



**DFM**  
Danish National Metrology Institute

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**EMRP –**  
**European Metrology Research Programme**  
**Danske aktiviteter indenfor partikelmetrologi**

Metrologidag 20. maj 2014



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Denmark



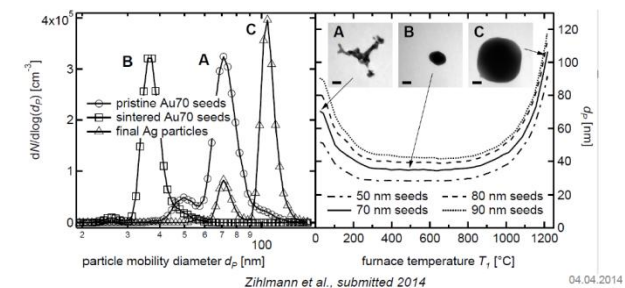
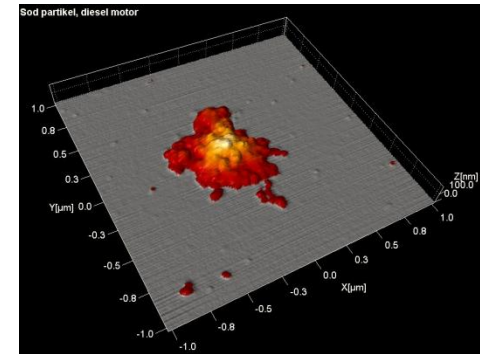
# Overview

§ Particle Emission -  
et europæisk “Joint Research Project”

§ Sodpartikler -  
målestørrelser – udfordringer – anbefalinger

§ Partikelmetrologi i Danmark -  
Videnstransfer – serviceydelser – forskningsopgaver

§ Opsummering



# Sodpartikler: regulative opgaver

- § Sodpartikler: urene kulpartikler fra ufuldstændig forbrænding af hydrocarboner,  
- ) WHO (2012) “kræftfremkaldende”  
- ) stor forårsager af global opvarmning

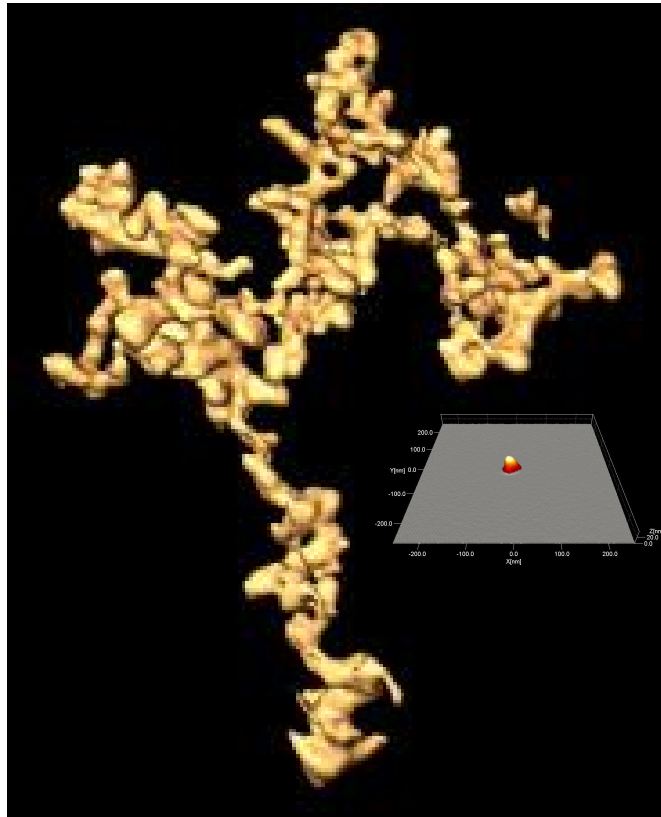


Foto: autobild

- § Euro 5 / 6 standards regulation 715/2007 for diesel vehicles (i kraft siden 2013):  
„Maks. 600 milliarder partikler per kørt km“



# Sodpartiklernes mange ansigter



ζ

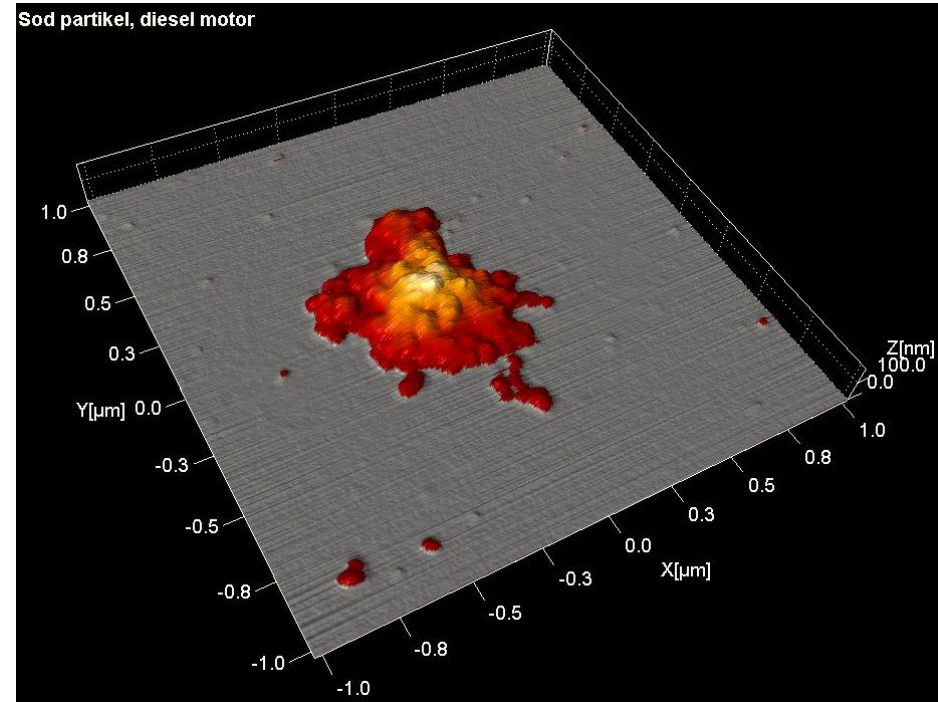
800 nm

ϕ

ζ

500 nm

è



Dieselpartikler ( $\sim 1 \mu\text{m}$ ) består af primærpartikel af kulstof (30 nm)

# PartEmission (2011-2014)

et fælles forsknings project (JRP) støttet af  
Europæisk Metrologi Forsknings Program (EMRP)



**DFM**

Danmarks Nationale Metrologiinstitut



**JRC**

EUROPEAN COMMISSION

*ie*

Institute for Energy



**METAS**

**NPL**

National Physical Laboratory



**MIKES**

Partikelreference-  
størrelse  
form  
material  
tællemetode

**TROPOS**

Leibniz Institute for  
Tropospheric Research

**PTB**

**EMRP**

European Metrology Research Programme  
• Programme of EURAMET

The EMRP is jointly funded by the EMRP participating countries  
within EURAMET and the European Union

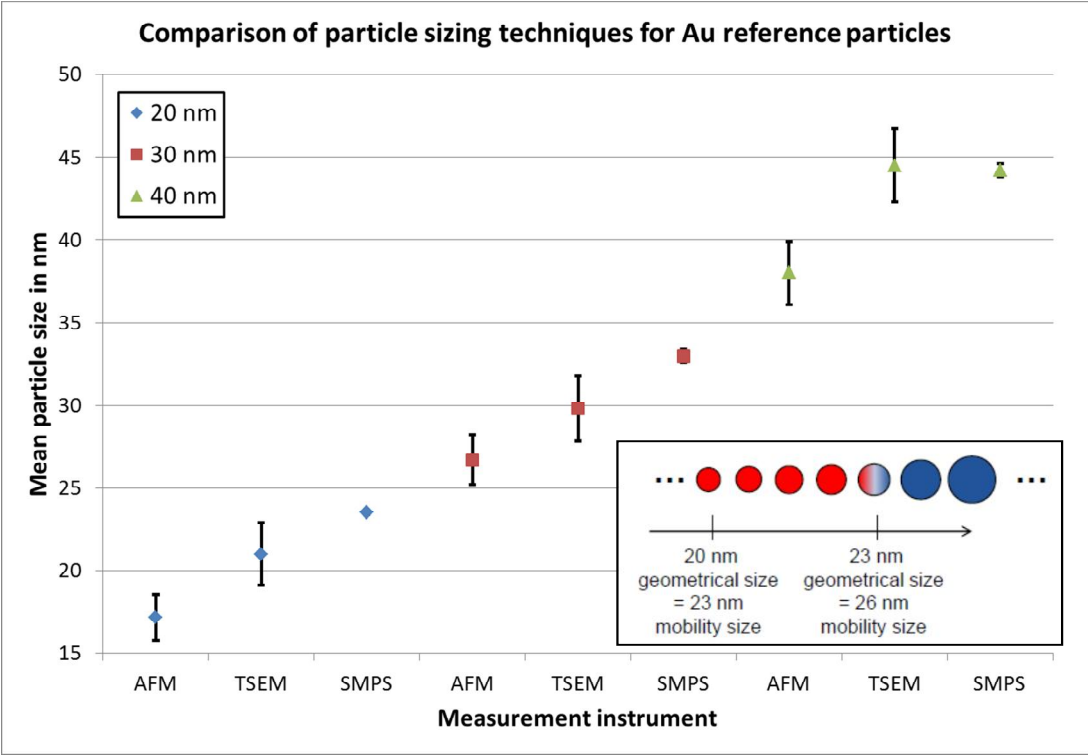


**Emerging requirements for measuring pollutants  
from automotive exhaust emissions**

*<http://www.ptb.de/emrp/partemission.html>*

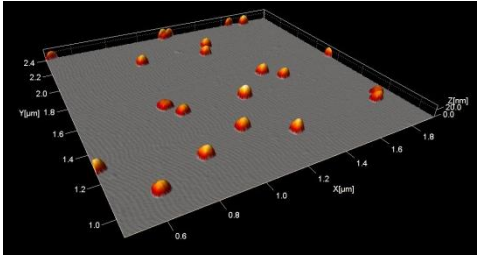
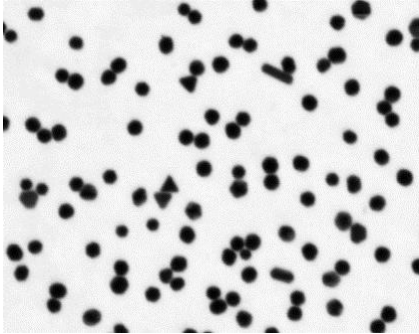


# PartEmission / partikelstørrelse: Kalibrering af partikeltæller med reference partikler



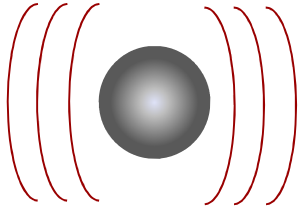
At the nanometer scale, the shells of different physical interaction do not necessarily coincide with the compact particle shape.

SEM:  
diameter



AFM:  
height

European Metrology Research Project on Particle Emission  
<http://www.ptb.de/emrp/partemission-publications.html>,  
ENV02 WP1 D1.1.1.pdf



SMPS:  
Mobility diameter



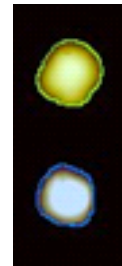
# PartEmission / partikelmorfologi: Ækvivalens af forskellige reference partikler

miniCAST 41 nm

miniCAST 23 nm

graphite 41 nm

Paired populations	$E_n$
$M_{23} - M_{41}$	-0.3
$M_{23} - G_{41}$	6.0
$M_{41} - G_{41}$	3.5

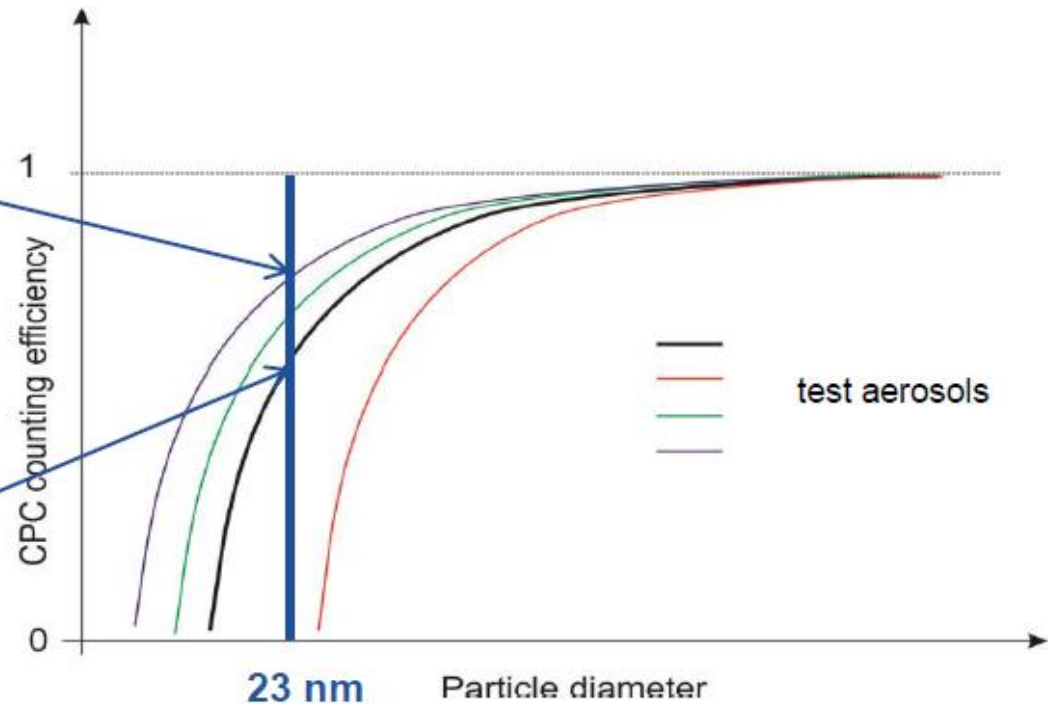
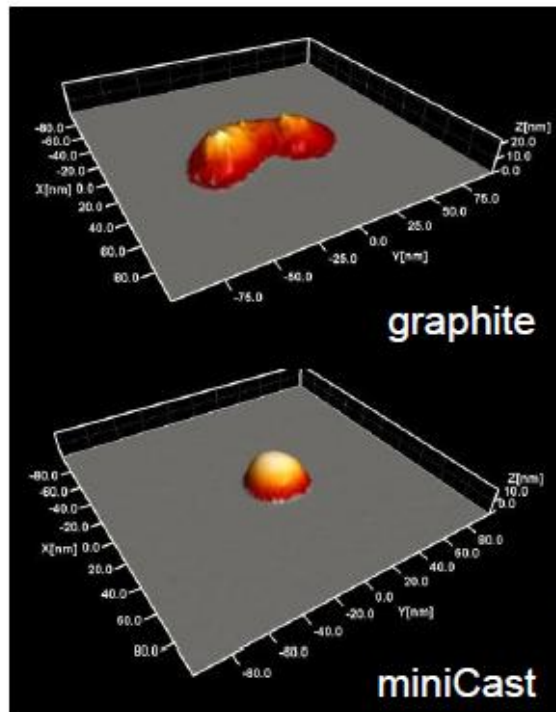


Roundness  
(a.u.)

Average	$M_{23}$	0.85
	$M_{41}$	0.87
	$G_{41}$	0.60
Standard uncertainty of average	$M_{23}$	0.02
	$M_{41}$	0.06
	$G_{41}$	0.04



# PartEmission: Skal reference aerosol "efterligne" sodpartikler ?



Test med forskellige partikelmaterialer viser, at partikeltæller er følsomme for materiale, form og størrelse.

Heterogen nukleerede (sintrede) sølv partikler "machtet" bedst.



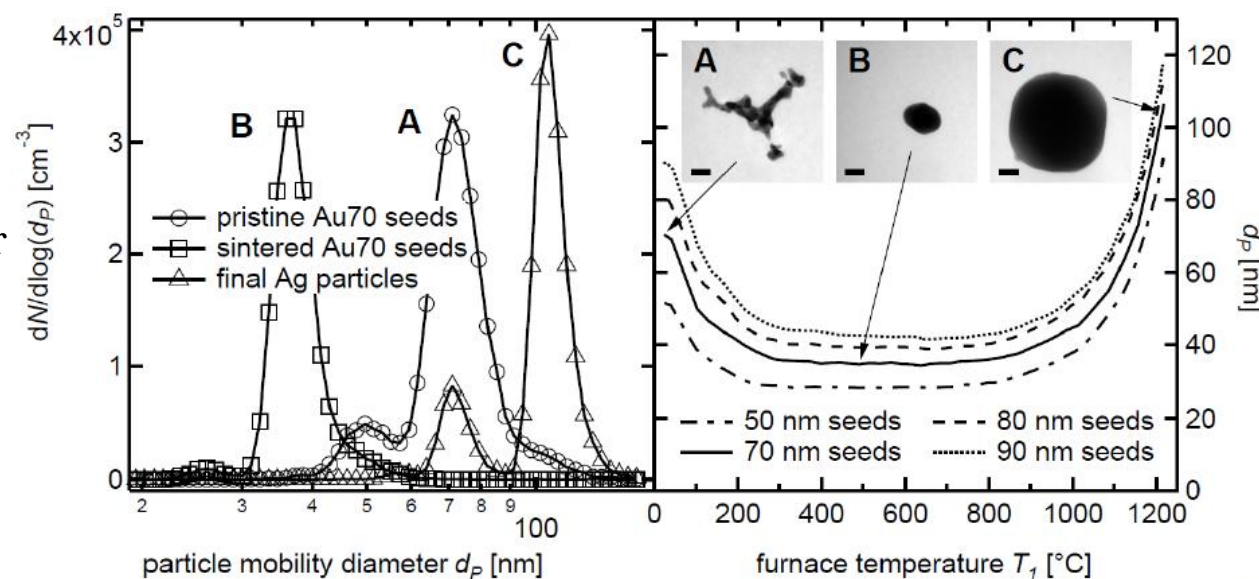
# PartEmission: Nuværende anbefalinger (projektet udløber 2014)



- Kalibrering af tællerne sker ved  $>80$  nm med kugleformede reference partikler
- Mobilitetsdiameteren  $< 80$  nm beregnes efterfølgende iht. ISO 15900
- Der findes ikke én type "sod-lignende" reference partikler, men sintrede sølvpartikler anbefales pga. aerosol performance/reproducerbarhed

## Fremtidens opgave

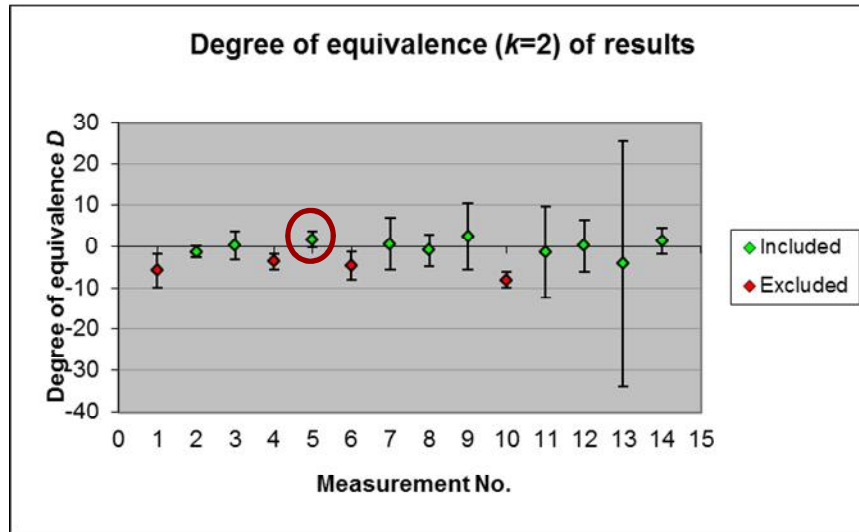
- Kalibreringsydelse for partikelantal i sod-aerosoler skal etableres efter behov



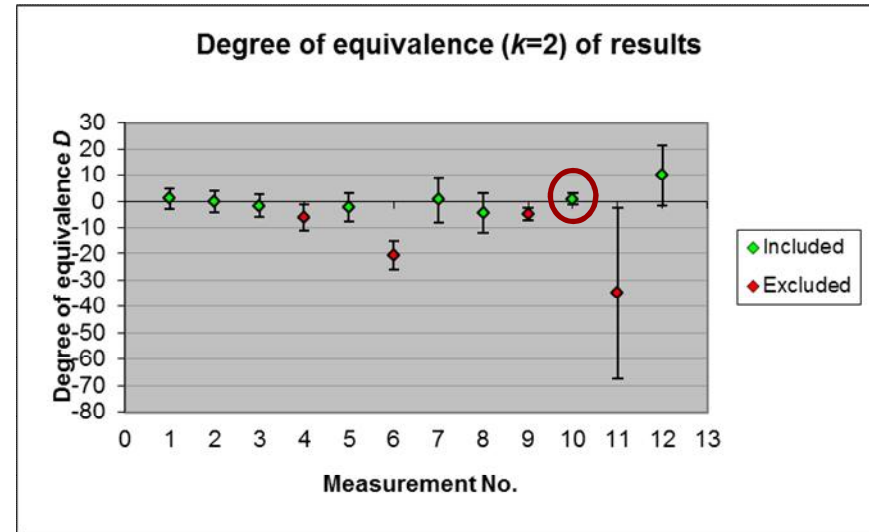
04.04.2014



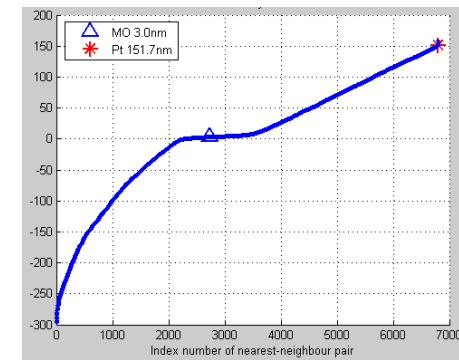
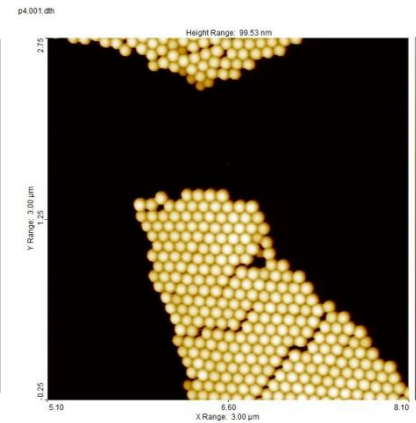
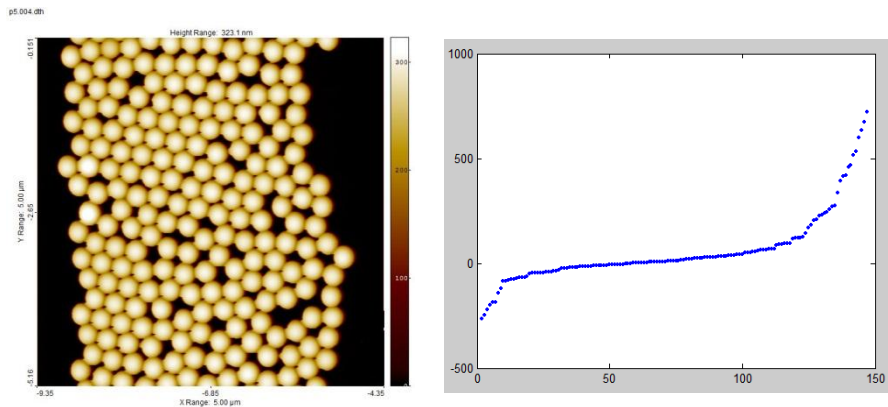
# Good performance during international comparison "Particle size" 2012/13 (preliminary results)



0.100 µm particles



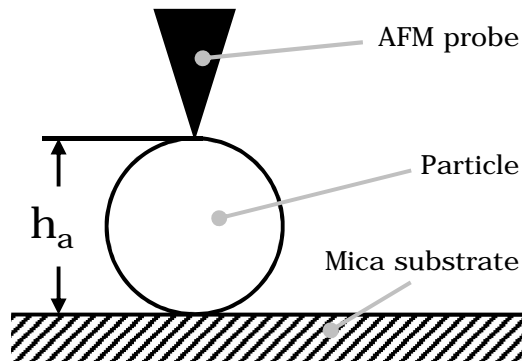
0.300 µm particles



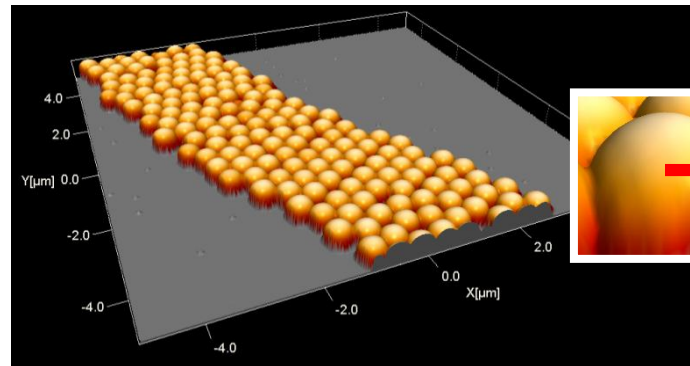


# Size-certified nanometer particles traceable to the meter with a metrological Atomic Force Microscope

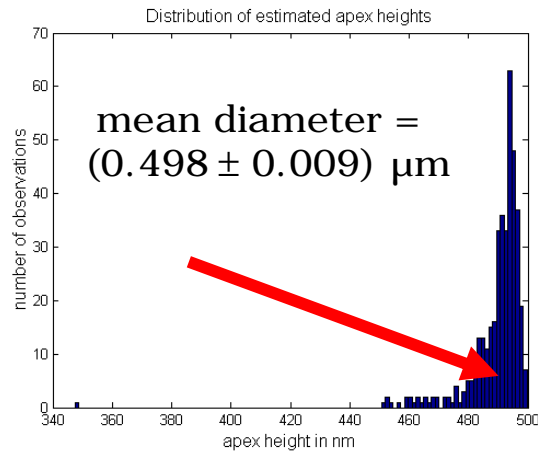
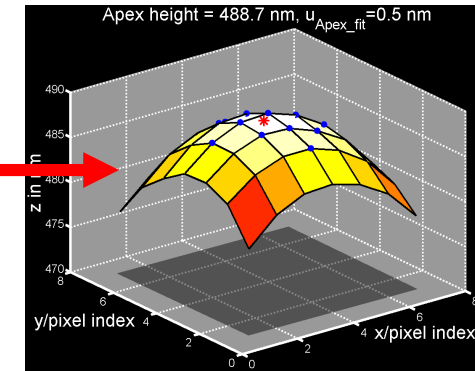
## Measurement method



## AFM image of particle sample



## Data analysis



Result: diameter distribution  
3308 KDI

**DANAK**  
CAL Reg. nr. 266

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Report no. N0200  
Page 1 of 6

**Calibration certificate**  
**Nanoparticle diameter**

Customer	
Address	
Telephone/Fax	
Contact person	

Certification

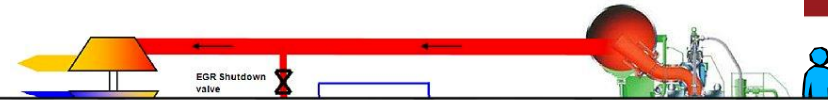
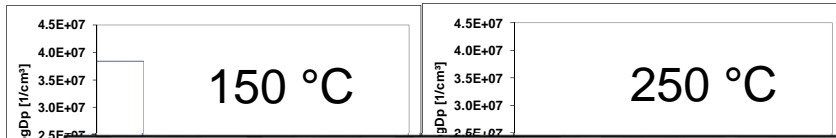
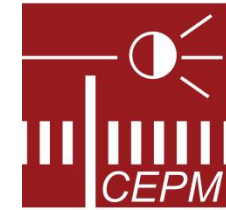


Diameter ranges:  
from 100 nm  
to 5 000 nm

Uncertainties:  
from 5 nm  
to 50 nm

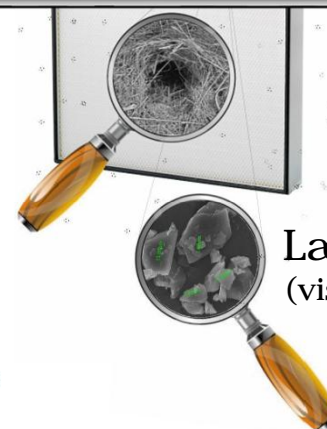
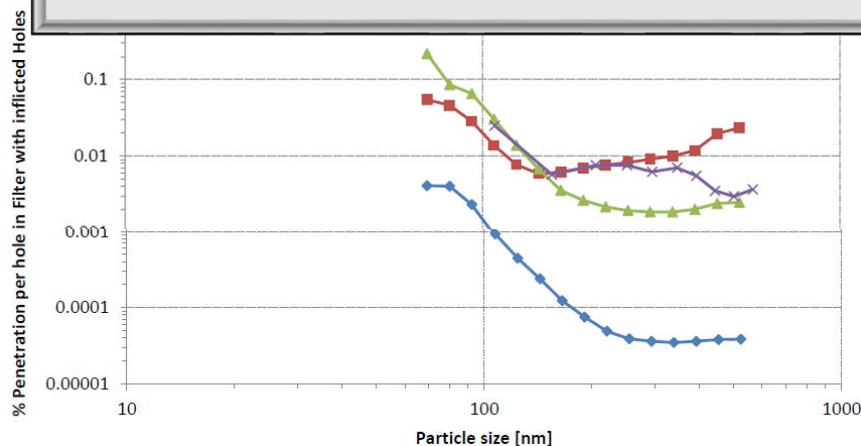
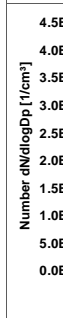
Certified dispersion (15 ml)

GTS- samarbejde DFM/Teknologisk Institut/Force Technology fortsættes i  
**Centre of Excellence for Partikel Metrologi**



**Serviceydelser i Danmark i GTS-regi**

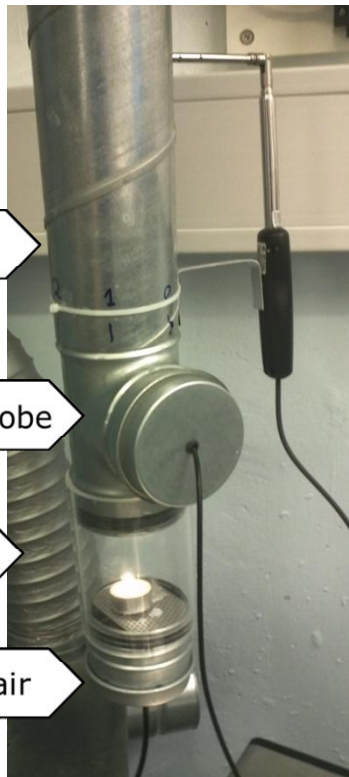
- Konsulentytelser (bl.a. for Miljøstyrelsen)
- Måling af luftbårne partikler i indeklimaet i tog
- Måling af suspenderede partikler for dansk industri
- Måling af ultrafine partikler i udeluften omkring lufthavn
- Måling af sammensætning og emissioner af ultrafine partikler fra fly og køretøjer i lufthavn
- Måling af ultrafine partikler fra vådscurber
- Opmåling af partikler indlejret i faststof ved brug af microCT scanner
- Akkrediteret gennemsnitsdiameter af en partikelpopulation for reference brug
- Partikelstørrelsesfordeling i suspensioner
- Brydningsindeks af partikler i suspensioner
- Akkrediteret kalibrering af partikeltæller for rene rum



Innovationskonsortium  
 NaKIM (2010-2013)

**Lækage i HEPA-filtre**  
 (viste målinger: Teknologisk Institut)

# Forbrændingspartikler fra lyse

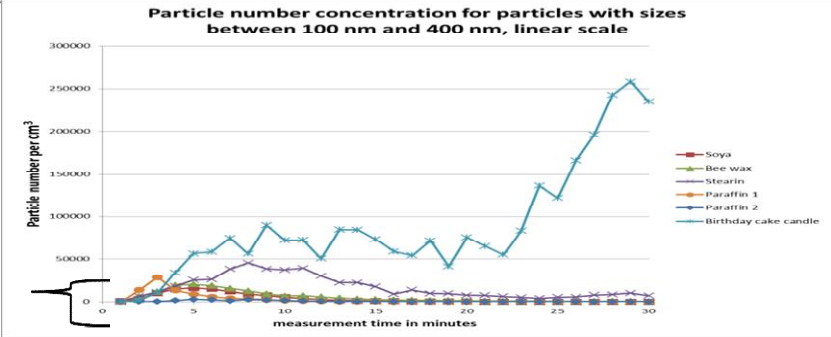
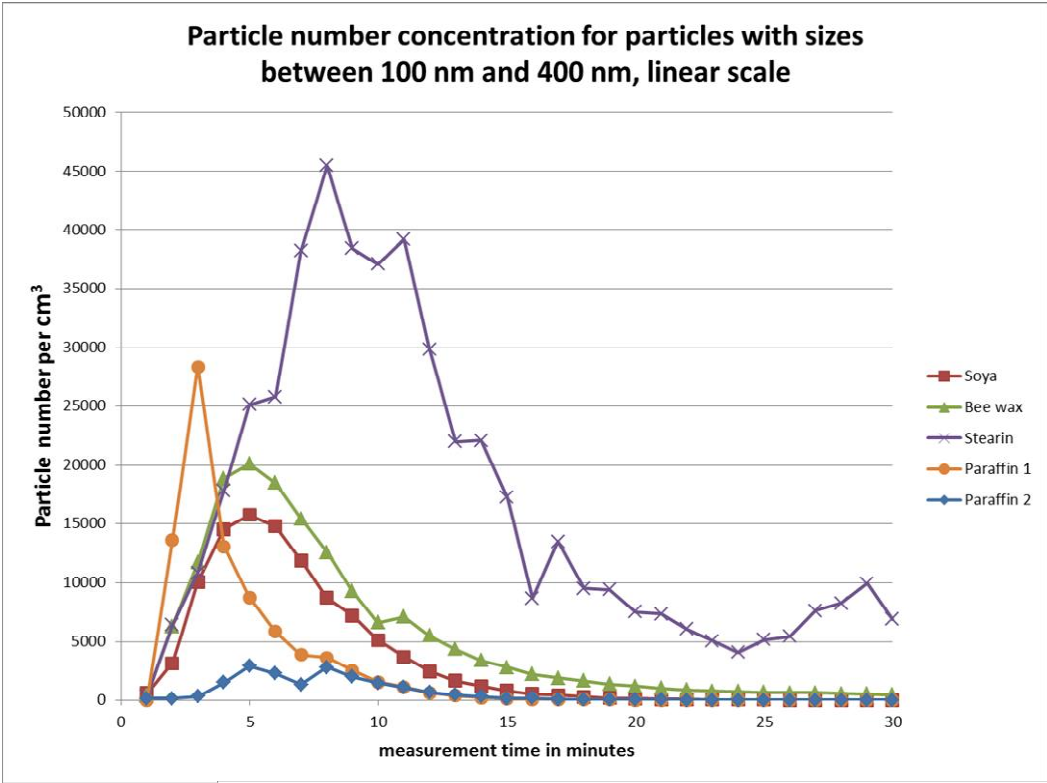


2 m chimney

Particle counter probe

Combustion chamber

Inlet for filtered clean air





# Renrum klasser – global kvalitetssikring igennem standardisering

Class	maximum particles/m <sup>3</sup>					
	≥0.1 μm	≥0.2 μm	≥0.3 μm	≥0.5 μm	≥1 μm	≥5 μm
ISO 1	10	2.37	1.02	0.35	0.083	0.0029
ISO 2	100	23.7	10.2	3.5	0.83	0.029
ISO 3	1,000	237	102	35	8.3	0.29
ISO 4	10,000	2,370	1,020	352	83	2.9
ISO 5	100,000	23,700	10,200	3,520	832	29
ISO 6	1.0 × 10 <sup>6</sup>	237,000	102,000	35,200	8,320	293
ISO 7	1.0 × 10 <sup>7</sup>	2.37 × 10 <sup>6</sup>	1,020,000	352,000	83,200	2,930
ISO 8	1.0 × 10 <sup>8</sup>	2.37 × 10 <sup>7</sup>	1.02 × 10 <sup>7</sup>	3,520,000	832,000	29,300
ISO 9	1.0 × 10 <sup>9</sup>	2.37 × 10 <sup>8</sup>	1.02 × 10 <sup>8</sup>	35,200,000	8,320,000	293,000



**ISO 14644-1**  
**ISO 21501-4**

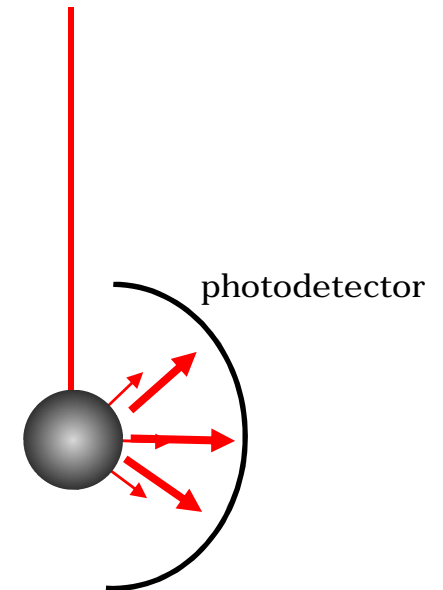
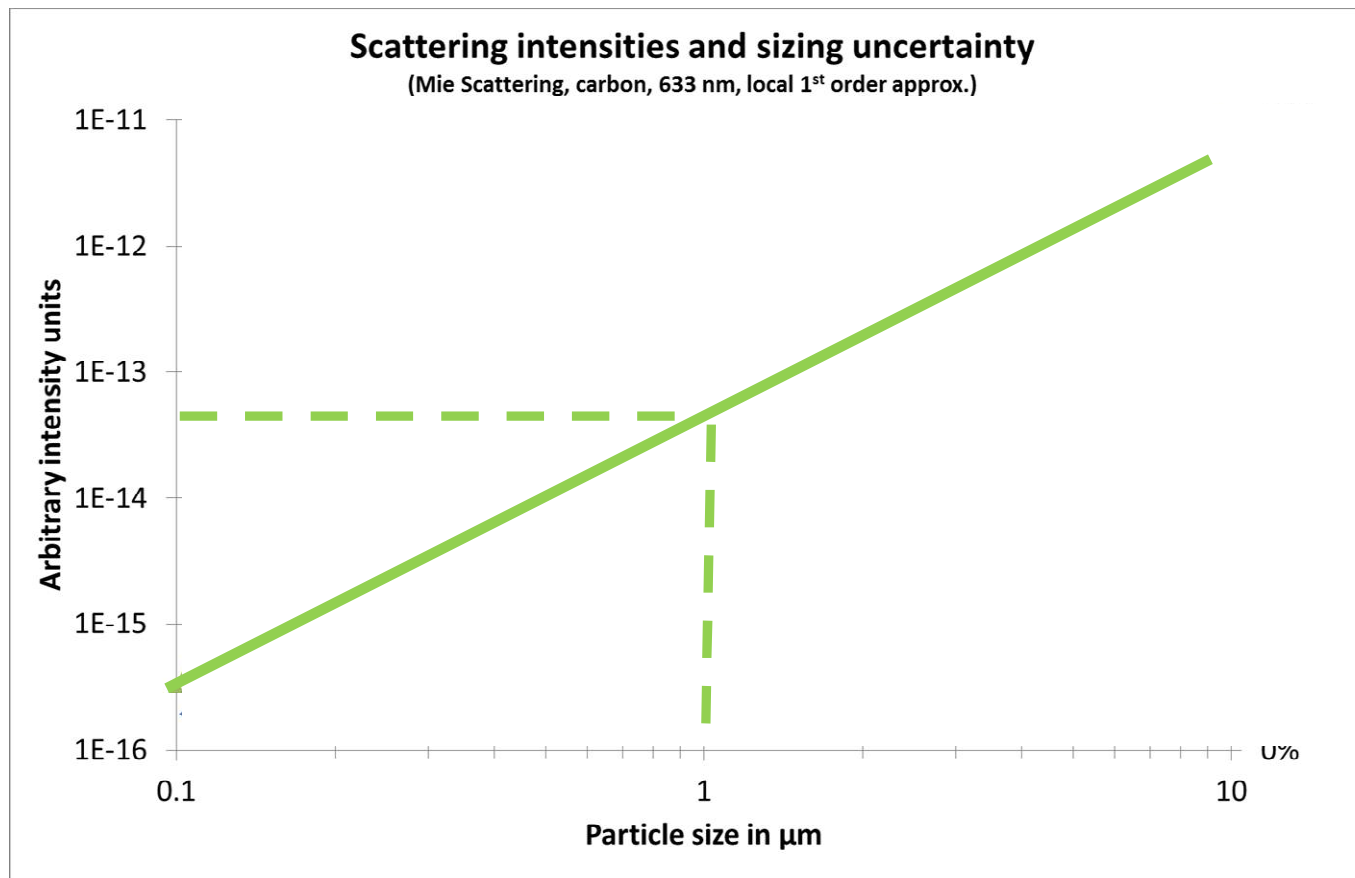
**Danske eksperter bidrager  
aktivt i ISO udvalg**





# Physical limitations of optical particle counters

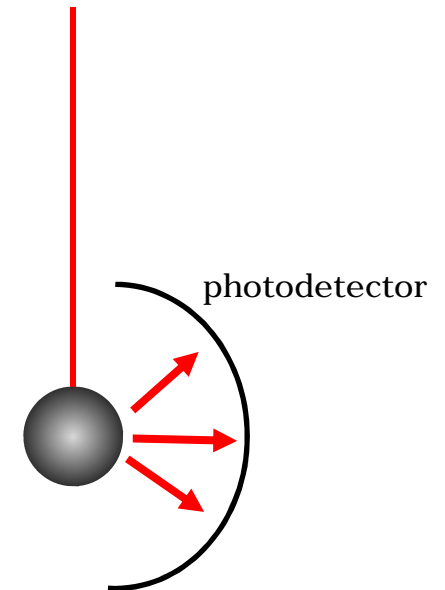
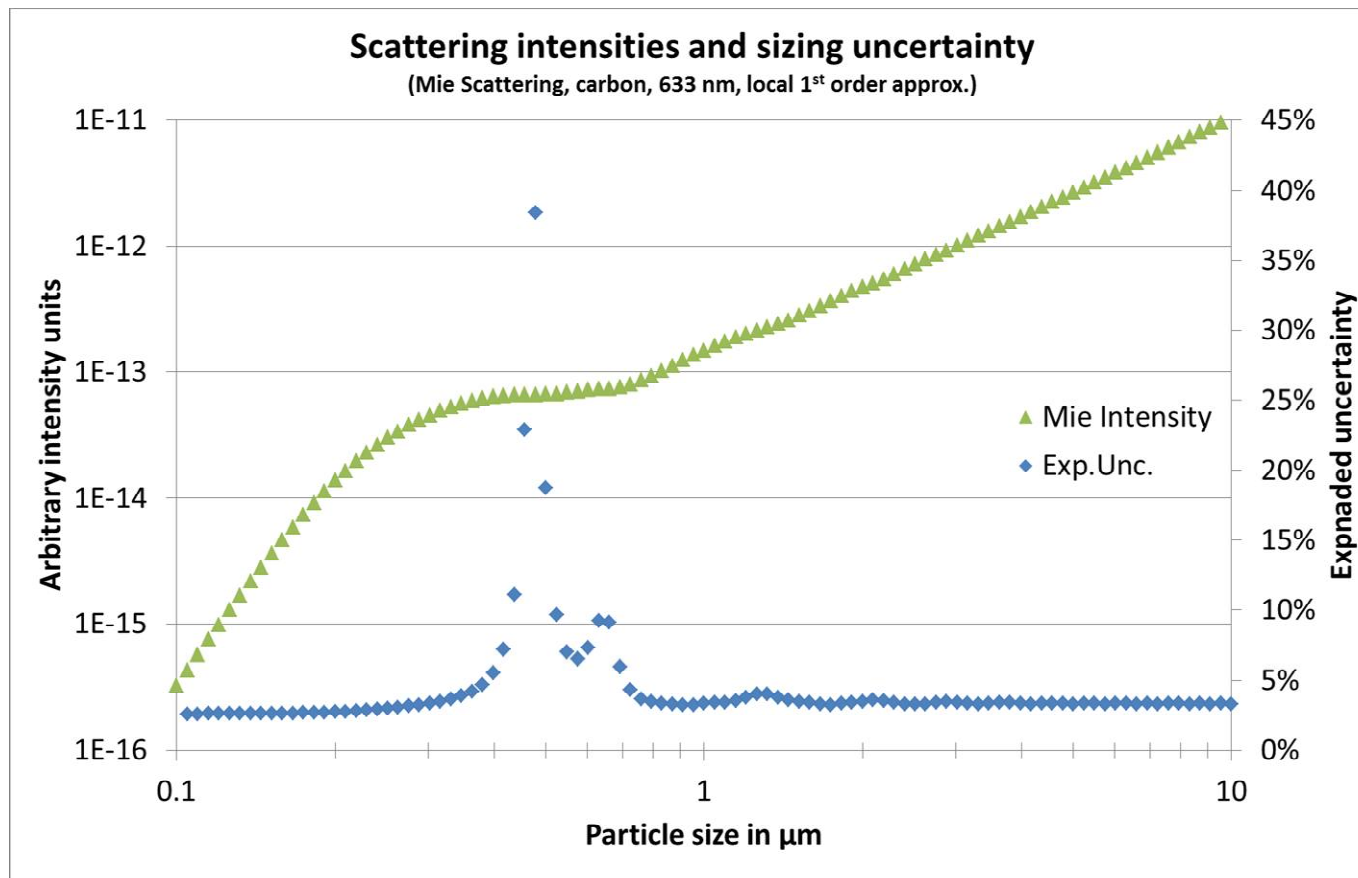
Light scattering particle counters use a photodetector to measure the intensity of the light scattered from a particle.





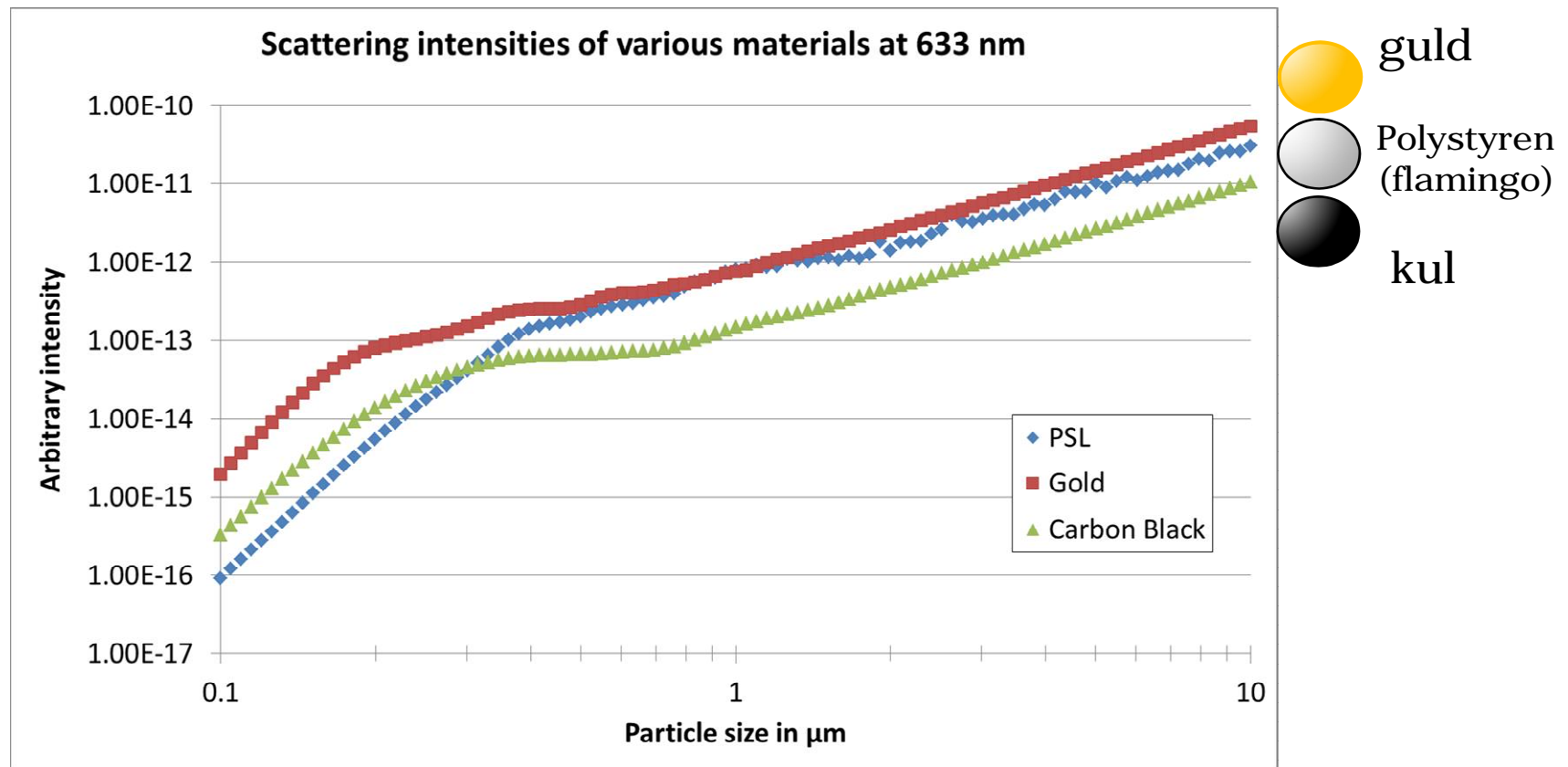
# Physical limitations of optical particle counters

Light scattering particle counters use a photodetector to measure the intensity of the light scattered from a particle.





# Physical limitations of optical particle counters





# Akkrediteret ydelse til kalibrering af partikeltæller i rene rum

Expanded uncertainties on particle number concentration:

5% sizes 0.1  $\mu\text{m}$  to 1.999  $\mu\text{m}$   
8% sizes 2.0  $\mu\text{m}$  to 6  $\mu\text{m}$

Dilution and Homogenization 4  
(max 300 L/min)

Exhaust 6

3 Drying of aerosol

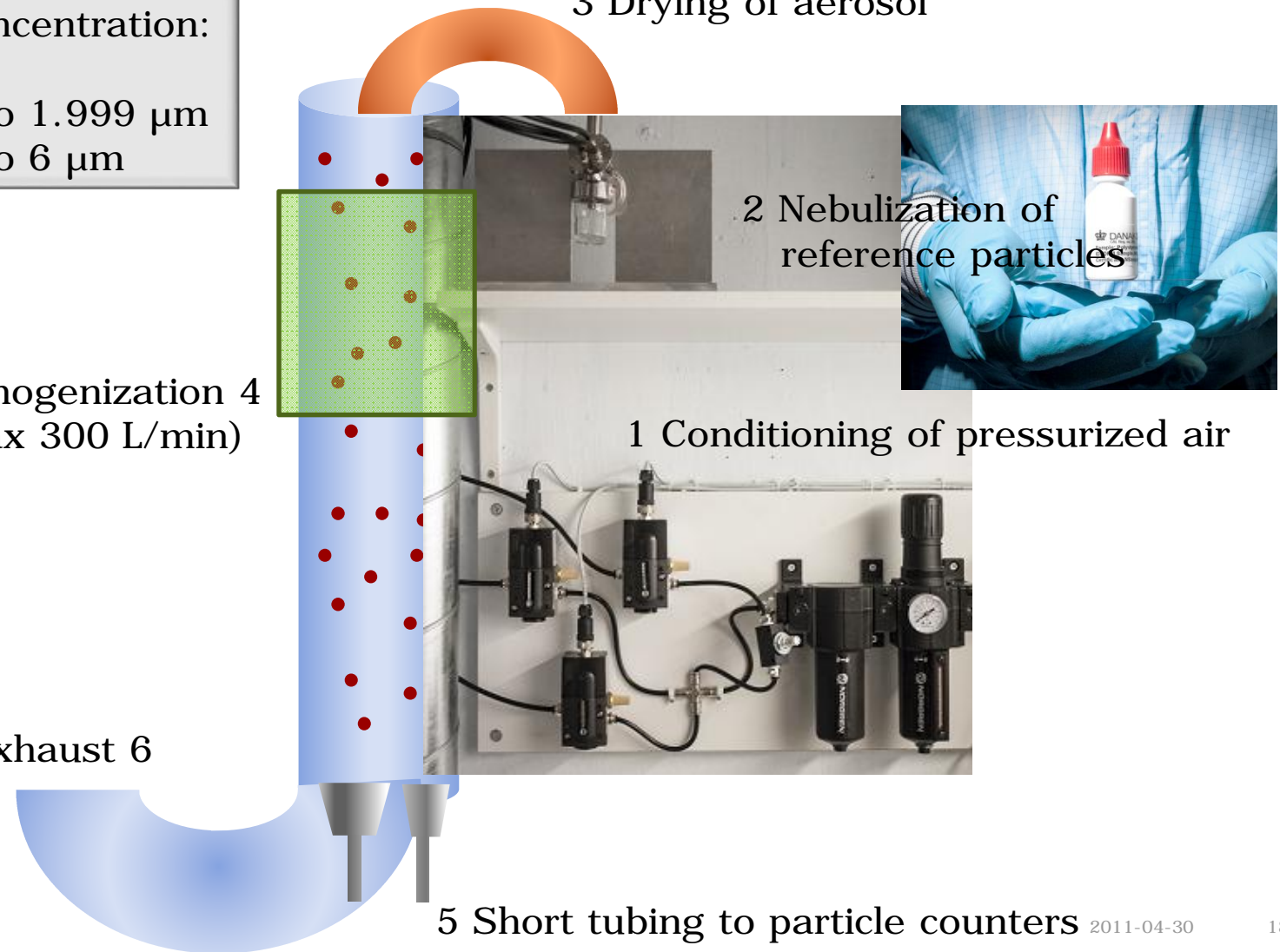
2 Nebulization of reference particles

1 Conditioning of pressurized air

5 Short tubing to particle counters

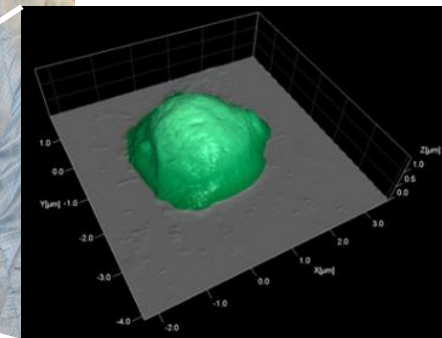
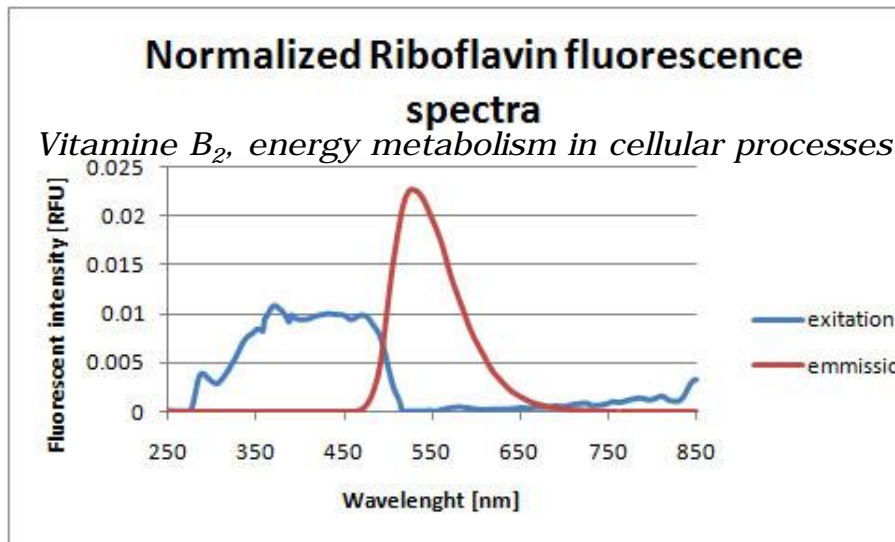
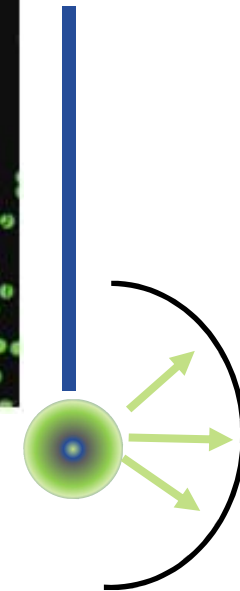
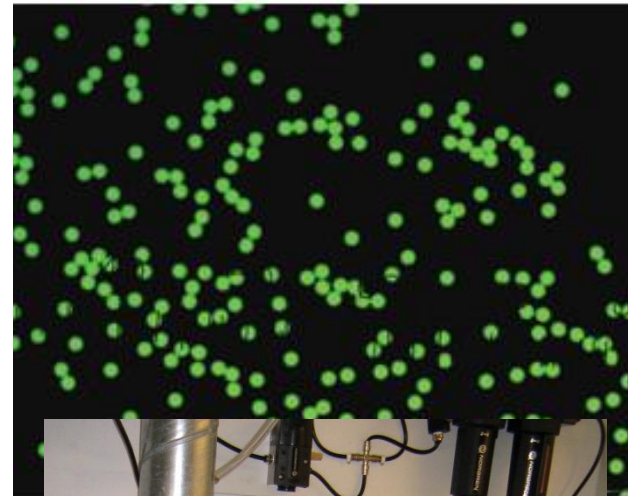
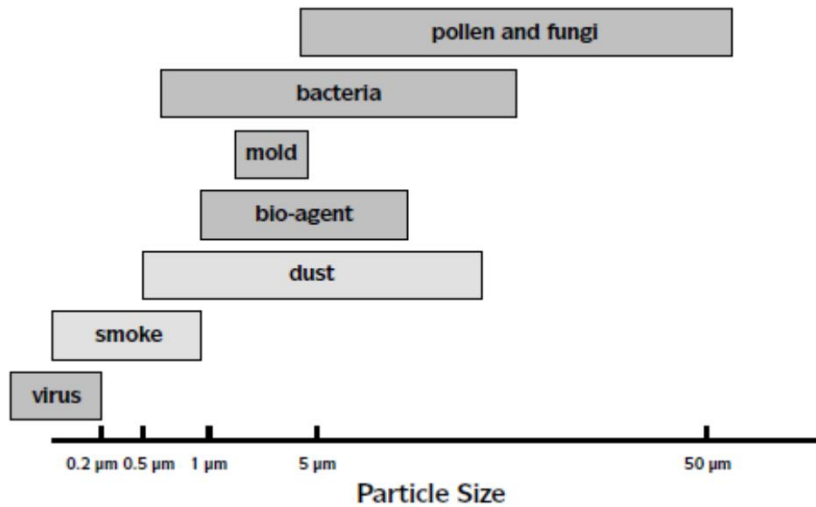
2011-04-30

18





# Fremtidige udfordringer – detektering af levedygtige partikler



*Fungi spores  
penicillium chrysogenum*



# Opsummering – Dansk partikel metrologi 2014

- § Danmark bidrager succesrig i internationale partikel aktiviteter (forskning, sammenligninger, standardisering)
- § Videnhjemtagning er essentielt for at fortsætte opbyggelsen af ekspertise og opretholde konkurrencedygtighed
- § Danske serviceydelser (i GTS fællesskab) inkluderer myndighedsbetjening, aerosolanalyse og –udmåling, akkrediterede kalibreringsydelser
- § I tæt samarbejde med industrielle partner motiveres og initieres videregående og relevant state-of-the-art forskning

