UT-BATTELLE

Triple-Axis Spectroscopy

Mark Lumsden

HFIR

SNS
SPALLATION NEUTRON SOURCE

OAK RIDGE NATIONAL LABORATORY
U. S. DEPARTMENT OF ENERGY

References

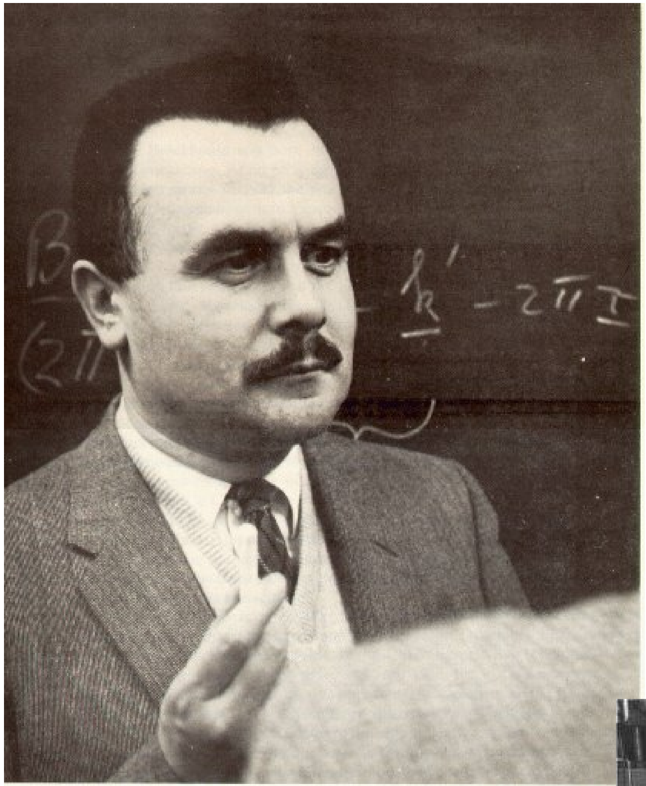
G.L. Squires, **"Introduction to the Theory of Thermal Neutron Scattering"**
http://www.amazon.com/Introduction-Theory-Thermal-Neutron-Scattering/dp/048669447X

S.W. Lovesey, **"Theory of Neutron Scattering from Condensed Matter"**

G. Shirane, S.M. Shapiro, J.M. Tranquada, "**Neutron Scattering with a Triple-Axis Spectrometer: Basic Techniques"**
http://www.amazon.com/Neutron-Scattering-Triple-Axis-Spectrometer-Techniques/dp/0521025893/

History

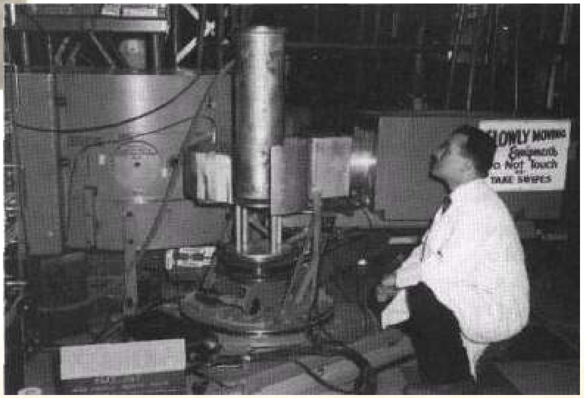
1994 Nobel Prize in Physics



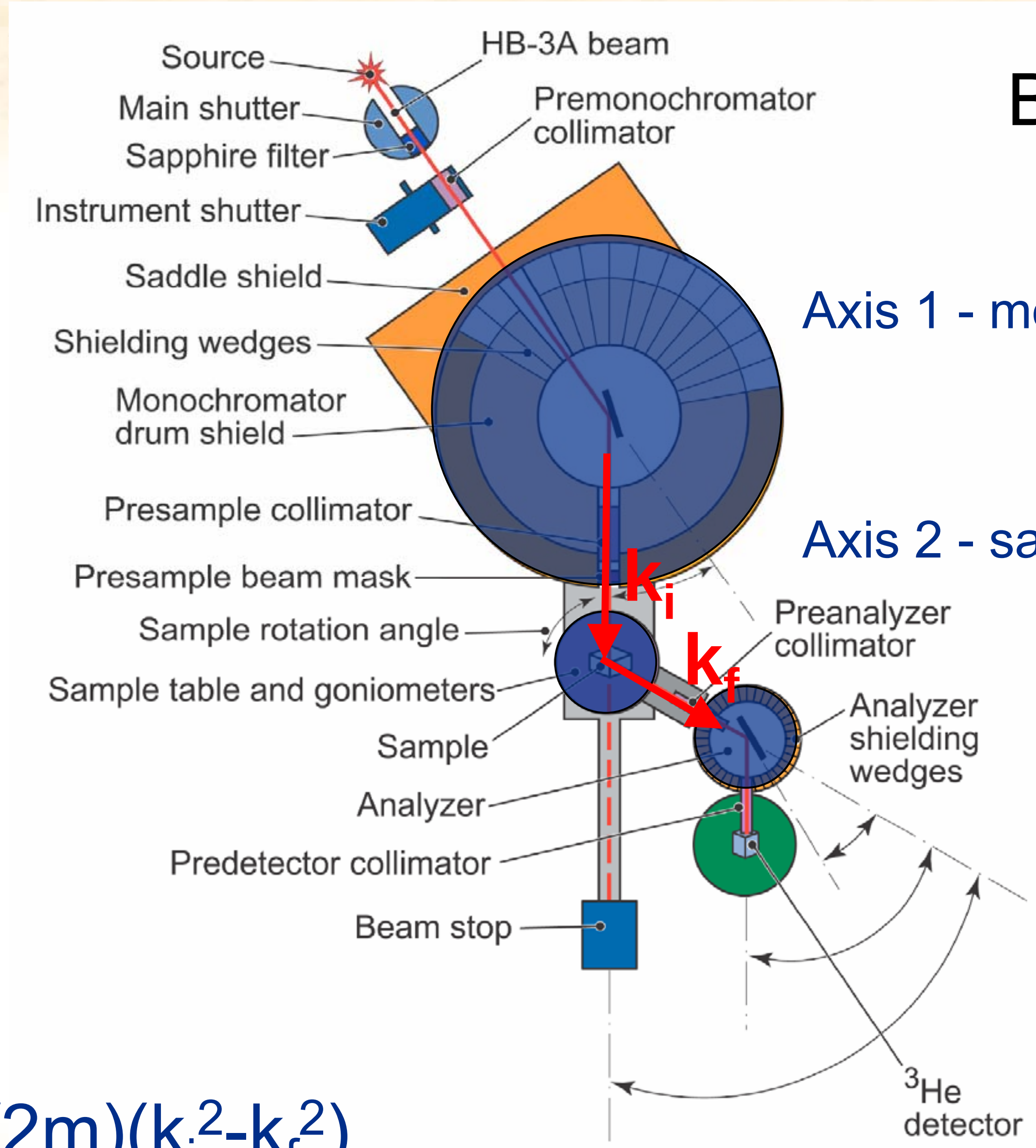
"for pioneering contributions to the development of neutron scattering techniques for studies of condensed matter"

Clifford G. Shull:
"for the development of the neutron diffraction technique"

Bertram N. Brockhouse:
"for the development of neutron spectroscopy"



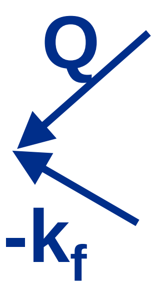
Bragg's Law:
$\lambda = 2d \sin\theta$



Axis 1 - monochromator

Axis 2 - sample

Axis 3 - analyzer



$Q = k_i - k_f$

$\Delta E = (\hbar^2/2m)(k_i^2 - k_f^2)$

recip_space_2.avi

TAS Instruments at HFIR: HB1A Ames Laboratory Fixed E_i TAS

Fixed E_i=14.6 meV

Good for elastic and lower energy inelastic measurements (<~7meV).
Very clean beam
(very little $\lambda/2$ contamination:
$\lambda/2 \sim 10^{-4}\ \lambda$)

Instrument scientist: Jerel Zarestky

TAS Instruments at HFIR: HB1 Polarized TAS

Instrument scientist: Andrey Zheludev

Currently, unpolarized with vertically focusing PG002 monochromator.

Polarized capabilities should be ready in late 2008.

Original instrument where Moon, Riste, and Koehler did initial polarized triple-axis measurements Phys. Rev. **181** 920 (1969).

TAS Instruments at HFIR: HB3 TAS

Instrument scientist: Mark Lumsden

<u>Choice of 3 monochromators:</u>

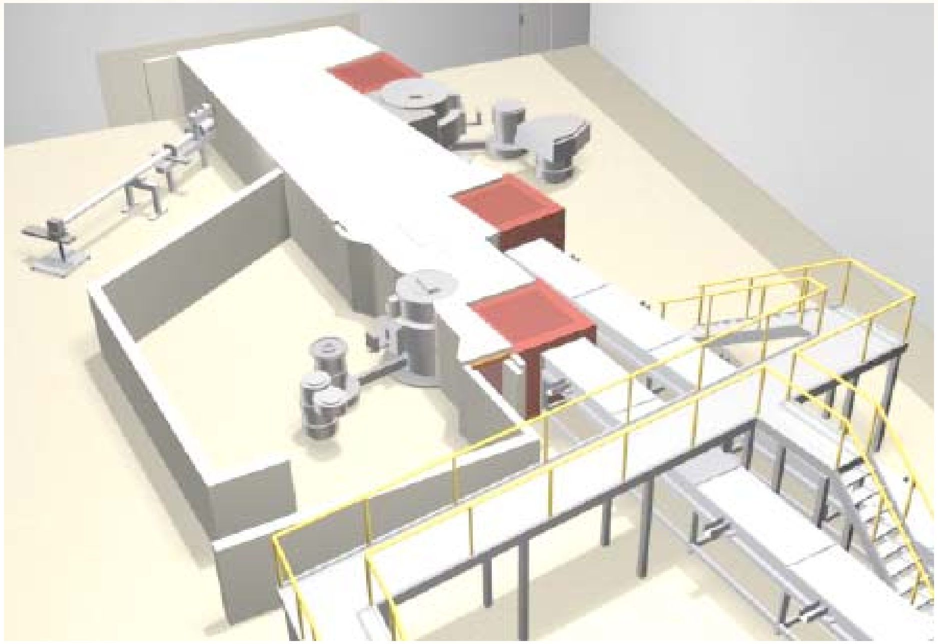
PG 002 – best intensity

Si 111 – no $\lambda/2$

Be 002 – good resolution at higher energies

All vertically focused.
Approximate beam height at sample is about 1”

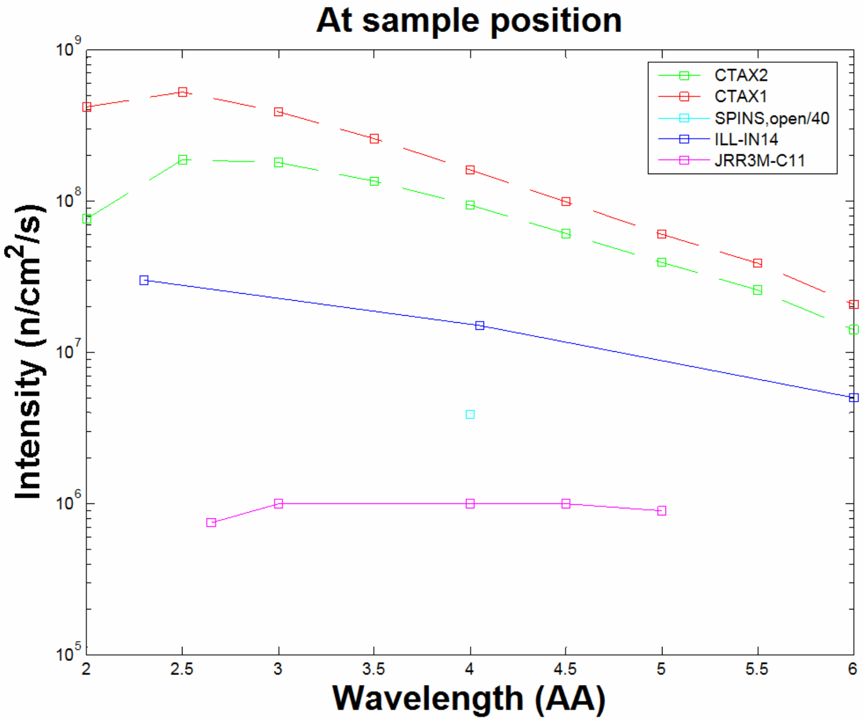
TAS Instruments at HFIR:
Future – cold TAS instruments



US-Japan Cold TAS – CG4C:

E_i 2-20 meV

Flux ~ 10^8 n/cm²/s



CG1 TAS (VICTOR):

IDT Formed – guide in place

Highest flux cold TAS in world

Multi-blade analyzer

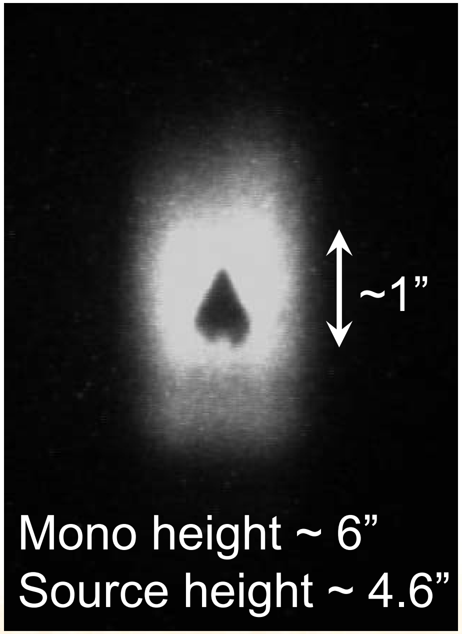
Experimental Configuration - Monochromators

Monochromator	d-spacing	Comment
PG 002	3.35 Å	Very high reflectivity – best intensity
Si 111	3.135 Å	No $\lambda/2$ but lower intensity (good signal:noise)
Be 002	1.79 Å	Smaller d-spacing – good resolution at higher ΔE
Heusler	3.437 Å	Polarizing monochromator

All vertically focusing:

$$\frac{1}{L_{Source-Mono}} + \frac{1}{L_{Mono-Sample}} = \frac{2\sin(\theta_M)}{R}$$

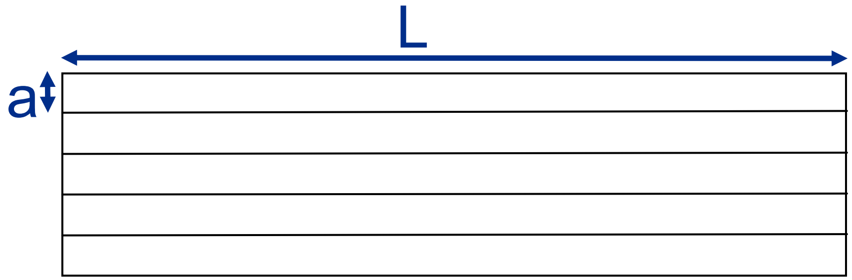
As lengths are fixed, need to adjust R for each θ_M to maintain focus



Experimental Configuration - collimators

Collimations:

Source-Monchromator
Monochromator-Sample
Sample-Analyzer
Analyzer-Detector



Collimation $\alpha = a/L$

Helps define resolution – example (Rescal):

48’-40’-40’-120’ E_f=14.7meV ΔE=0.92meV I=1
48’-20’-20’-120’ E_f=14.7meV ΔE=0.75meV I~0.44

http://www.ill.fr/Computing/resources/software/matlab/

Role of filters

Reduce fast neutron background – significant source of background on all TAS instruments

Bragg's Law: $\lambda = 2d \sin\theta$

If satisfy scattering condition for λ **you will also for $\lambda/2$, $\lambda/3$, … assuming reflections with d/2, d/3, … exist.**

Want a filter to transmit λ with high efficiency but reject $\lambda/2$ and $\lambda/3$ (higher orders aren't typically relevant due to reactor spectrum)

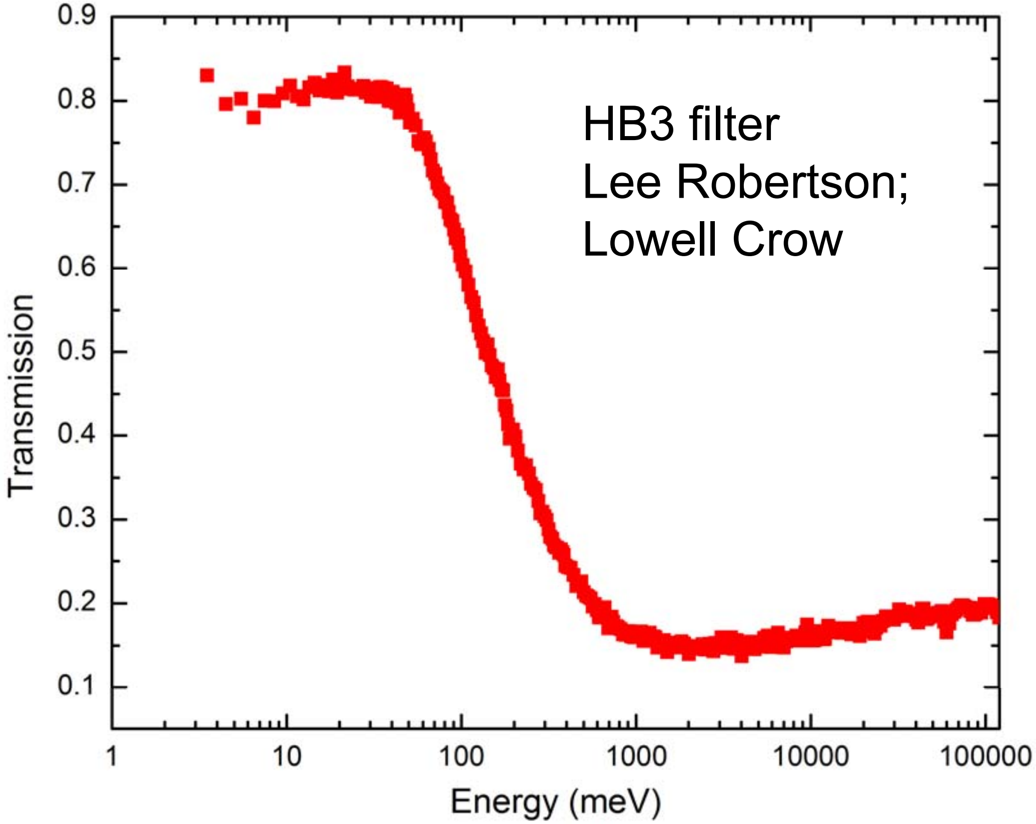
Experimental Configuration - filters

Sapphire fast neutron filter

Present on all instruments

Inelastic phonon and multiphonon processes dominate at high energies reducing transmission

Greatly reduces fast neutron background of instruments



Experimental Configuration - filters

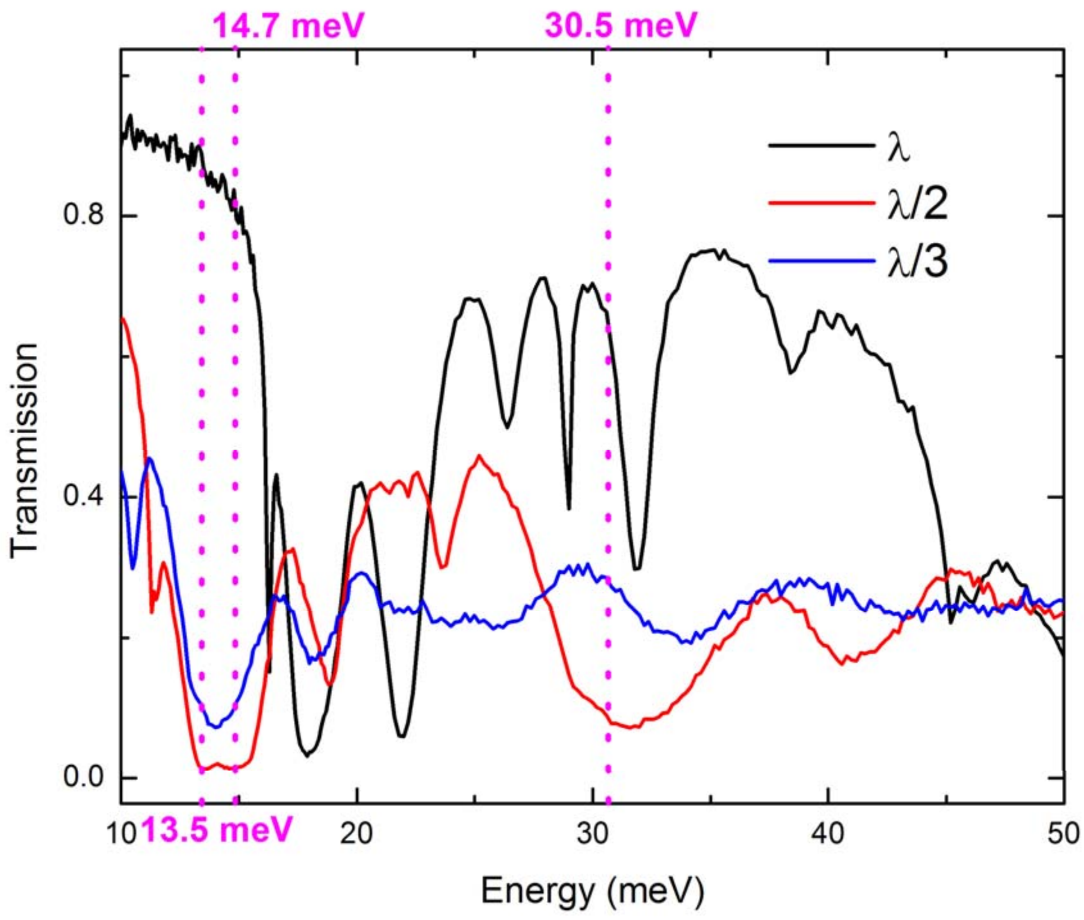
PG Filter:

PG with c-axis along beam direction.

Bragg scattering occurs for k values where

$2\,k\,\sin(90-\phi_{hkl})=\mathbf{G_{hkl}}$

Where ϕ_{hkl} is angle between reciprocal lattice vector $\mathbf{G_{hkl}}$ and c-axis.



Experimental Configuration - filters

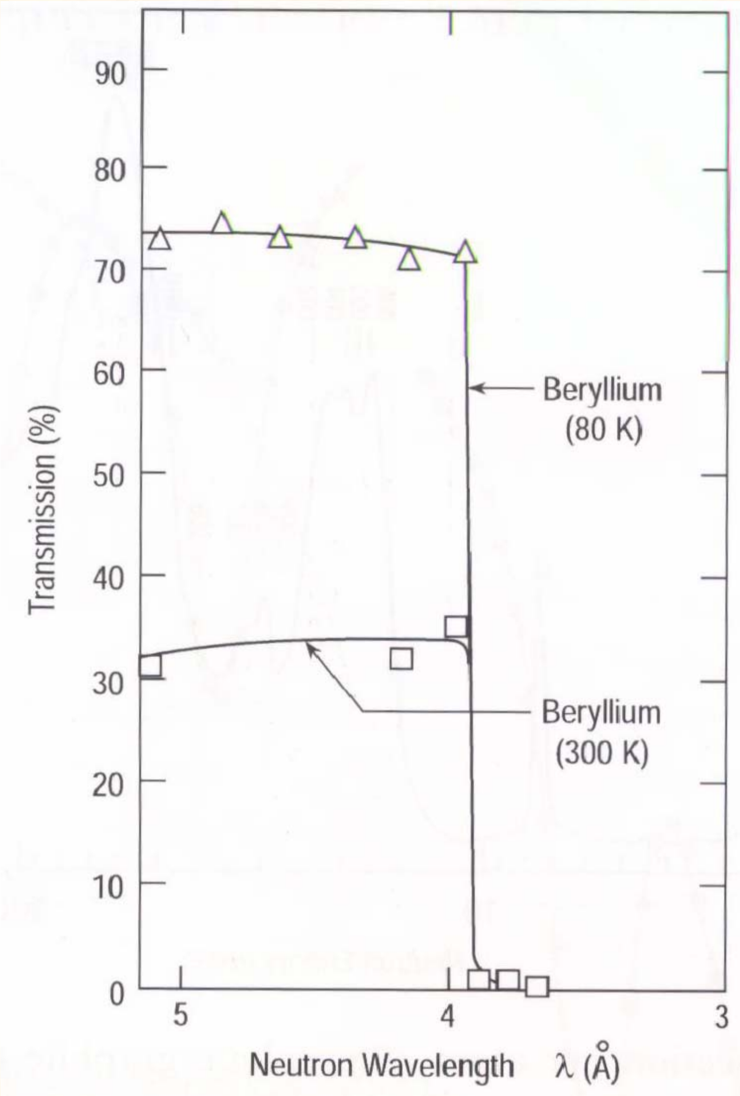
Be/BeO filter:

Bragg scattering filters

Max wavelength for which Bragg scattering can occur is:

$\lambda_{cutoff}=2d_{max}$

$\lambda/2d < 1$: for $\lambda>\lambda_{cutoff}$, can have no Bragg scattering and beam gets transmitted.



TAS Experiment – before arriving

<u>Sample size:</u>

Powder – typically 10-50g

Single crystal: > 300 mg (preferred > 1g)

Often need to coalign several crystals to attain enough mass.

TAS Experiment – before arriving

Characterize sample before arriving – at least, examine with x-rays. NOTE: x-rays examine near-surface while neutrons examine bulk.

Know what scattering plane you want to explore – TAS measures in a plane

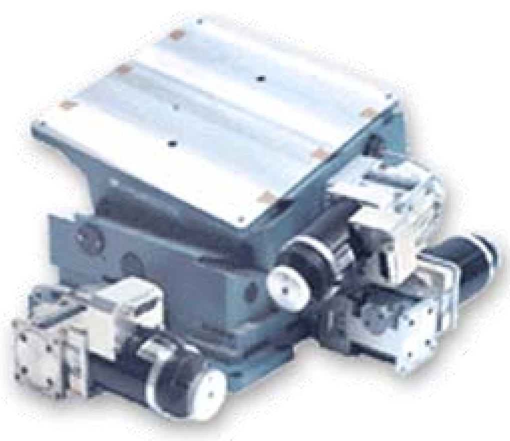
Know structure and come with a list of Bragg reflection intensities

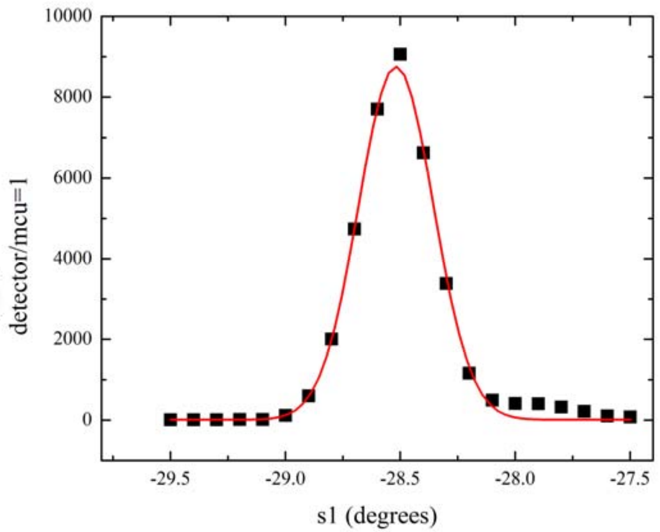
TAS Experiment – align sample

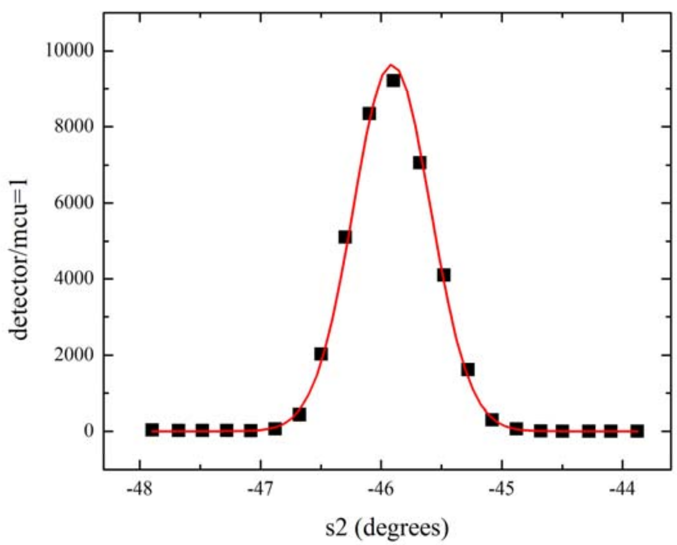
Software at ORNL uses UB matrix to define orientation.

M.D. Lumsden, J.L. Robertson, and M. Yethiraj, J. Appl. Cryst. 38 (2005) 405.

Perform transverse and longitudinal scans through 2 or more reflections. Also scan arcs at each reflection. Input the lattice constants. Tell the computer what the reflections are (for instance, the current spectrometer angles give me the (200) Bragg peak).

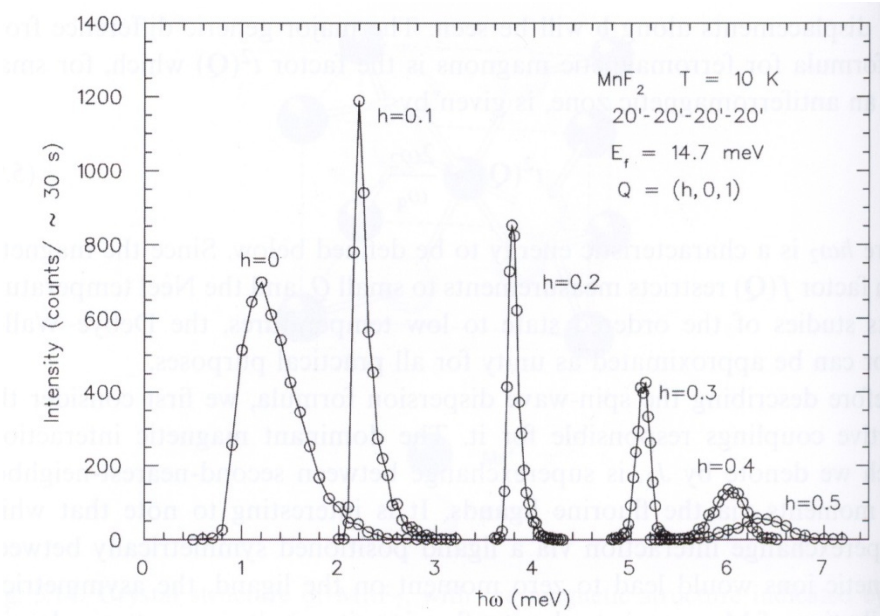


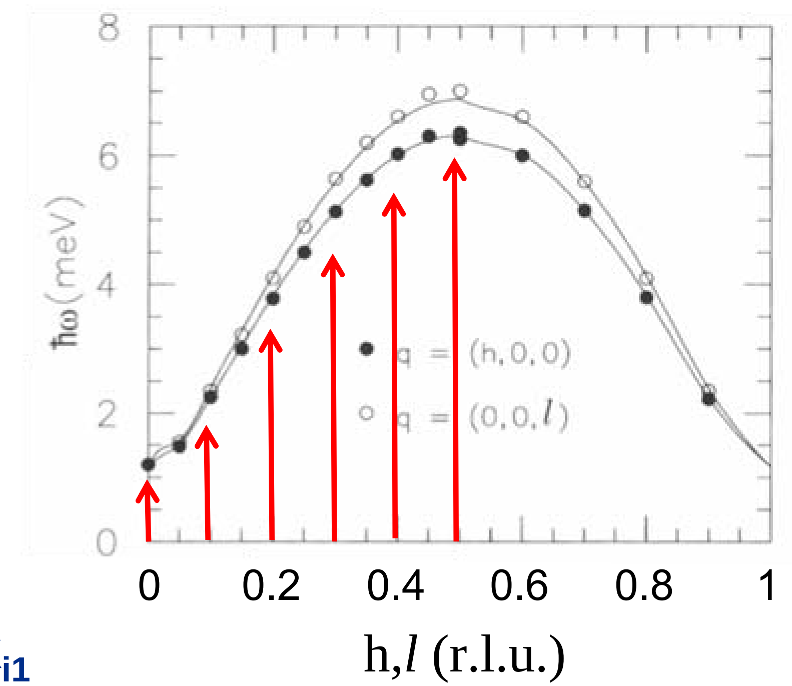


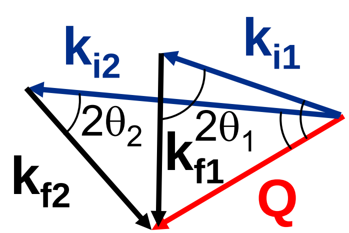


TAS Experiment – scans

Constant-Q scan – concept developed by Brockhouse



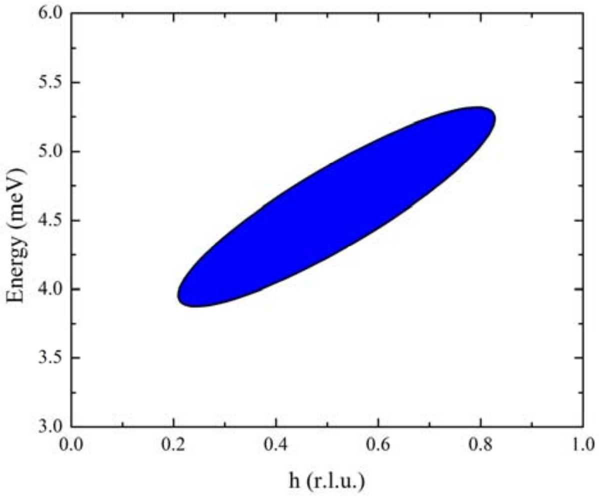




TAS Experiment – resolution

The resolution of the TAS is dependent on:

1. Monochromator/analyzer d-spacing
2. Monochromator/analyzer mosaic spreads
3. Sample mosaic spread
4. Collimations (S-M; M-S; S-A; A-D)
5. Distances (not in simplest approximation)

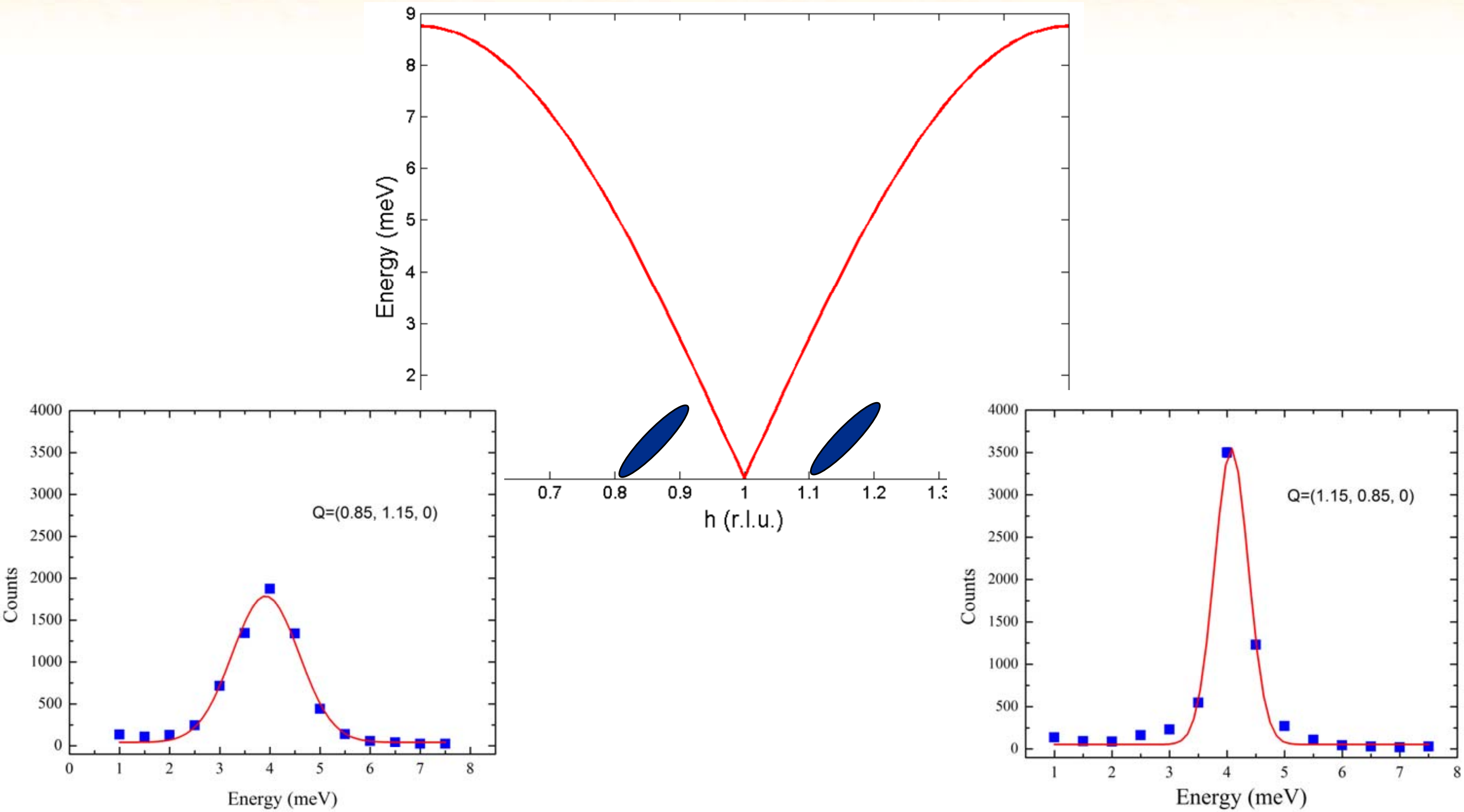


Shape: cigar-like ellipsoid

What we measure:

$$I(\vec{Q}_0, \omega_0) \propto \iint R(\vec{Q} - \vec{Q}_0, \omega - \omega_0) S(\vec{Q}, \omega) d\vec{Q} d\omega$$

TAS Experiment – resolution - focusing



TAS Experiment – resolution

Proper way to analyze data is to convolute expected S(**Q**,ω) with instrumental resolution.

Reslib - A. Zheludev (MATLAB)
http://neutron.ornl.gov/~zhelud/reslib/index.html

References:

M. J. Cooper and R. Nathans, *Acta Cryst.* (1967) **23**, 357-367**.**

N. J. Chesser and J. D. Axe, *Acta Cryst.* (1973) A**29**, 160-169.

M. Popovici, *Acta Cryst.* (1975) A**31**, 507-513.