 

CCLRC Rutherford Appleton Laboratory

ISIS

Time of Flight instrumentation for powder diffraction :

techniques, applications and new perspectives.

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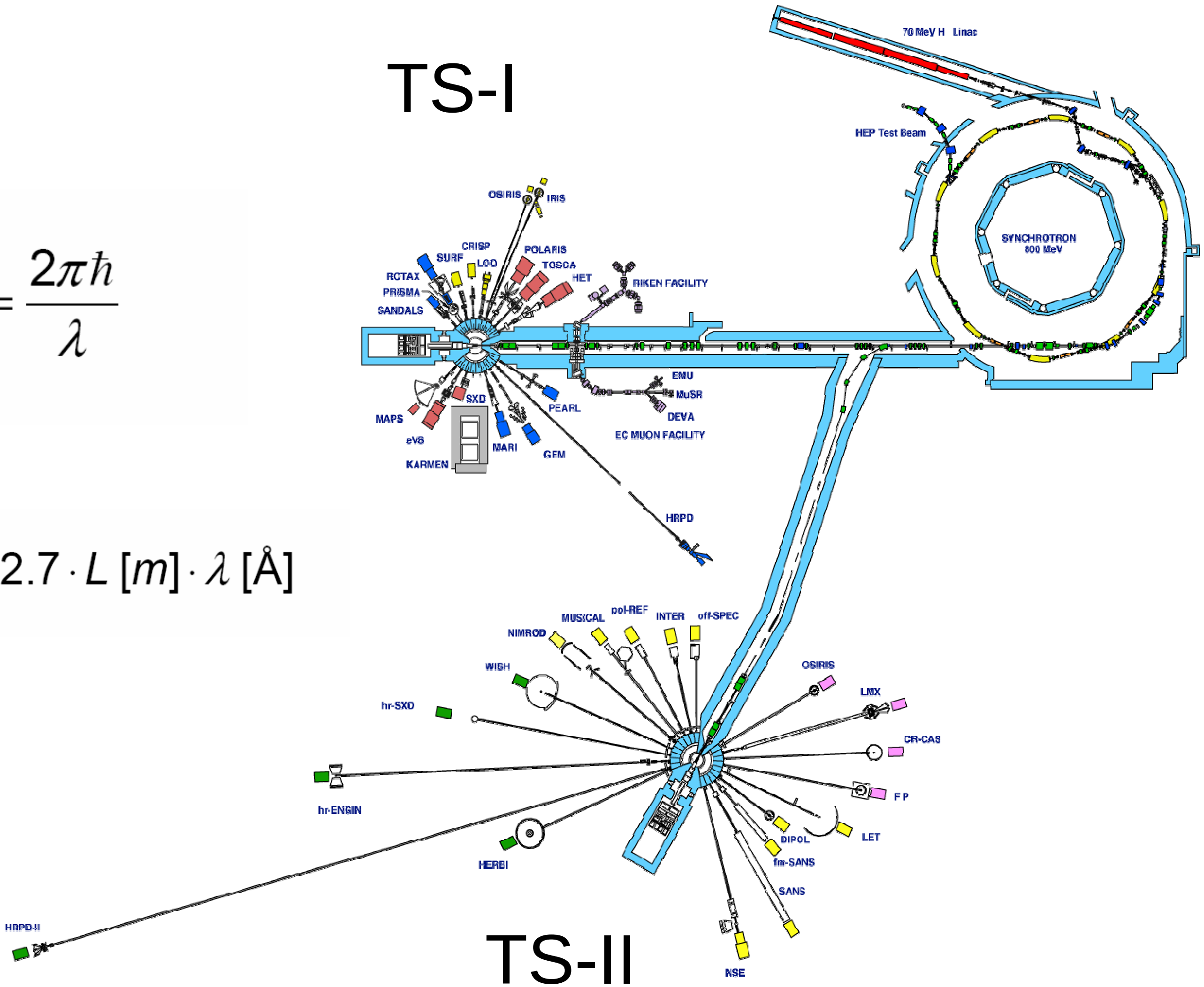
ISIS Facility, Rutherford Appleton Laboratory

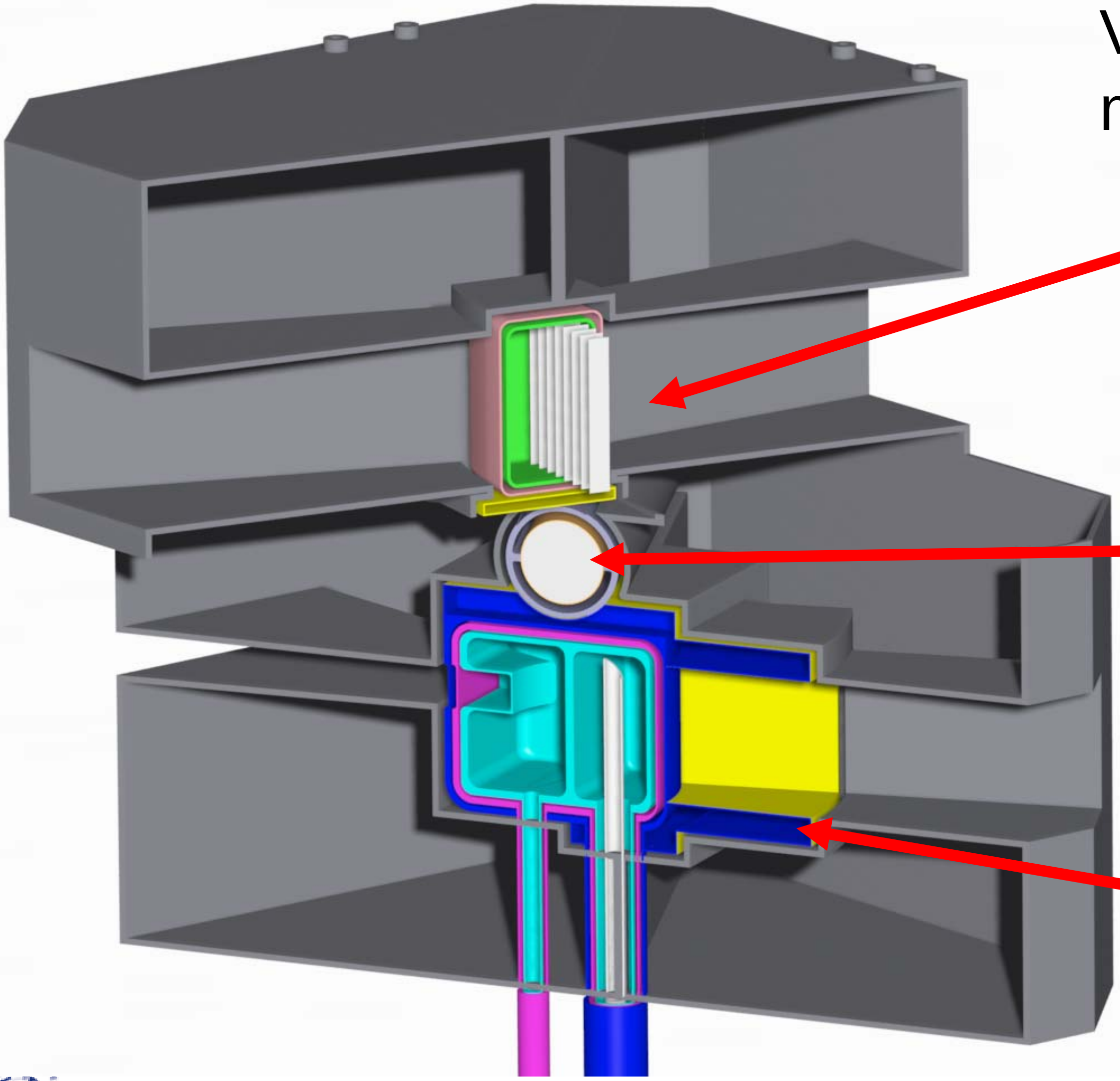
Les Rencontres de Saint-Aubin, Mar 2 2006

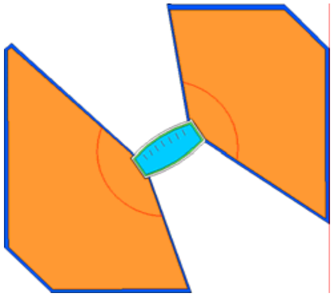
$$p_i = \hbar k_i = \frac{2\pi\hbar}{\lambda}$$

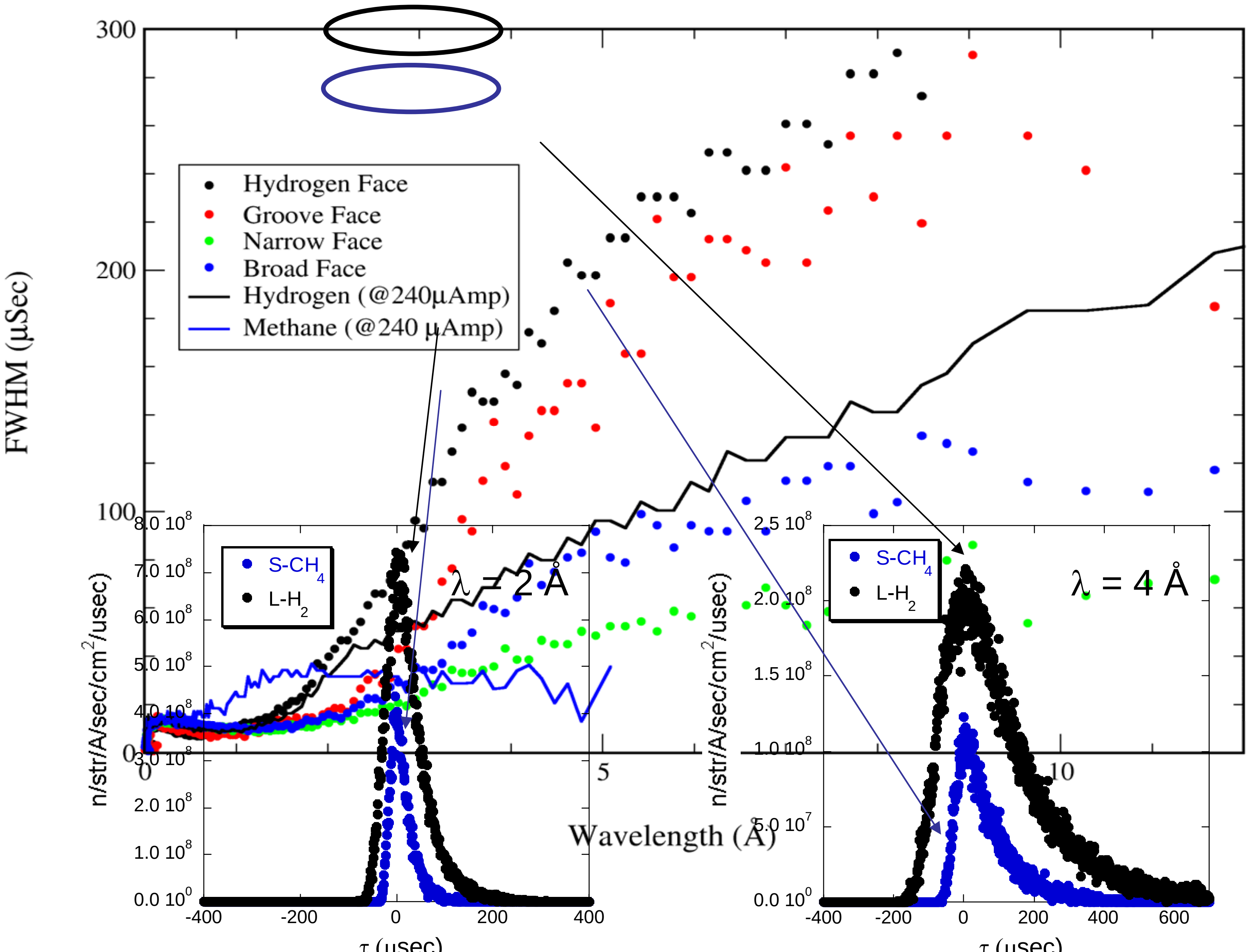
$$t\,[\mu\text{sec}] = 252.7 \cdot L\,[m] \cdot \lambda\,[\text{Å}]$$



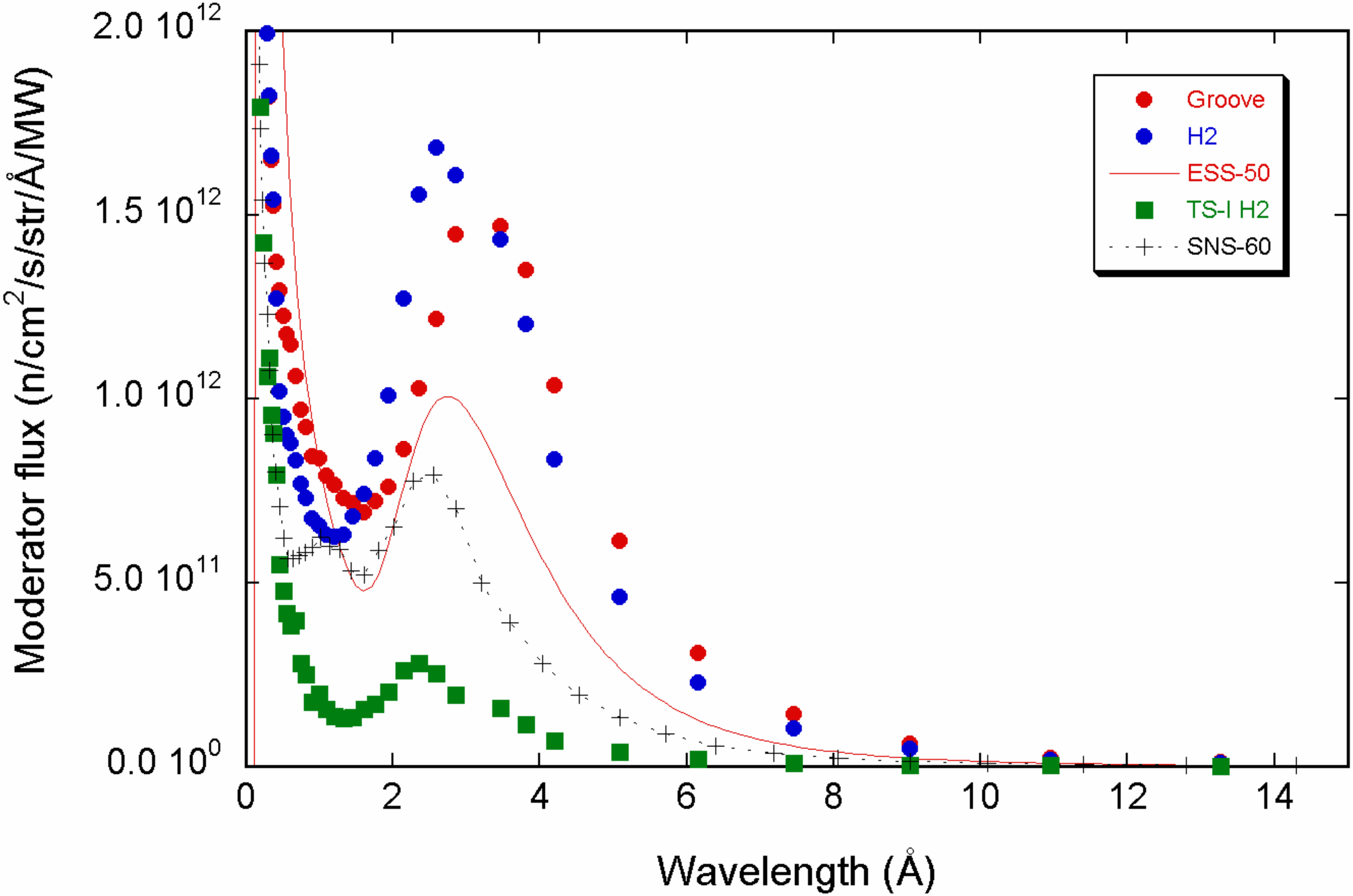
Views of the Moderator Engineering





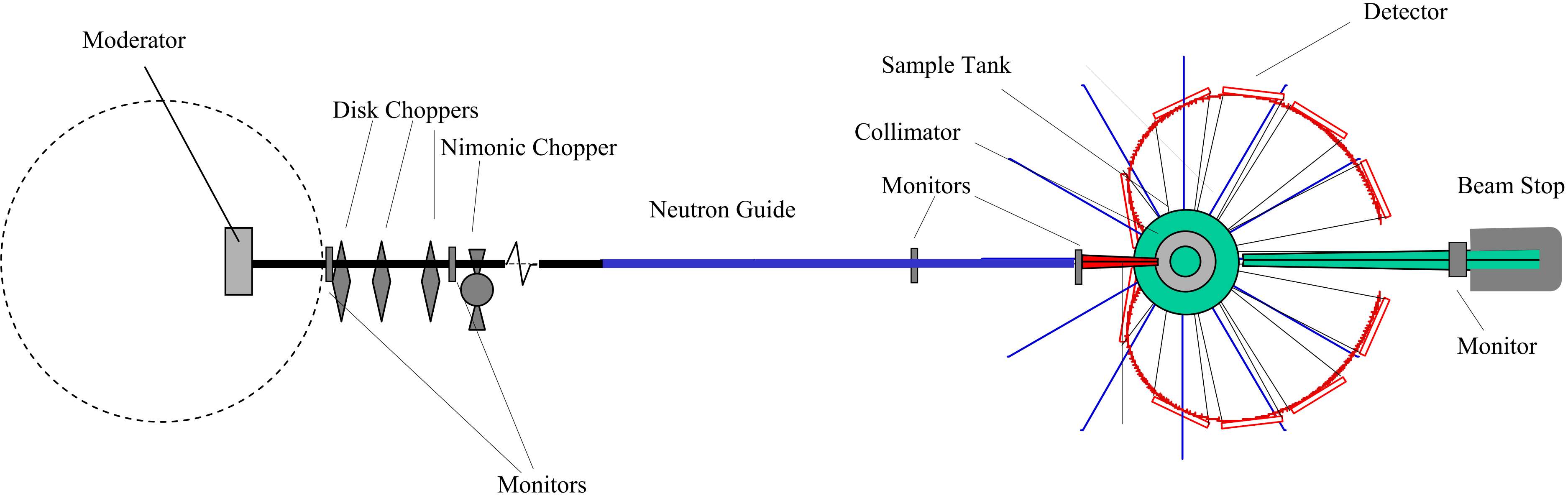


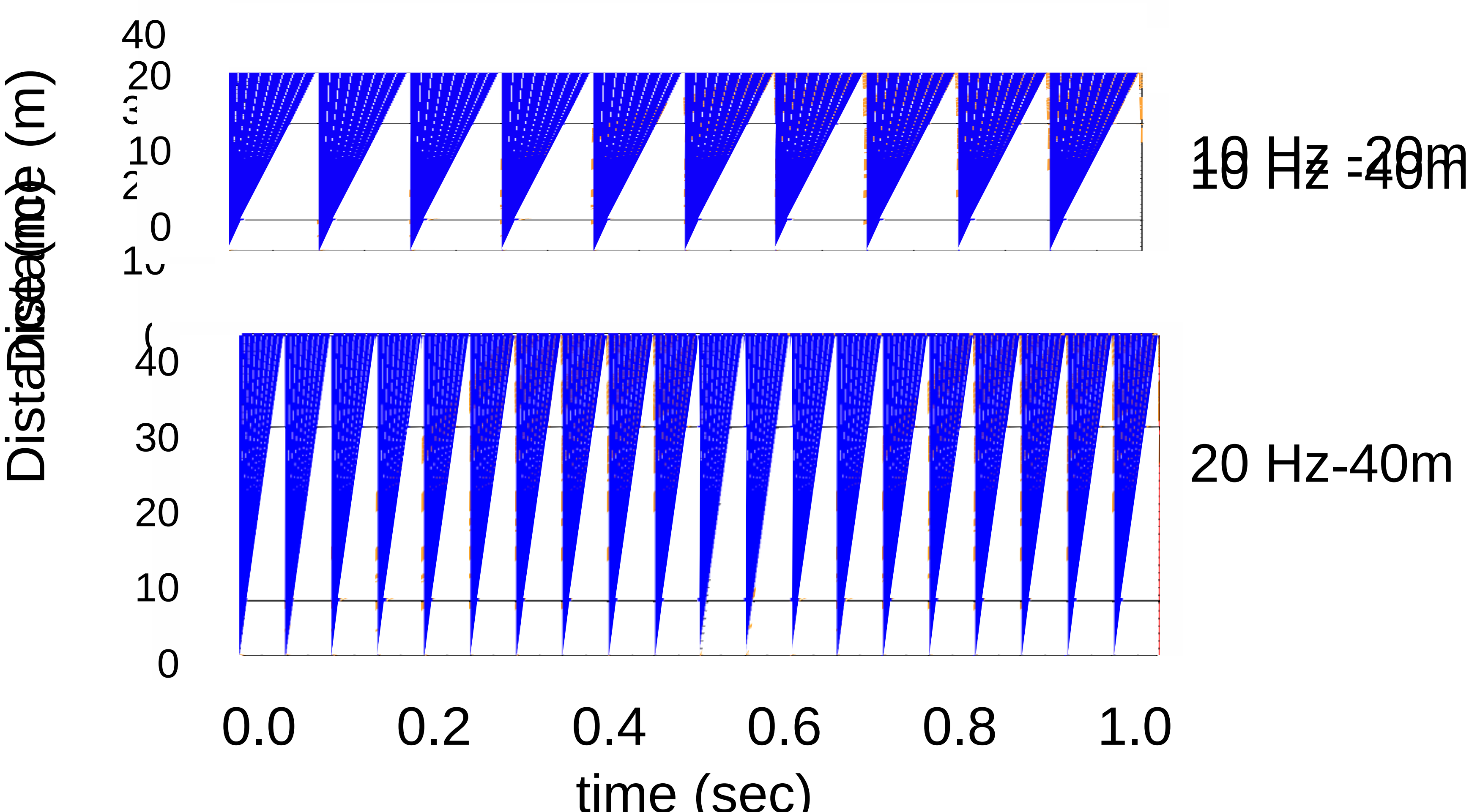
FWHM (μSec)
300
200
100
0
0
5
10
Wavelength (Å)
Hydrogen Face
Groove Face
Narrow Face
Broad Face
Hydrogen (@240μAmp)
Methane (@240 μAmp)
λ = 2 Å
λ = 4 Å
S-CH4
L-H2
n/str/A/sec/cm2/usec)
τ (μsec)
8.0 10^8
7.0 10^8
6.0 10^8
5.0 10^8
4.0 10^8
3.0 10^8
2.0 10^8
1.0 10^8
0.0 10^0
-400
-200
0
200
400
2.5 10^8
2.0 10^8
1.5 10^8
1.0 10^8
5.0 10^7
600



Groove
H2
ESS-50
TS-I H2
SNS-60
Moderator flux (n/cm2/s/str/Å/MW)
Wavelength (Å)
2.0 10^12
1.5 10^12
1.0 10^12
5.0 10^11
0.0 10^0
0
2
4
6
8
10
12
14

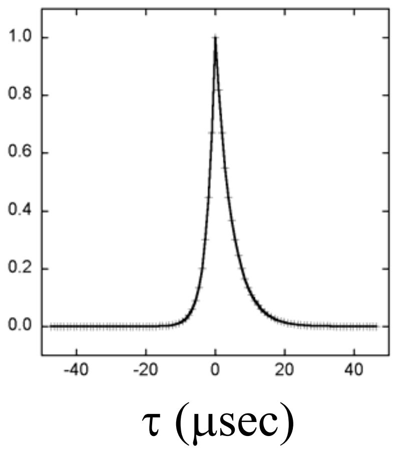
A generic TOF powder diffractometer

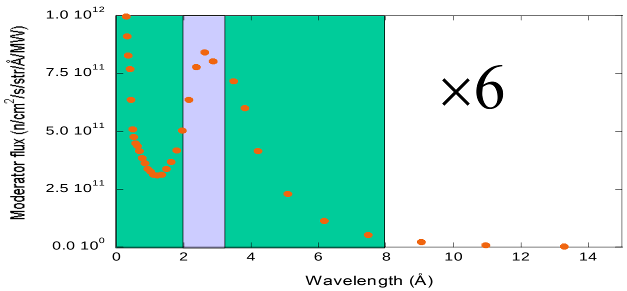


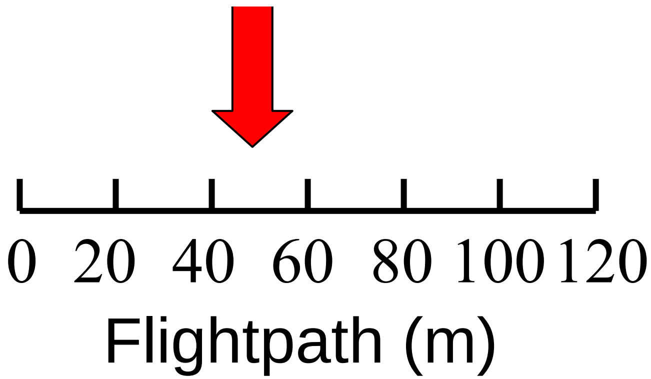


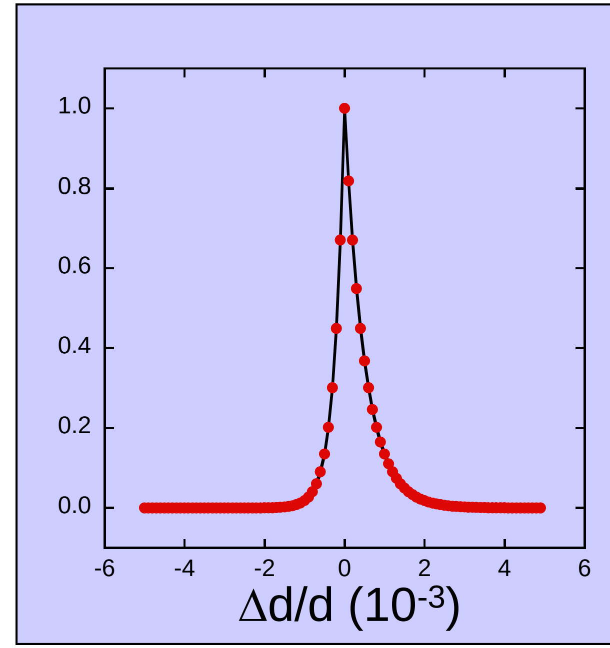
40
20
10
0
10 Hz -20m
10 Hz -40m
Distance (m)
40
30
20
10
0
20 Hz-40m
0.0
0.2
0.4
0.6
0.8
1.0
time (sec)

Decoupled Moderator

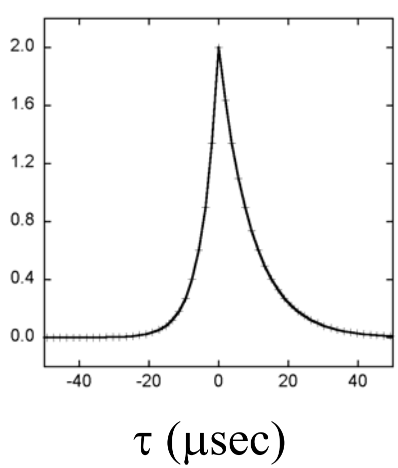


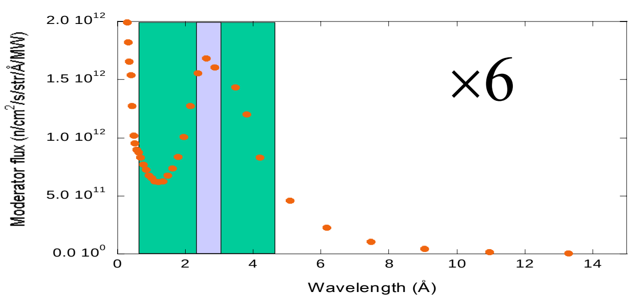


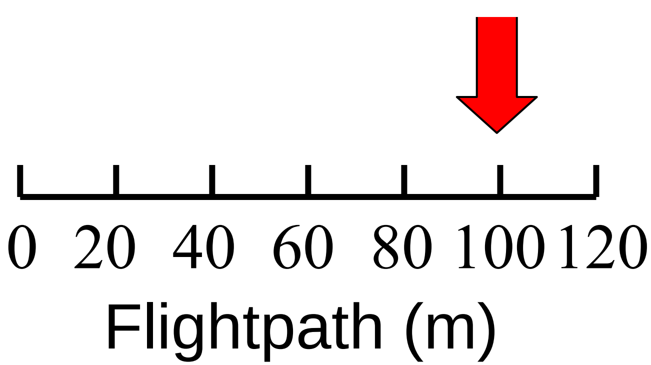


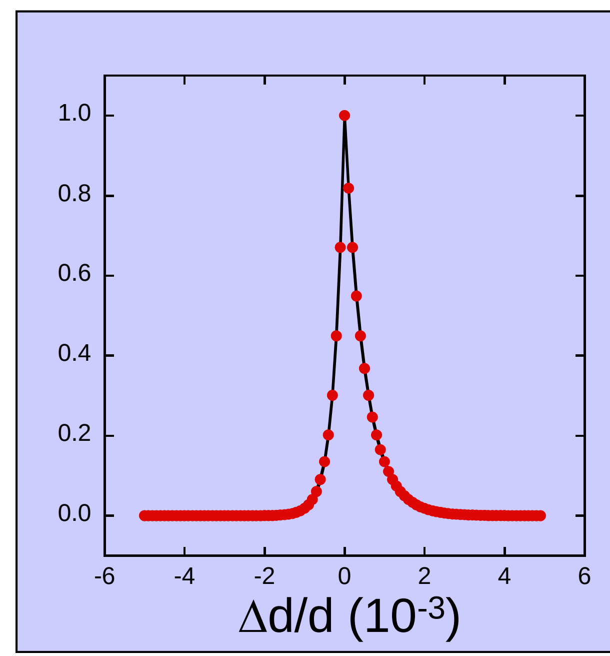


Coupled Moderator









Diffractometers: Flux on Sample

Optical efficiency

Source solid angle

Dispersive Resolution

$$\Phi_{sam} = \left\langle \varepsilon \cdot P \cdot \rho_{dis} \cdot \Omega_s \right\rangle_{\Delta\lambda}$$

$$P = \frac{\lambda \cdot \Sigma(\lambda)}{\nu \cdot \tau} \left[n \cdot \sec^{-1} \cdot sterad^{-1} \cdot cm^{-2} \right]$$

Incident spectrum

Peak Brilliance

Repetition rate

Pulse width

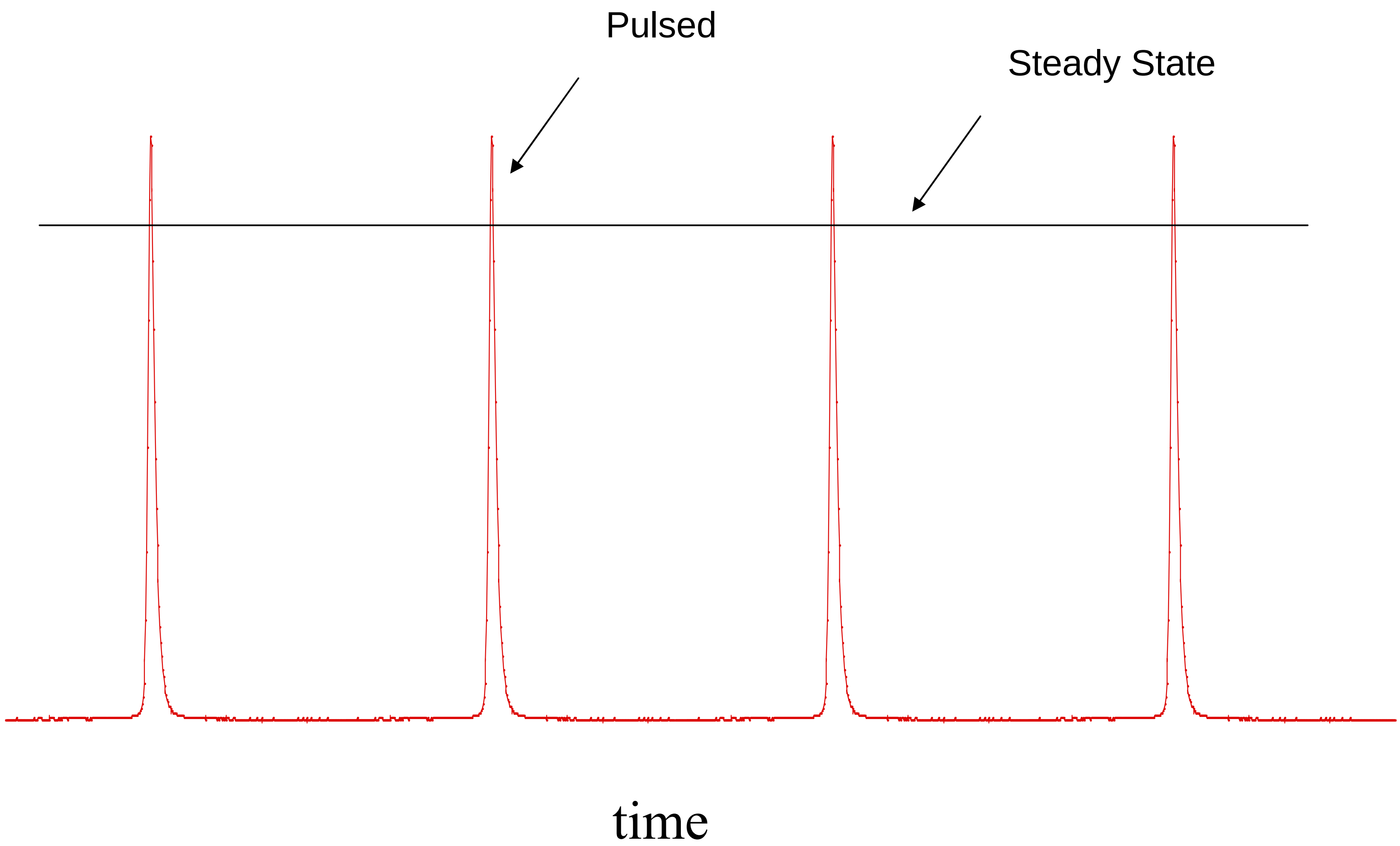
Diffractometers: Dispersive resolution

$$\rho_{dis} = \frac{\Delta t}{t}$$ TOF

$$\rho_{dis} = \frac{\Delta v}{v}$$ Velocity Selector

$$\rho_{dis} = \left(\frac{\Delta \lambda}{\lambda}\right)^{*}$$ Monochromator

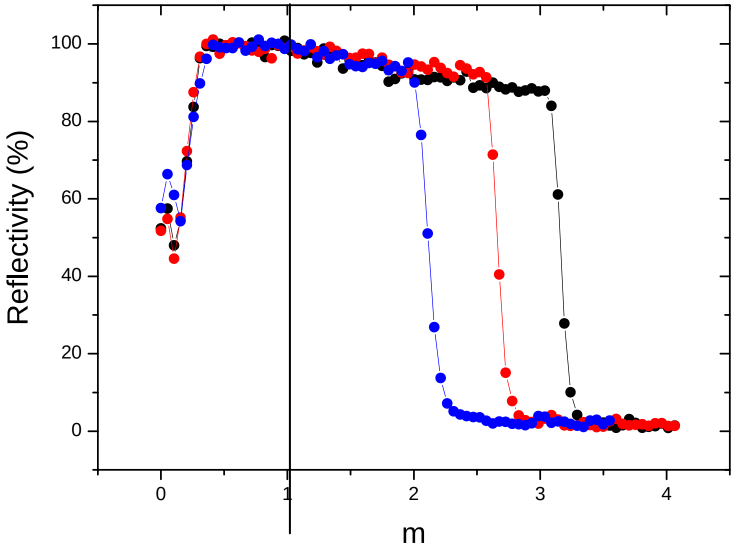
Peak Brilliance



Supermirrors

Direct view of the moderator :
- Resolution depends on L
- $\Phi \alpha$ solid angle α $1/L^2$
  so the distance is a limiting factor

Using optics :
- Can transport beam up to very large distance .
- However reflectivity drops rapidly in the "supermirror" region



~ 0.1° at 1 Å

(Measurements from Mirrotron).

- m=1 ,reflectivity about 99%
- above m=1, loose around 10% per extra m.

θ_C=0.0998.m.λ ~ 0.1m.λ

Powder cross section

$$\left(\frac{\partial\sigma}{\partial\Omega}\right)_{coh} = n_c \frac{(2\pi)^3}{v_0}\sum_{\tau}\delta^{(3)}(\mathbf{q}-\tau)\left|F(\tau)\right|^2 \quad [\mathrm{cm}^2]$$

$$\Leftarrow \quad \delta^{(3)}(\mathbf{x}) = \frac{\delta(r)\delta(\theta)\delta(\phi)}{r^2\sin\theta}$$

$$\delta(f(x)) = \left(\frac{\partial f(x)}{\partial x}\right)^{-1}\delta(x)$$

$$\left(\frac{\partial\sigma}{\partial\Omega}\right)^{pow}_{coh} = \frac{N}{4\pi}\frac{(2\pi)^3}{v_0}\sum_{\tau}\frac{\delta(q-\tau)}{q^2}\left|F(\tau)\right|^2 \quad [\mathrm{cm}^2]$$

$$\Leftarrow \quad \delta(q) = \frac{1}{k\cos\theta}\delta(2\theta) = \frac{\lambda^2}{4\pi\sin\theta}\delta(\lambda)$$

Angle-dispersive

$$\int\left(\frac{\partial\sigma}{\partial\Omega}\right)^{pow}_{coh} d2\theta = \int\frac{N}{4\pi}\frac{(2\pi)^3}{v_0}m_\tau\frac{\delta(2\theta-2\theta_\tau)}{k q^2\cos\theta}\left|F(\tau)\right|^2 d2\theta$$

$$= \frac{1}{4\pi}\frac{N}{v_0}m_\tau\left\{\frac{\lambda^3}{2\sin\theta\sin 2\theta}\right\}\left|F(\tau)\right|^2$$

$$= \frac{1}{4\pi}\frac{N}{v_0}m_\tau\left\{2d^3\tan\theta\right\}\left|F(\tau)\right|^2 \qquad [\mathrm{cm}^2]$$

Wavelength-dispersive

$$\int\left(\frac{\partial\sigma}{\partial\Omega}\right)^{pow}_{coh} d\lambda = \int\frac{N}{4\pi}\frac{(2\pi)^3}{v_0}m_\tau\frac{\lambda^2\delta(\lambda-\lambda_\tau)}{4\pi q^2\sin\theta}\left|F(\tau)\right|^2 d\lambda$$

$$= \frac{1}{4\pi}\frac{N}{v_0}m_\tau\frac{\lambda^4}{8\sin^3\theta}\left|F(\tau)\right|^2 = \frac{1}{4\pi}\frac{N}{v_0}m_\tau\left\{2d^4\sin\theta\right\}\left|F(\tau)\right|^2 \quad [\mathrm{cm}^2\ \mathrm{A}^{-1}]$$

Powder cross section (TOF)

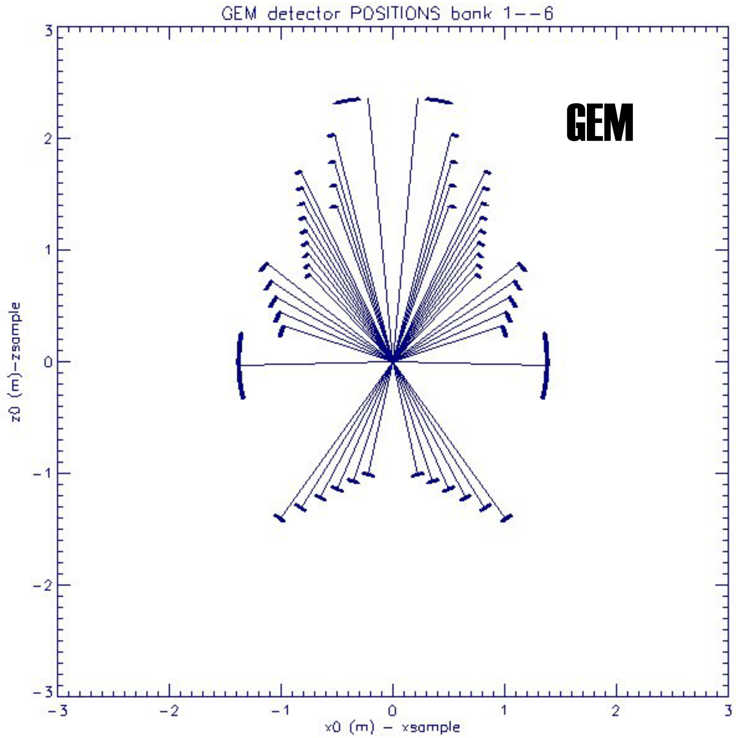
$$\int \left(\frac{\partial\sigma}{\partial\Omega}\right)_{coh}^{pow} d\lambda = \int \frac{N}{4\pi}\frac{(2\pi)^3}{v_0} m_\tau \frac{\lambda^2\,\delta(\lambda-\lambda_\tau)}{4\pi\, q^2 \sin\theta}\left|F(\tau)\right|^2 d\lambda$$

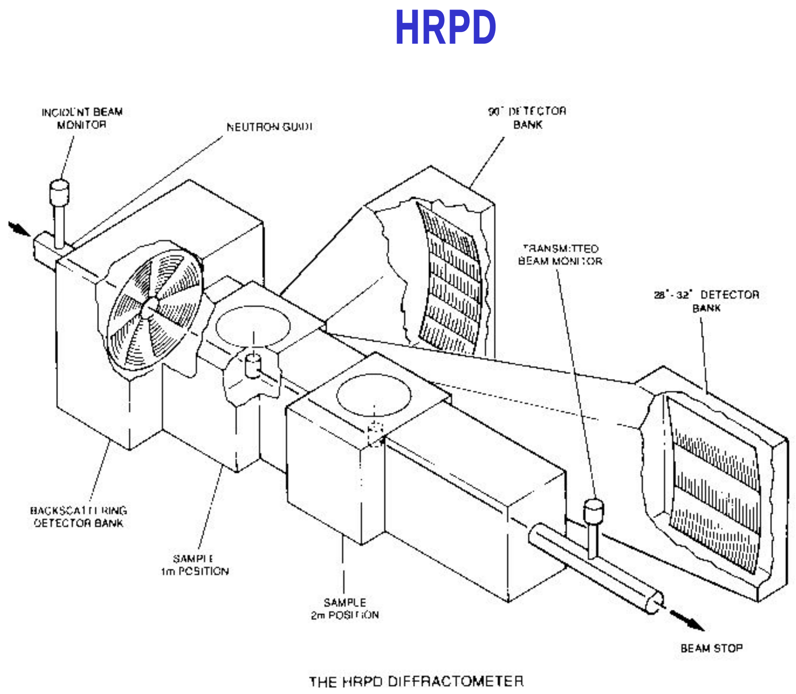
$$= \frac{1}{4\pi}\frac{N}{v_0} m_\tau \frac{\lambda^4}{8\sin^3\theta}\left|F(\tau)\right|^2 = \frac{1}{4\pi}\frac{N}{v_0} m_\tau \left\{2d^4\sin\theta\right\}\left|F(\tau)\right|^2 \quad [\text{cm}^2\ \text{A}^{-1}]$$

$$\int \left(\frac{\partial\sigma}{\partial\Omega}\right)_{coh}^{pow} d\lambda = \int \frac{N}{4\pi}\frac{(2\pi)^3}{v_0} m_\tau \frac{\lambda^2\,\delta(\lambda-\lambda_\tau)}{4\pi\, q^2 \sin\theta}\left|F(\tau)\right|^2 d\lambda$$

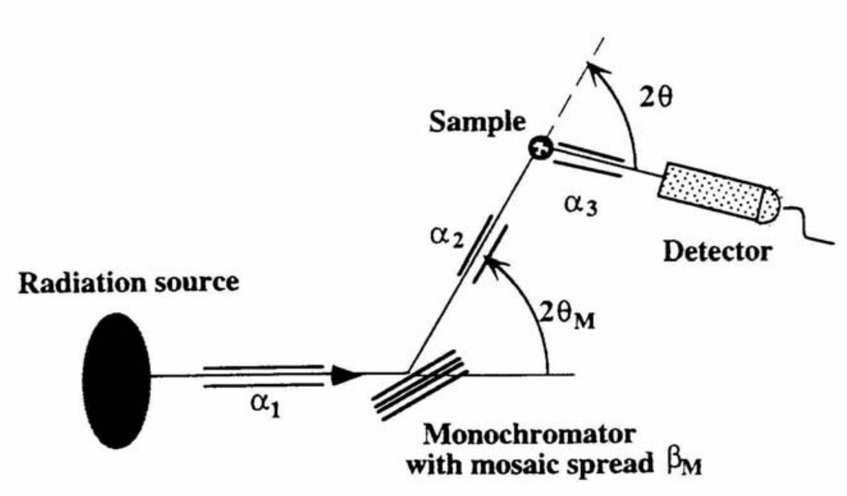
$$= \frac{1}{4\pi}\frac{N}{v_0} m_\tau \frac{\lambda^4}{8\sin^3\theta}\left|F(\tau)\right|^2 = \frac{1}{4\pi}\frac{N}{v_0} m_\tau \left\{2d^4\sin\theta\right\}\left|F(\tau)\right|^2 \quad [\text{cm}^2\ \text{A}^{-1}]$$

TOF Instrument designs





Resolution



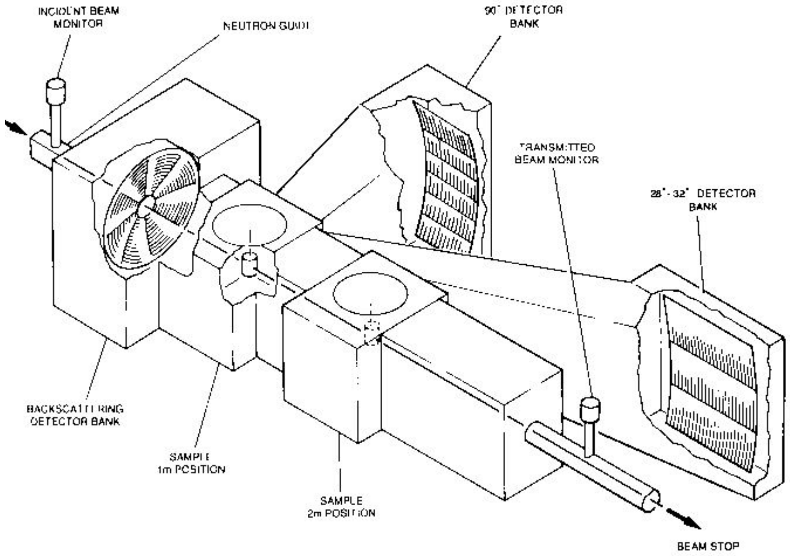
$$\frac{\Delta d}{d} = \frac{1}{2}\sqrt{W\cot^2\theta + V\cot\theta + U}$$

$$U = \frac{\alpha_1^{\;2} + \alpha_2^{\;2}}{\tan^2\theta_M}$$

$$V = -\frac{2\alpha_2^{\;2}}{\tan\theta_M}$$

$$W = \alpha_2^{\;2} + \alpha_3^{\;2}$$

HRPD

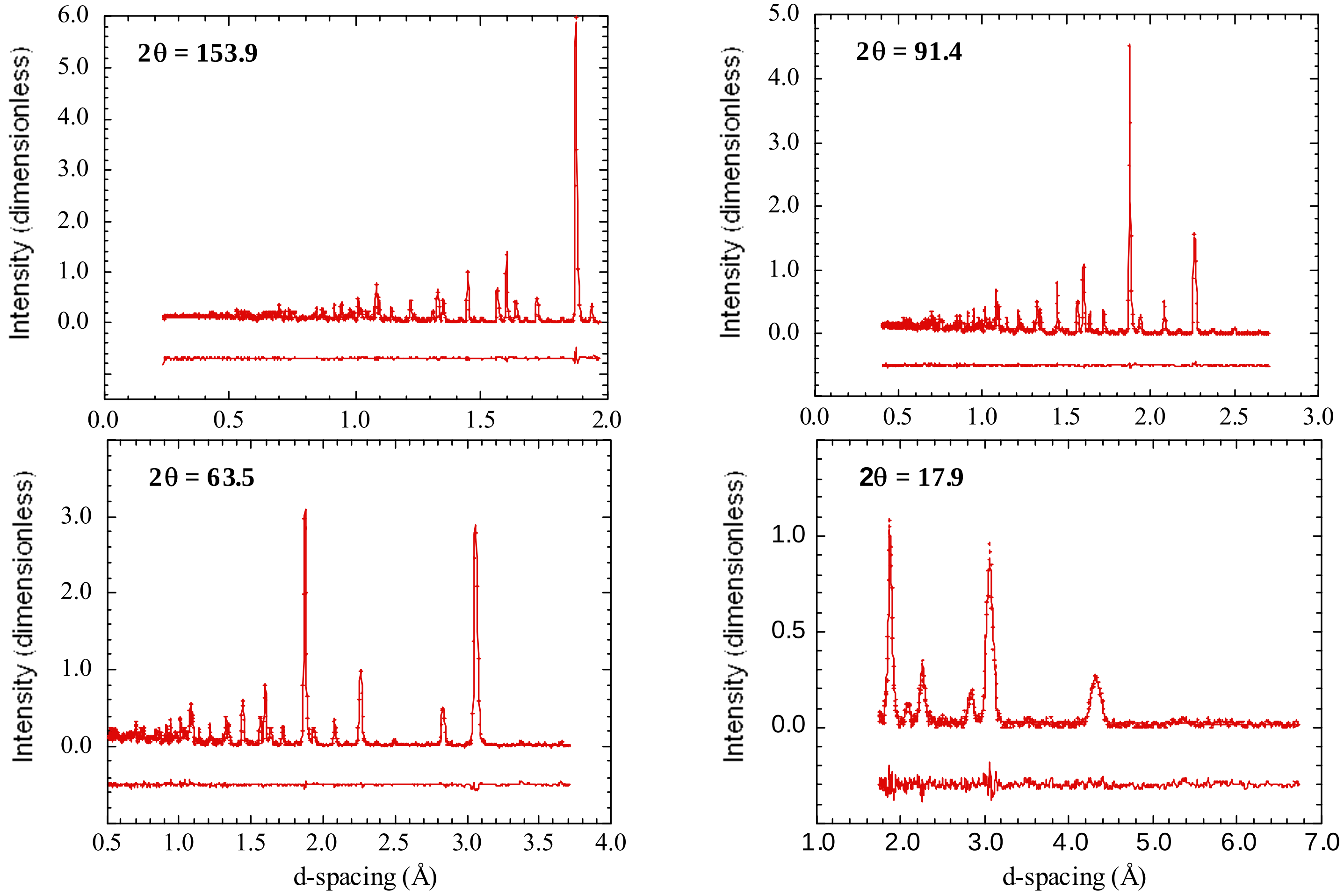


THE HRPD DIFFRACTOMETER

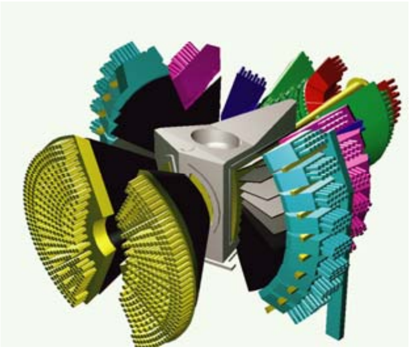
$$\left(\frac{\Delta d}{d}\right)_{TOF} = \sqrt{\left(\frac{\Delta t}{t}\right)^2 + \left(\frac{\Delta L}{L}\right)^2 + (\cot\theta \cdot \Delta\theta)^2}$$

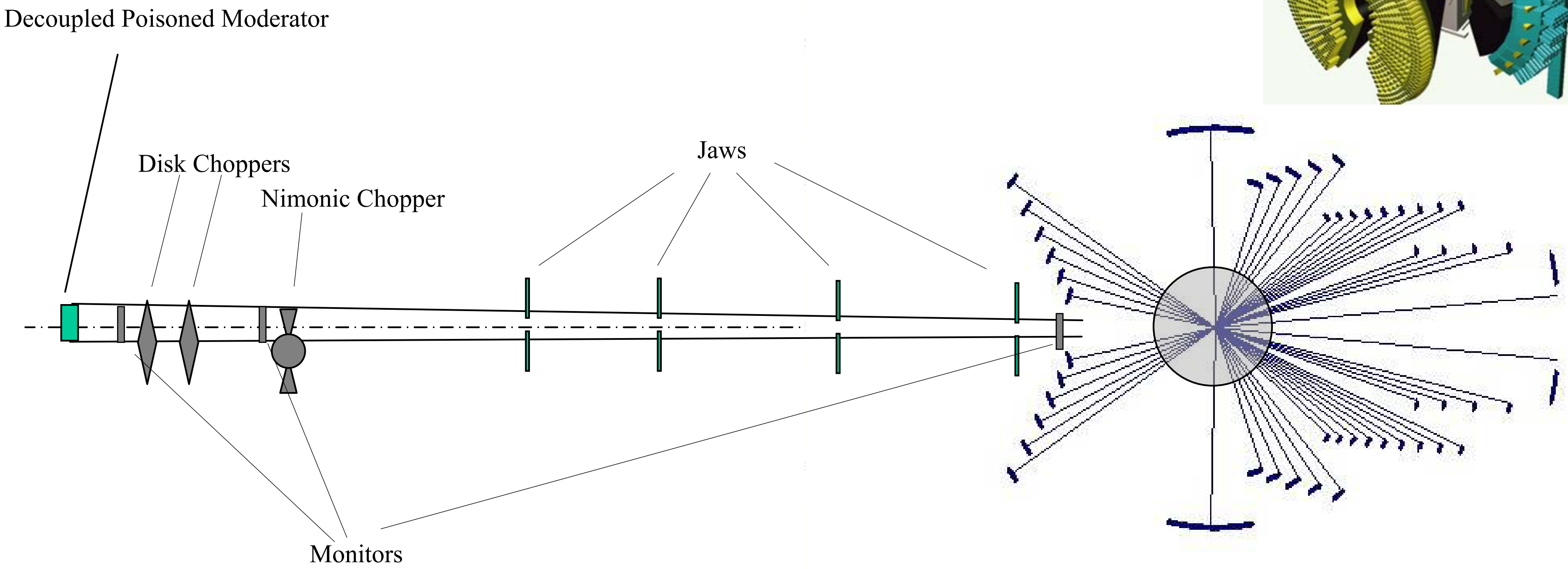
TOF data structure

Y_2O_3 powder 3 g 1 min run



The “classic” design

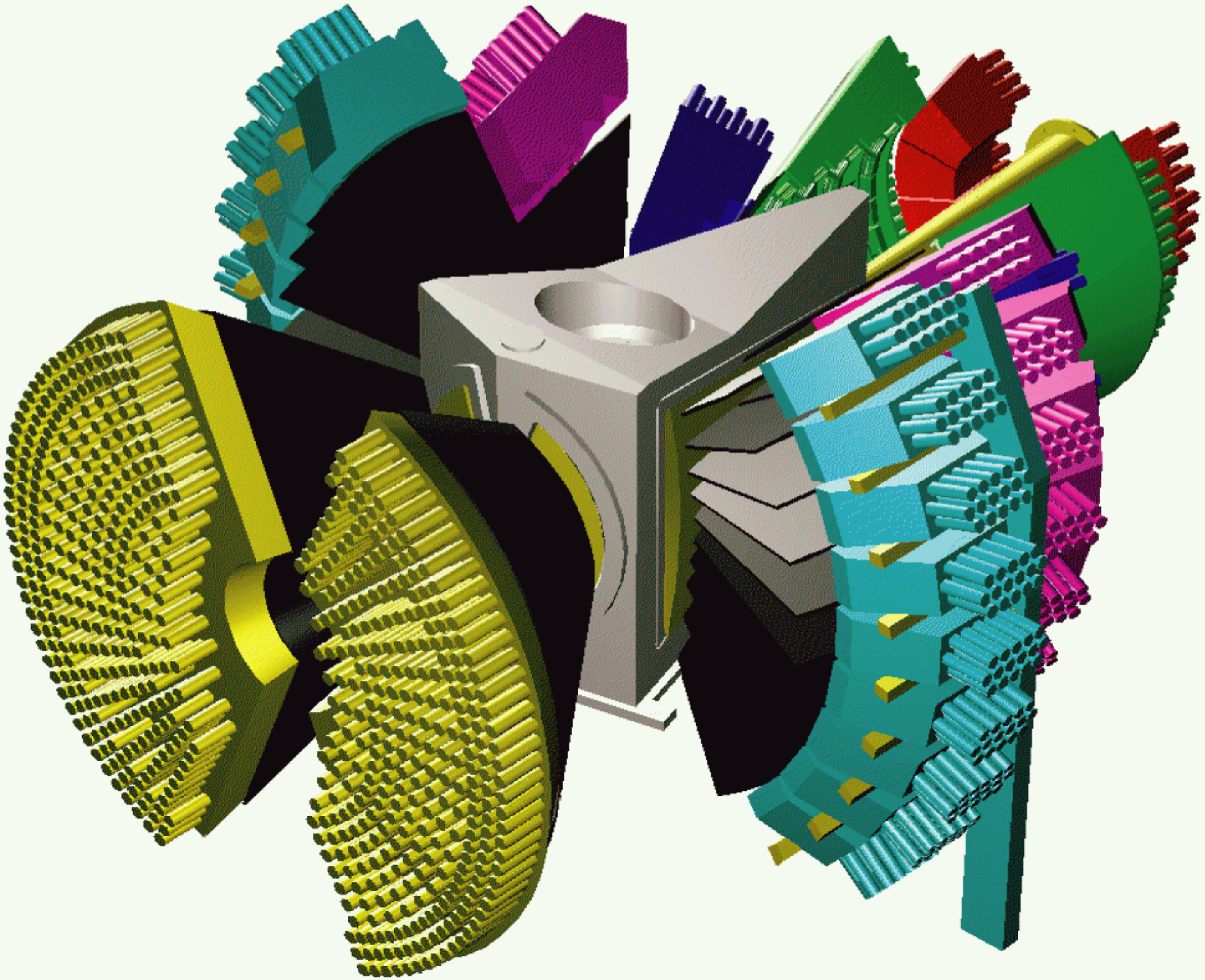




+ Direct view of the moderator (no losses)
+ High flux at short wavelengths.
+ Good bandwidth ($\Delta\lambda=3957/\nu/l_{tot}$).

- Problem to cool moderator below 100 K - long wavelengths.
- No focussing = relatively low flux. Can recover with solid angle but…
- Fragmented data structure.

MEDUSA Full Feature Shader

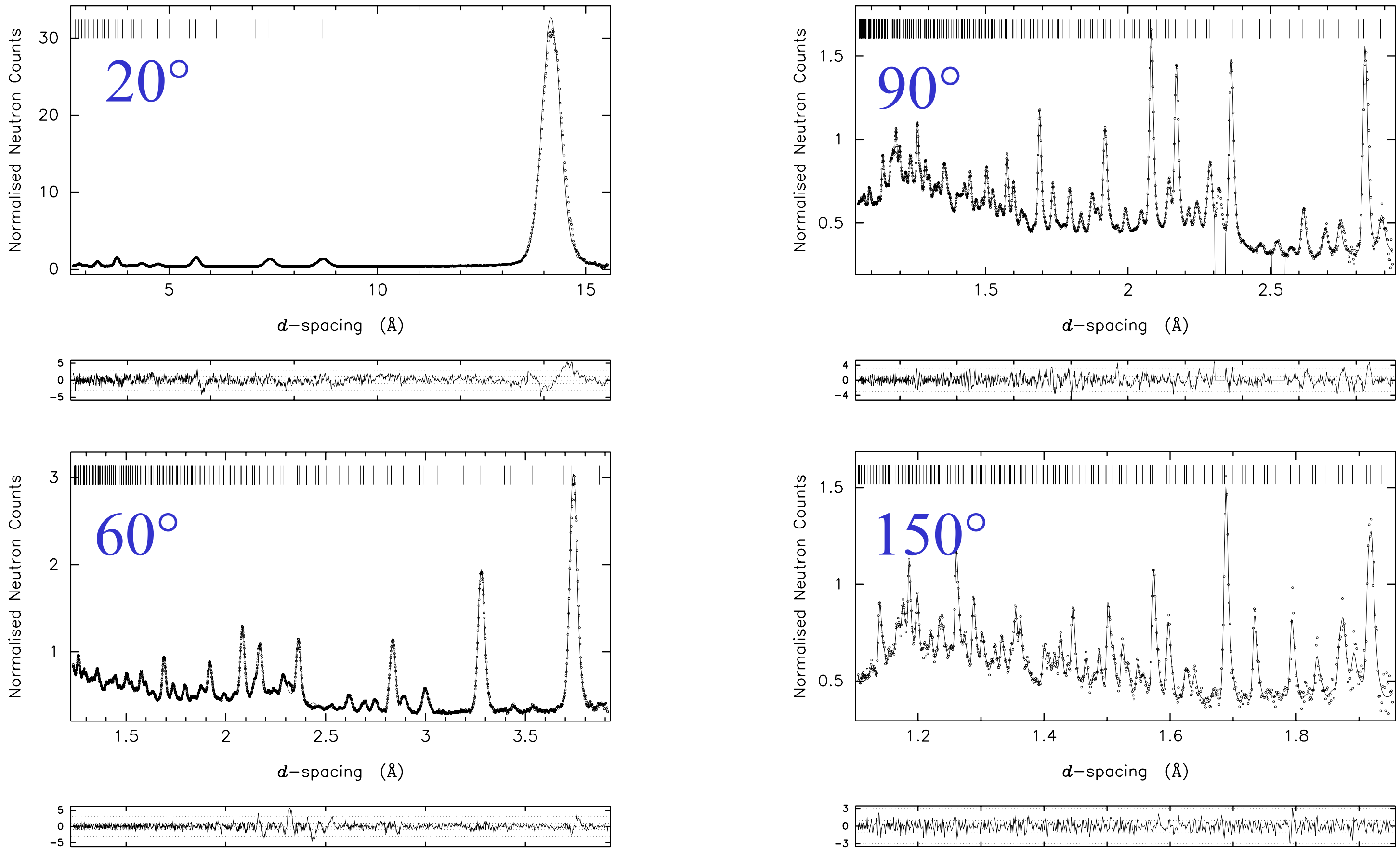



22/2/200


3/7/2000

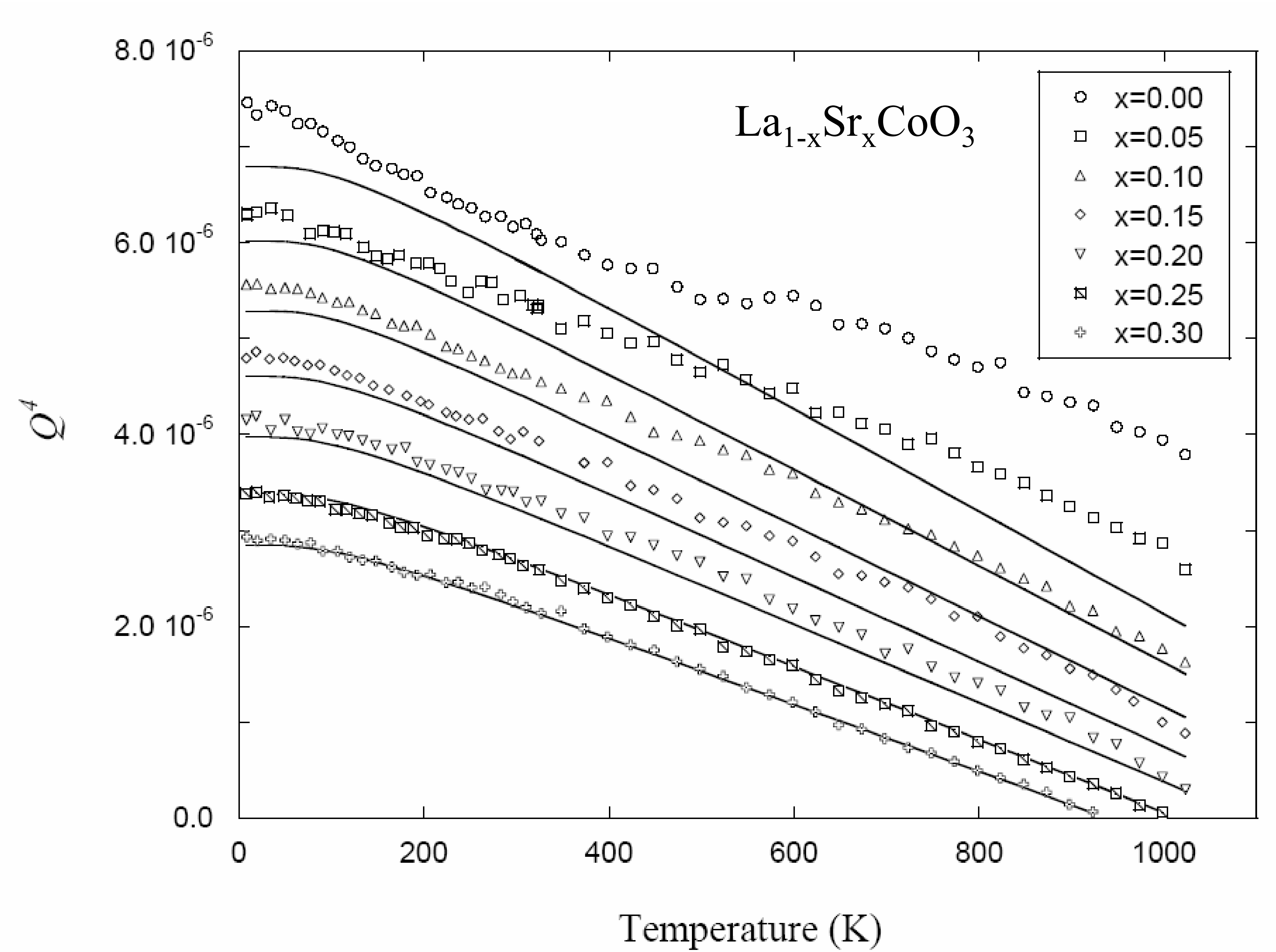
80 cm

Location of adsorbed species in NO-reduction catalysts



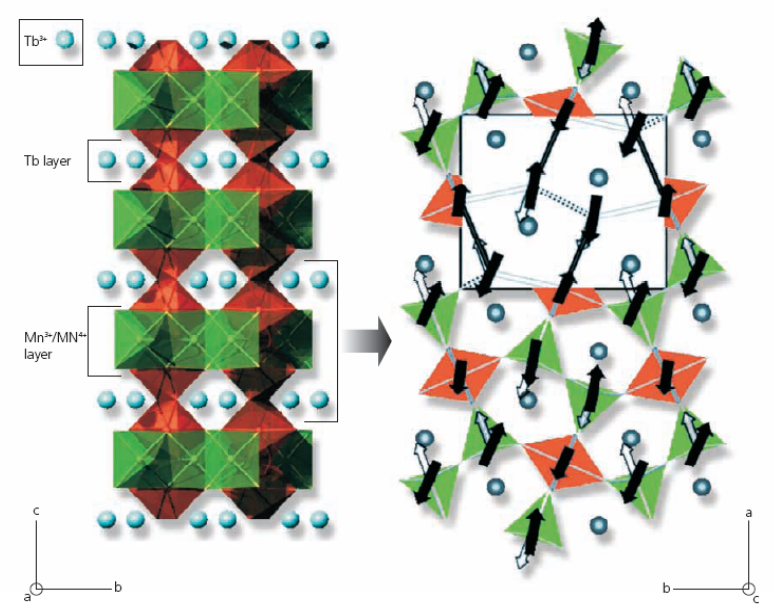
Refined data from pristine Cu-exchanged zeolite Y at 77 K collected in only 30 mins using all current detector banks on GEM. The structural evolution of the framework and of the adsorbed NO ligands was studied as a function of temperature and NO gas overpressure.

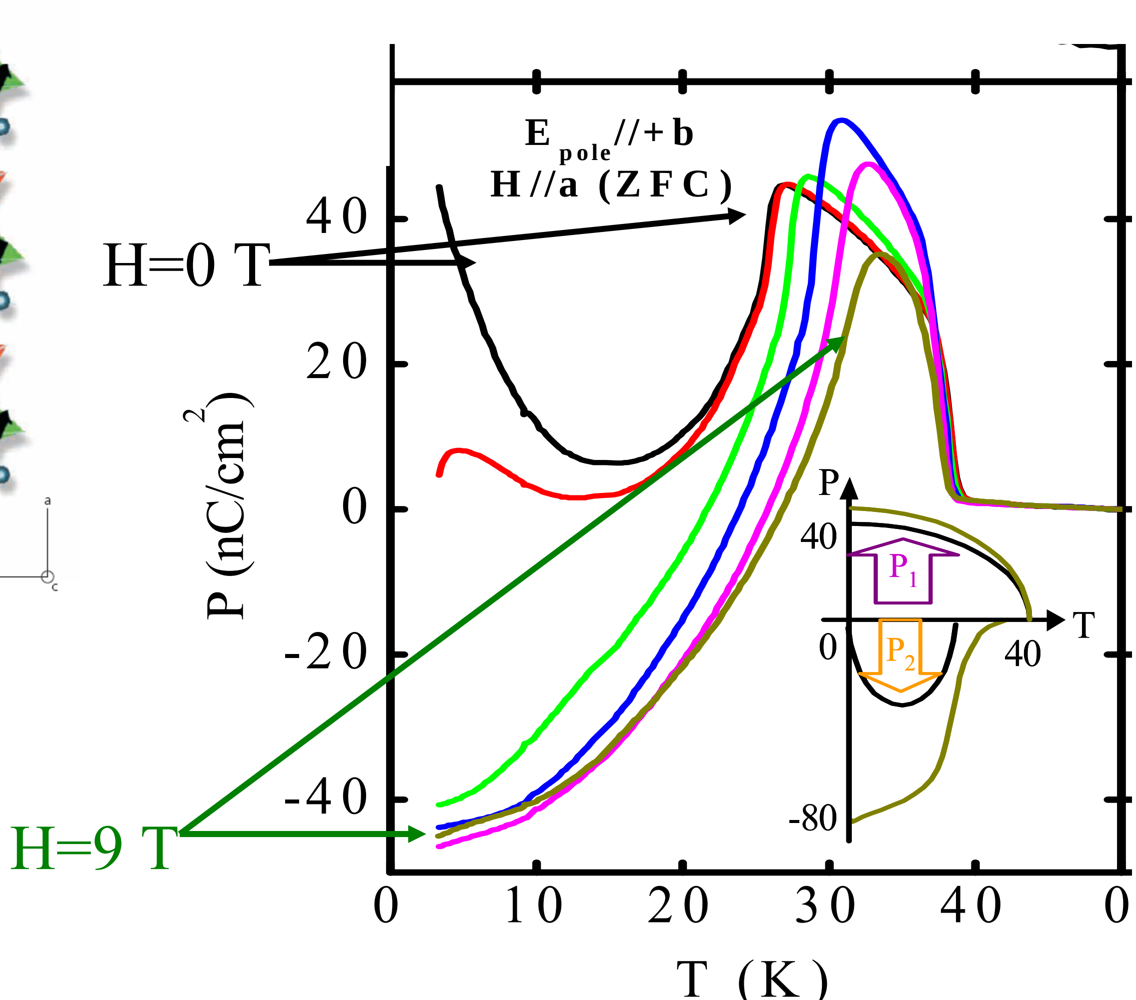
G C Hardy, M J Rosseinsky, Dept. of Chemistry, University of Liverpool & R M Ibberson, P G Radaelli, ISIS.



La1-xSrxCoO3
x=0.00
x=0.05
x=0.10
x=0.15
x=0.20
x=0.25
x=0.30
8.0 10-6
6.0 10-6
4.0 10-6
2.0 10-6
0.0
Q4
0
200
400
600
800
1000
Temperature (K)

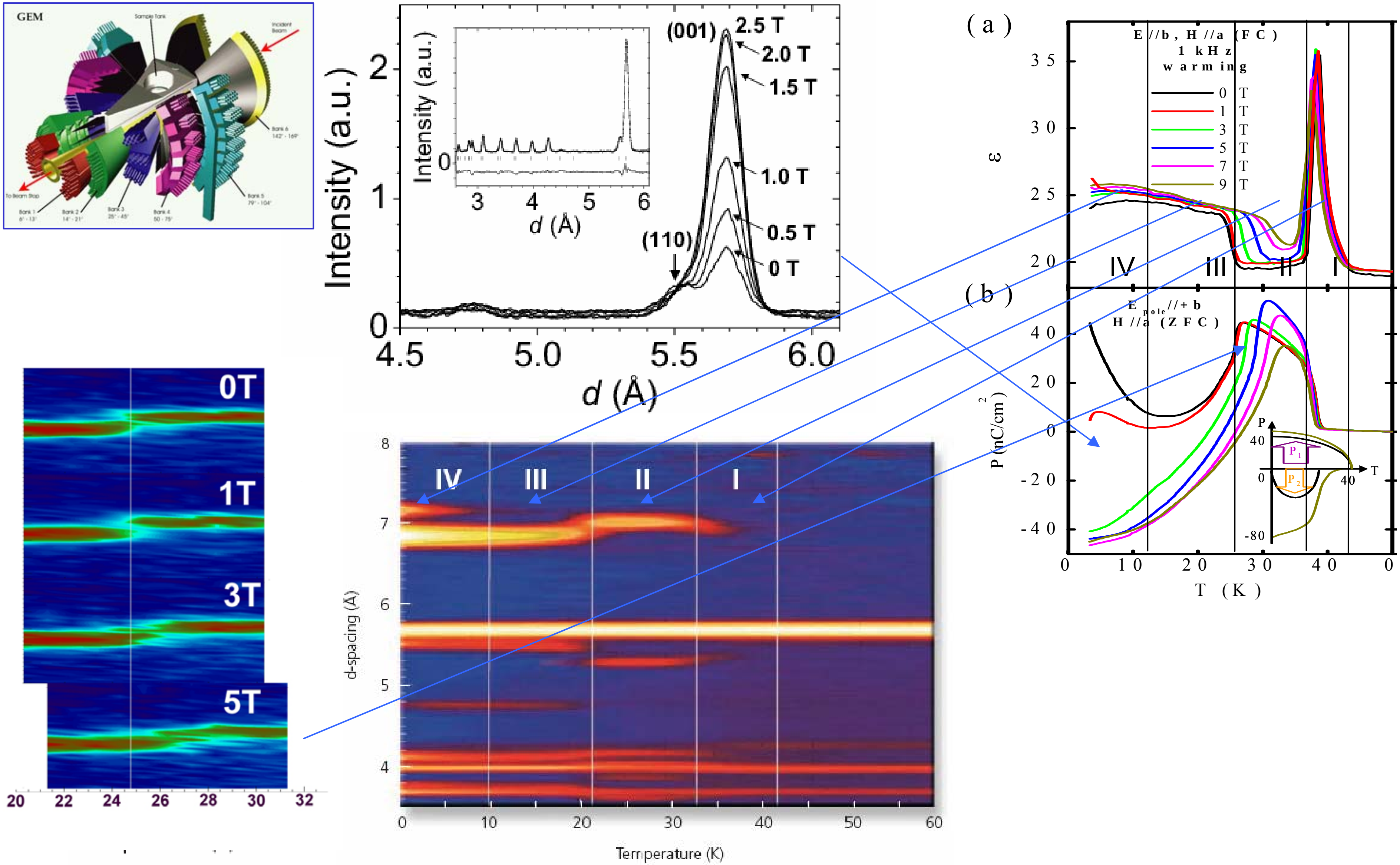
Multiferroics: $\mathit{RE}Mn_2O_5$





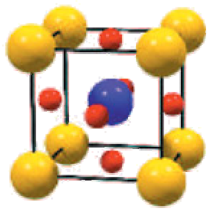
1. N. Hur *et al.* Nature, **429**, 392 (2004)
2. L.C. Chapon *et al.,* Phys. Rev. Lett. **93**, 177402 (2004)
3. G. Blake *et al.*, Phys. Rev. B **71**, 214402 (2005)

<u>*RE*Mn$_2$O$_5$: temperature and field dependence</u>

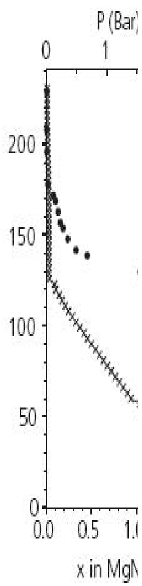


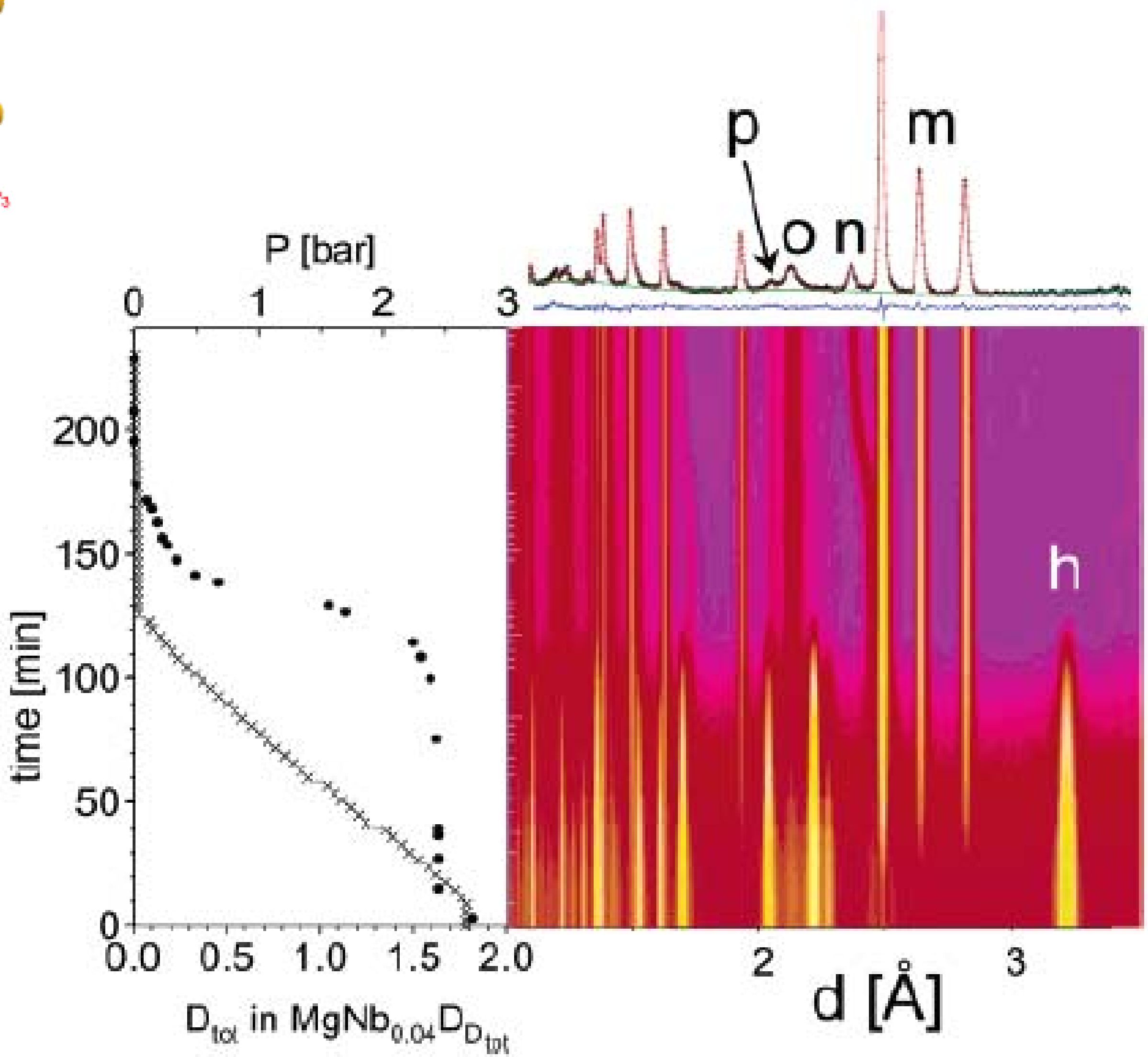
Hydrogen sorption of Nb-catalysed, nanostructured Mg

F M Mulder, H G Schimmel (TU-Delft, The Netherlands), J Huot (Université du Québec à Trois-Rivières, Canada) and L C Chapon (ISIS)



$Nb(Mg_{0.333}Nb_{0.667})O_3$

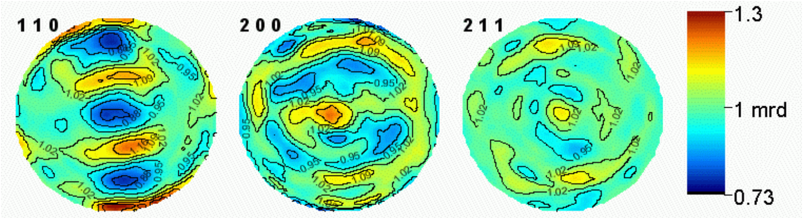


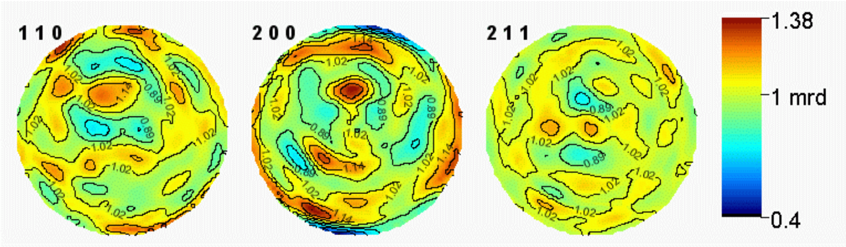


17th/19th century iron armour plates

Sylvia Leever, J. Dik
TU Delft, NL

D. Visser
ISIS&NWO,NL





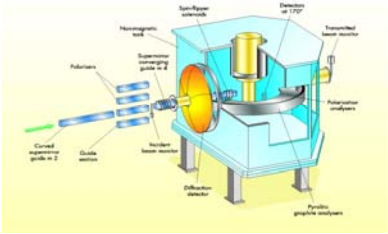
wt%: 99.9 Fe, 0.2 Fe_3C, 0.2 FeO

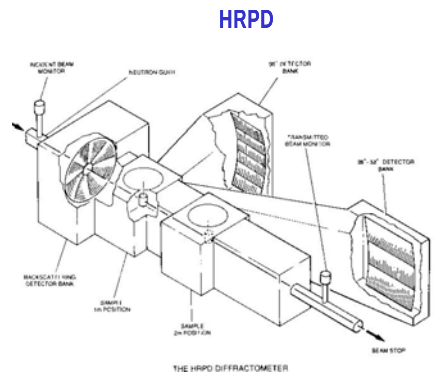
→ 0.002 wt% C

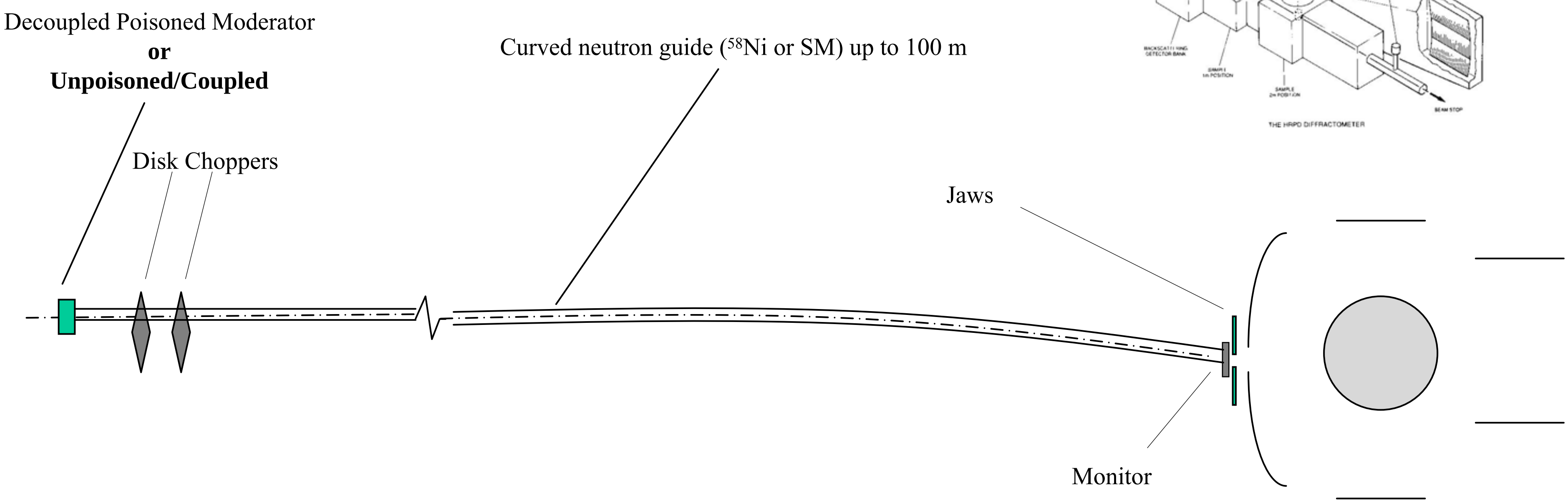
wt%: 97.3 Fe, 2.5 Fe_3C, 0.2 Fe_3O_4

→ 0.17 wt% C

The “long ‘uns”





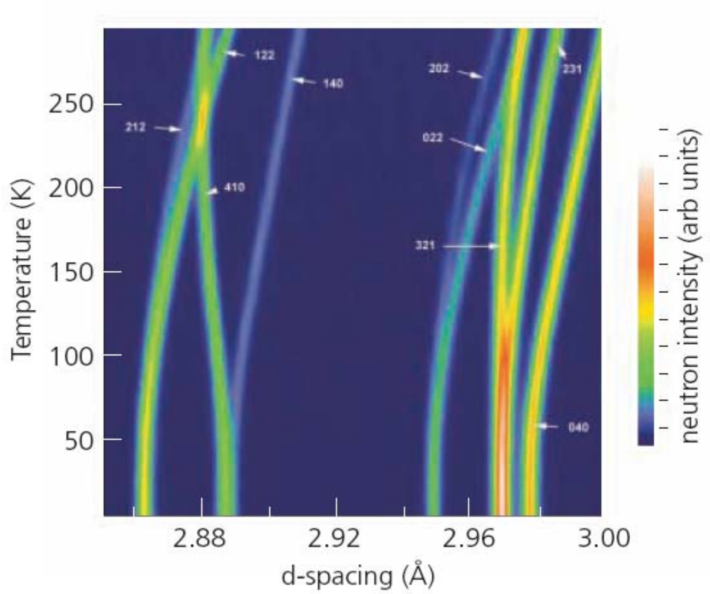


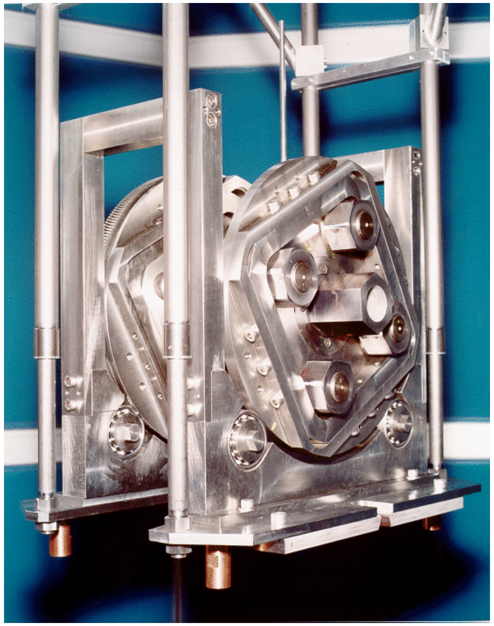
- \+ Sharp pulse structure – w. poisoned moderator can reach particle size limit in backscattering
- \+ Resolution truly independent on d-spacing in backscattering
- \+ Can accommodate focussing.
- \+ Coupled moderators can be colder (20 K)

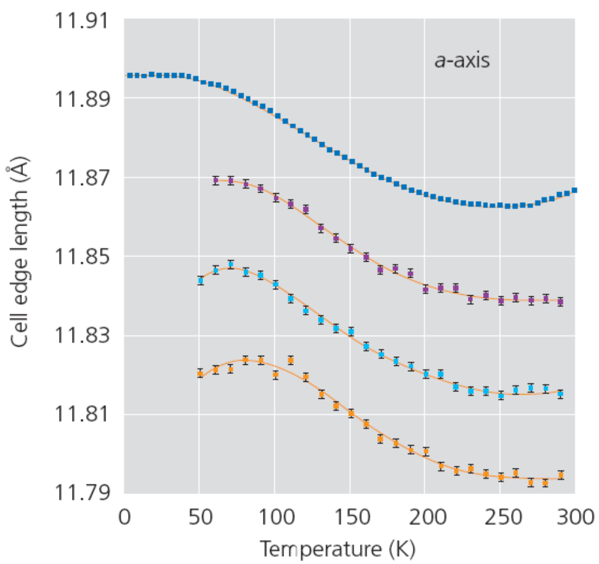
- \- Need to reduce the repetition rate to archive sufficient BW -> TS2.
- \- Need to transport neutrons efficiently – optics.

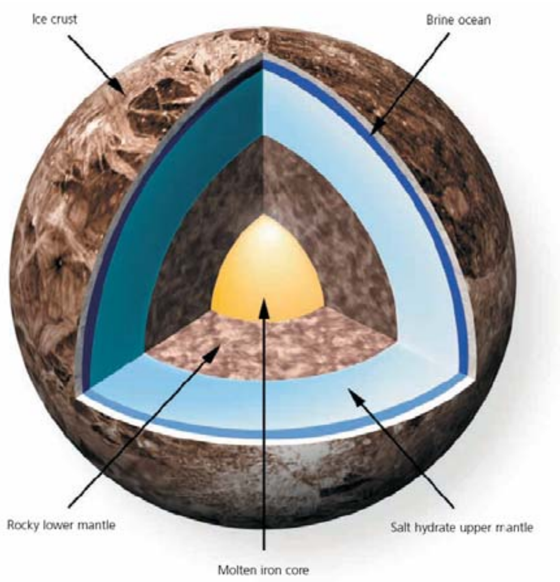
High Pressure studies: Epsom salt on the moons of Jupiter

A D Fortes, M Alfredsson, J P Brodholt, L Vocùadlo, I G Wood, (University College London) and K S Knight (ISIS)
ISIS Annual Report 2004.









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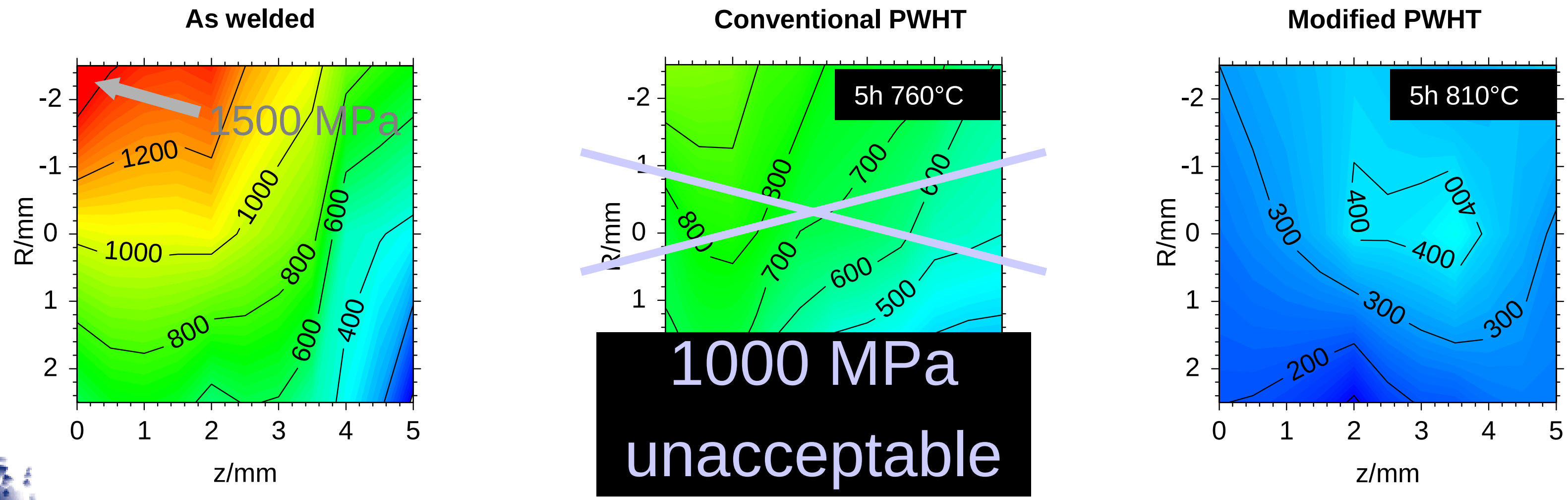
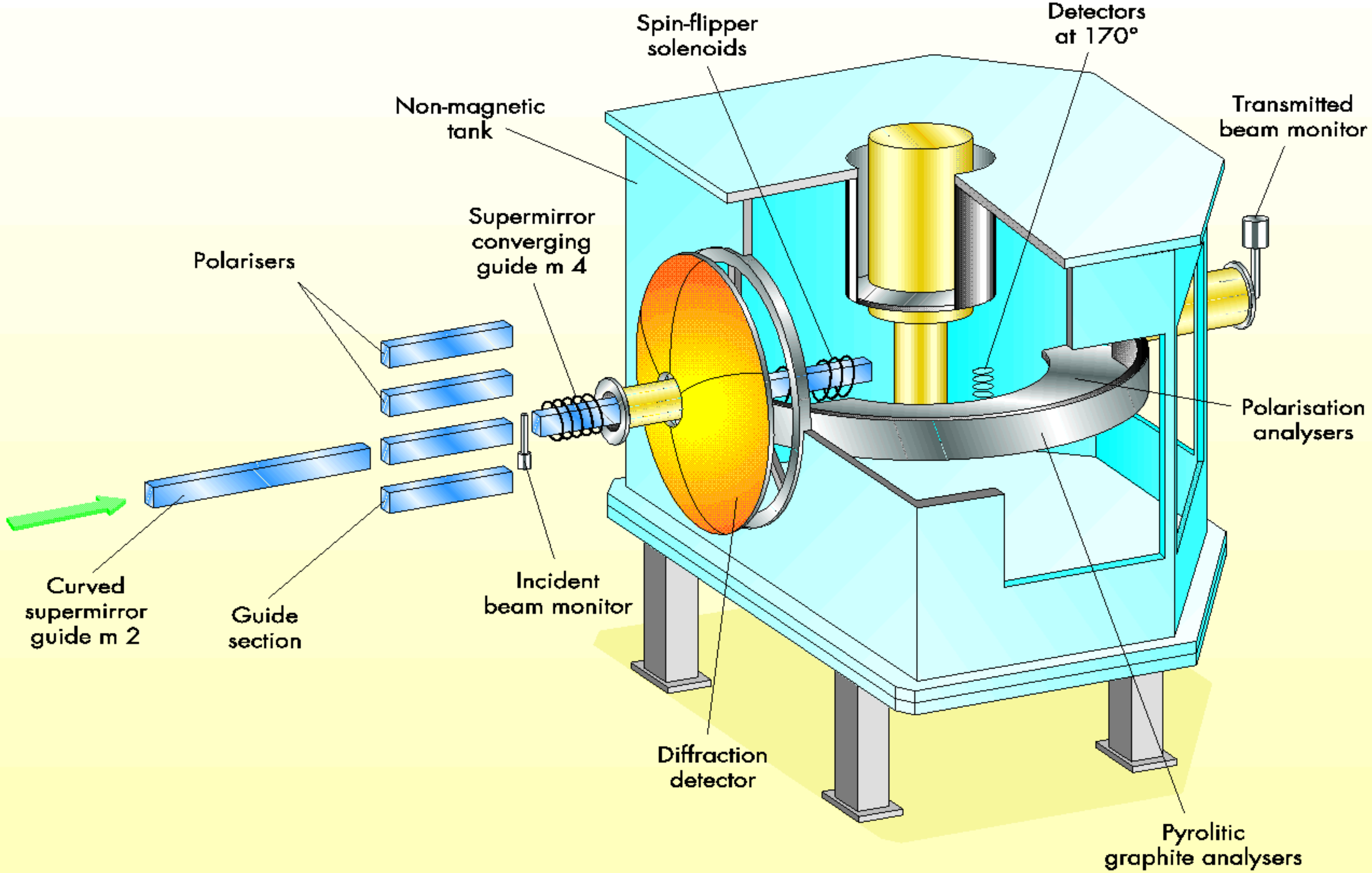
Inertia friction welding


Rolls-Royce plc. Compressor rotor factory (CRF)

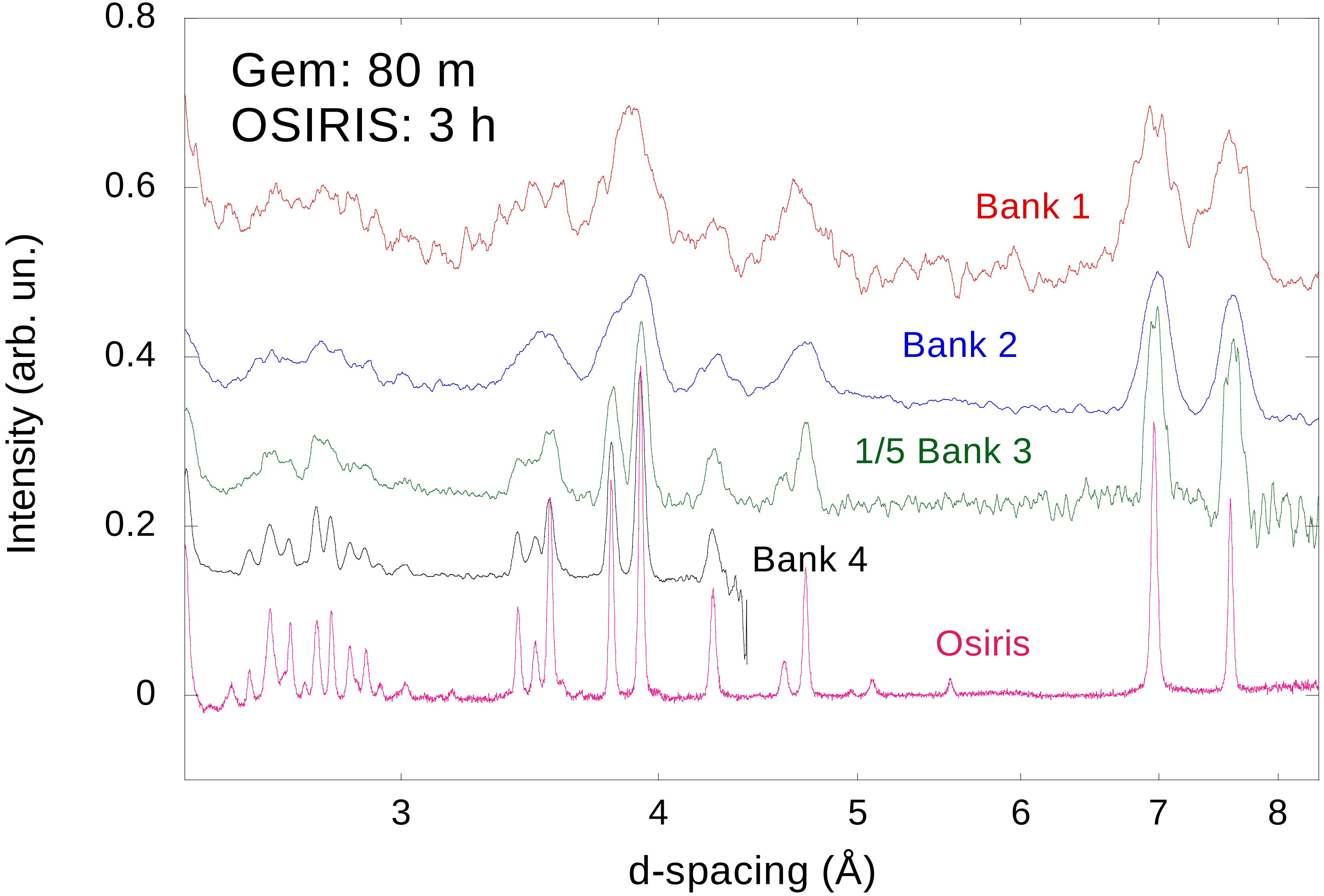


ISIS

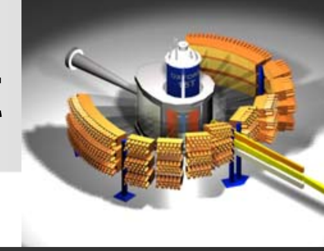
OSIRIS


Spin-flipper
solenoids
Detectors
at 170°
Non-magnetic
tank
Transmitted
beam monitor
Supermirror
converging
guide m 4
Polarisers
Polarisation
analysers
Curved
supermirror
guide m 2
Guide
section
Incident
beam monitor
Diffraction
detector
Pyrolitic
graphite analysers

GEM-OSIRIS Comparison
Magnetic Diffraction



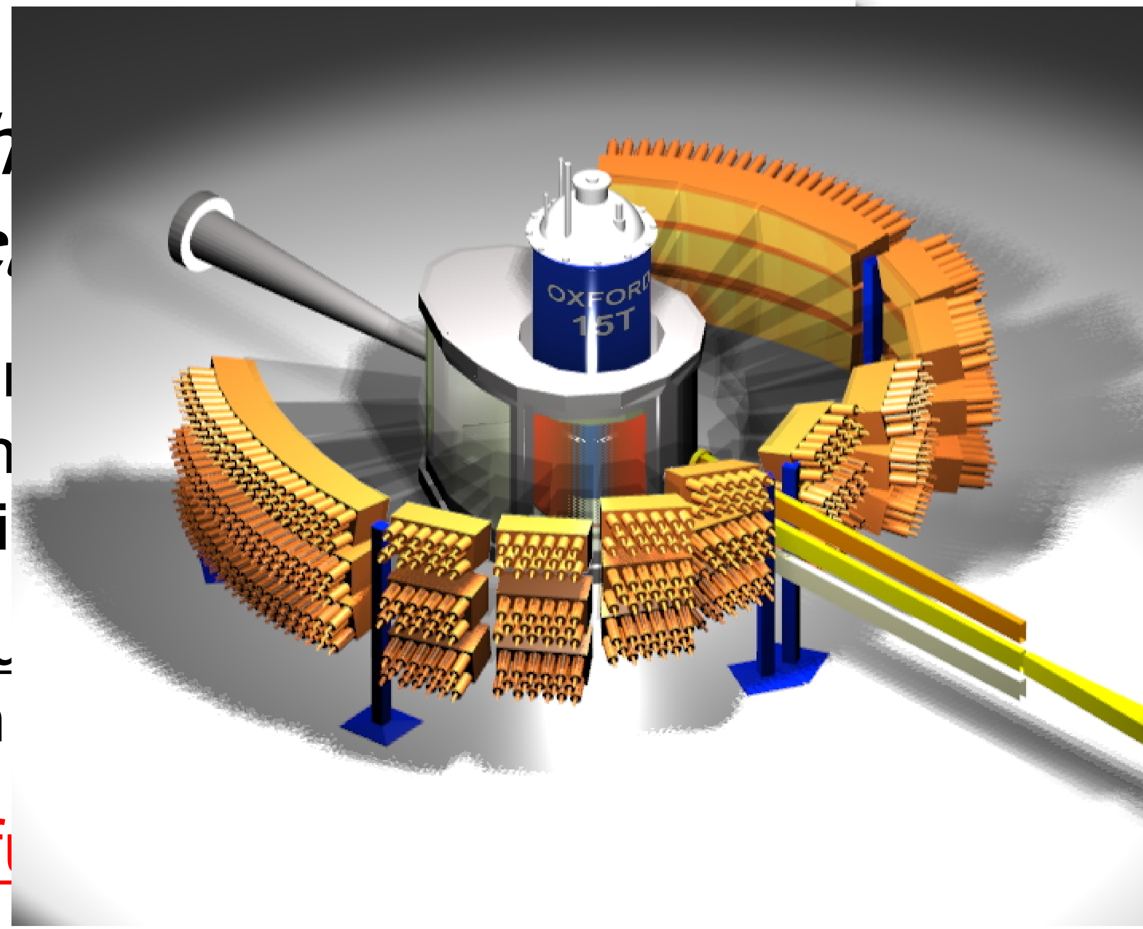
GOALS of the WISH project



To build th
diffrac

- TOF instrume
scattering. Th
towards the hi
- Need cold neu
Perfect match

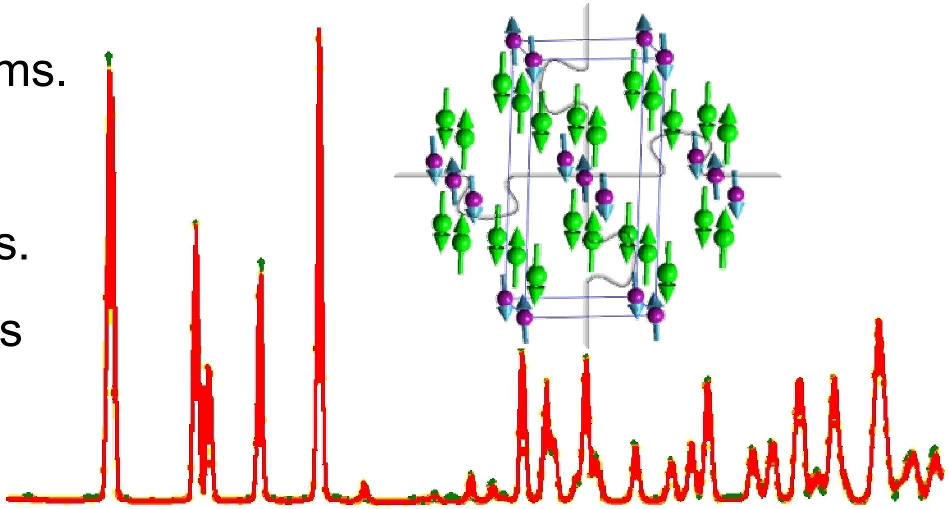
Status: fu



Paolo G. Radaelli

WISH scientific themes

- Magnetism in ionic and covalent systems.
- Model and designer magnetic systems.
- Metallic magnets.
- Magnetic clusters and nano-particles.
- Magnetism under extreme conditions (pressure, magnetic field).
- Large unit-cell structures.



Initial requirements :

- WISH is primarily a powder diffractometer to be optimised for magnetic studies, but with a full 2D detector for SX studies.
- Dedicated 15 Tesla magnet

Phase 2 construction upgrade:

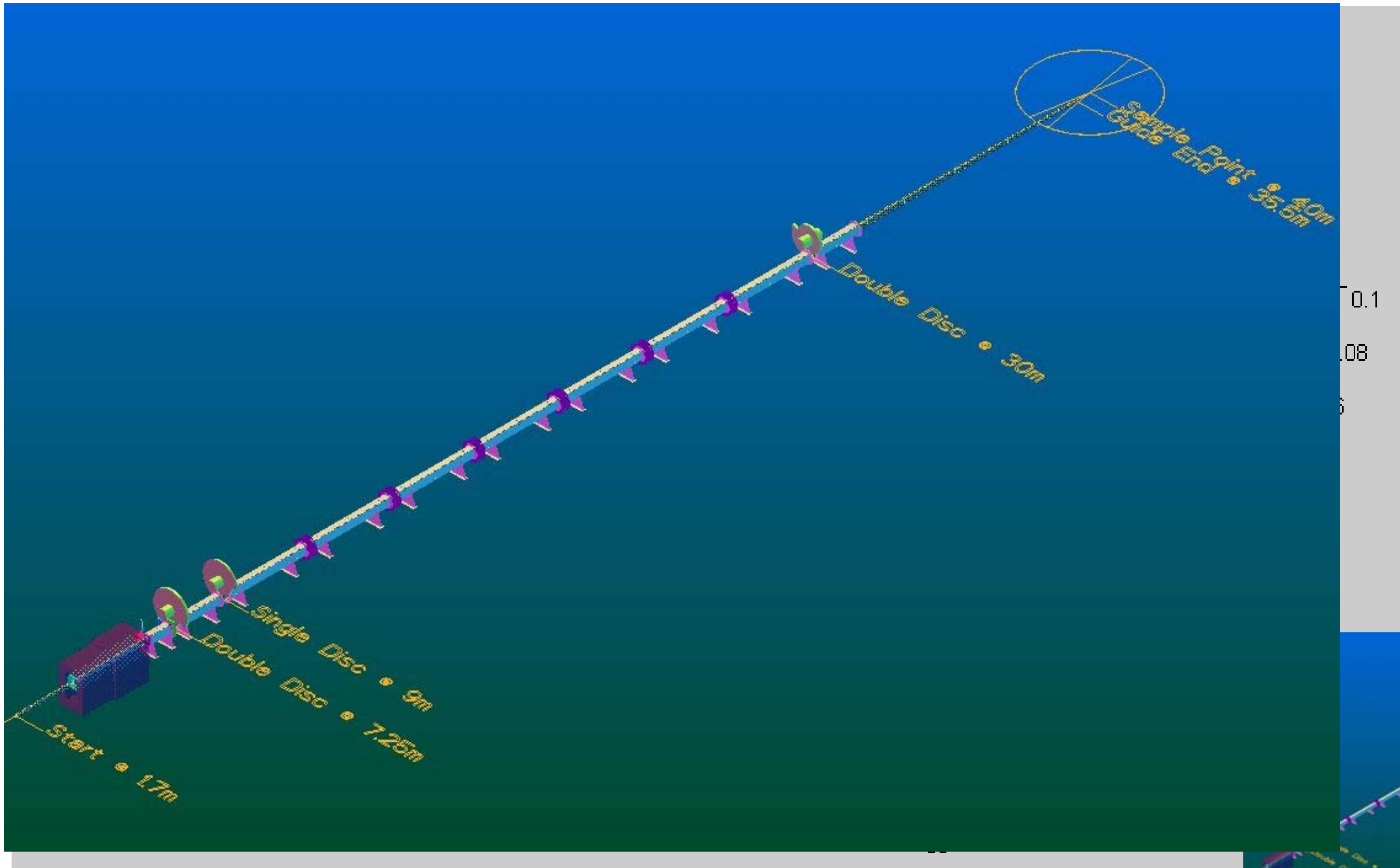
- Polarization device, flipping ratios, spin-density distributions

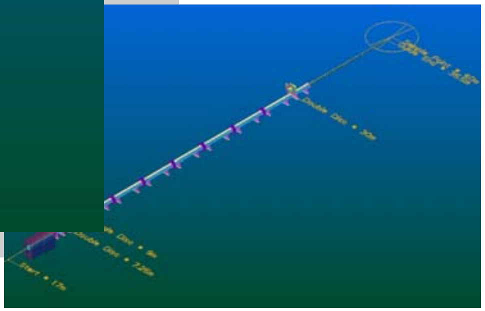
WISH Overview

Moderator	Decoupled, Unpoisoned Solid Methane, broad side
Incident Wavelengths	1.5 – 15 Å
Single-frame bandwidth	8 Å
d-spacing range	0.7 – 50 Å
L1	40m
L2	1-2.5 m
Flight path	Elliptical guide+ tunable divergence (slit collimation)
Choppers	3 disc choppers (50-10Hz)
Detectors	^{3}He linear PSD detectors covering all scattering angles between 10º and 175º.
Beam size	20 mm x 40mm (unfocussed) to 1 mm x 1mm (super-focussed)
Optimal frequency	10 Hz
Sample/detector tank	Radial Collimator, 2m diameter vacuum tank
Sample environment	All standard equipment + dedicated 15 T cryomagnet

CCLRC

Guide



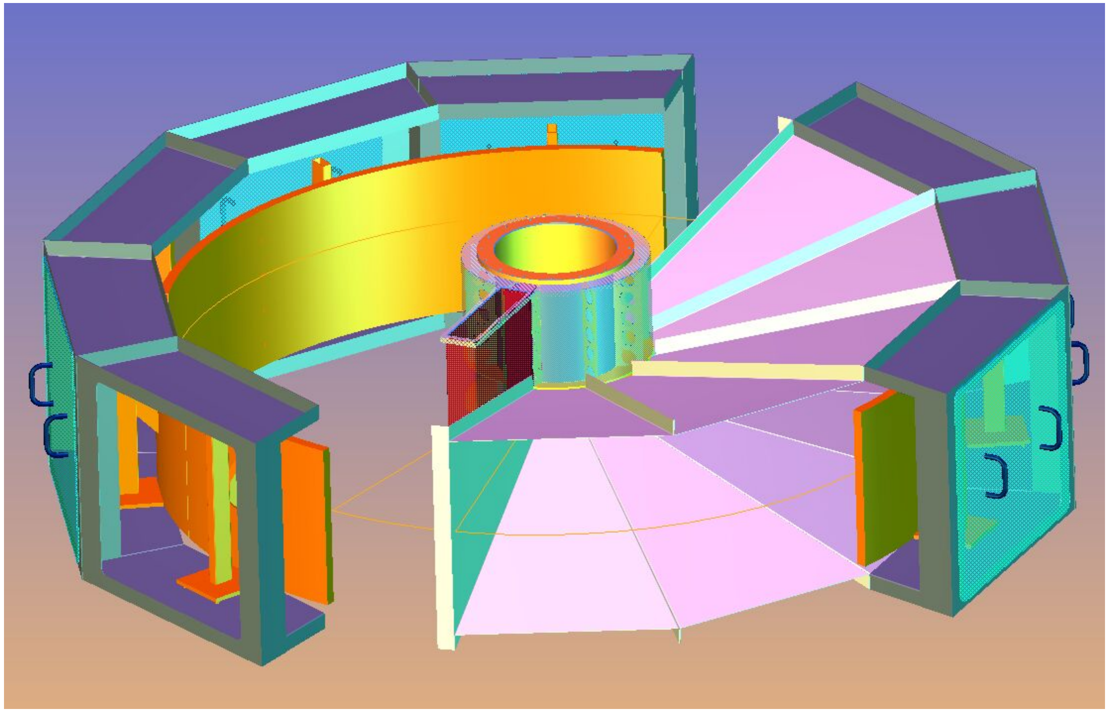


- Guide entrance : 40 x 80 mm
- Guide exit : 22 x 44 mm
- Moderator and sample positioned at ellipse extremes
- 0.5 m sections with 0.5 mm breaks every 1.5 m.

TS2 ISIS
second target station project

WISH Detector

- ^{3}He tubes PSD , 8mm diameter 125 pixels (8 mm resolution)
- 1 m long detectors (28 degrees azimuthal angle) at 2.2 m from sample position.
- Insensitive to magnetic field.
- Need good vertical resolution to reconstruct Debye-Scherrer cones.
- This option will enable single-crystal studies.
- Cover 10-170 degrees 2θ on both sides (~1200 tubes).
- Tubes on a 10 mm pitch.



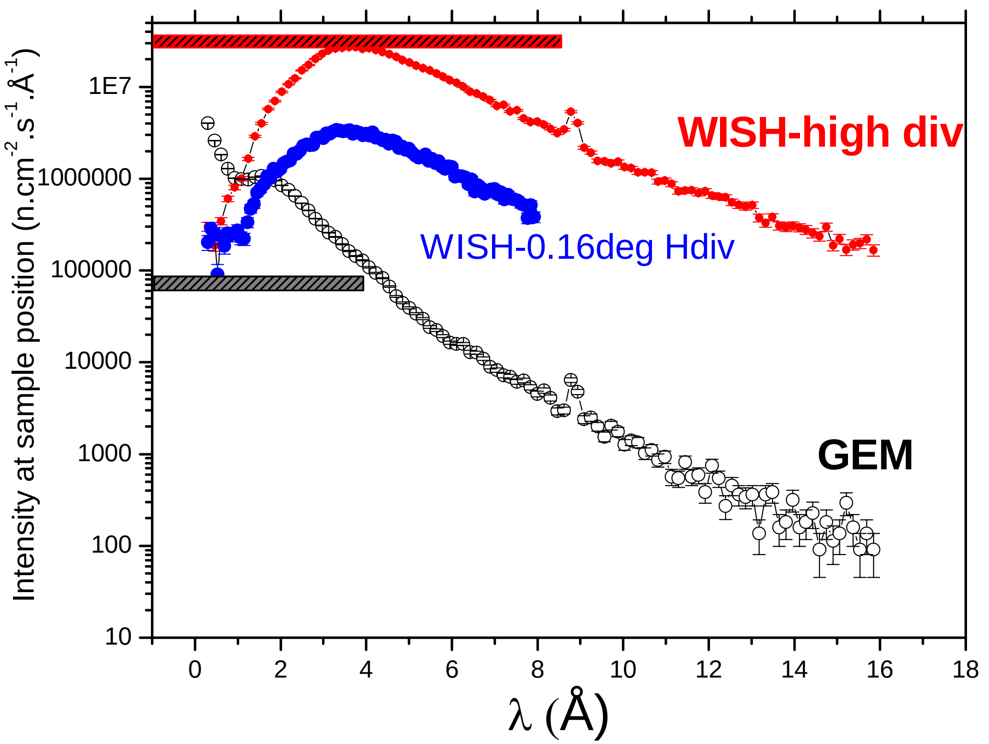
- Initially considered secondary flight path under vacuum

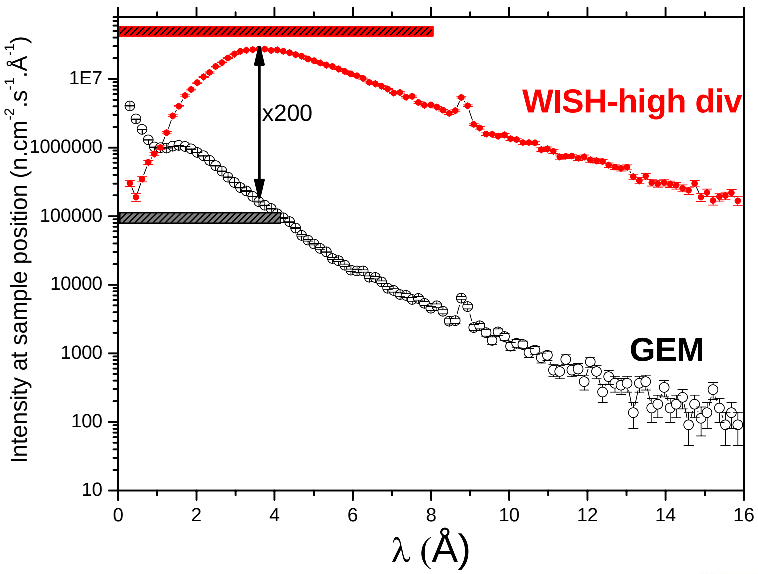
- Current Design : Secondary flight path under Ar atmosphere

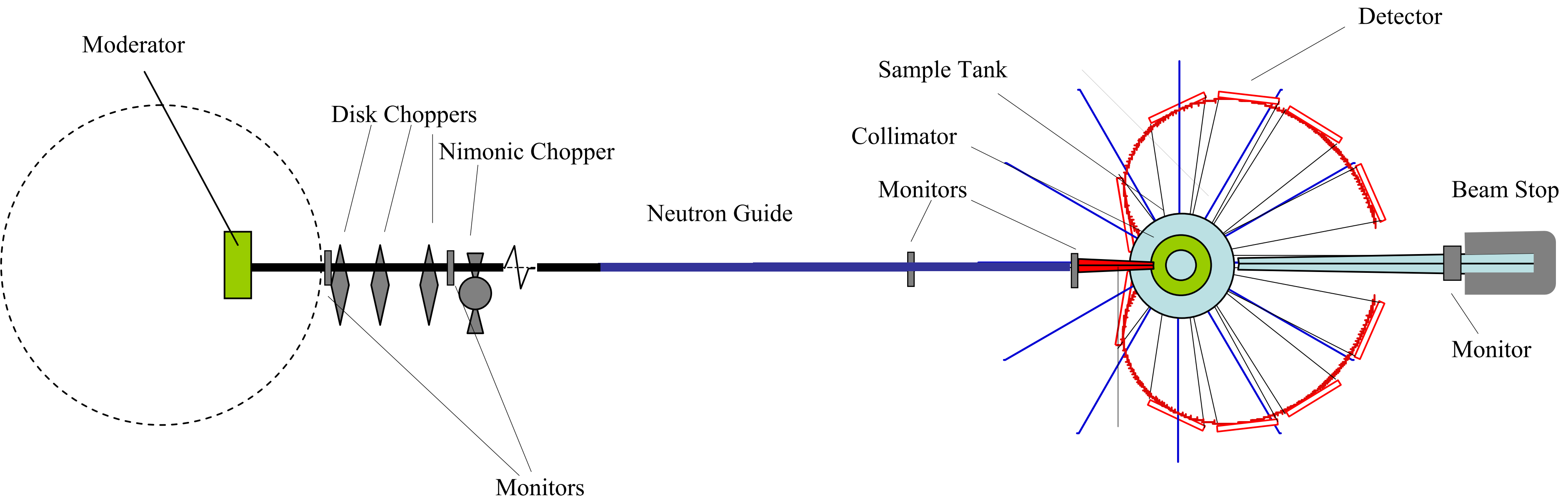
Flux at sample position

- Integrated flux is 1.2.10^8 n/cm^2/s at sample position (50 times GEM).

- Peak flux 200 times GEM at 4 Å in high divergence mode
- Peak flux 20 times GEM at 4 Å with same horizontal divergence






Moderator
Disk Choppers
Nimonic Chopper
Neutron Guide
Sample Tank
Collimator
Monitors
Detector
Beam Stop
Monitor
Monitors