Characterizing Spherical Nanoparticles by Small-Angle X-ray Scattering to Validate New Interferometry Techniques

SURF Summer Colloquium 2021

Daven Shah

Gaithersburg Campus | NIST

Background

Small Angle Scattering (SAS) vs. Far Field Interferometry (INFER)

- Both identify structural features between 1nm – 1µm

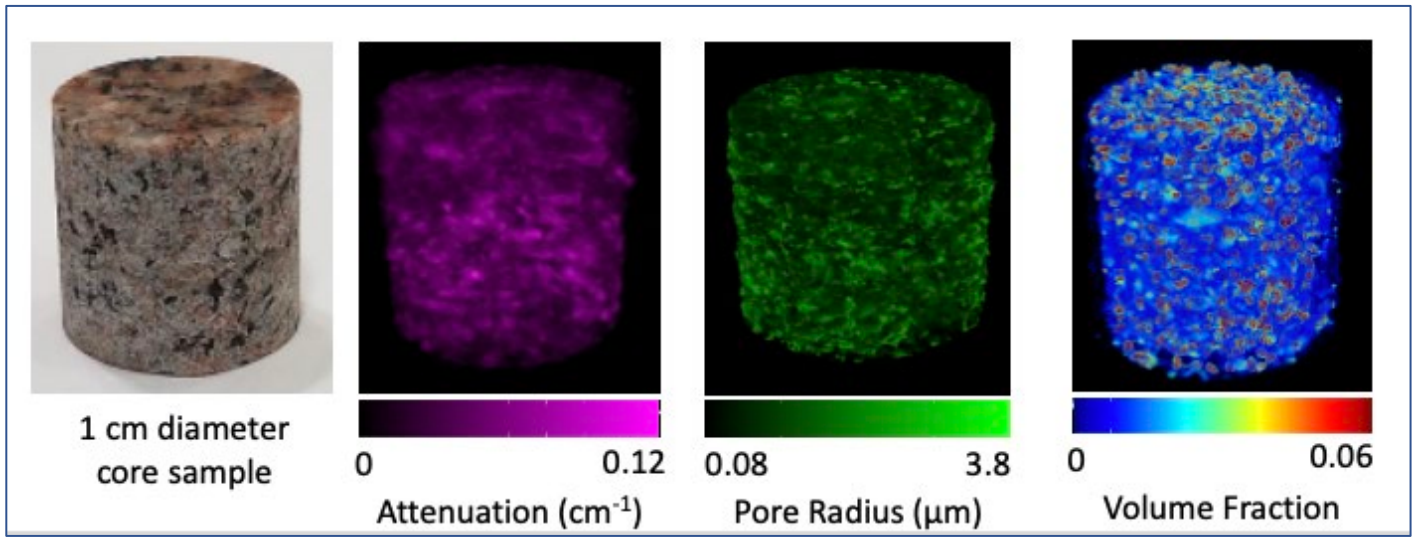
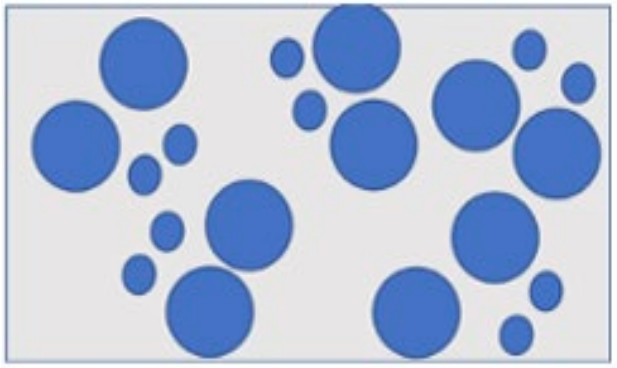
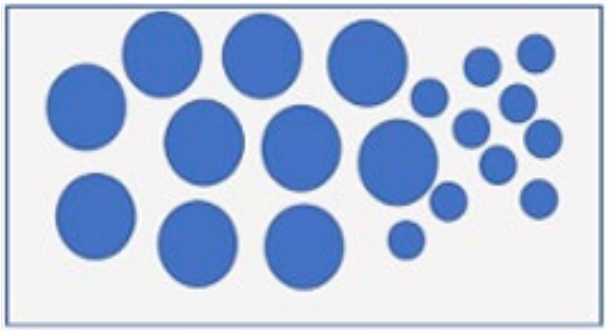


Image Credit: Dan Hussey. NCNR Cold Imaging Station.



SANS/SAXS Interpretation



INFER Interpretation

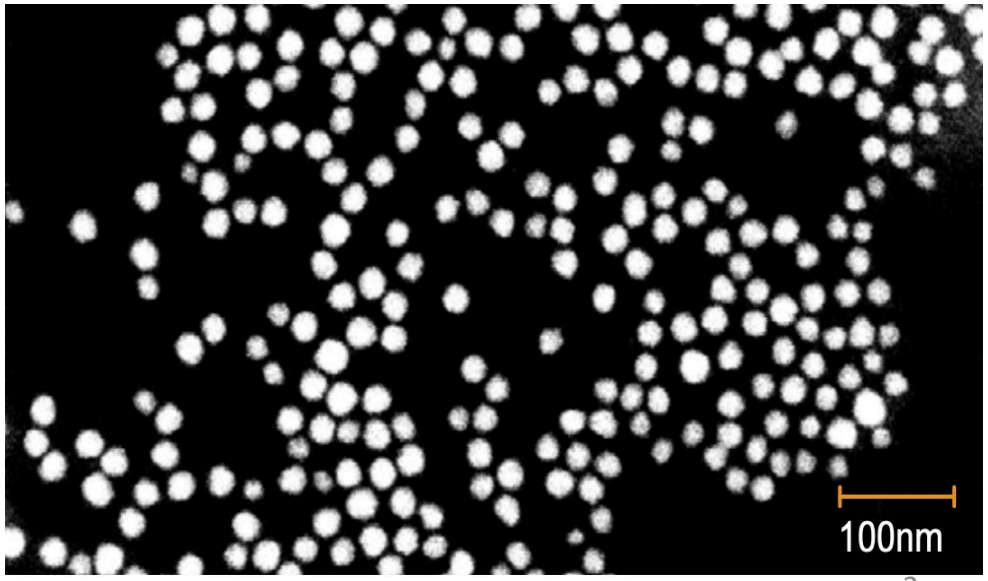
Project goals:

- Construct Library of scattering data for spherical nanoparticles
- Validate INFER data based on scattering data
- Further analyze systems of nanoparticles other than spheres for validation

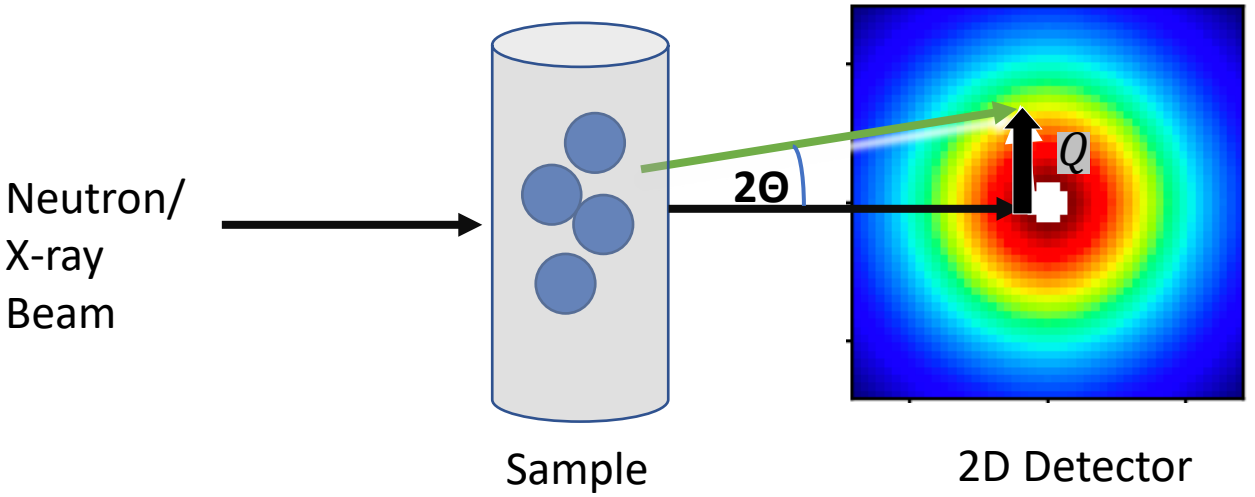
Ludox Silica Spherical Nanoparticles

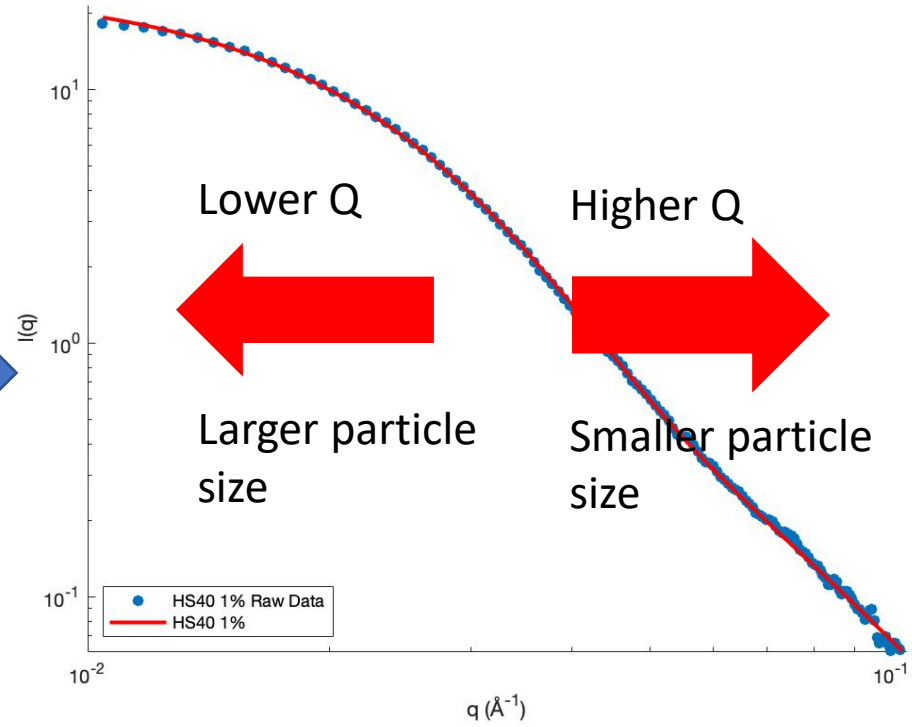
- Well Characterized
- Generally Monodispersed
- Have a charge and counterion

Ludox	Listed Radius (nm)	Net Charge	Counterion
SM	3.5 nm	-	Na^+
LS	6 nm	-	Na^+
CL	6 nm	+	Cl^-
HS40	6 nm	-	Na^+
TM50	11 nm	-	Na^+



Small Angle Scattering



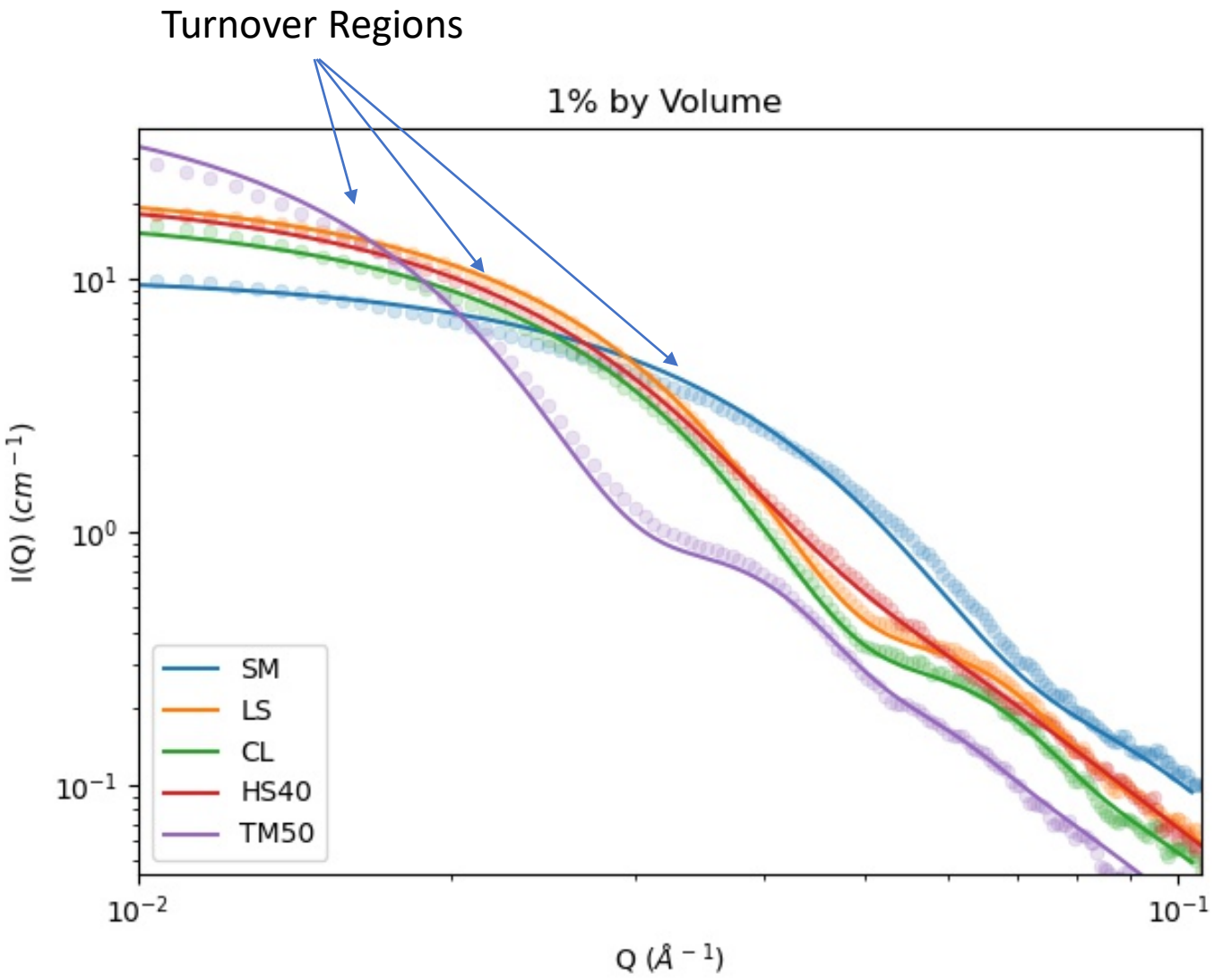


Intensity vs Q plot

$$Q = \frac{2\pi}{d} = \frac{4\pi}{\lambda}\sin(\theta)$$

I(Q) ~ P(Q) S(Q)
P(Q): Form Factor
S(Q): Structure Factor

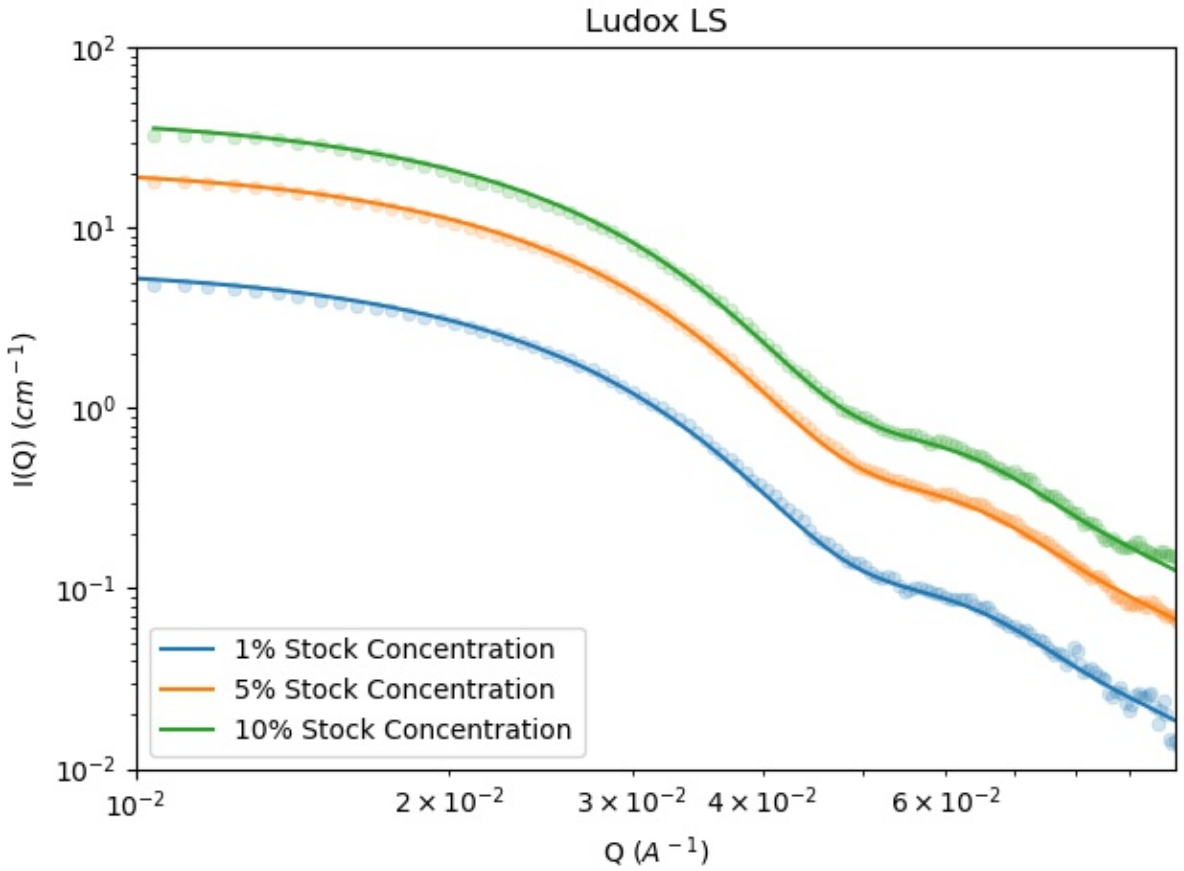
Understanding The Sphere Form Factor



Ludox	Scale	Radius (nm)	Polydispersity
SM	$4.59 \times 10^{-3} \pm 1 \times 10^{-5}$	4.51±0.01	0.292
LS	$4.81 \times 10^{-3} \pm 1 \times 10^{-5}$	7.94±0.01	0.160
CL	$3.80 \times 10^{-3} \pm 1 \times 10^{-5}$	7.94±0.01	0.165
HS40	$4.52 \times 10^{-3} \pm 1 \times 10^{-5}$	6.57±0.01	0.350
TM50	$3.96 \times 10^{-3} \pm 1 \times 10^{-5}$	13.02±0.01	0.141

LS Concentration Series: Form Factor Parameters

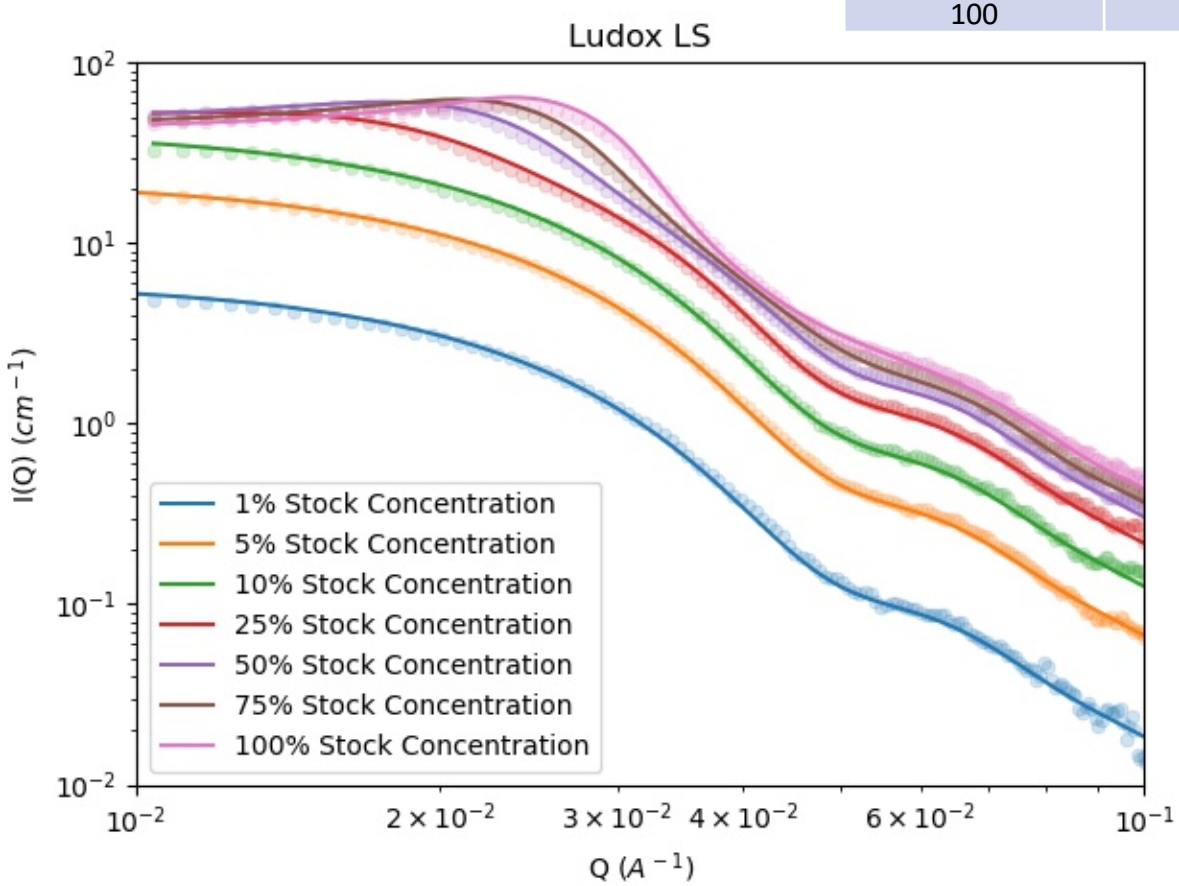
% Stock Concentration	Scale	Radius (nm)	Polydispersity
1	$1.306 \times 10^{-3} \pm 4 \times 10^{-6}$	7.925	18.00%
5	$4.76 \times 10^{-3} \pm 1 \times 10^{-5}$	7.925	18.00%
10	$8.92 \times 10^{-3} \pm 3 \times 10^{-5}$	7.925	18.00%



I(Q) ~ P(Q)
P(Q): Form Factor

LS Concentration Series: Form Factor Parameters

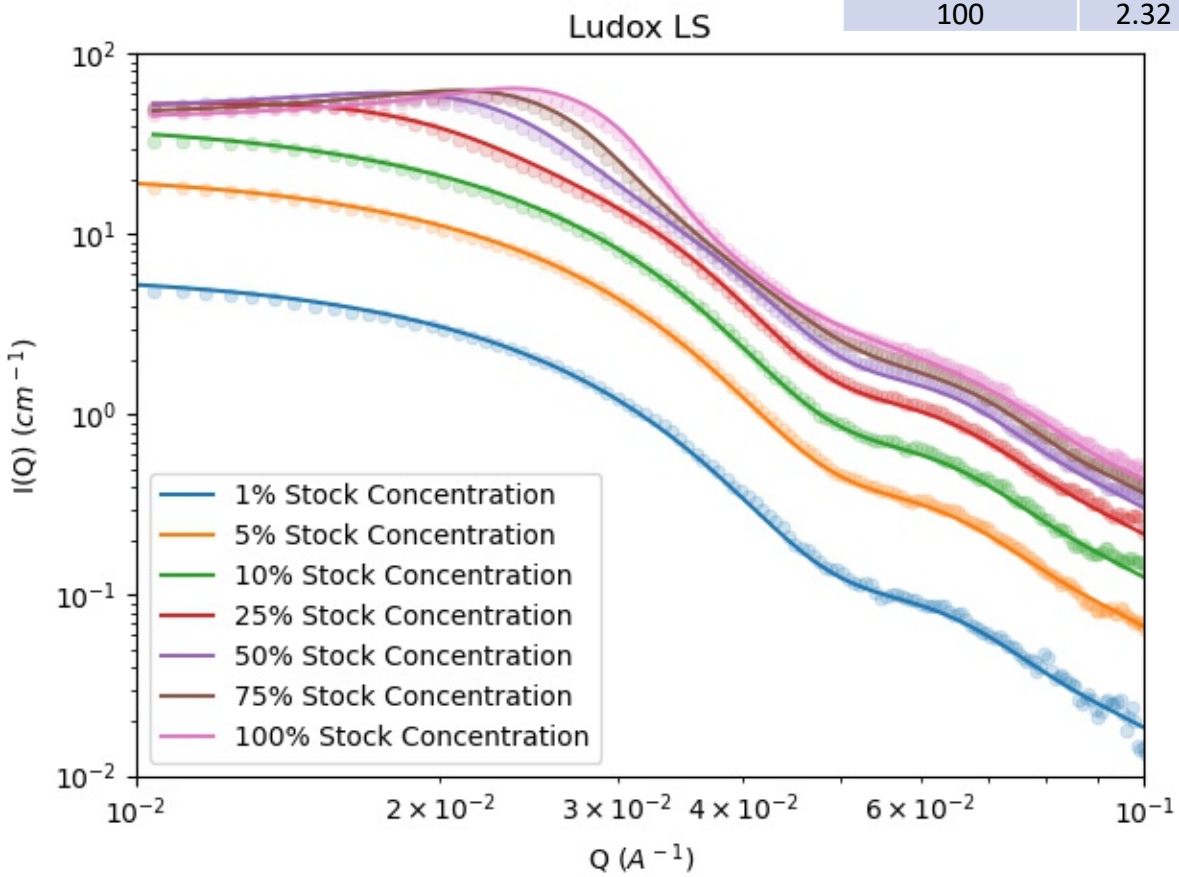
% Stock Concentration	Scale	Radius (nm)	Polydispersity
1	$1.306 \times 10^{-3} \pm 4 \times 10^{-6}$	7.925	18.00%
5	$4.76 \times 10^{-3} \pm 1 \times 10^{-5}$	7.925	18.00%
10	$8.92 \times 10^{-3} \pm 3 \times 10^{-5}$	7.925	18.00%
25	$5.3 \times 10^{-1} \pm 2 \times 10^{-2}$	7.925	18.00%
50	$3.40 \times 10^{-1} \pm 4 \times 10^{-3}$	7.925	18.00%
75	$2.69 \times 10^{-1} \pm 2 \times 10^{-3}$	7.925	18.00%
100	$2.32 \times 10^{-1} \pm 2 \times 10^{-3}$	7.925	18.00%



I(Q) ~ P(Q)
P(Q): Form Factor

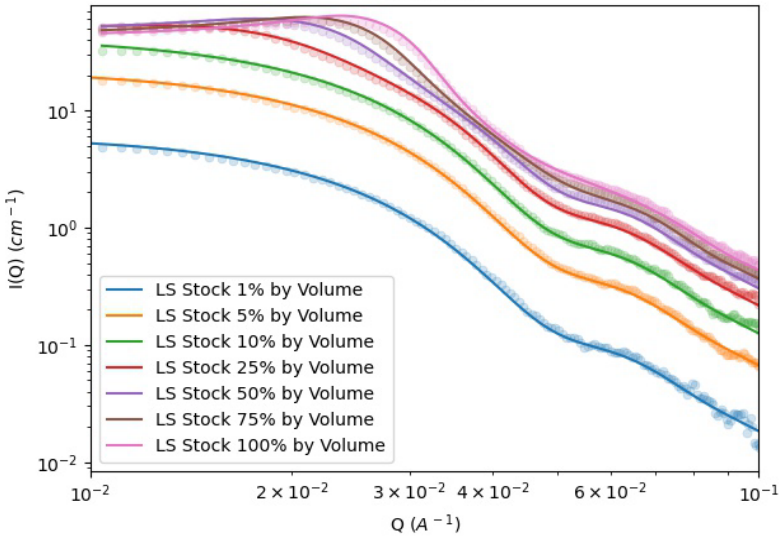
LS Concentration Series: Structure Factor Parameters

% Stock Concentration	Scale	Radius (nm)	Polydispersity	Volume Fraction	Charge (e-)	Salt Concentration (mM)
1	$1.306 \times 10^{-3} \pm 4 \times 10^{-6}$	7.925	18.00%	-		
5	$4.76 \times 10^{-3} \pm 1 \times 10^{-5}$	7.925	18.00%	-		
10	$8.92 \times 10^{-3} \pm 3 \times 10^{-5}$	7.925	18.00%	-		
25	$5.3 \times 10^{-1} \pm 2 \times 10^{-2}$	7.925	18.00%	$2.87 \times 10^{-2} \pm 8 \times 10^{-4}$	198.73	5.73
50	$3.40 \times 10^{-1} \pm 4 \times 10^{-3}$	7.925	18.00%	$6.36 \times 10^{-2} \pm 7 \times 10^{-4}$	198.73	5.73
75	$2.69 \times 10^{-1} \pm 2 \times 10^{-3}$	7.925	18.00%	$9.60 \times 10^{-2} \pm 7 \times 10^{-4}$	198.73	5.73
100	$2.32 \times 10^{-1} \pm 2 \times 10^{-3}$	7.925	18.00%	$1.33 \times 10^{-1} \pm 1 \times 10^{-3}$	198.73	5.73



I(Q) ~ P(Q) S(Q)
P(Q): Form Factor
S(Q): Form Factor

Conclusions



- Different kinds of Ludox provide diverse fits
- Concentration Effects
  - Lower concentrations of Ludox nanoparticles fit best with the sphere form factor
  - Higher concentrations of Ludox nanoparticles require both the sphere form factor and the Hayter-Penfold structure factor
- Next Steps to further validate INFER approach:
  - Observe effects on Hayter-Penfold Structure factor after modifying parameters
  - Conducting other dilutions series to verify Hayter-Penfold Structure Factor
  - Analyze nanoparticles that require other form factors/structure factors

Acknowledgements

- Caitlyn Wolf and Katie Weigandt (Mentors)
- NCNR Directors
- SURF Directors

NIST