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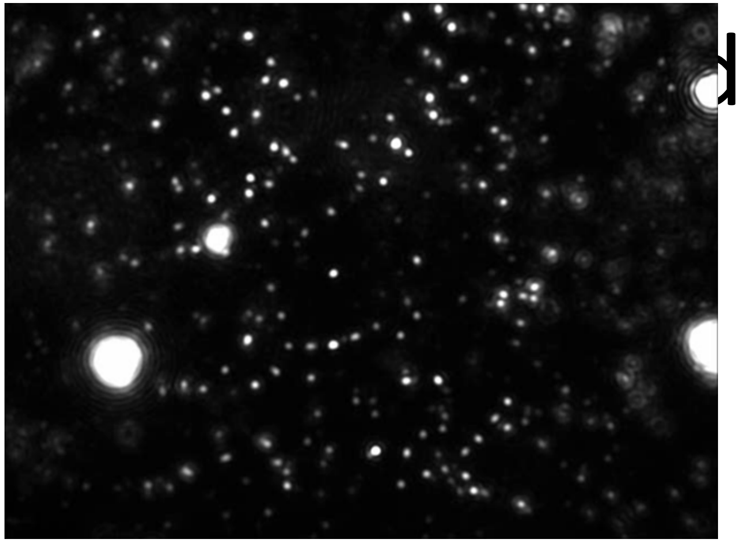
New techniques for nanoparticle characterization

ds



Dr. Victoria Coleman

Nanometrology Section, Physical Metrology Branch

National Measurement Institute Australia

IMRE Training Day, 1 July 2011

Specific trade names and company products are mentioned to adequately specify the experimental procedure and equipment used. Such identification does not imply recommendation or endorsement by the National Measurement Institute Australia, nor does it imply that the products are necessarily the best available for the purpose.

measurement.gov.au

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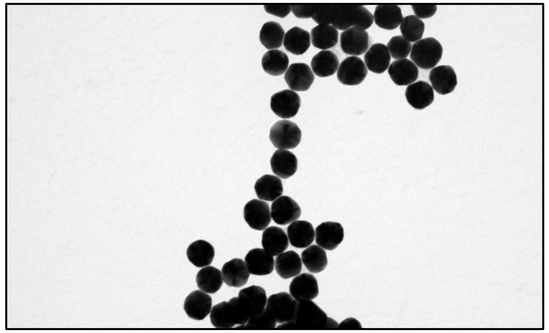
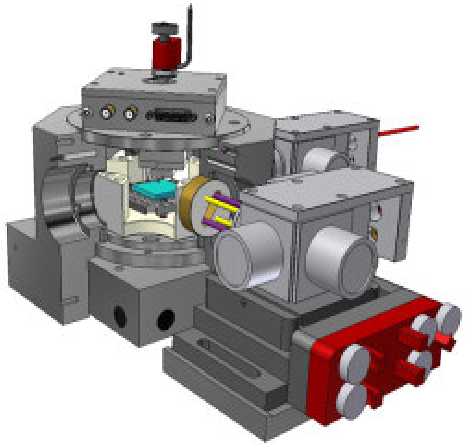
NMIA Nanometrology

Metrological SPM

Nanoparticle Characterisation Laboratory



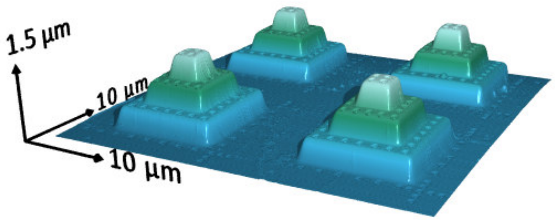


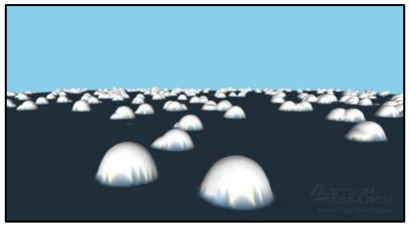
Gratings, step heights

Standard reference particles

- Initial focus particles suspended in liquids

- Traceability to the metre
- Calibration of SPM standards

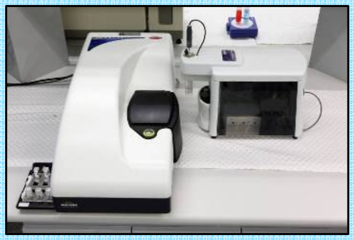




NMIA Nanoparticle Characterization Lab

Dimensional properties (size)

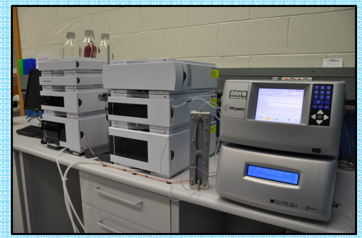
Light scattering

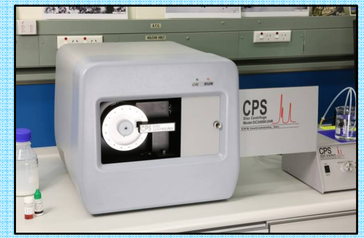


Static and dynamic light scattering,
Nanoparticle tracking analysis,
Laser diffraction

Electro zone sensing

Classification techniques



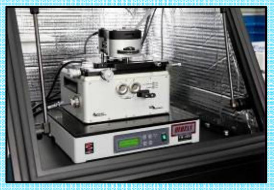


Field flow fractionation,
Differential centrifugal sedimentation,
Microseiving

Microscopy

Optical

Atomic force

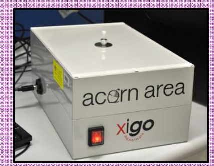


+ access to scanning and transmission electron microscopy facilities

Surface area & porosity

Gas adsorption

NMR



Surface charge

Streaming current potential,
Electrophoretic mobility

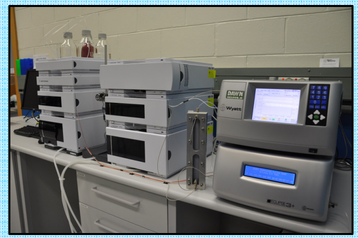
Mass/density

Microchannel resonator

Dimensional properties (size)

Light scattering

Classification techniques



Field flow fractionation,
Differential centrifugal sedimentation,

Electro zone
sensing

Nanoparticle tracking analysis

"New" characterization techniques

Surface area & porosity

NMR

Surface charge

Mass/density

Microchannel resonator

Light scattering

Dimensional properties (size)

Classification techniques

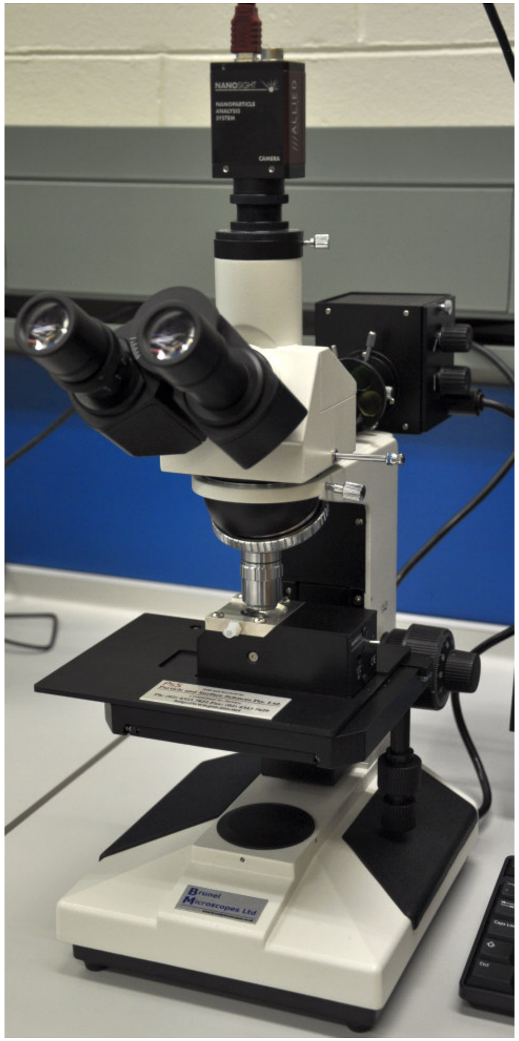
Nanoparticle tracking analysis

Differential centrifugal sedimentation

“New” characterization techniques

Mass/density

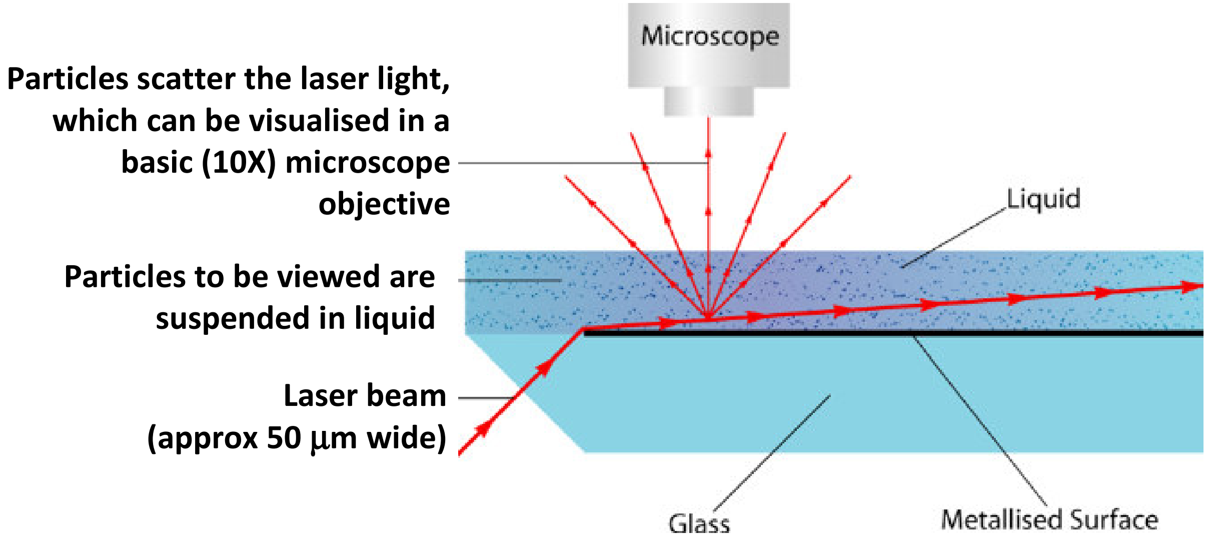
Microchannel resonator

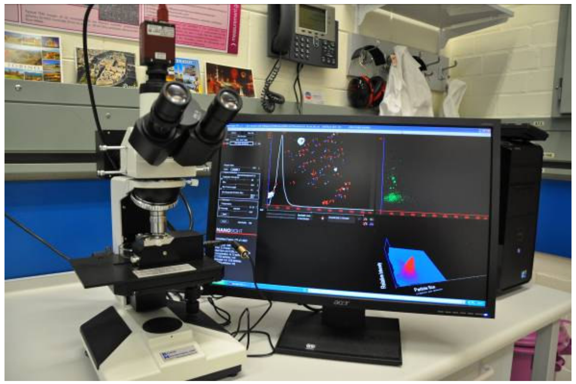


Nanoparticle tracking analysis

LM10

(Nanosight, UK)

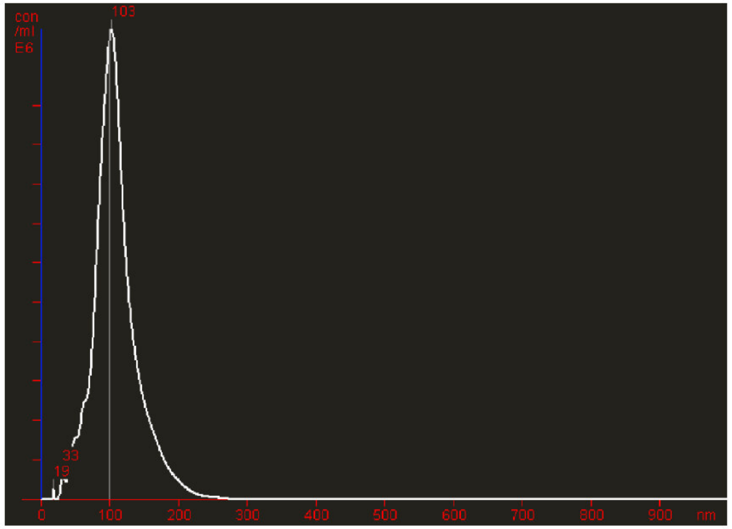




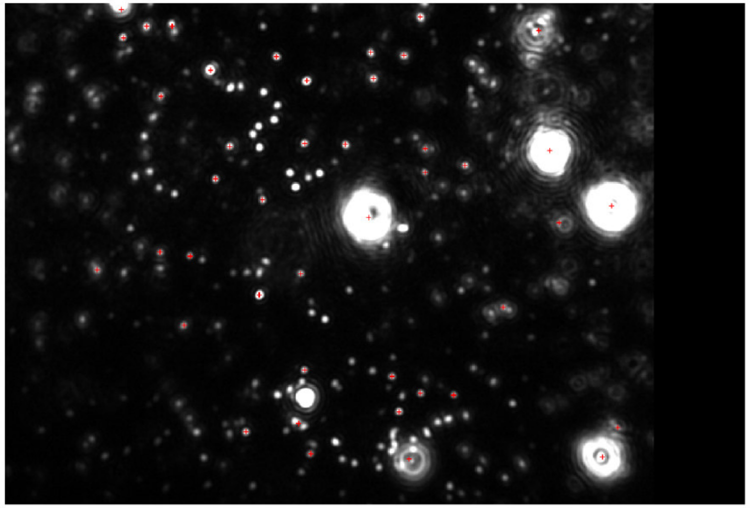
Fast facts: Nanoparticle tracking analysis

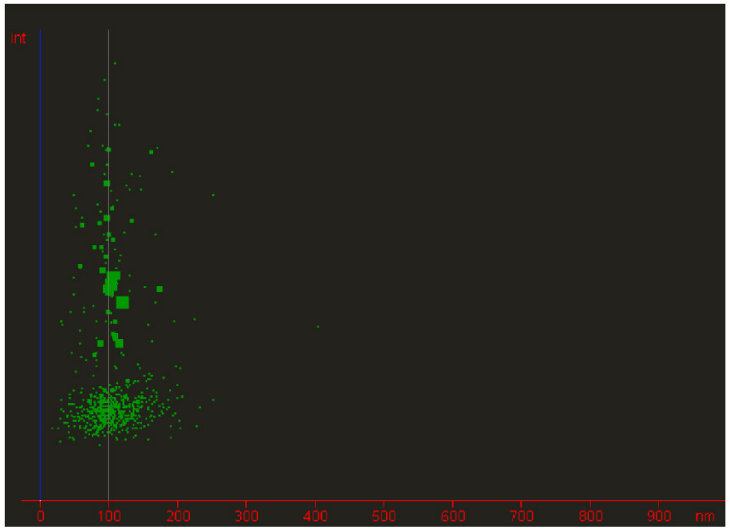
Size range	20 nm – 2µm (lower range is dependant on scattering properties and instrument configuration: @ 638 nm, PSL ~ 70 nm, Au ~20 nm)
Measurand	Diffusion length
Analysis principle	Brownian motion (Stokes-Einstein equation)
Key assumptions	Spherical particles, particle motion is in 2D
Concentration range	~1×10^8 particles per mL
Sample requirements	Fluid needs to be optically transparent
Pre-requisite knowledge	Measurement temperature, viscosity of dispersant*

Example results - NTA

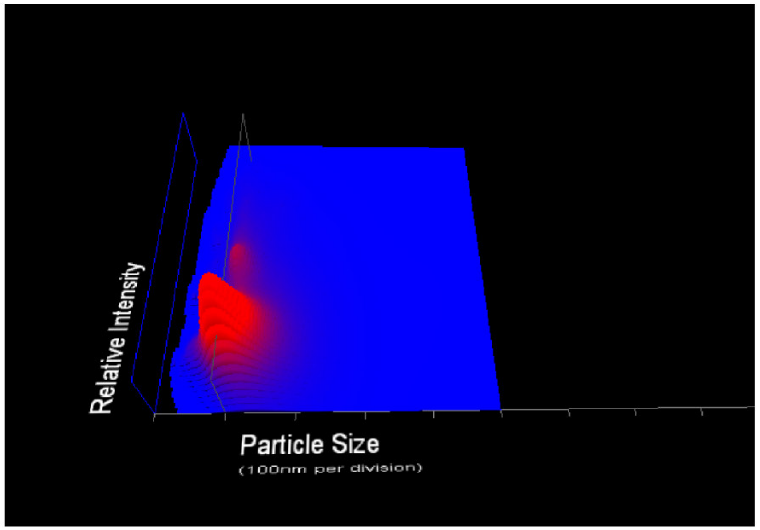


Particle Size / Concentration


Sample Video Frame



Particle Size / Relative Intensity



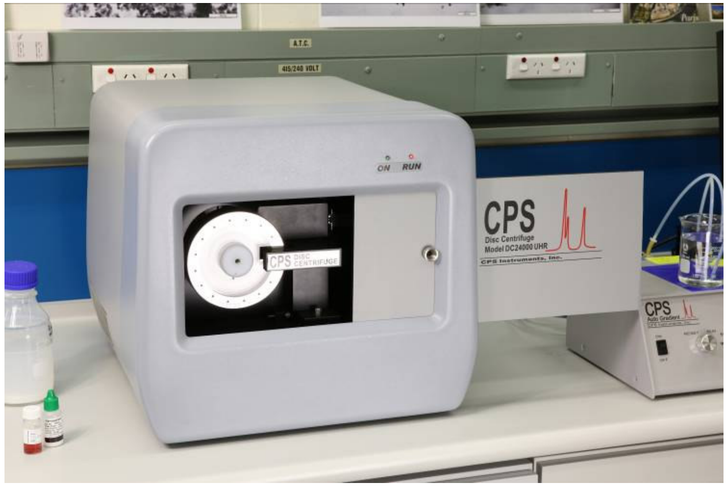
Particle Size / Relative Intensity 3D plot

Other Features – NTA

- Can measure particle number concentration (particles/mL)
- Can qualitatively differentiate between particles of different composition based on scattering intensity
- Different systems with different laser wavelengths, camera resolutions and temperature control are available

Limitations – NTA

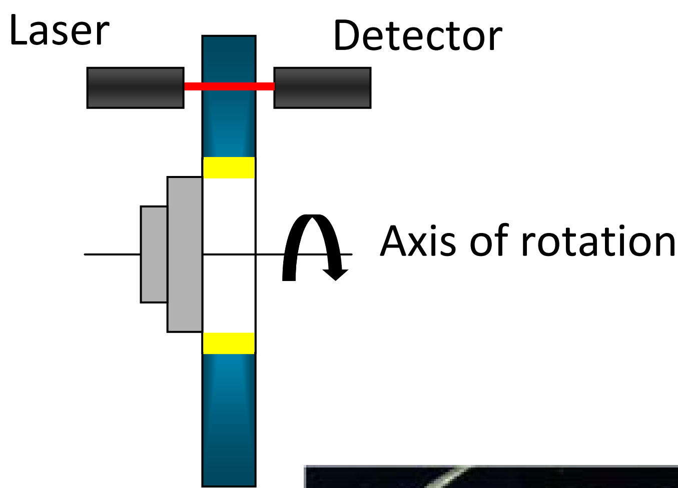
- Strong dependence on operator through choice of settings for imaging and analysis
- Limited statistical relevance
- Dilution

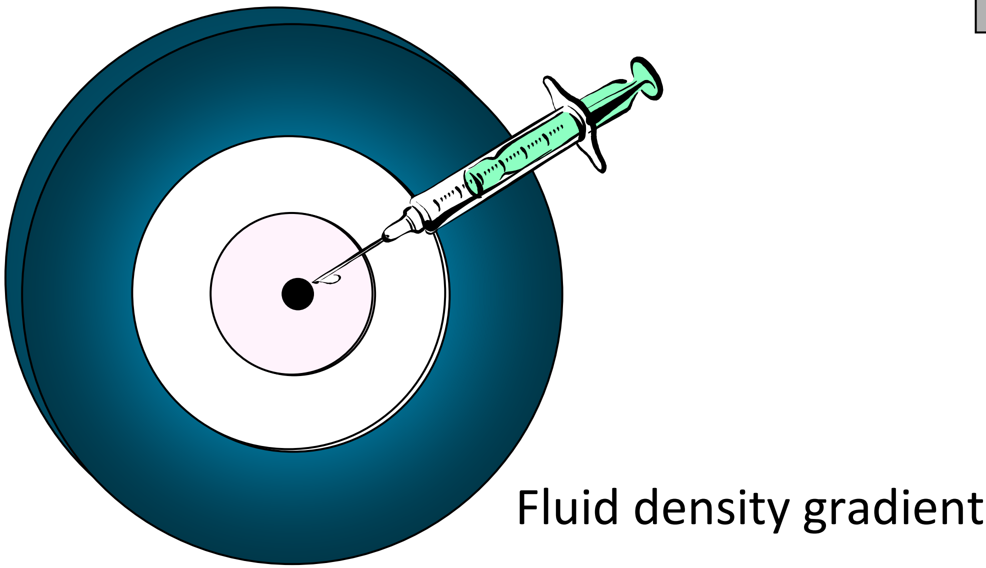


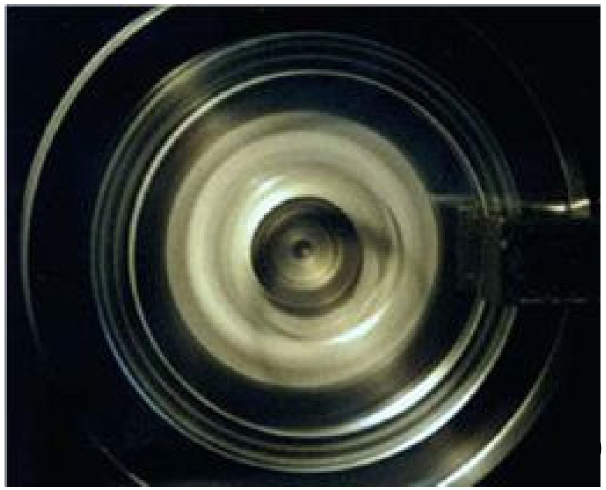
Differential centrifugal sedimentation (DCS)

24000UHR

(CPS Instruments, USA)



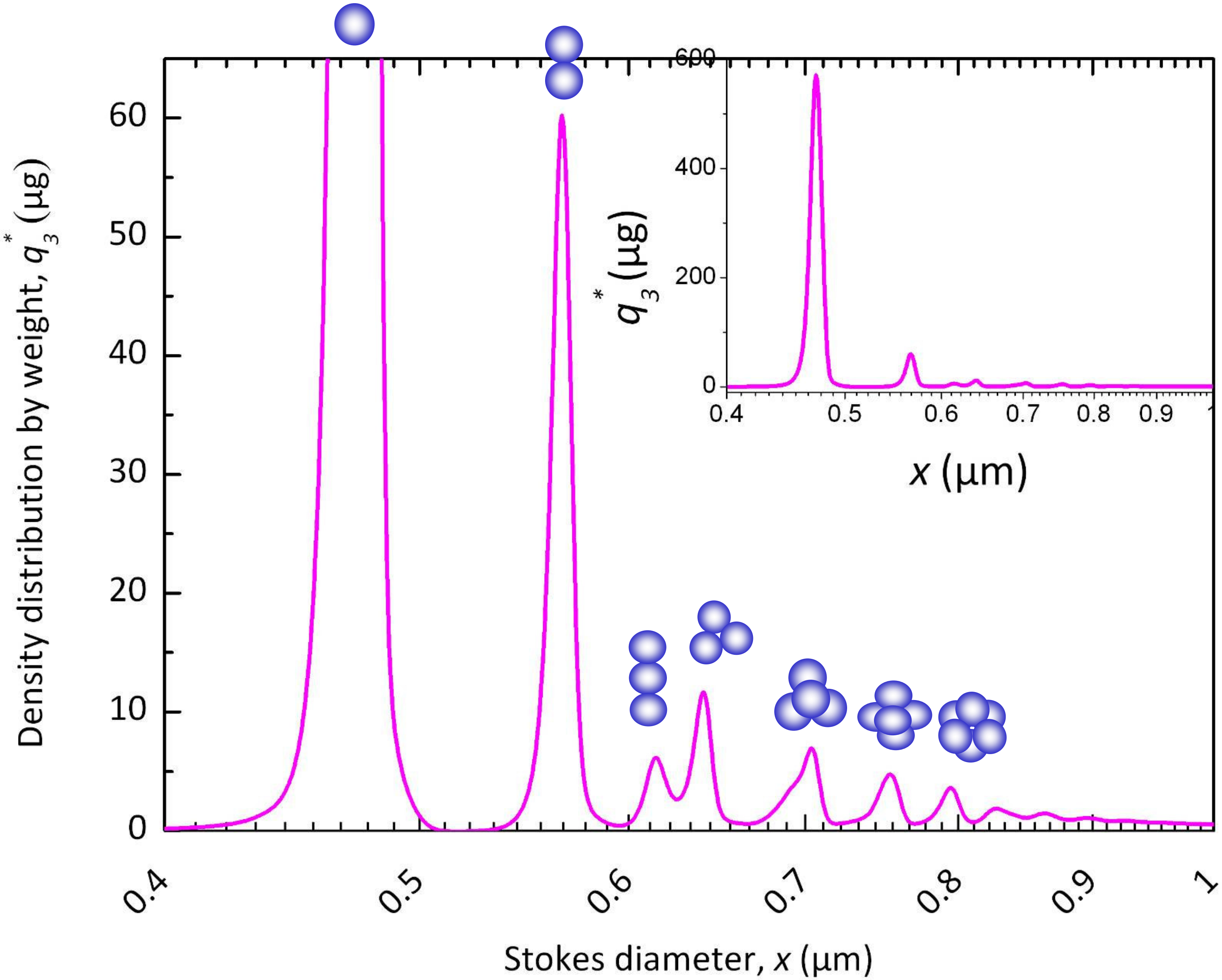




Fast facts: DCS

Size range	5 nm – 10µm (Rotation speed can be varied up to 24000 rpm)
Measurand	Sedimentation time from injection to detection
Analysis principle	Stokes sedimentation
Key assumptions	Laminar, uniform flow
Concentration range	~ 50 μgL^{-1} – $1mgL^{-1}$
Sample requirements	Sample must have a greater density than the fluid gradient and have homogeneous composition.
Pre-requisite knowledge	Particle **density** and the **optical properties** of particle and gradient fluid (to convert measured intensity distribution to weight and number distributions)

Example results – DCS



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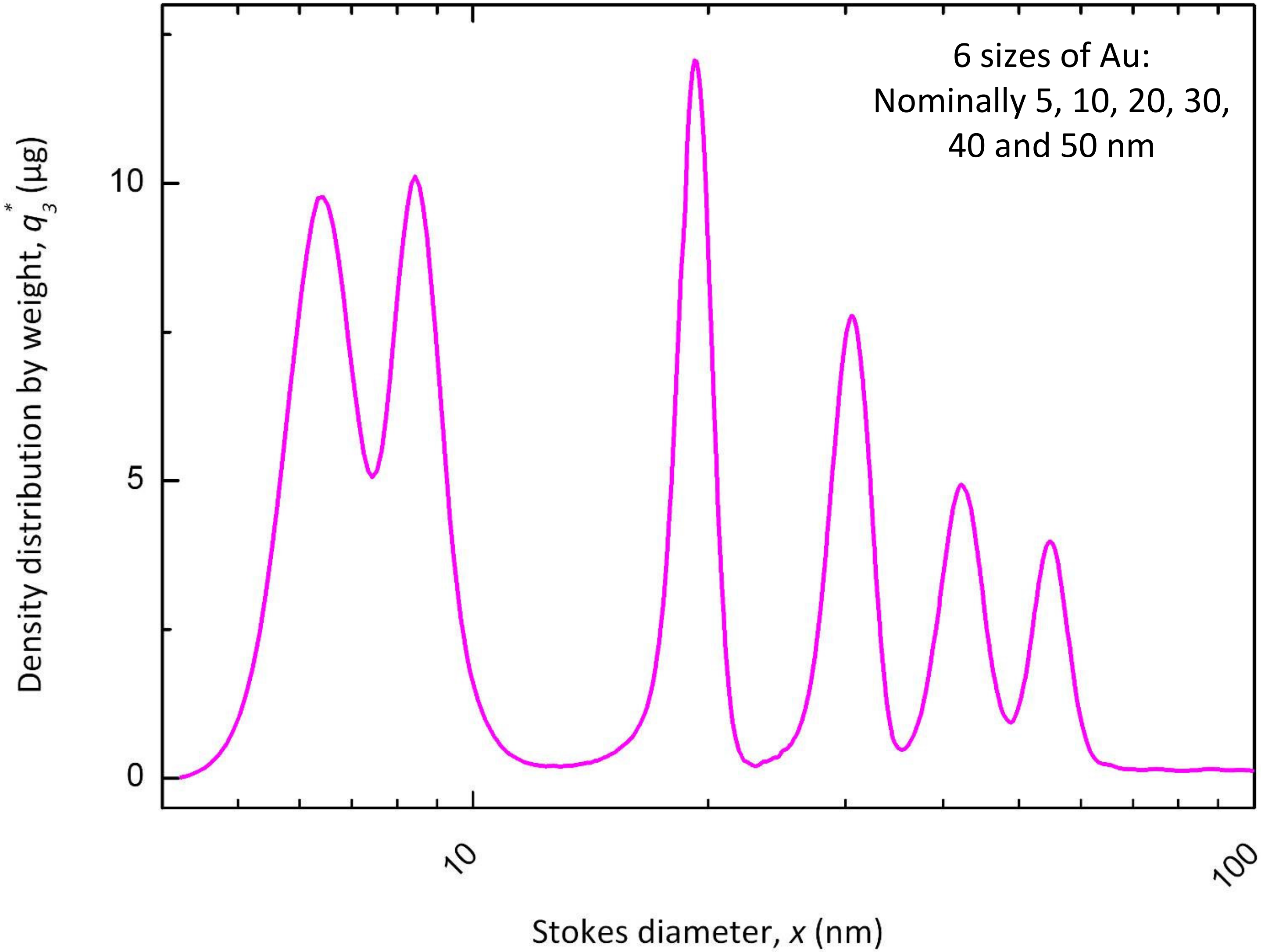
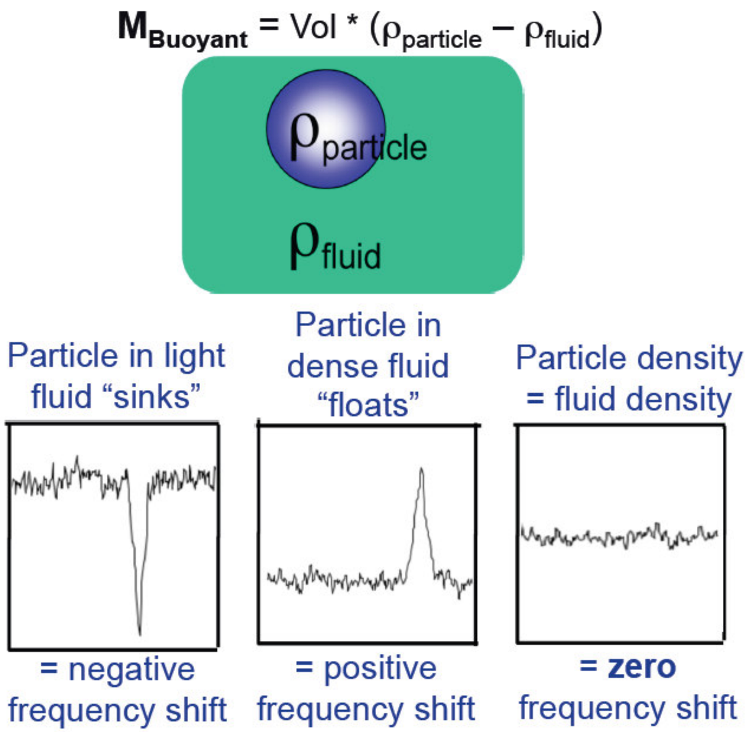
Example results – DCS



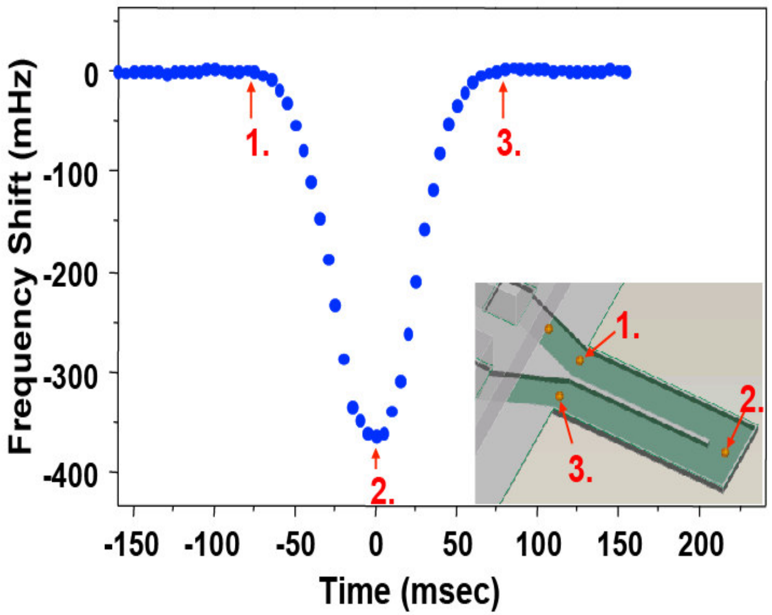
Limitations – DCS

- Sample needs to have homogeneous composition, density, porosity
- Optical properties of sample need to be known
- Calibration run must be performed before every measurement: lack of applicable certified reference materials

Microchannel Resonator
Archimedes particle metrology system
(Affinity Biosensors, USA)



Weighing a Particle



AFFINITY BIOSENSORS

Images courtesy of Ken Babcock, Affinity Biosensors

Fast facts: Microchannel resonator

Size range	60 nm – ~5µm Lower limit is dependant on particle density. Instrument sensitivity is ~1 Femtogram.
Measurand	Buoyant mass determined by a shift in the resonant frequency of an oscillating cantilever with a buried microfluidic channel
Analysis principle	Archimedes principle
Key assumptions	Spherical geometry, material homogeneity
Concentration range	$1\times10^{7} – 1\times10^{9}$ particles per mL
Sample requirements	Sample should be free from agglomerates/aggregates larger than 8 µm
Pre-requisite knowledge	Density of particle* and suspending fluid must be known

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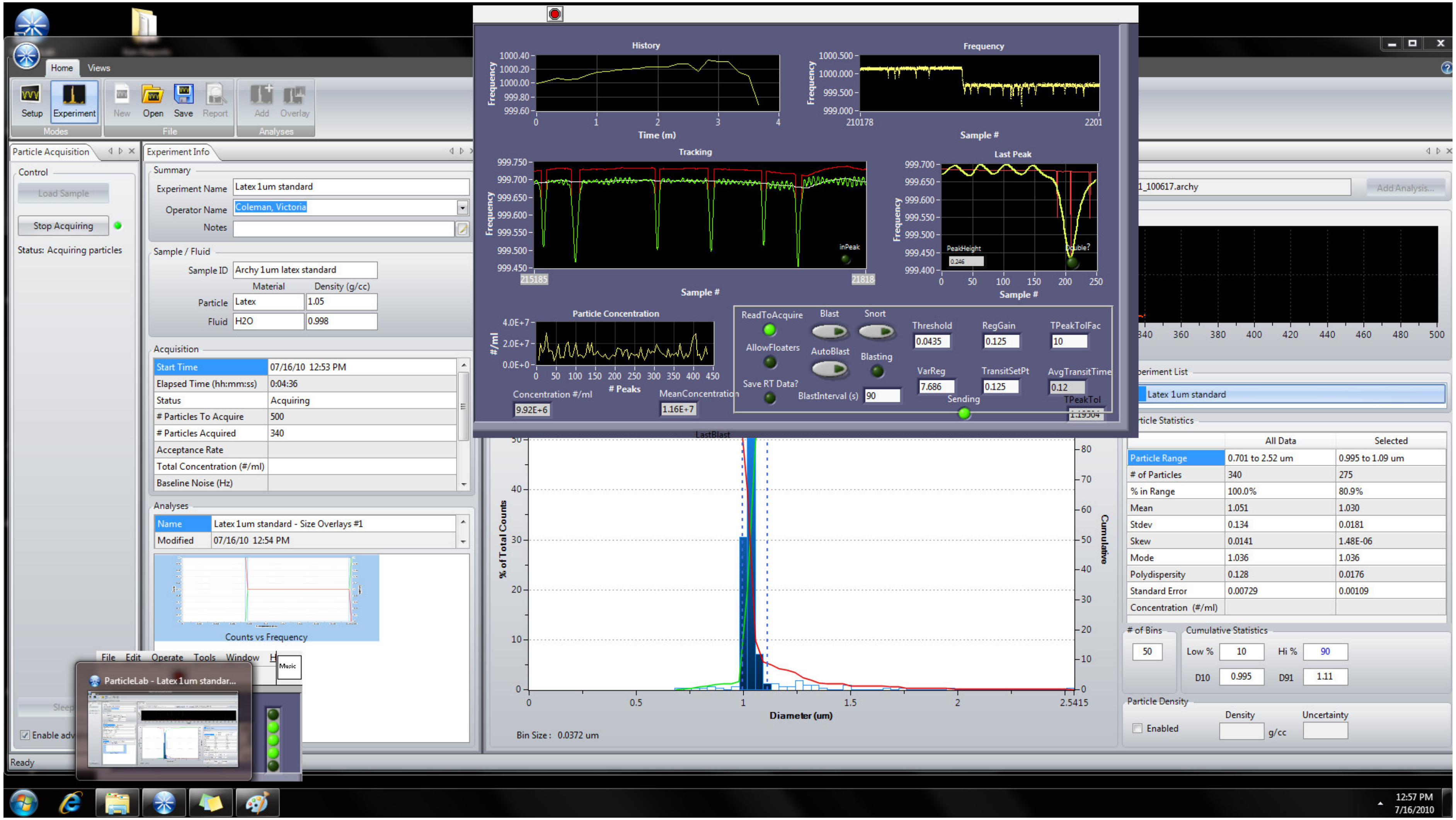
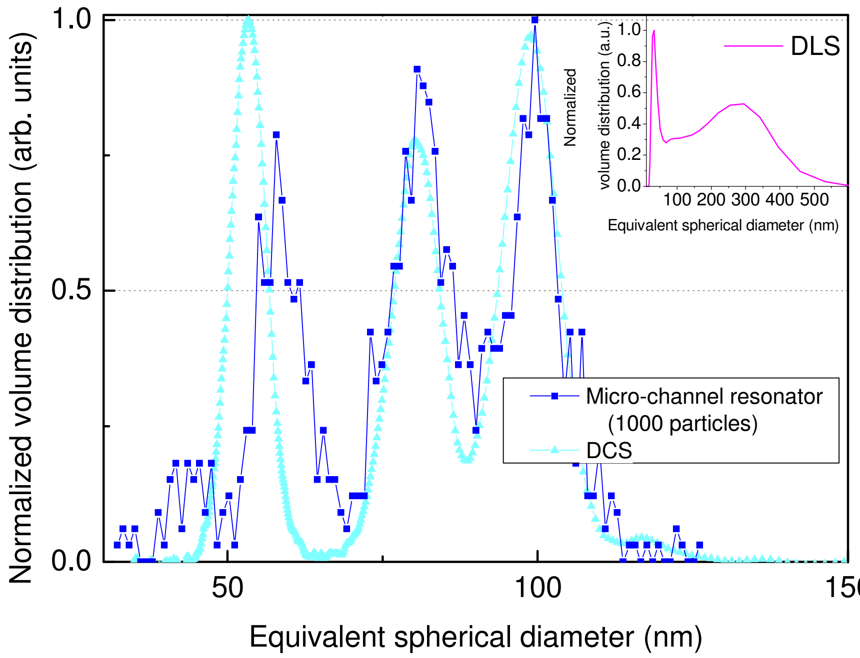
Example results – Microchannel resonator



More Features – Microchannel resonator

- Can measure 'floaters' – buoyant particles/bubbles
- Measurement in aqueous and non-aqueous fluids
- Can measure particle density
  - Measure same particle in two fluids of different known density (e.g. H_2O and D_2O)
- Can measure particle number concentration (particles/mL)
- And for the metrologists….
  - Simple model
  - Based on frequency measurements



Comparison of a trimodal Au sample measured by the microchannel resonator, disc centrifuge and dynamic light scattering

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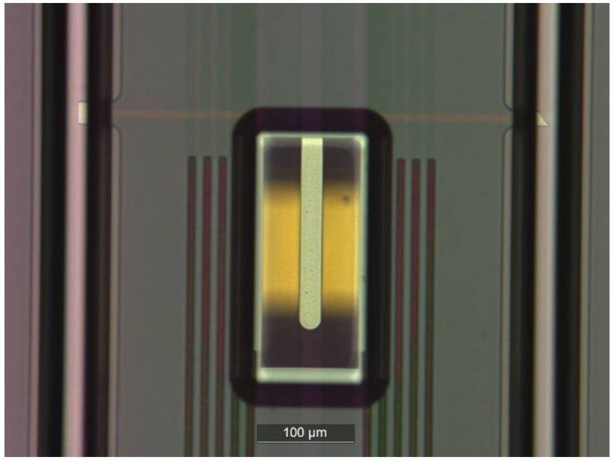
Limitations – Microchannel resonator

- Sensitivity is dependant on the channel size
  - Smallest channel currently available: 2 x 2 µm
  - For Au, this translates to a minimum size of ~60 nm
- Dilute suspensions required
- Aggregated samples may clog the sensor
- Sample needs to have homogeneous composition, density, porosity

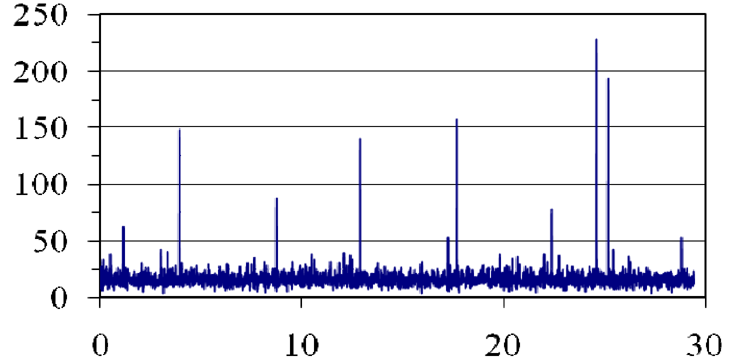


Other techniques

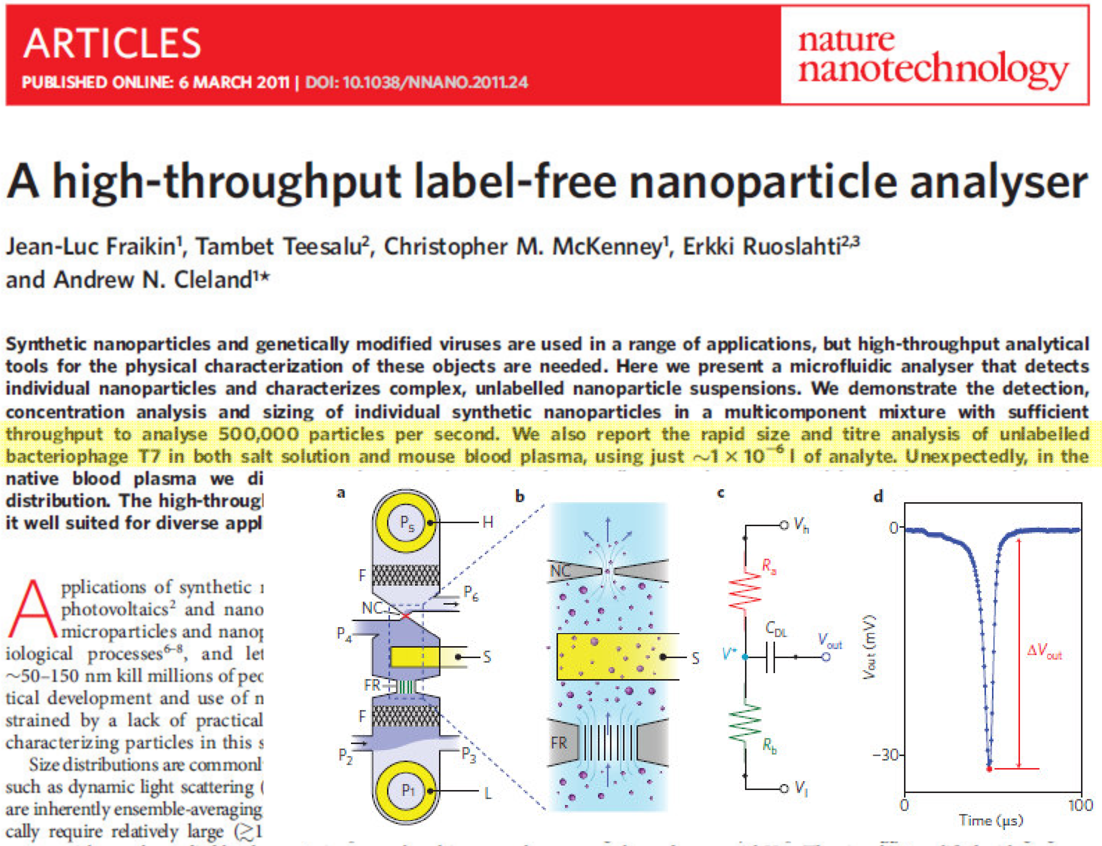
improvements are happening all the time!

iZon qNano (www.izon.com) – electrozone sensing with flexible nanopore

Real-time single-particle ICP-MS



Quantomix (www.quantomix.com) – wet cell SEM technology
40 and 400 nm Au and SiO2 particles in suspension


ARTICLES
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nature nanotechnology

A high-throughput label-free nanoparticle analyser

Jean-Luc Fraikin[1], Tambet Teesalu[2], Christopher M. McKenney[1], Erkki Ruoslahti[2,3] and Andrew N. Cleland[1*]

Synthetic nanoparticles and genetically modified viruses are used in a range of applications, but high-throughput analytical tools for the physical characterization of these objects are needed. Here we present a microfluidic analyser that detects individual nanoparticles and characterizes complex, unlabelled nanoparticle suspensions. We demonstrate the detection, concentration analysis and sizing of individual synthetic nanoparticles in a multicomponent mixture with sufficient throughput to analyse 500,000 particles per second. We also report the rapid size and titre analysis of unlabelled bacteriophage T7 in both salt solution and mouse blood plasma, using just ~1 × 10^{-6} l of analyte. Unexpectedly, in the native blood plasma we di distribution. The high-throug it well suited for diverse appl

Applications of synthetic photovoltaics[2] and nano microparticles and nanop iological processes[6–8], and let ~50–150 nm kill millions of pec tical development and use of n strained by a lack of practical characterizing particles in this Size distributions are commonl such as dynamic light scattering are inherently ensemble-averaging cally require relatively large (≳1

Silver bullet for particle sizing?

- As yet there is no single ideal characterisation technique
- All techniques make assumptions
- Different techniques measure different quantities
- Characterising particles in-situ in complex matrices or in concentrated slurries/suspensions is challenging
- Complex particle systems need complex descriptors – what is ‘size’?

Use as many techniques as possible: you can never know too much about your sample!

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