FRM II
Forschungs-Neutronenquelle
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MLZ
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Neutron diffraction

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Properties of neutrons

- 1932: Discovery of neutrons

James Chadwick
Nobel prize in physics 1935

James Chadwick
1891–1974

$^{9}\text{Be} + {}^{4}\text{He} \rightarrow {}^{12}\text{C} + n$

Neutrons are
- Electrically neutral
- Isotope-sensitive
- Spin sensitive
- Motion sensitive

Wave–particle duality (DeBroglie)

$E = m_n v^2/2 = k_B T = (\hbar k)^2/2m_n$

$\mathbf{k} = 2\pi/\lambda = m_n \mathbf{v}/\hbar$

	Energy
cold neutrons:	E = 1 meV
	E = 5 meV
thermal neutrons:	E = 25 meV
	E = 50 meV

Wavelength	n-Wavevector	Velocity	Frequency
l = 9.0446 Å	k = 0.6947 1/Å	v = 437 m/s	v = 0.2418 THz
l = 4.0449 Å	k = 1.5534 1/Å	v = 978 m/s	v = 1.2090 THz
l = 1.8089 Å	k = 3.4734 1/Å	v = 2187 m/s	v = 6.045 THz
l = 1.2791 Å	k = 4.9122 1/Å	v = 3093 m/s	v = 12.090 THz

Neutron energies

- Typical fission energies: 1-2 MeV are too high for practical applications

– **hot neutrons:**

moderated at 2000C

0.1-0.5 eV, 0.3-1 Å, 10 000 m/s

– **thermal neutrons:**

moderated at 40° C

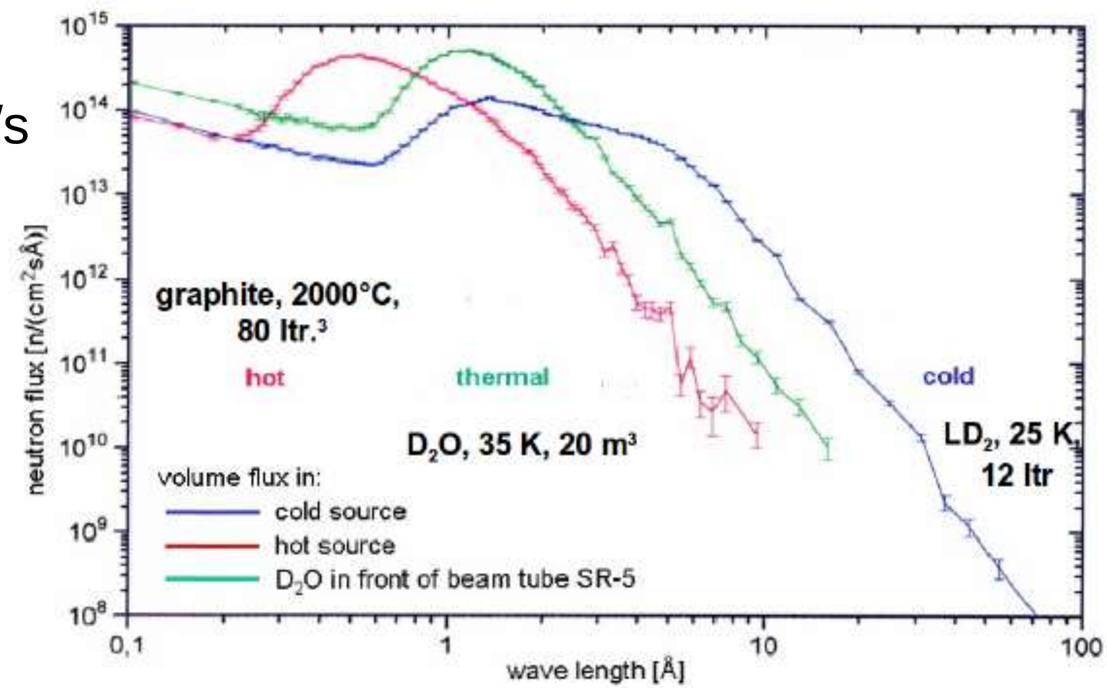
0.01-0.1 eV, 1-4 Å, 2000 m/s

– **cold neutrons:**

moderated at -250° C

0-0.01 eV, 0-30 Å, 200 m/s

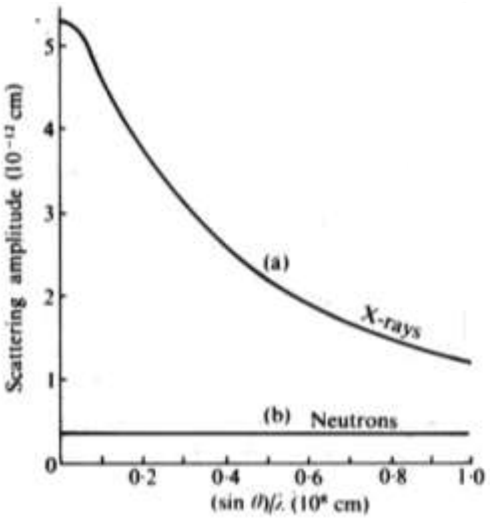
Spectral distribution of neutrons at FRM II



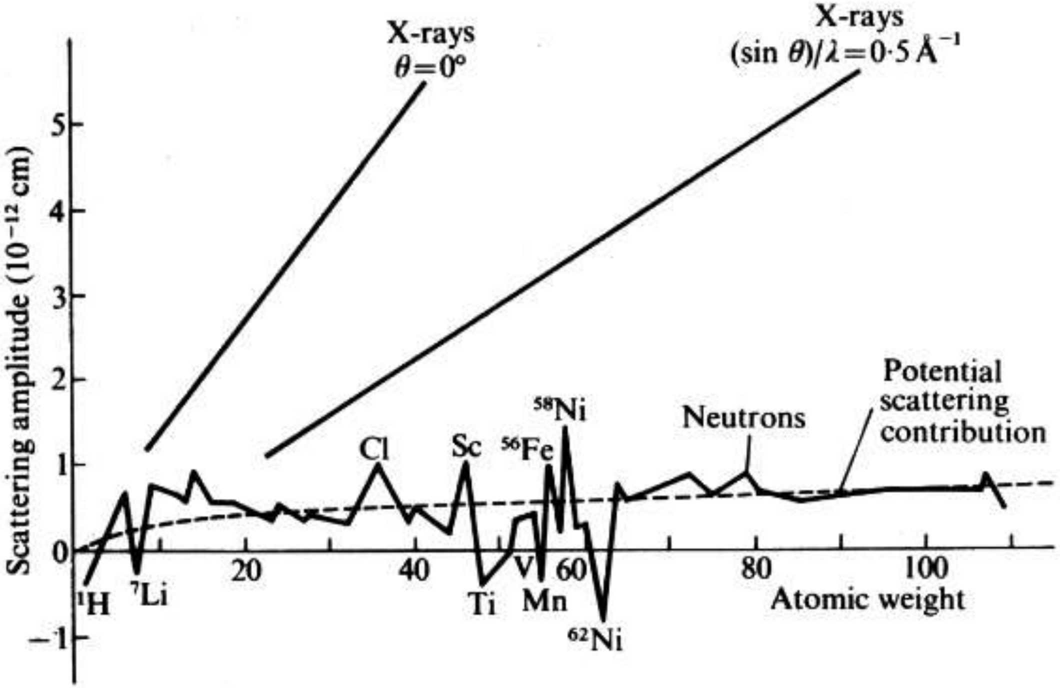
- under-moderated cold source provides also short wavelength $\lambda \geq 1$ Å
- on the expense of intensity at very long wavelength $\lambda \geq 15$ Å

Neutrons vs. X-rays

Particle wave	Electromagnetic wave
Mass	No mass
Spin ½	spin 1
Magnetic dipole moment	No magnetic dipole moment
Neutrons interact with the nucleus	X-ray photons interact with the electrons
Scattering power independent of 2θ	Scattering power falls off with 2θ



Neutron scattering length



x10	σ_{coh} [barn]		σ_{coh} [barn]	isotopes
	0.66	H 1	1.76	1, 2
	24	C 6	5.55	
	416	Mn 25	1.75	
	450	Fe 26	11.22	
	522	Ni 28	13.30	58, 60, 62
	1408	Pd 46	4.39	
	2986	Ho 67	8.06	
	5631	U 92	8.90	

Advantages of neutron scattering

The energy of thermal neutrons is in range of meV → Neutrons weakly perturb the experimental system, i.e. non-destructive.

Neutrons are deeply penetrating into the matter → Studies of bulk samples or processes under realistic conditions (in complex environments).

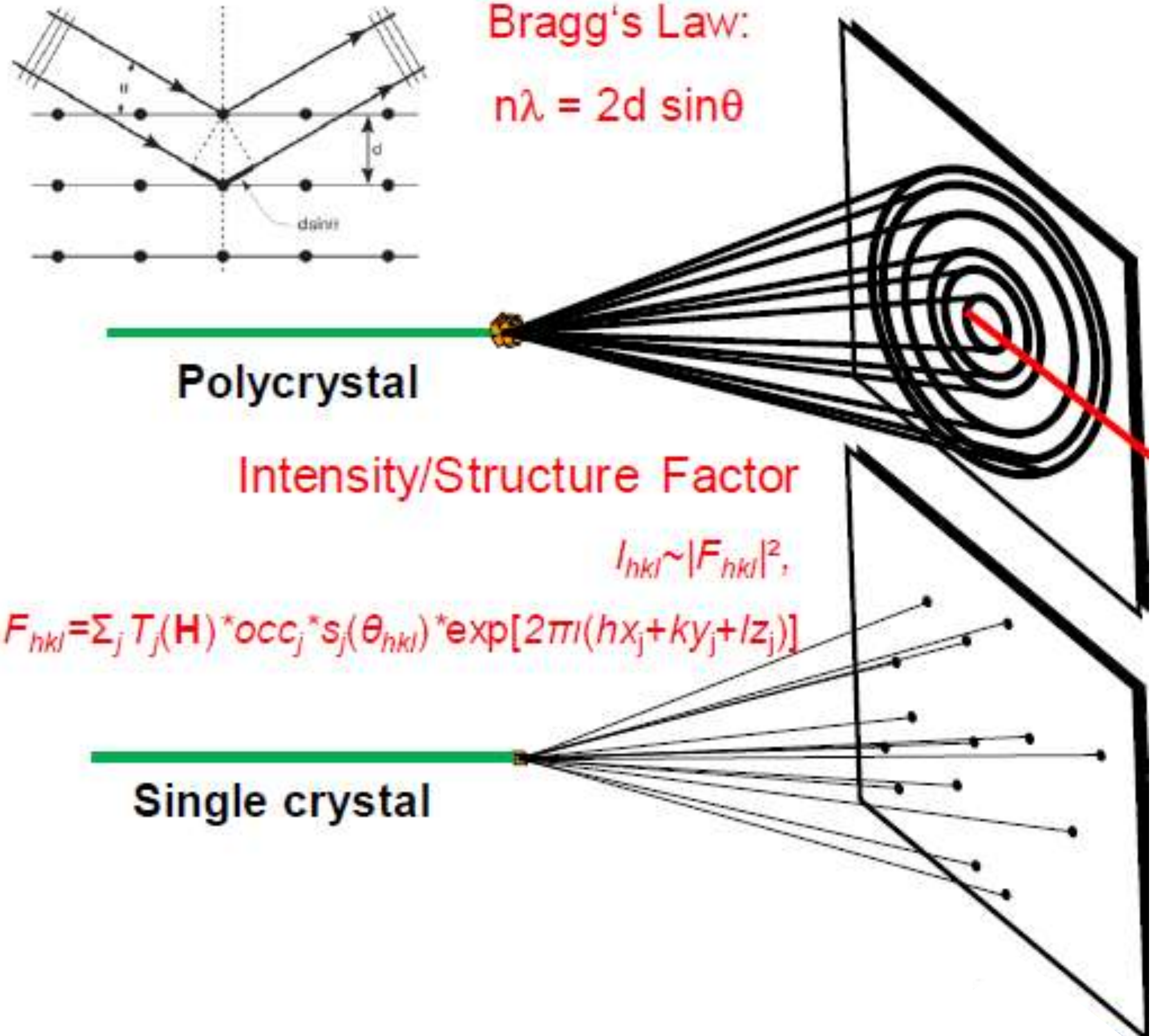
Neutrons interact with nucleous (strong force interaction) → Neutrons can localize light atoms (e.g. hydrogen, lithium) in the presence of heavier ones and to distinguish isotopes (additional contrast) and neighboring elements from Periodic Table. Accurate Debye-Waller (displacement parameter) determination.

The wavelength of thermal neutrons is similar to interatomic spacings. Neutron scattering length not depending on momentum transfer ($\sin(\theta)/\lambda$). → Details of the crystal structure. Studies of bulky samples – better particle average. Accurate lattice parameters and atomic coordinates, effects of microstructure.

Diffraction on powders and single crystals

Bragg's Law:

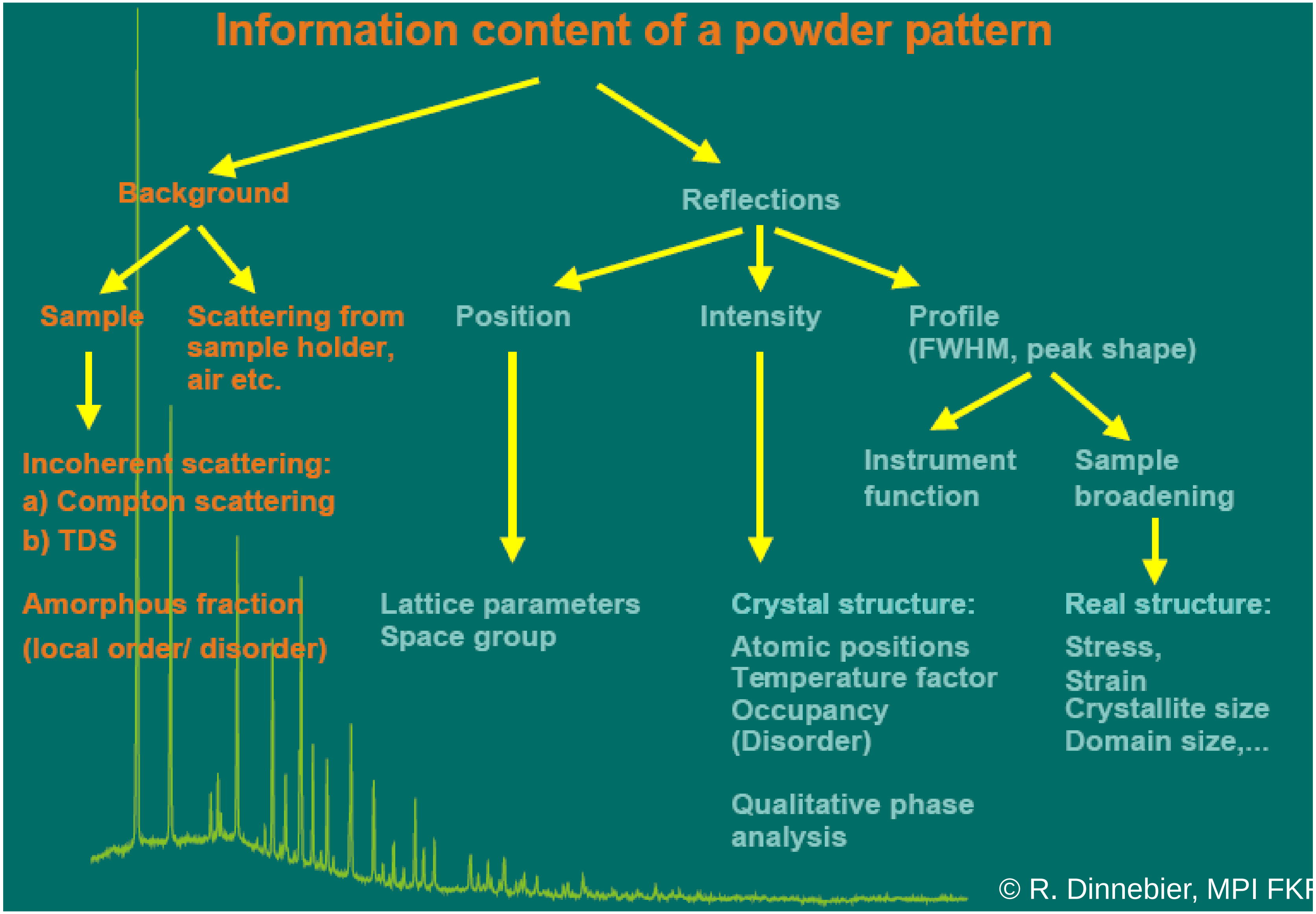
$n\lambda = 2d \sin\theta$



Intensity/Structure Factor

$I_{hkl} \sim |F_{hkl}|^2,$

$F_{hkl} = \Sigma_j T_j(\mathbf{H}) * occ_j * s_j(\theta_{hkl}) * \exp[2\pi i(hx_j + ky_j + lz_j)]$

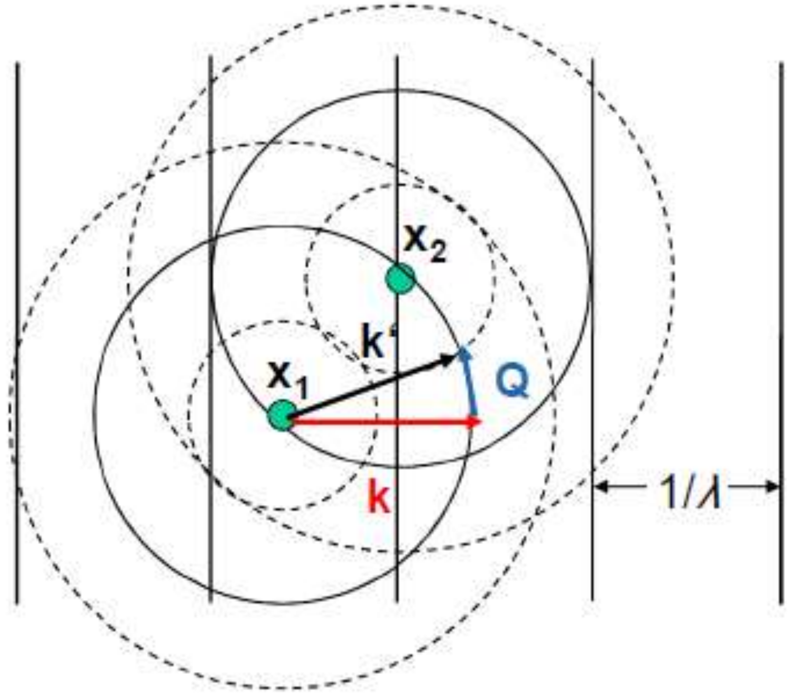

Information content of a powder pattern
Background
Reflections
Sample
Scattering from sample holder, air etc.
Position
Intensity
Profile (FWHM, peak shape)
Incoherent scattering:
a) Compton scattering
b) TDS
Instrument function
Sample broadening
Amorphous fraction
(local order/ disorder)
Lattice parameters
Space group
Crystal structure:
Atomic positions
Temperature factor
Occupancy
(Disorder)
Real structure:
Stress,
Strain
Crystallite size
Domain size,...
Qualitative phase analysis
© R. Dinnebier, MPI FKF

Elastic scattering on crystal lattice

more than one scatterer,
elastic scattering: |k‘| = |k|

Interference with **k‘ = k + Q**
Q: scattering vector

phase shift between spherical waves
along **k** : $\delta = \mathbf{k}\,(\mathbf{x}_2 - \mathbf{x}_1)/|\mathbf{k}|$
along **k‘**: $\delta' = \mathbf{k}'(\mathbf{x}_2 - \mathbf{x}_1)/|\mathbf{k}'|$
$\rightarrow \delta' - \delta = \mathbf{Q}(\mathbf{x}_2 - \mathbf{x}_1)$



Scattering Amplitude $s(\mathbf{Q}) = s_1 \exp(i\mathbf{Q}\mathbf{x}_1) + s_2 \exp(i\mathbf{Q}\mathbf{x}_2)$ for 2 scatterers

n scatterers in given volume: $s(\mathbf{Q}) = \sum_{j=1}^{n} s_j \exp(i\mathbf{Q}\mathbf{x}_j)$

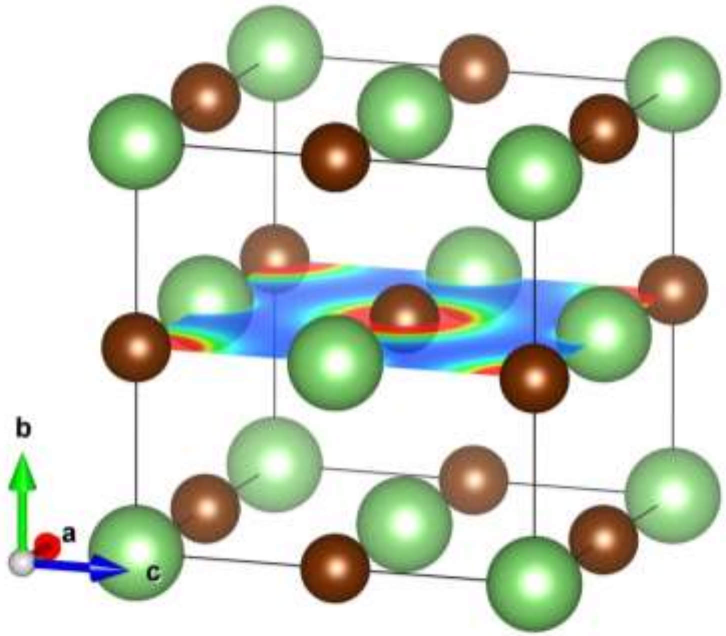
⇒ Structure factor $F(Q) \sim \sum_{i=1}^{n_{EZ}} s_i(Q) e^{i\vec{Q}\vec{x_i}}$

Concept of scattering density

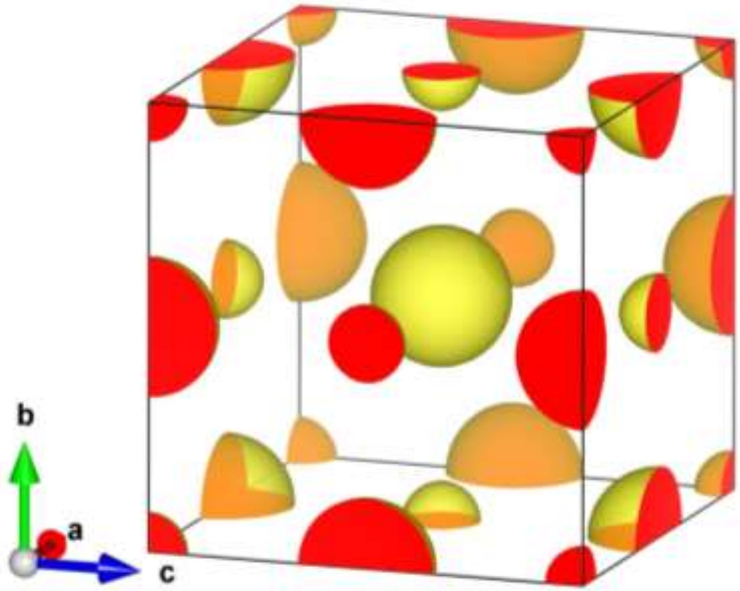
„Diffraction is not measuring atomic positions, but periodic scattering densities averaged in time and space"

$$\rho_{xyz} = \frac{1}{V} \sum_{h=-\infty}^{h=+\infty} \sum_{k=-\infty}^{k=+\infty} \sum_{l=-\infty}^{l=+\infty} \left| \mathbf{F}_{hkl}^{obs} \right| \cos\left[2\pi (hx + ky + lz) - \alpha_{hkl} \right]$$

Example: crystal structure of LiBr

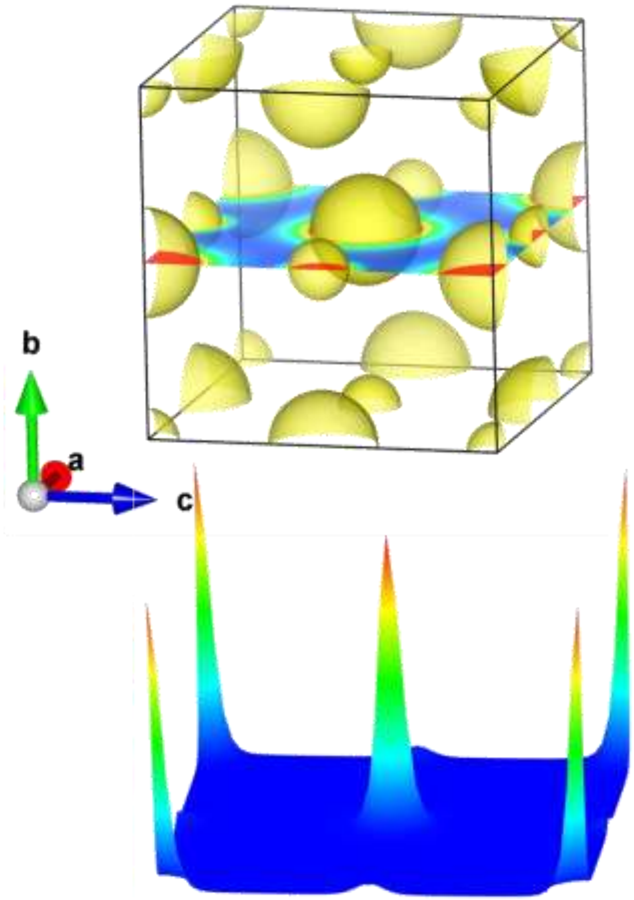


Simulated electron densities

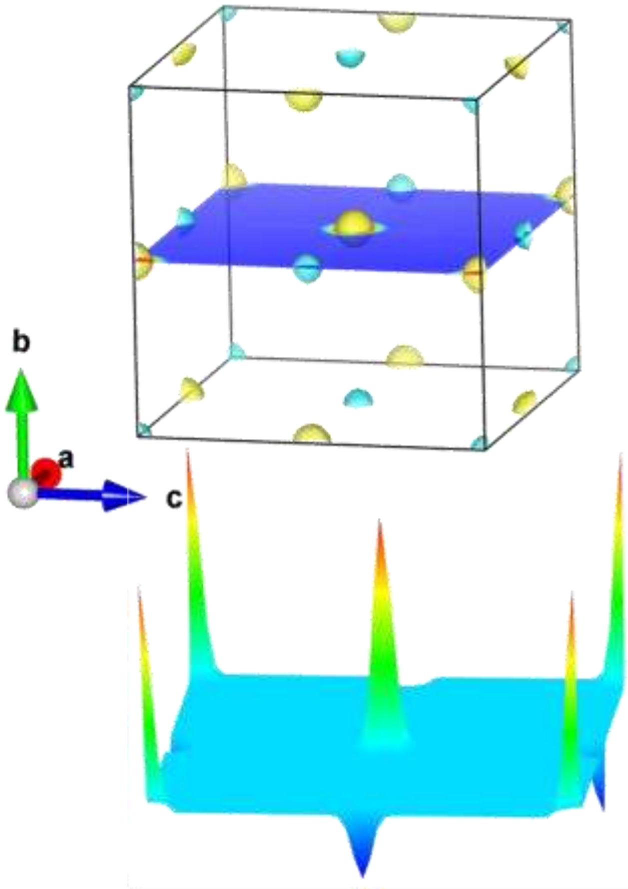


Electron vs. Nuclear densities (X-ray vs. Neutron diffaction)

Simulated electron densities



Simulated nuclear densities



Elastic scattering on crystal lattice

$$\mathbf{F}(\mathbf{h}) = \sum_{j=1}^{n} g^j t^j(s) f^j(s) \exp(2\pi i \mathbf{h} \cdot \mathbf{x}^j)$$

Structure amplitude

Total number of atoms

$s=\sin(\theta_{hkl})/\lambda$

Scattering factor

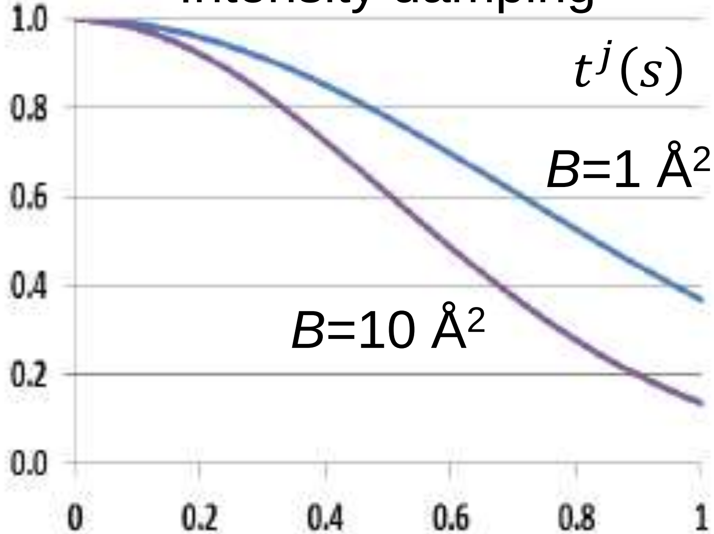
h=[h,k,l]

Occupation factor

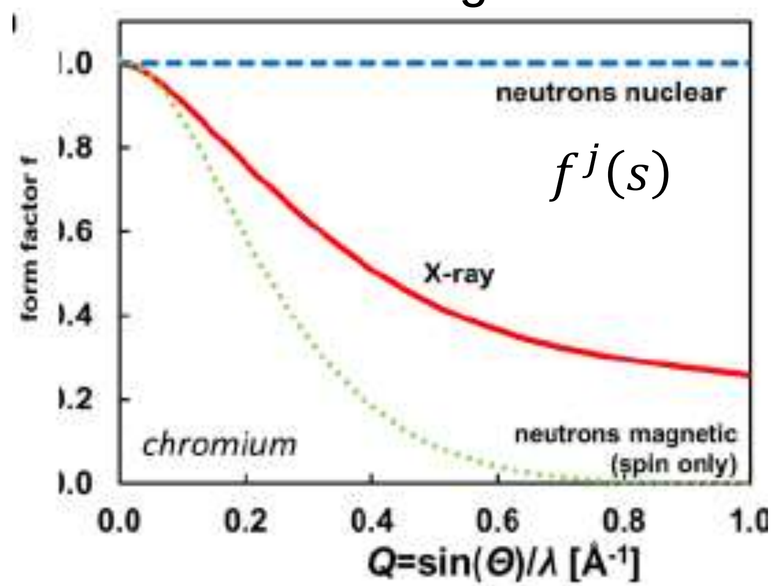
Temperature factor

x=[x,y,z]

Intensity damping



Scattering factor



Limitations of diffraction

1. Time and space(volume) averaged information

2. Phase problem

Since F_{hkl} is an amplitude

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

In general, F is imaginary ....
so

$$F = A + iB$$

and

$$F^*F = (A - iB)(A + iB)$$

3. Resolution effects $2\sin(\theta)/\lambda = 1/d_{min}$

limited info on *F*(Q) →
limited accuracy for *s*(x)!

A. Blake, W. Clegg, et al.; IUCr Book Series, Oxford University Press (2009)

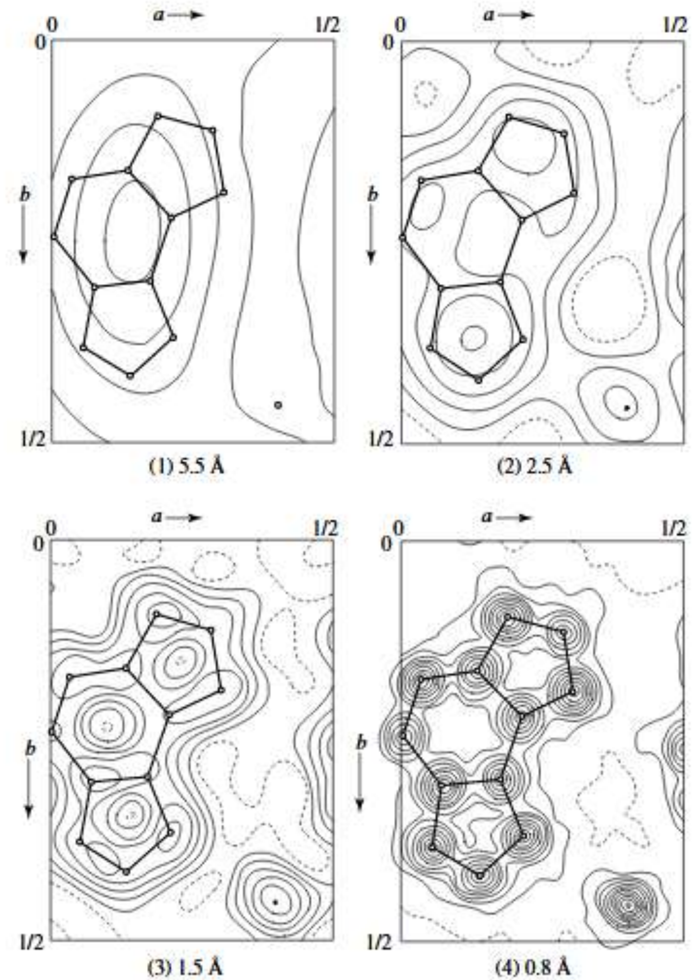
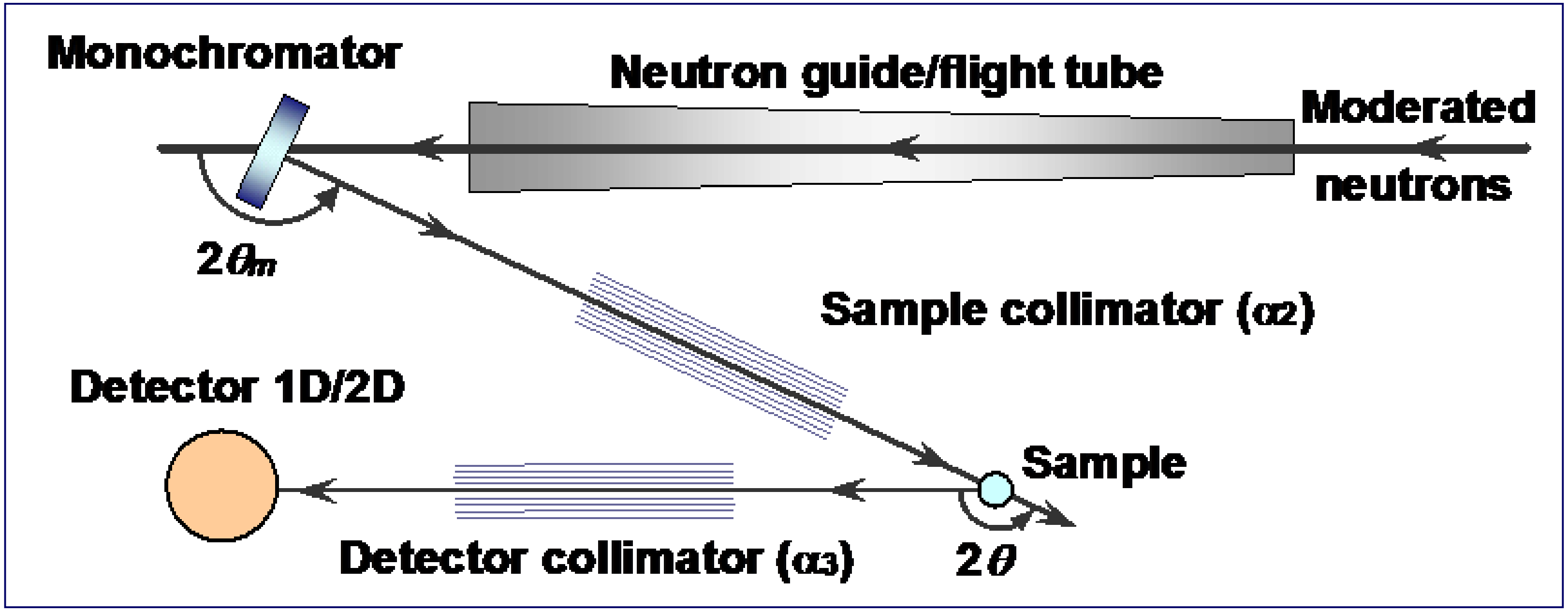
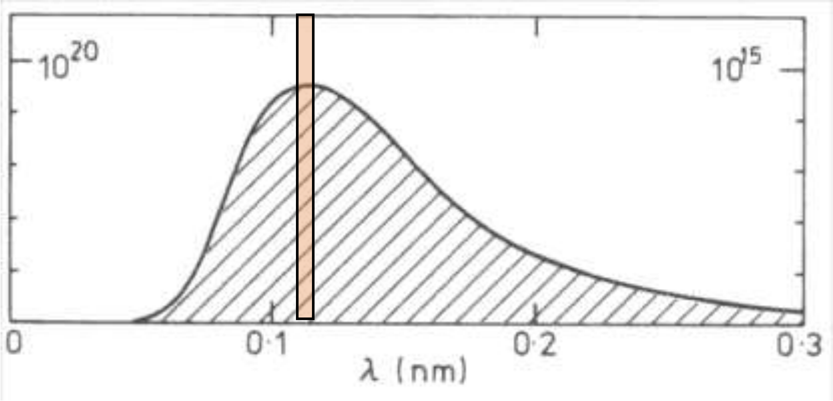


Fig. 1.9 The electron density calculated from a diffraction pattern of limited extent, indicated by the decreasing values of d_{min} from (1) to (4).

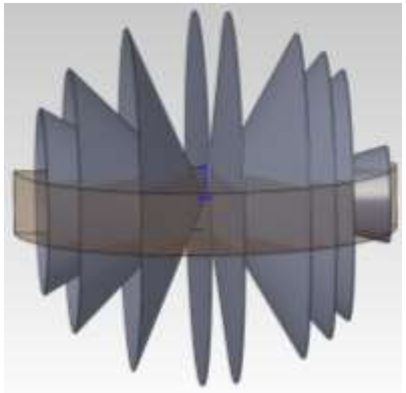
Schematics of two-axis diffractometer



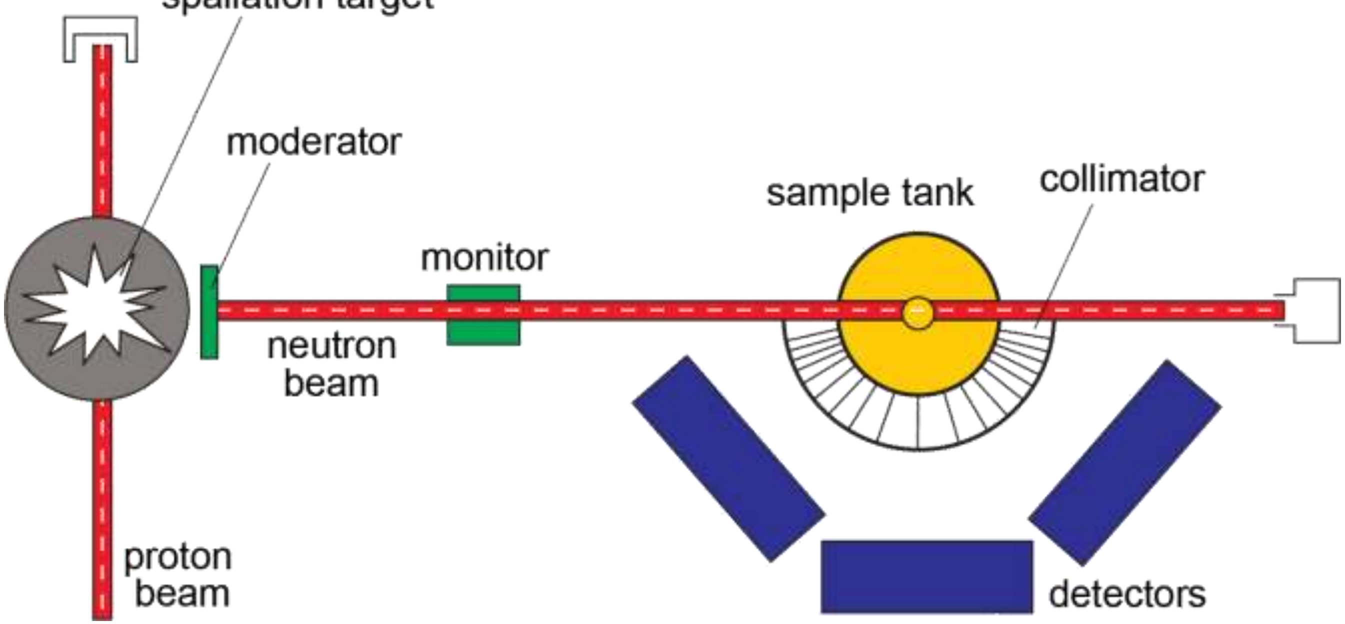
Reactor flux



Diffraction signal in space

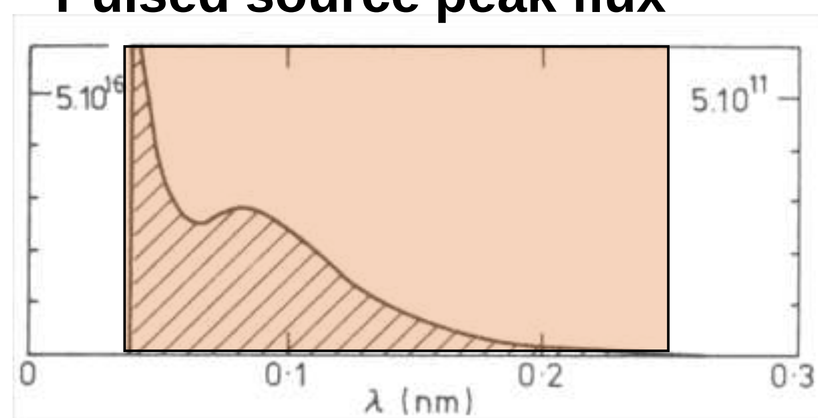


Neutron diffraction (time-of-flight)



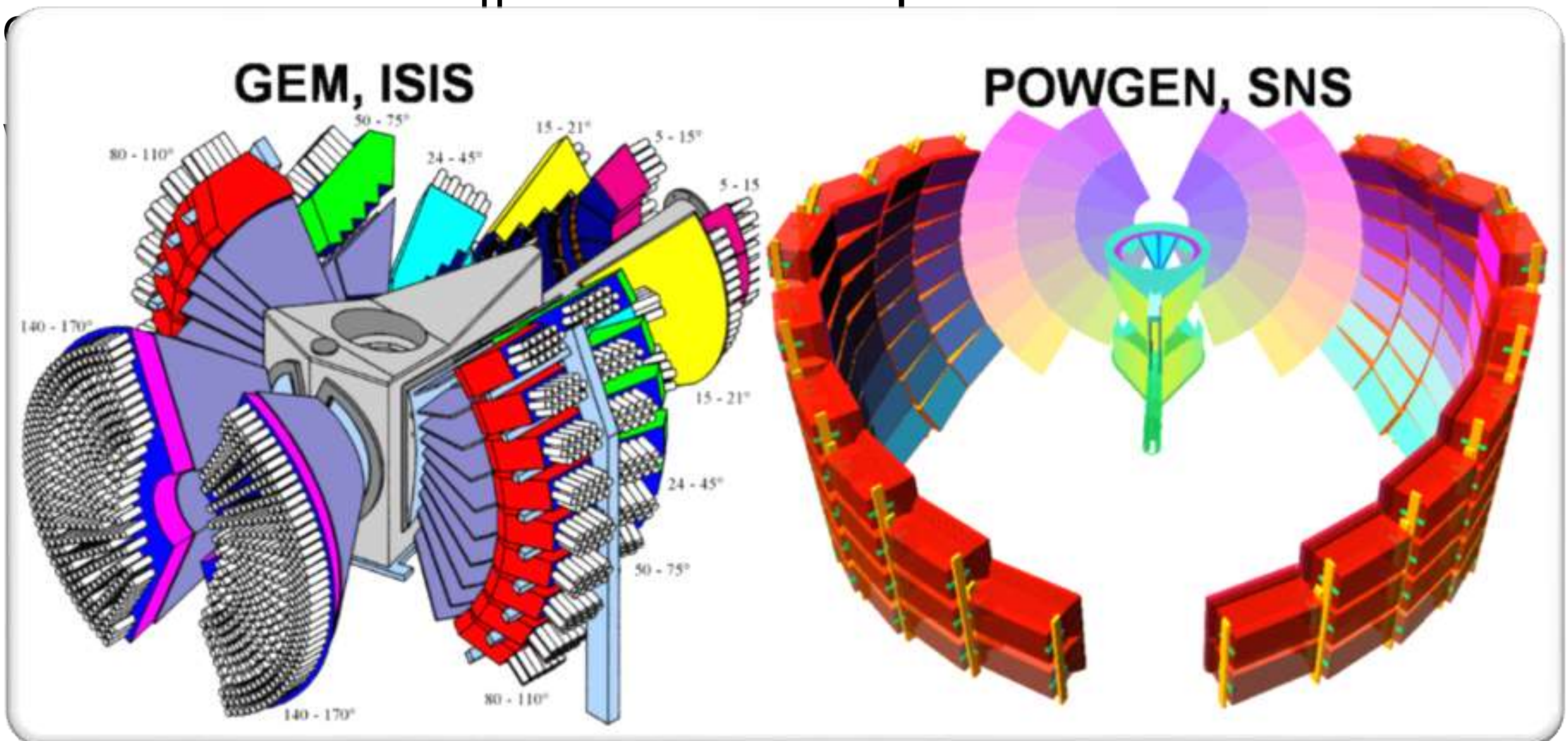
t – time of flight
L – total flight path
$\sin\theta$ – diffraction angle
d_{hkl} – lattice d-spacing

Pulsed source peak flux



- We are measuring d, so two variables, θ and λ;
- In lab X-ray diffraction, λ is constant, θ variable;
- In time-of-flight (t.o.f), θ is constant, λ variable;

Neutron powder diffraction (time-of-flight)



Use many separate detectors and sum the counts recorded in each to measure I(Q) with good counting statistics in less time

Single Crystal Diffraction Activities at MLZ