USANS/BT5
Group 1

Chinedu Umeasiegbu, Andrew Longhini, Nathan Hould, David Stalla, Shu Wang, Dongcui Li, Melissa Dao, Steven Howell

NCNR Neutron Summer School
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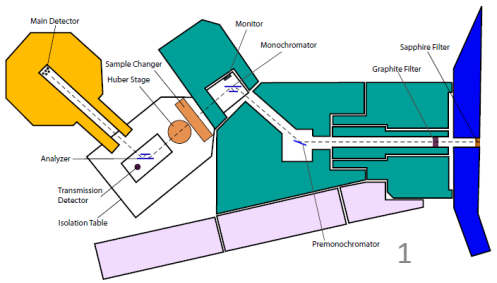


National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

SANS and USANS Investigation of Oil Uptake by Micellar Gels

The Question?

USANS

Experimental Set-up

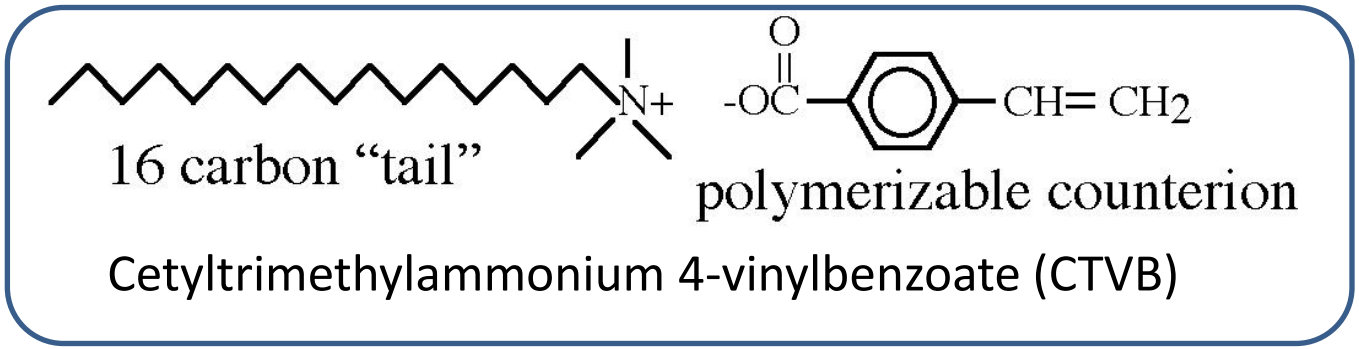
Raw Data/Initial Fits

Global Model

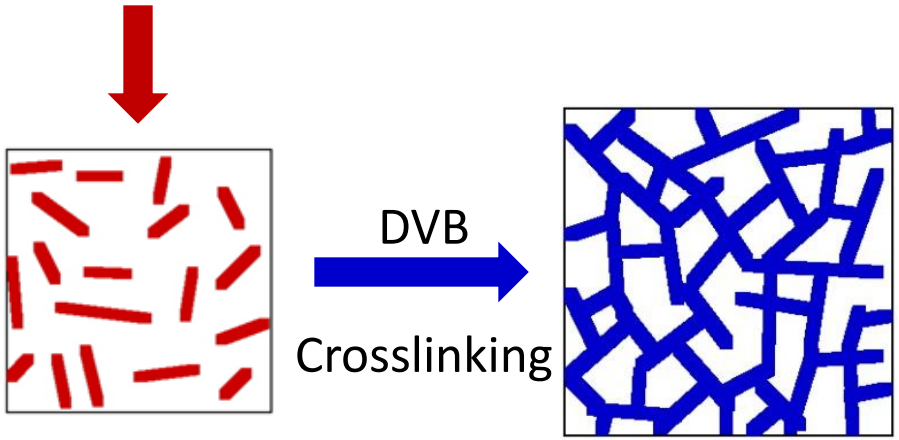
What we learned

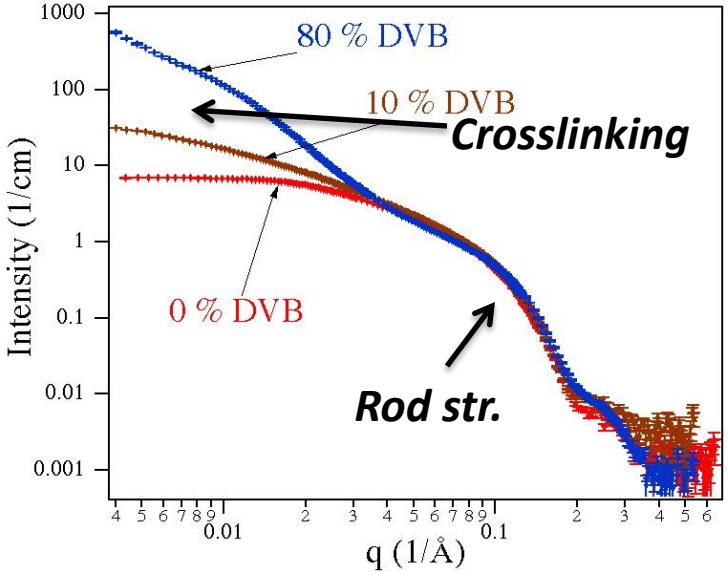
Conclusions

Background



Cetyltrimethylammonium 4-vinylbenzoate (CTVB)

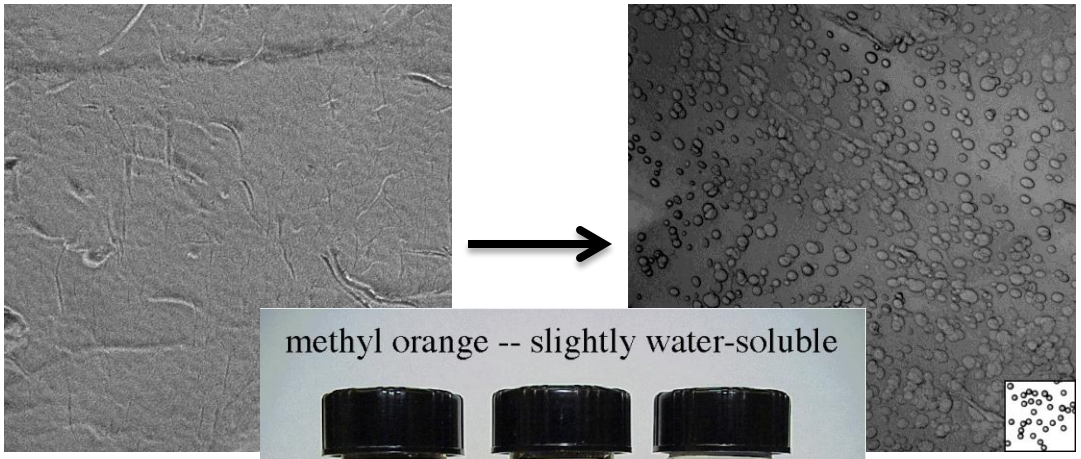




- Space filling gel: Retains cylindrical structure of micelles
- Various organic materials can be solubilized in these micellar gels

Background: Oil Uptake

- Uptake of toluene in water creates the formation of droplets of unknown composition
- Optical micrographs reveals the droplets are micron sized
  - Oil?
  - Air?
  - Water?
  - Something else?



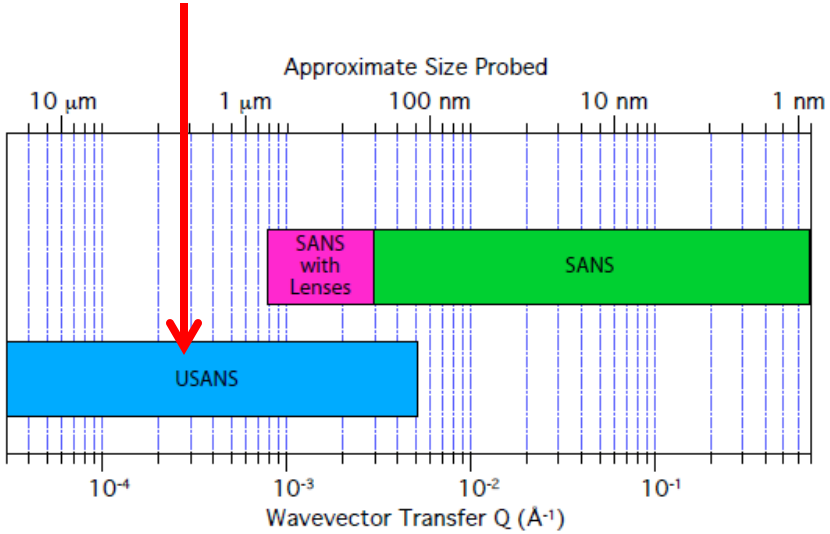
0 day

7 days

Objectives

What *size* are the droplets?

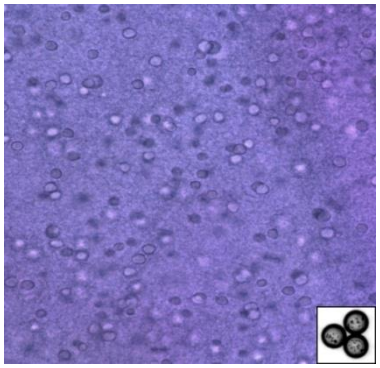
- Optical microscopy
- Electron microscopy
- DLS or SLS
- NMR
- **USANS**



Q: $3e^{-5}$~ $3e^{-3}$Å^{-1} → 0.5~10μm

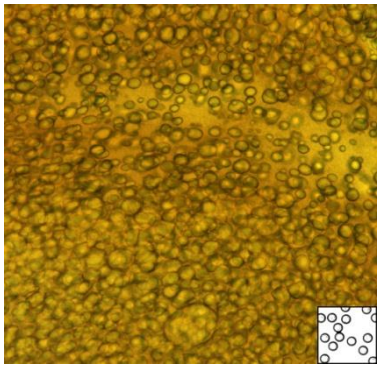
What *are* the droplets?

- EDS
- Staining



50x

Toluene-soluble dye:
Colored droplets?



20x

Water-soluble dye:
Un-colored droplets?

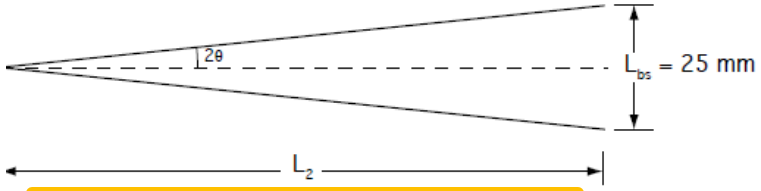
Quite inconclusive…

Microscopy cannot make the ID

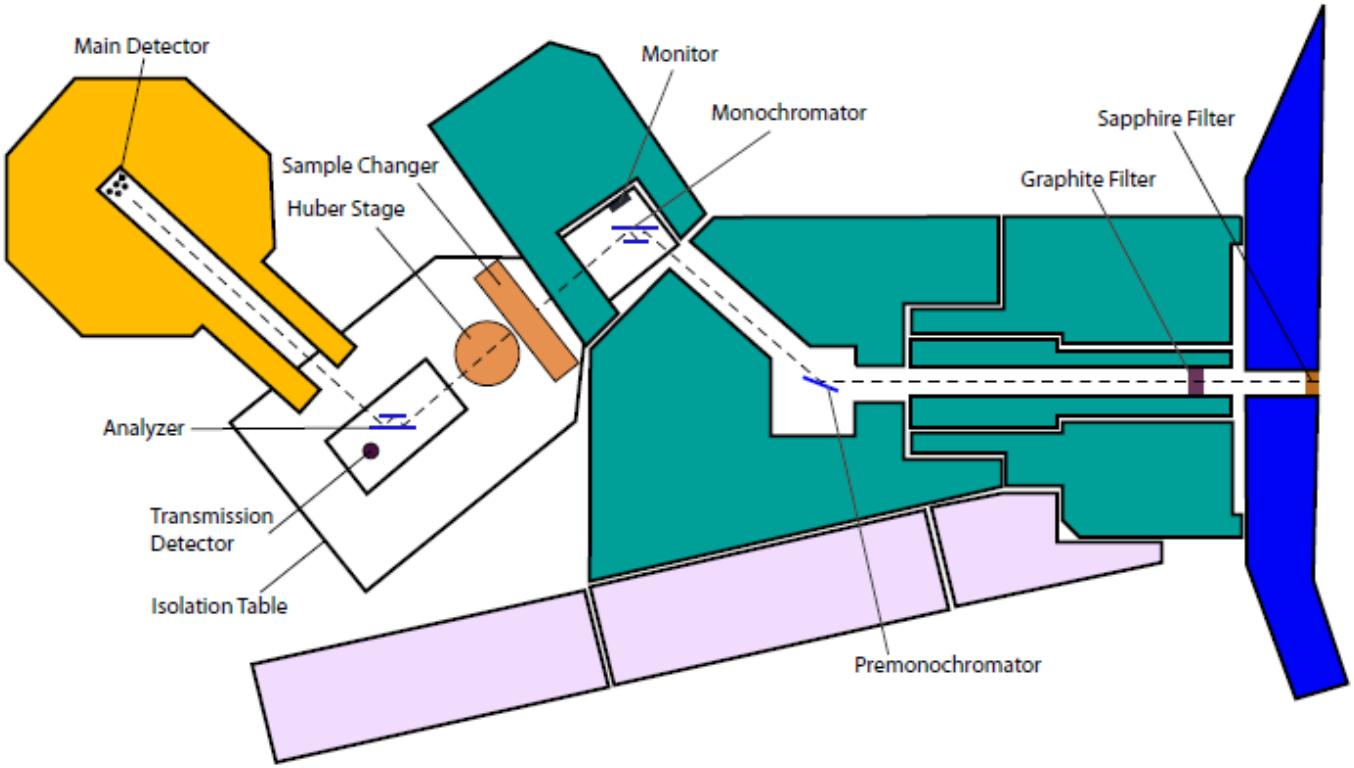
USANS Instrument

$$Q = \frac{4\pi}{\lambda} sin\theta \quad \tan 2\theta = \frac{L_{bs}/2}{L_2}$$

$$Q = 3 \times 10^{-5}\ \text{Å}^{-1}, \qquad \lambda = 6\text{Å}$$



$\rightarrow$ $L_2 = 443$ m!



Differences from SANS

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- Mult
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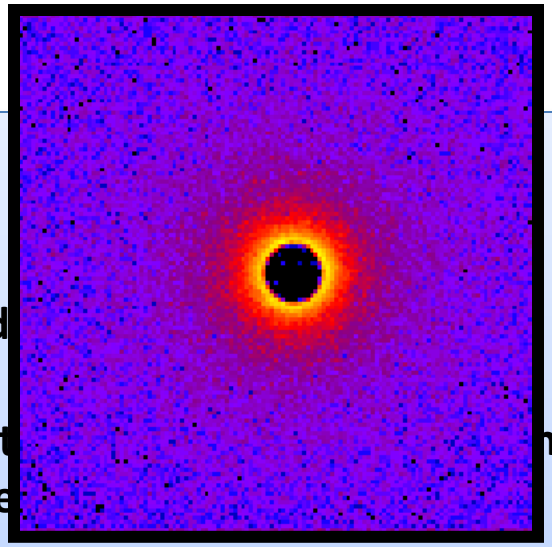
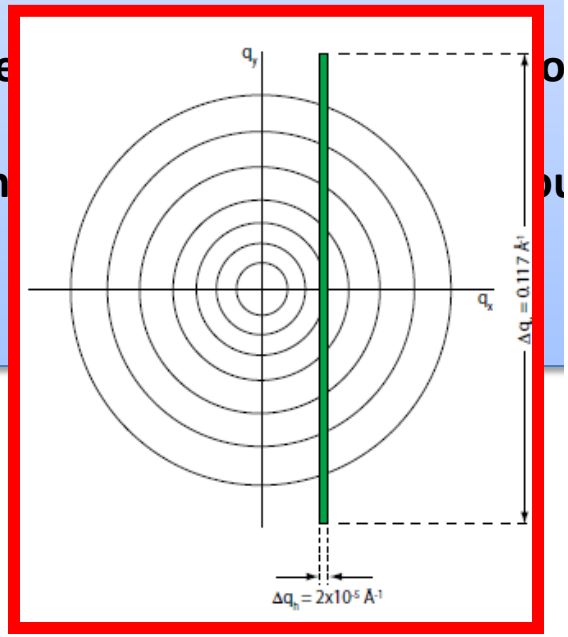
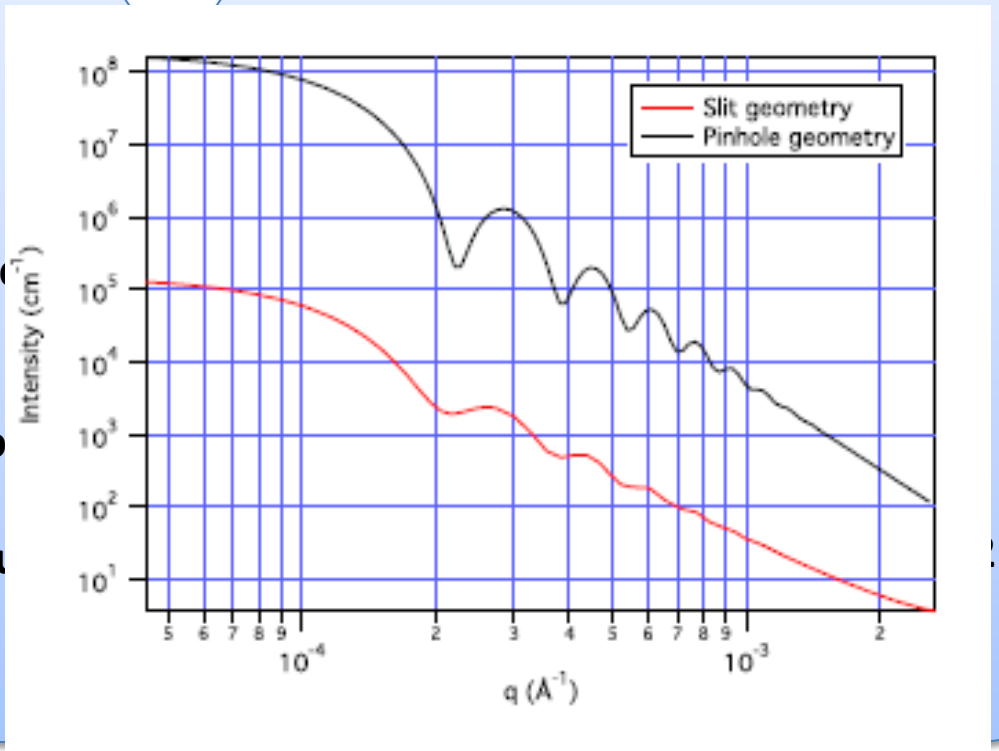
- Colle
- Coun







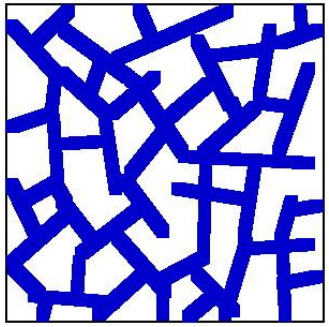
Experimental Plan

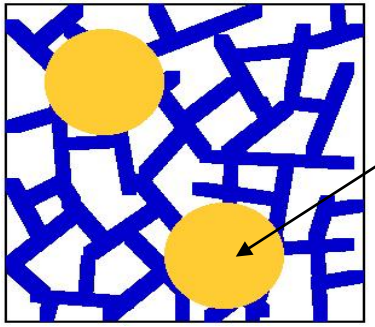
Mystery "Blobs"…

Water?

Air?

Oil?...





What are these?

What do we know?

SLD of the gel matrix, oil component…

Roughly the size and shape of the "blobs"…

What we will do on USANS?

1. ***Plan of samples***
2. Instrumental configuration: $3e^{-5}$~ $3e^{-3}Å^{-1}$
3. Data collection
4. Data reduction
5. ***Data Analyzing***
6. ***Key learning from USANS***

Experimental Plan

D_2O
6.32 e-6Å^{-2}

H_2O
-0.52 e-6Å^{-2}

Gel CM
0.53 e-6Å^{-2}

S#1 S#2

D-Toluene
5.6 e-6Å^{-2}

H-Toluene
0.94 e-6Å^{-2}

S#3 S#4

Gel D_2O
5.7 e-6Å^{-2}

Gel D_2O
5.7 e-6Å^{-2} S#5

Empty Cell S#6

If , $I(q)_{\#1} / I(q)_{\#2} \approx 150$,
not ~~water~~ nor ~~air~~

$$I(q) \propto (\Delta\rho)^2$$

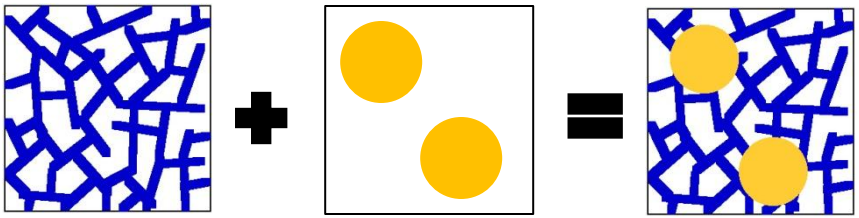
#3 & #4 cross check
Reverse the contrast

#5 measure pure gel structure

#6 essential for data reduction

What to measure?

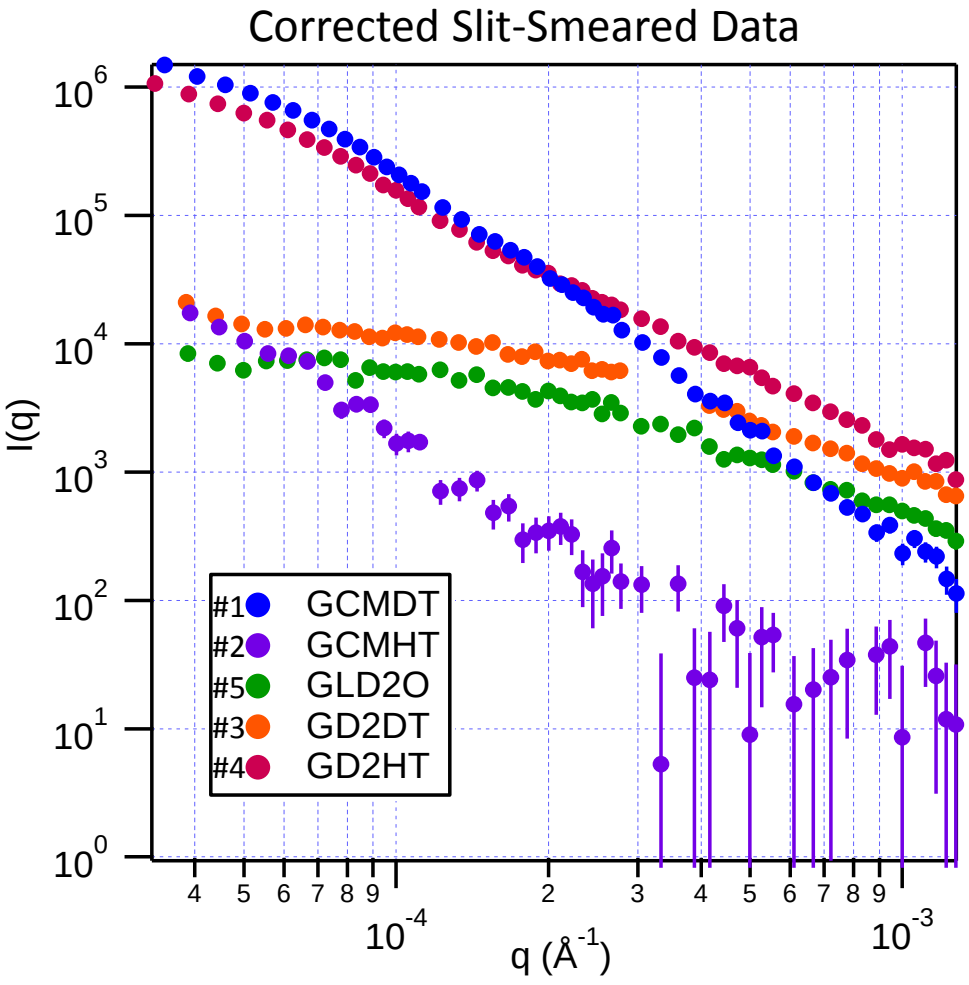
- Mystery "Blobs"…
  Water?
  Air?
  Oil?
- Structures of Gel
  Structures?



$I(q)_{\#5}+I(q)_{\#1}=I(q)_{\#4}$

#1 GCMDT
#2 GCMHT
#5 GLD2O
#3 GD2DT
#4 GD2HT

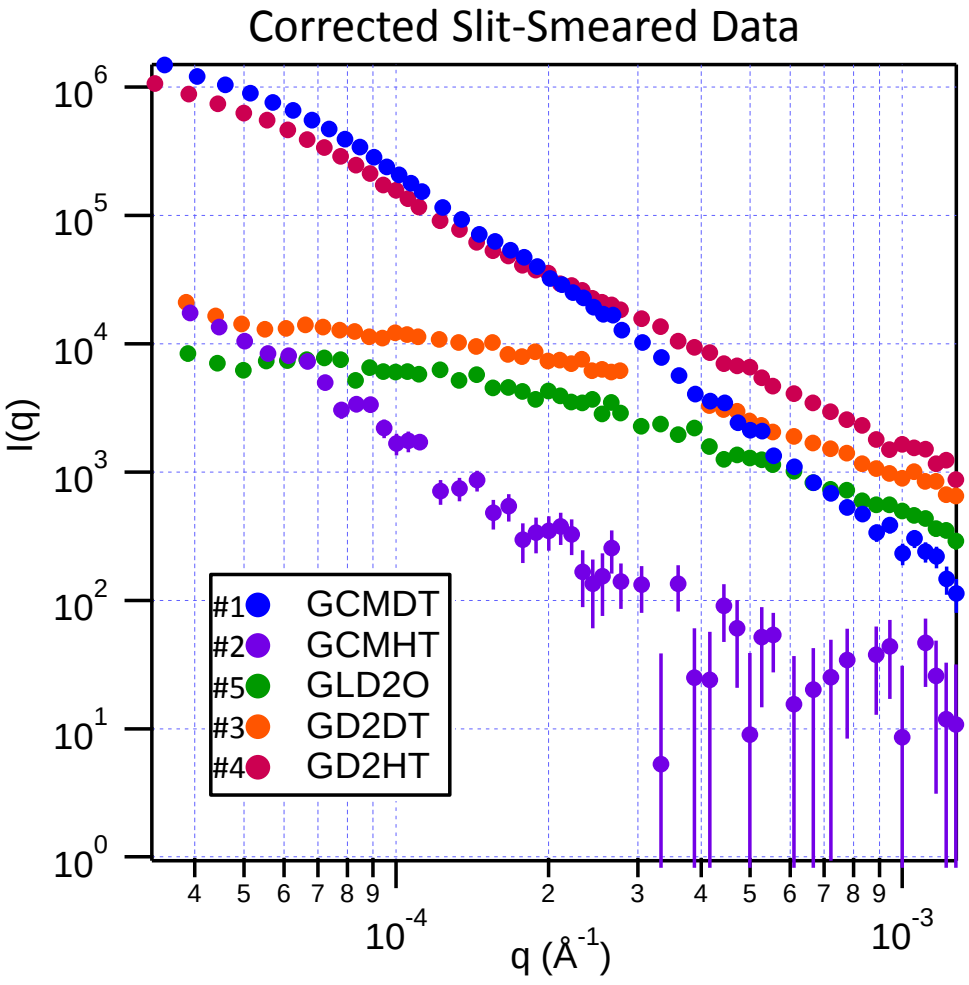
Data Analysis: Data Reduction



Data Reduction:

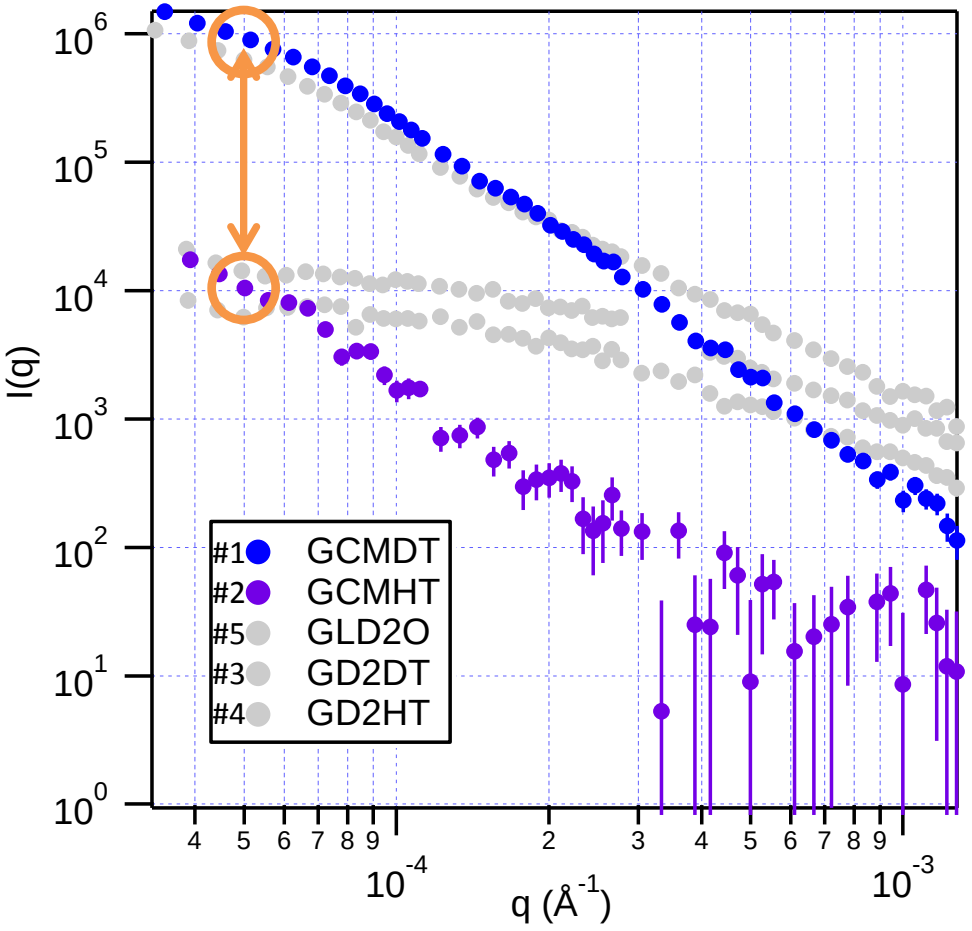
1. Join raw data sets
2. Shift to zero angle and exclude direct beam
3. Transmission is automatically calculated
4. Subtract the Background (blocked beam) and the Empty Cell

Data Analysis: Sanity Check



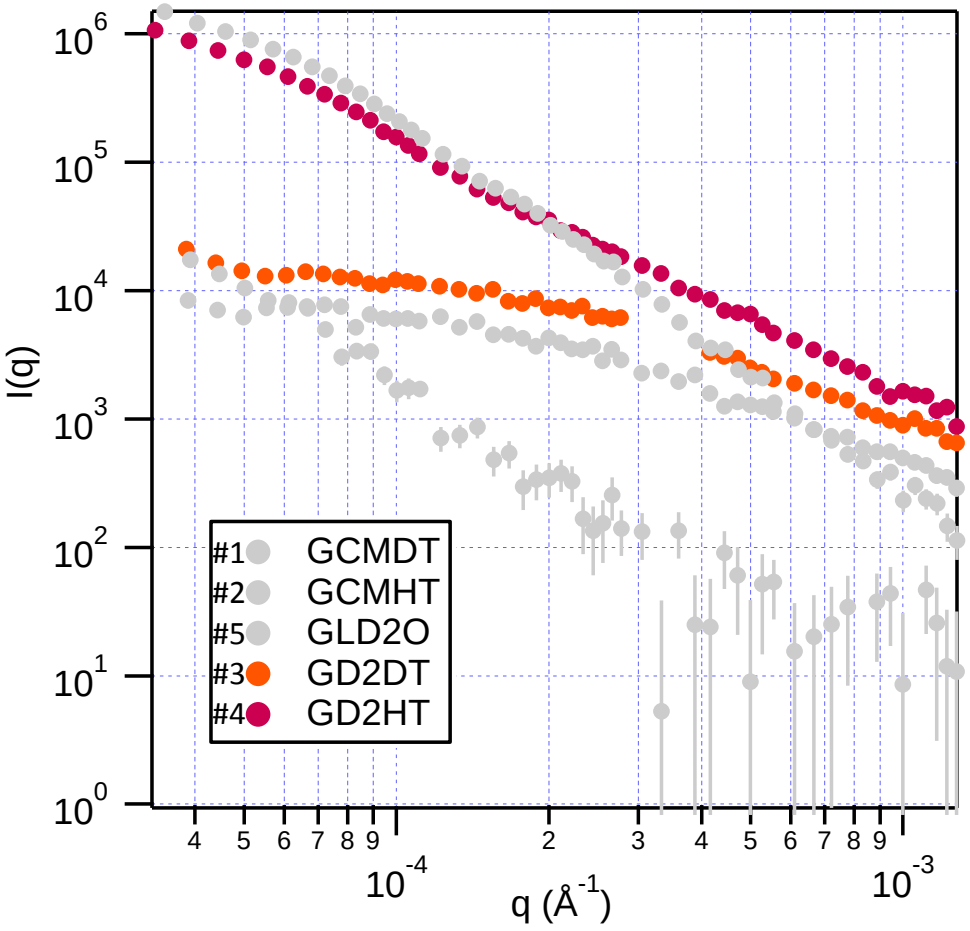
- Good scattering data
- Obvious structure present
- Intensities vary

Data Analysis: Matched Gel



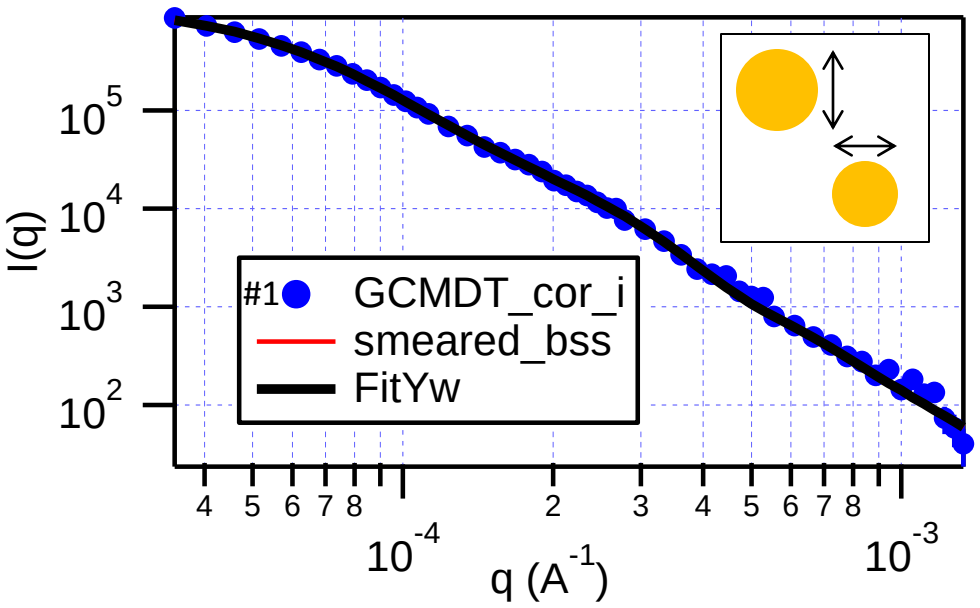
- Large difference
  - Air scattering would be identical
  - Water/Solvent would not scatter
- $I_D / I_H \approx 100$
- Oil is in the “blobs”

Data Analysis: Deuterated Gel



- Reversing the contrast indeed reversed the scattering intensity

Data Analysis: GCMDT and GLD2O



Sample: #1 GCMDT

- Used to determine the approximate size of the droplets
- Bimodal Schultz Sphere Model

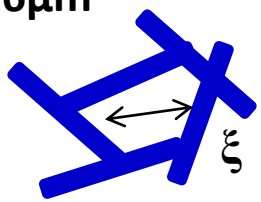
$$I(q) = \left[\left(\tfrac{4\pi}{3}\right)^2 N_1 \Delta\rho_1^2 \int_0^\infty f(R_1) R^6 F^2(qR_1) dR\right] + \left[\left(\tfrac{4\pi}{3}\right)^2 N_2 \Delta\rho_2^2 \int_0^\infty f(R_2) R^6 F^2(qR_2) dR\right]$$

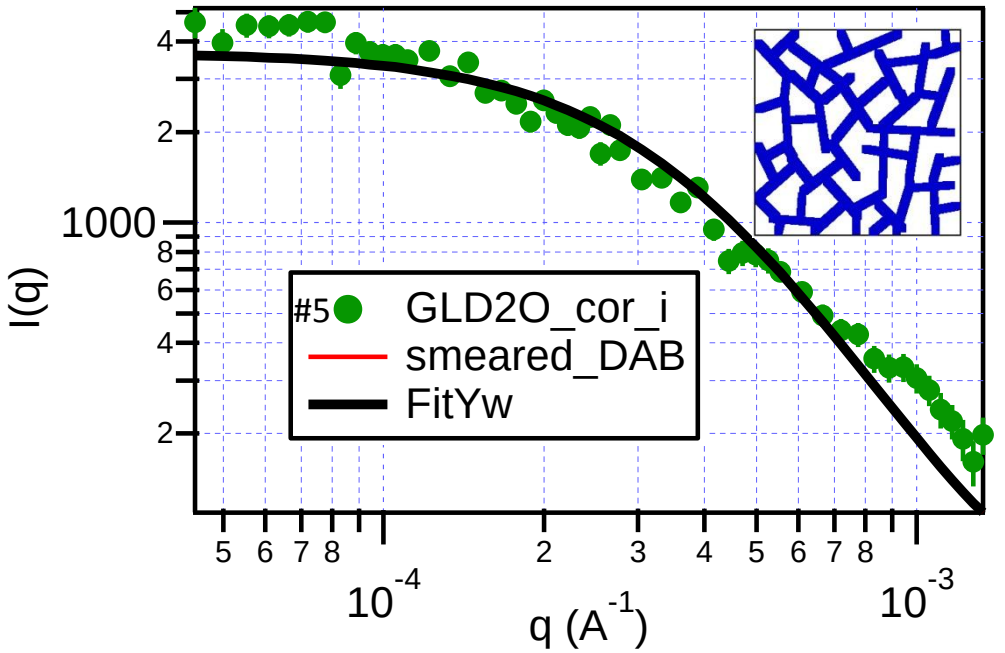
$$F(x) = \frac{3\left[\sin(x) - x\cos(x)\right]}{x^3}$$

Sample: #5 GLD2O

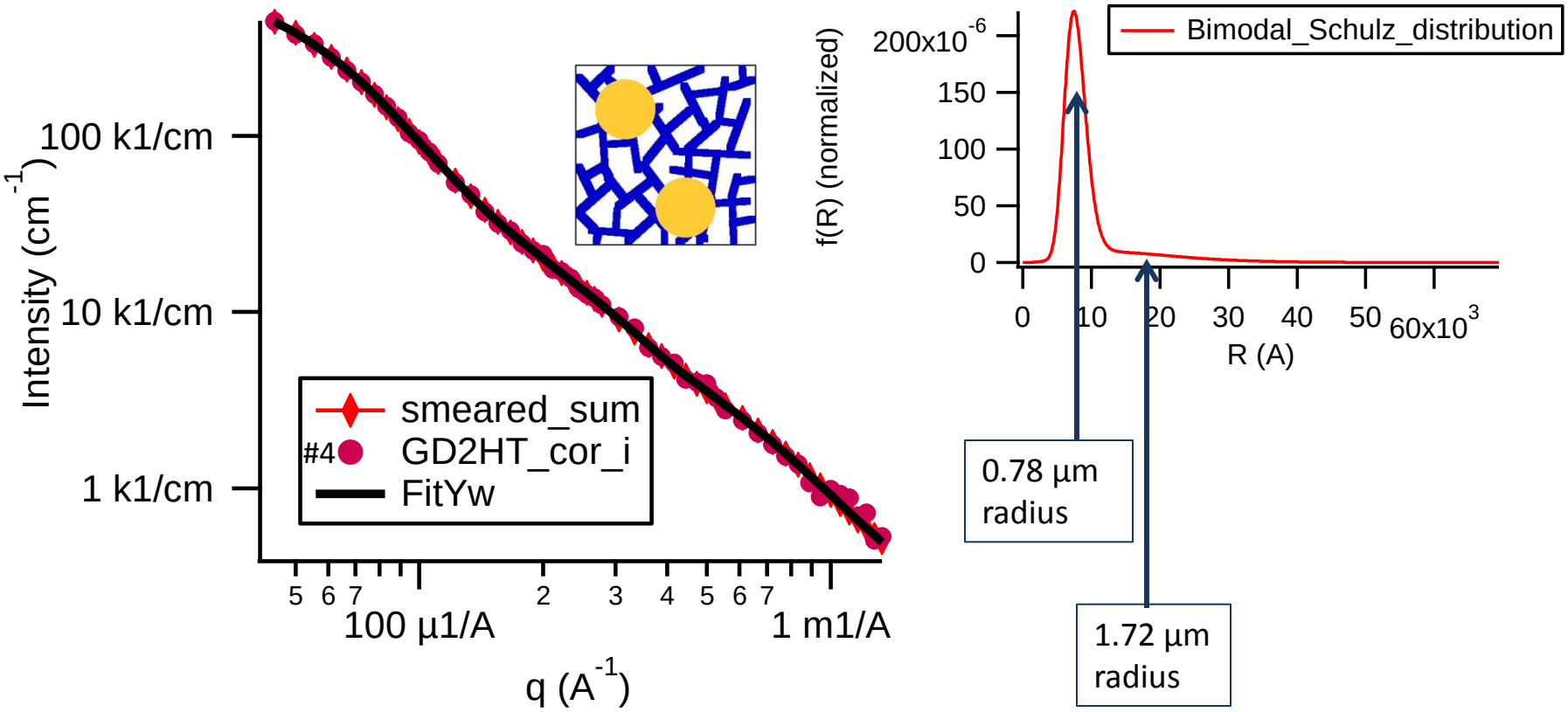
- Used to determine the approximate size of the gel
- DAB model was used because it does not require a specific shape
- **Correlation length: 0.26µm**

$$I(q) = \frac{A\xi^3}{\left[1+(q\xi)^2\right]^2}$$





Data Analysis: GD2HT



Sample: #4 GD2HT

- Contains data about droplets and gel size
- Combines models determined by GCMDT and GLD2O
- Bimodal distribution determine droplets are mostly small droplets with radius of 0.78µm

Conclusions

- The droplets are identified as solubilized oils by USANS
- The size of the drops is about 1.6 µm
- The basic structure of the micelles is retained, solubilizing oil in the core of micelles
- Why large oil droplets form is still an open question

Acknowledgements

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We Love Neutrons, because neutrons never lie!