# SANS sample environments at NIST past, present, and future: From lessons learned to new opportunities

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CHRNS
Center for High Resolution Neutron Scattering
NIST

# Sample Environment: what is it?

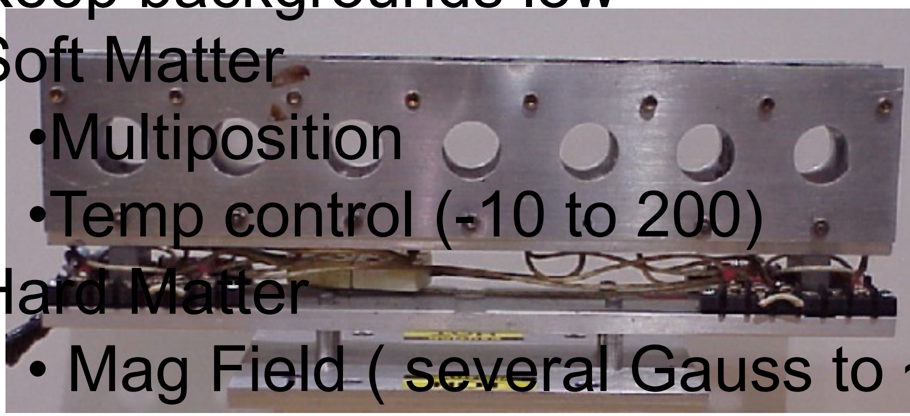
- Apply an environment "field" to the sample (B,T,E,P, $\sigma,\gamma$ etc)
- Make simultaneous measurement of various properties
  - Is it necessary? There exists two camps on this issue still
- RELATED – new techniques closely coupled to environment:
  - TISANE (e.g. high frequency oscillating fields)
  - Time Resolved (e.g. stop flow)
  - Polarization and Polarization analysis…
- INCREASINGLY – multiples of these.

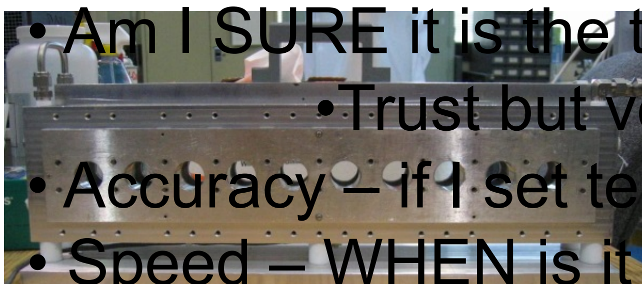
The past decade has seen a rapid move towards ever more complex systems using ever more complex "environments"

# Sample Environment: Don't Forget the Basics

In the quest for ever more sophisticated environments one can easily lose sight of the basics.

- Keep backgrounds low
- Soft Matter
  - Multiposition
  - Temp control (-10 to 200)
- Hard Matter
  - Mag Field ( several Gauss to ~10 Tesla?)
  - Low Temp (2K can do a lot)
- Am I SURE it is the temp I say it is? How do I know?
  - Trust but verify!!!
- Accuracy – if I set temp to x.yy is the sample at x.yy?
- Speed – WHEN is it at x.yy?





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Acquiring new multizone changer

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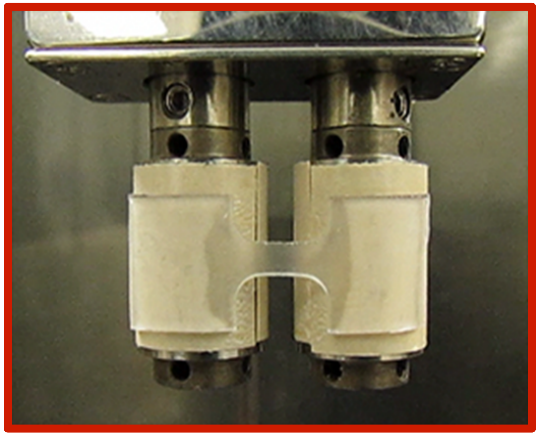
# Sample Environment: Infinite variety – how to choose

- Must be driven by an actual scientific program NOT a hypothetical one.
  - Internal program
  - collaboration with "local" power user
  - Approved experiments – need team with a variety of skills and/or collaborate with long distance power users.

Examples of different user interactions



# Sample Environment: Some Current NIST Directions

## *Pressure – driver: I.S. program + nSOFT*

Syringe pump driven


Control software

- Compatible with :
  - aqueous and organic liquids
  - corrosive solutions
  - heated fluid
  - liquefied gases
  - viscous fluids or colloids
  - flammable and explosive gases
- Temperature 0 to 60C at present Higher temp being developed
- Pressure to 690 bar

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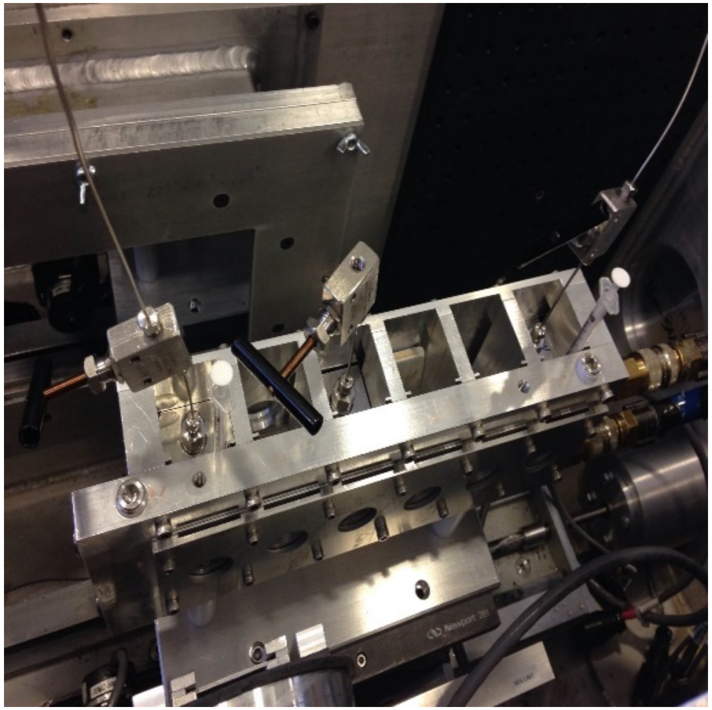
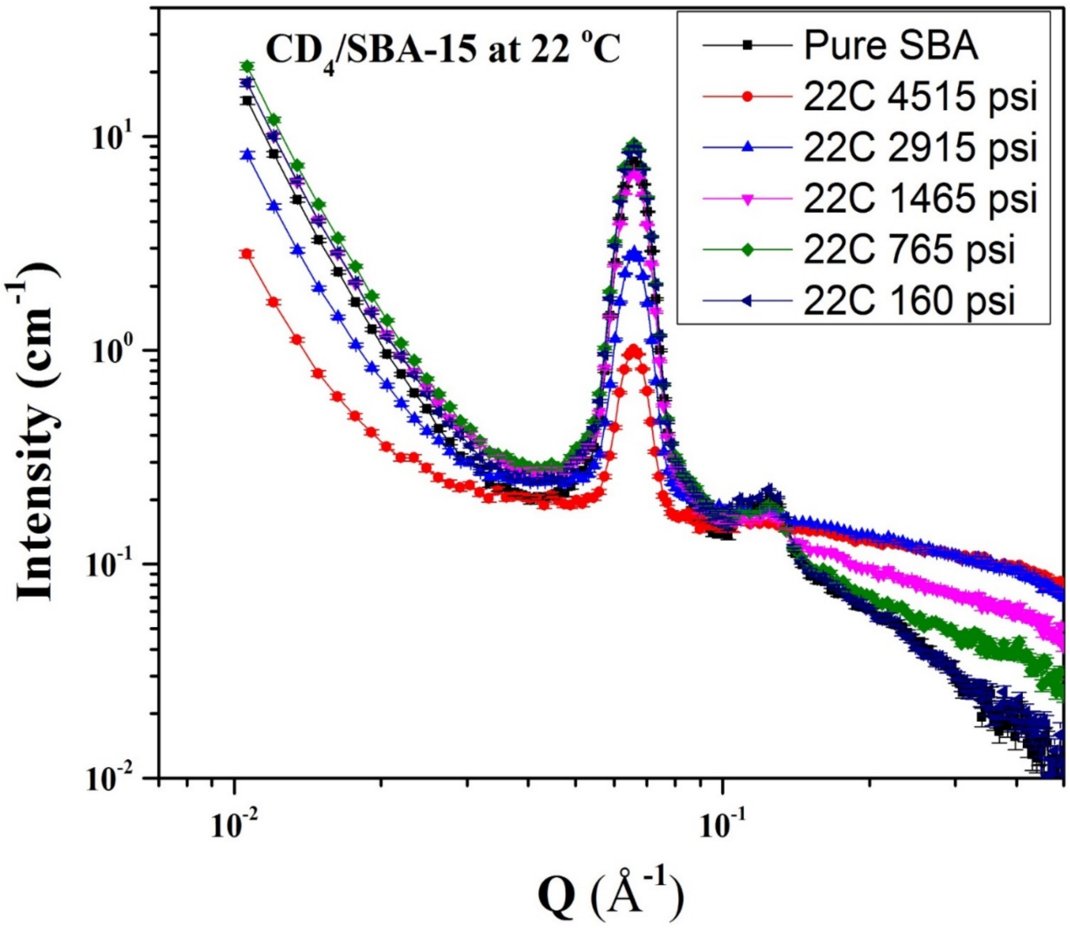
# Sample Environment: Some Current NIST Directions

## *Pressure – driver: I.S. program + nSOFT*



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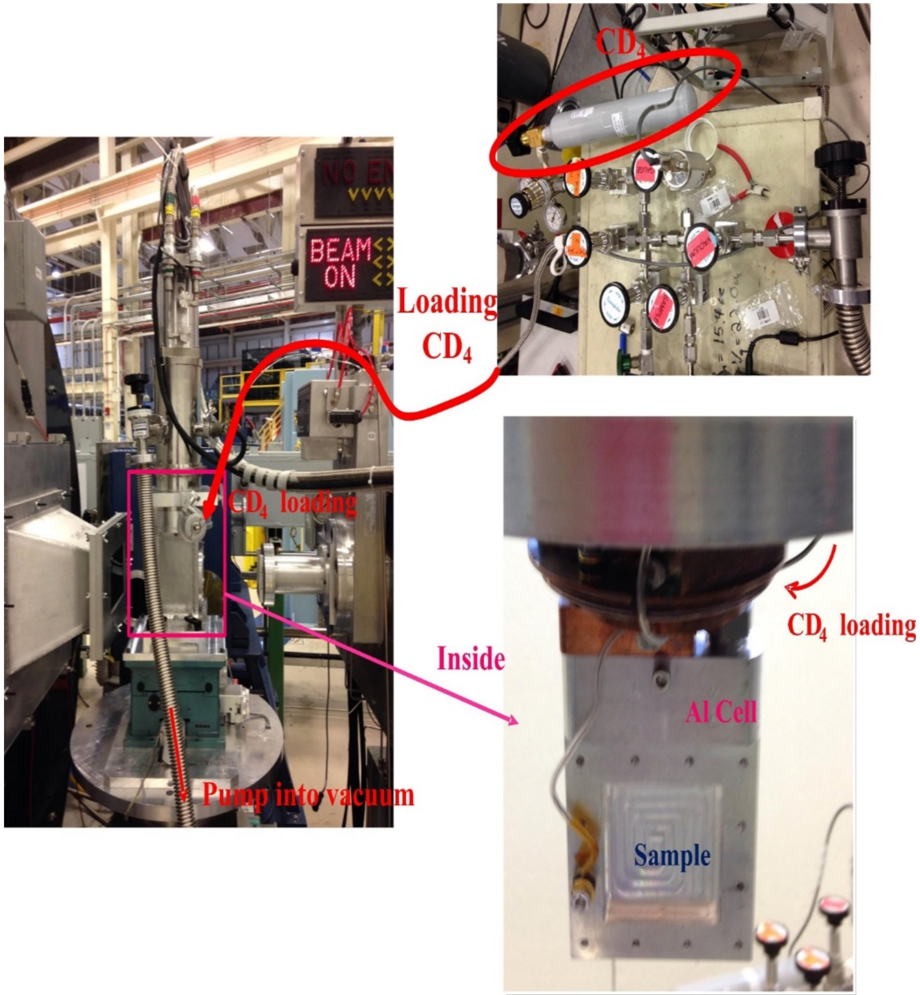
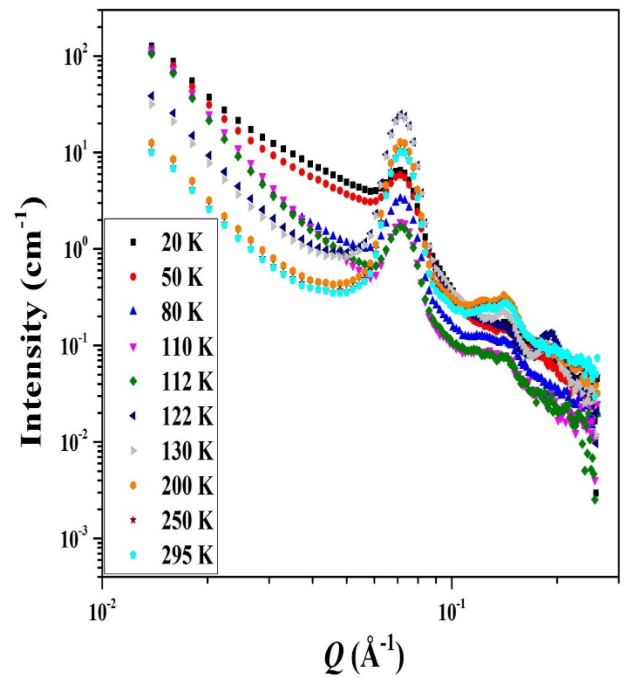
# Sample Environment: Some Current NIST Directions

## *Pressure – driver: I.S. program + nSOFT*




Pressure at Low temperature

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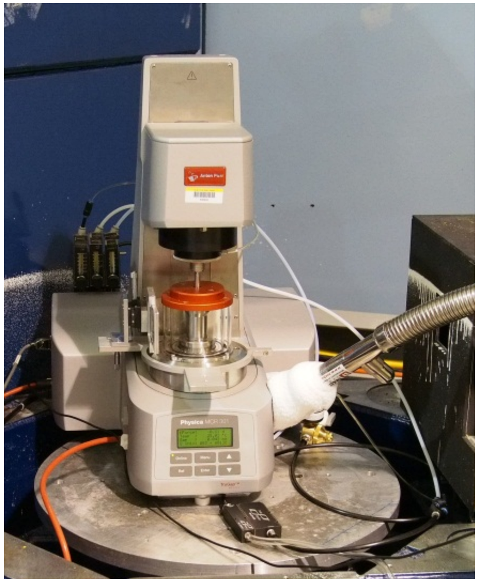
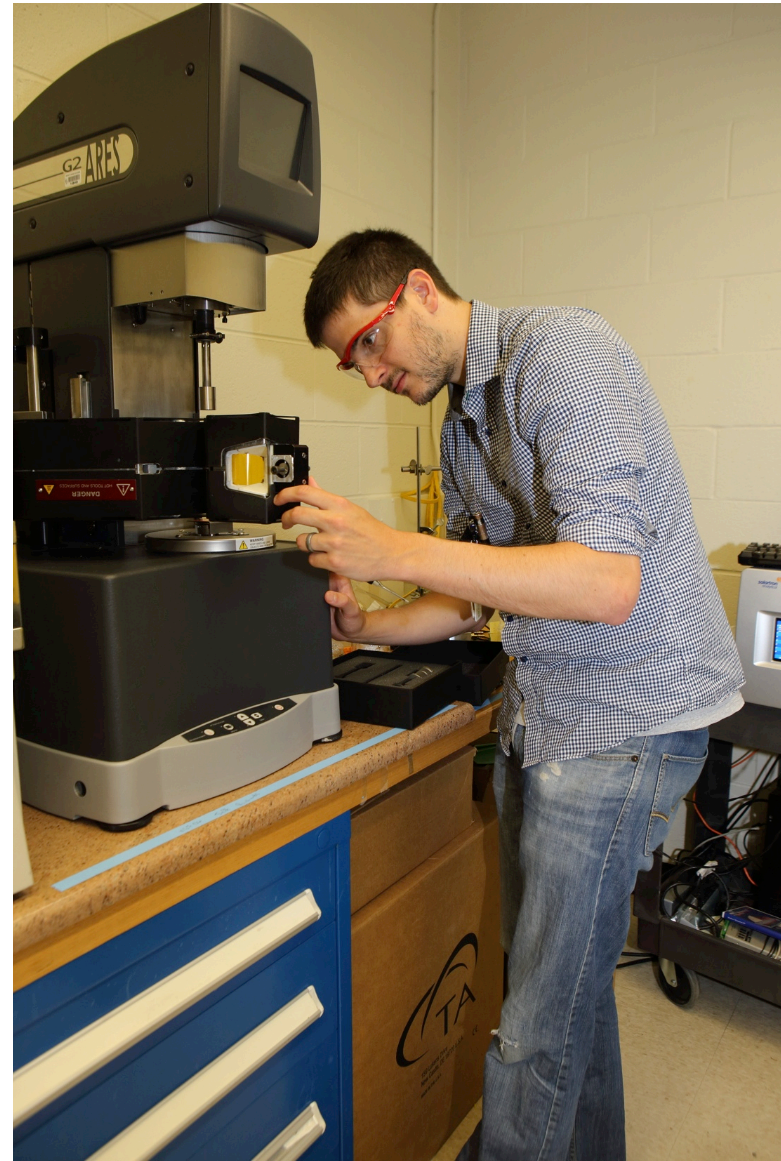
# Sample Environment: Some Current NIST Directions

## *Flow: new rehometer - driver: long standing partnership*



New Ares G2 strain controlled Rheometer will add dielectric spectroscopy

Good for shear banding, conducting polymer solutions, flow batteries

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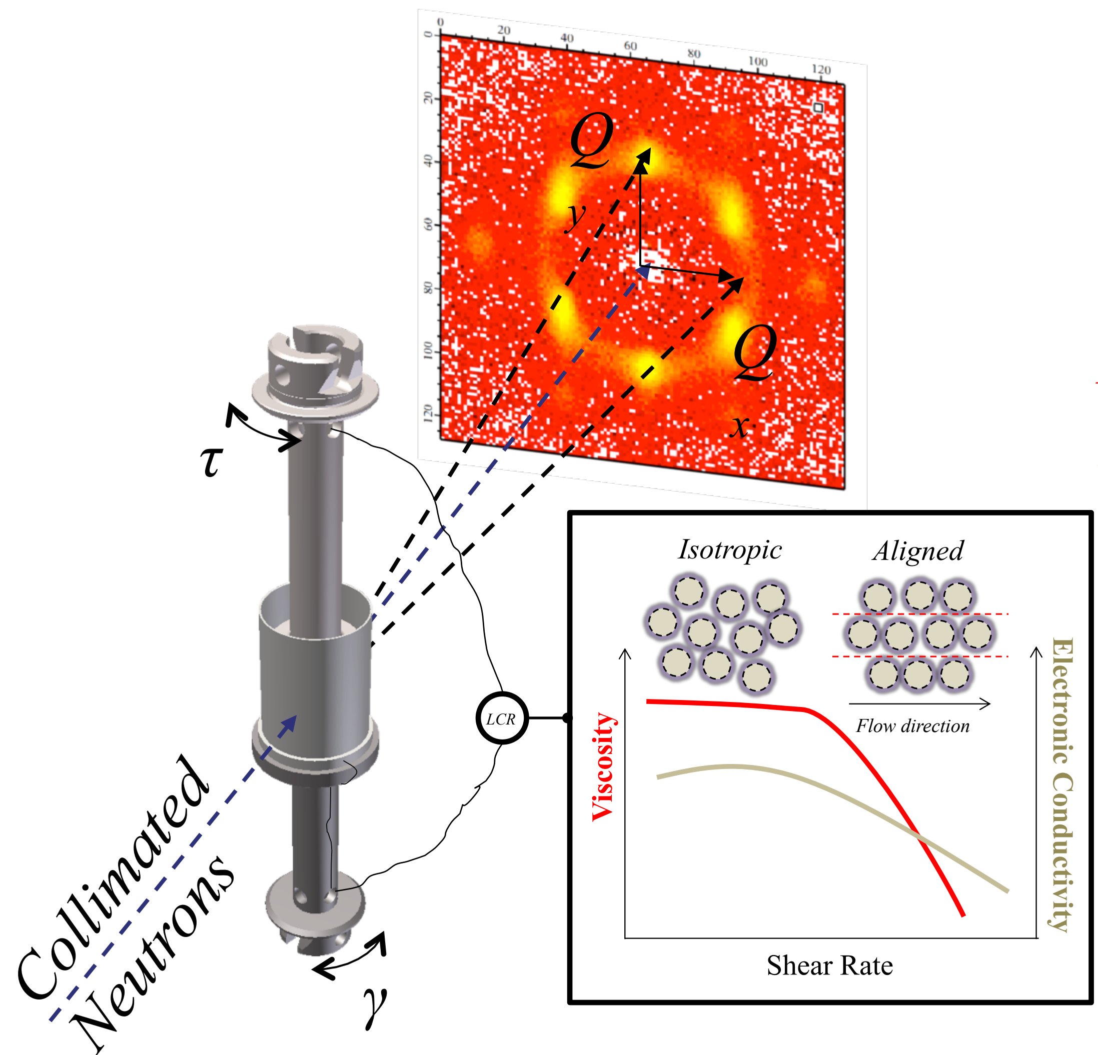
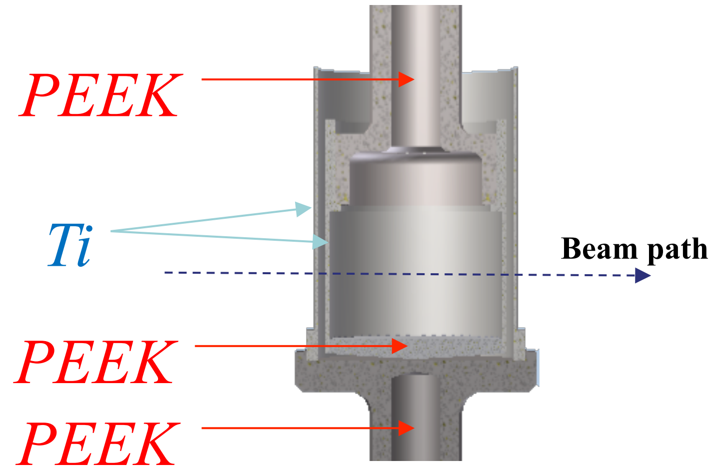
# Sample Environment: Some Current NIST Directions

## *Flow: Dielectric Rheosans – driver: flow battery partnership*




## Specifications:

- Polyethyletherketone (PEEK) shafts for electrical isolation.
- Titanium (Ti) walls serve as transparent (to neutrons) conductors – 2 mm total thickness.
- 25/27 OD/ID Couette design
- Electrical connections provide for continuous measurement under steady flow conditions.
- Installed on ARES G2 with Oven capable of -90-350°C temperature range

***Part of new Ares G2 capabilities***

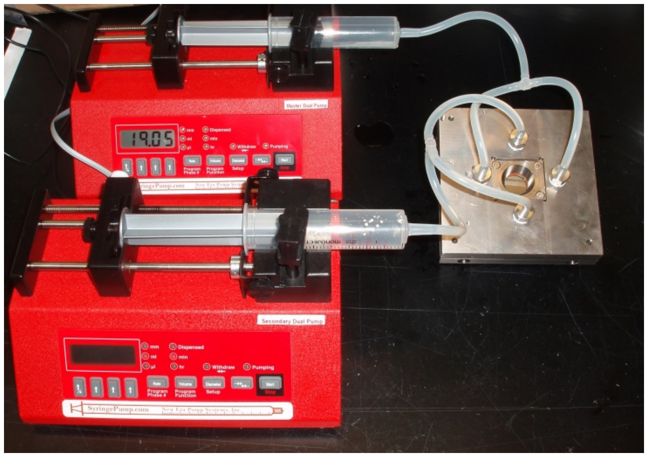
# Sample Environment: Some Current NIST Directions

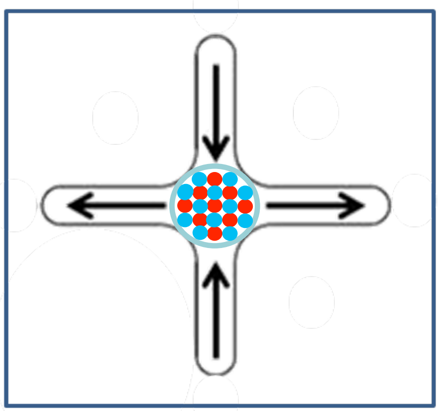
## *Flow elongatinal – driver: I.S. program + nSOFT*

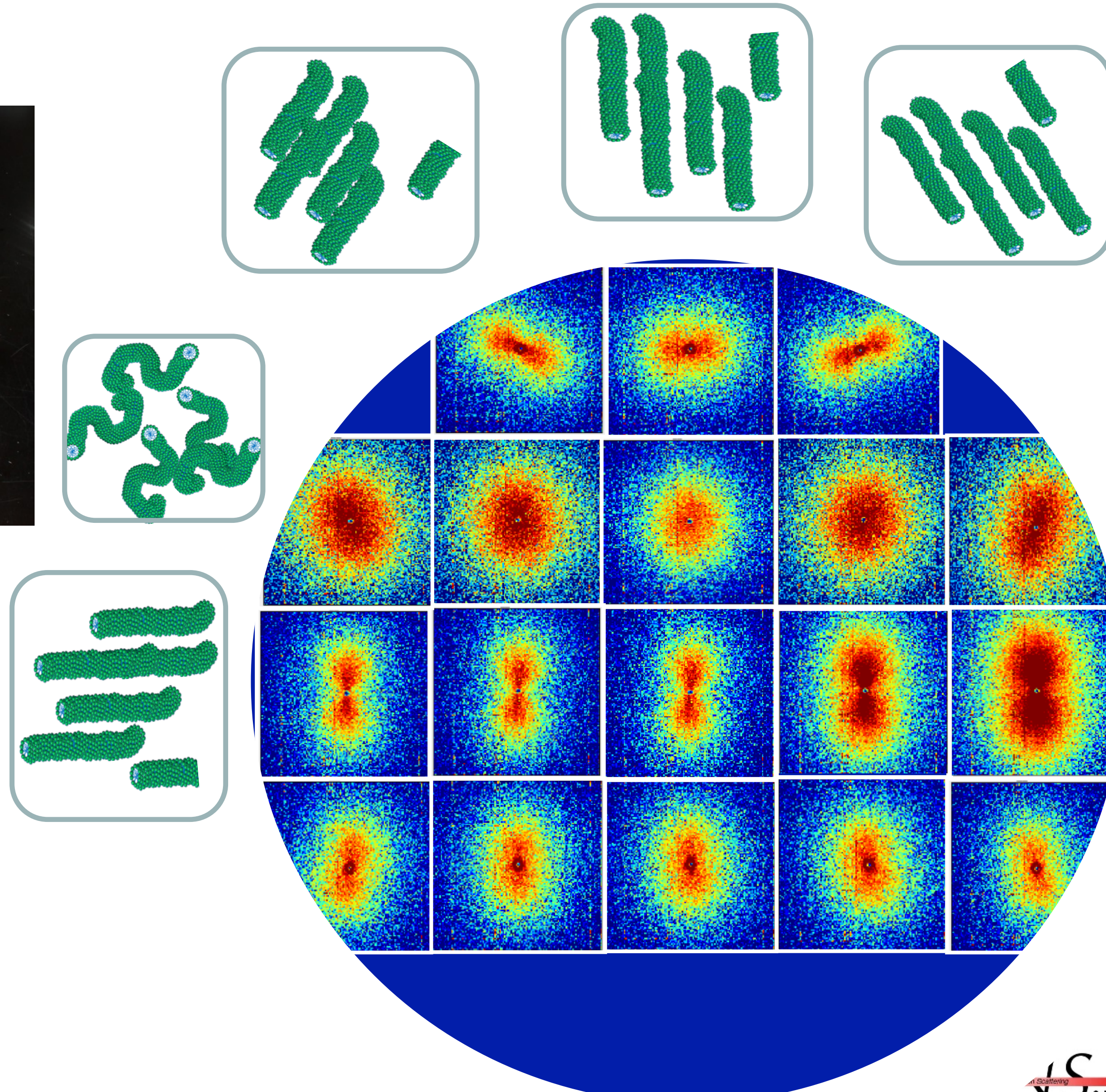
- Temperature control ~10°C to 80°C
- Multiple inlet and outlet ports
- Interchangeable flow channel geometries
- Optional pressure sensors at outlets and inlets
- Continuous unidirectional flow with syringe pumps
- Start-Stop flow with event mode trigger

# Sample Environment: Some Current NIST Directions

## *Flow extensional – driver: I.S. program + nSOFT*





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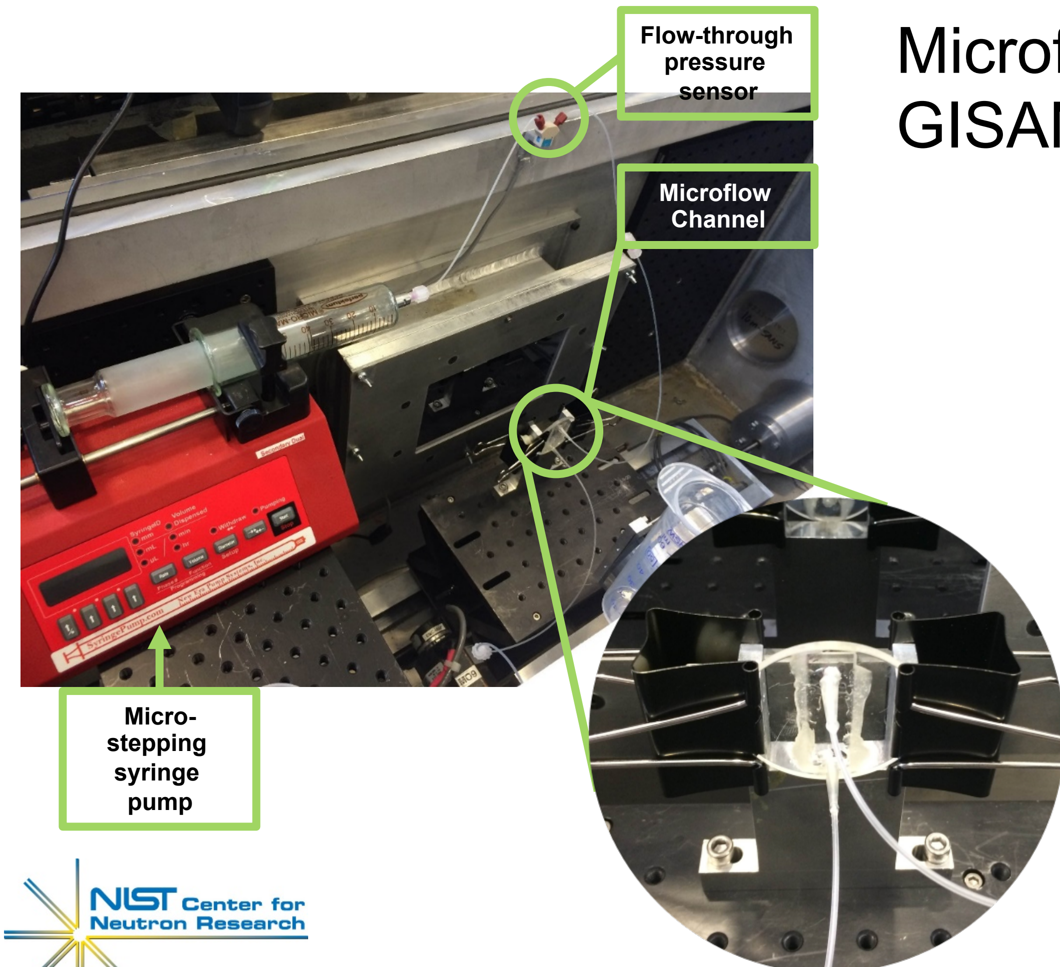
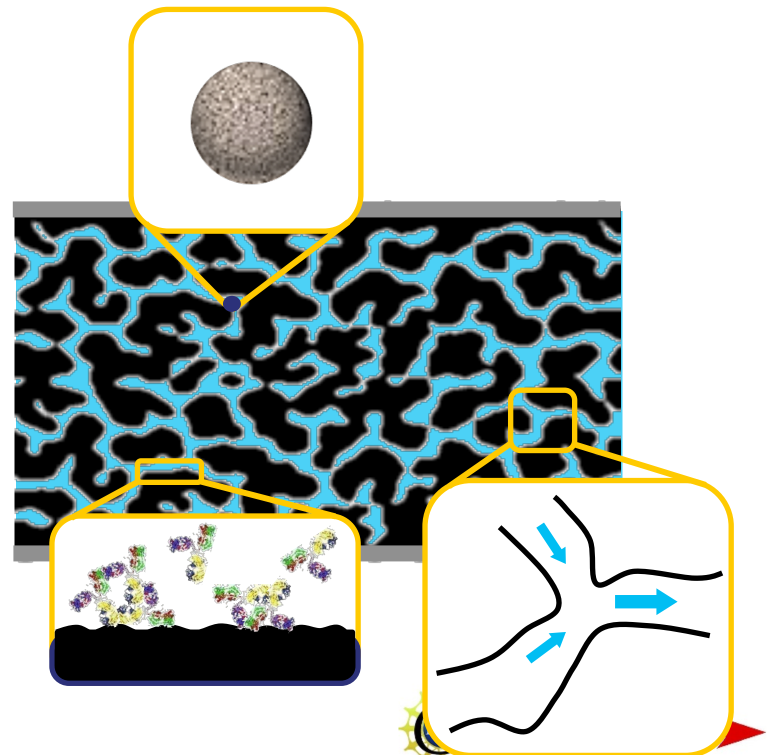
# Sample Environment: Some Current NIST Directions

## *Flow: The Next Generation – driver: I.S. programs + nSOFT*

New IMS to look at high shear in constrained geometries under varying conditions of pressure and temperature




Microfluidics, porous materials, GISANS



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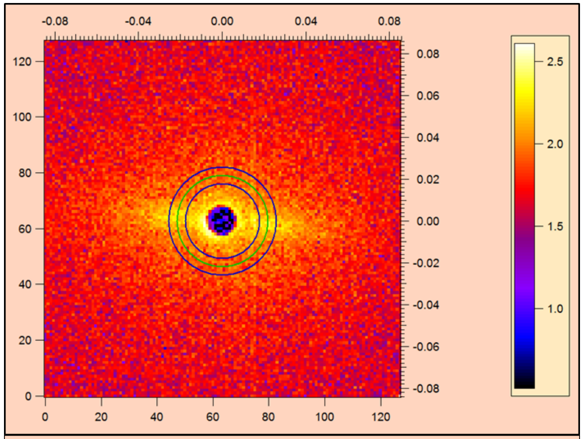
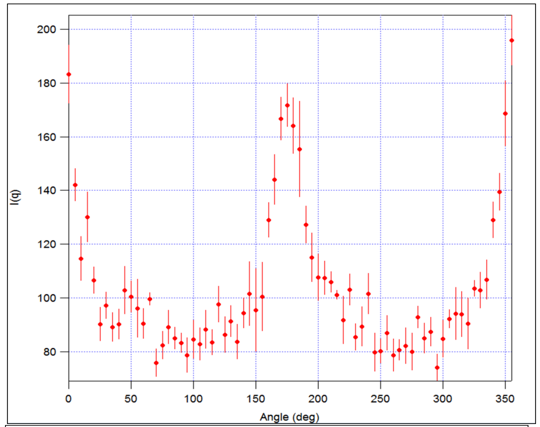
# Sample Environment: Some Current NIST Directions

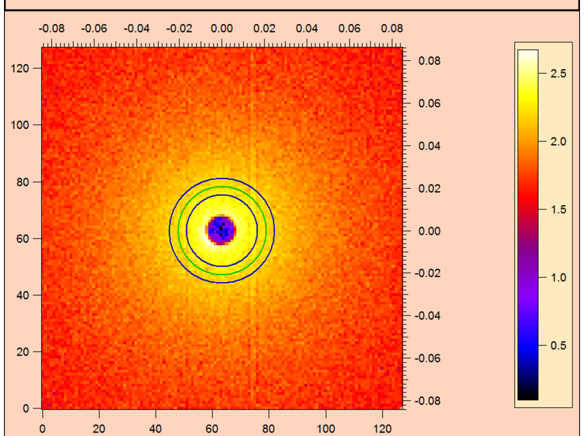
## *Flow: The Next Generation – driver: I.S. programs + nSOFT*

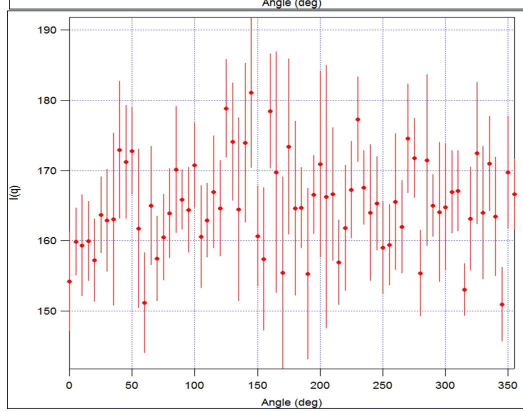
### CTAB/NaSal Worm-like Micelles Under High Shear Rates






$\dot{\gamma} = 1.50 \times 10^6\ s^{-1}$ (10 μm channel)






$\dot{\gamma} = 0.0\ s^{-1}$ (10 μm channel)

| Channel Thickness | Maximum Tested Shear Rate | Flow Rate Required |
|---|---|---|
| 100 $\mu$m | 1.49 x $10^6$ $s^{-1}$ | 19.9 mL/min |
| 10 $\mu$m | 1.50 x $10^6$ $s^{-1}$ | 0.2 mL/min |

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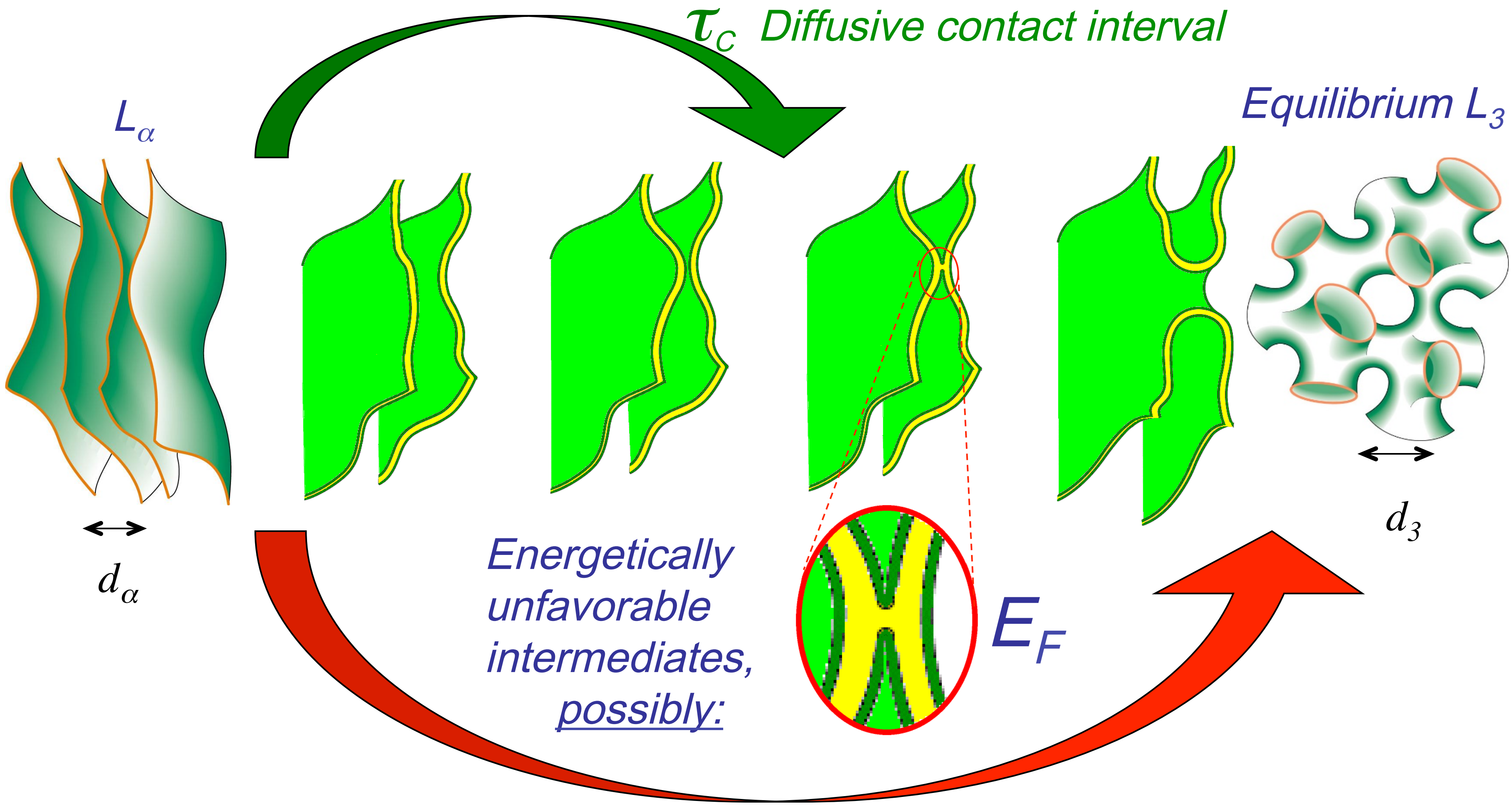
# Sample Environment: Data Acquisition Integration

Competing interests and philosophies:

- Native to the acquisition software
  - drivers added by request by experts – usually DAQ Team
- Driven independently of acquisition Software
  - Simple Ready, Acknowledge (and trigger?)
- Simple interface from acquisition software ➔ Some kind of API
  - but simple (e.g. transfer of commands to an RS-232)?
  - or more complex (interfacing with Labview or C programs?

Question: do we want to enable experiments? Or only measurements? There are NOT the same – can I quickly add a new SE or a new feature to my SE on Sunday afternoon or at 3:00 in the morning?

# Energetics of passage formation




Topological relaxation time $\tau_R = \tau_C \exp[-E_F/k_BT]$

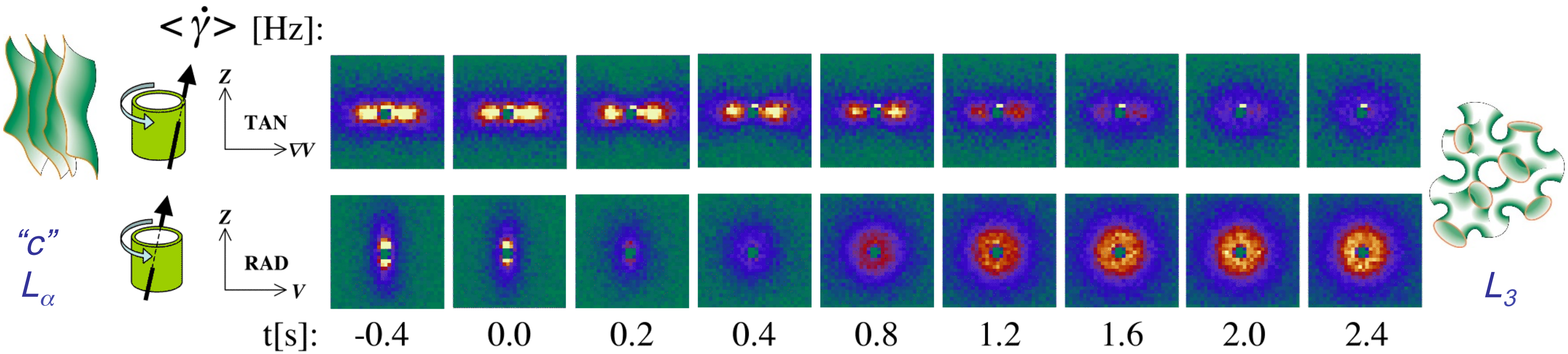


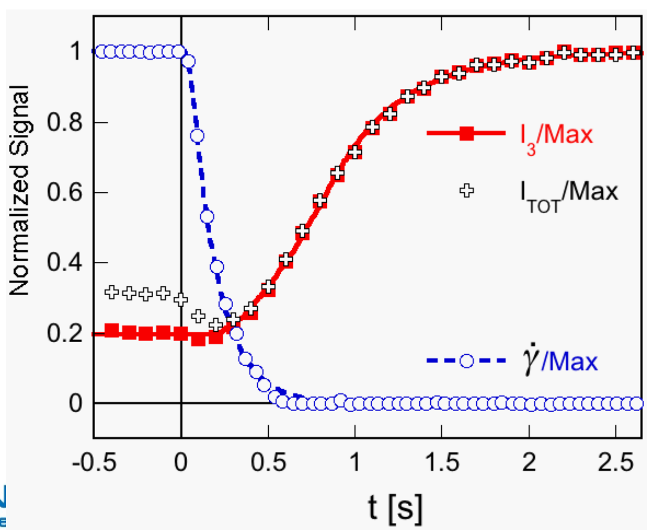
S. T. Milner, M.E. Cates and D. Roux, *J. Phys.* (Paris) 51, 2629 (1990)

# Determination of $\tau_R$ - t-SANS

*t-SANS Shear-induced $L_\alpha$ to equilibrium $L_3$ relaxation*

*$\phi$=5vol% CPCl-hexanol in 40vol% dextrose-brine ($\eta_s$=16.3cP)*




*Shear aligned at*

$$\dot{\gamma}\eta_s / \phi^3 \sim 3\times10^8 cP/s$$

*~center $L_\alpha$ signal plateau*

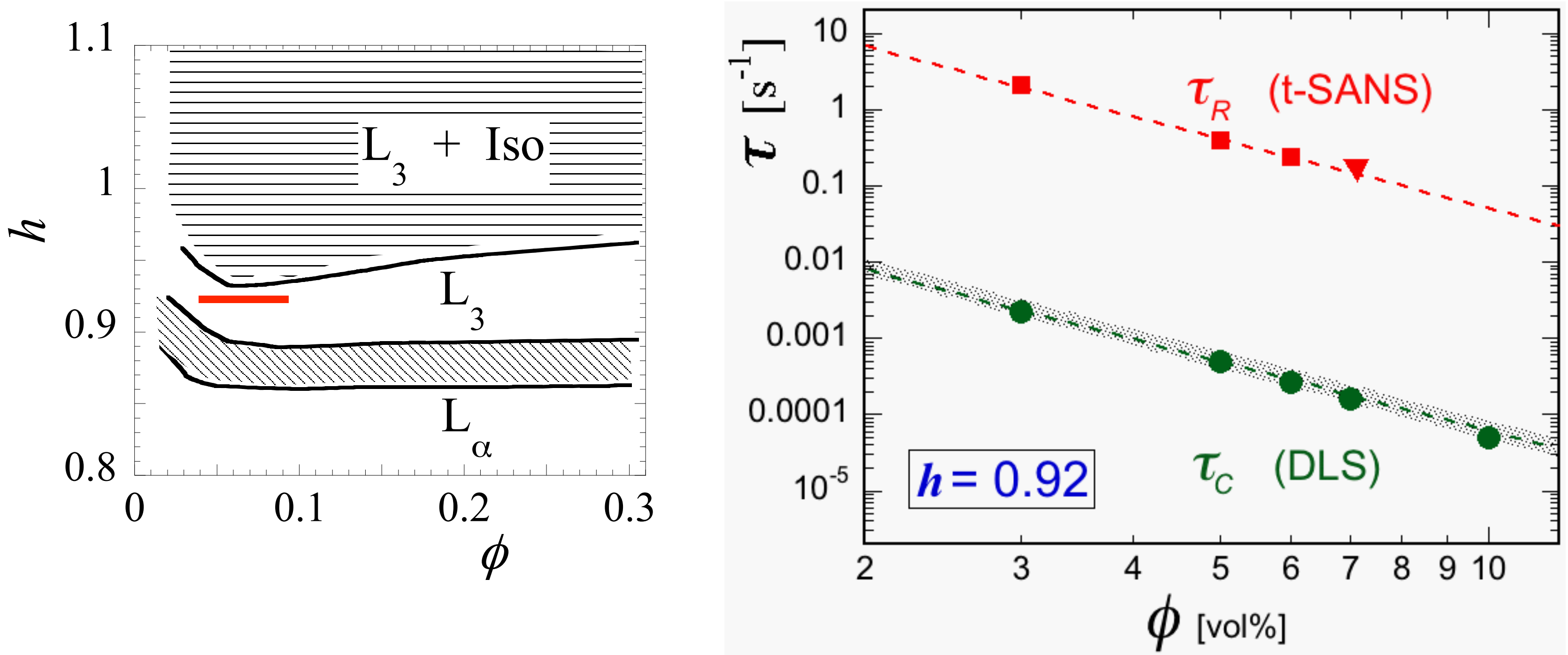
*When Couette cell is stopped*
*$L_3$ signal (passages) re-established*

**$\tau_R$=0.40±0.08 s**

# $\tau_R$ and $\tau_C$ versus membrane volume fraction $\phi$

*Constant membrane composition, i.e. properties*

*Dilution series: $d_3\phi \approx const$, $d_\alpha\phi \approx const$*



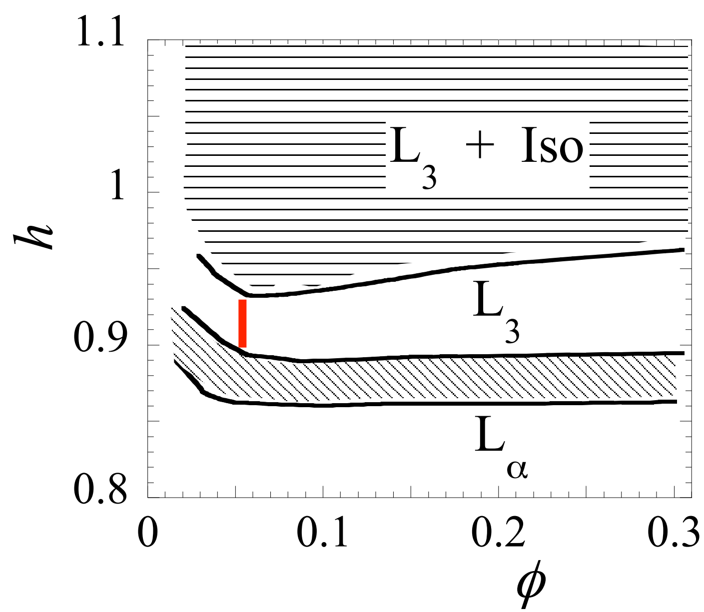

*$\tau_R$ and $\tau_C$ scale as $\phi^{-3}$ (as you might expect per shear response)*

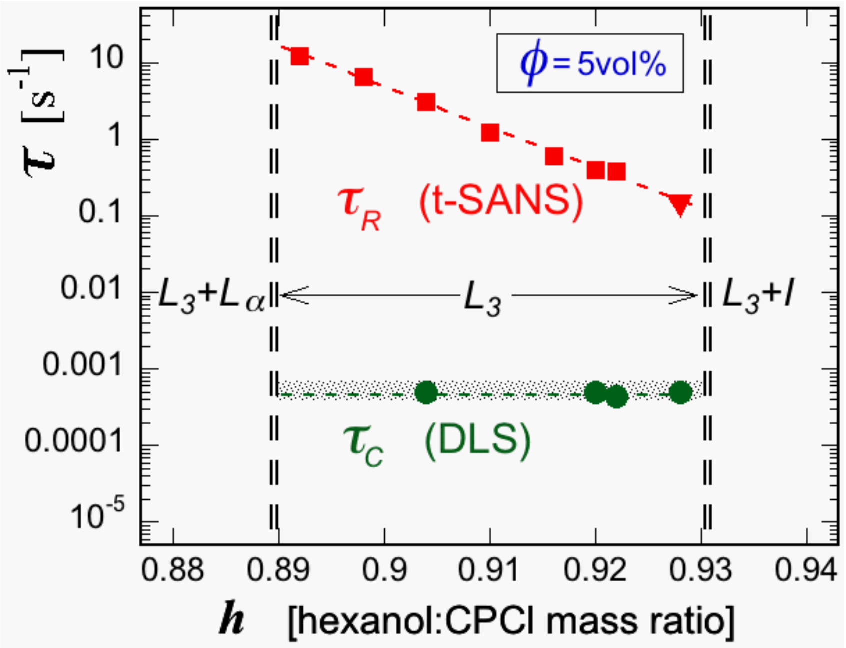
*Constant Arrhenius relationship (<u>**despite**</u> ~3X scaling)*

*$\tau_R = \tau_C \exp[-E_F/k_BT] \Rightarrow E_F = 6.7k_BT$ (170 meV)*

# $\tau_R$ and $\tau_C$ versus membrane composition $h$

*Change membrane composition, i.e. properties across $L_3$ phase region for constant $\phi$*




*Increasing hexanol to CPCl mass ratio $h$*

*⇒ Increasing Gaussian curvature modulus of membranes*

*⇒ Decreasing energy cost of passages (and stalk structures)*

*4% increase in $h$ $E_F = 10.3k_BT$ (260 meV) down to $5.8k_BT$ (150 meV)*

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L. Porcar, W. A. Hamilton, P.D. Butler, and G.G. Warr, *Physical Review Letters*, **93**, 198301 (2004)

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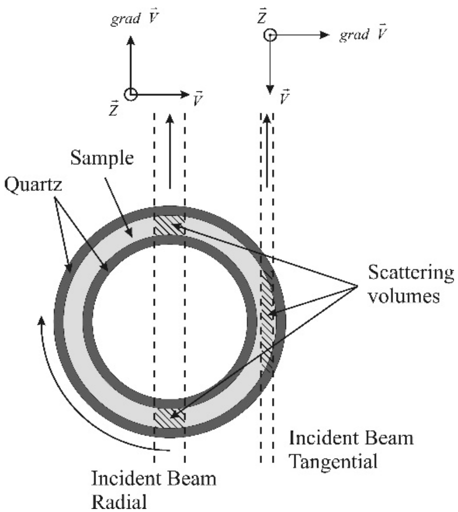
# Sample Environment: What does the data mean?

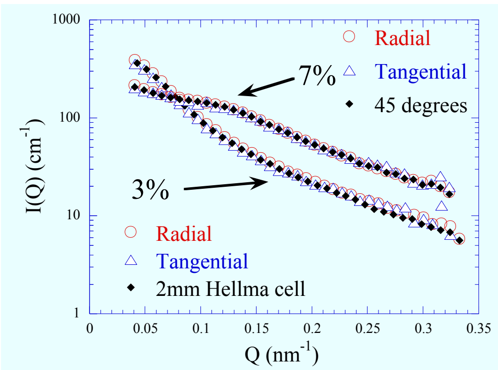
## *Data Reduction*

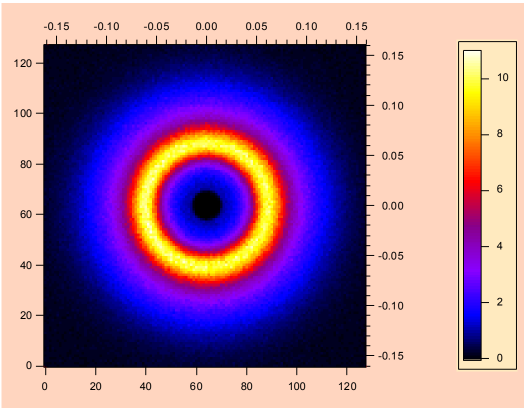



Example: Shear Cell Tangential

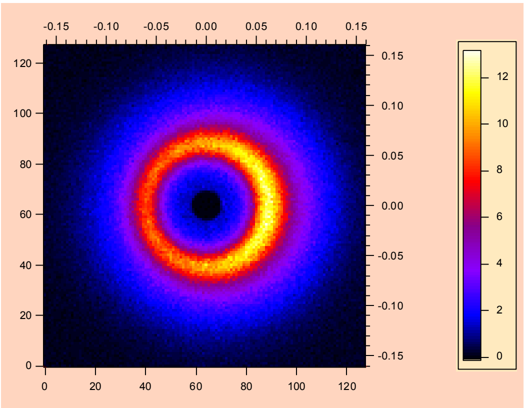
Sample thickness?

Sample absorption?

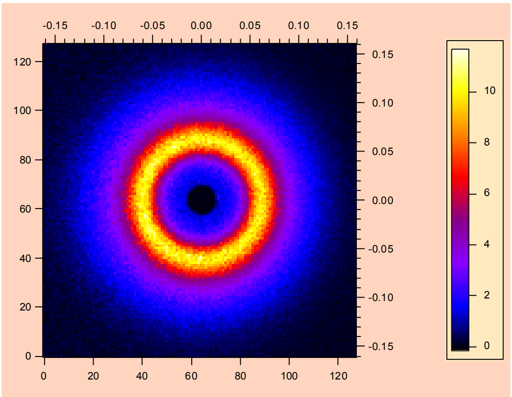


radial



tangential



Corrected tangential

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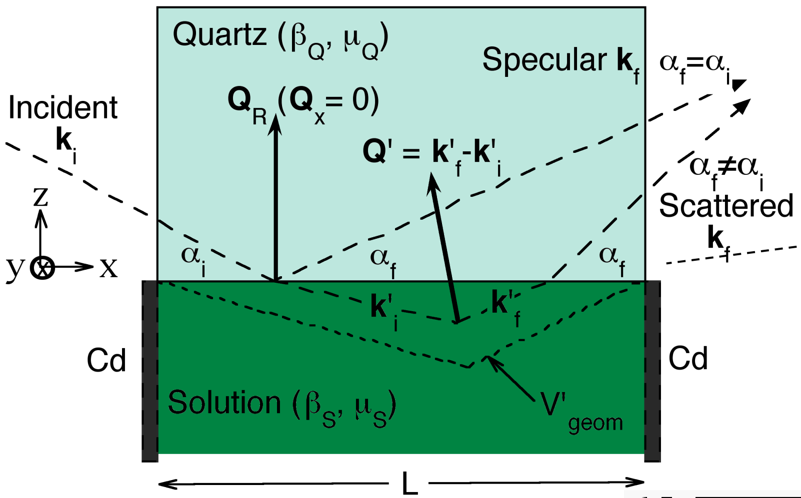
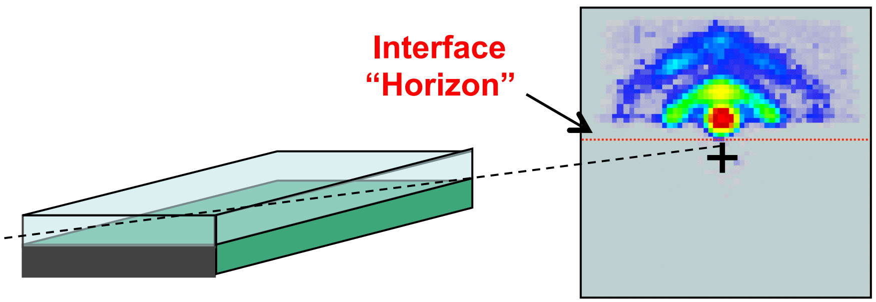
# Sample Environment: What does the data mean?

## *Data Reduction*

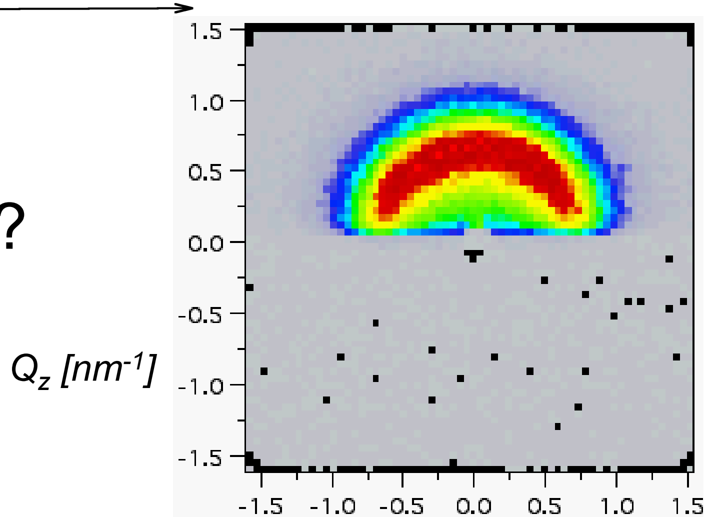
### Example: NSSANS (NOT GISANS)

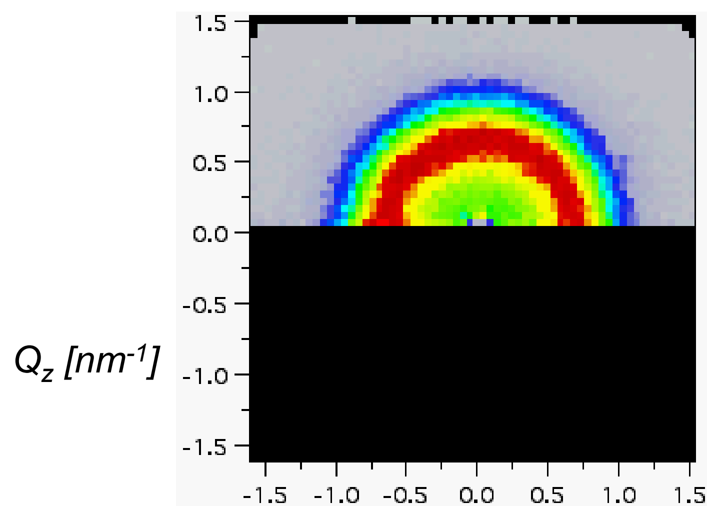


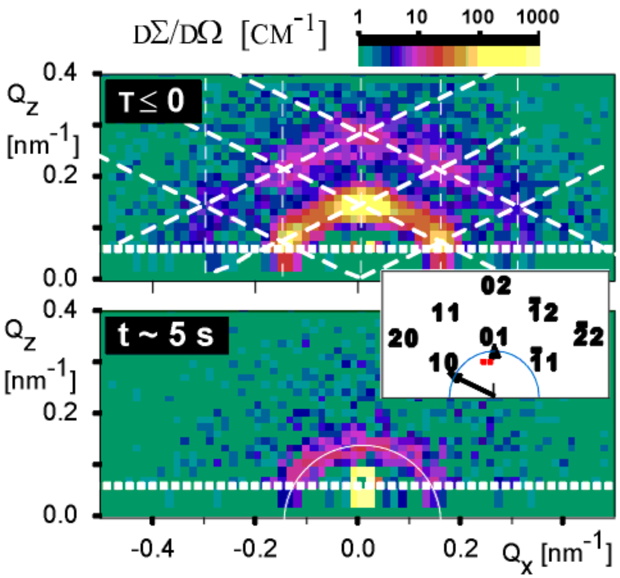

SANS 2D Detector

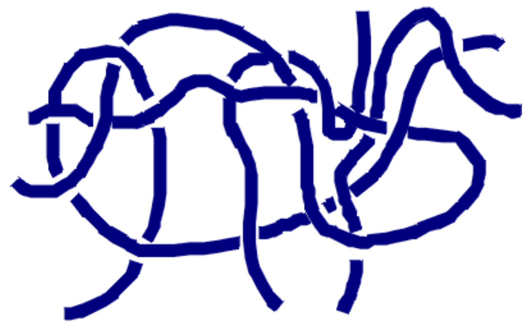
What is q?
Sample volume?
What is I0?

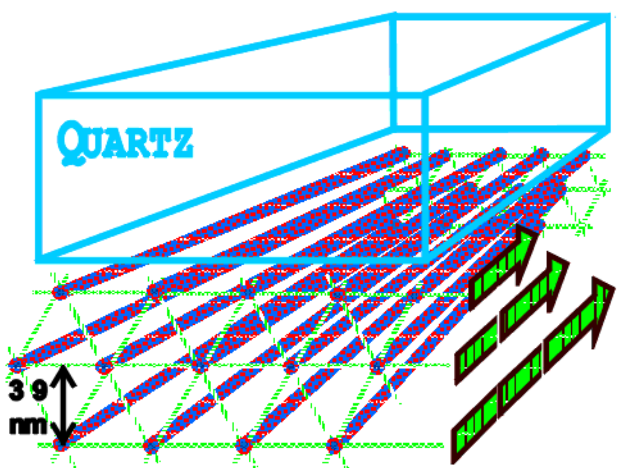


# 20mM 70/30 CTA 3,5Cl/Br Wormlike micelles






<u>Phys. Rev. Let.</u> 72, 2219 (1994)
<u>J. Phys. Chem.</u> 100, 442-445 (1996)
<u>Faraday Discussion</u> 104, 65 (1997)
<u>Phys. Rev. E</u> 60., R1146 (1999)

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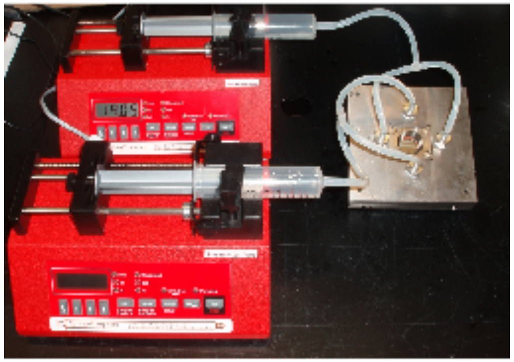
# Sample Environment Conclusions
## *Current directions*

- Multizone temp holders
- FLOW
  - 1,3 and 2,3 RheoSANS … and 1,2 next
  - Extensional flow
  - Rheomicrofludics (pressure driven flow – 10^6/200 atm/ 200C
  - GI-FlowSANS
  - Dielectric RheoSANS
- Pressure – particularly for gas loading but also upgrade 3kbar
- Displex for soft matter – “standard liquid cell holder 2 pos”
- Vapor sorption/flow = nSOFT
- Humidity? Elec field, oscillating magnetic field and pressure

# Sample Environment Conclusions
## *Guiding Principles*

- Get the basics RIGHT …. And stay vigilant.
- Always develop new capabilities around strong "local" scientific programs.
- Make sure our acquisition system remains flexible enough for experiments as well as measurements.
- Need for flexible reduction software.
- Have a team capable of modifying exiting capabilities for one off user needs.
- ALWAYS be opportunistic!
- KISS and don't be shy



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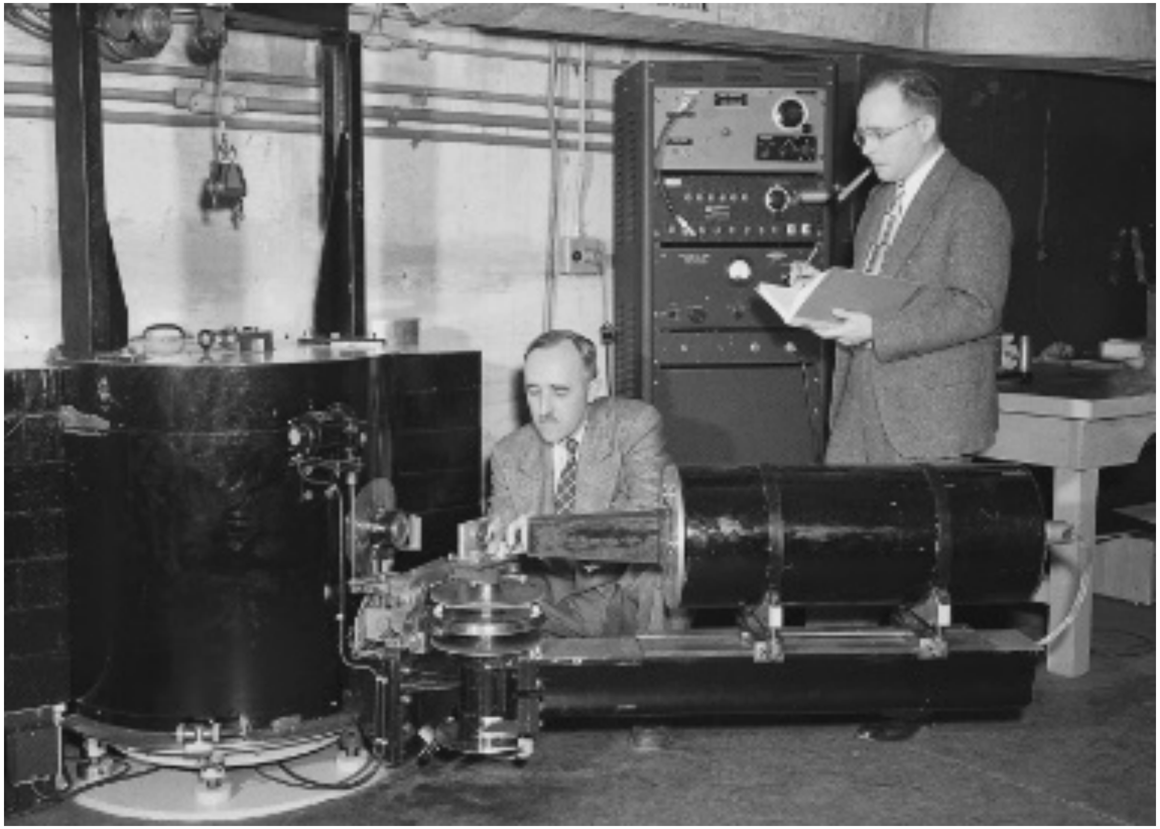
# Sample Environment

# QUESTIONS?
# DISCUSSION?

# Sample Environment: in the beginning was the beam



Then came:

- Minimize air path
- Temperature
- Mag field
- …..
- Shear
- Pressure

Just put in beam and measure

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