Diffraction and Instruments for Neutron Diffraction

Paul Henry

Instrument Scientist – ISIS/STFC, UK

Adj. Prof. in Neutron Scattering – CTH, Göteborg, Sweden

Who am I?

- University education (solid-state chemist)
  - Chemistry Degree (with supplementary Bio-chemistry)
  - D. Phil. in Solid State Chemistry “Fullerene Intercalation Compounds”
  - 7 years Post Doctoral experience
    - High-pressure, zeolites, magnetic materials, superconductors, pigments, isotopes
- Instrument scientist (powder diffraction)
  - 5 years as co-responsible for D20 at ILL, Grenoble
  - 2.5 years responsible E9 at HZB, Berlin
  - 5.5 years responsible for powder diffraction at ESS, Lund
  - Since Jan 2017 senior instrument scientist at ISIS, Didcot
  - Since May 2013 Adjunct Professor in Neutron Scattering at CTH, Göteborg
- Research interests
  - Hydrogenous materials and neutrons
  - In-situ sample environment development
  - Materials research
  - Instrument development
  - Neutron diffraction community development

Outline

- Neutron scattering methods & science
- Key concepts
- Diffraction measurements
- Aspects influencing diffractometer design
  - Source type
  - Q range
  - Q resolution
  - Detectors
  - Availability
  - CW or TOF?
  - Powder or single crystal?
- Instrument examples
  - CW powder
  - CW single crystal
  - TOF powder
  - TOF single crystal

Neutron scattering & science

Neutron scattering methods

DIFFRACTION
Molecular structure from crystalline samples

POLARIZED NEUTRONS
Magnetic structure & dynamics

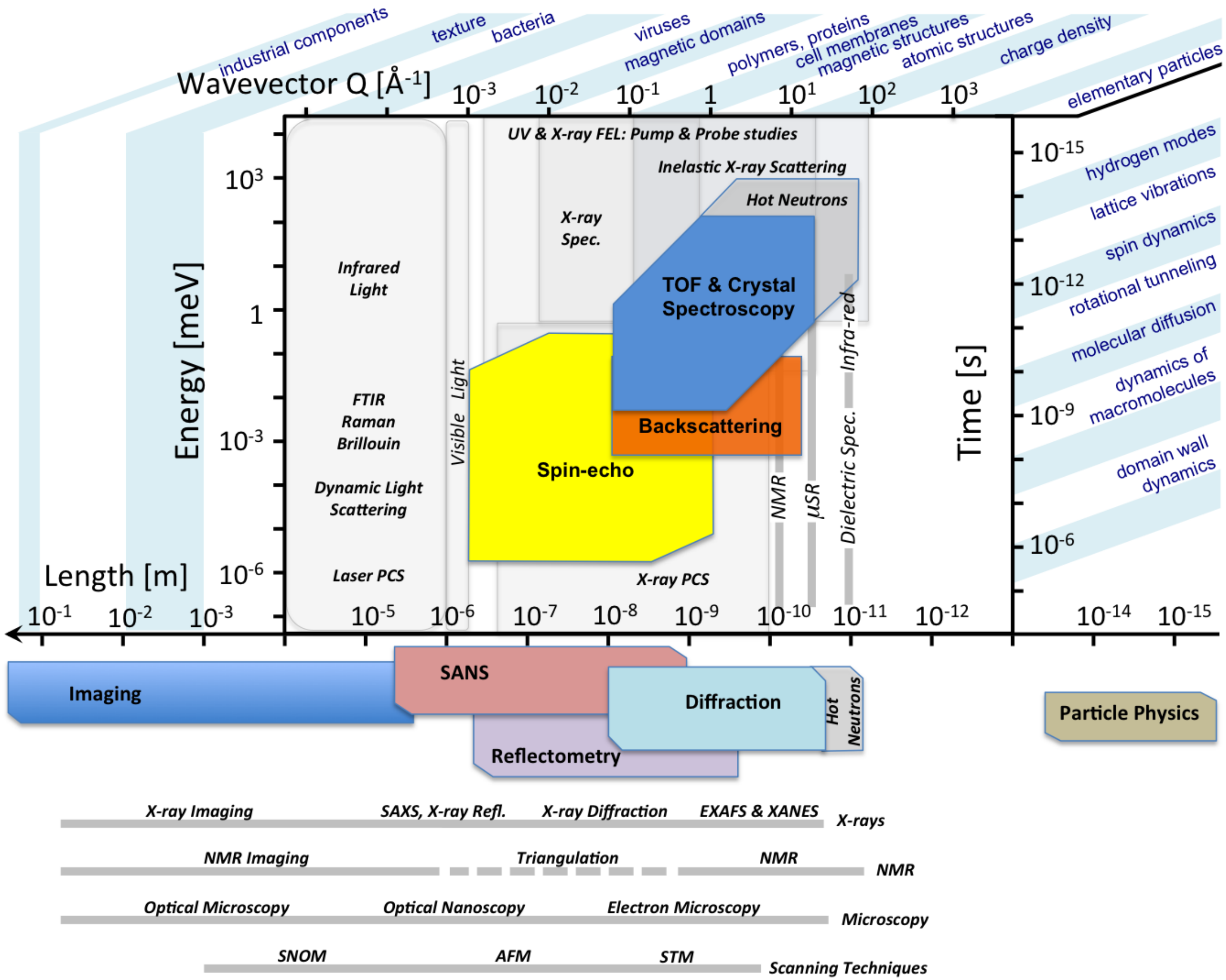
IMAGING
2D or 3D

SMALL-ANGLE SCATTERING
Large-scale structures, composite materials

NEUTRON SPECTROSCOPY
Atomic & molecular motions, vibrations, bond strenghts

REFLECTOMETRY
Surfaces & interfaces

NEUTRON SPIN ECHO
Slow dynamics



Science & Technology Facilities Council
ISIS

Why diffraction?

- Diffraction is the tool we use to determine structure
- Structure determines the physical properties of a material
- To tailor physical properties we must understand structure

Most diffraction measurements use powders. Why?

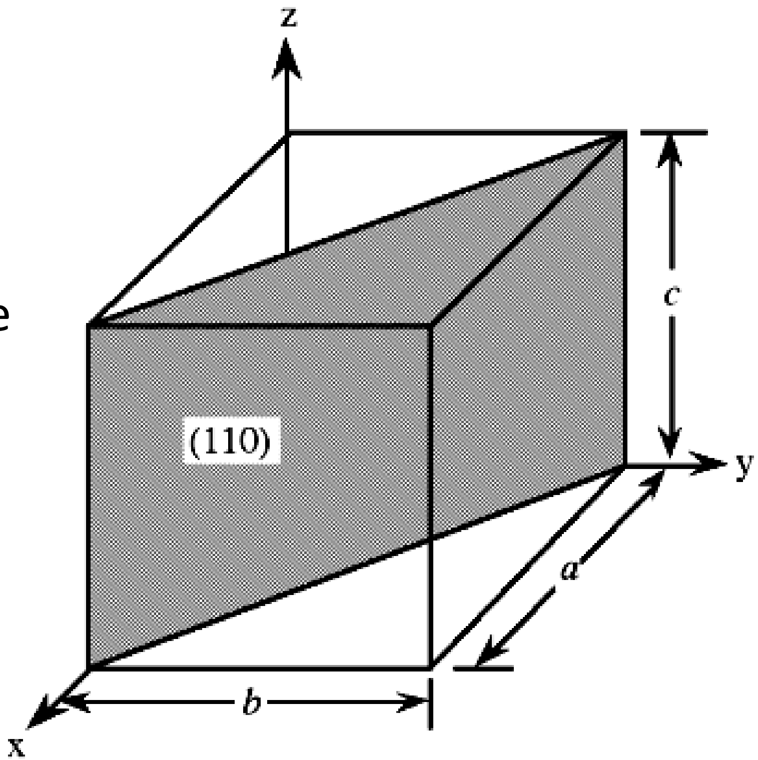
- Many materials are difficult to make as single crystals
- Most practical uses require bulk materials
- Functional materials may require non-perfect crystals or mixed systems
- Phase transitions destroy or fracture single crystals

Uses of diffraction

- Check purity of a sample
- Identify known phases
- Identify new phases
- Collect data for structural analysis
- Follow phase transitions
- Construct phase diagrams (T, P, B, etc)
- Study chemical processes *in situ*
- Monitor particle sizes
- Analyse residual stress within materials
- Process control
- Etc…

Key concepts

Miller indices / planes

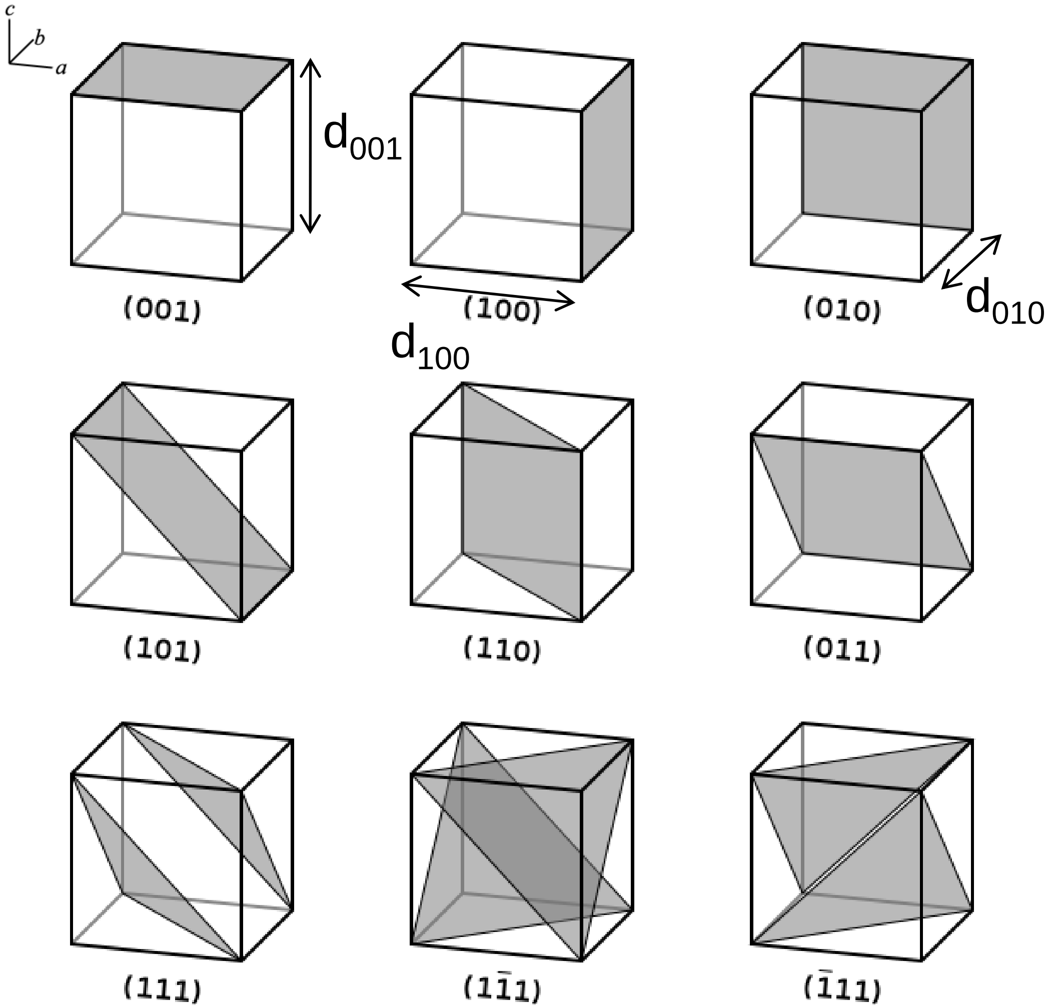


Unit cell planes can be defined by the notation called Miller indices. The Miller index is given as a hkl number where *h*, *k*, and *l* are reciprocals of the plane with the *x*, *y*, and *z* axes.

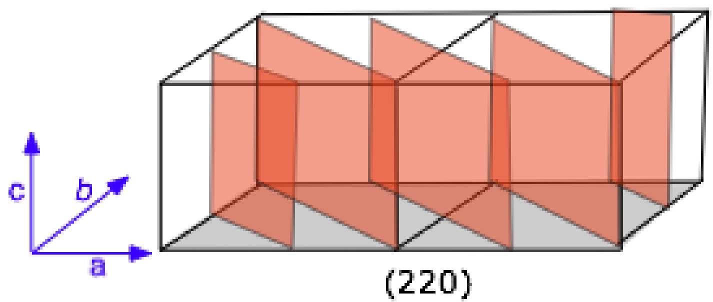
To obtain the Miller indices of a given plane requires the following steps:

Step 1. The plane in question is placed on a unit cell.
Step 2. Find its intercepts with each of the crystal axes.
Step 3. The reciprocal of the intercepts are taken.
Step 4. Multiply by a scalar to get a ratio of integers.

Miller indices

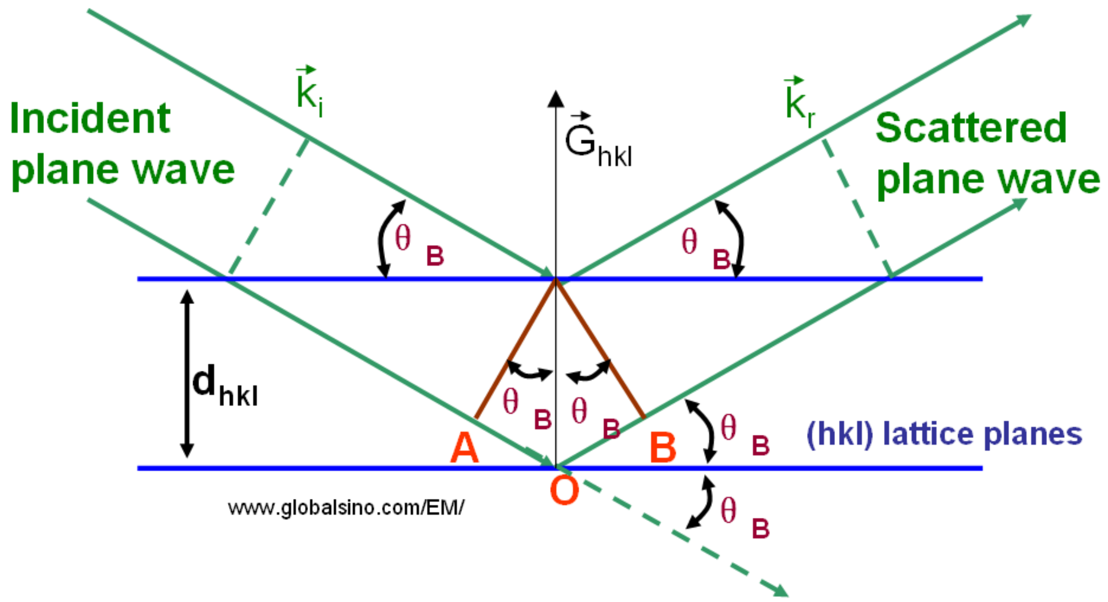


The higher the Miller index the less distance there is between equivalent planes, dividing the unit cell into ever smaller slices



For higher symmetry cells interplane distances are identical $d_{001} = d_{010} = d_{100}$ for cubic

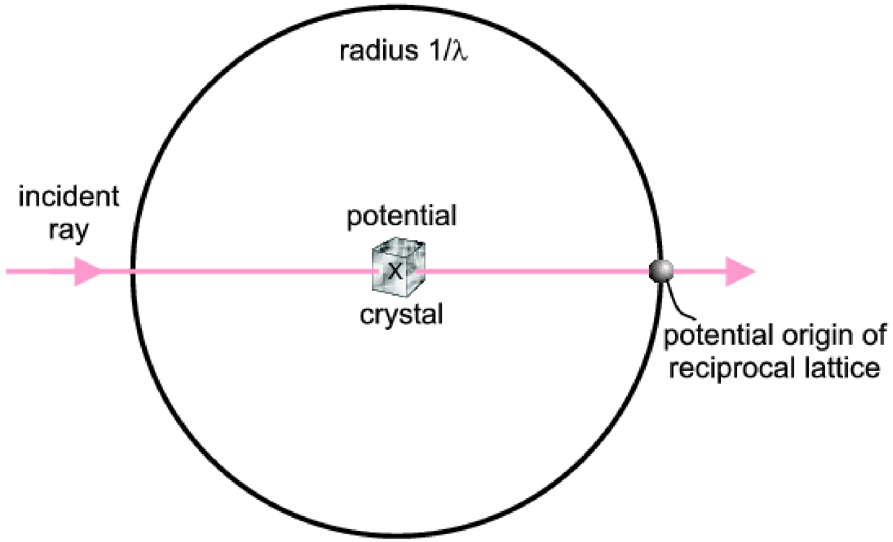
The Bragg equation



- Constructive interference occurs when the waves reflected from adjacent scattering planes remain in phase – diffraction peak is observed
- The path difference travelled by waves between adjacent planes must be an integral multiple of the wavelength

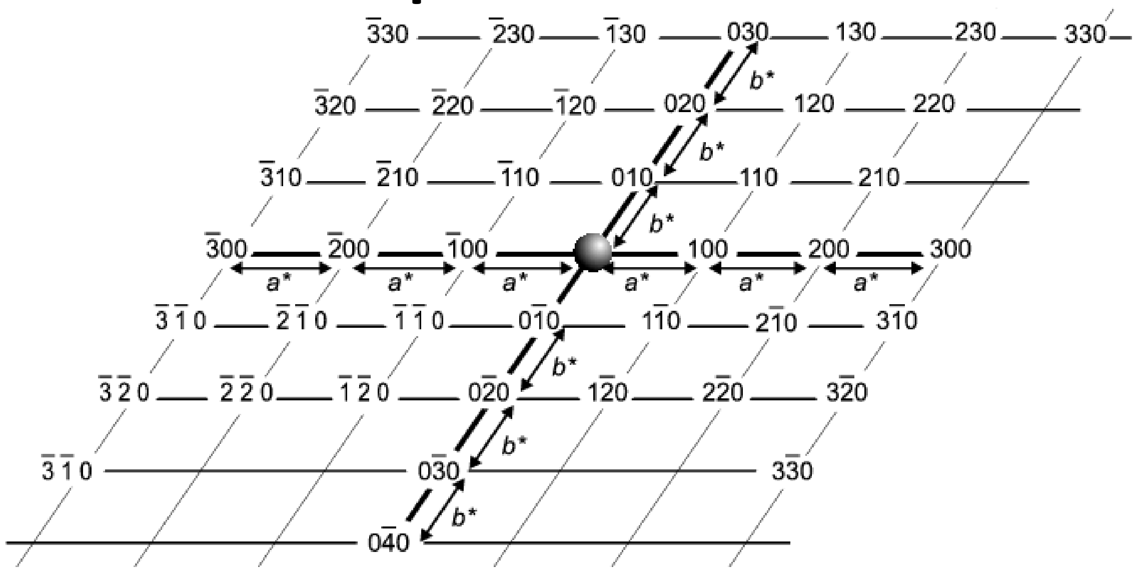
$$n\lambda = 2d\sin\theta$$

Ewald sphere



- A sphere of radius 1/λ (2-D projection shown above)
- Potential diffracted X-rays/neutrons can be along any radius from the centre of the sphere to the circumference (including out of plane in the projection above). This represents the experimental possibilities (λ, possible 2θs)

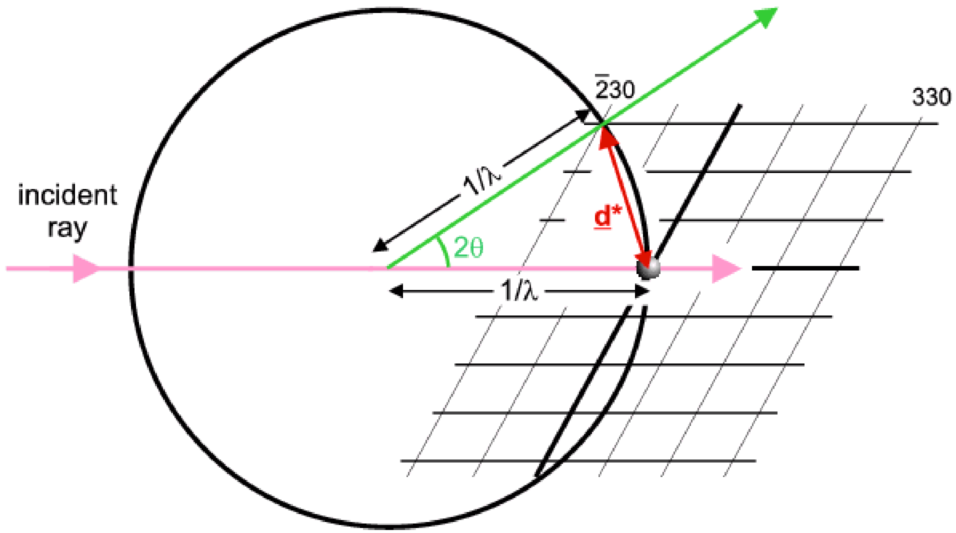
Reciprocal lattice



- Alternative view of the crystal structure (hk0 plane illustrated)
- The reciprocal lattice consists of points which represent diffraction possibilities
- Each point can be labeled with a Miller index
- The units of this lattice are a*, b* and c* and any point can be reached using the vector equation d* = ha* + kb* + lc*

Science & Technology Facilities Council
ISIS

Condition for observing Bragg diffraction

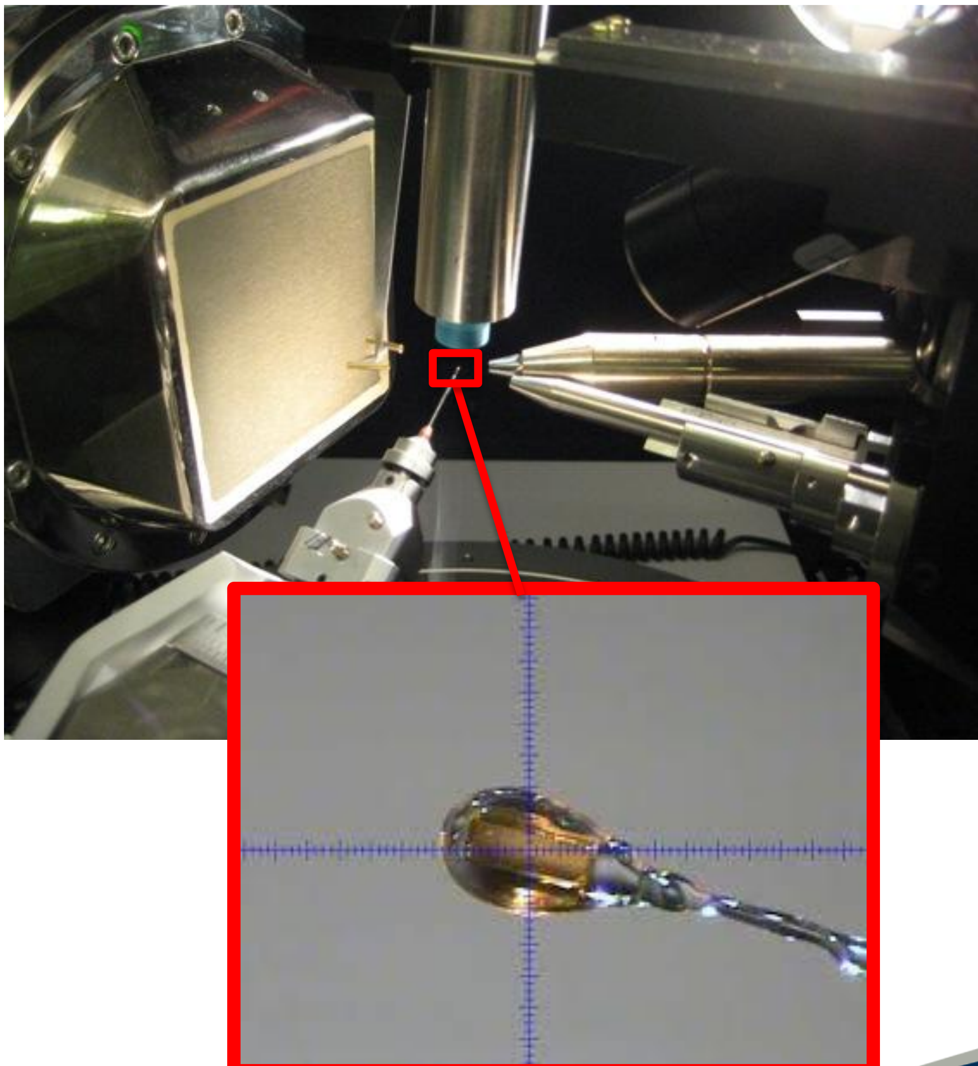


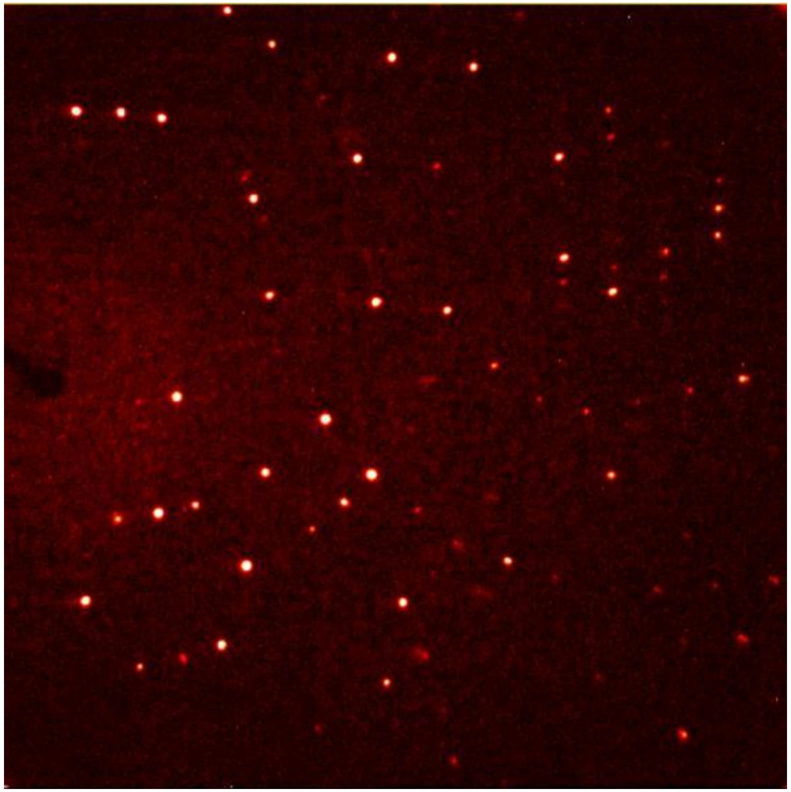
- Diffraction observed when a reciprocal lattice point intersects Ewald sphere
- Crystal rotation brings other lattice points into contact with Ewald sphere
- The vector from origin to lattice point is d* (reciprocal lattice spacing) is red – it is exactly equal to 1/d and its direction is perpendicular to the hkl plane
- The direction of the diffracted ray is indicated in green

Science & Technology Facilities Council ISIS

Diffraction measurements

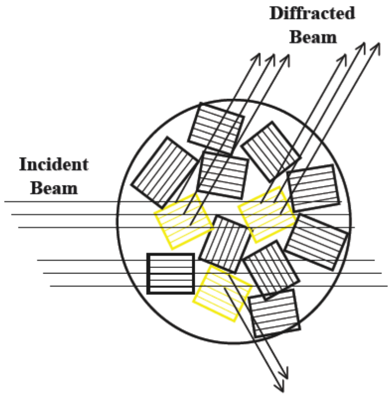
Single crystal diffraction

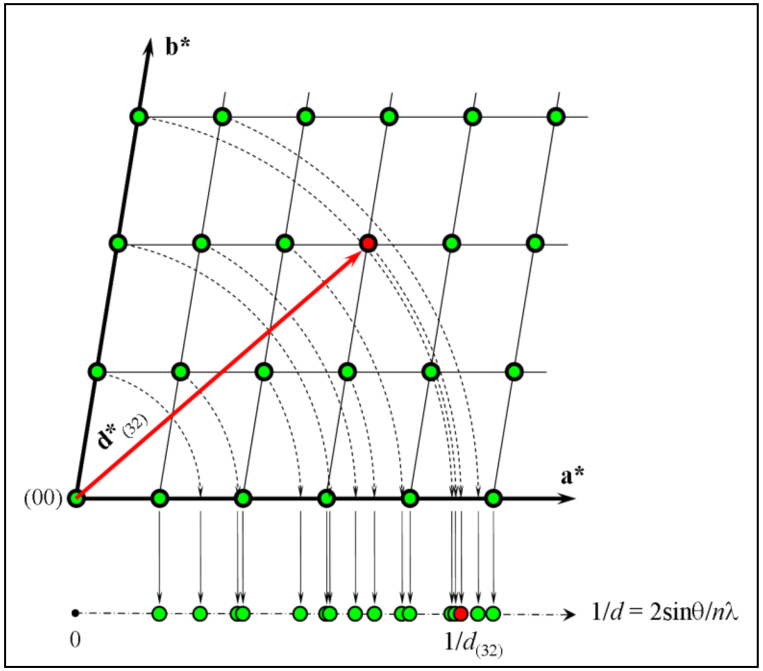




Science & Technology Facilities Council
ISIS

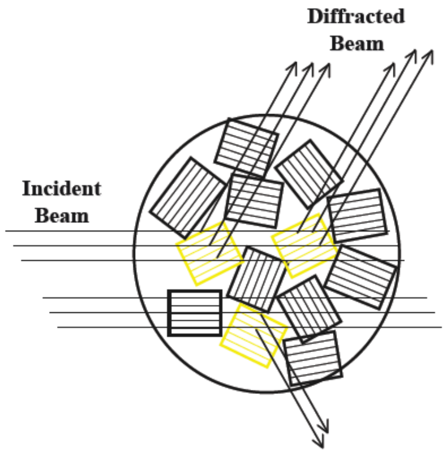
Powder diffraction in reciprocal space

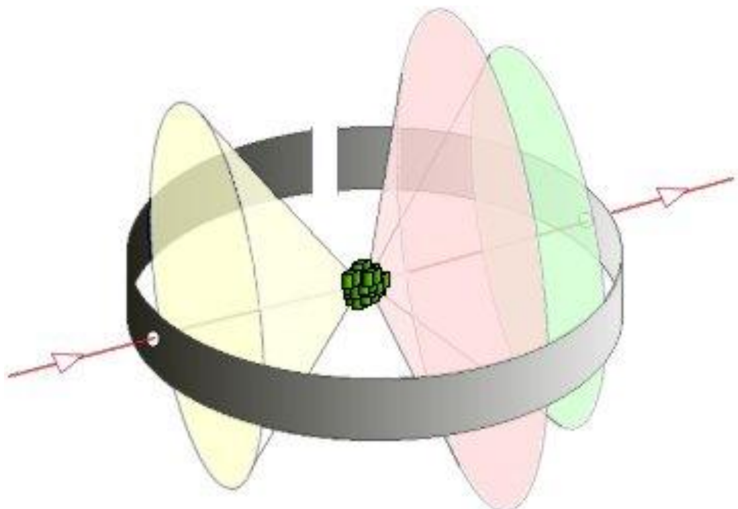


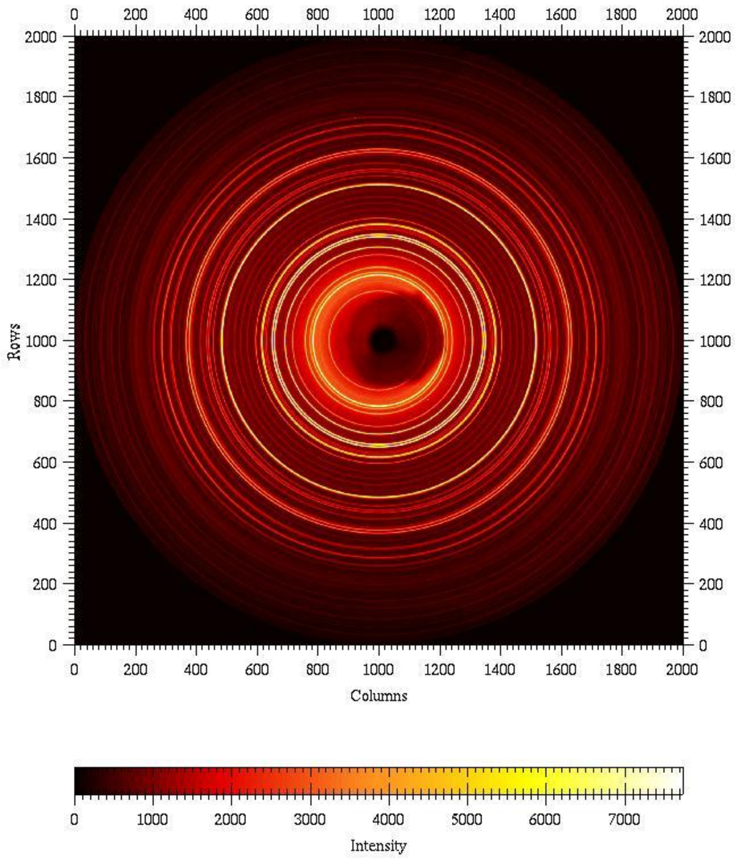


- Many crystallites with random orientation mean that each reciprocal lattice point will occur in every orientation possible, broadening into the surface of a sphere with radius d*
- The intersection of the Ewald sphere and the reciprocal lattice becomes a cone (intersection of 2 spheres)
- The directions of the vectors are lost and only the lengths of the reciprocal lattice vectors are measurable with powder diffractometers
- 3-D information collapsed into 1-D

Powder diffraction

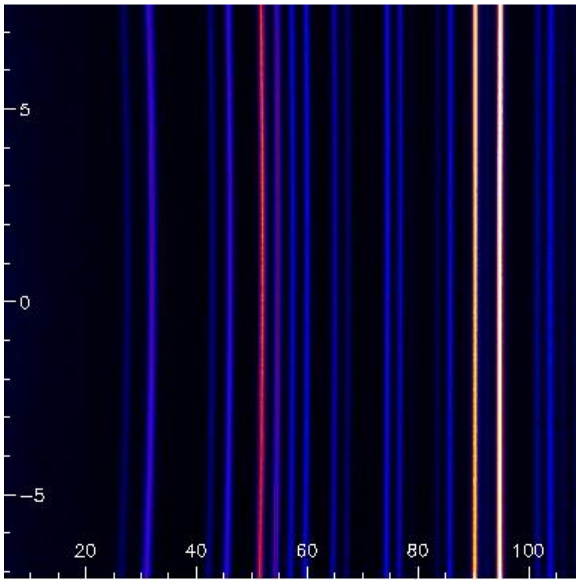


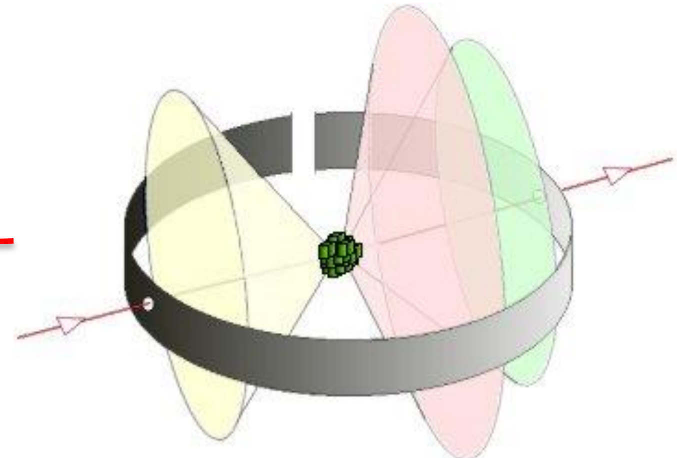


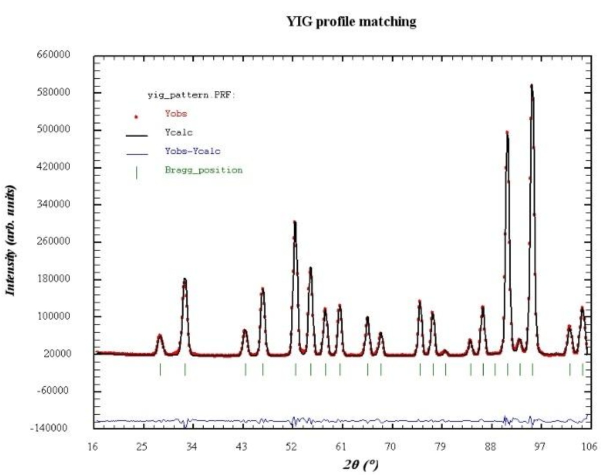


Science & Technology Facilities Council
ISIS

Powder diffraction





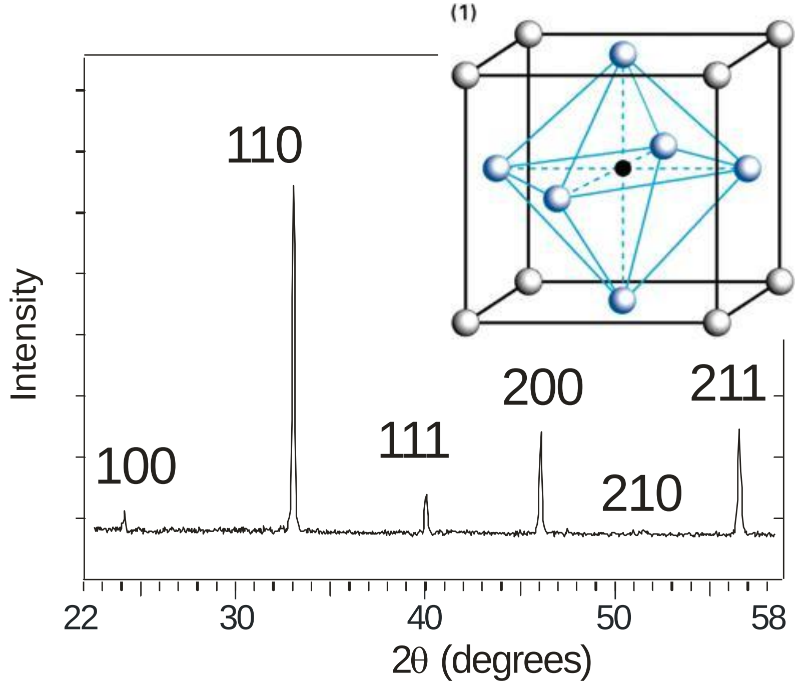


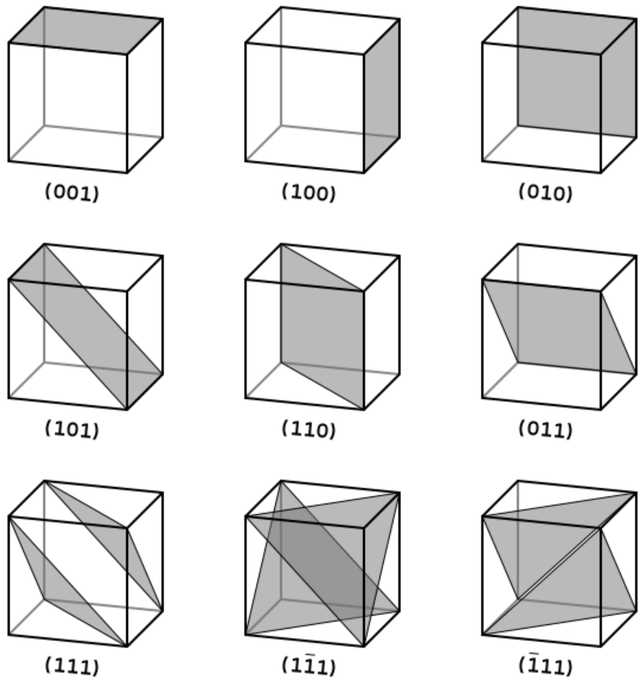
Typical monochromatic powder diffractometer

- Area or point detector that scans scattering angle, intersecting the Debye-Scherrer cones
- Collapse data into 1-D diffraction pattern

Science & Technology Facilities Council
ISIS

Miller plane equivalence in powder diffraction



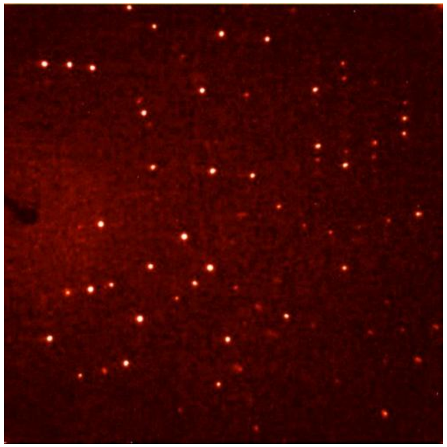


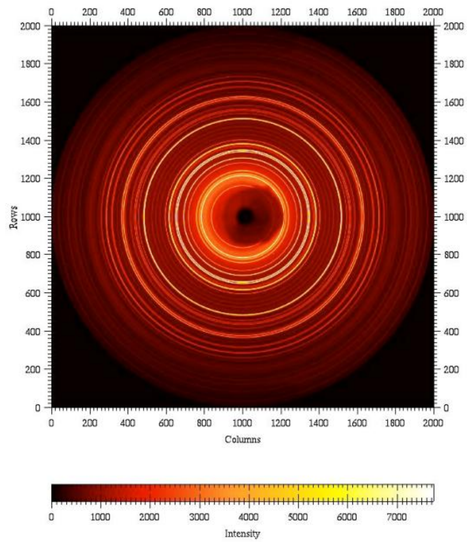
All equivalent planes occur at same scattering angle

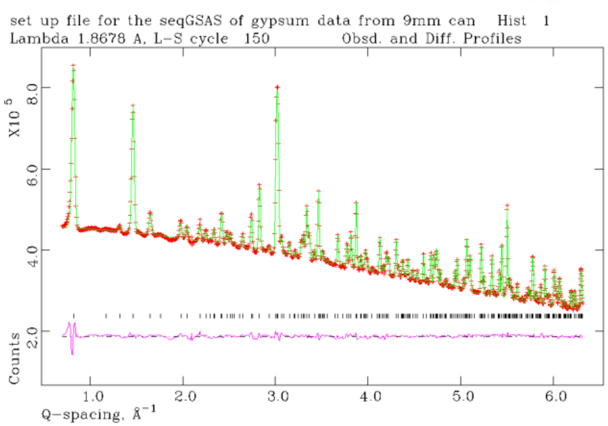
All planes separated by the same distance occur at one scattering angle in powder diffraction

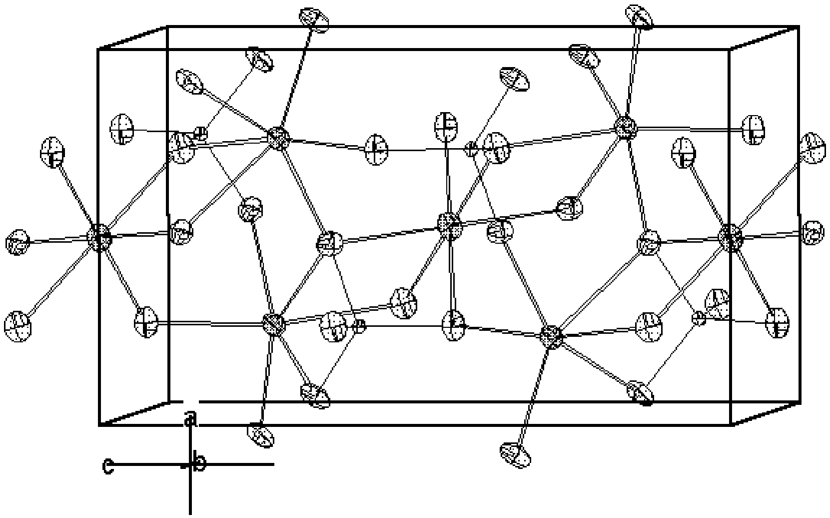
e.g. (511) and (333) occur at same 2θ for a cubic material

Structure solution and refinement









Science & Technology Facilities Council
ISIS

Intensity and structure factor

$$I_{hkl} \propto |F_{hkl}|^2$$

Measured intensity proportional to F_{hkl}^2 and so we cannot tell whether F_{hkl} is positive or negative – the Phase problem

$$F_{hkl} \propto \Sigma f_i \exp[2\pi i(hx_i + ky_i + lz_i)] \exp(-U_i Q^2/2)$$

f_i is the scattering power (form factor of the ith site i.e. (x_i, y_i, z_i) and incudes fractional occupancy

Contribution of the ith site to the F_{hkl} in question

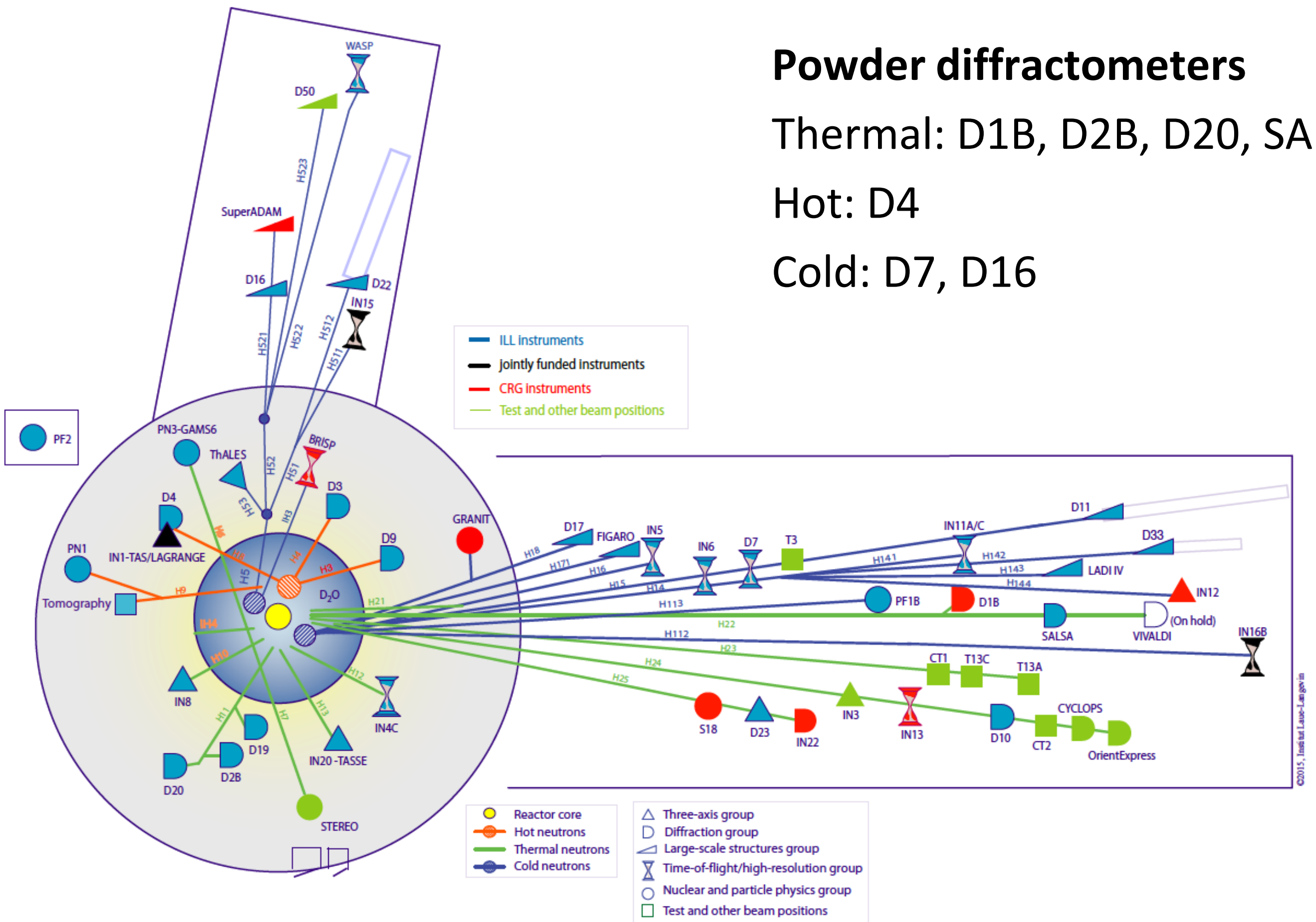
Atomic displacement of the ith atom site

See diffraction workshop for details

Science & Technology Facilities Council ISIS

Aspects influencing diffractometer design

Diffraction at a continous source: ILL



Powder diffractometers

Thermal: D1B, D2B, D20, SALSA

Hot: D4

Cold: D7, D16

Single crystal diffractometers

Thermal: D19, D10, CYCLOPS, VIVALDI, D23, OrientExpress

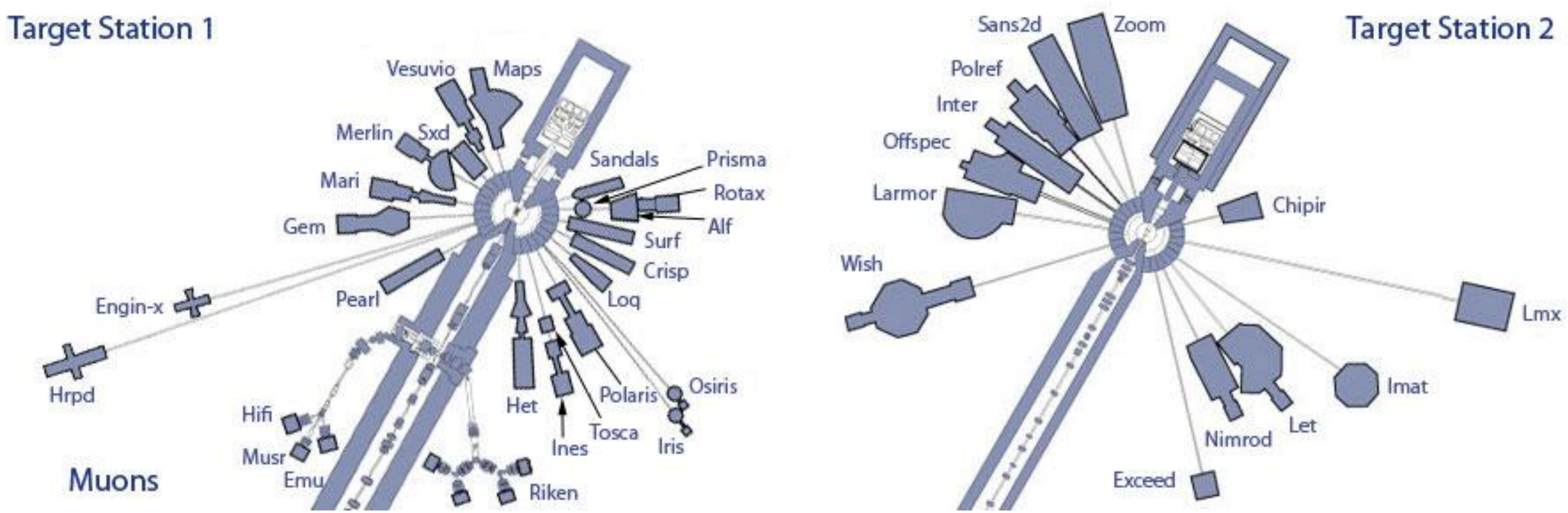
Hot: D3, D9

Cold: LADI-III, D7

Half of instrument suite are diffractometers

Science & Technology Facilities Council
ISIS

Diffraction at a pulsed source: ISIS



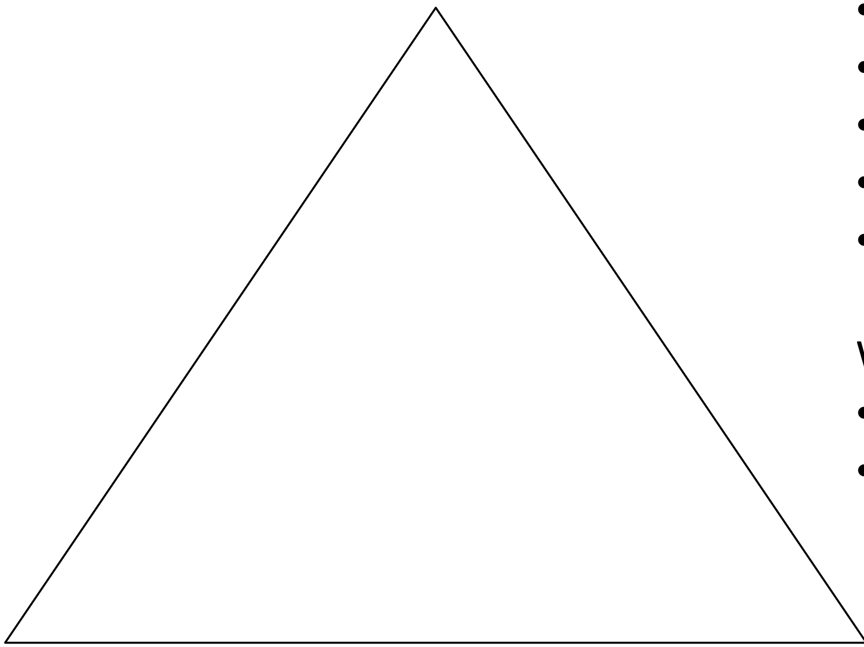
HRPD	WISH
ENGIN-X	IMAT
GEM	(LMX)
SXD	(EXCEED)
INES	SANDALS
PEARL	NIMROD
POLARIS	OSIRIS

Almost half of the instrument suite are diffractometers or carry significant diffraction capability

Moderators

	cold	thermal	hot
moderator	liquid D_2	Liquid D_2O	graphite
moderator temperature	20K	300K	2000K
neutron wavelength	3→20Å	1→3Å	0.3→1Å
sample lengthscale	1Å→100 nm	0.3→5Å	0.1→2Å
sample timescale	1kHz→1 THz	0.1→10 THz	1→100 THz

Designing diffractometers



But also:

- Unit cell volume
- Sample environment restrictions
- Need for *in situ* capability
- Sample size
- Sample state
- Etc…

Which come from:

- Science case requirements
- Available budget!

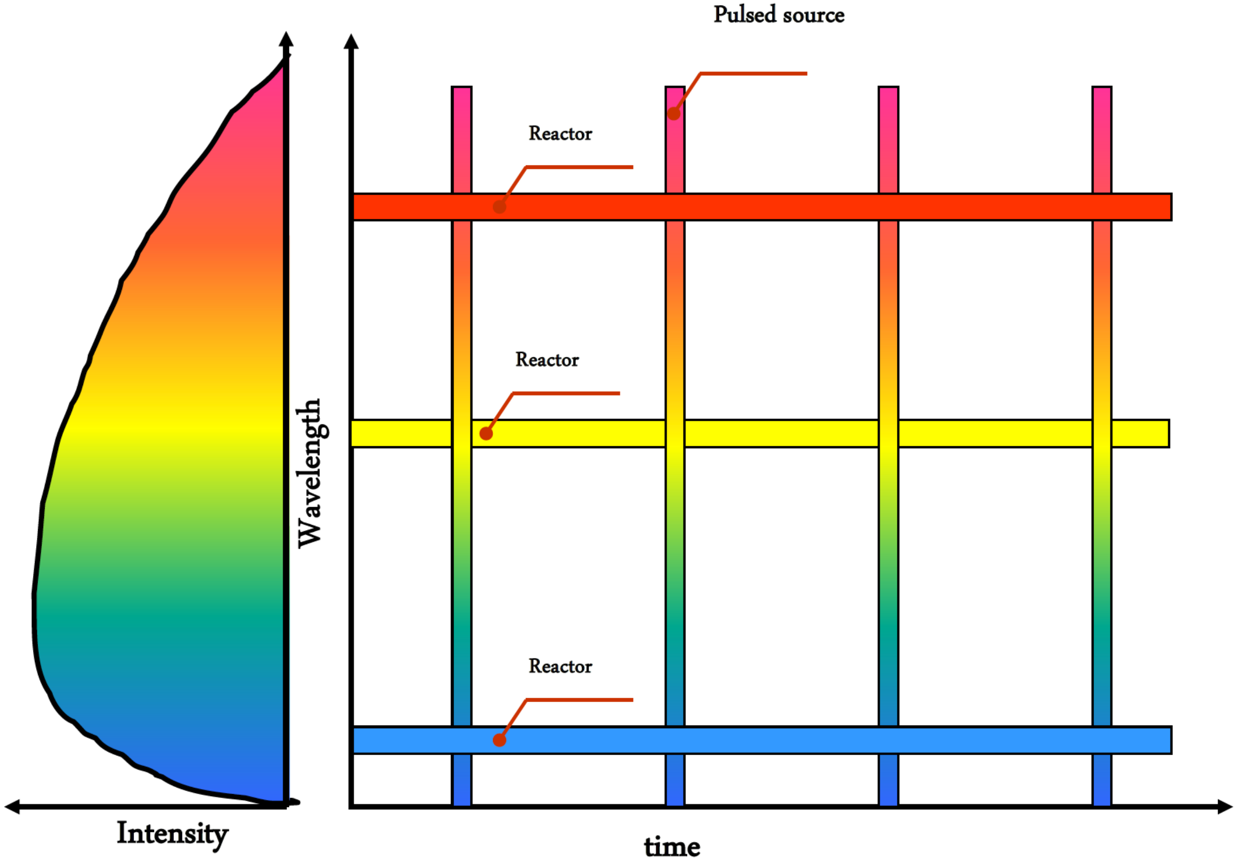
Types of diffractometer

$$\lambda = 2d\sin\theta$$

- Monochromatic (CW)
  - Fix wavelength and scan detector angle
  - Multiple 2θ required to cover Q(d) spacing range
  - Q(d) spacing limit $4\pi/\lambda$ ($2\pi/d$)
  - Instrumental count rate factors: Source power, monochromator reflectivity, detector coverage and efficiency, etc
- TOF
  - Fix detector angle and scan wavelength
  - Single 2θ covers range of Q(d) space
  - Q(d range) determined by λ_{max}, λ_{min} and θ
  - Instrumental count rate factors: Source power, moderator performance, beam transport efficiency, detector coverage and efficiency, etc

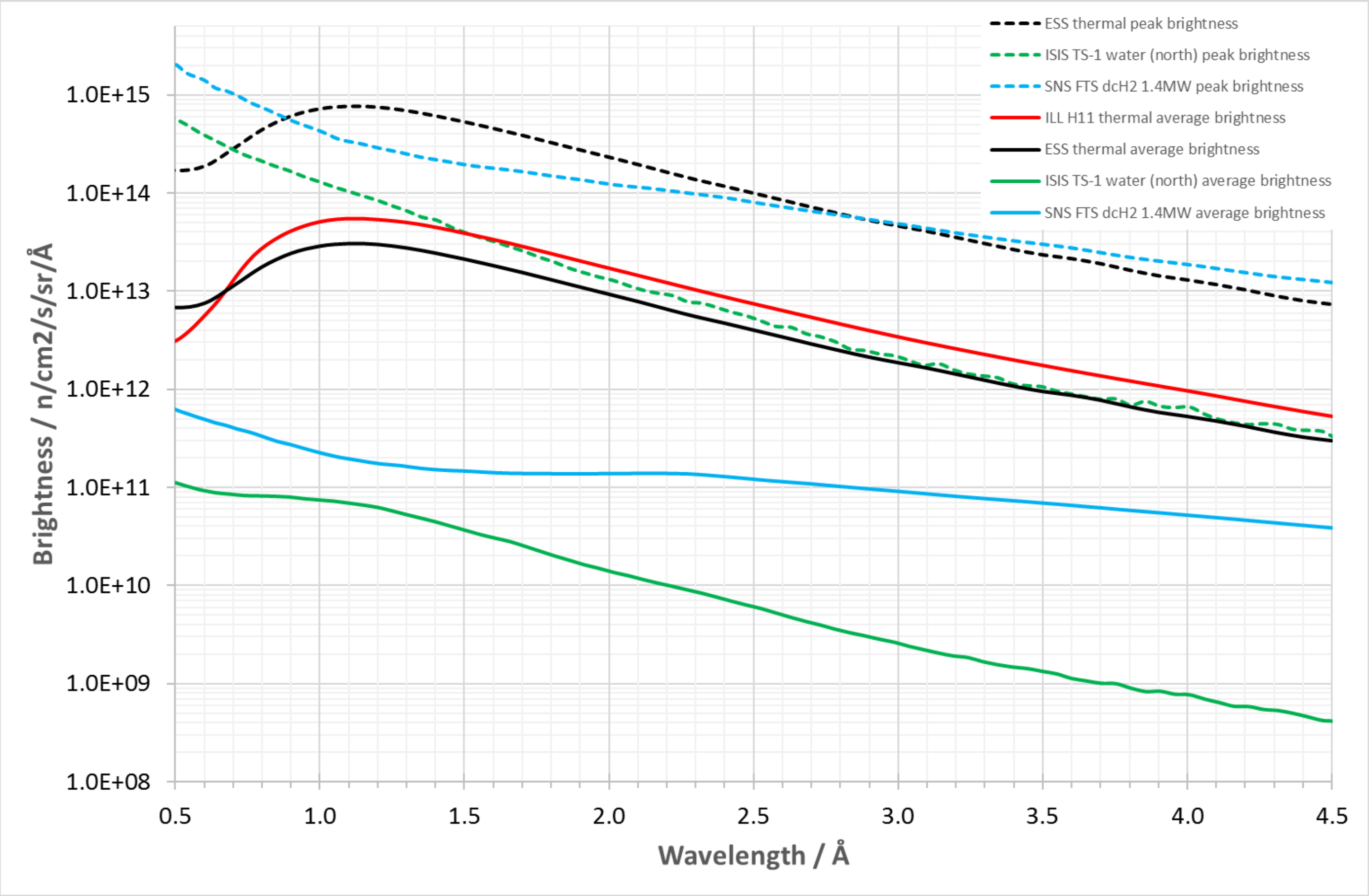
Science & Technology Facilities Council ISIS

Influence of source type on CW or TOF



Some of the neutrons all of the time or all of the neutrons some of the time

Influence of source brightness on CW or TOF



High peak brilliance good for TOF

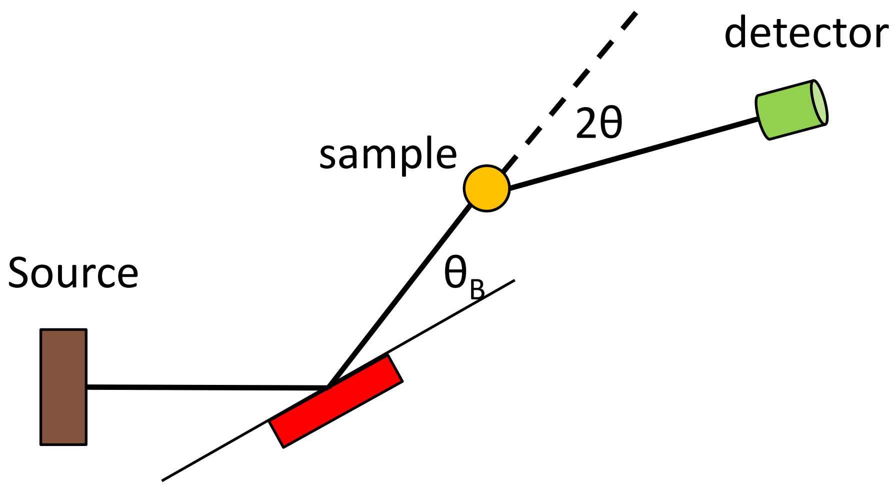
High time-average brilliance good for CW

Science & Technology Facilities Council

ISIS

Instrument layouts

CW

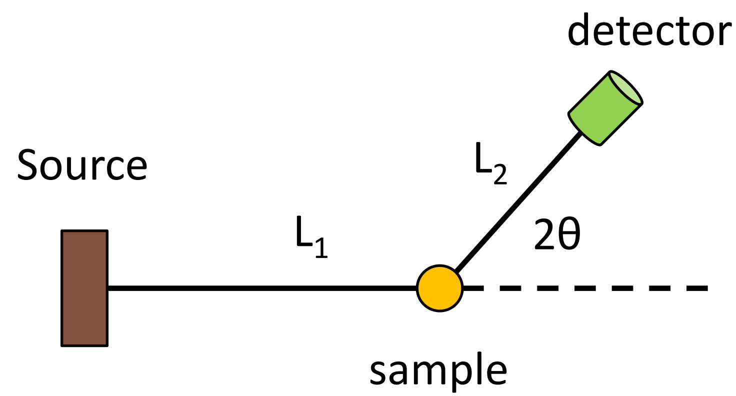


$$\lambda = \frac{2d_c \sin\theta_B}{n}$$

$$\frac{\Delta\lambda}{\lambda} = \frac{\delta d}{d} + \cot\theta\delta\theta$$

Correlation between λ and θ_B

TOF

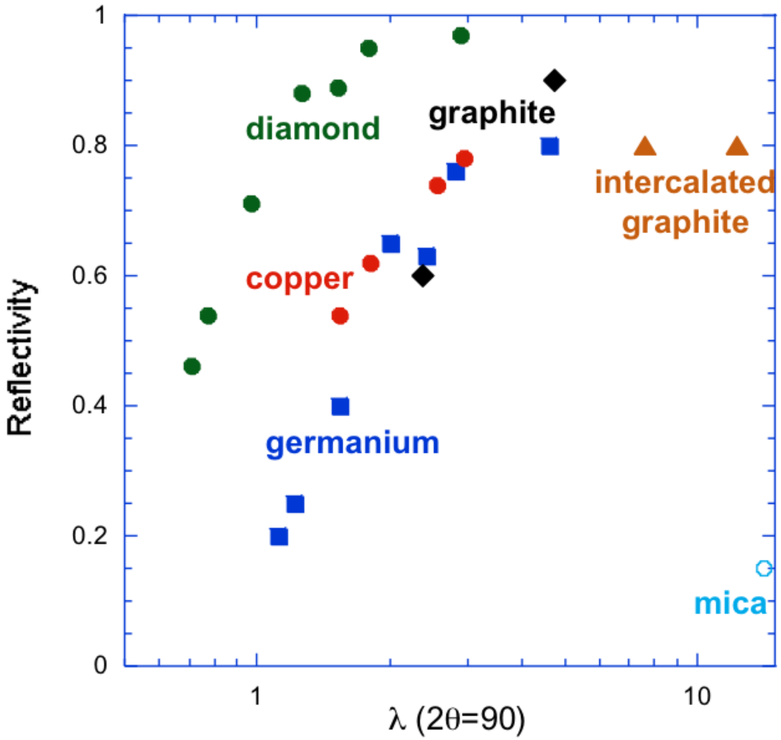


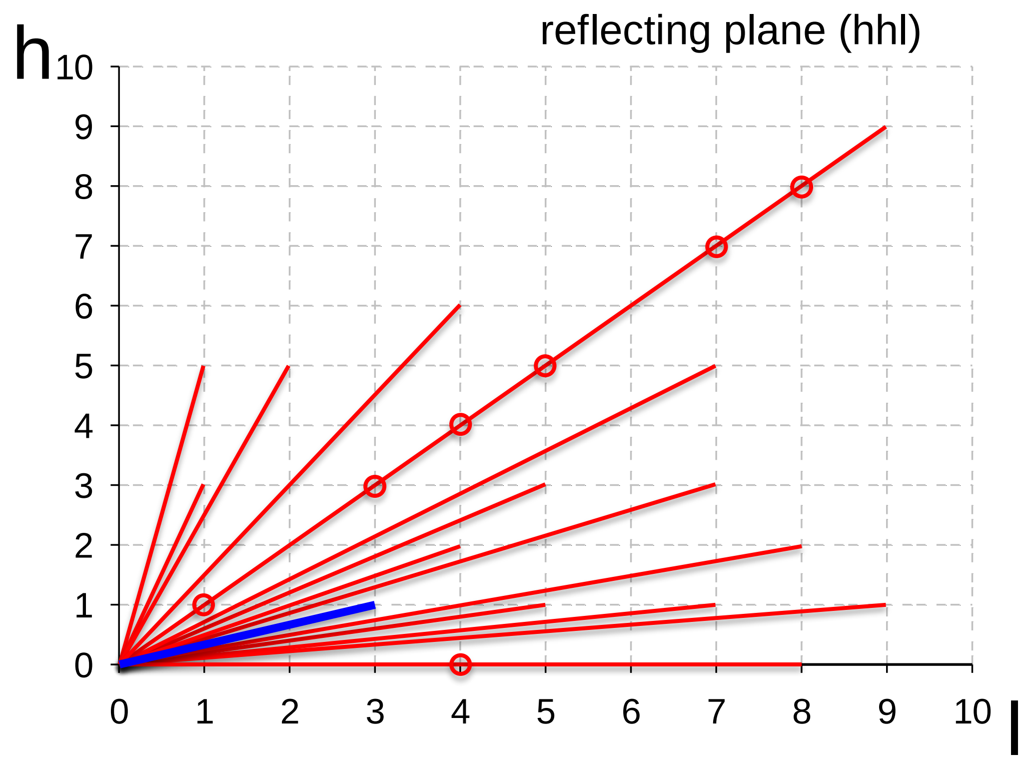
$$\lambda = \frac{3956}{v} = \frac{3956\,(t - t_0)}{L_1 + L_2}$$

$$\Delta\lambda \sim \delta t_0, \delta t, \delta L$$

No correlation between λ and θ

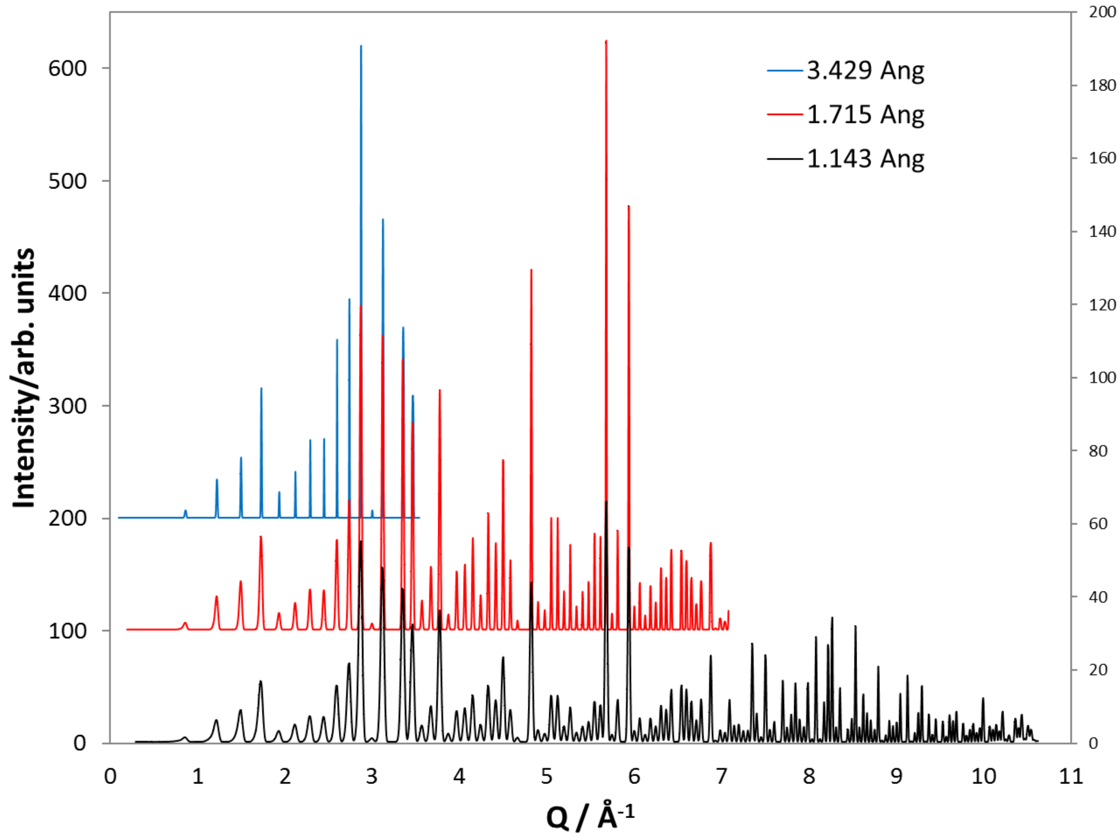
Monochromator materials





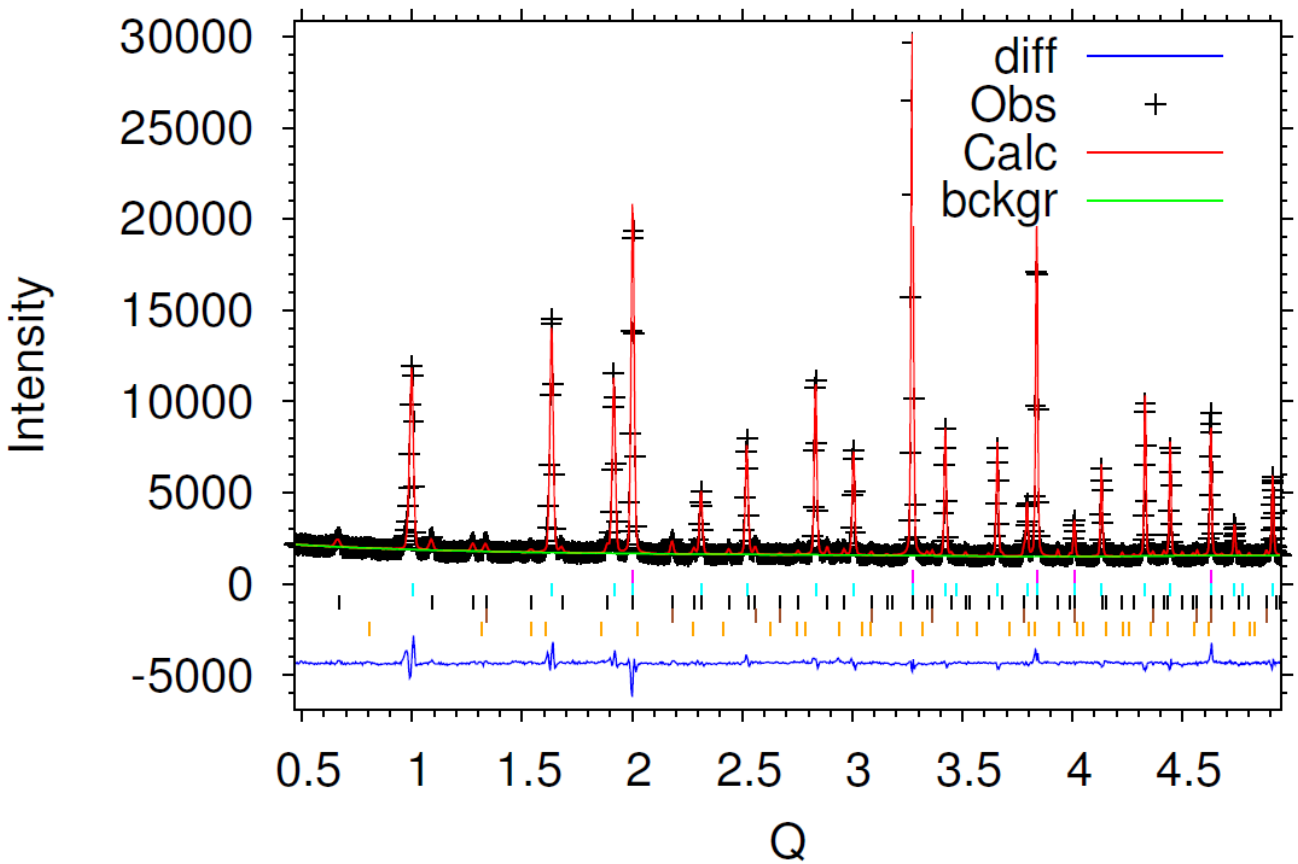
Choose material and plane to access necessary Q-range
Reflectivity falls as wavelength decreases

Q range with monochromators



Shorter wavelengths access higher Q but have lower reflectivity

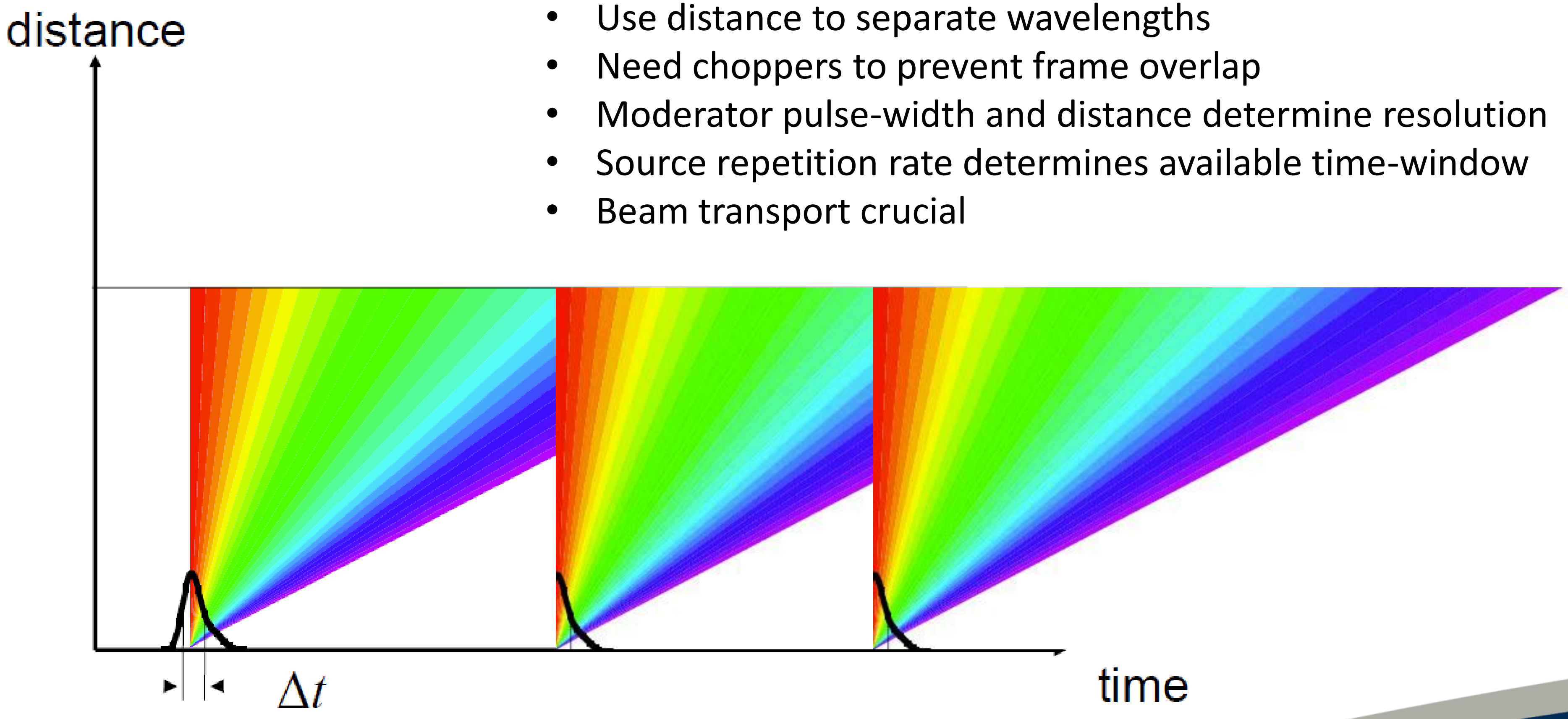
Higher reflection order contamination



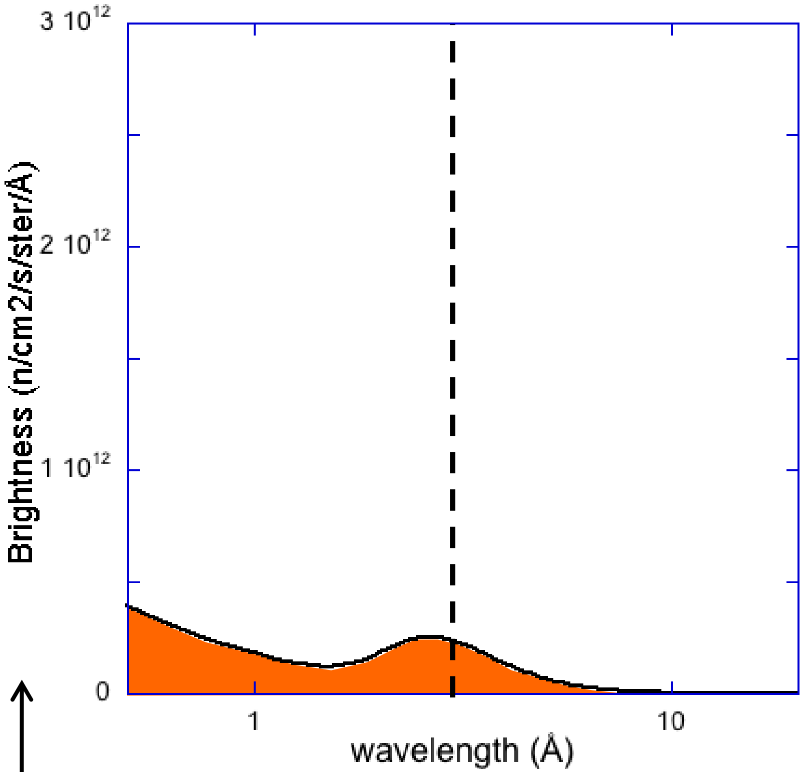
High reflection order contamination complicates analysis with CW data

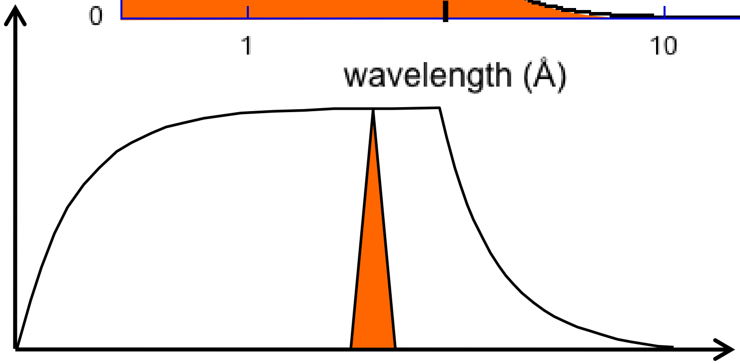
TOF method

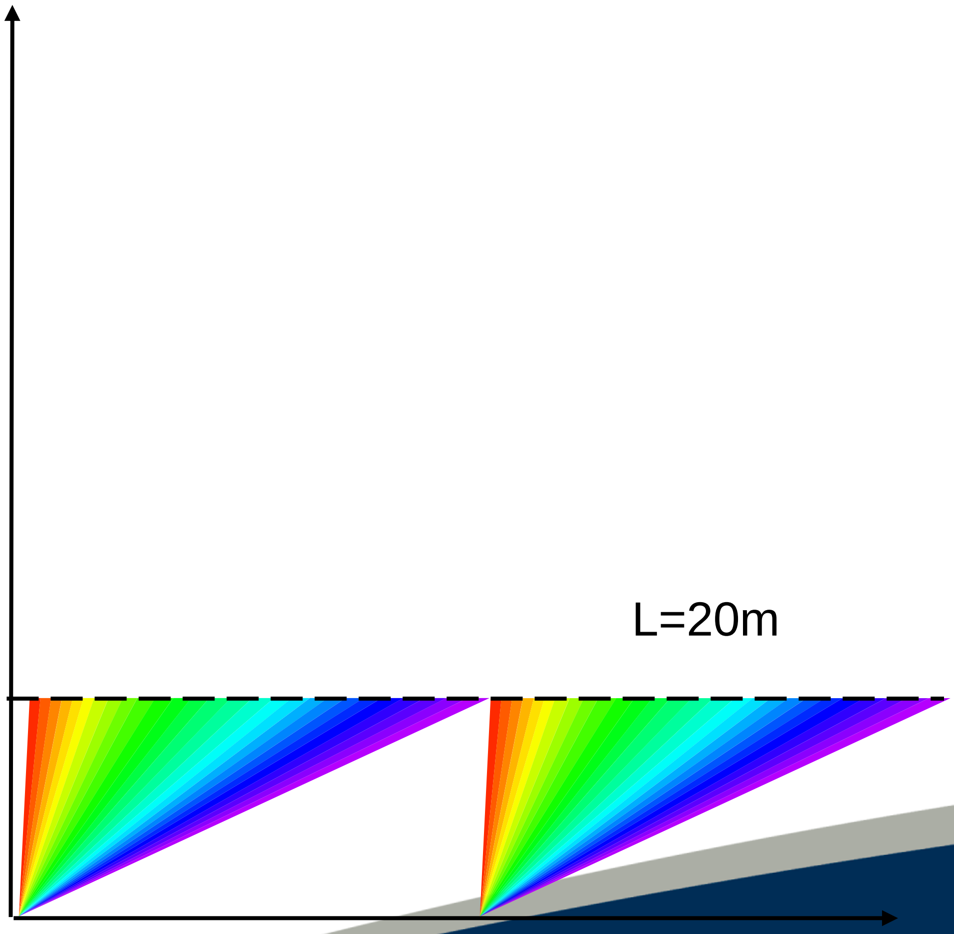
- Use distance to separate wavelengths
- Need choppers to prevent frame overlap
- Moderator pulse-width and distance determine resolution
- Source repetition rate determines available time-window
- Beam transport crucial



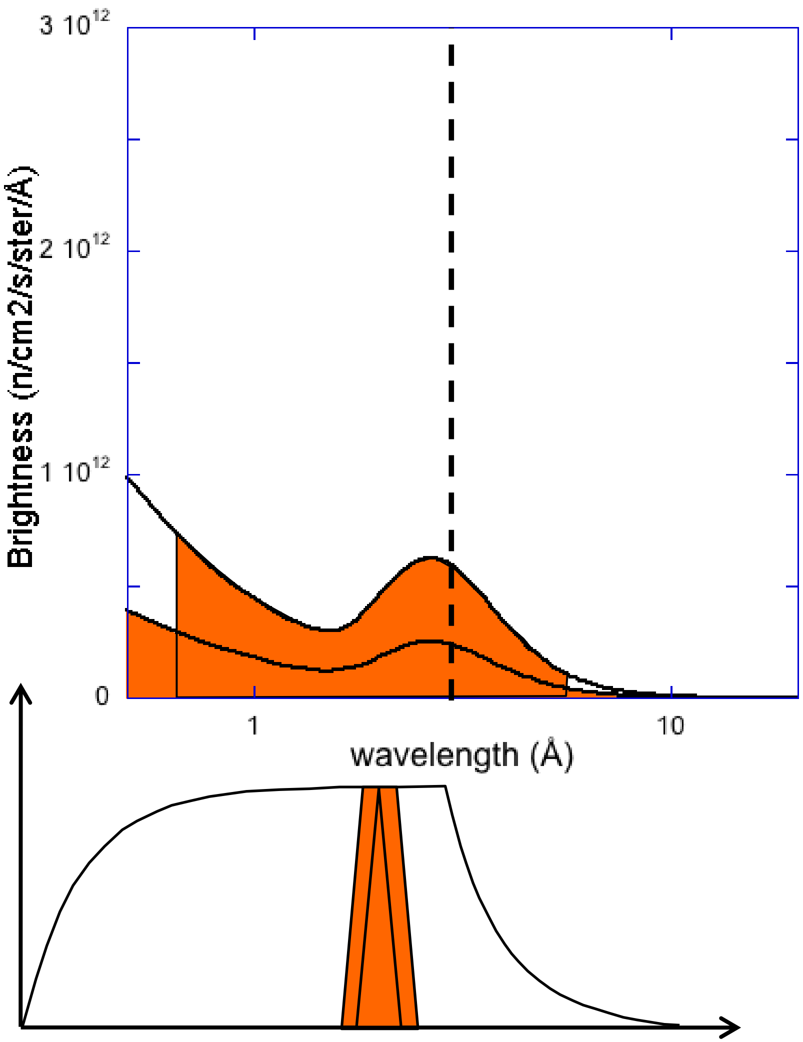
Q range with TOF: available bandwidth

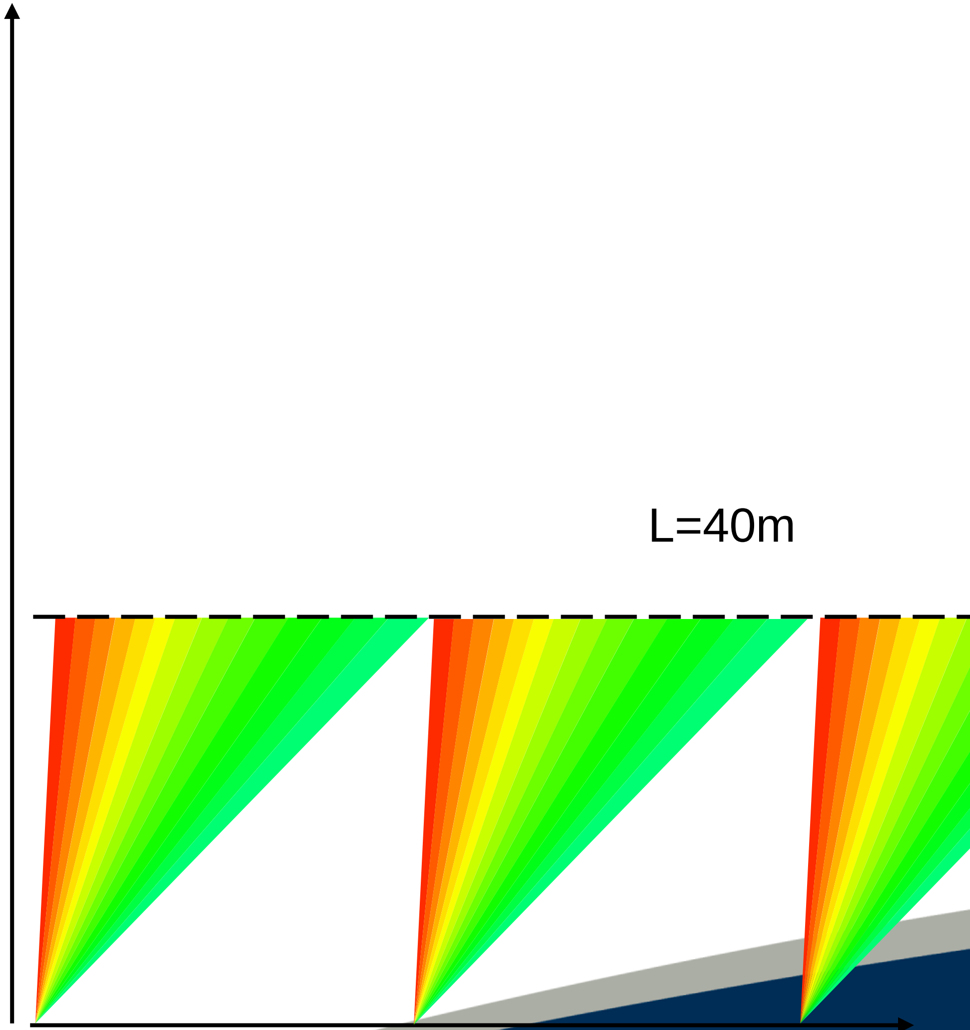






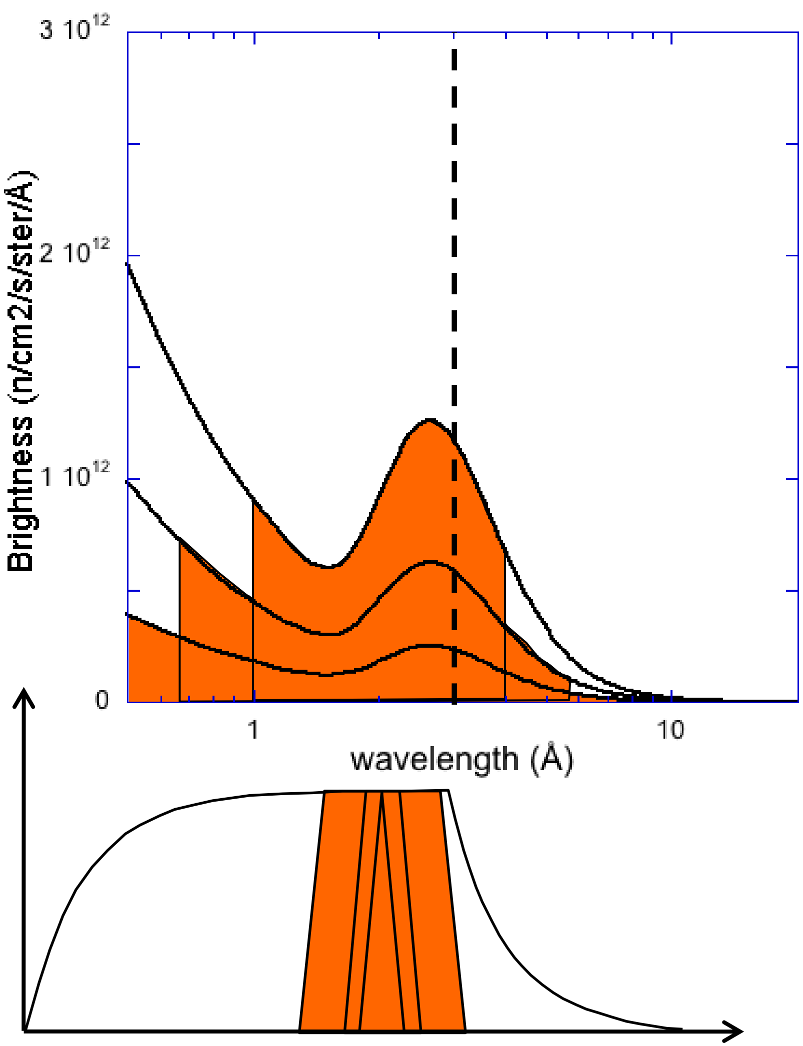
Q range with TOF: available bandwidth

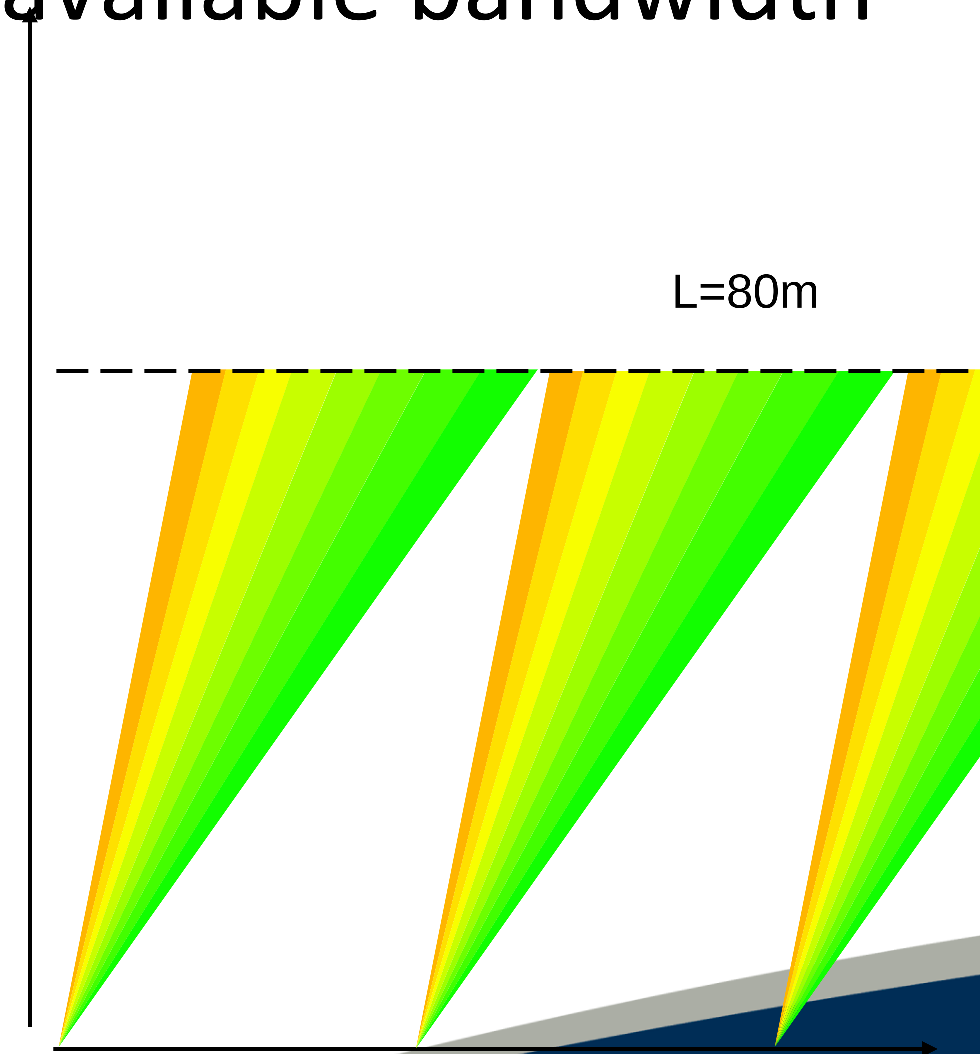




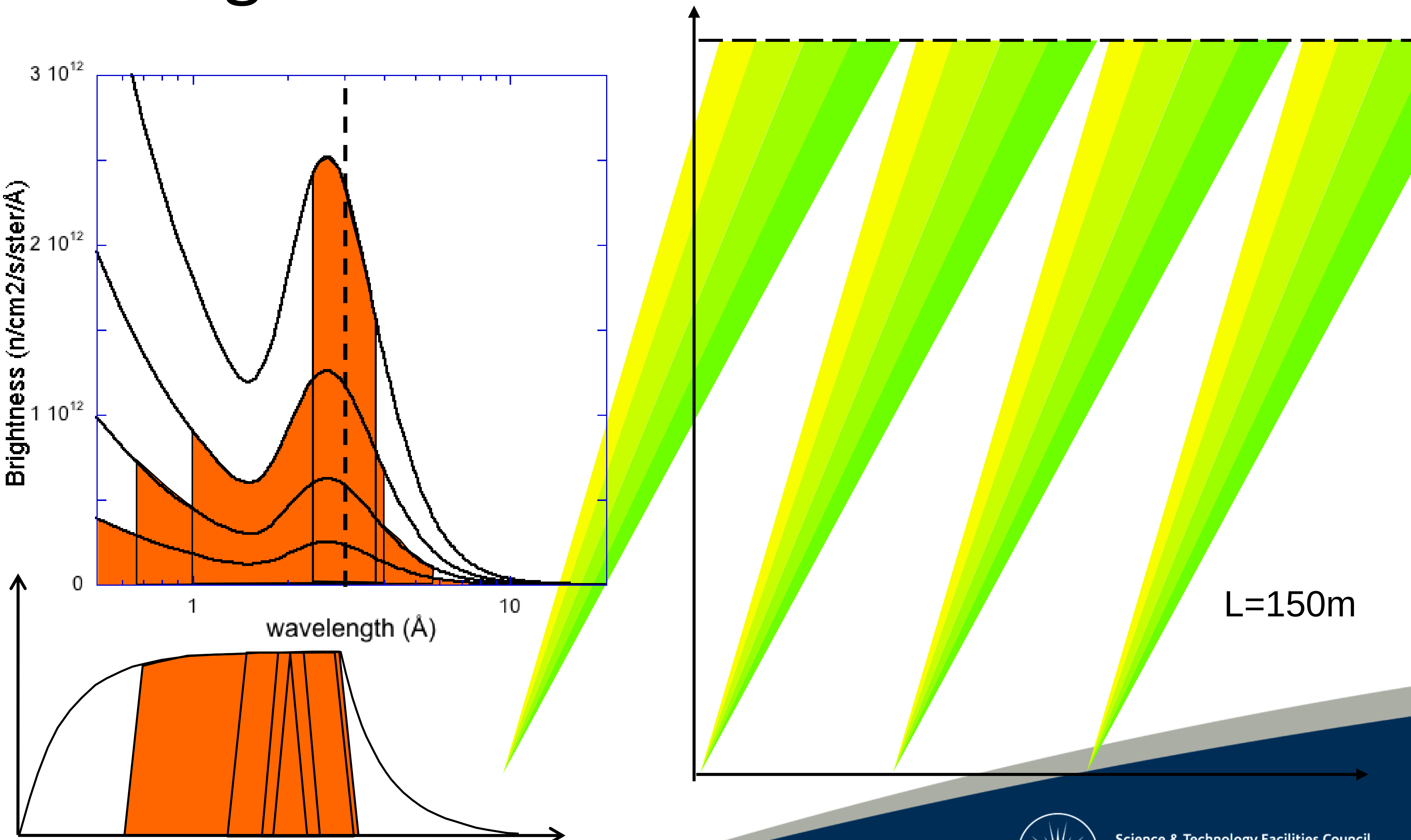
Science & Technology Facilities Council
ISIS

Q range with TOF: available bandwidth





Q range with TOF: available bandwidth



CW or TOF: Q-range summary

For CW:

- For monochromatic instruments the Q_{max} is $4\pi/\lambda$ i.e. when $\sin\theta = 1$, $\theta = 90°$, $2\theta = 180°$
- If a high Q_{max} is required a shorter wavelength must be used.
- Shorter wavelengths are produced by higher order hkl planes
- Reflectivity is lower for shorter wavelengths
- Realistic Q_{max} of around 25 $Å^{-1}$

For TOF:

- Q_{max} depends on λ_{min} and detector θ.
- λ_{min} can be much lower than for the CW case allowing $Q_{max} > 100$ $Å^{-1}$
- λ_{min} determined by the moderator, transport characteristics of the guide and which frame the instrument is working in

CW or TOF: Q resolution

Monochromatic

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

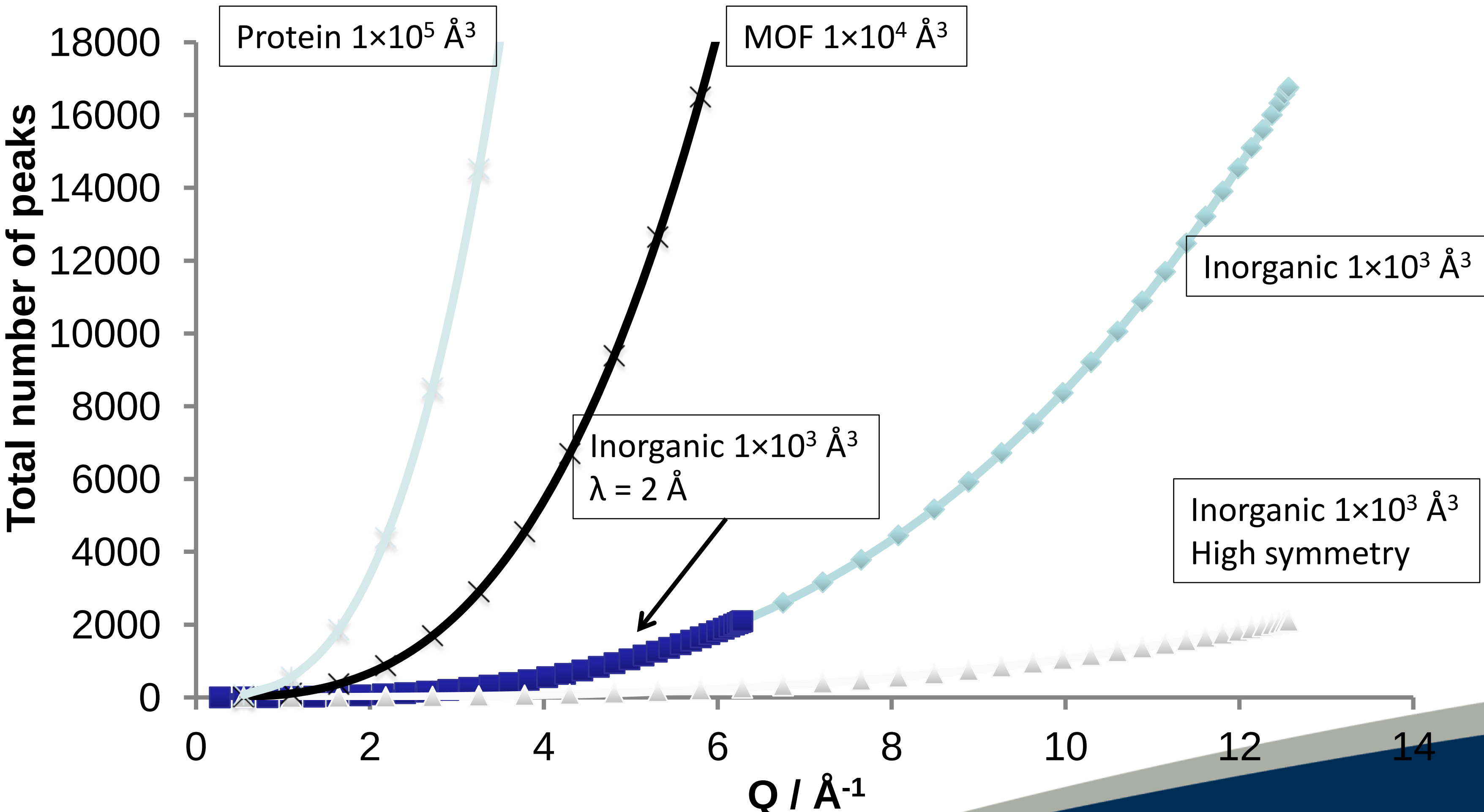
- U, V and W are functions of the collimation and U, V also takeoff angle to the monochromator
- Resolution minimum found near the takeoff angle of the monochromator $2\theta_M$
- Higher takeoff angle gives higher resolution for identical wavelength
- Wavelength produced by monochromator is takeoff angle dependent for any particular hkl plane

Time-of-flight

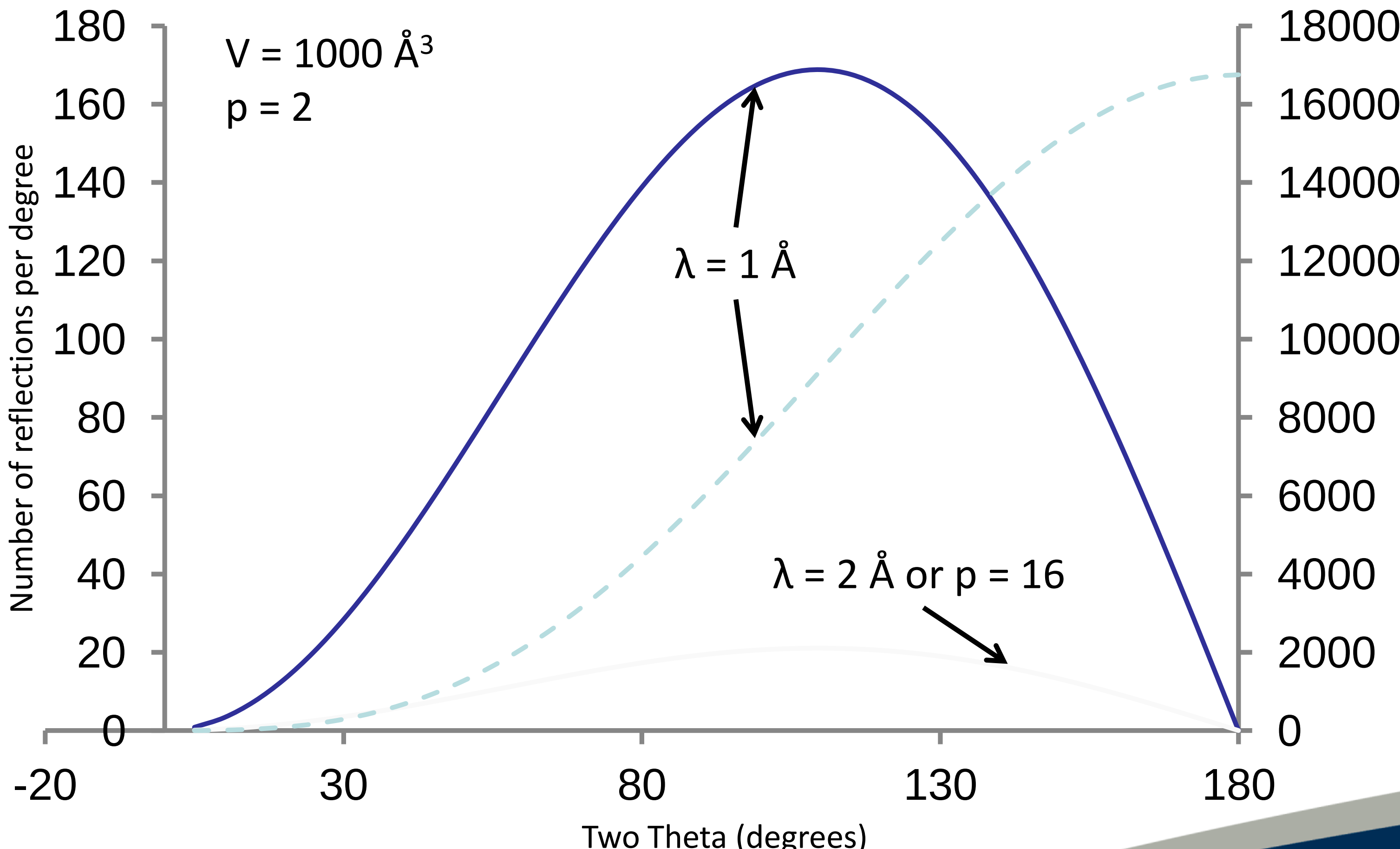
$$\Delta d/d = \left[\Delta\theta^2 \cot^2\theta + \left(\Delta t/t\right)^2 + \left(\Delta L/L\right)^2\right]^{1/2}$$

- Δθ is the angular uncertainty
- The main component of Δt is the moderation time of the neutron
- ΔL is the flight path uncertainty of the neutron mainly due to the finite width of the moderator
- First term can be minimised by moving to higher scattering angle
- Second and third terms minimised by increasing instrument length

Number of possible reflections



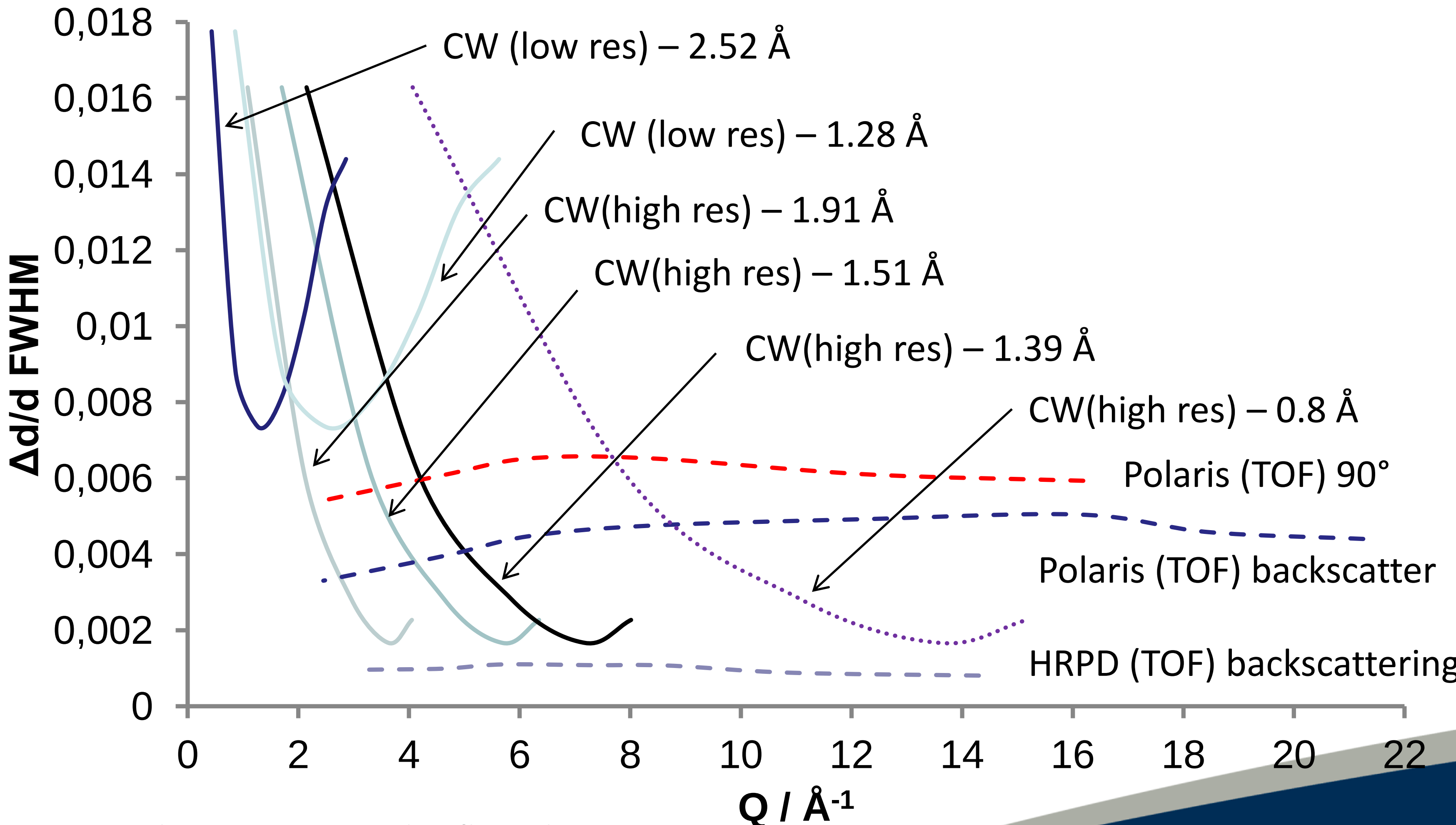
Reflection density for CW



CW instruments designed to have best resolution at highest peak density

Science & Technology Facilities Council ISIS

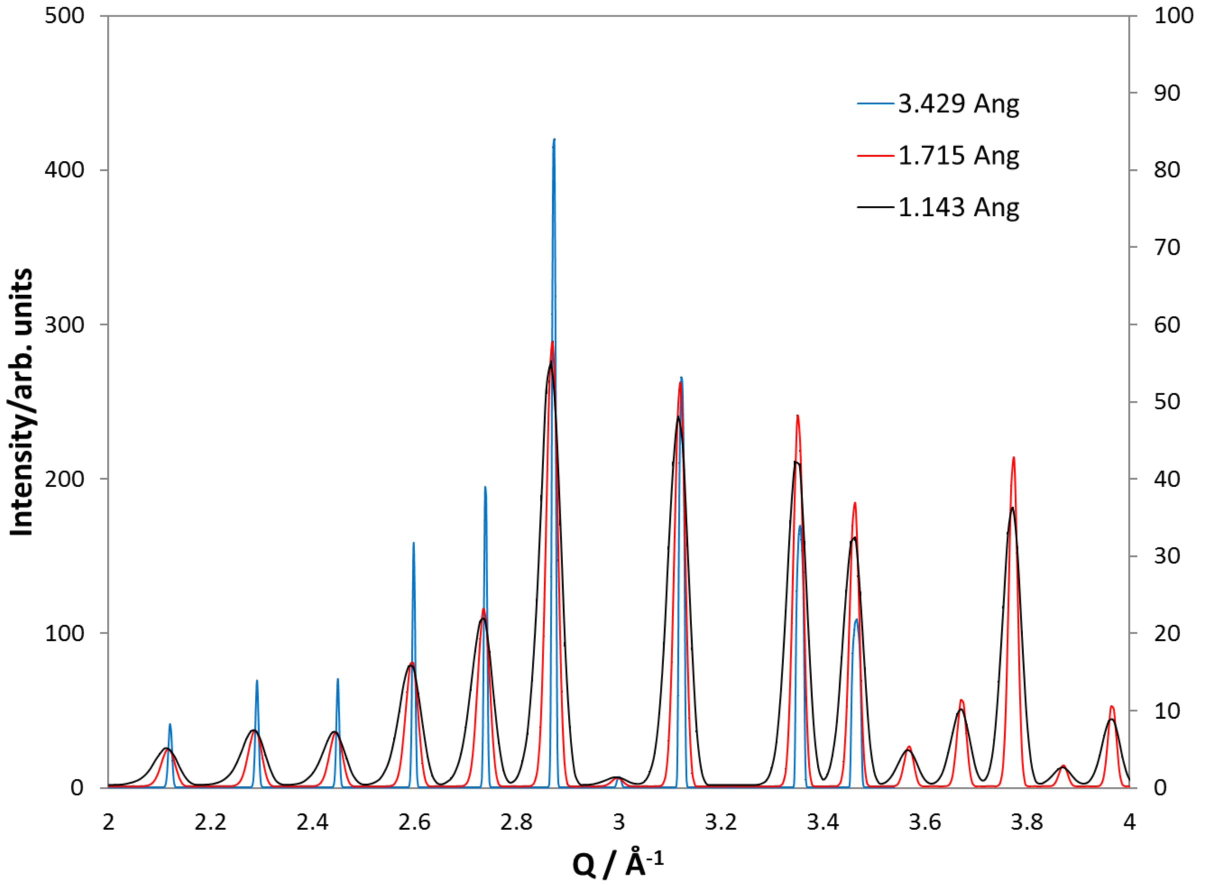
Resolution functions CW v TOF



TOF Q-resolution tends to be flat, change at high Q caused by moderator residence time

Science & Technology Facilities Council ISIS

CW Q resolution example



Choose wavelength to match Q resolution required by science in a given Q range

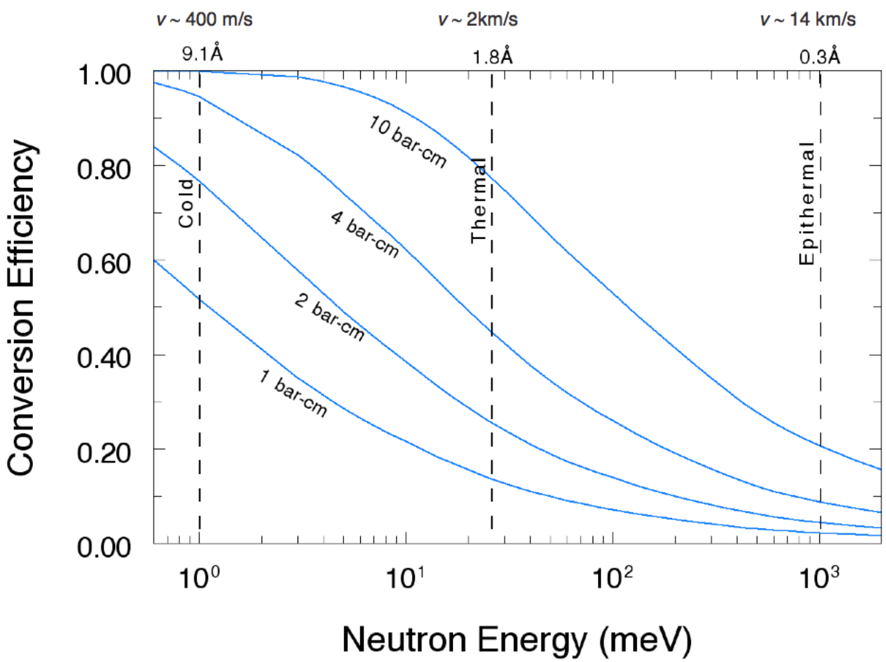
Science & Technology Facilities Council ISIS

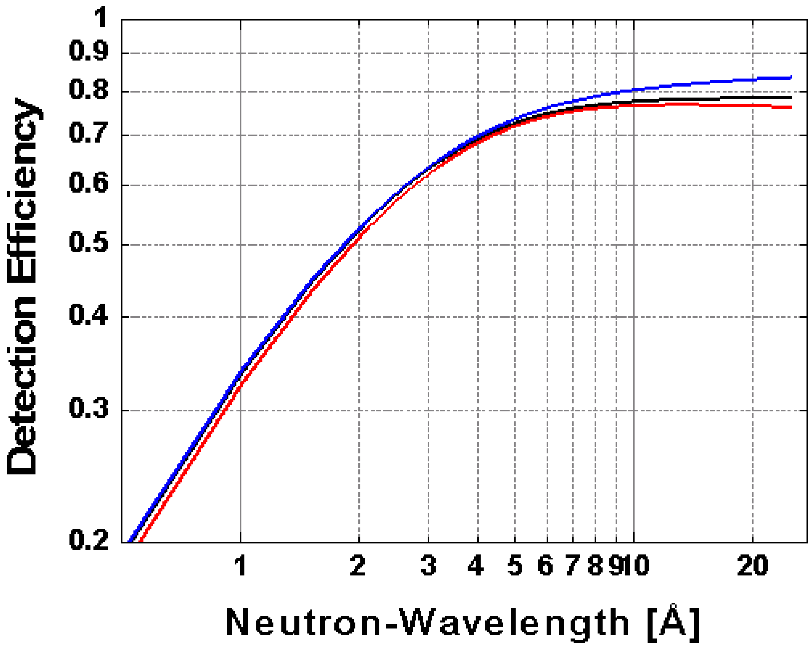
CW or TOF: Q resolution

- CW:
  - Simple, symmetric peakshape function
  - Best resolution where diffraction peak density is highest in scattering angle
  - Different wavelength can be used to give Q resolution where required
  - Different takeoff angle can be used to change resolution function and wavelength
  - Instrument can be high Q resolution but with very limited Q range

- TOF:
  - Complex asymmetric peakshape related to moderator characteristics
  - Instrument length and moderator give wavelength band and overall resolution
  - Q resolution almost constant for a given detector bank so increasing peak density with Q can be an issue
  - Q resolution improved by moving to higher scattering angle detector bank
  - Q range determined by scattering angle of detector bank

Detection efficiency





^3He detection efficiency as a function of detection depth. (from Radeka, Neutrons & photon detector workshop, 2012)

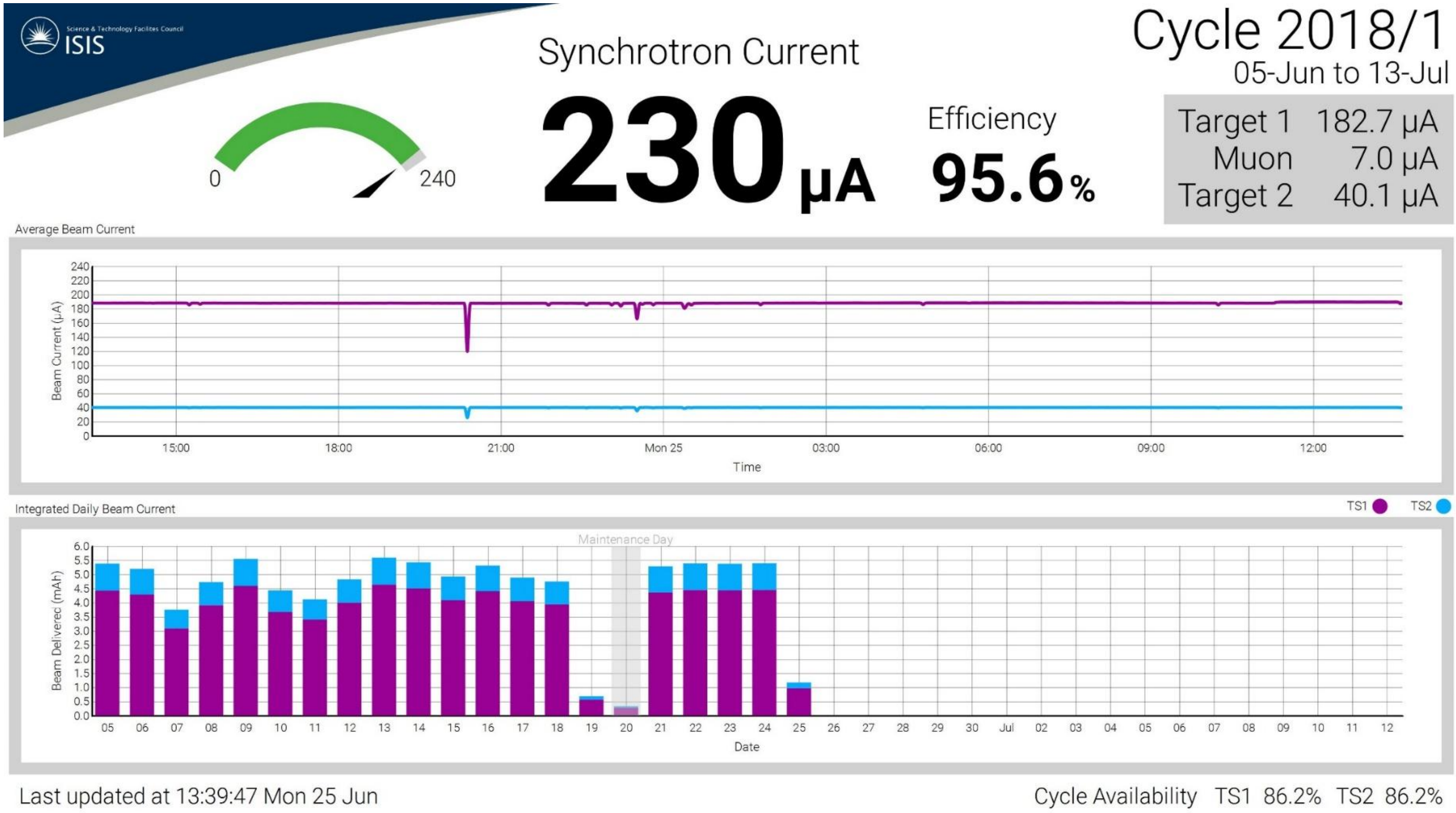
https://portal.slac.stanford.edu/sites/conf_public/nxd2012/presentations/VR_Neutron%20gas%20dets_Aug1_2012.pdf

Predicted detector efficiency CASCADE-detector for 20 ^{10}B layers

http://www.physi.uni-heidelberg.de/Forschung/ANP/Cascade/Projekt/results.php?lang=en

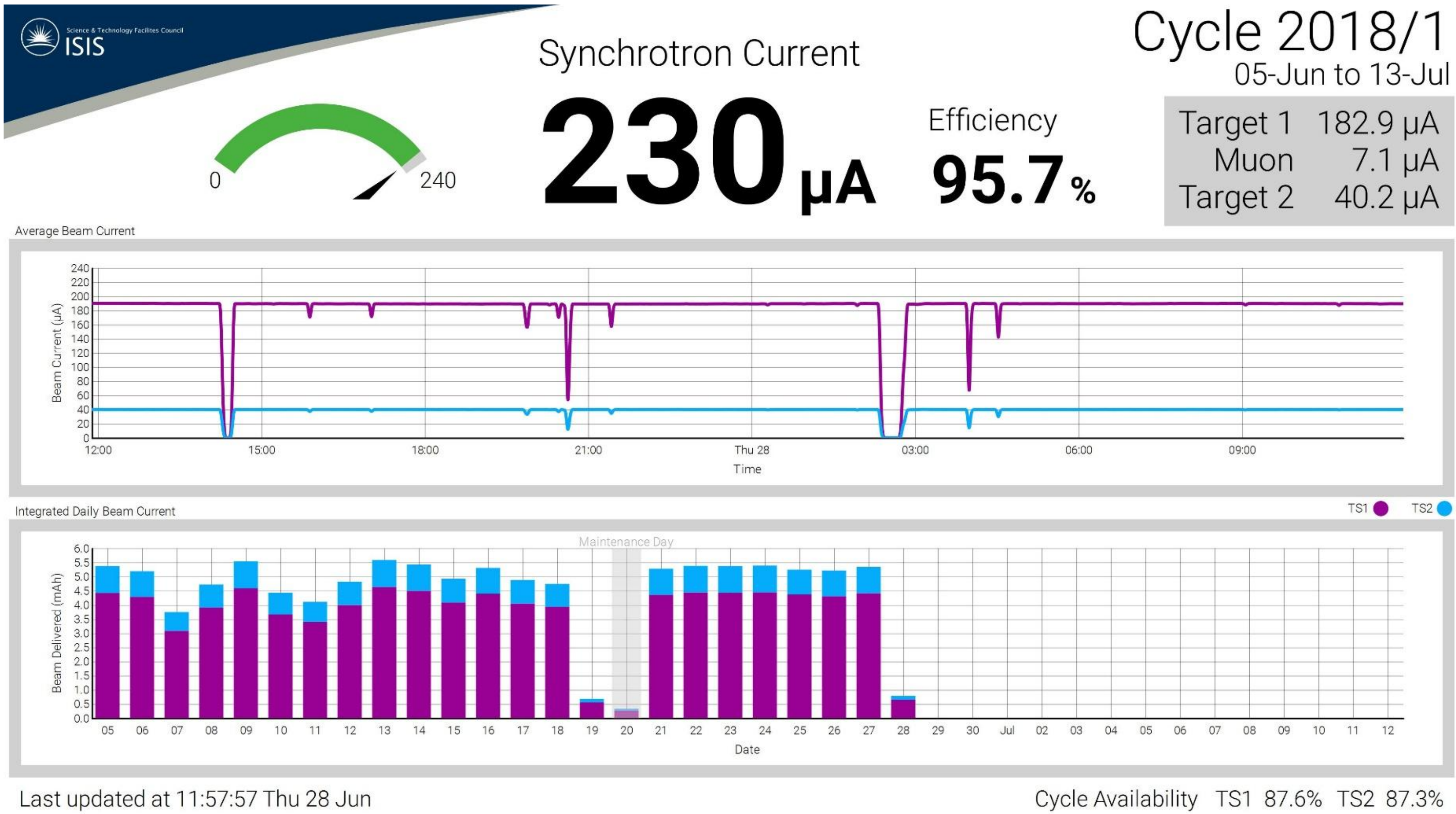
Science & Technology Facilities Council ISIS

Pulsed source availability



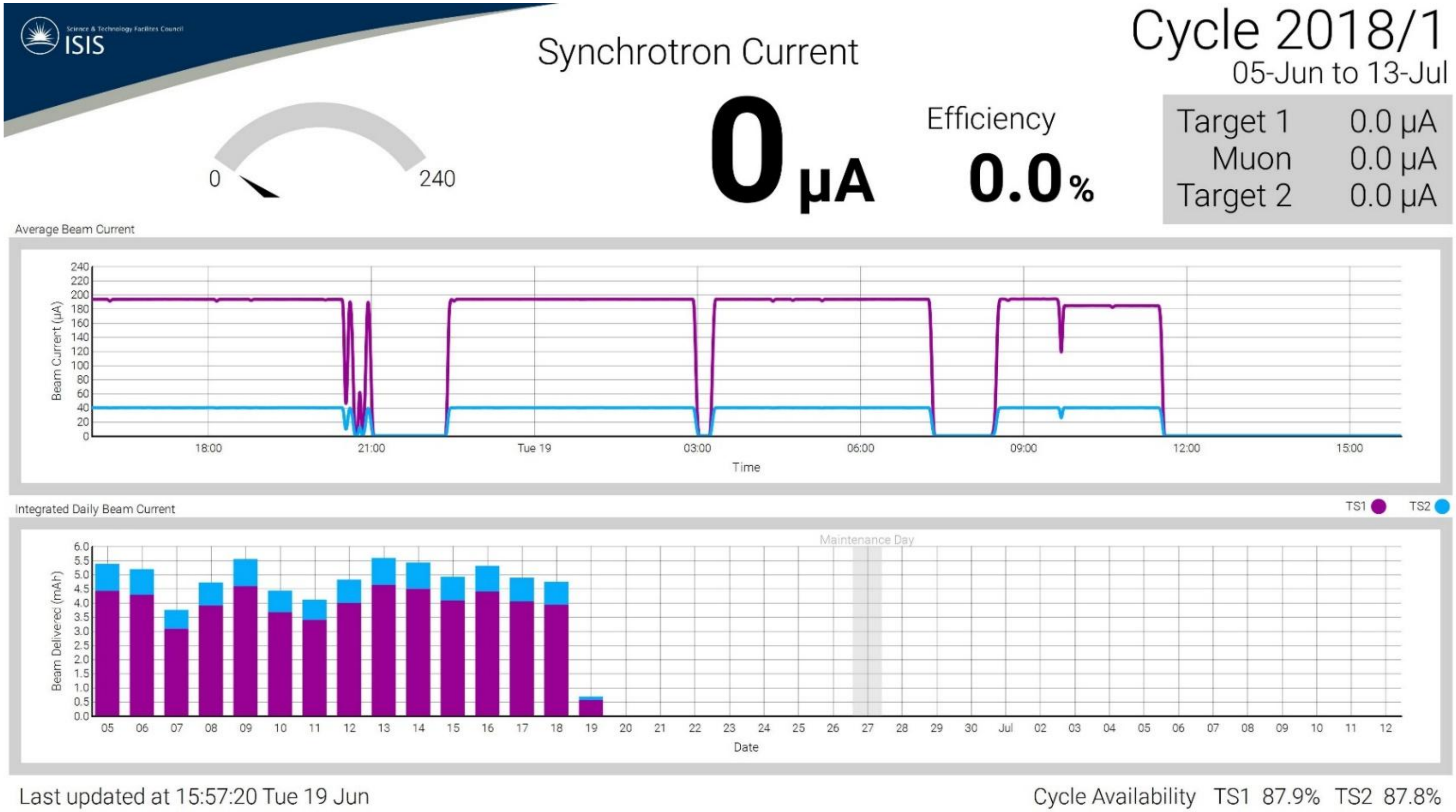
A very good day in terms of beam
All experiment types possible

Science & Technology Facilities Council ISIS



A good day in terms of beam – still possible issues with *in situ* and time resolved experiments

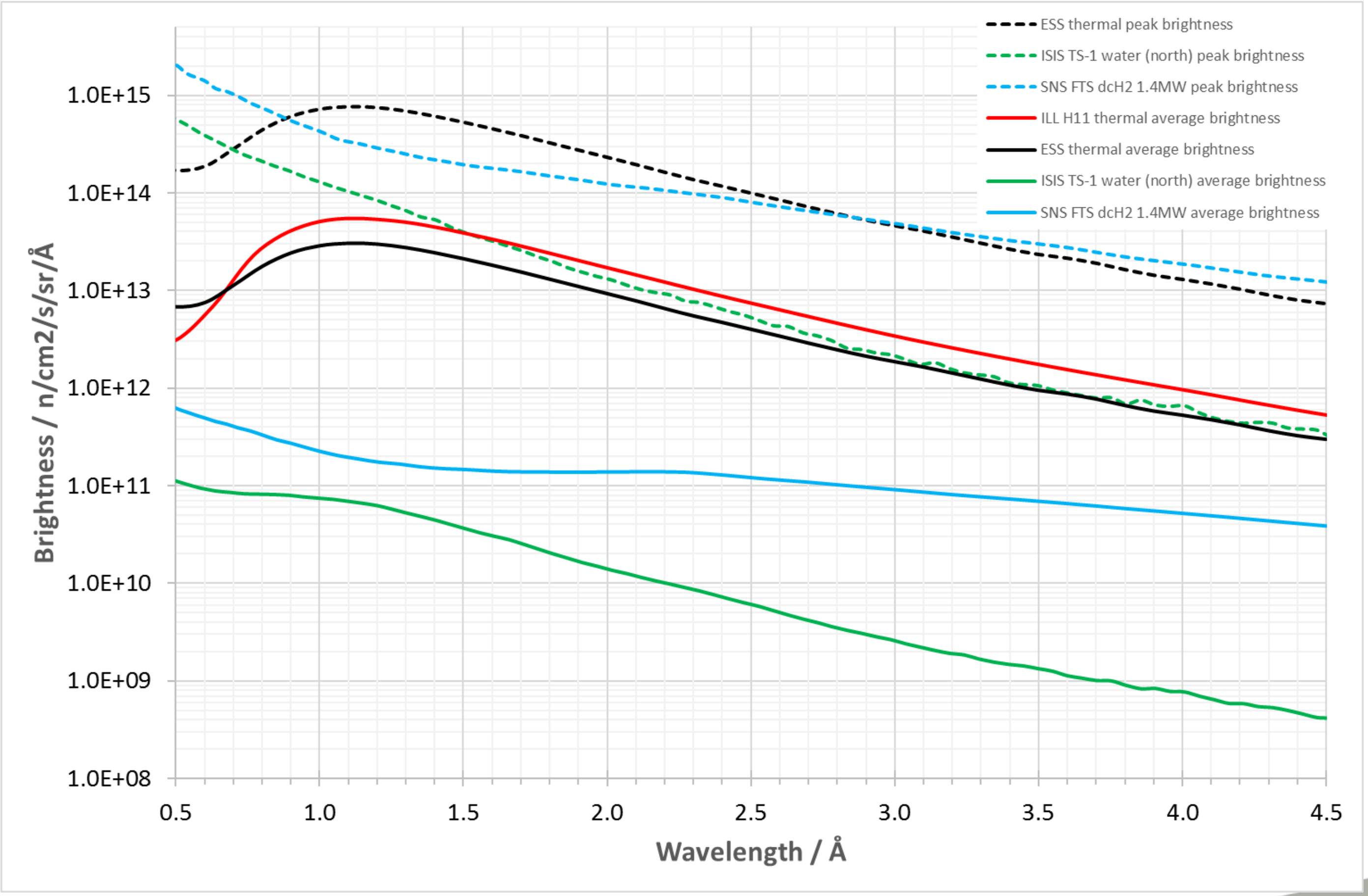
Pulsed source availability



A bad day – any time resolved experiment is compromised

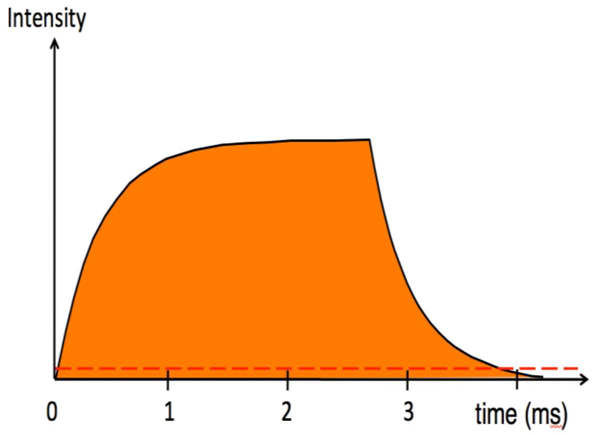
Science & Technology Facilities Council ISIS

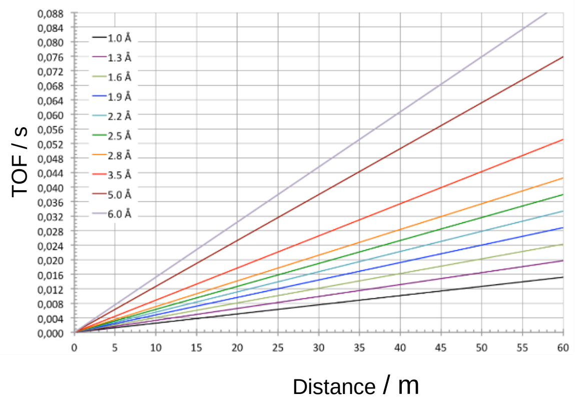
CW at a long pulse source: ESS

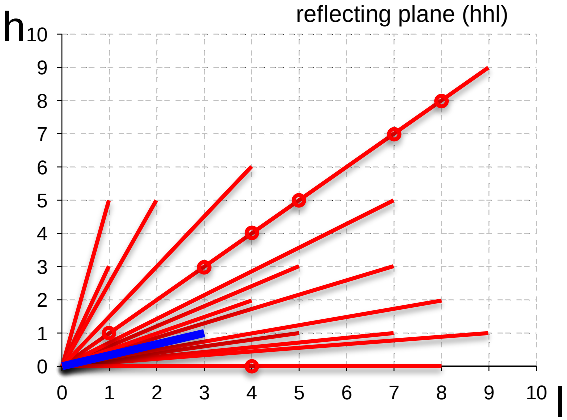


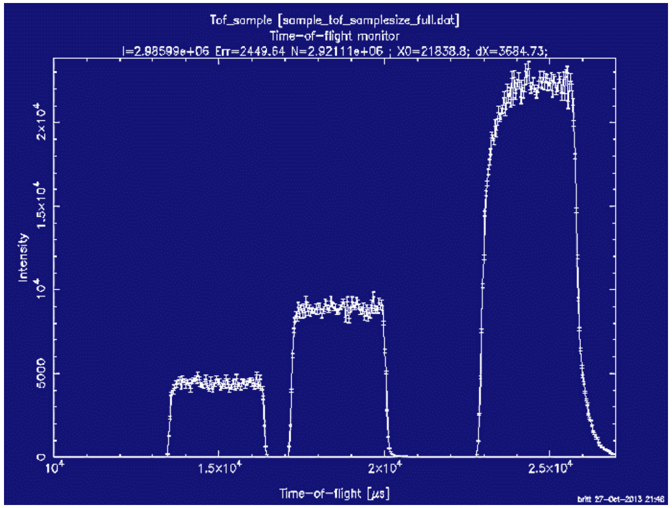
High peak brilliance good for TOF but also
High time-average brilliance good for CW

CW at a long pulse source: ESS





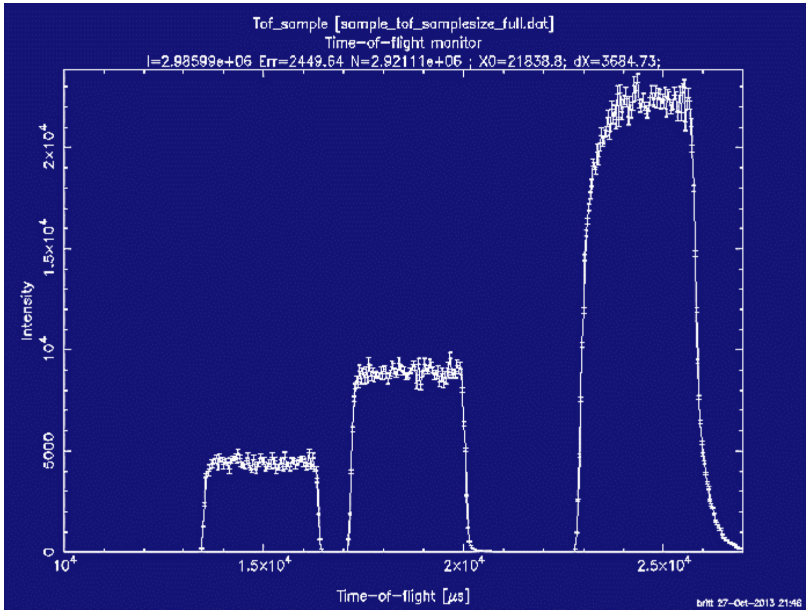


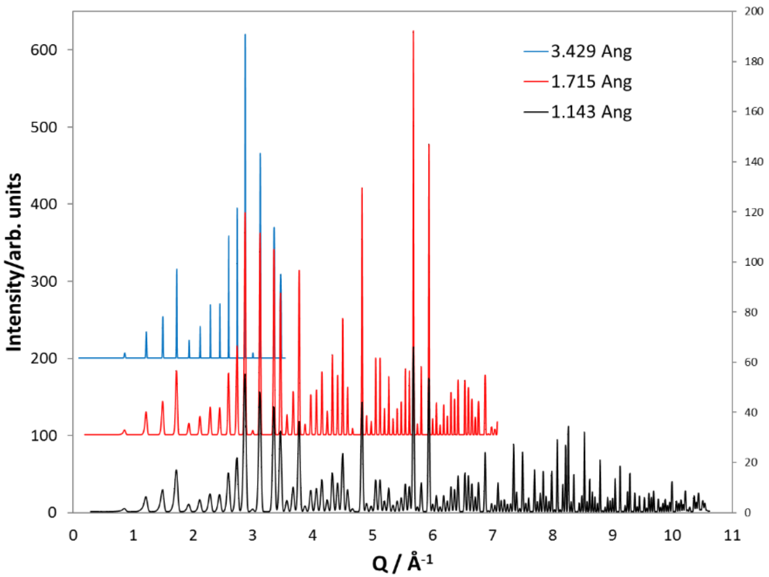


- Use distance to separate higher order monochromator reflections in TOF at the detector
- Develop new monochromator materials
- Access wider Q range
- Tune Q resolution

Science & Technology Facilities Council
ISIS

CW at a long pulse source: ESS





- Combine several current CW instrument capabilities in one simultaneous measurement
- No wavelength contamination
- Lower instrument background
- New science possibilities

Science & Technology Facilities Council
ISIS

Summary: Instrument types

- Reactors build CW instruments*
  - Low peak brilliance, high time-average brilliance
  - Variable reflectivity from monochromators limit low λ use
  - High Q not easily reached
  - Match moderator and monochromator take-off angle to Q range and resolution
  - Beam always on

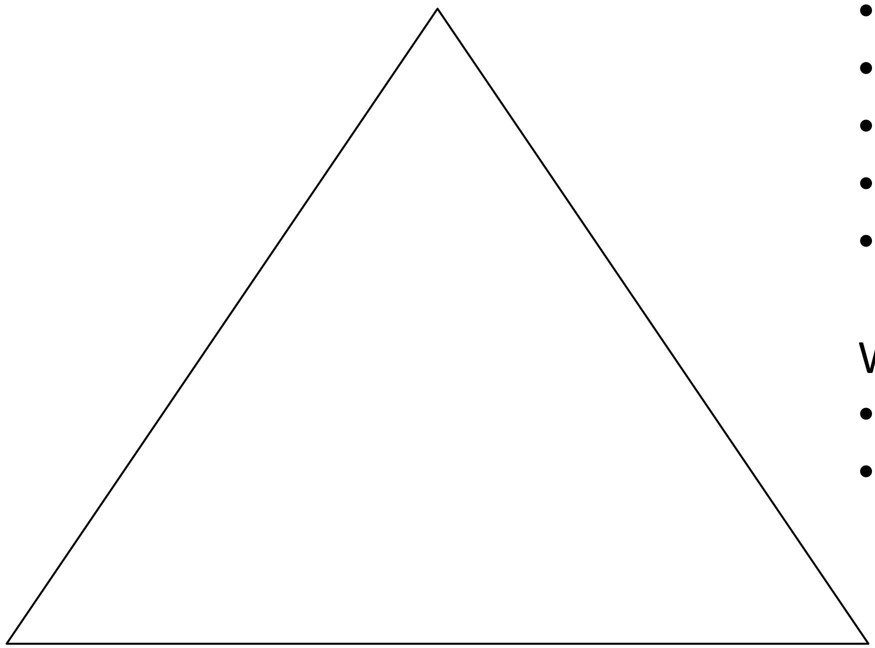
*Except when significantly restricted geometry constraints from science case necessitate use of TOF

- Pulsed sources build TOF instruments#
  - High peak brilliance, low time-averaged brilliance
  - Require efficient beam transport
  - High Q possible
  - Increase instrument length to improve resolution at expense of bandwidth
  - Variable Q range and resolution from detector angles
  - Beam availability can compromise science

#Remains to be seen for long pulse sources

Science & Technology Facilities Council
ISIS

Why so many diffractometers?



But also:

- Unit cell volume
- Sample environment restrictions
- Need for *in situ* capability
- Sample size
- Sample state
- Etc…

Which come from:

- Science case requirements
- Available budget!

Single crystal or powder?

Depends on the scientific problem:

- Unambiguous structure determination – single crystal
  - Beware extinction and absorption issues
- In situ studies – powders
  - Generally the only practical option
- Fast measurements – powders
  - Larger samples
- High background materials (such as incoherent scattering) – single crystal
  - BUT is possible with powder
- Multi-component systems investigations – powder
- Structural phase transitions – powder
  - Crystals tend to shatter
- Real systems – powders
- Very small samples – single crystal
  - Can become difficult to get a powder average

CW or TOF?

Depends on the scientific problem:

- Structure refinement – either but TOF preferred as complexity increases
- *In situ*, time resolved studies – CW
- Parametric studies – either but CW probably faster for T, P, B mapping
- Fast measurements – either (flux for CW, detector coverage for TOF)
- Small samples – either (lowest background instrument)
- PDF studies – TOF to access high Q
- Magnetic structure – CW preferred but TOF catching up
- Polarised neutron work – traditionally CW but TOF developing
- Hydrogenous materials – CW still preferred but TOF developing
- Large unit cells – either source type, but will be Laue methods (quasi-TOF)
- High pressure – TOF offers wider Q range, CW higher flux
- Engineering applications – either, pick depending on Q range
- Texture – either

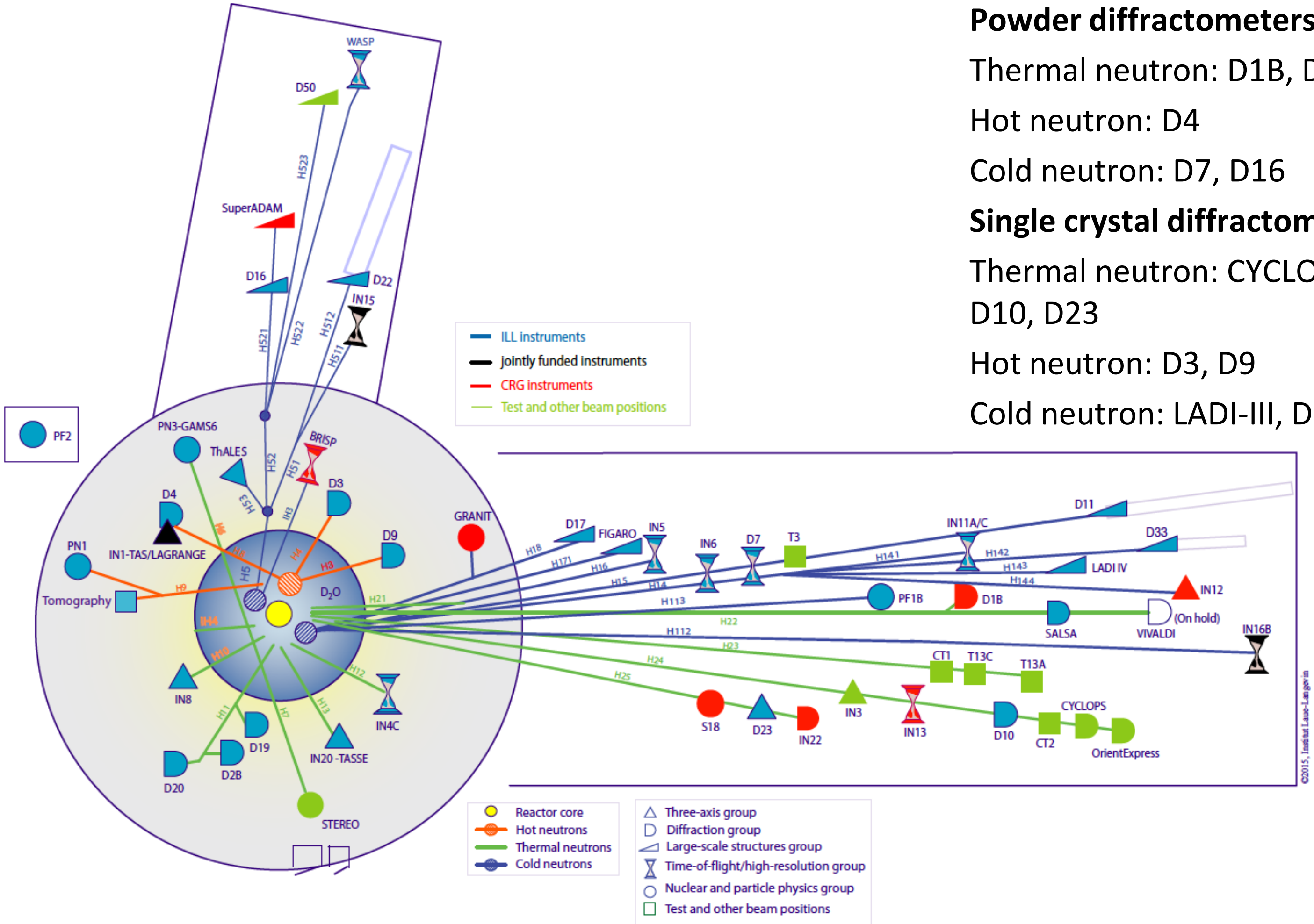
Instrument examples

- Earliest diffractometers
- Continuous wavelength powder (thermal)
  - High resolution powder
  - High flux powder
  - Variable resolution powder
- Time-of-flight powder
  - High resolution powder
  - Medium resolution powder (×2)
  - Specialised powder
- Single crystal diffractometers

Earliest CW diffractometer

Science & Technology Facilities Council
ISIS

Diffraction instruments at a reactor: ILL



Powder diffractometers

Thermal neutron: D1B, D2B, D20, SALSA

Hot neutron: D4

Cold neutron: D7, D16

Single crystal diffractometers

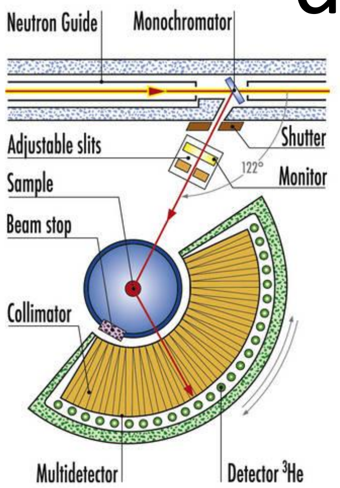
Thermal neutron: CYCLOPS, OrientExpress, D19, D10, D23

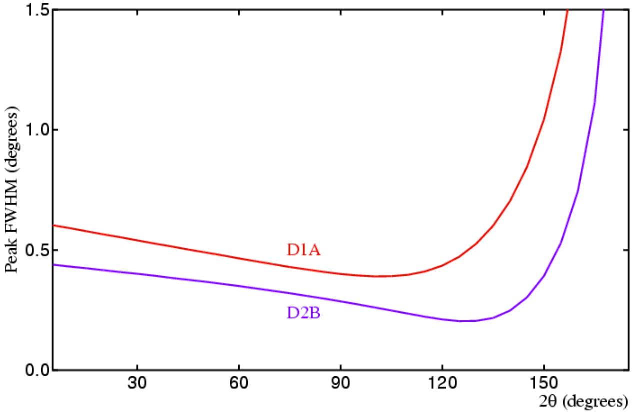
Hot neutron: D3, D9

Cold neutron: LADI-III, D7

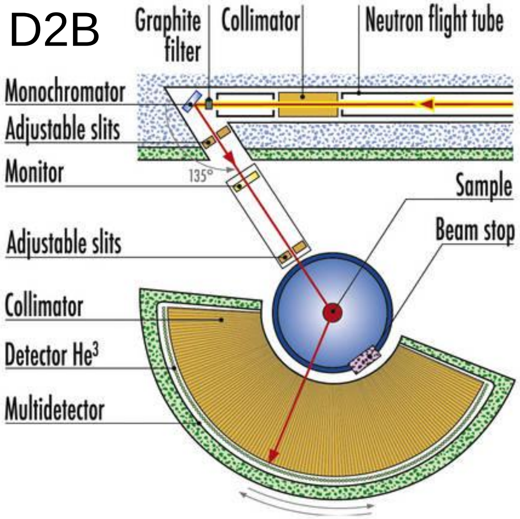
CW, High resolution, thermal powder diffractometers: D1A, D2B

D1A





D2B



Science: Inorganics, small molecule, magnetism,
Q range up to about 12 $Å^{-1}$

http://www.ill.eu/instruments-support/instruments-groups/

CW, High resolution powder diffractometers: D1A, D2B

Instrument	D1A	D2B
Takeoff angle / °	122	135
Flux / n cm^{-2} s^{-1}	10^6	10^6 to 10^7
Beam (h × w) / mm	30 × 20	50 × 20
Detectors	25 ^3He × 10 cm h	128 ^3He × 30 cm h
Wavelengths	Ge(hhl)	Ge(hhl)
Δd/d Resolution	2-3 × 10^{-3}	Min 5 × 10^{-4}
Background	Very Low (60 m)	Low (15 m)
Average data collection time	3-24 hrs	0.25-4 hrs

Similar instruments at all continuous sources:
Echidna (ANSTO), Spodi (FRM-II), BT-1 (NIST), 3T2 (LLB), HB-2A (HFIR) etc…

http://www.ill.eu/instruments-support/instruments-groups/

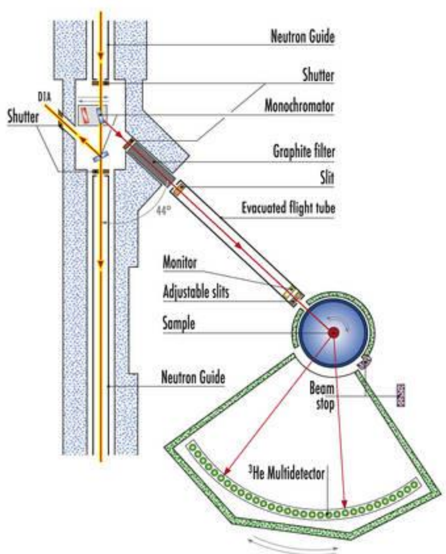
Science & Technology Facilities Council ISIS

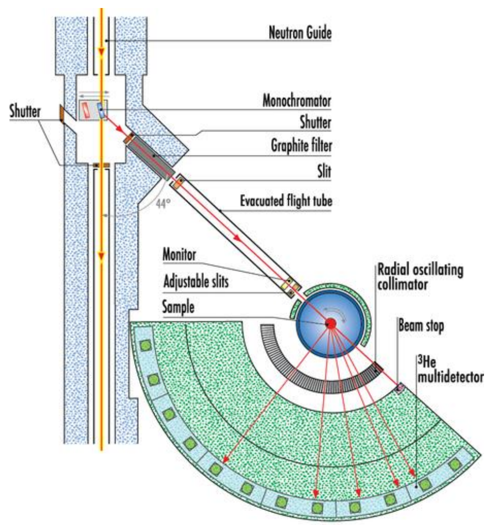
CW, High flux, thermal powder diffractometers: D1B, D20

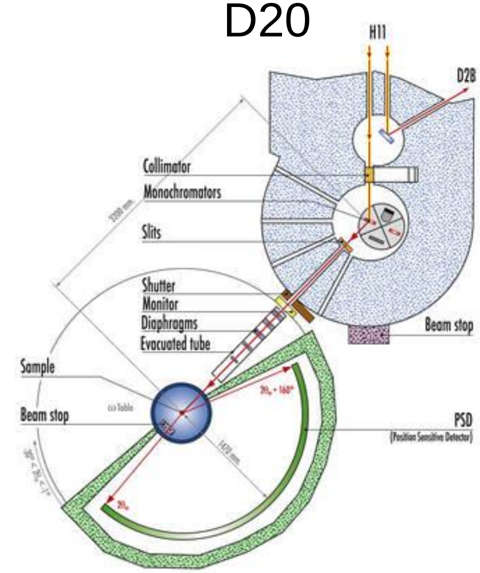
D1B

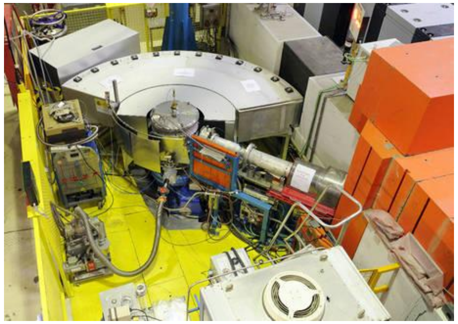
newD1B

D20









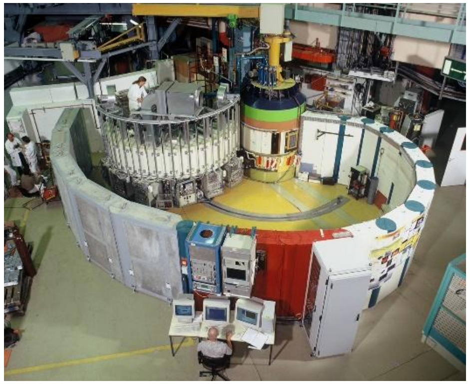
CW, High flux, thermal powder diffractometers: D1B, D20

Instrument	D1B (old) / D1B (new)	D20
Takeoff angle / °	44	28, 42 (±2)
Flux / n cm^{-2} s^{-1}	6.5×10^6 HOPG(002) 0.4×10^6 Ge(311)	4.2×10^7 HOPG(002) 9.8×10^7 Cu(200) 42° 3.2×10^7 Cu(200) 28°
Beam (h × w) / mm	50 × 20	50 × 20
Detectors	80° multi-wire / 128° multi-wire 0.2° separation / 0.1° separation 400 channels / 1280 channels	153.6° micro-strip detector 0.1° separation 1536 channels
Wavelengths / Å	2.52, 1.28	2.42, 1.30, 0.87
Δd/d Resolution	$> 1 \times 10^{-2}$	$> 1 \times 10^{-2}$
Background	Medium (low with ROC)	Medium/High (low with ROC)
Average data collection time	5-10 mins / 1-5 mins	<1 min

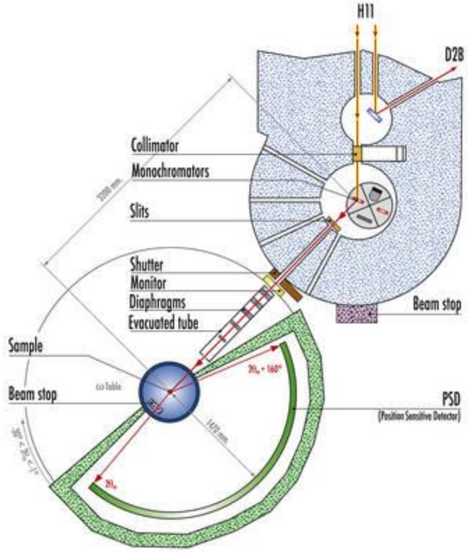
Fewer comparable instruments: Wombat (ANSTO), G4.1 (LLB), HB-2C (HFIR)

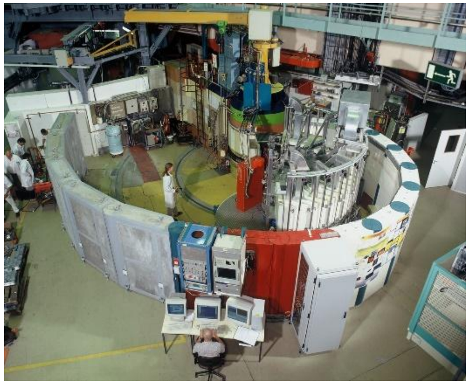
Science & Technology Facilities Council
ISIS

CW, variable resolution, thermal powder diffractometer: D20

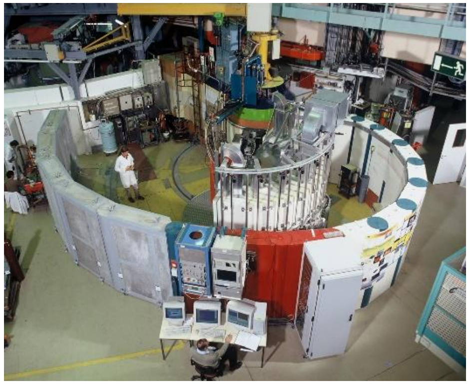


120°

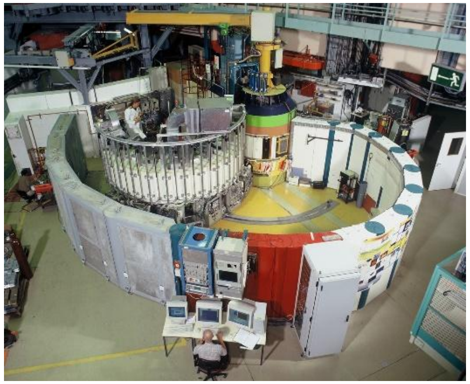




28°



42°



90°

65°

Photos courtesy T. Hansen ILL

Science & Technology Facilities Council
ISIS

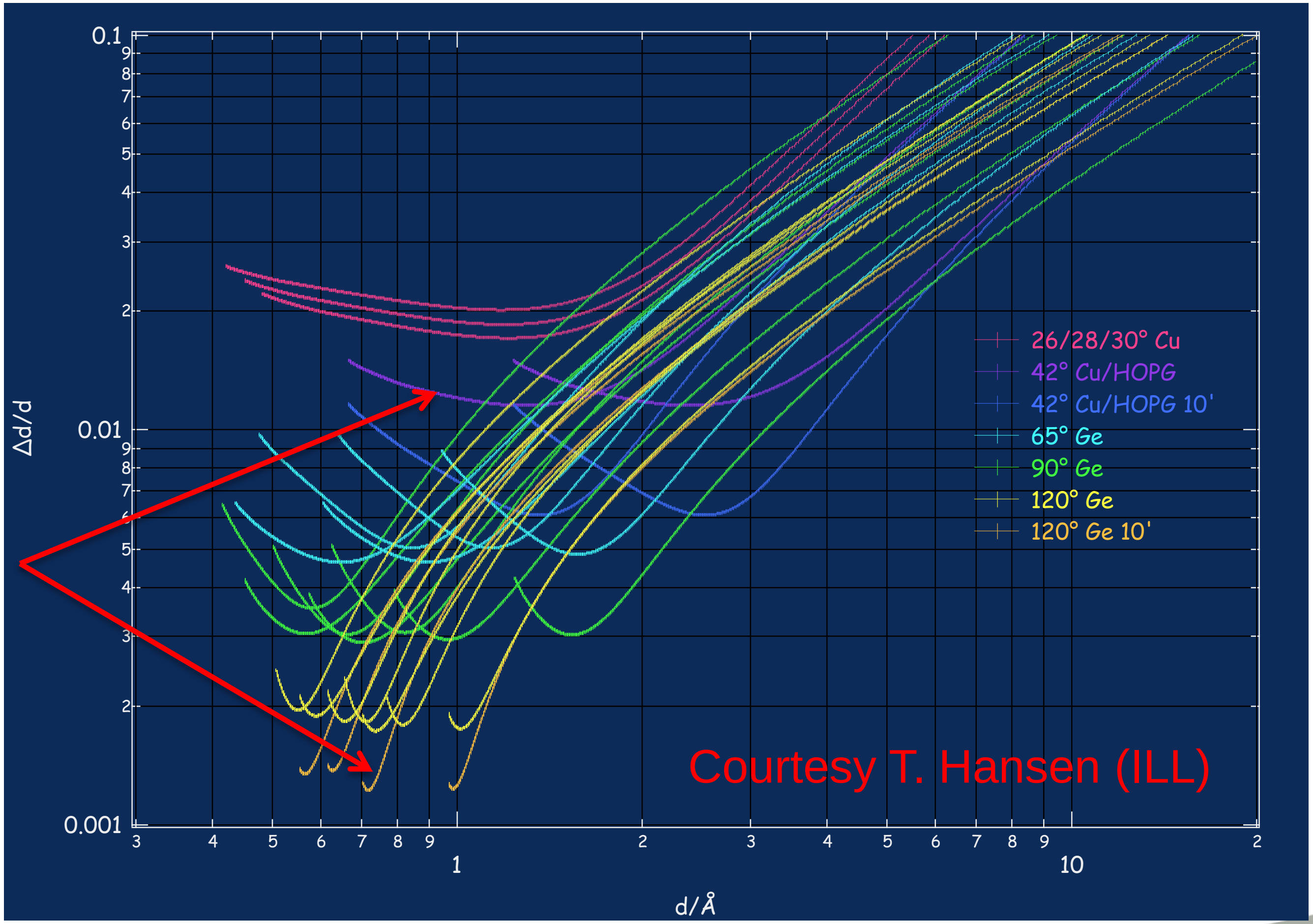
CW, variable resolution, thermal powder diffractometer: D20

Instrument	D20 (high flux)	D20 (high takeoff angle)
Takeoff angle / °	28, 42 (±2)	65, 90, 120 (±2)
Flux / n cm^{-2} s^{-1}	4.2×10^7 HOPG(002) 9.8×10^7 Cu(200) 42° 3.2×10^7 Cu(200) 28°	8.0×10^6 Ge(115) 7.5×10^6 Ge(117) 4.0×10^6 Ge(119)
Beam (h × w) / mm	50 × 20	50 × 20
Detectors	153.6° micro-strip detector 0.1° separation 1536 channels	153.6° micro-strip detector 0.1° separation 1536 channels
Wavelengths / Å	2.42, 1.30, 0.87	variable 0.8-3 Ge(hhl/00l/hhh)
Δd/d Resolution	$> 1 \times 10^{-2}$	See next slide
Background	Medium/High (low with ROC)	Medium/High (low with ROC)
Average data collection time	<1 min	5-15 mins (30 times faster than similar counting statistics on D2B)

Even fewer contemporary instruments: HRPT (PSI), Wombat (ANSTO has potential)

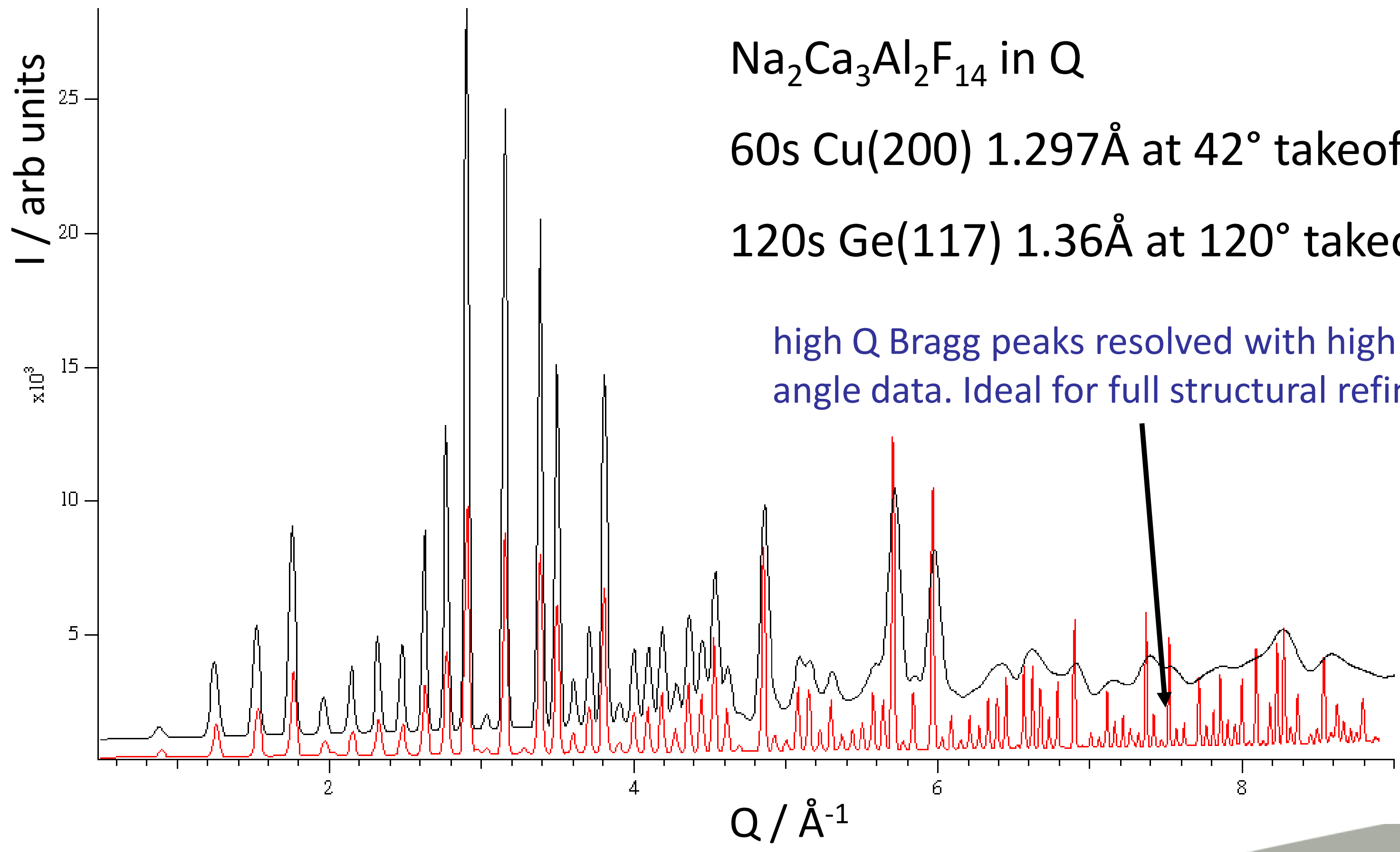
Science & Technology Facilities Council ISIS

Tune resolution using θ_B



compare

Low θ_B v high θ_B: Q resolution v count-rate



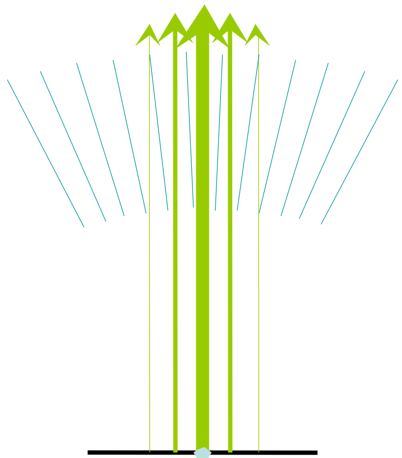
$Na_2Ca_3Al_2F_{14}$ in Q

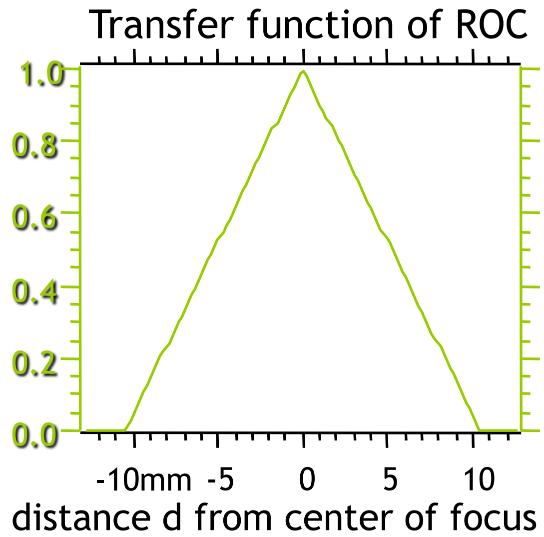
60s Cu(200) 1.297Å at 42° takeoff (black)

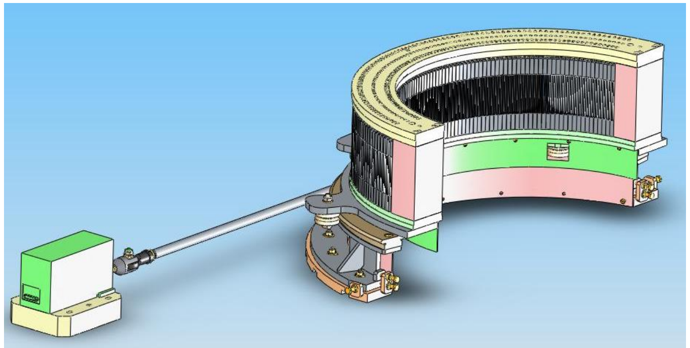
120s Ge(117) 1.36Å at 120° takeoff (red)

high Q Bragg peaks resolved with high take off angle data. Ideal for full structural refinement

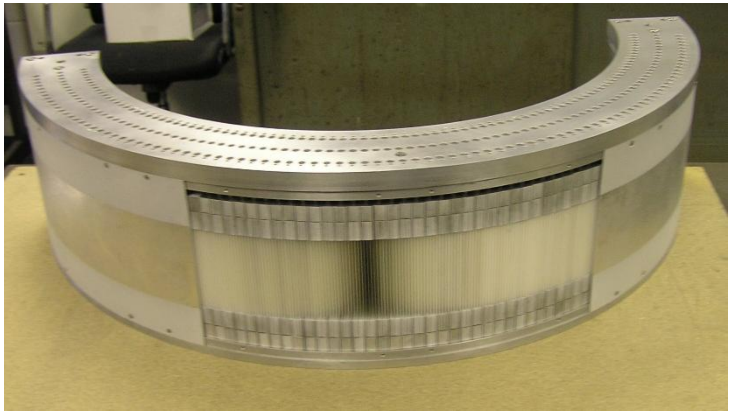
Parasitic scattering on instruments with area detectors: D20 collimator

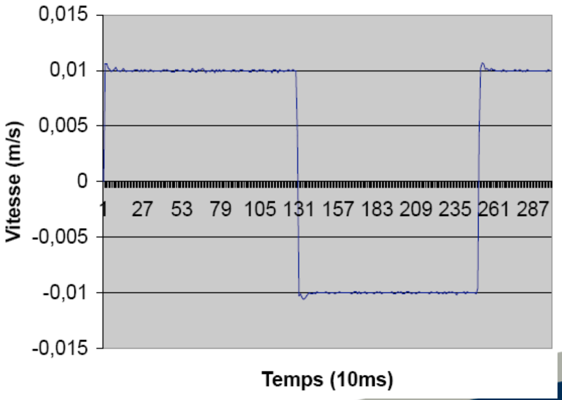




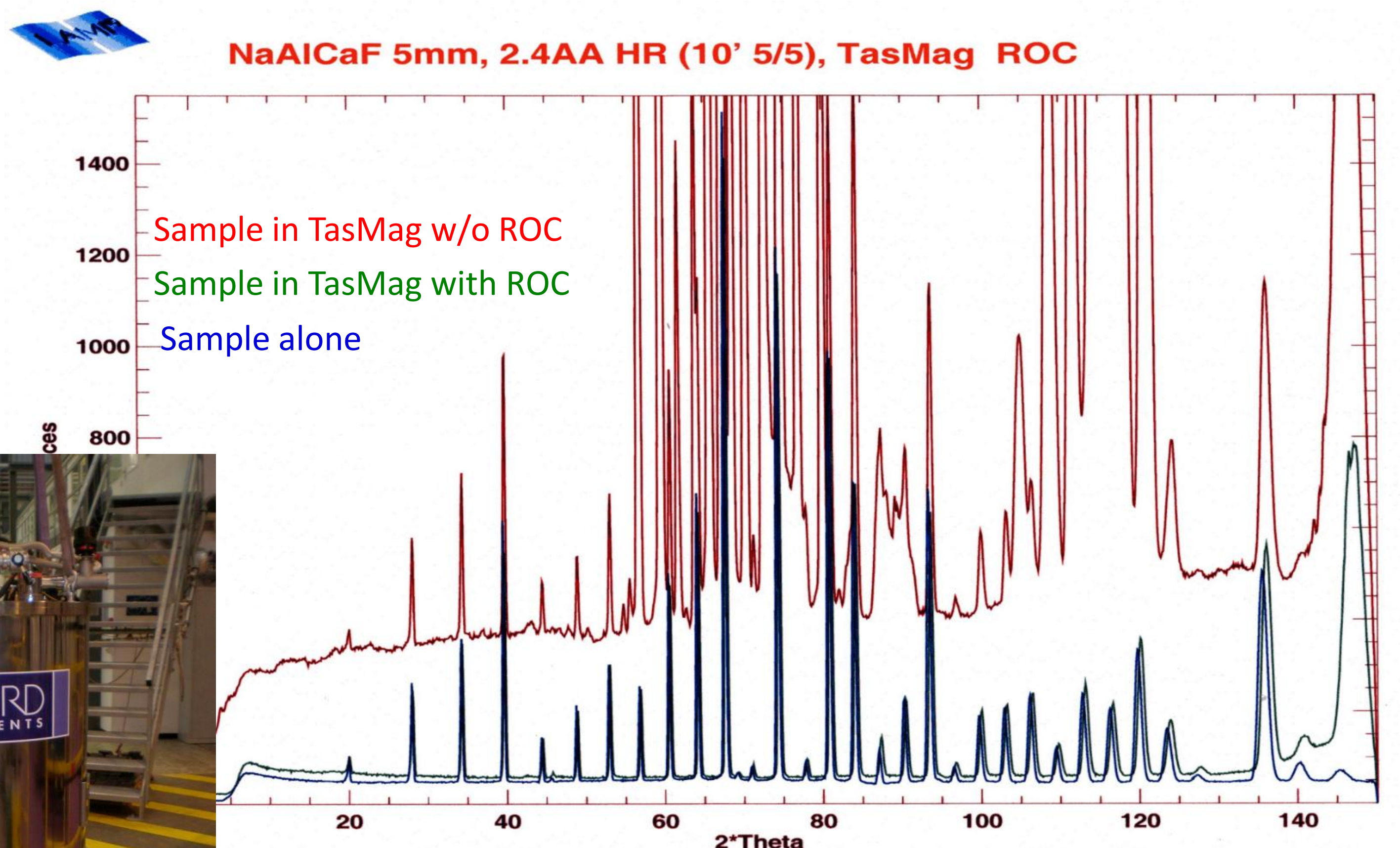


Photos Courtesy ILL

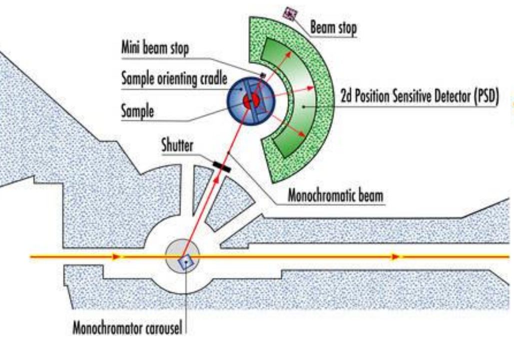




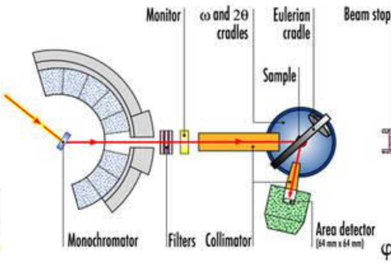
Parasitic scattering on instruments with area detectors: D20 example



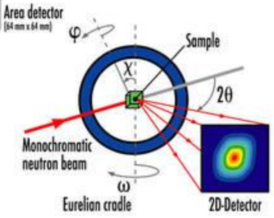
CW single crystal diffraction

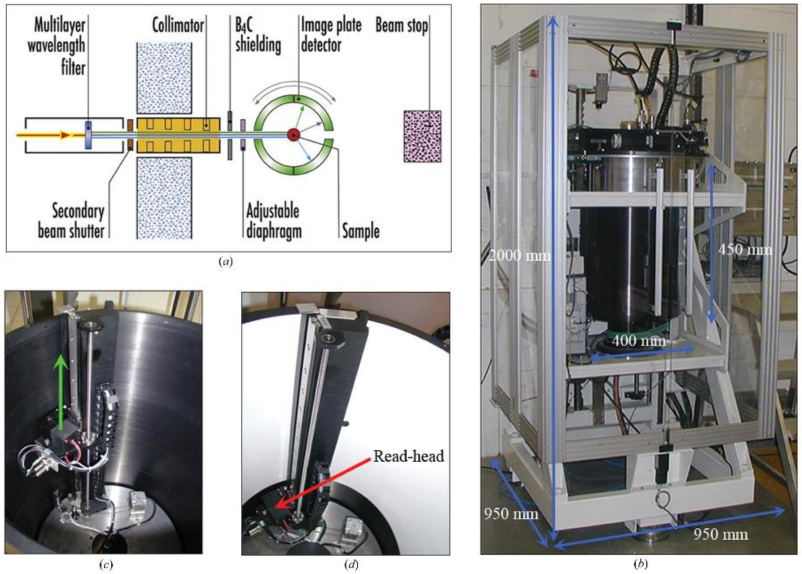


D19



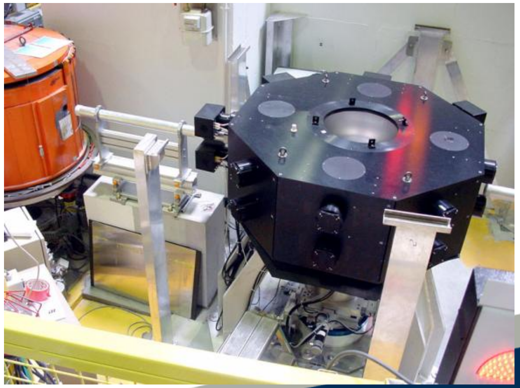
D9





↑

LADI-III



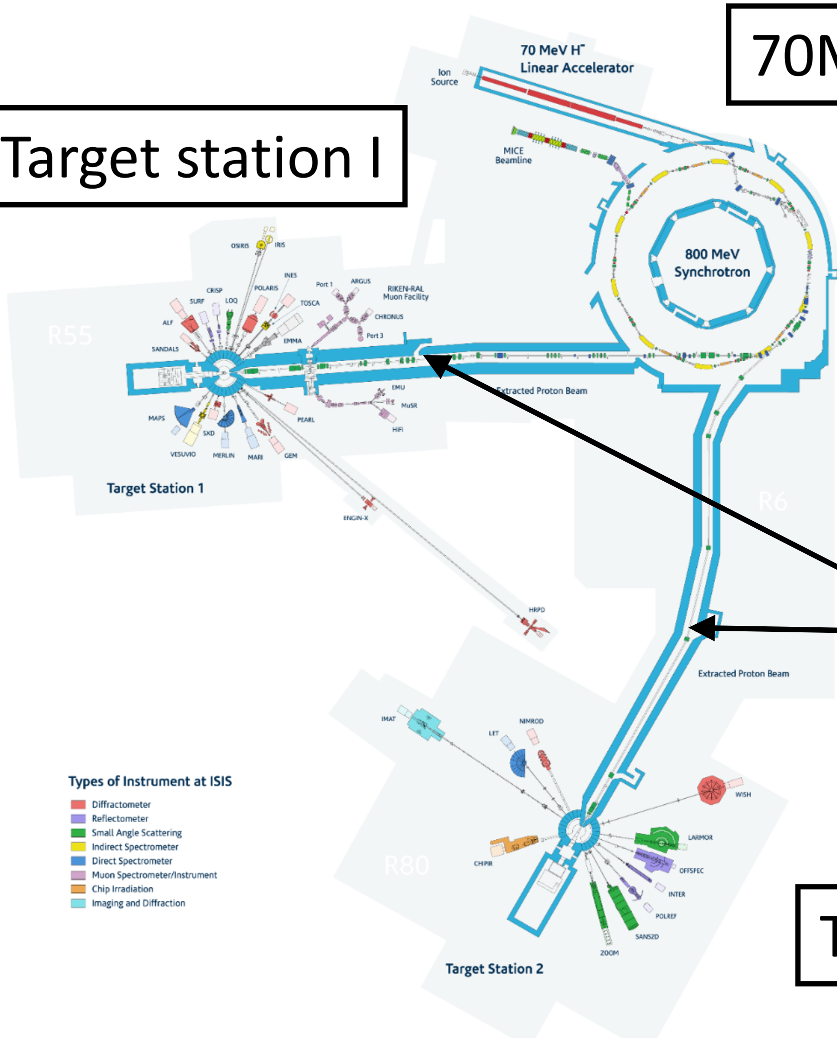
CYCLOPS

- Monochromatic and Laue type instruments are represented
- Q-range of interest and unit cell volume determine whether hot, thermal or cold neutron spectrum required for both instrument types

http://www.ill.eu/instruments-support/instruments-groups/

Science & Technology Facilities Council
ISIS

Pulsed source: ISIS



70MeV H^- Linac

Target station I

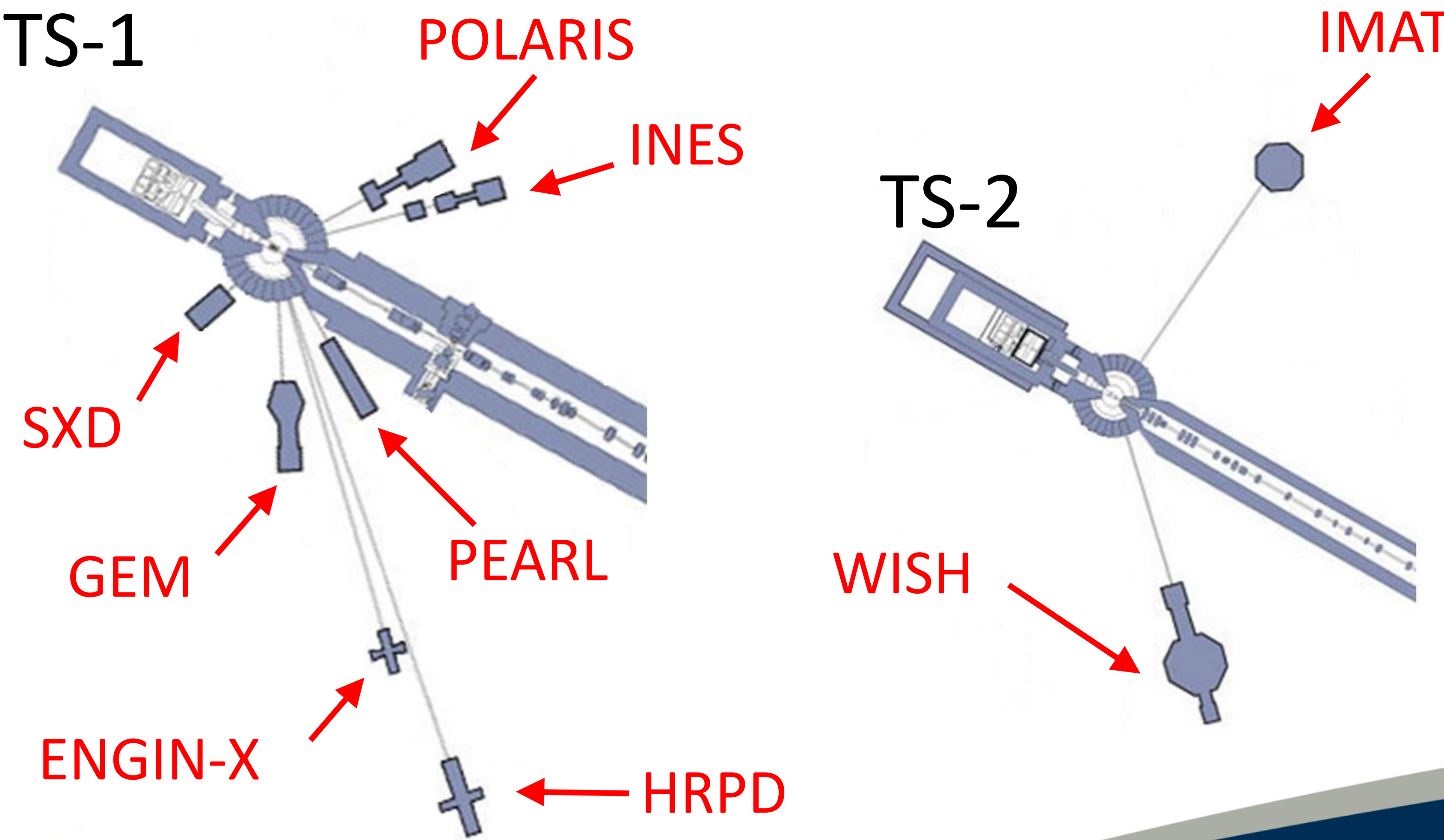
50Hz 800MeV Synchrotron

Extracted proton beams

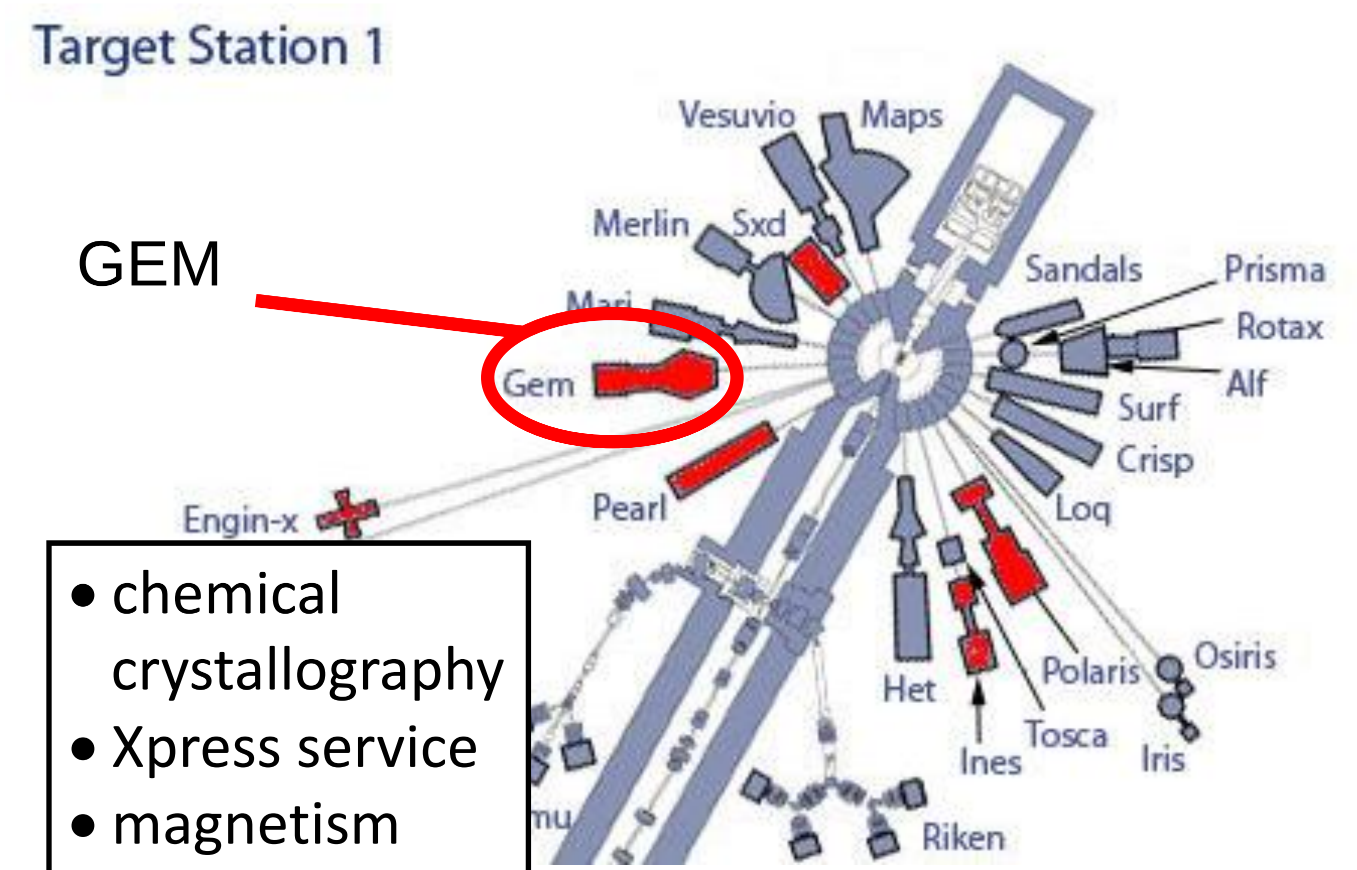
Target station II

Science & Technology Facilities Council ISIS

Diffraction at a pulsed source: ISIS

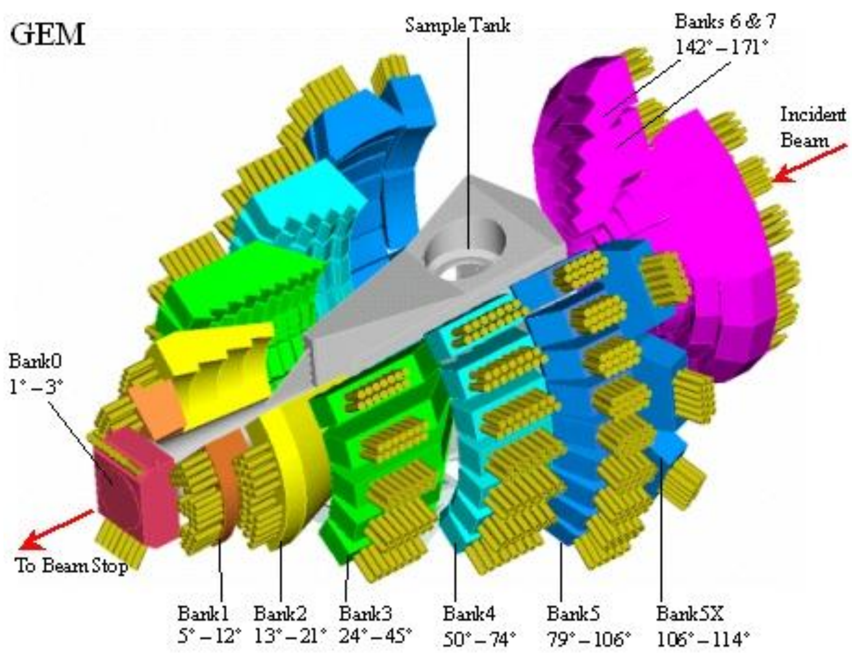


GEM: high intensity powder diffraction



- chemical crystallography
- Xpress service
- magnetism
- PDF studies
- glasses

GEM: high intensity powder diffraction

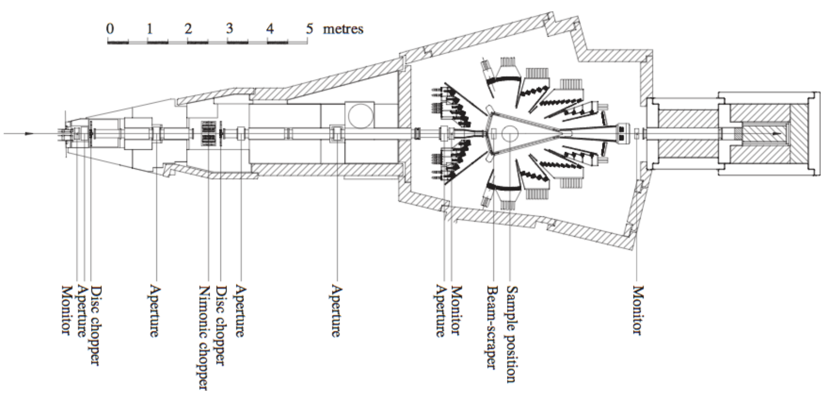


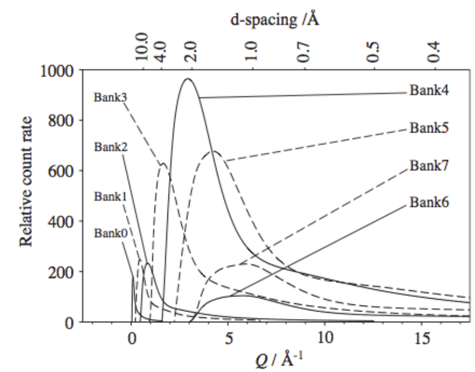
Initially constructed in the late 1990s this powder/liquids diffractometer hybrid changed the way TOF diffraction instruments were designed and built

http://wwwisis2.isis.rl.ac.uk/disordered/gem/gem_home.htm

GEM: high intensity powder diffraction

Detector Bank	Scattering angle 2θ (deg)	Range in azimuthal angle ϕ (deg)	Secondary flight path L_2 (m)	Number of detector elements/ modules	Solid angle Ω (sr)	Resolution $\Delta Q/Q(\%)$	Minimum accessible momentum transfer $Q_{\min}$ (Å^{-1})
Bank0	1.21–3.18	±90.0	2.757–2.767	80/4	0.008	5–10	0.04
Bank1	5.32–12.67	±45.0	2.365–2.376	330/6	0.056	4.7	0.17
Bank2	13.44–21.59	±43.4	1.477–2.100	320/4	0.093	2.4	0.43
Bank3	24.67–45.61	±42.5	1.077–1.893	900/10	0.478	1.7	0.79
Bank4	50.07–74.71	±44.4	1.028–1.436	1400/14	0.988	0.79	1.56
Bank5	79.07–106.60	±44.5	1.376–1.383	2160/18	1.135	0.51	2.35
Bank5X	106.02–114.19	±42.7	1.377–1.387	720/18	0.378	0.5	2.95
Bank6	142.50–149.72	±69.3	1.544–1.738	560/14	0.280	0.34	3.50
Bank7	149.98–171.40	±66.6	1.035–1.389	800/10	0.443	0.35	3.57

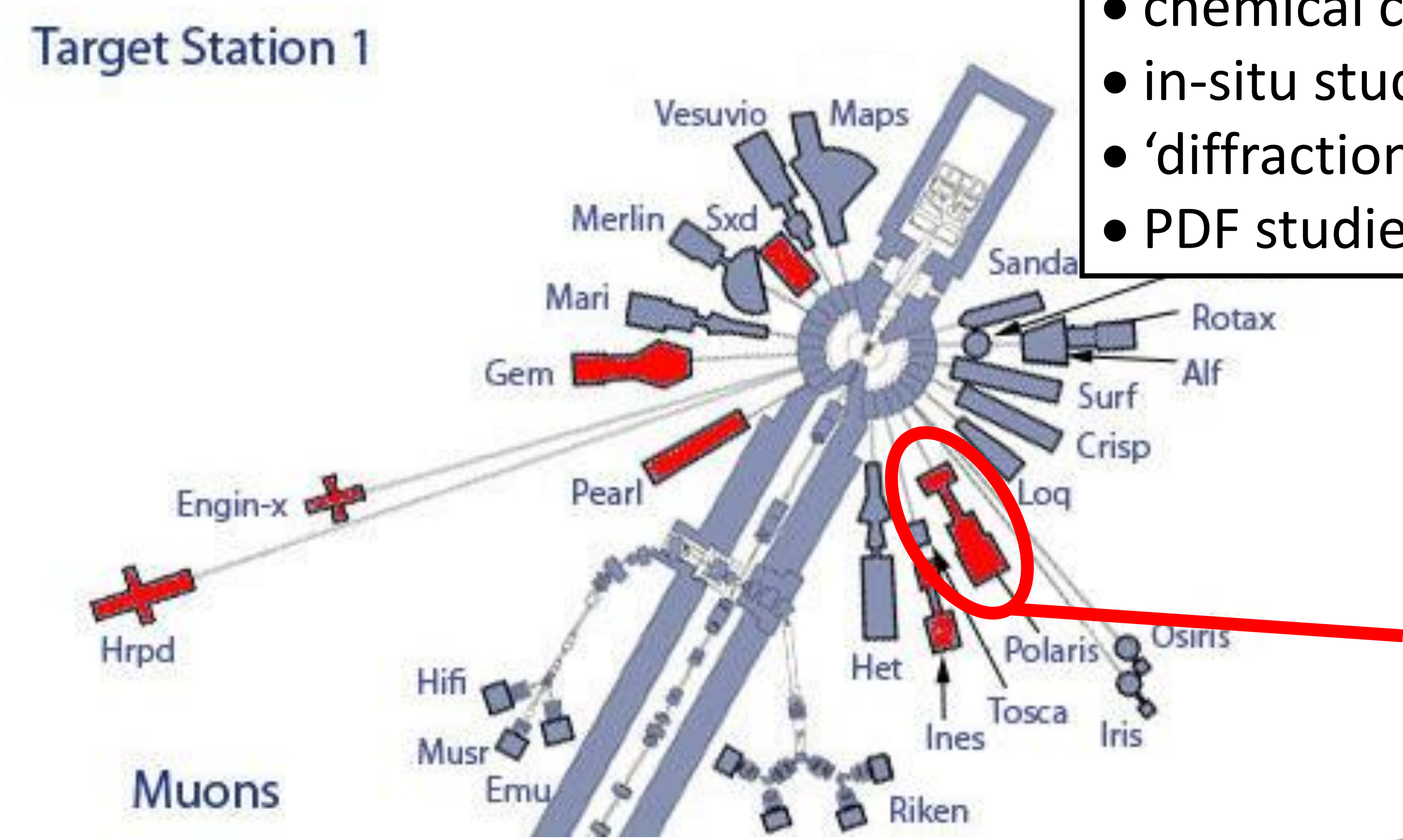




From Hannon, NIMA (2005), **551**, 88-107

Polaris: high intensity powder diffraction

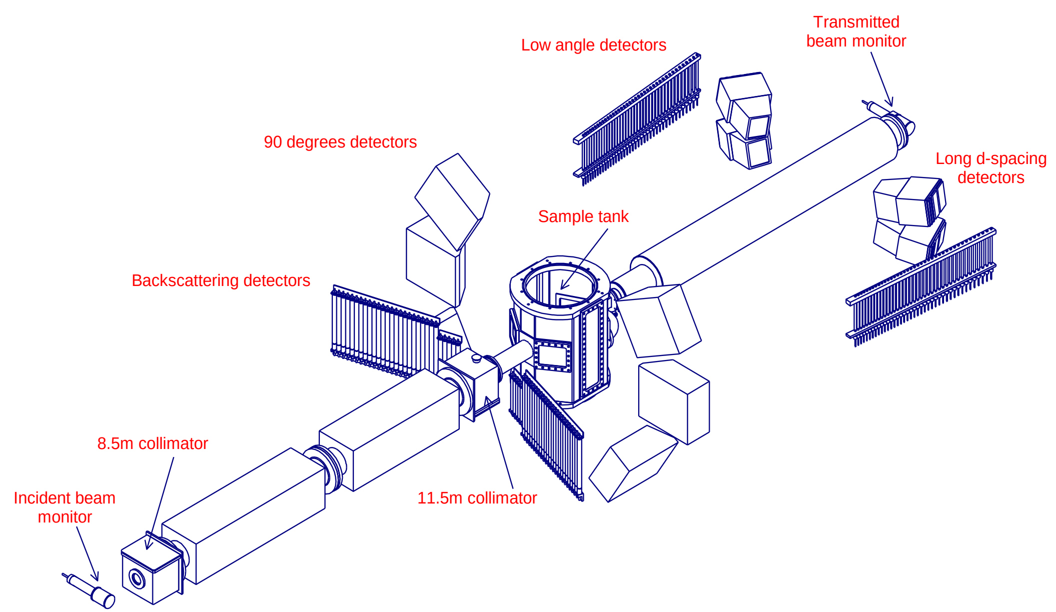
- chemical crystallography
- in-situ studies
- ‘diffraction plus’
- PDF studies

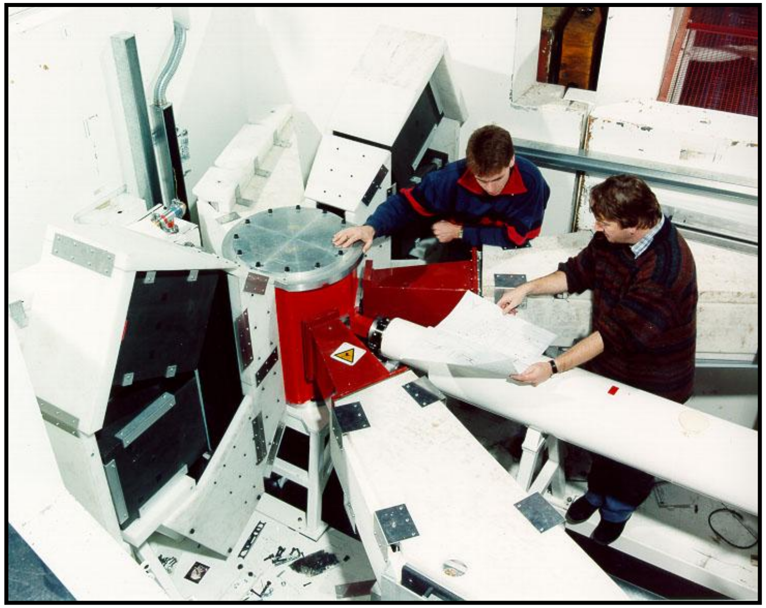


Polaris

Science & Technology Facilities Council
ISIS

Polaris: old configuration





Compare with GEM:
- Higher sample flux
- Wider bandwidth
- Lower resolution
- Hotter spectrum
- Lower detector coverage

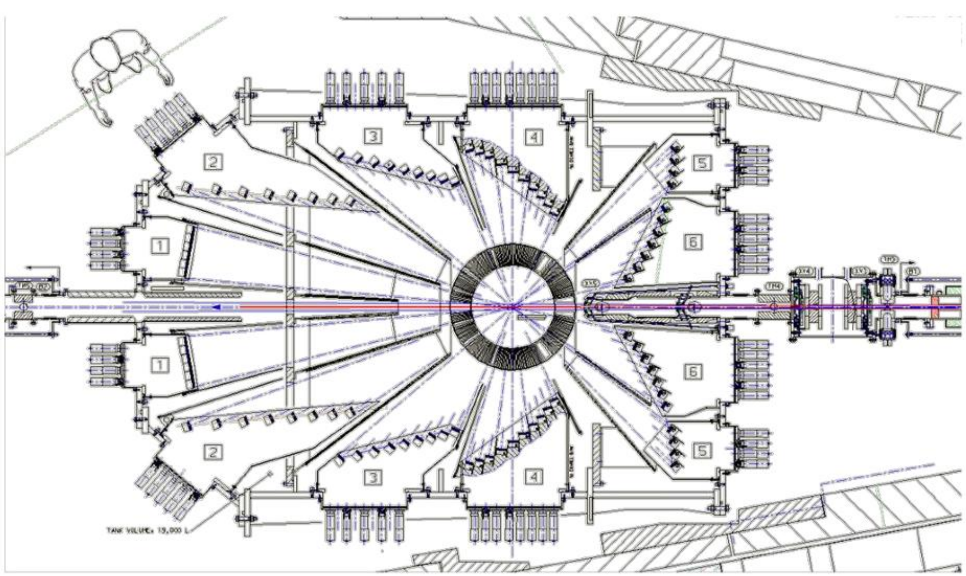
http://www.isis.stfc.ac.uk/instruments/polaris/polaris4643.html

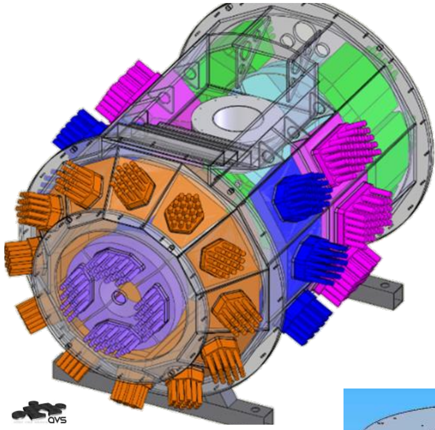
Polaris: old configuration

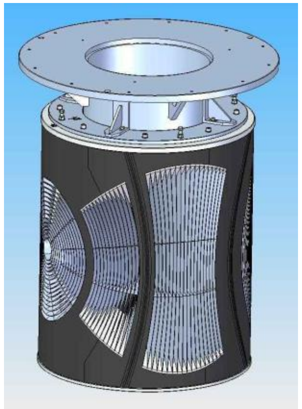
bank position (label)	low angle (A)	low angle (B)	backscattering (C)	90 degrees (E)
detector type	^{3}He	ZnS	^{3}He	ZnS
no. of elements	2 x 40 = 80	4 x 20 = 80	2 x 29 = 58	6 x 36 = 216
L_2 (m)	1.72 - 2.65	~2.2	0.65 - 1.35	~0.80
2θ range	28°< 2θ < 42°	13°< 2θ < 15°	130°< 2θ < 160°	83°< 2θ < 97°
Ω (ster)	0.046	0.009	0.29	0.48
Δd/d	$\sim 1 \times 10^{-2}$	$\sim 3 \times 10^{-2}$	$\sim 5 \times 10^{-3}$	$\sim 7 \times 10^{-3}$
d-range (Å)	0.5 - 8.3	0.5 - 21.6	0.2 - 3.2	0.2 - 4.0
Q-range ($Å^{-1}$)	0.75 - 12.6	0.3 - 12.6	2.0 - 31.4	1.5 - 31.4

- Good workhorse instrument for powder diffraction
- High Q accessible for disordered materials investigation using the PDF method
- Some in situ capability but limited by count-rate
- Compatible with a wide range of restricted geometry sample environment

Polaris upgrade







- Increase primary flight path to 14 m
- Optimise each detector bank to give constant resolution
- Increase detector coverage
- Design a collimator to reduce background and parasitic scattering

http://www.isis.stfc.ac.uk/instruments/polaris/polaris-upgrade-poster11575.pdf

Science & Technology Facilities Council
ISIS

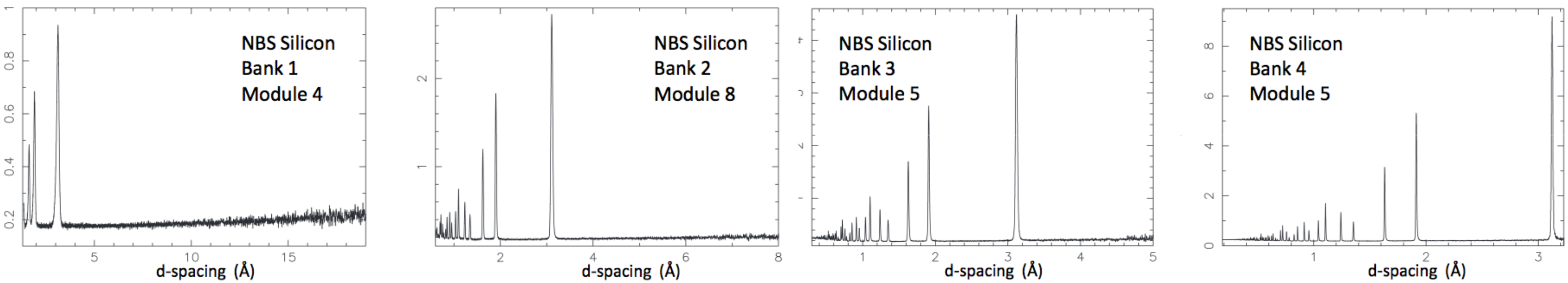
Polaris upgrade

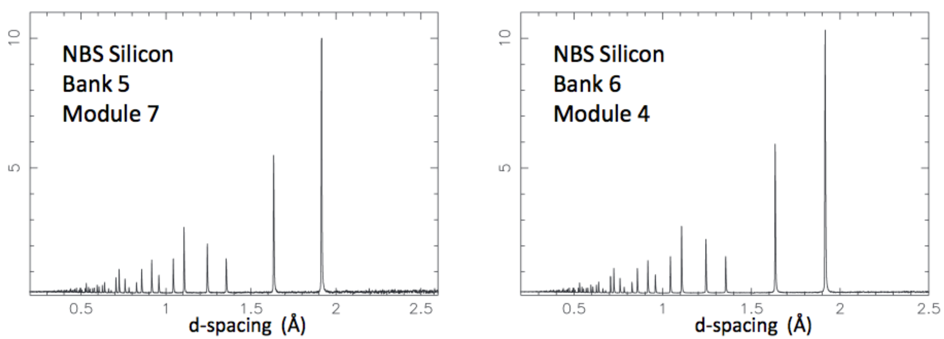
http://www.isis.stfc.ac.uk/instruments/polaris/polaris4643.html

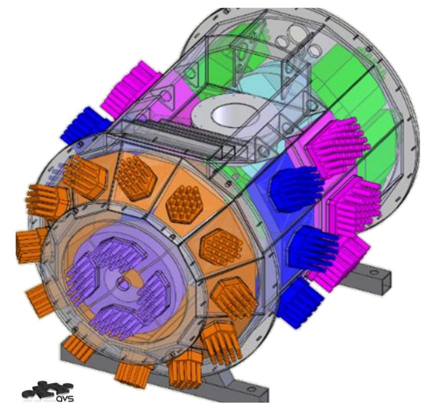
Science & Technology Facilities Council
ISIS

Current Polaris

http://www.isis.stfc.ac.uk/instruments/polaris/polaris-upgrade---first-diffraction-pattern12763.pdf



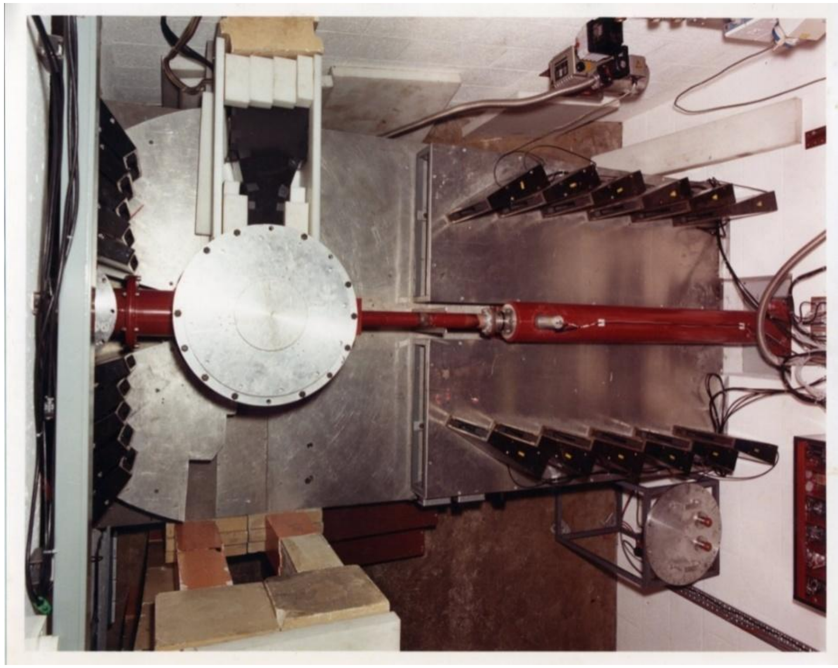


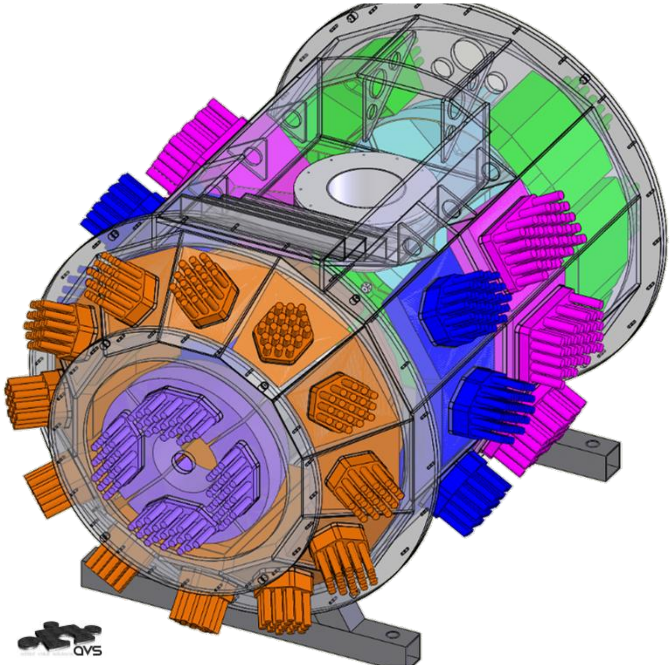


Bank 1 – cyan
Bank 2 – green
Bank 3 – pink
Bank 4 – blue
Bank 5 – orange
Bank 6 - purple

- Increased count rate ×3 at high scattering angle to >20 for low angle banks
- Resolution improvement e.g. bank 5 and 6 of 3×10^{-3} cf. 5×10^{-3}
- Improvement in data at high Q

Polaris 1995 v 2013

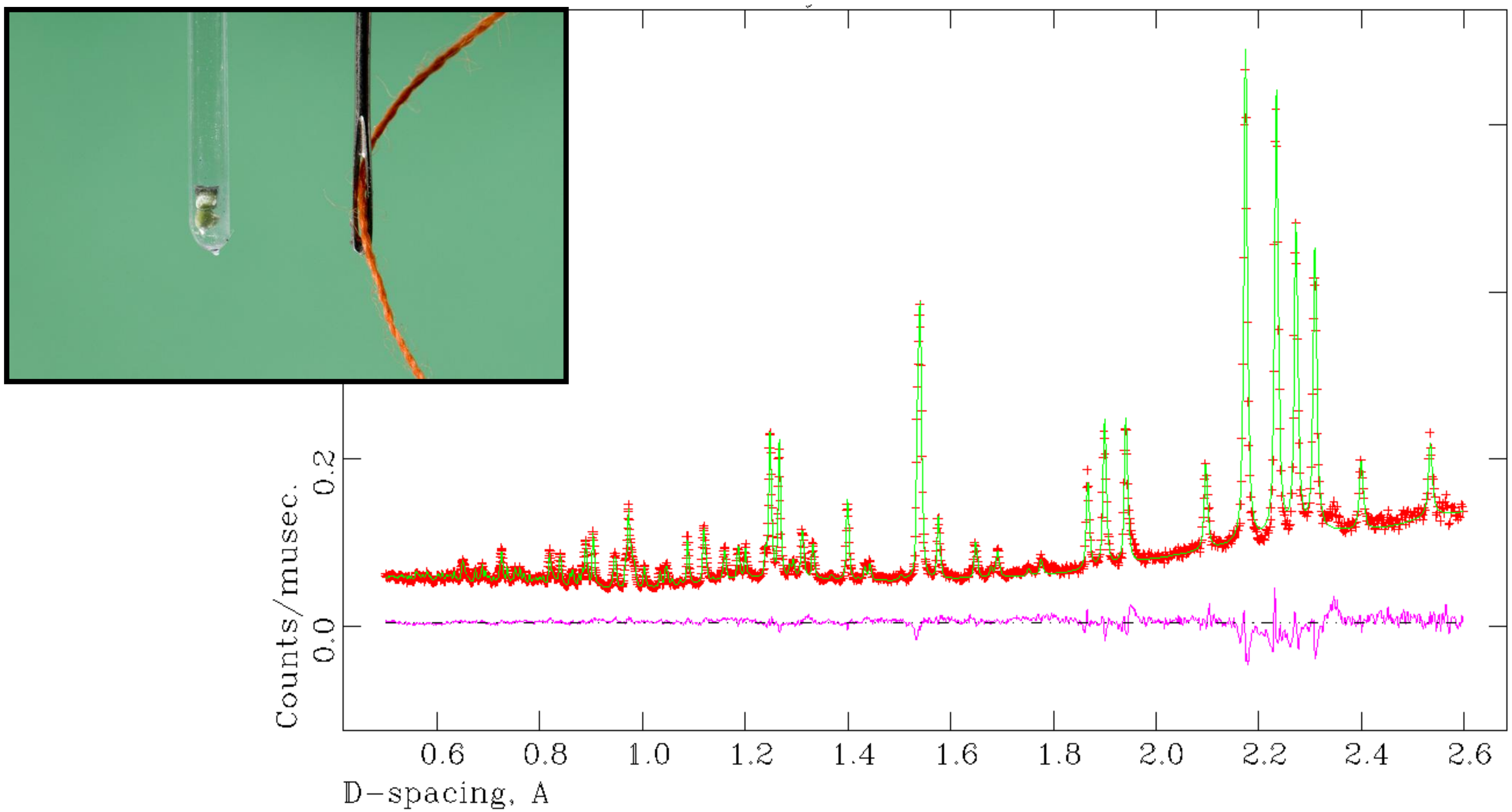




- 1995 500 mg 24+ hrs
- 2013 500 mg 15-20 minutes with increased Q-range

Contemporary instruments NOVA and iMateria (J-PARC), POWGEN-3 (SNS)

Polaris: pushing boundaries in sample size

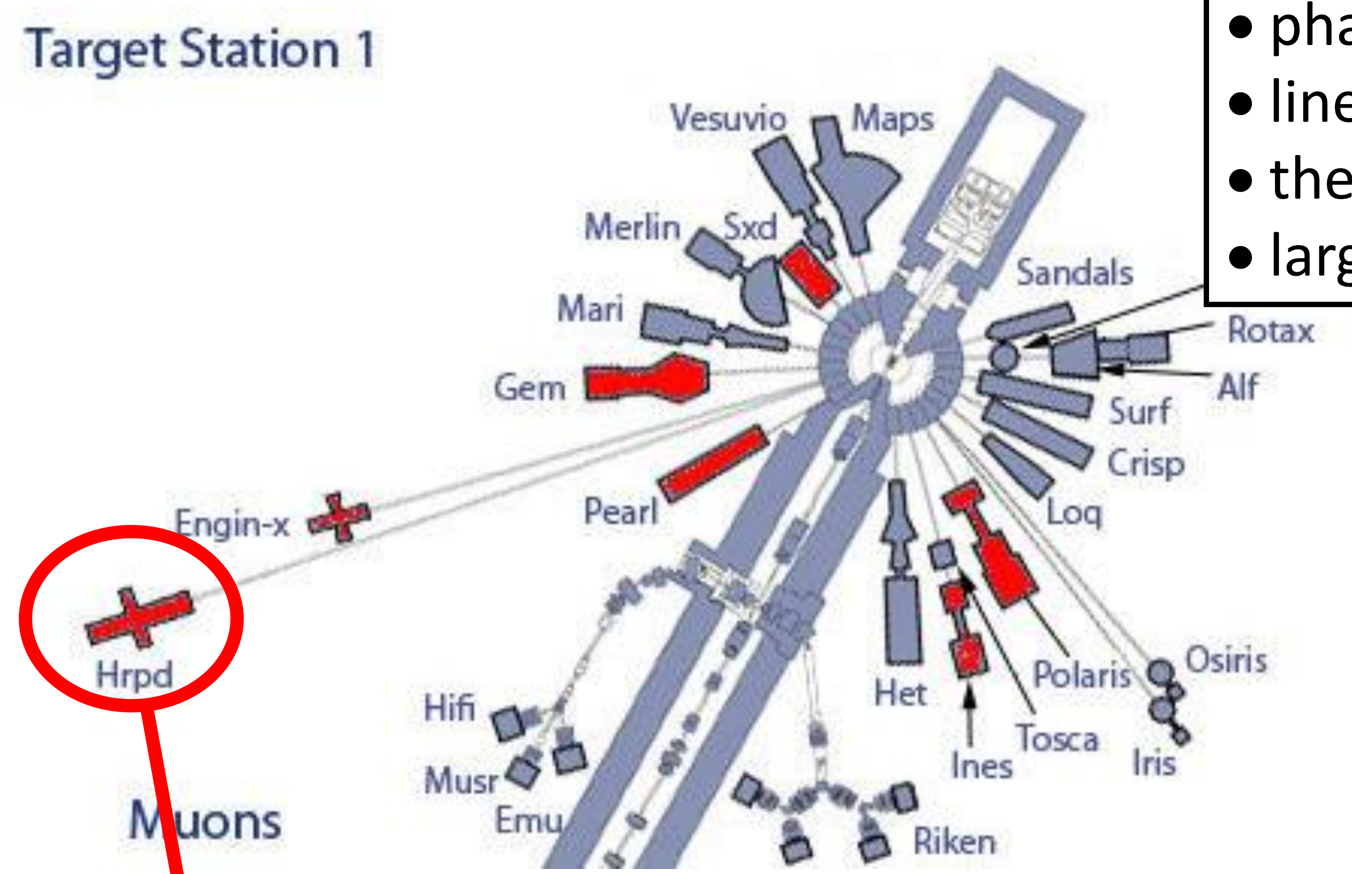


~1mm^3 sample of $NaNiF_3$ phase prepared at high p + high T

Lindsay-Scott *et al*, J. Appl. Cryst., **47**, 1939 (2014)

Science & Technology Facilities Council
ISIS

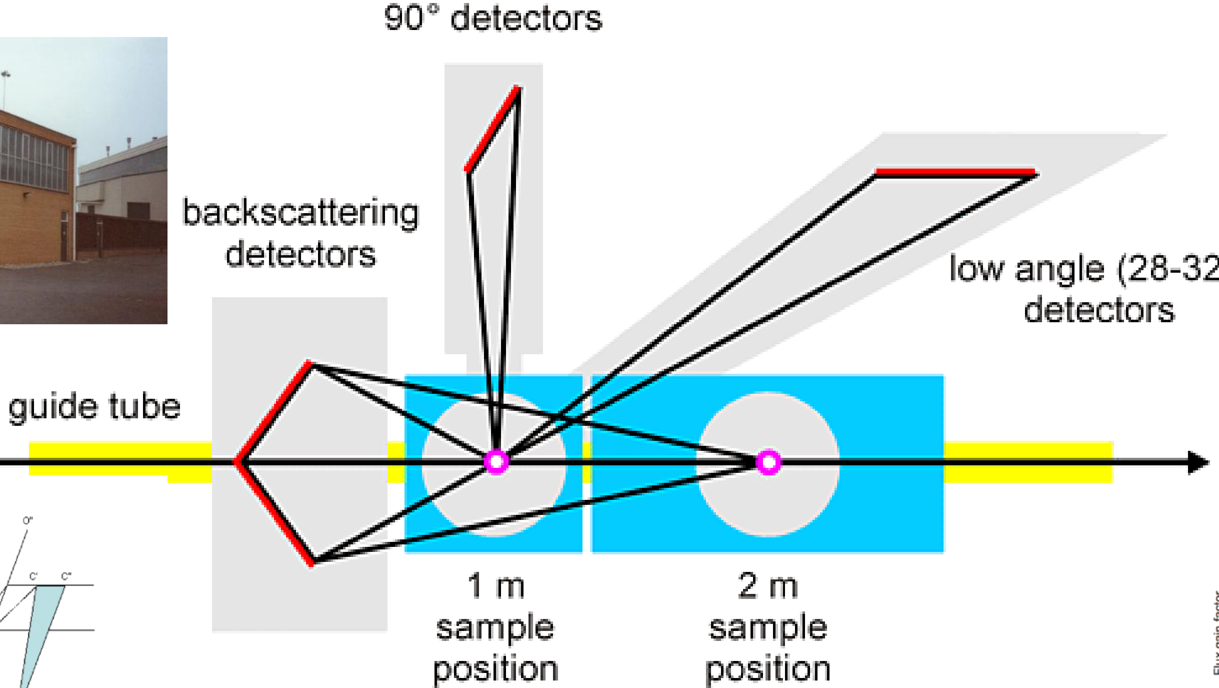
HRPD: high resolution powder diffraction

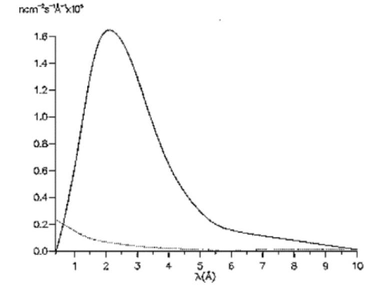


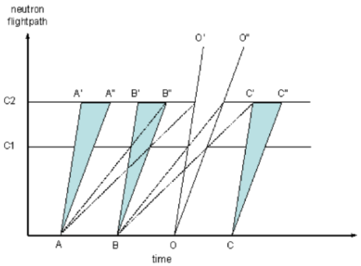
- phase transitions
- line broadening
- thermal expansion
- large unit cells

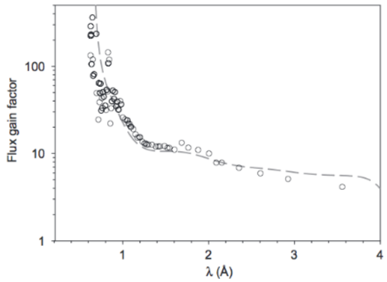
HRPD

HRPD: high resolution powder diffraction









- Approximately 100 m primary flight path
- 100K methane moderator – peak flux around 2 Å
- Operates at 5 or 10 Hz cf. TS-1 50 Hz

http://www.isis.stfc.ac.uk/instruments/hrpd/hrpd.html

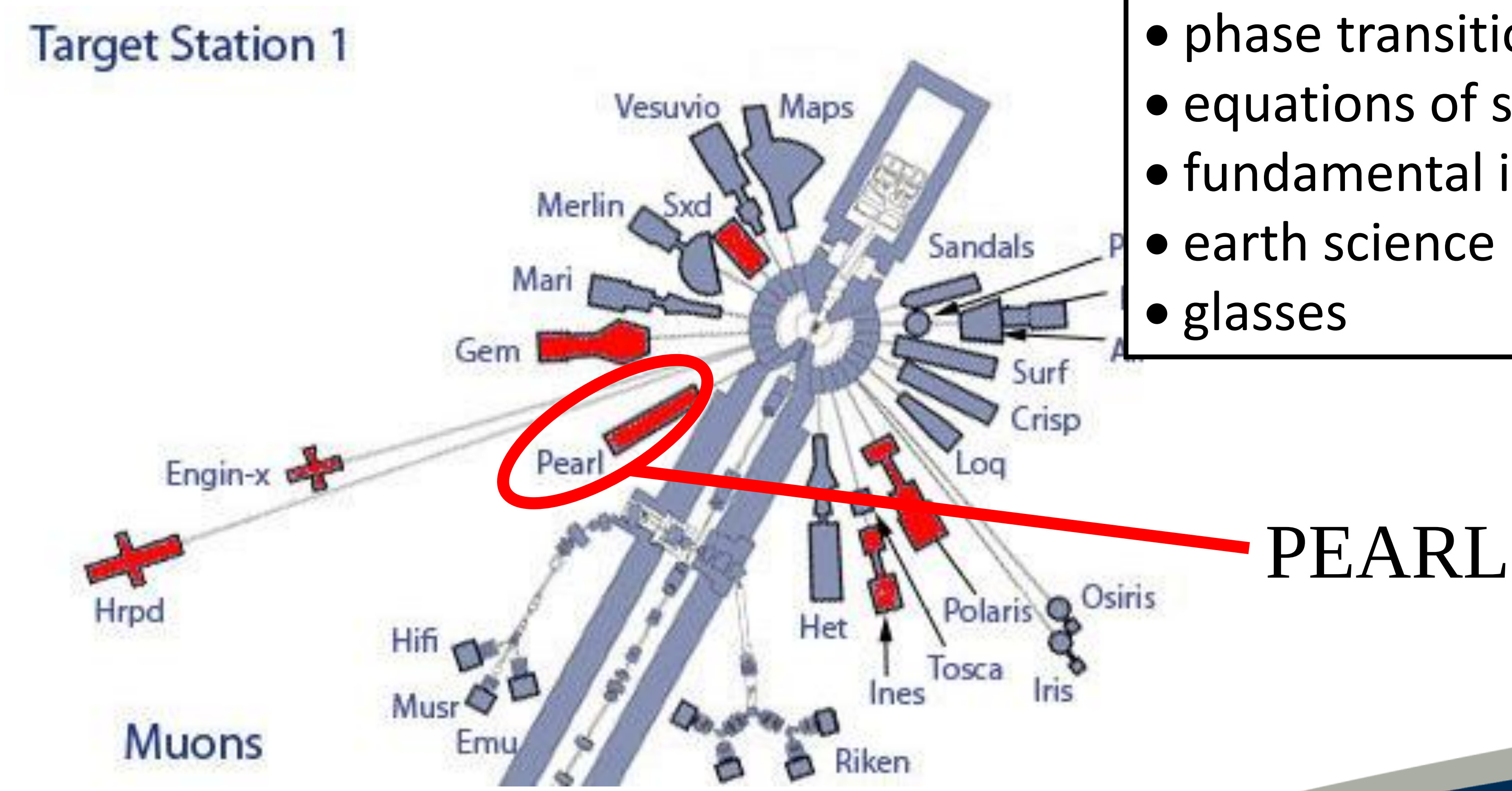
HRPD: high resolution powder diffraction

Table 1. HRPD Detector Bank Details			
	Backscattering	**90°**	**Low Angle**
Detector Specification	ZnS scintillator	ZnS scintillator	½" 10atm He^3 gas tubes
Geometry	60 rings: $7 < r_1 < 8.5$cm $35.5 < r_{60} < 37$cm 8 Octants: 4147cm^2	Slab: 20 x 20cm 66 x 3mm elements 6 Modules: 2400cm^2	72 tubes: (20cm active length) 8 tubes/module 9 Modules: 1800cm^2
Fixed Scattering Angle	$160° < 2\theta < 176°$ (1m)	$87° < 2\theta < 93°$	$28° < 2\theta < 32°$
Solid Angle (Ω)	0.41 ster (1m)	0.08 ster	0.01 ster
Resolution (Δd/d)	~ 4-5 x 10^{-4}	~ 2 x 10^{-3}	~ 2 x 10^{-2}
***d*-spacing range (30-230ms)**	~ 0.6 - 4.6Å 0.25 – 4.6 Å	~ 0.9 - 6.6Å 0.4 – 6.6 Å	~ 2.2 - 16.5Å 1.0 – 16.5 Å

- Large backscattering detector to minimise $\cot^2\theta$ in resolution term
- High resolution at intermediate Q
- Long flight path to reduce Δt/t and ΔL/L uncertainties
- Pulse skipping to increase bandwidth (@50 Hz 0.4 Å)
- Good combination of parameters for a high resolution TOF diffractometer

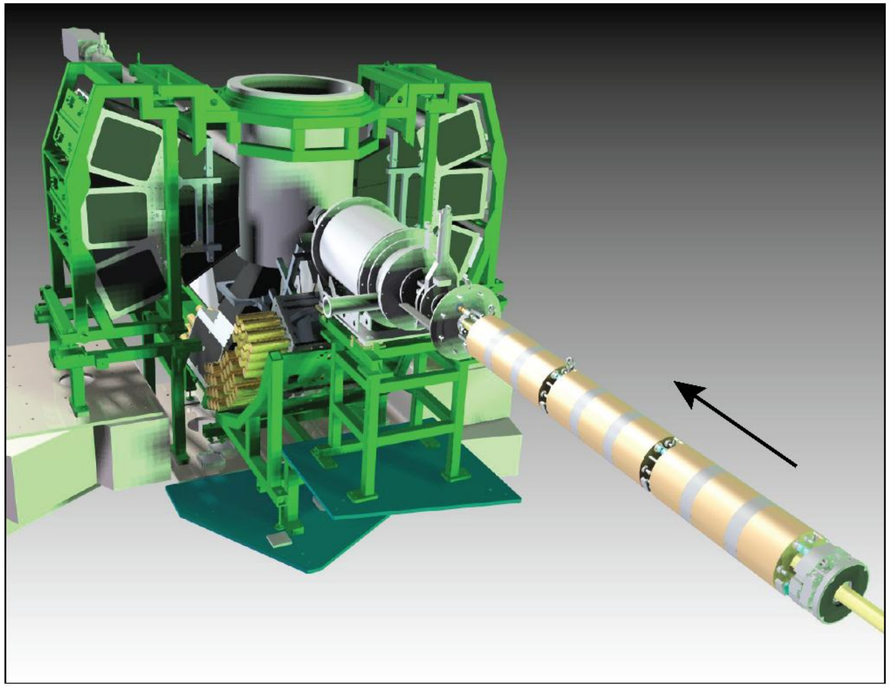
Contemporary instrument sHRPD (J-PARC), no current analogue at SNS

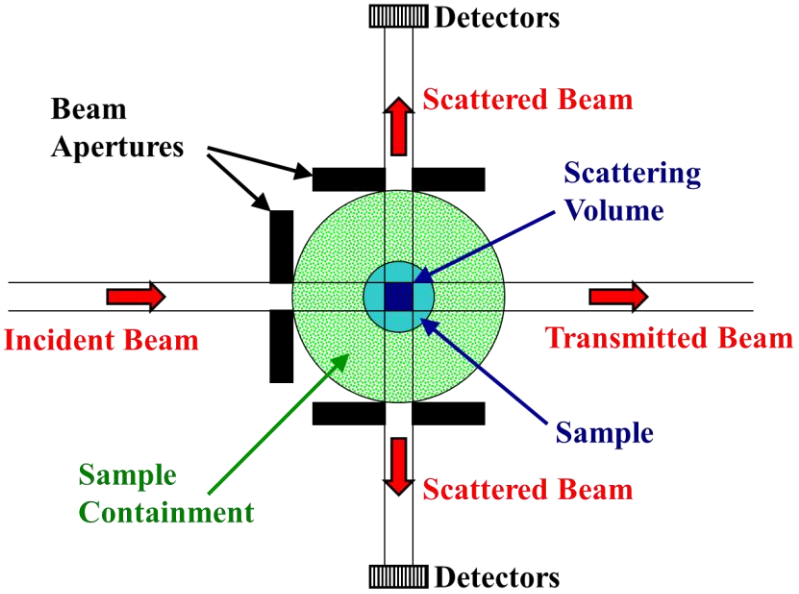
PEARL: high pressure diffraction



- phase transitions
- equations of state
- fundamental ices
- earth science
- glasses

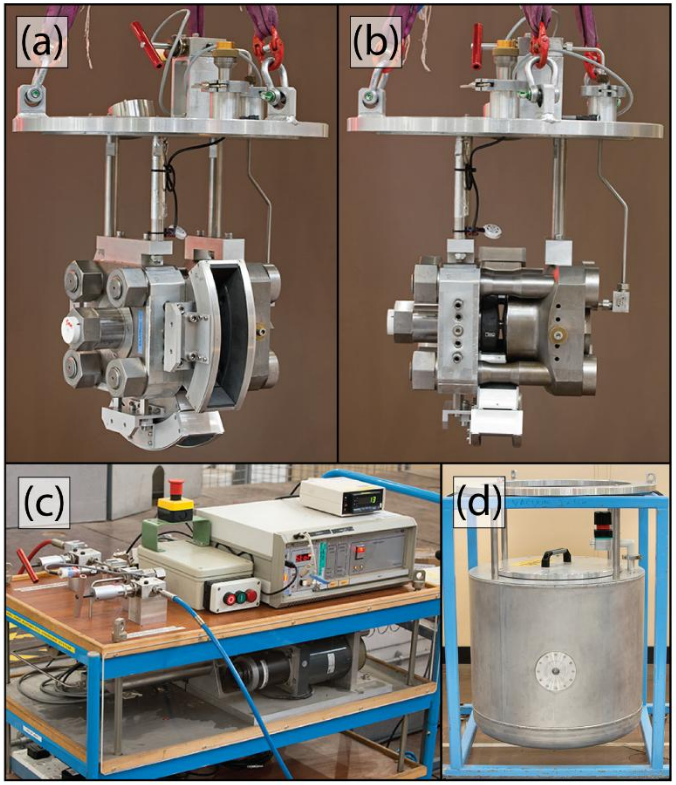
PEARL: high pressure diffraction



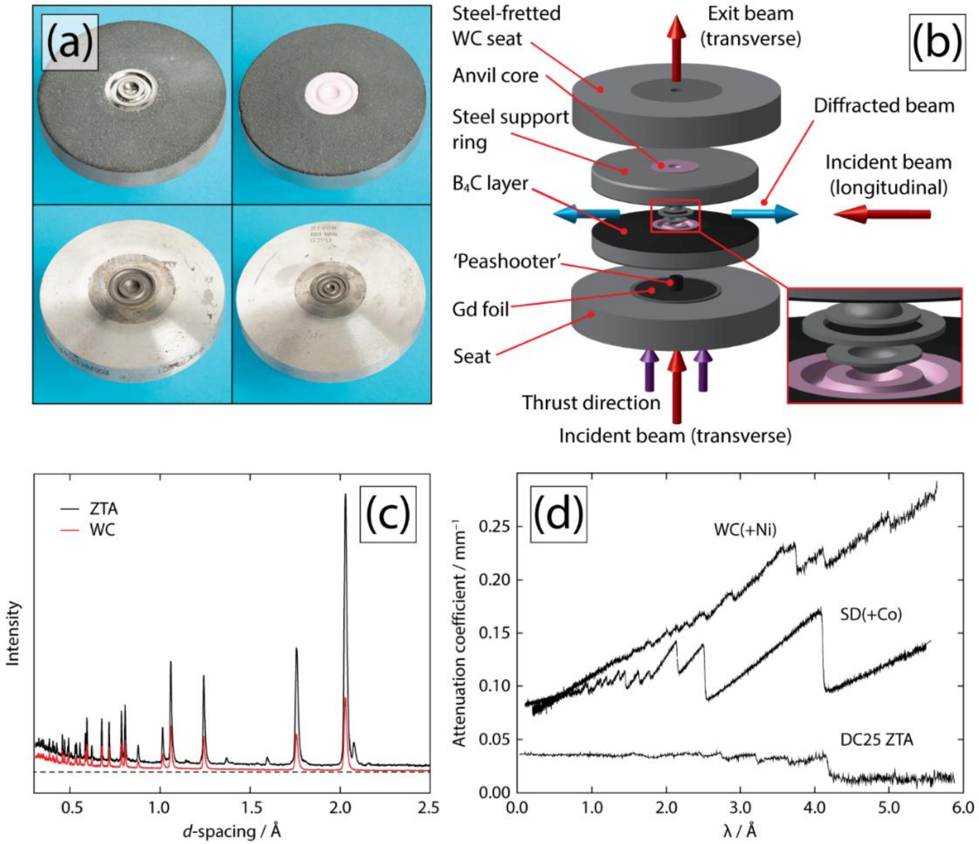


Instrument geometry designed to match sample environments

PEARL: high pressure diffraction



Paris Edinburgh V3 press

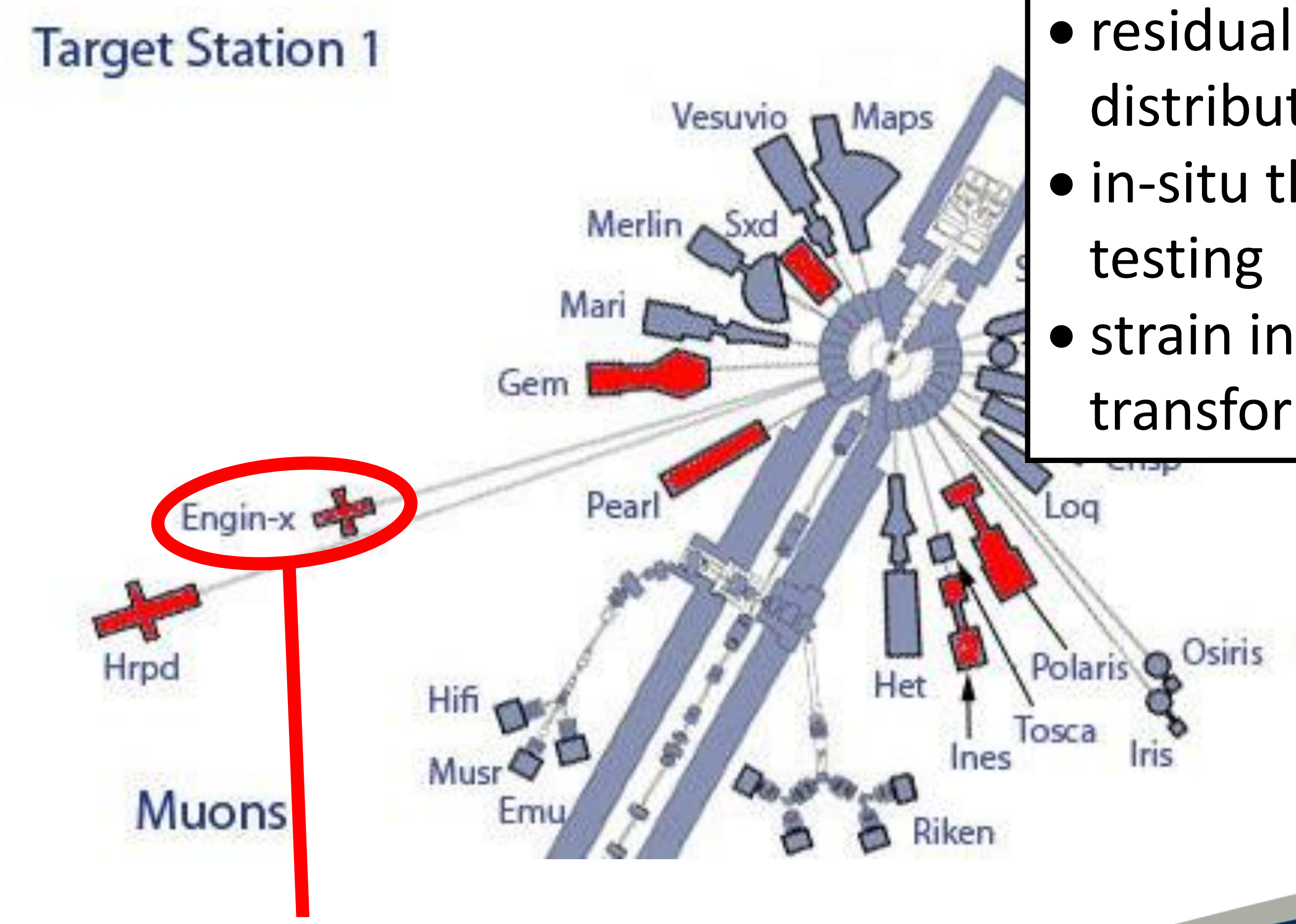


Anvil material properties

See C.L. Bull, N.P Funnell, M.G. Tucker, S. Hull, D.J. Francis, W.G. Marshall, *High Pressure Research* 2016, DOI: 10.1080/08957959.2016.1214730

Contemporary instruments: Planet(J-Parc), SNAP (SNS)

ENGIN-X: engineering diffraction



ENGIN-X

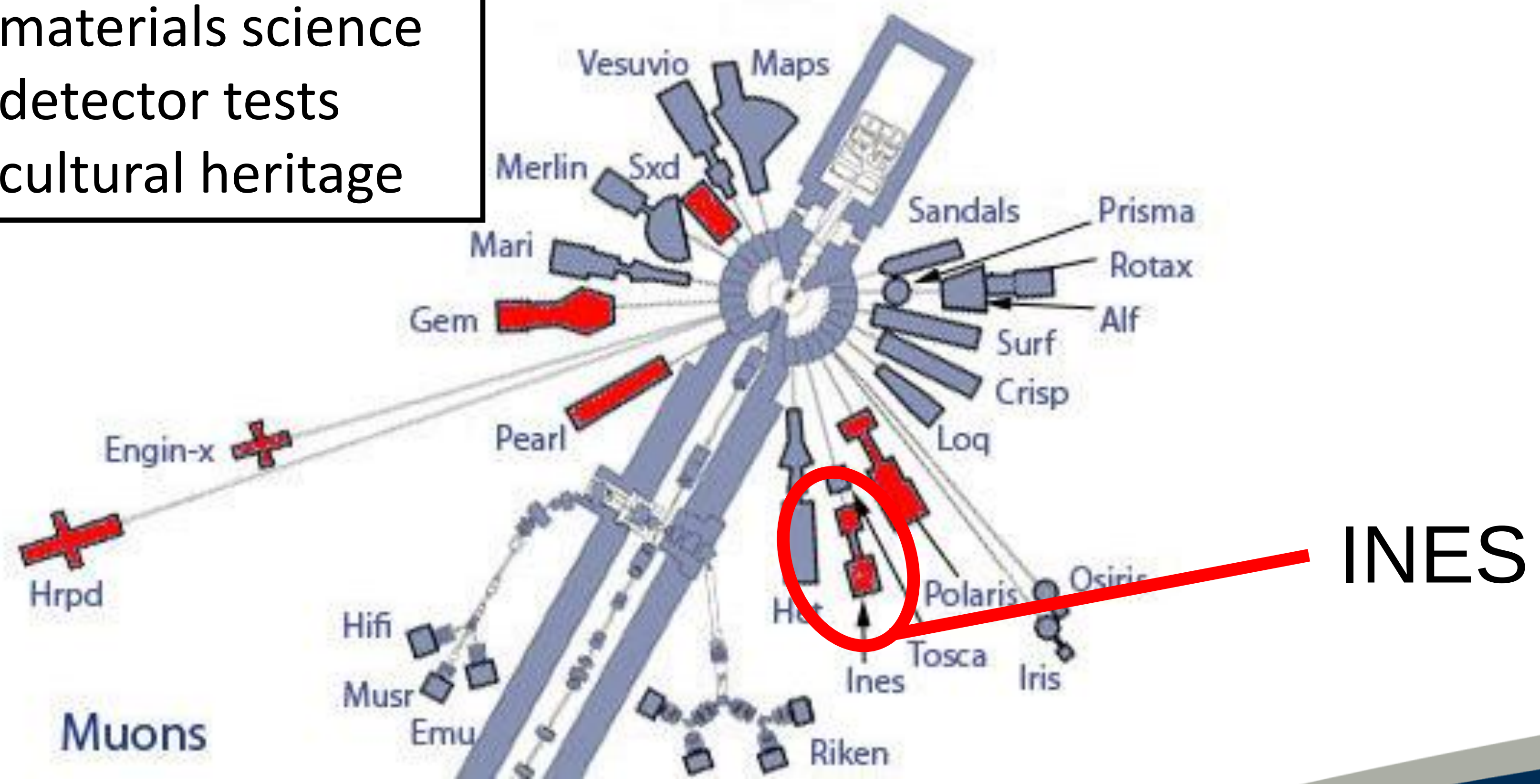
- residual stress distributions
- in-situ thermomechanical testing
- strain induced phase transformations

ENGIN-X: engineering diffraction

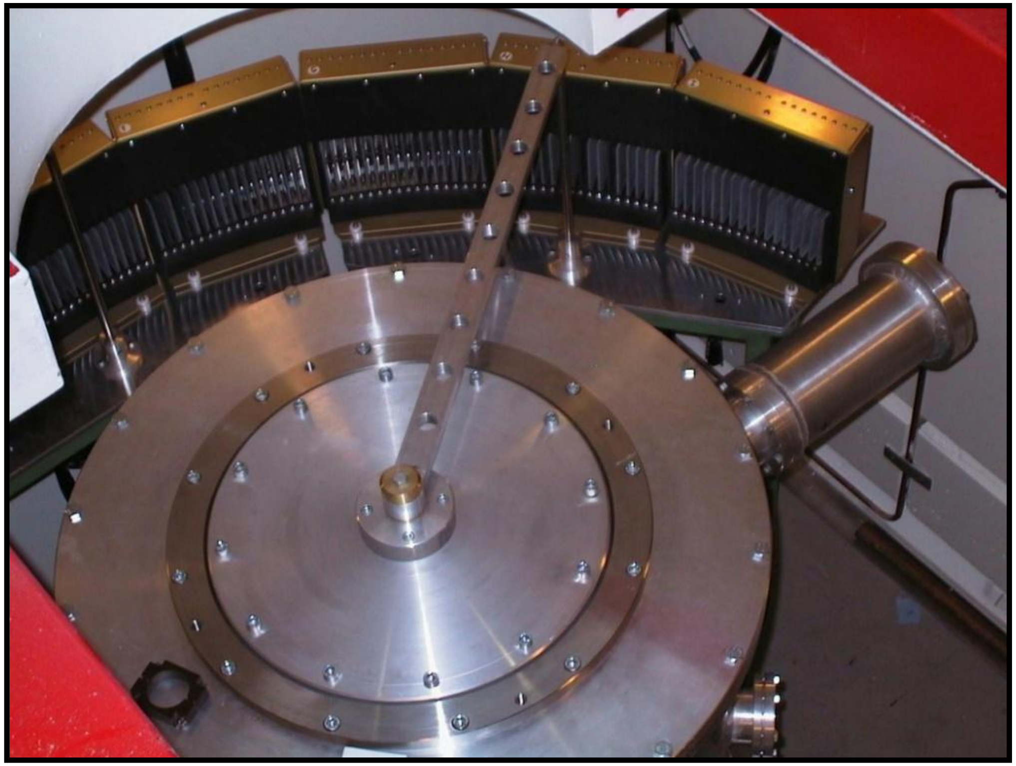
Contemporary instruments: Vulcan (SNS)

INES: joint CNR-ISIS instrument

- powder diffraction
- materials science
- detector tests
- cultural heritage



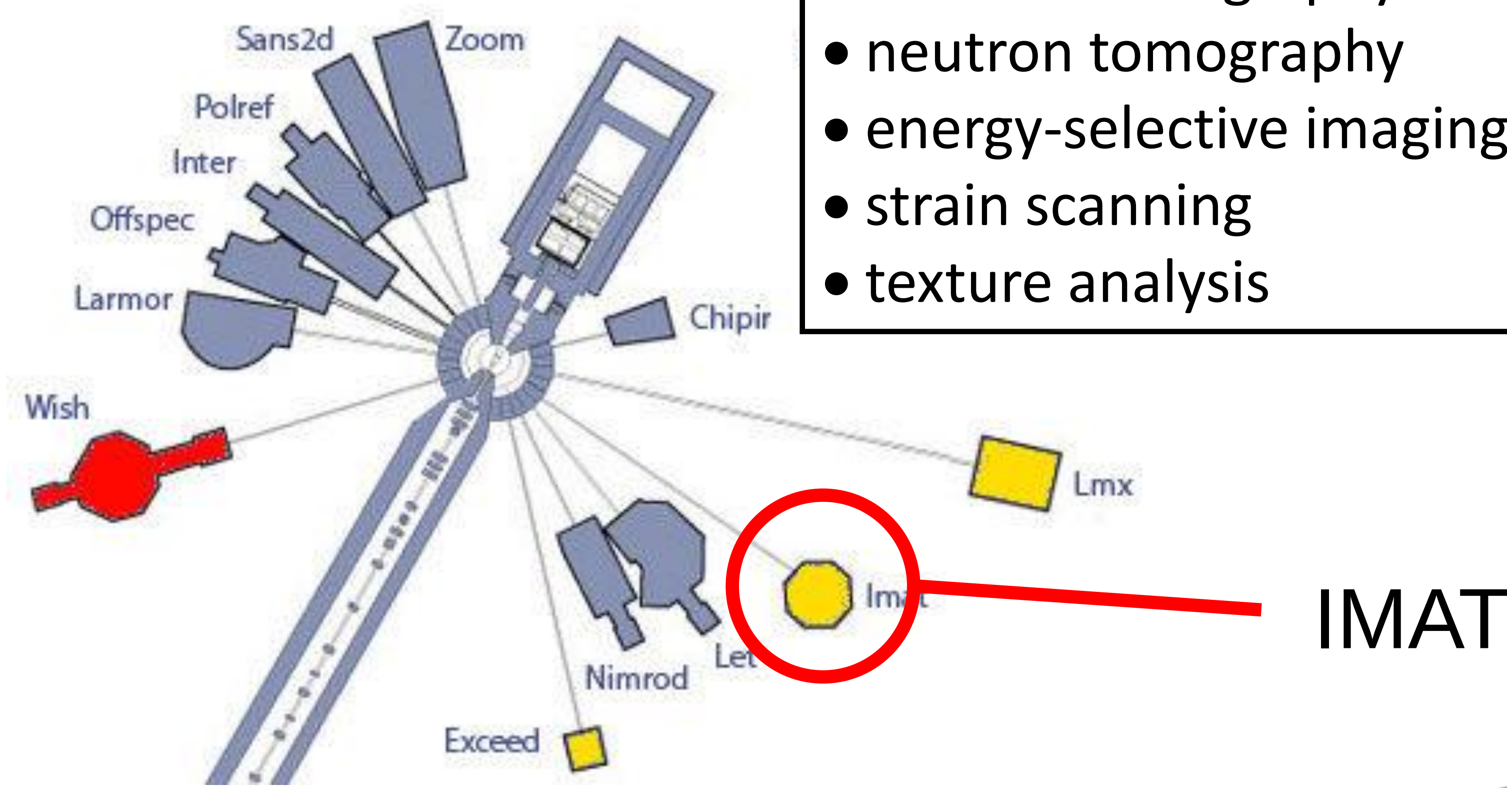
INES for cultural heritage



General purpose diffractometer for materials characterisation and cultural heritage studies. Built and managed by the Italian National Research Council (CNR) within a co-operation agreement with STFC.

IMAT: imaging and diffraction

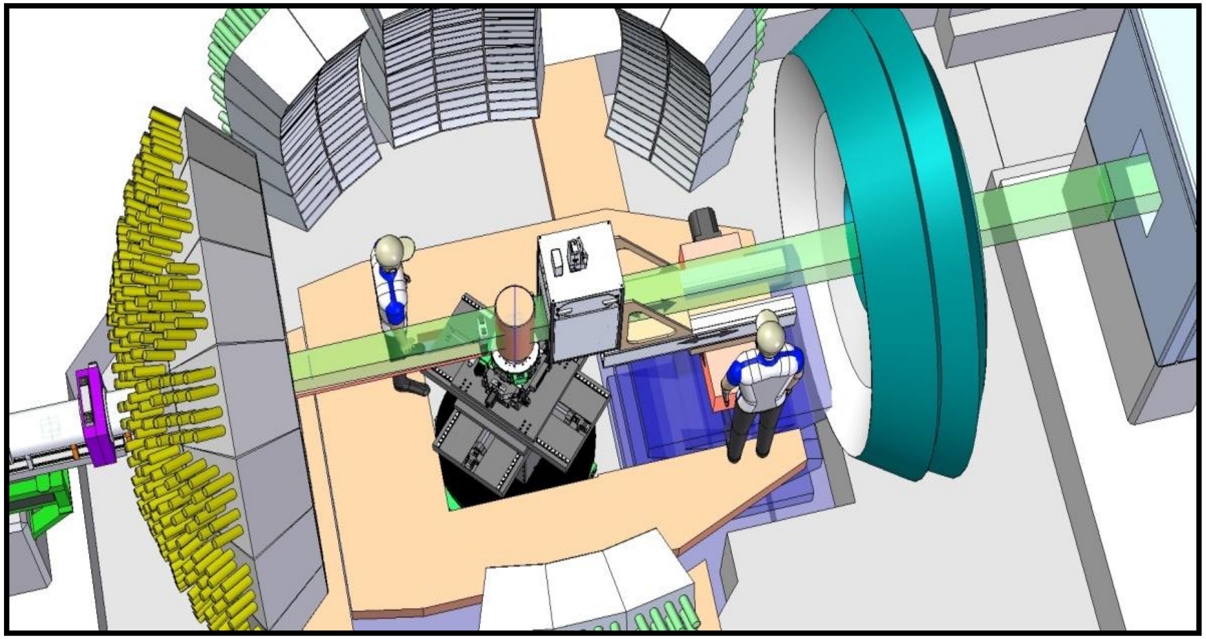
- neutron radiography
- neutron tomography
- energy-selective imaging
- strain scanning
- texture analysis



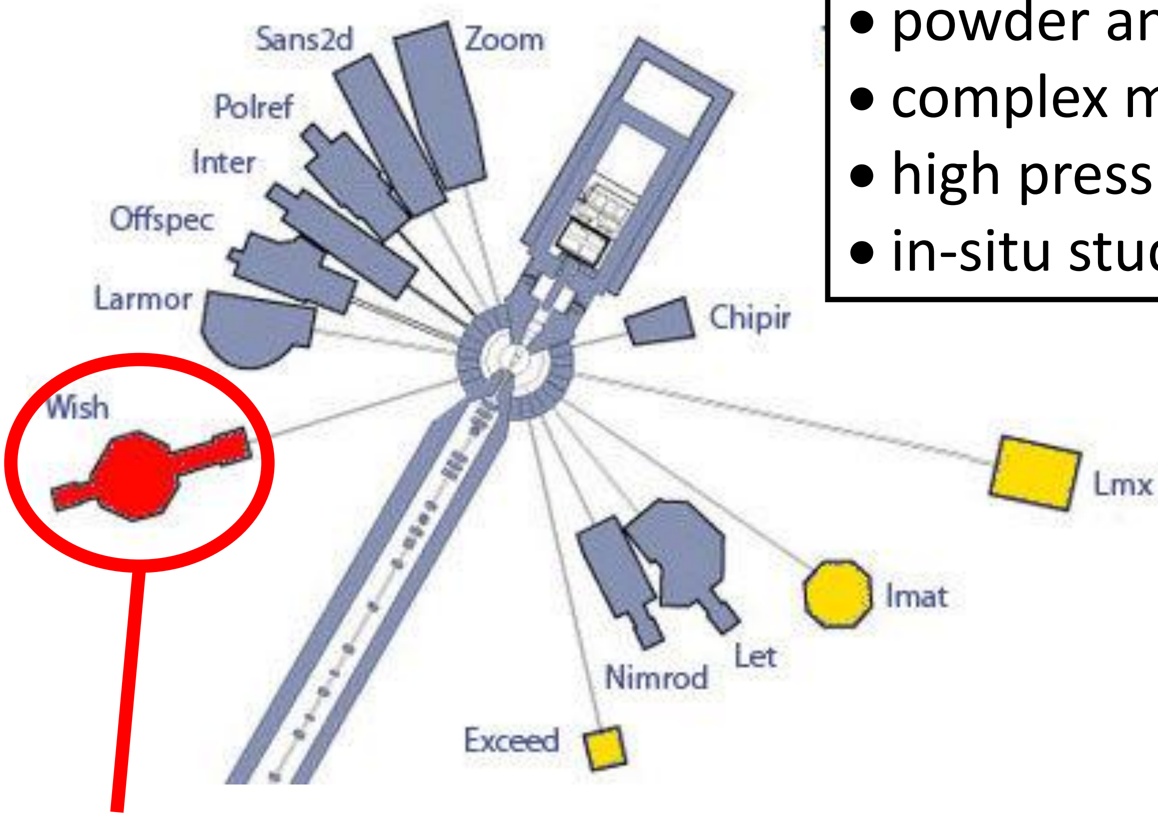
IMAT: imaging and diffraction

Dual-use instrument: neutron imaging and diffraction

- Conventional neutron radiography and tomography
- Novel energy-selective (Bragg-edge) imaging techniques
- Simultaneous strain and texture measurements



WISH: magnetic diffraction

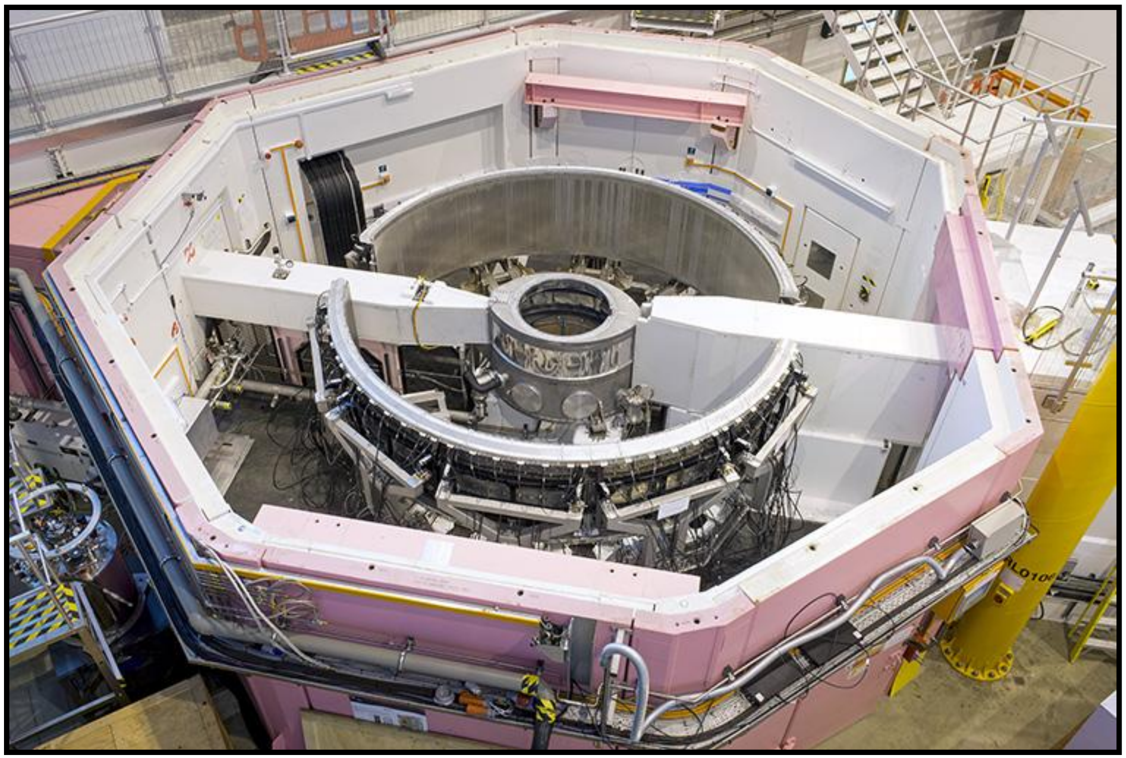


- powder and single crystal
- complex magnetism
- high pressure
- in-situ studies

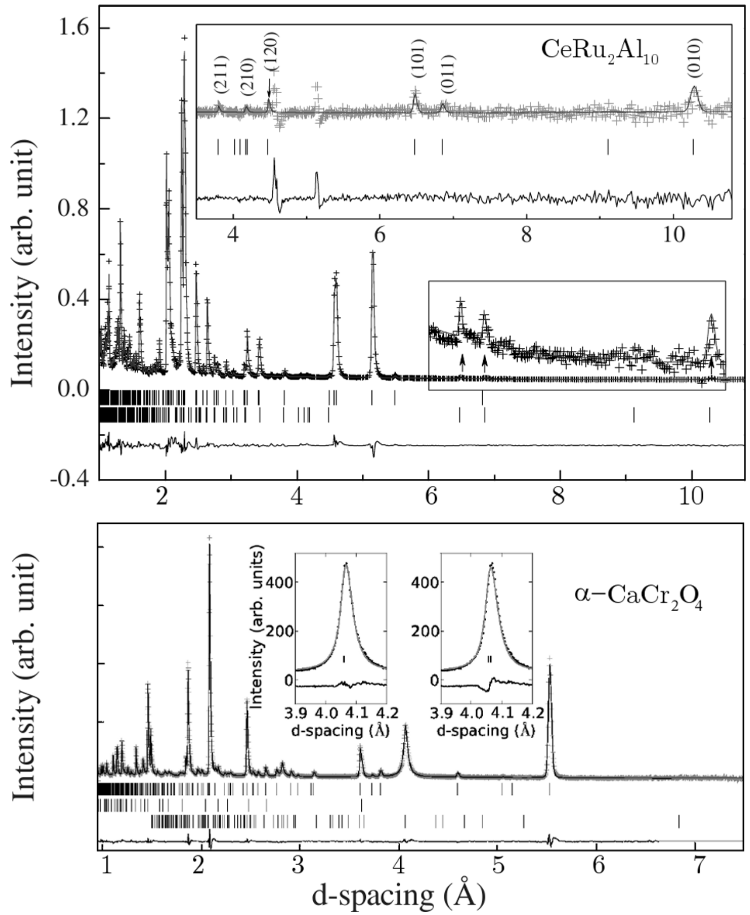
WISH

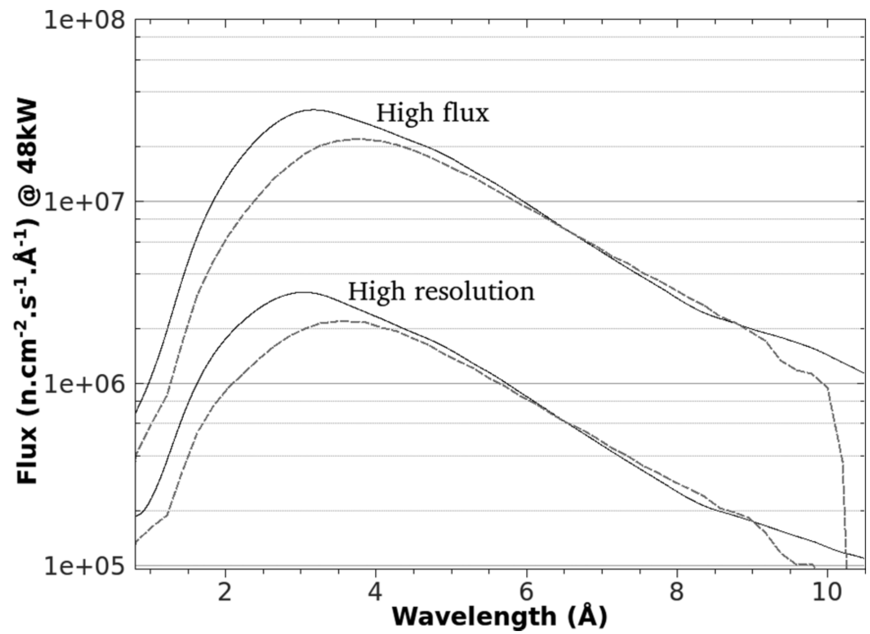
WISH: magnetic diffraction

The magnetic moment of the neutron interacts with magnetic fields caused, for example, by unpaired electron spins in a material.



WISH: Magnetic diffraction

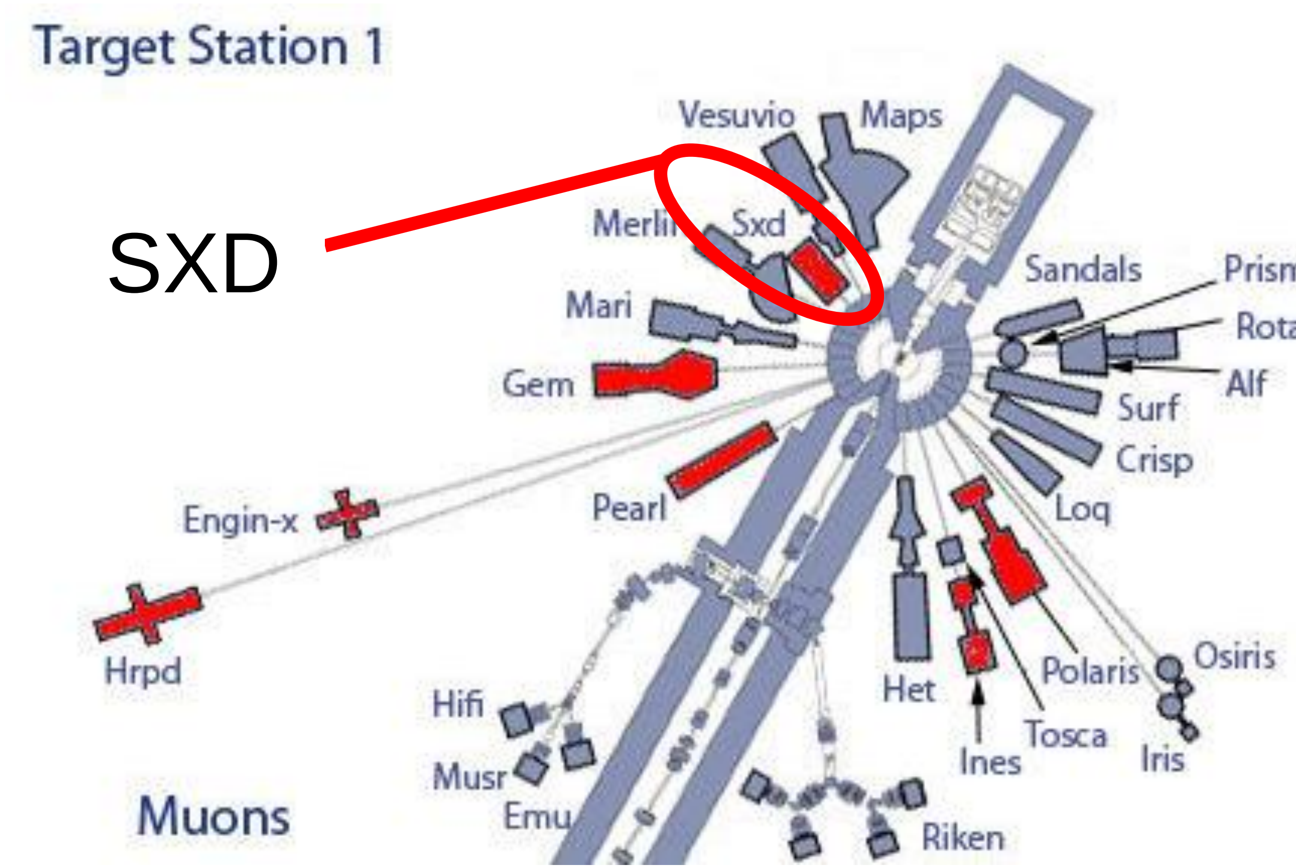




See Chapon *et al*. Neutron News (2011), **22(2)**, 22-25

Science & Technology Facilities Council
ISIS

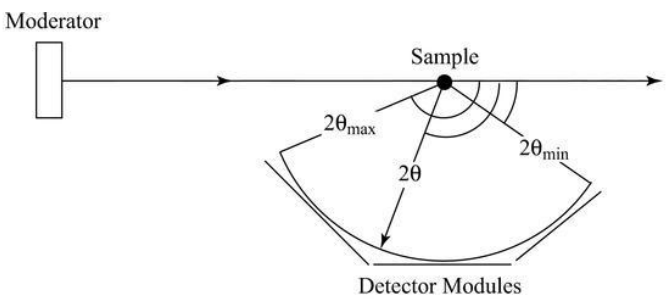
SXD: single crystal diffraction

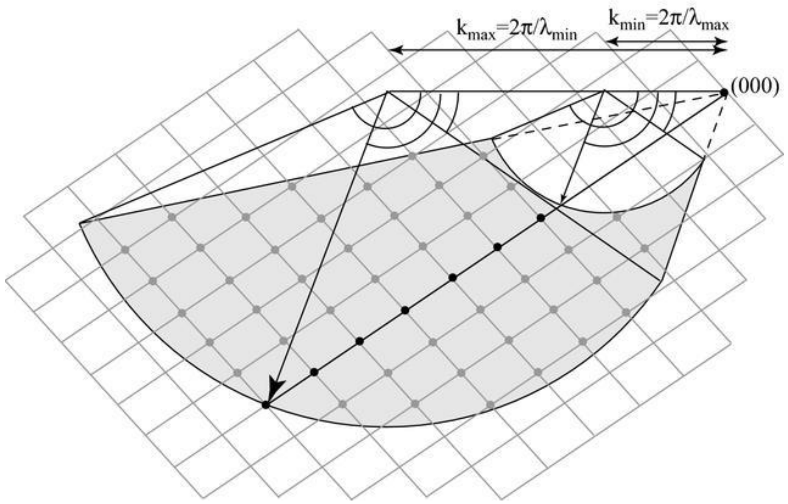


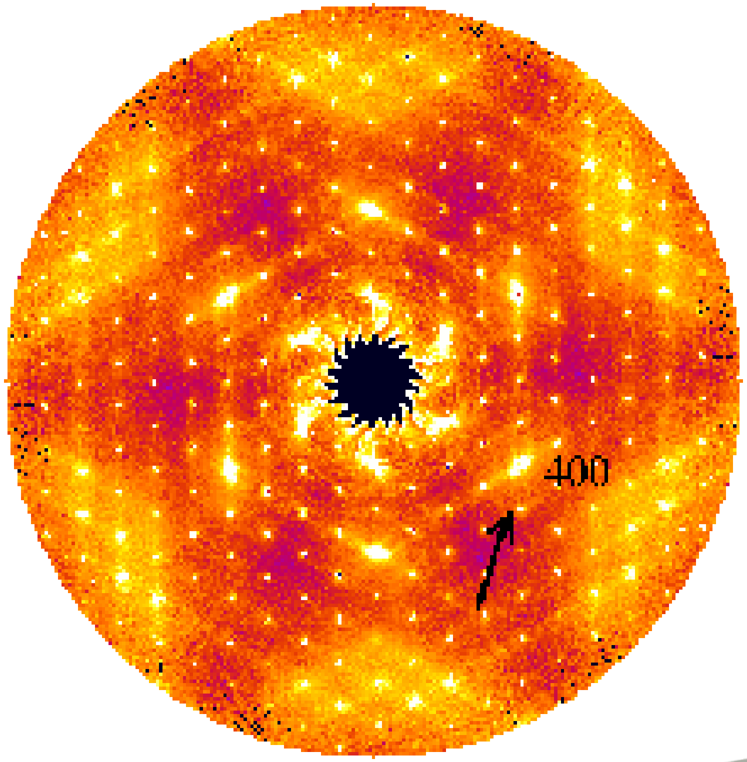
- chemical crystallography
- Q space mapping
- incommensurate structures
- high pressure

TOF Laue method

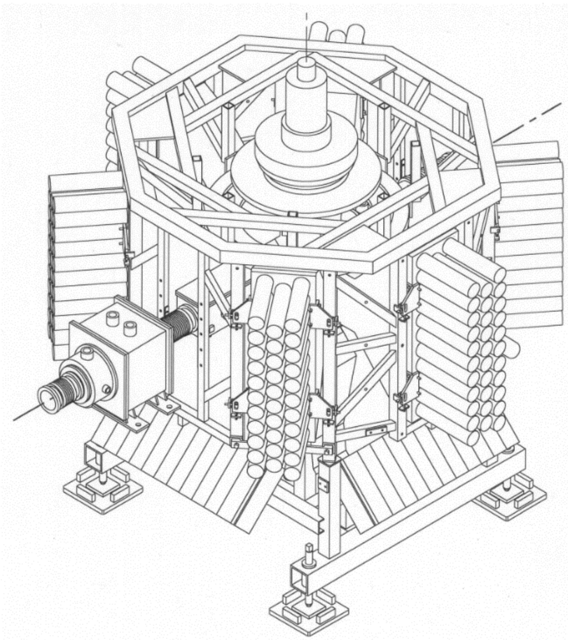
SXD uses the ‘time-of-flight Laue’ method to scans a large volume of reciprocal space at each crystal orientation.





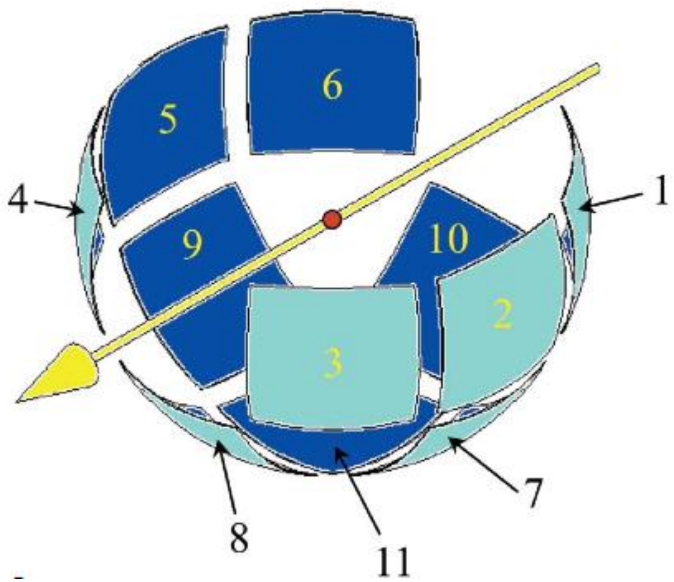


SXD: single crystal diffraction



SXD: single crystal diffraction

- H_2O moderator poisoned at 2 cm
- 0.2 – 10 Å wavelength band
- Primary flight path 8.3 m
- Beam size < 15 mm
- Eleven 192 × 192 mm^2 detectors (3 × 3 mm^2 resolution)



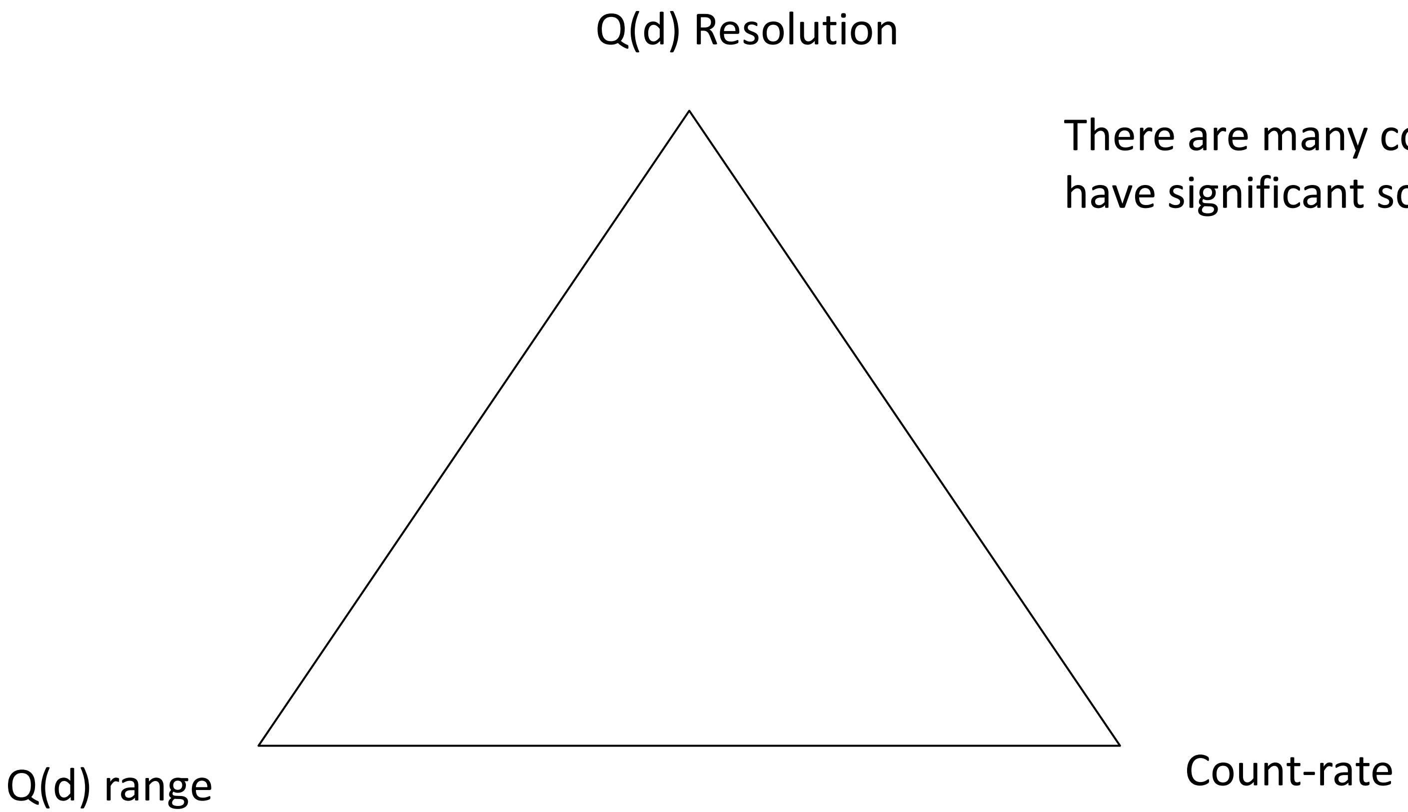
Unlike an image plate set-up the detectors are continuously read-out as a function of TOF allowing spatial overlap to be resolved in the TOF channel while minimising background

Keen *et al.* J. Appl. Cryst. (2006), **39**, 714-722) http://www.isis.stfc.ac.uk/instruments/sxd/sxd4813.html

Contemporary instruments: Topaz (SNS), Senju (J-Parc), Mandi (SNS)

Science & Technology Facilities Council ISIS

Why so many diffractometers?



There are many combinations that have significant science cases.

Diffractometer design: final words

Define a science case!!

Instruments are designed and built to perform science

The science case determines requirements

Instruments are designed and constructed to meet those requirements