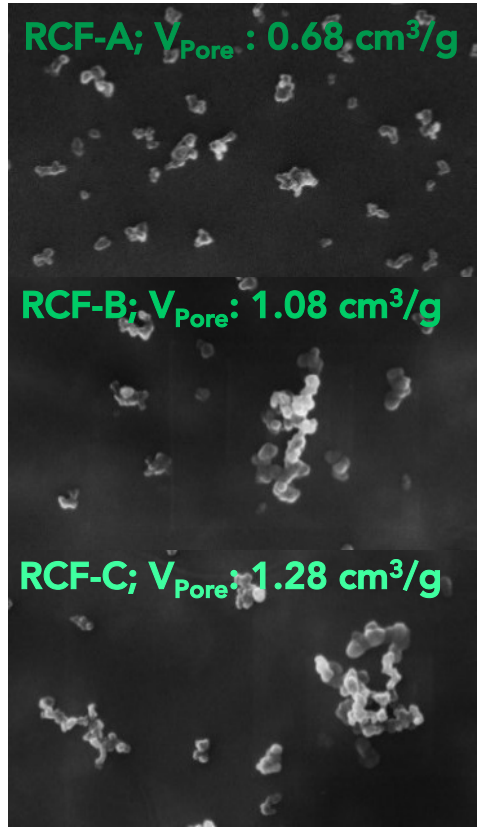
- constant sphericity
- constant fractal index
- heavier and larger particles
- lower density
- higher pore volume

sample preparation:

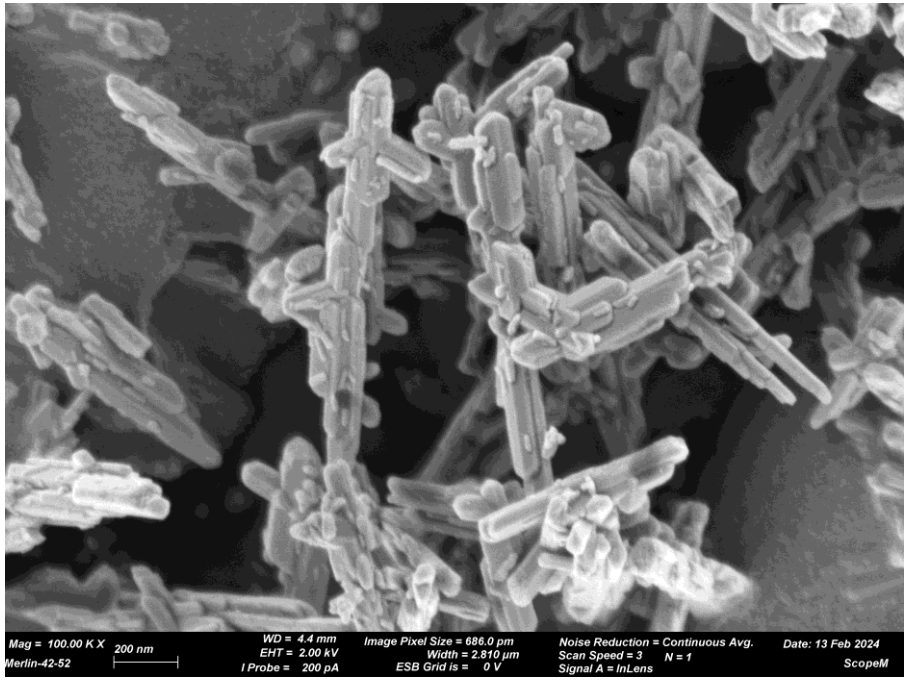
dry-dispersion

(sampling from reactor)

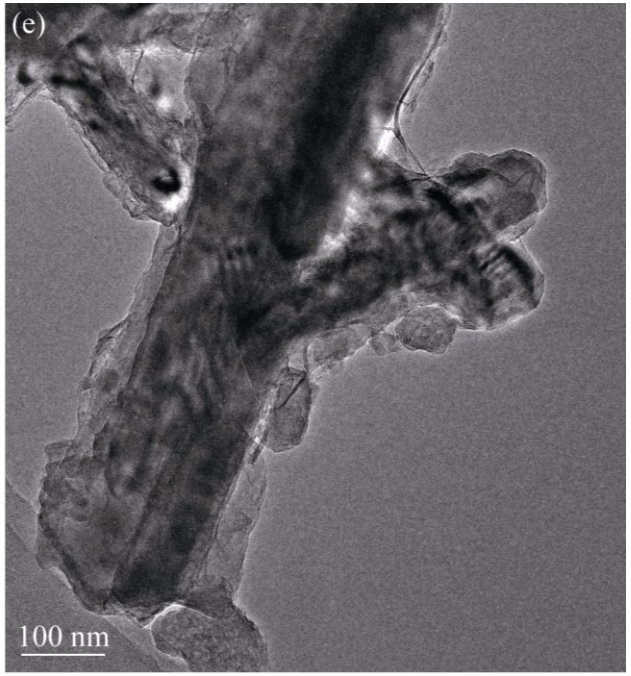




Multicomponent system



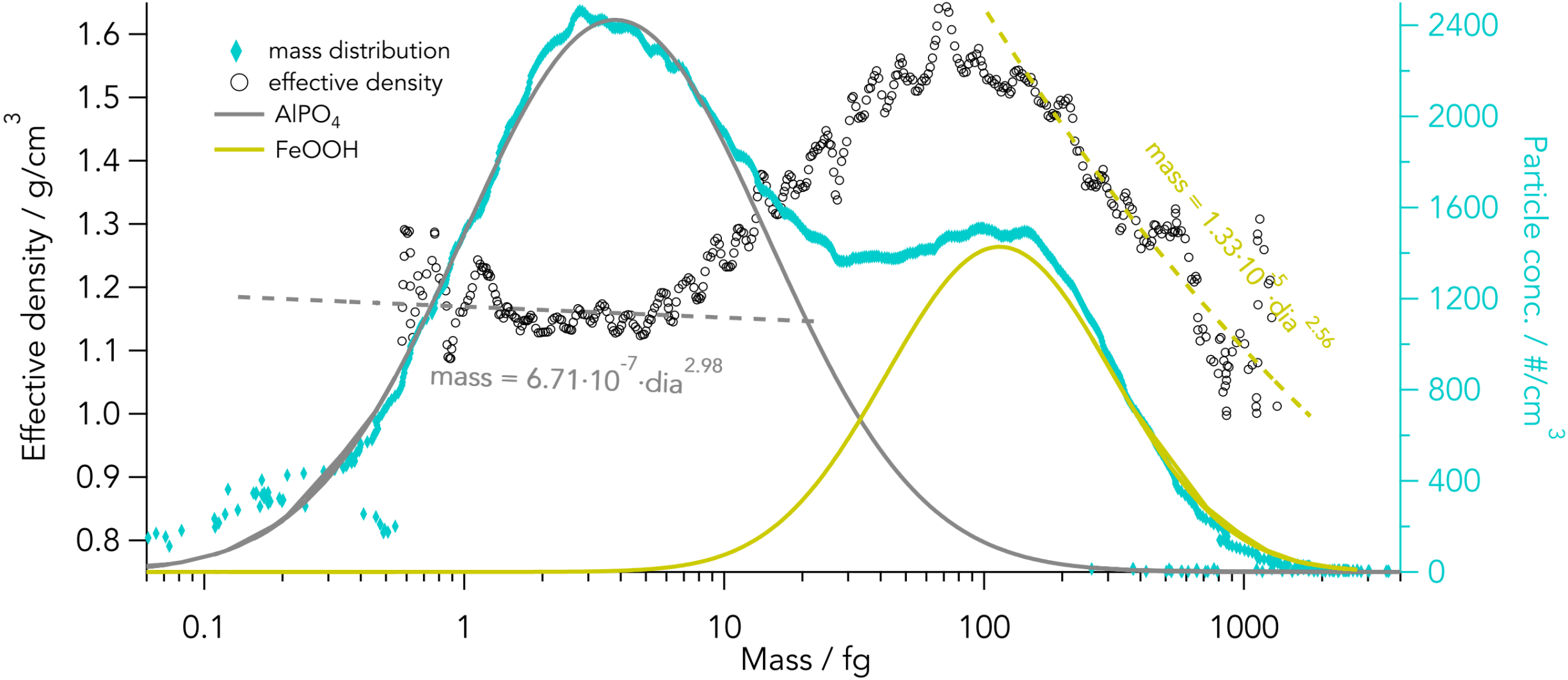
Uncoated FeOOH



FeOOH + $AlPO_4$ shell

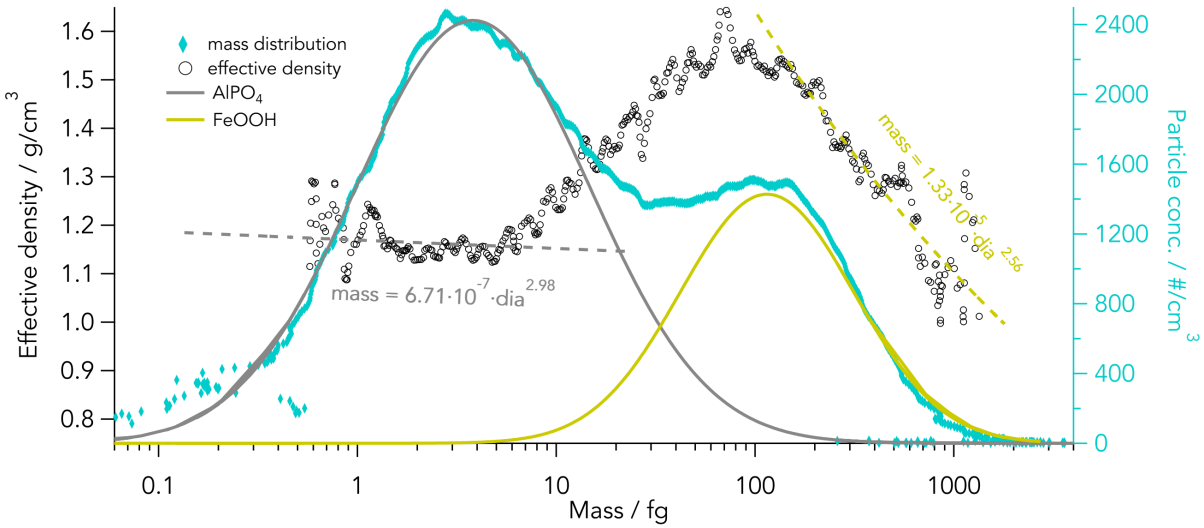
- Different refractive indice → laser diffraction?
- Undefined particle density → sedimentation analysis?
- What are the constituent particles?

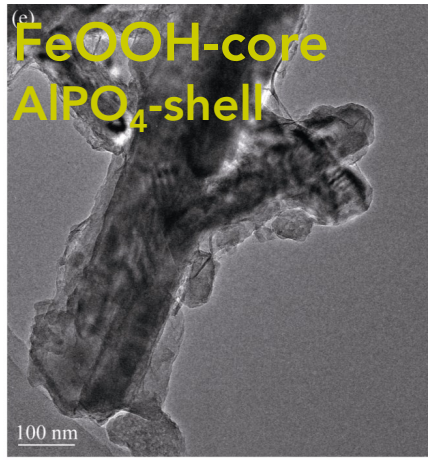
FeOOH Pigment with $AlPO_4$ coating



FeOOH Pigment with $AlPO_4$ coating

Median mass	Particles per gram	Mass fraction		«Fractal»-index
		measured	spec-sheet	
$AlPO_4$ isolated non-aggregated particles				
3.8 fg	$11 \cdot 10^{12}$ /g	**10%**	**26%**	2.98
FeOOH rods: 100 x 700nm @ 4.0 g/cm³ (+coating) m_{rod}: 29fg (+7fg)				
115 fg	$4.7 \cdot 10^{12}$ /g	90%	(74%)	2.56



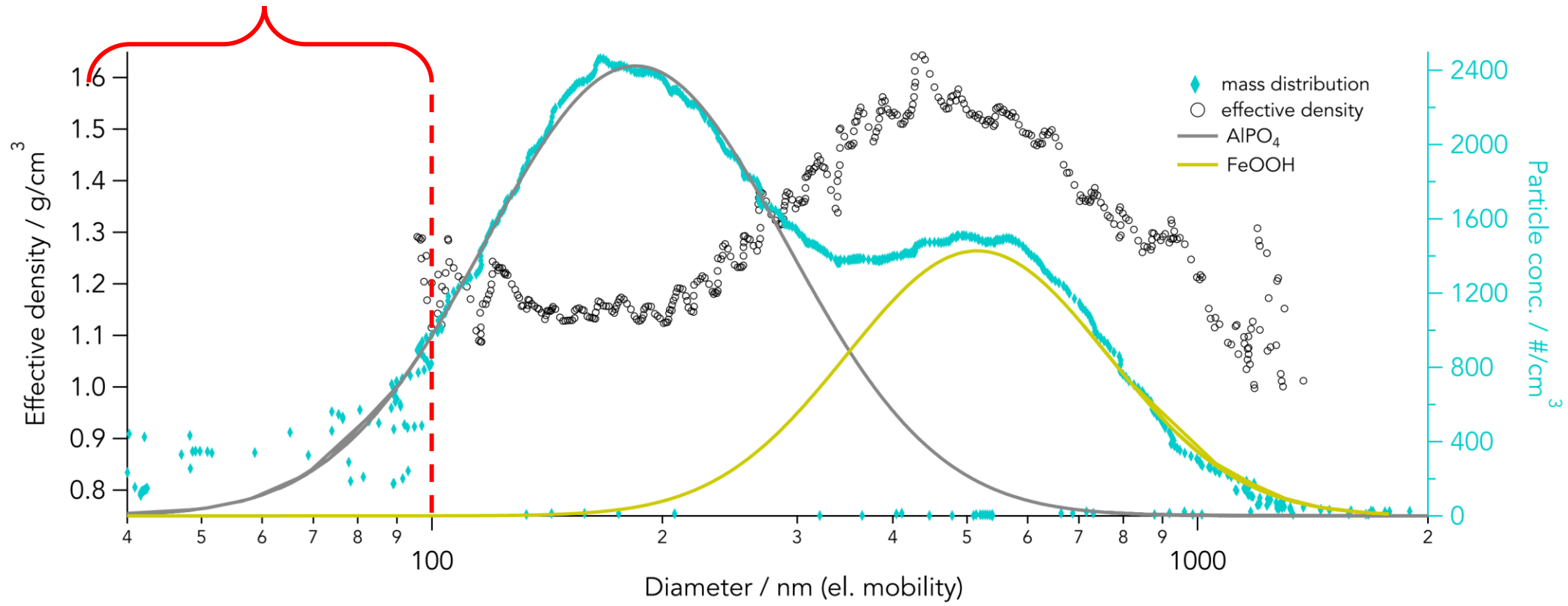


- 40% of $AlPO_4$ does not adhere to the FeOOH core
  → reduced color strength, loss of material, …

Definition of a nanomaterial according to the European Commision

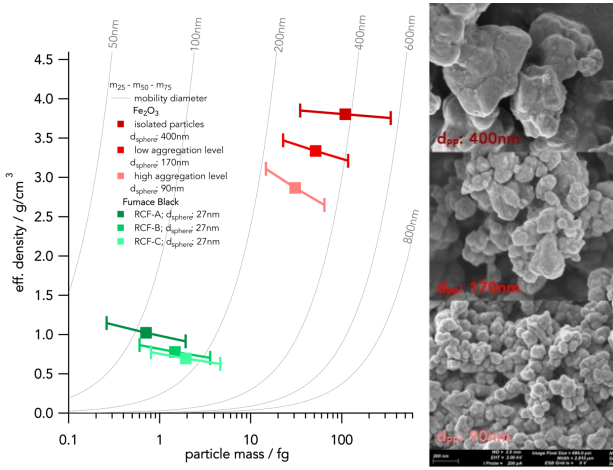
„ … 50 % or more of the particles in the number size distribution […] in the size range 1 nm -100 nm.“

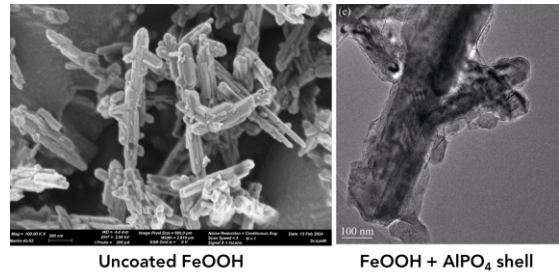
nano particle content: 6% or $9.4 \cdot 10^{11}$ nanoparticles per gram

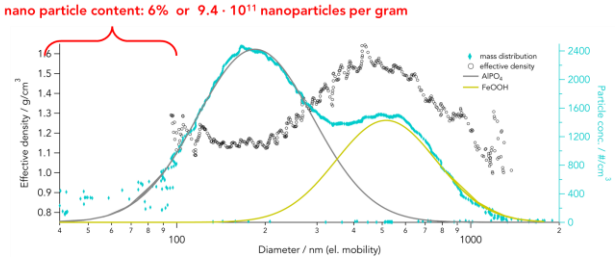


Summary

- Rapid analysis of particle size and structure
  - THE mass and A diameter
- Multicomponent systems require a multiparameter analysis
- (Nano-)particle concentration in a powder







Nanoparticle release vs. dispersion intensity
Titanium dioxide, bismut vanadate, pearlescent pigment
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