Hands-On Neutron Spin Echo Spectroscopy

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Outline

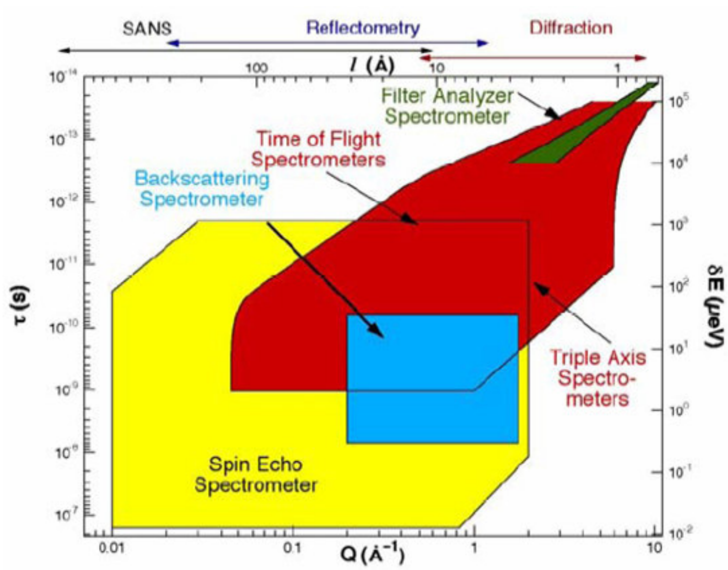
- What a user (you!!!) need to know to propose and perform a successful NSE experiment.
- How a NSE measurement is performed. The data reduction:
  - Exercise.
- Typical examples of data analysis.
  - Shape Fluctuations in a microemulsion.
  - Determination of the bending modulus of the membrane in a lipid vesicle.

What You Should Know of NSE

The preparation is often the most important part of a QENS experiment.

- NSE characteristics.
- NSE technical concept.
- NSE science.
- NSE measurements details.

NSE Characteristics

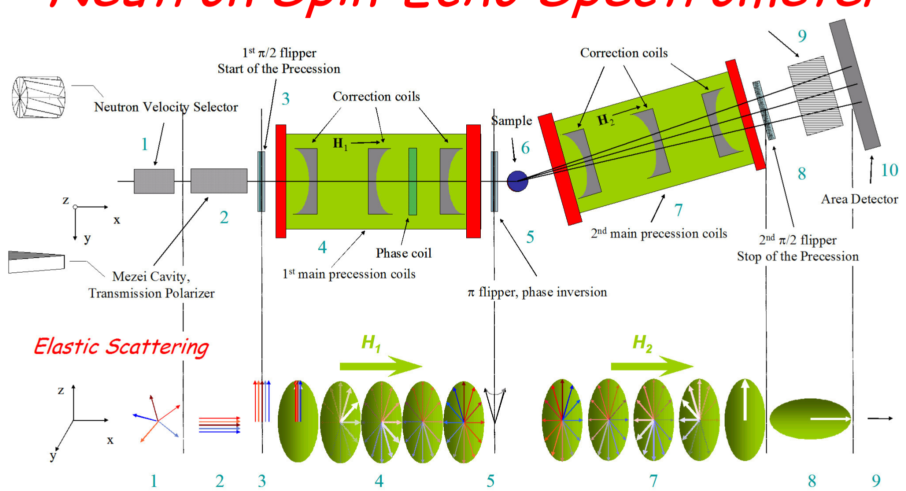


NSE Characteristics

- It is the QENS technique with the highest energy resolution.
- The accessible Q range extends to a portion of the small-angle region.
- It works in the time domain.

  - The instrumental resolution can be simply divided out.
- It can cover up to ~4 orders of magnitude in time.

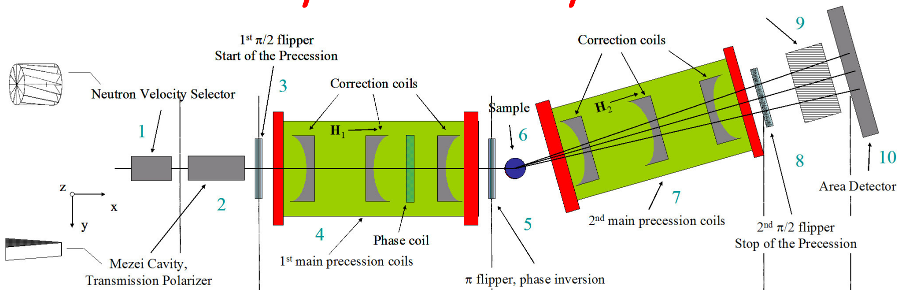
Neutron Spin-Echo Spectrometer

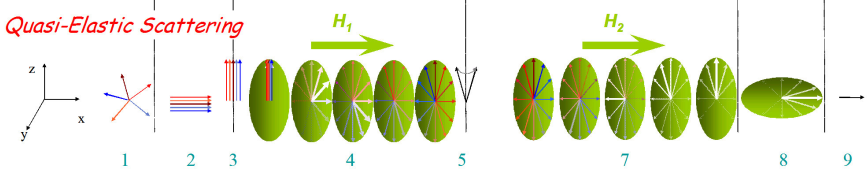


$$\bar{\varphi} = \left\langle \gamma \frac{\int B_0 dl}{v} - \gamma \frac{\int B_1 dl}{v} \right\rangle_{f(\lambda)}$$

Echo Condition: $J_0 = J_1$ $\quad \bar{\varphi} = 0$ $\quad P_x = 1$

Neutron Spin-Echo Spectrometer





$$\varphi = \left\langle \gamma \frac{\int B_0 dl}{v(\lambda)} - \gamma \frac{\int B_1 dl}{v(\lambda) + \delta v} \right\rangle \approx \gamma \frac{m^2 \lambda^3}{2\pi h^2} J_0 \omega + \gamma \frac{m}{h}(J_0 - J_1)\lambda$$

$$P_x = \langle \cos(\varphi) \rangle = \iint f(\lambda) S(Q,\omega) \cos\left[\gamma \frac{m^2 \lambda^3}{2\pi h^2} J_0 \omega + \gamma \frac{m}{h}(J_0 - J_1)\lambda\right] d\lambda d\omega$$

The Fundamental Equations OF NSE

$$P_x = \langle \cos(\varphi) \rangle = \int f(\lambda) \cos\left[\gamma \frac{m}{h}(J_0 - J_1)\lambda\right] d\lambda \times \int S(Q,\omega) \cos\left[\gamma \frac{m^2 \lambda^3}{2\pi h^2} J_0 \omega\right] d\omega$$

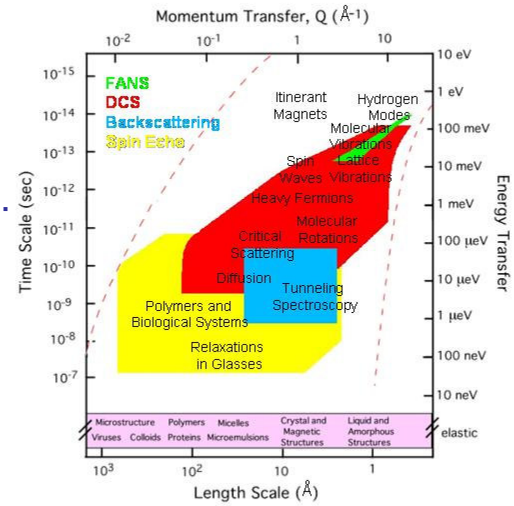
FT of the wavelength distribution — FT of the Dynamic Structure Factor

$$P_x\left(\Delta J^{ph_i}, Q, t\right) = P_s\left(\Delta J^{ph_i}\right) \frac{\int S(Q,\omega) \cos[\omega t] d\omega}{\int S(Q,\omega) d\omega}$$

NSE measures the Fourier Transform of the Dynamic Structure Factor

What Science on NSE

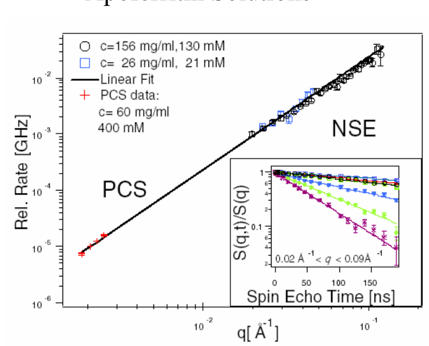
- Diffusion.
  - Collective diffusion: Effects of S(Q) and H(Q).
- Shape Fluctuations.
  - Microemulsion.
  - Membranes.
  - Worm-Like Micelles.
- Glasses.
- Polymer.

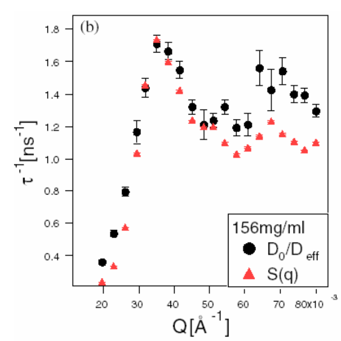


A Few Examples…

Collective Diffusion

Apoferritin Solutions





$$\frac{S(Q,t)}{S(Q,0)} = \exp\left[-D_0 Q^2 t\right]$$

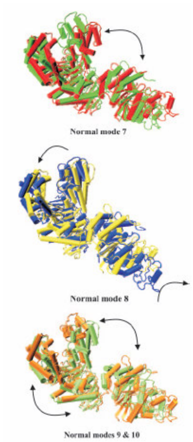
$$\frac{S(Q,t)}{S(Q,0)} = \exp\left[-D_{eff} Q^2 t\right] = \exp\left[-\frac{D_0 Q^2 t H(Q)}{S(Q)}\right]$$

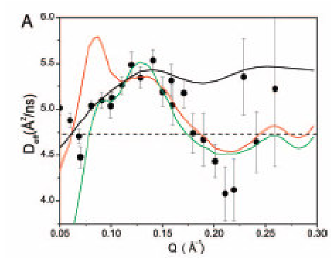
Haußler W. and Farago B., *J. Phys.: Condens. Matter*, **15**, S197 (2003)

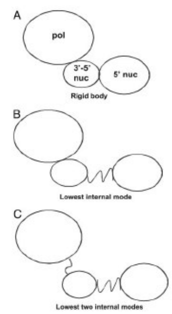
A Few Examples…

Protein Internal Dynamics

Taq Polimerase





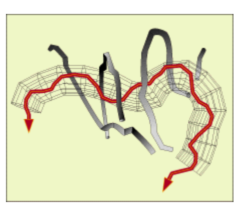


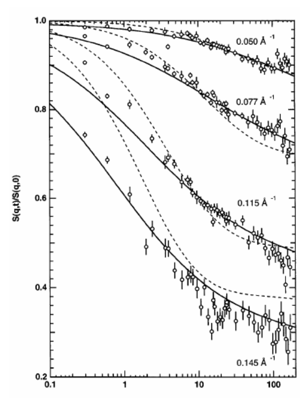
Bu Z., Biehl R., Monkenbusch M., Richter D., and Callaway D.J.E., *Proceedings of the National Academy of Sciences*, **102**, 17646 (2005)

A Few Examples… Polymers I

Reptation

PolyEthylene



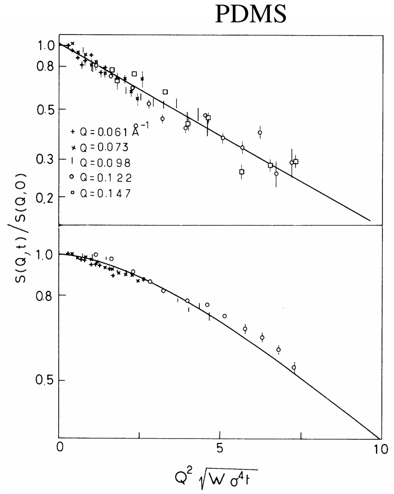


Schleger P., Farago B., Lartigue C., Kollmar A. and D. Richter, *Phys. Rev. Lett.*, **81**, 124 (1998)

A Few Examples… Polymers II

Rouse Dynamics

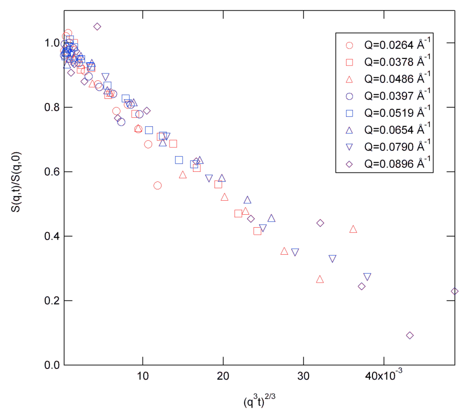
PDMS



Richter D., Ewen B., Farago B., and Wagner T., *Phys. Rev. Lett.*, **62**, 2140 (1989)

Zimm Dynamics

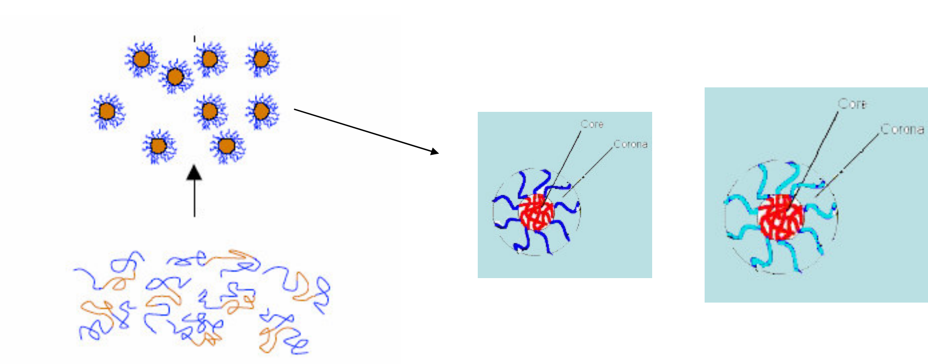
PS/d-Octane



A Few Examples…

Corona Dynamics in Pluronic Triblock Copolymer Micelles

F68 0.30 gr/cm^3



Yardimci H., Chung B., Harden J.L., and Leheny R.L., *J. Chem. Phys.*, **123**, 244908 (2005).

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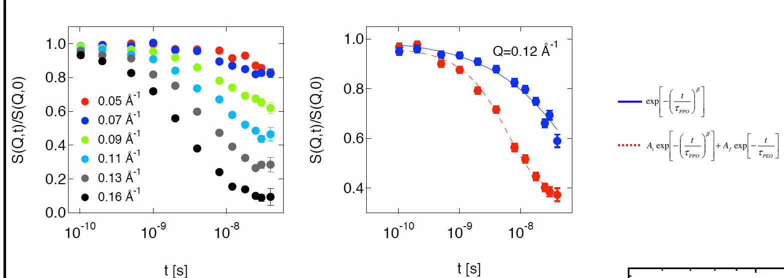

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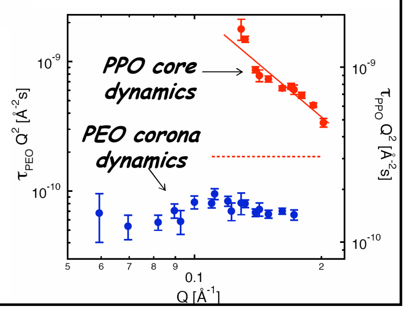
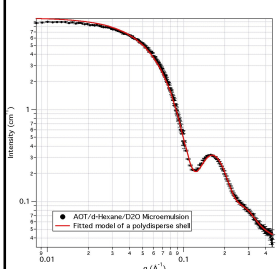
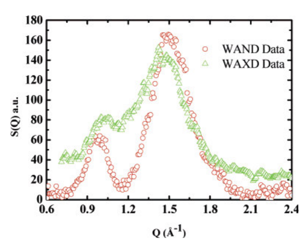
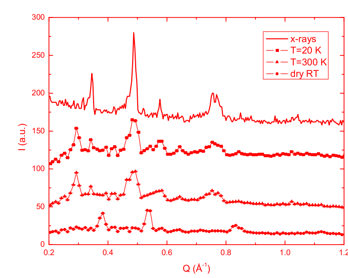
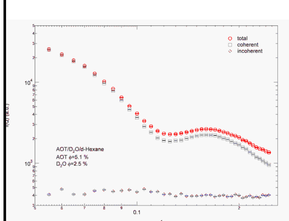


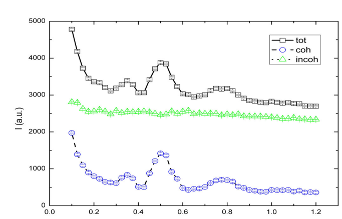
The Details:
Choosing the right Q











What You Should Know of NSE Take Home Messages

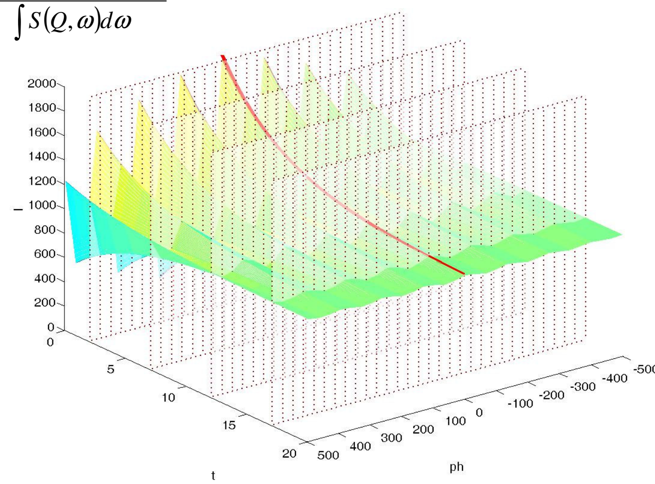
- The Q-t range accessible to NSE is broad and is best for the investigation of slow processes on large-medium length scales.
- The High Energy Resolution of NSE is achieved by encoding the neutron velocity into its Spin state.
- NSE is the instrument of choice for studying coherent (collective) dynamics in Soft Condensed Matter.
  - Collective Diffusion, Shape Fluctuation,…

The NSE Measurement

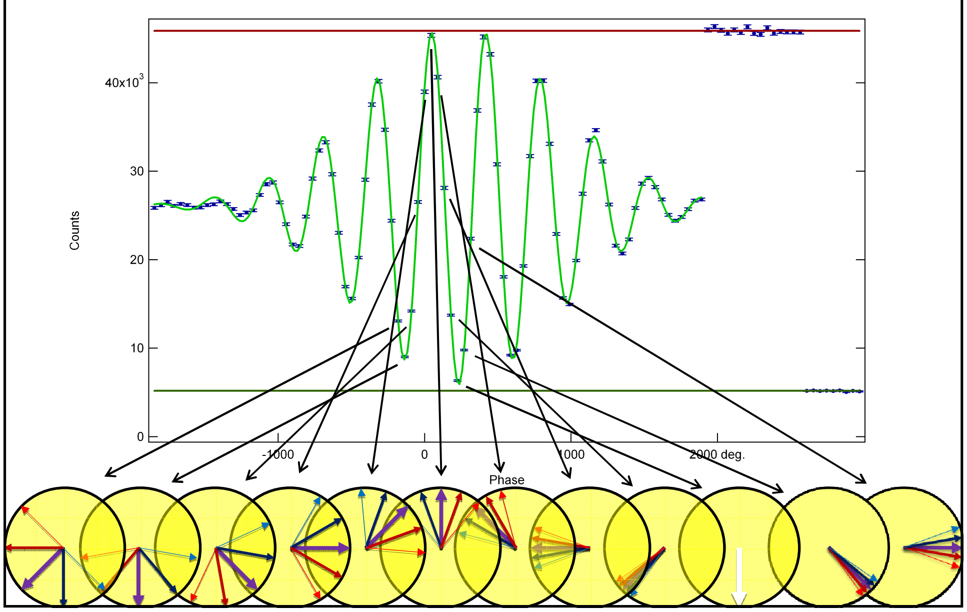
- What does NSE measure?
  – Polarization vs Phase and F_t.
  – How to Eliminate Instrument Dependent Signals.
- How using a 2D detector complicates things?
  – How DAVE helps us out.

Polarized Intensity vs phase and F_t

$$P_x\left(\Delta J^{ph_i}, Q, t\right) = P_s\left(\Delta J^{ph_i}\right)\frac{\int S(Q,\omega)\cos[\omega t]d\omega}{\int S(Q,\omega)d\omega}$$



Polarized Intensity vs phase



Fitting the echo

I_0 Average Intensity

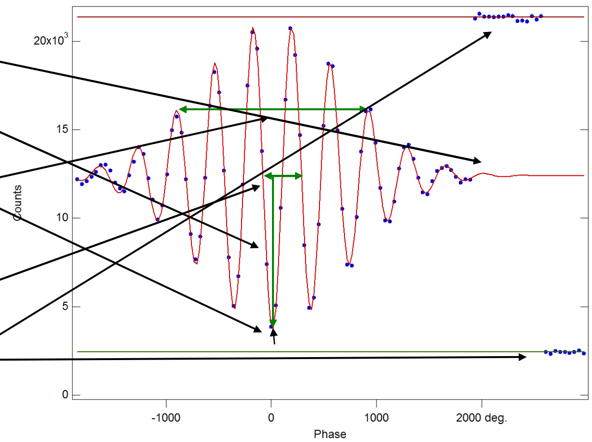
A Amplitude, related to I(Q,t)

ph_0 Echo point

σ Echo width, function of f(λ) width distribution

T Period, function of ⟨λ⟩

Static measurements (Up and Down) t=0 (J=0): ∫S(Q,ω)dω



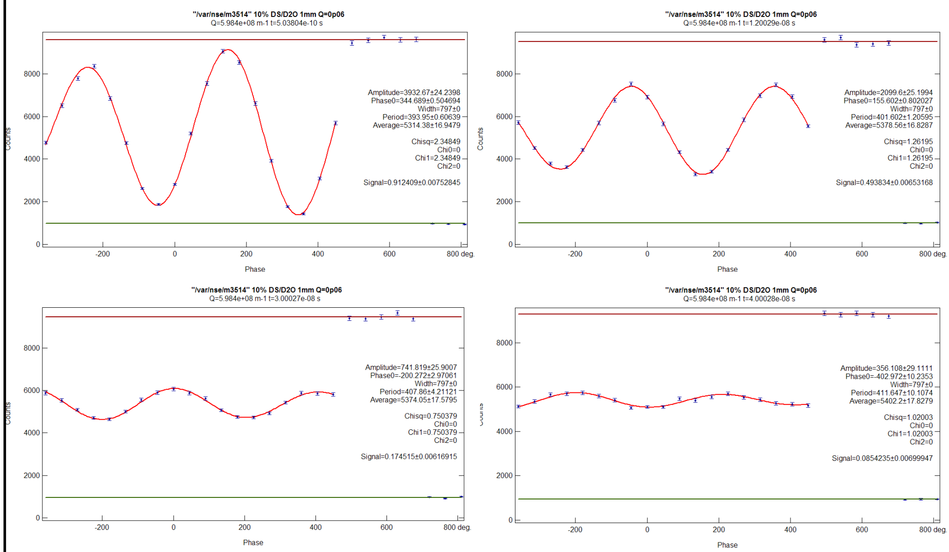
$$I^p = I_0 + A\exp\left[\frac{(ph-ph_0)^2}{2\sigma^2}\right]\cos\left[\frac{360}{T}(ph-ph_0)\right]$$

The Physical Information is All in the Amplitude

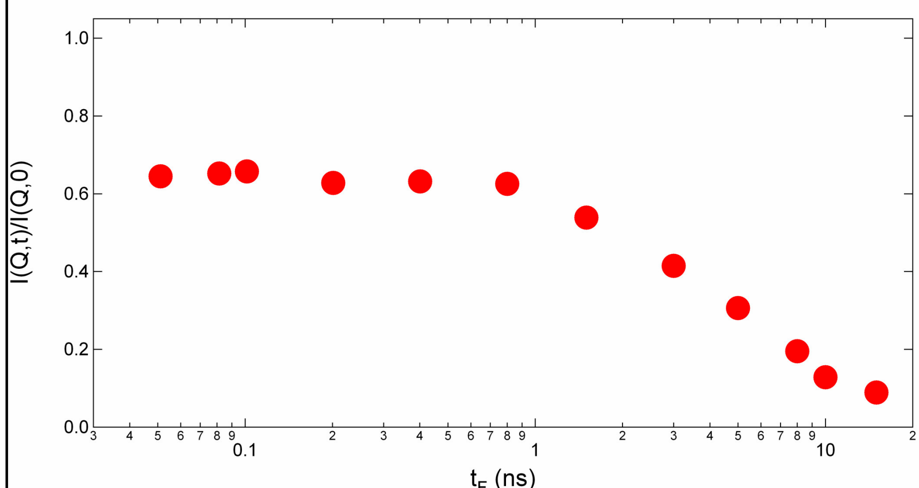
$$\frac{I(Q,t)}{I(Q)} \propto \frac{2A}{Up-Dwn}$$

Incidentally, in this way, both polarization and detector efficiency effects are taken care off.

Polarized Intensity vs F_t



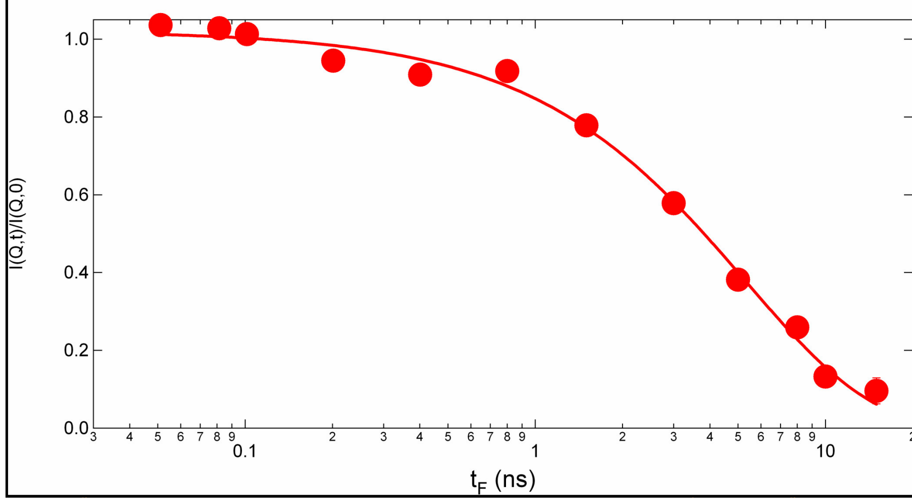
Resolution Normalization



$$\frac{I(Q,t)}{I(Q,0)} = \frac{2A/(Up - Dwn)}{2A^R/(Up^R - Dwn^R)}$$

Blank correction

$$\frac{I(Q,t)}{I(Q)}=\frac{2\left[A-(1-\phi)\dfrac{T}{T^{BKG}}A^{BKG}\right]\Big/\left[(Up-Dwn)-(1-\phi)\dfrac{T}{T^{BKG}}\left(Up^{BKG}-Dwn^{BKG}\right)\right]}{2A^{R}\big/\left(Up^{R}-Dwn^{R}\right)}$$



The NSE Measurement Take Home Messages

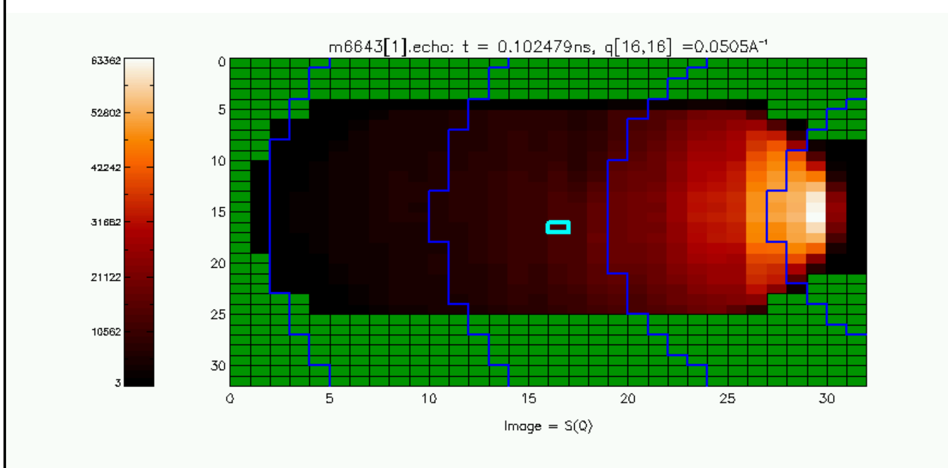
- The Physical Information is in the Echo Amplitude.
- But… You Have to Accurately Fit the Echo to get the Amplitude right.
- The Resolution can be simply divided out.

Using a Big Detector Makes Things Harder

Questions:

- What are the advantages of using a 2D detector?
- What are the problems that a 2D detector gives for the reduction of NSE data?

2D Detector



Total Data=$32\times32\times N_{phase}\times N_{Ft}\approx500000$

Dynamics of Soft Condensed Matter **ACNS 2008** Santa Fe, NM May 10th 2008

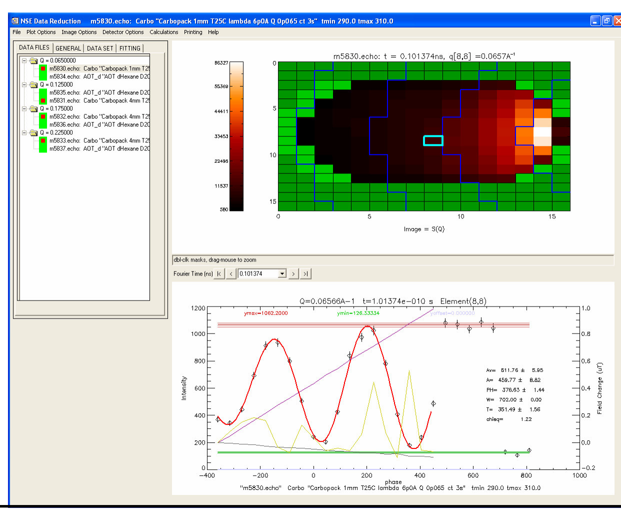
Questions II

- What are those thin blue lines?
- Does the polarized intensity change with the pixel position? Why?

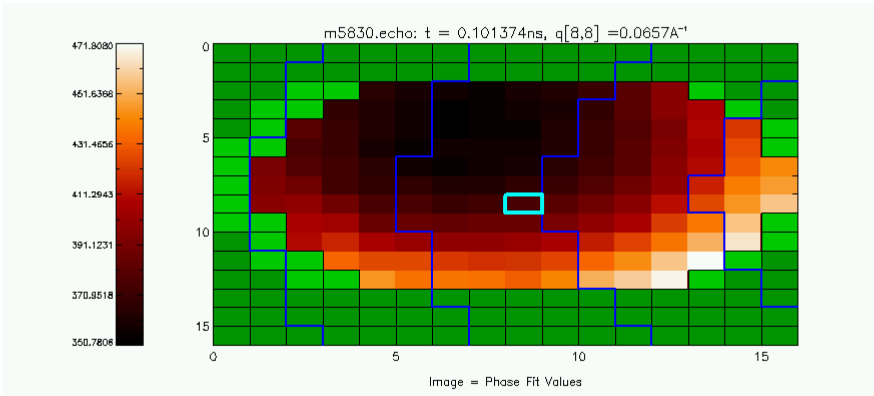
Up and Down	• Yes. Efficiency, Polarization.
I_0	• Yes.
A	• Yes. Q-dependence.
T	• No.
σ	• No.
Ph_0	• Yes. Field Integral: ∫Bdl.

2D Detector Analysis

The echoes at each detector pixel have to be fitted individually.



Phase Map



The Phase Map should be a smoothly varying function of the position on the detector and of the Fourier time.

Reducing NSE data with DAVE

1. Mask low intensity areas.
2. Fit the resolution.
3. Make sure the phase map is correct.
4. Remove poor resolution points.
5. Check the χ^2.

Importing the phase map

The phase map is sample independent. To fit your sample and background data just…

1. Import the phase map from the resolution.
2. Check the χ^2.

Calculate I(Q,t)

$$\frac{I(Q,t)}{I(Q)} = \frac{2\left[A-(1-\phi)\frac{T}{T^{BKG}}A^{BKG}\right] \Big/ \left[(Up-Dwn)-(1-\phi)\frac{T}{T^{BKG}}\left(Up^{BKG}-Dwn^{BKG}\right)\right]}{2A^{R} \Big/ \left(Up^{R}-Dwn^{R}\right)}$$

The Intermediate Scattering function values are calculated pixel by pixel and averaged, according to their weight, by Q areas.

Conclusion

At the end of the reduction process the I(Q,t) contains information about your sample only.