



What does it take to accurately measure concentration of nanoparticles in colloids

Jan “Kuba” Tatarkiewicz, PhD

VP Engineering

MANTA Instruments, Inc.



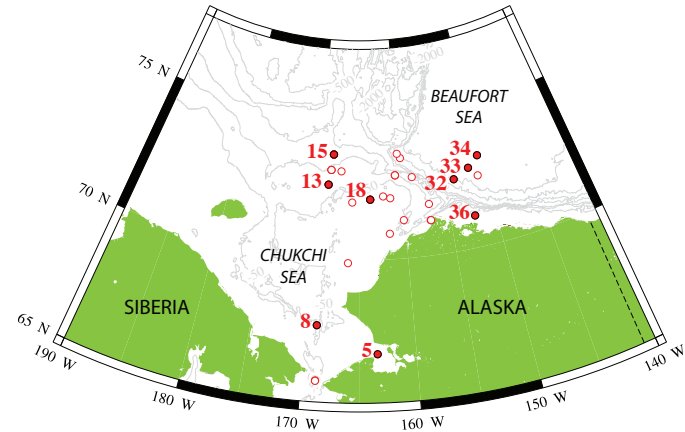
Counts

Volume

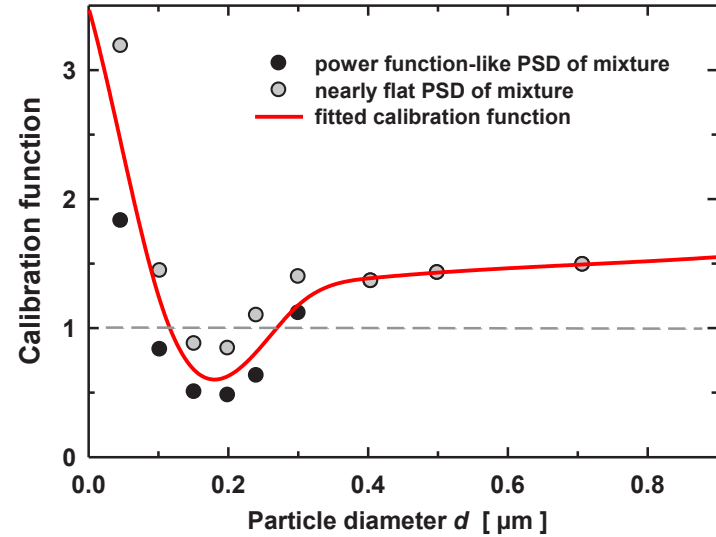
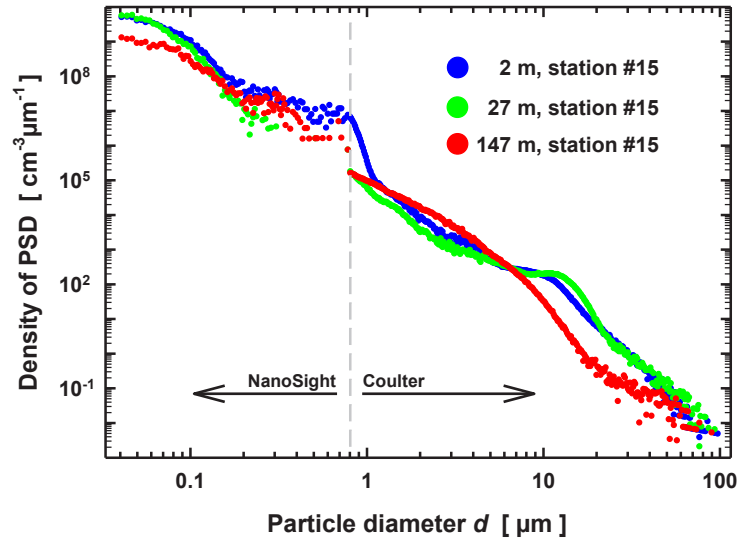
Beginnings



NASA sponsored ICESCAPE Project
two cruises on USCGC *Healy*
2010 - 2011



Results



J. J. Tatarikewicz, R. A. Reynolds, and D. Stramski *Counting and sizing of colloidal particles in the Arctic Ocean* 2012 Ocean Sci Meeting A0412

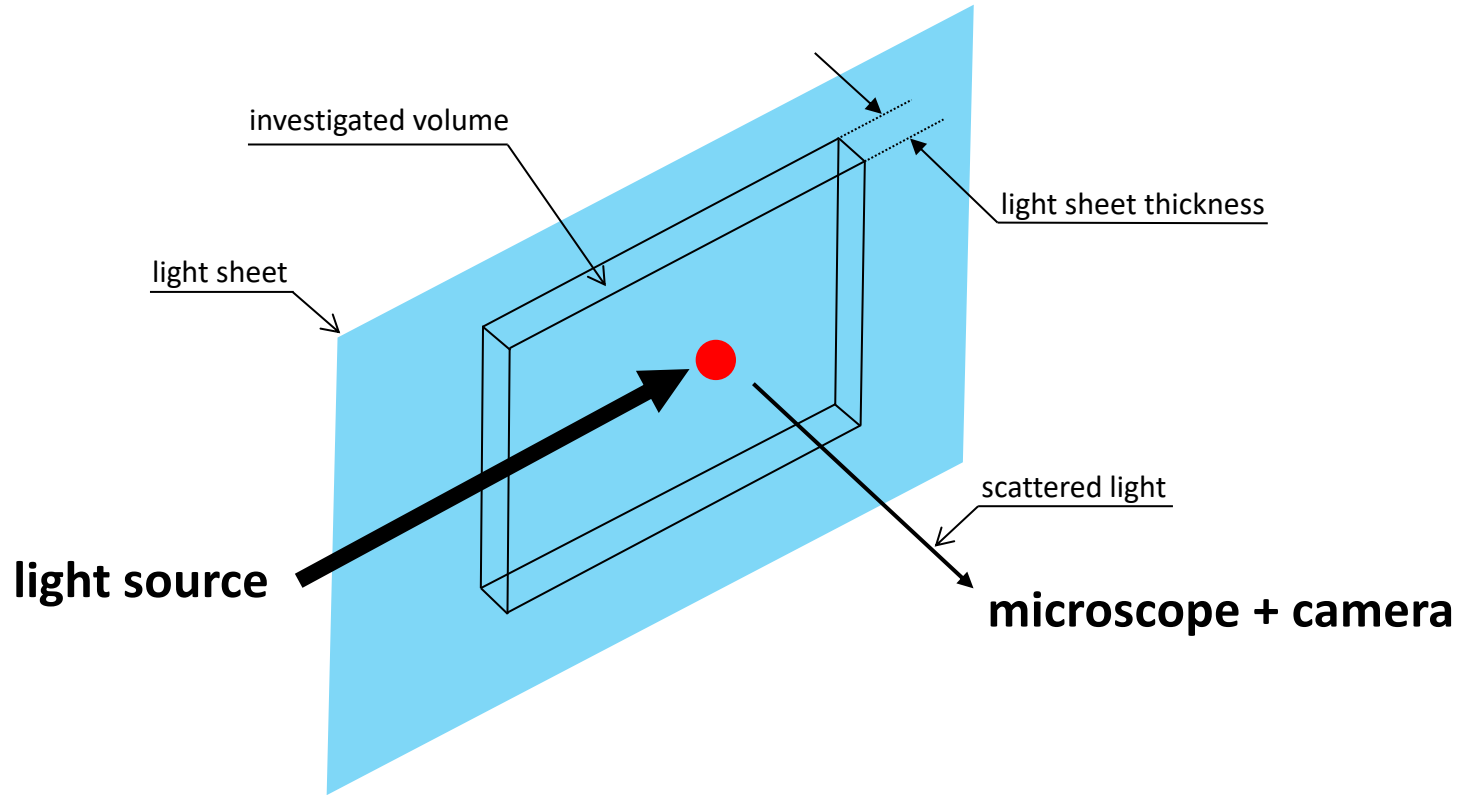
This is a very strange function...

2nd generation NTA

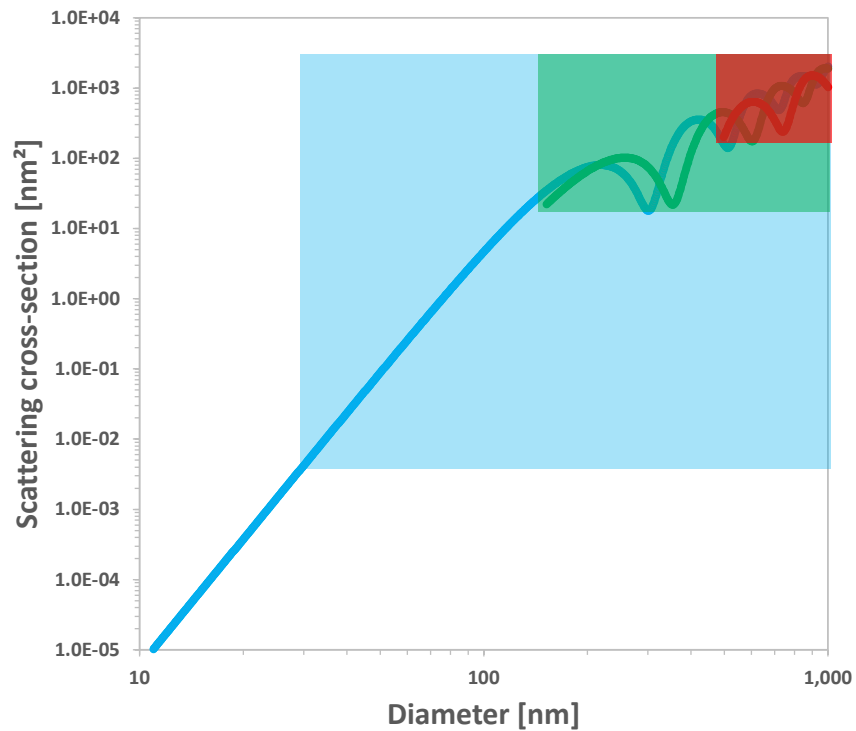


- **M**ultispectral **A**dvanced **N**anoparticle **T**racking **A**nalysis
- NSF grant for MRI #1126870, 2012-2014
- MANTA Instruments, Inc. founded in 2014
- US patents granted up to now:
 - 9541490, 9645070, 9857283, 9909972

Visualization



MANTA



Mie calculations for 445 nm, 520 nm and 635 nm polarized laser beams scattering on PSL in water ($n=1.337$), objective NA=0.28, $80^\circ\div 100^\circ$ integration

Sizes



- Mean Squared Distance MSD (2D, N frames track, n frames lag*):

$$MSD(n) = \frac{1}{N-n} \sum_{i=1}^{N-n} (x_{i+n} - x_i)^2 + (y_{i+n} - y_i)^2$$

- Diffusion coefficient D (least-squares fit of MSD as a function of n):

$$MSD(n) = (4 \cdot \Delta t \cdot D) \cdot n$$

- Hence hydrodynamic diameter: $d_h = \frac{k_B T}{3\pi\eta D}$

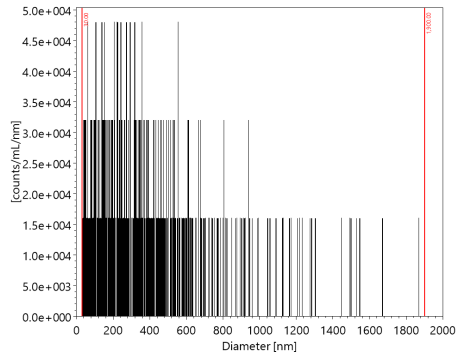
* ergodicity: assembly average \equiv time average

Statistics

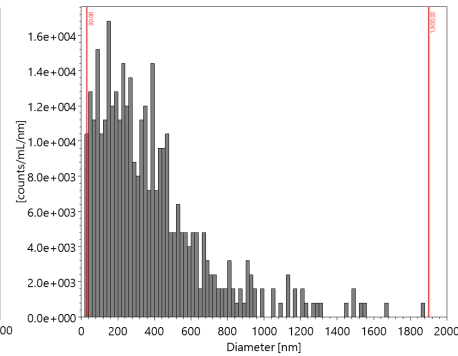


- Cramér-Rao statistics decides length of each track used for optimal MSD fitting
 - X. Michalet and A.J. Berglund *Optimal diffusion coefficient estimation in SPT*, Phys Rev **E85**, 061916 (2012)
- Binning diameters with different schemes (like equal or logarithmic widths) into density of particle-size distribution (PSD) with **variable investigated volume** (*explained later*)
- Statistical parameters of PSD (average size, standard deviation)

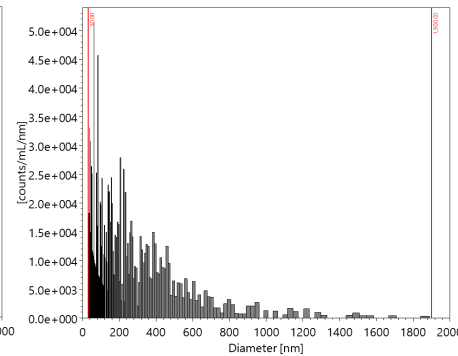
Mode?



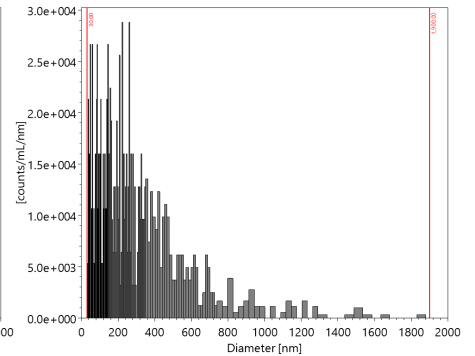
narrow equal bins



wide equal bins



logarithmic bins



variable bins

Counts



- 25 (*or more*) short videos (*300 frames each*) recorded*
- Track and count particles detected on 1st frame of each video
- Mixing sample between videos to get different aliquots (magnetic stirrer)
 - external fluidics for magnetic materials and low concentration samples – do **not** use sample flow during recording
- Proper PSD binning (*bin widths*) for polydispersity

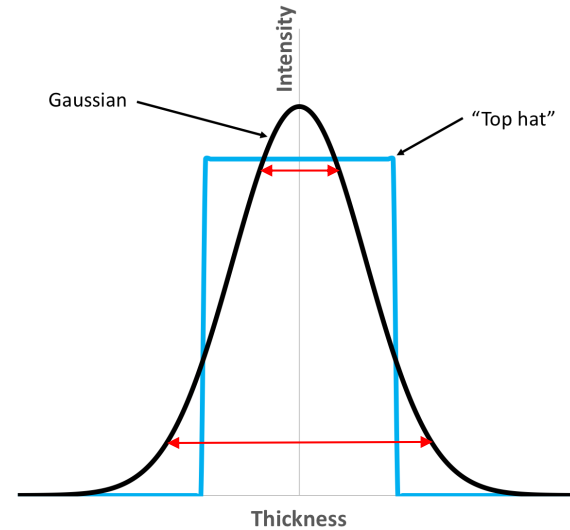
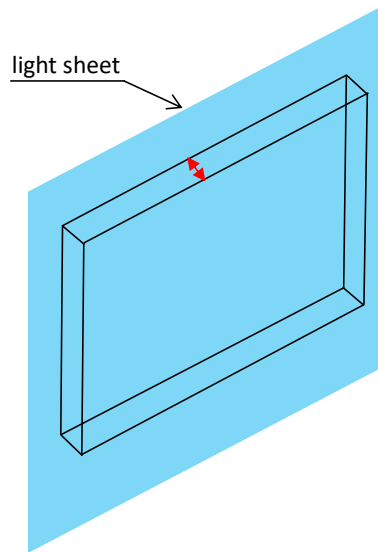
* typically between 100 and 150 particles tracked per video

Thickness



$$C = \frac{N}{V} \left[\frac{\text{counts}}{\text{mL}} \right]$$

$$V_0 \approx 2.5 nL$$



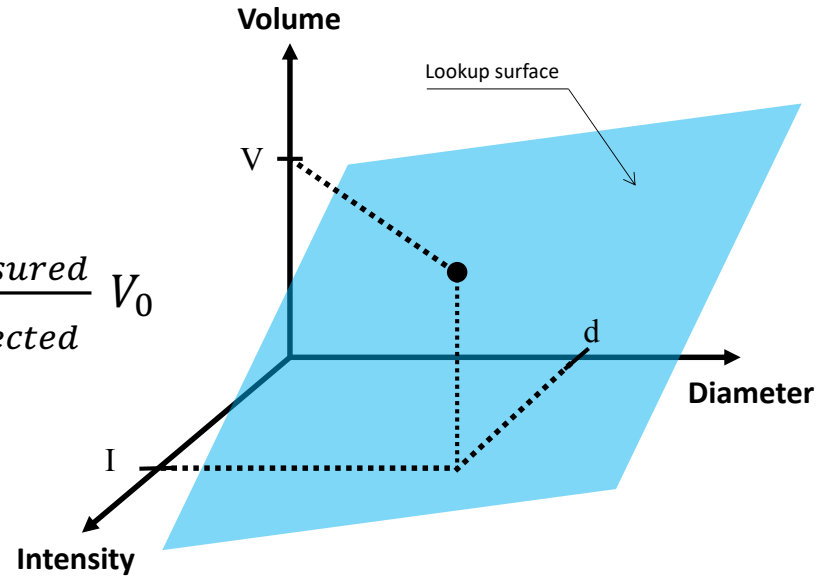
Volume



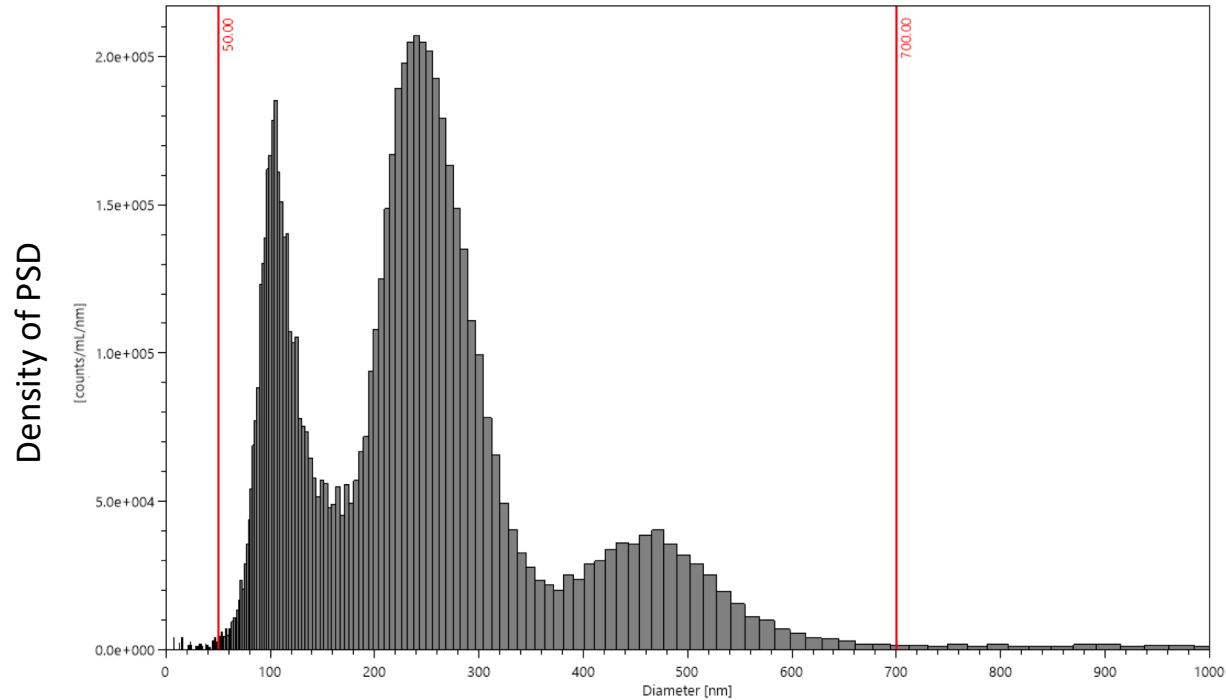
How to calibrate volume:

- Measure concentrations for standards of different sizes and made out of different materials (various RIs)
- Determine effective volumes
- Create look-up surface of volumes
- Extrapolate by using intensity of individual tracks and applying Mie scattering cross-section formula

$$V = \frac{N_{measured}}{N_{expected}} V_0$$



Histogram



Concentration from 50 nm to 700 nm = area of density of PSD histogram

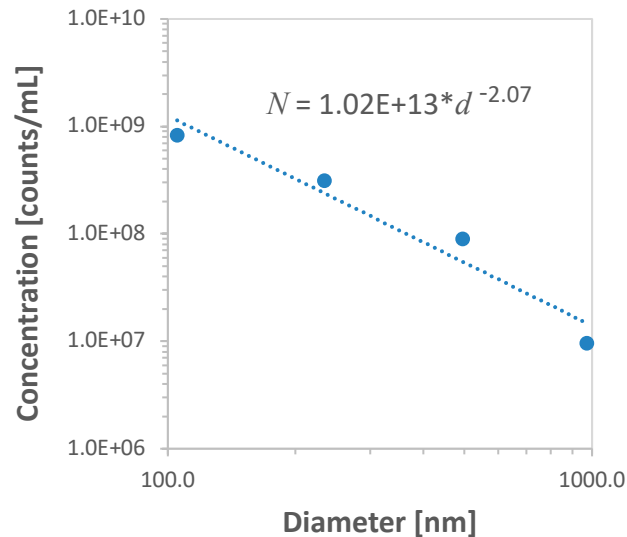
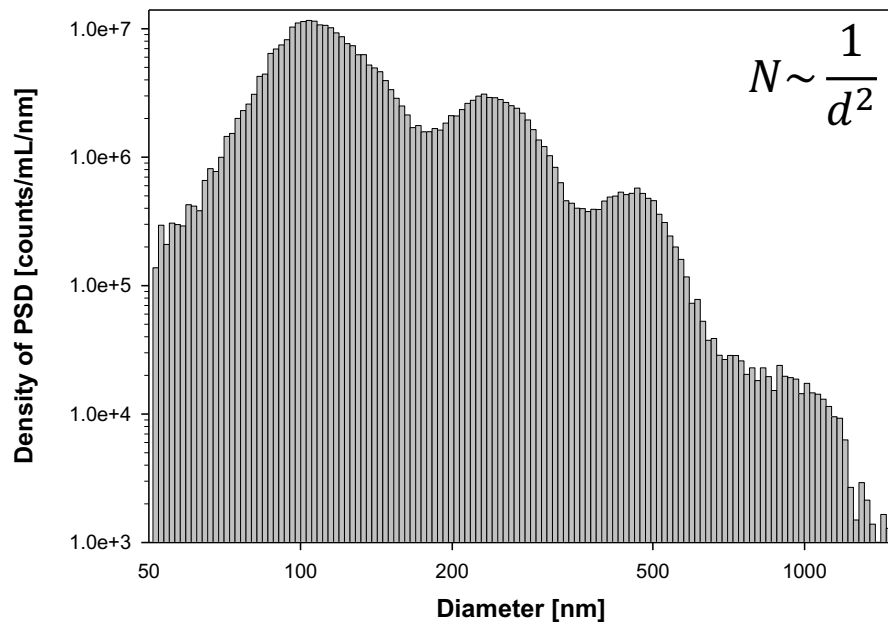
$$\delta_i = \sum_m \frac{1}{b_i \cdot V_m}$$
$$C_{kl} = \sum_{i=k}^l \delta_i \cdot b_i = \sum_{i=k}^l \sum_m \frac{1}{V_m}$$

Concentration



- Integrate density of PSD ($counts/mL/nm$) across sizes of interest, for example from 50 to 700 nm, to get concentration ($counts/mL$)
- Instruments are calibrated for optics scaling (nm/pix) and for laser(s) power (mW) (*manufacturing variability of active elements*)
- For unknown materials, extrapolate investigated volumes by using Mie scattering cross-sections of known test materials
- Use measured data with statistically significant number of counts, do **not** use fitted distributions (*PSD is **not** an invariant*)

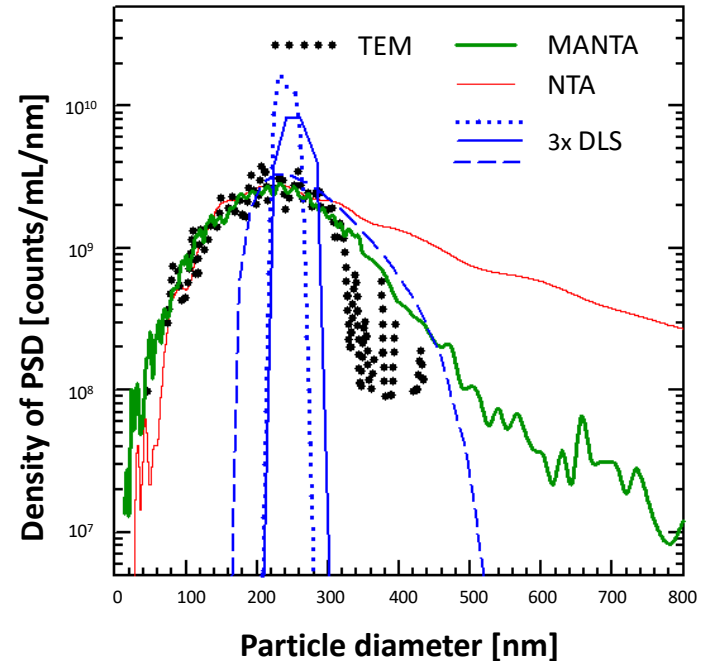
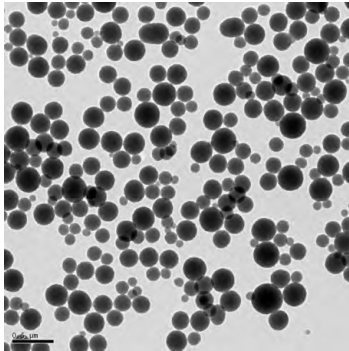
NIST exploratory mix



TEM, DLS & NTA vs. MANTA



α-lactalbumin nanoparticles
made as per Arroyo-Maya et al.
J Dairy Sci **95**, 6204 (2012)





Thank you

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