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School on Pulsed Neutrons: Characterization of Materials

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Neutron Sources & Scattering Techniques (3-4)

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The Abdus Salam
International Centre for Theoretical Physics

School on **Pulsed Neutron Sources: Characterization of Materials**
15-26 October 2007
Miramare, Trieste, Italy

Introductory Lecture:

Basics Concepts in Neutron Scattering Techniques and Neutron Sources (3-4)

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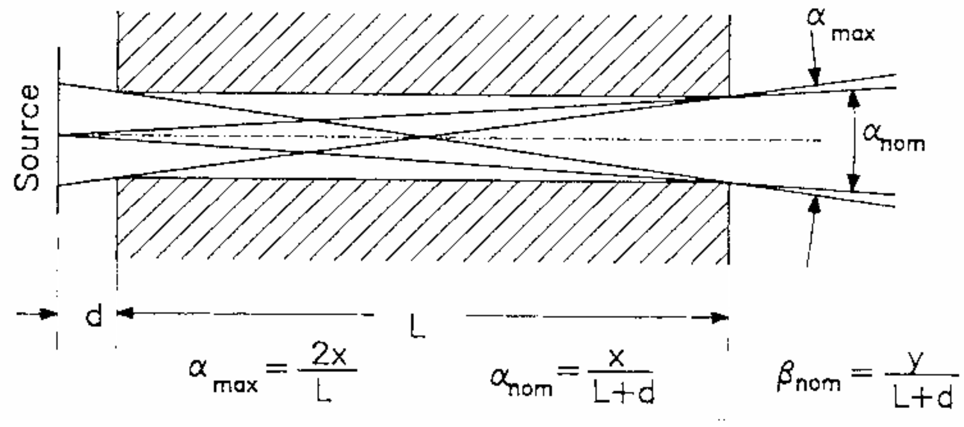
Part 3

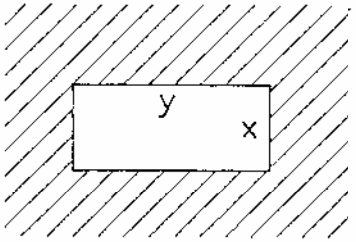
Phase Space Operators

The resolution of an instrument is controlled by phase space operators (devices that affect the magnitude and directions of $\underline{k}$ and $\underline{k}$')

PSOs Affecting k_x and k_y (perpendicular to $\underline{k}$) (1)

Beam holes and apertures





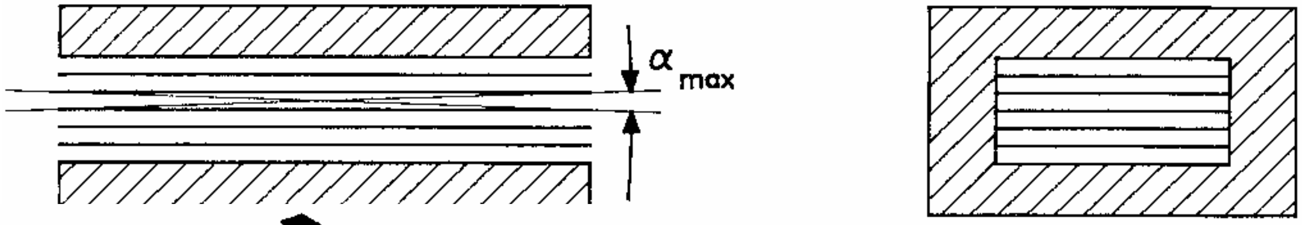
Solid angle:

$$d\underline{\Omega} = \frac{dxdy}{L^2} \cdot \frac{\underline{k}_z}{|\underline{k}_z|}$$

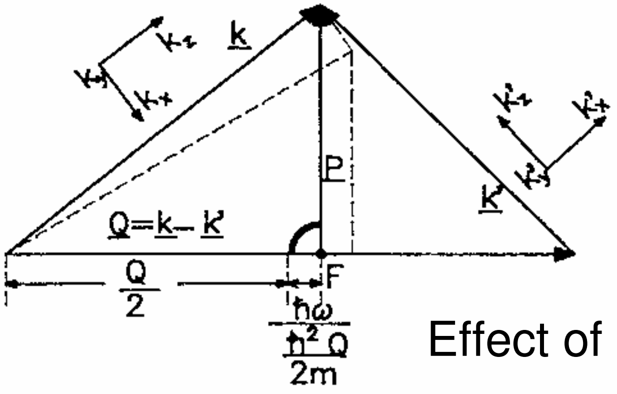
Transmitted intensity:

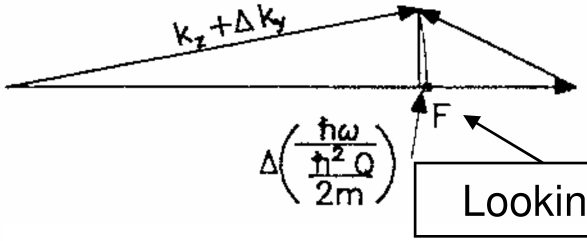
$$I(\underline{k}) = \int_{x,y} dxdy\phi(\underline{k})dk_x dk_y,$$

Soller collimators



The beam divergence in the direction perpendicular to the scattering plane affects the resolution only in 2nd order





Looking down along $\underline{P}$

Effect of a beam divergence in (left) and perpendicular to (right) the scattering plane

PSOs Affecting k_x and k_y (perpendicular to $\underline{k}$) (2)

Neutron guides

While the walls of a beam tube should be opaque for neutrons of all momenta, neutron guides are equipped with totally reflecting walls up to a certain value of $k_\perp$.

Refractive index: $n = v_M/v$

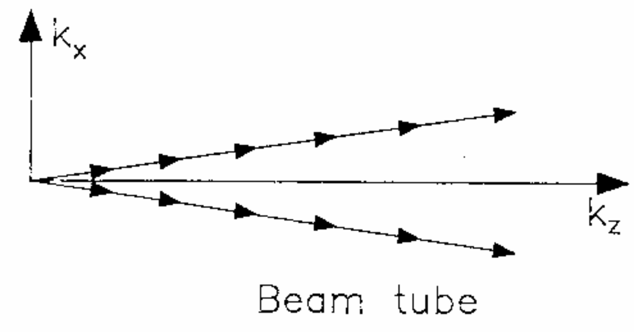
Using the Fermi pseudo potential $E=-2\pi(\hbar^2/m)Nb$

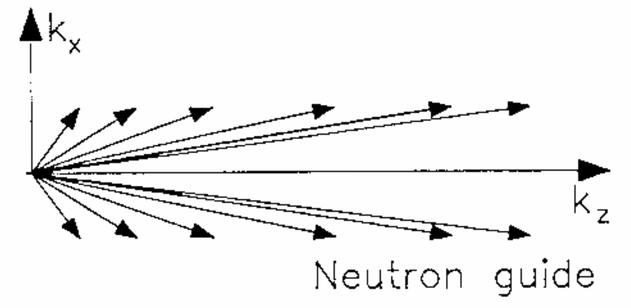
$$n = \frac{k_M}{k} = \sqrt{\frac{E_M}{E}} = \sqrt{\frac{E+\Delta E}{E}} \simeq 1 + \frac{1}{2}\frac{\Delta E}{E} \overset{\downarrow}{=} 1 - \frac{1}{2}\frac{2\pi\frac{\hbar^2}{m}\cdot N\cdot\bar{b}}{\frac{\hbar^2 k^2}{2m}} = 1 - \frac{2\pi}{k^2}\cdot N\bar{b} = \frac{\cos\gamma}{\cos\gamma_M}$$

Total reflection: $\cos\gamma_c \approx 1 - \frac{1}{2}\gamma_c^2 = n$

$$\Rightarrow \quad 1 - \frac{2\pi}{k^2}\cdot N\cdot\bar{b} = 1 - \frac{1}{2}\gamma_c^2 = 1 - \frac{1}{2}\left(\frac{\Delta k_x}{k}\right)^2$$

or $\Delta k^{max} = \sqrt{4\pi N\cdot\bar{b}}$ For the maximum value of $k_\perp$

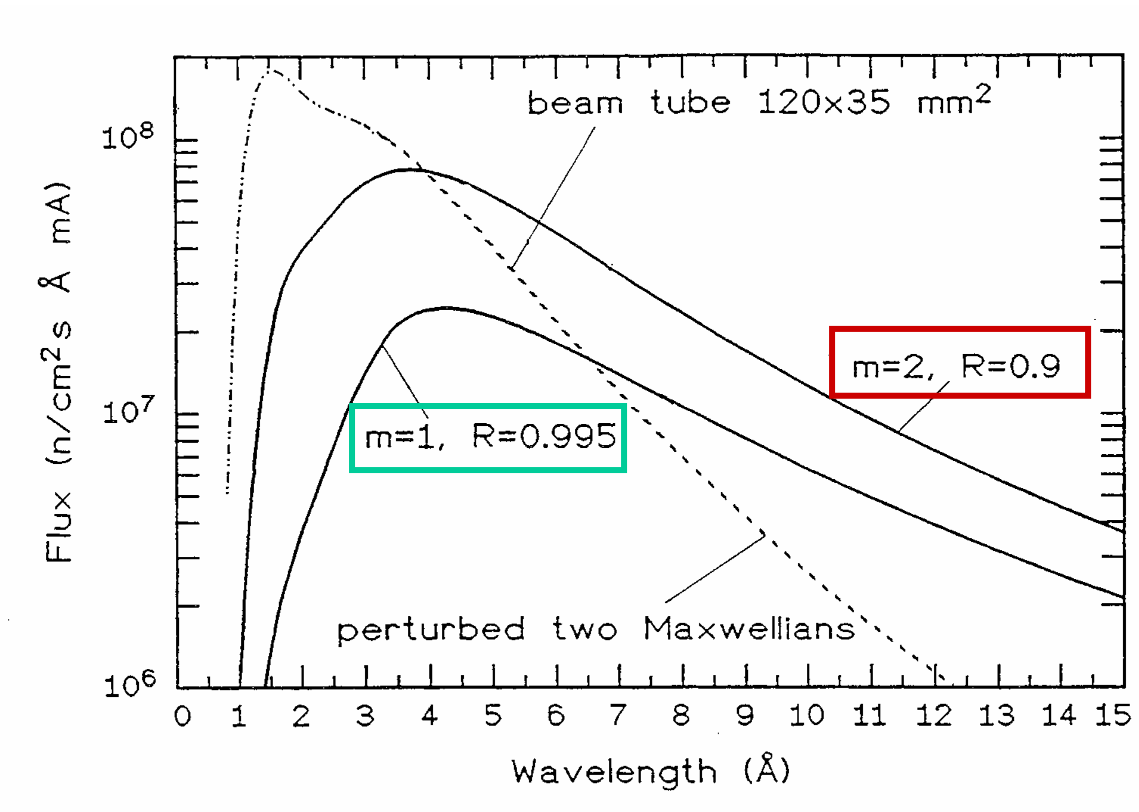




Modern neutron guides are equipped with *supermirrors*, which increase the critical angle to 3-4 times its value for natural nickel

PSOs Affecting k_x and k_y (perpendicular to <u>k</u>) (3)

Neutron guides (cntd.)



Calculated neutron spectra for the SINQ cold moderator for a 6.5 m long beam tube and 50 m long guides with m=1 (natural nickel) and m=2

The gain for m=2 boils down from a factor of 4 to a factor of two at longer wavelengths due to the poorer reflectivity R

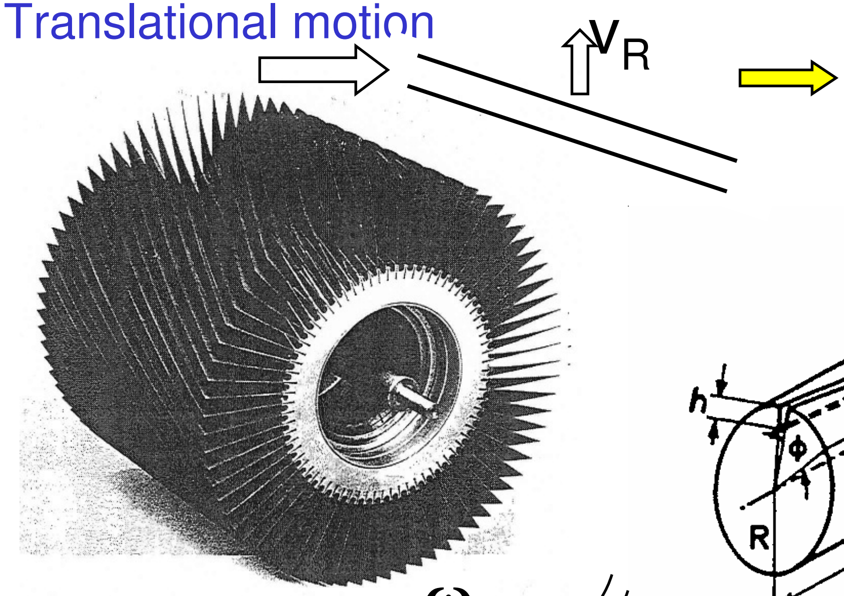
Neutron guides are usually curved in order to eliminate high energy neutrons from the transmitted spectrum. As a consequence the energy spectrum varies along the width of the guide.

A side effect of the lower reflectivity of supermirrors is the need for significantly more shielding along the length of the guides

PSOs Affecting k_x, (k_y) and k_z (1)

Moving collimators

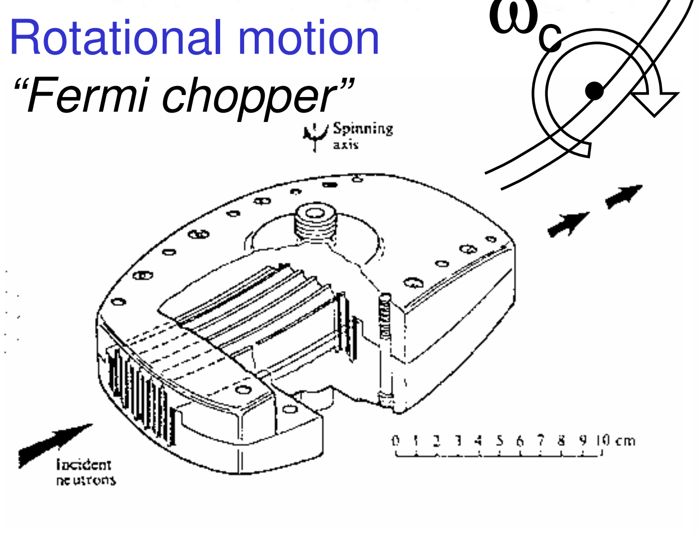
Translational motion

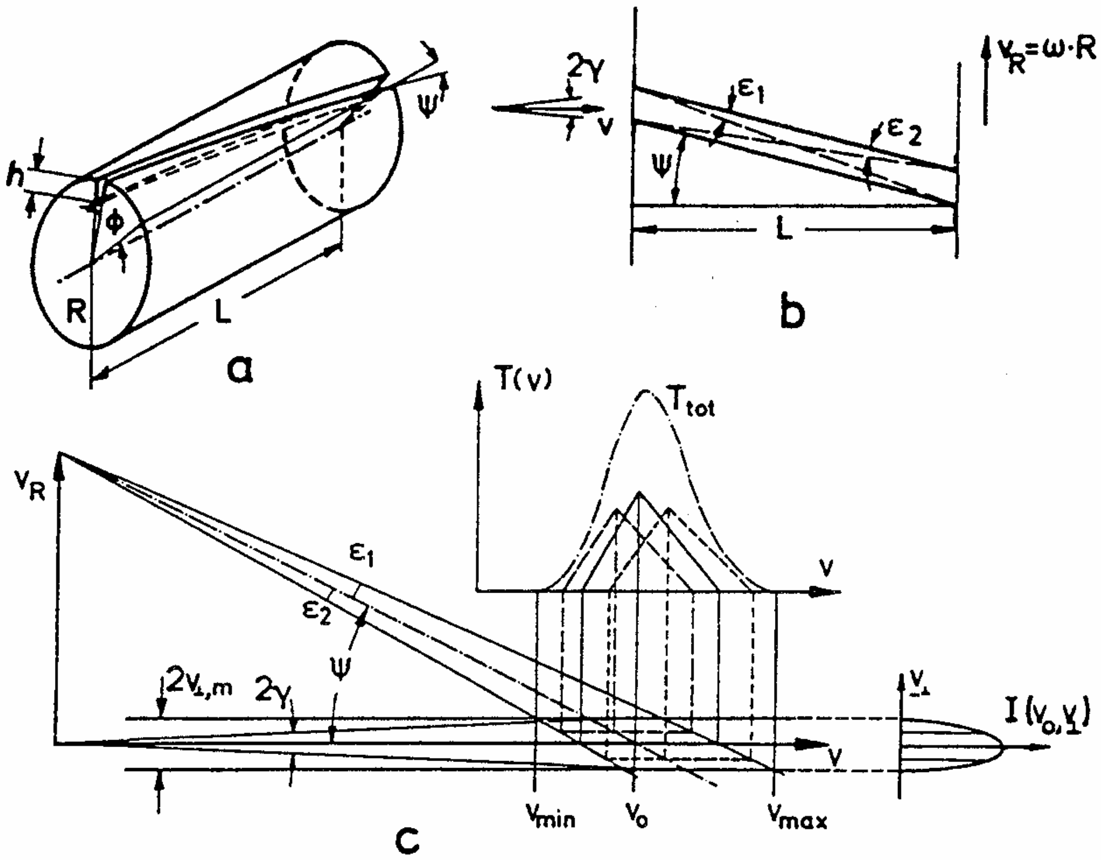


The resolution of a *mechanical velocity selector* can be varied (in certain limits) by changing its tilt angle ψ

Rotational motion

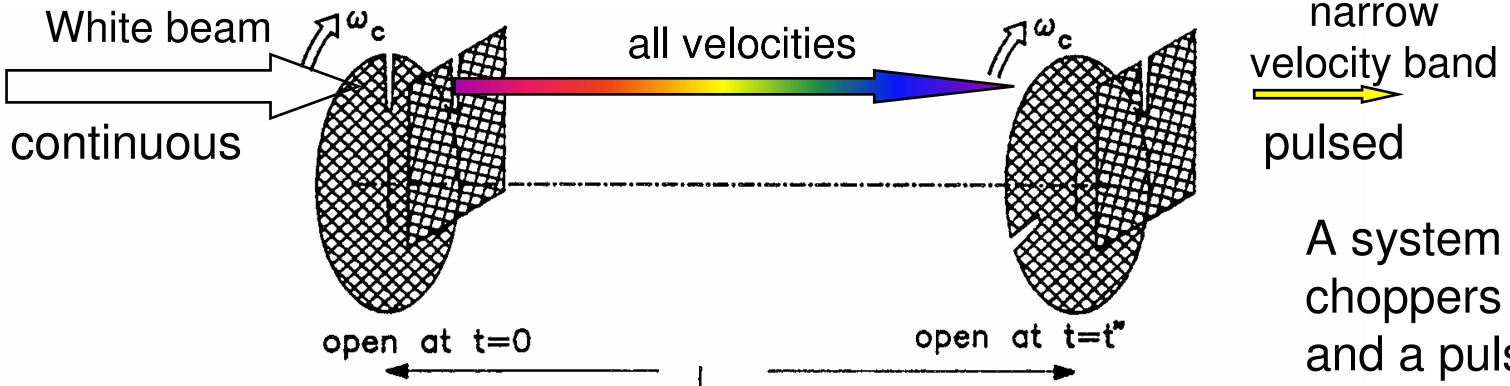
“Fermi chopper”





PSOs Affecting k_x, (k_y) and k_z (2)

The double chopper system

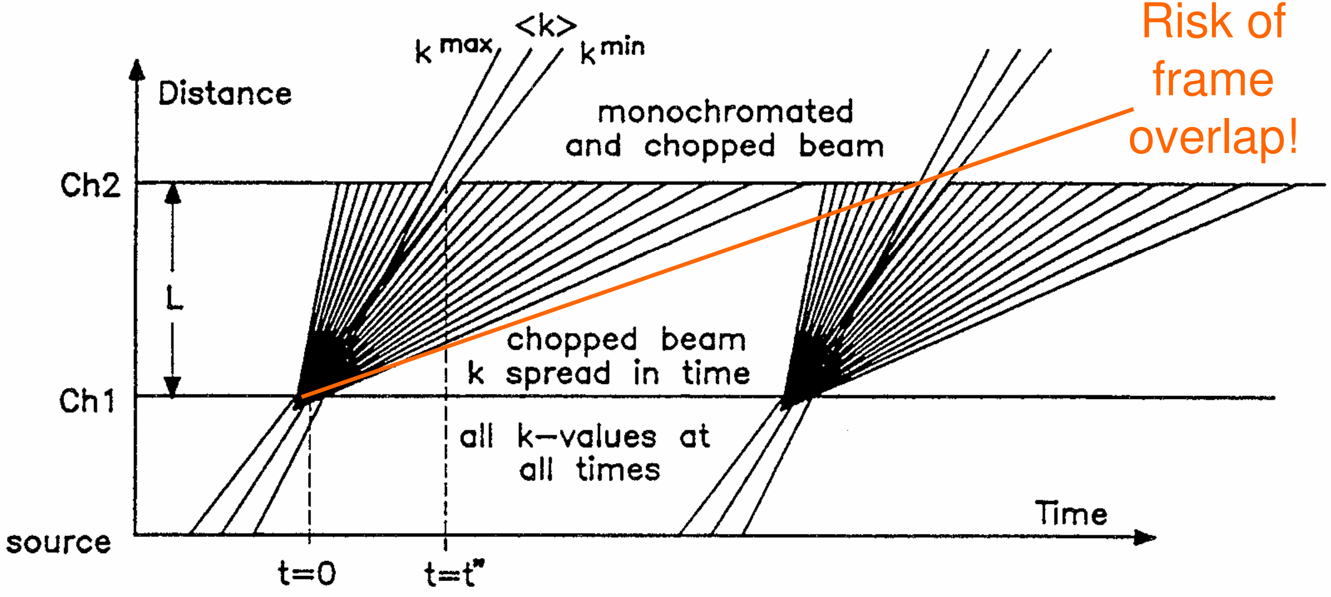


$$\langle k \rangle = \frac{m}{\hbar} \langle v_n \rangle = \frac{m}{\hbar} \frac{L}{t^*}$$

A system of two choppers (or a chopper and a pulsed source) can produce a pulsed beam of “monochromatic” neutrons (small Δk_z).

The chopper slits act as apertures limiting k_x (and k_y). A neutron guide between the choppers can reduce this effect.

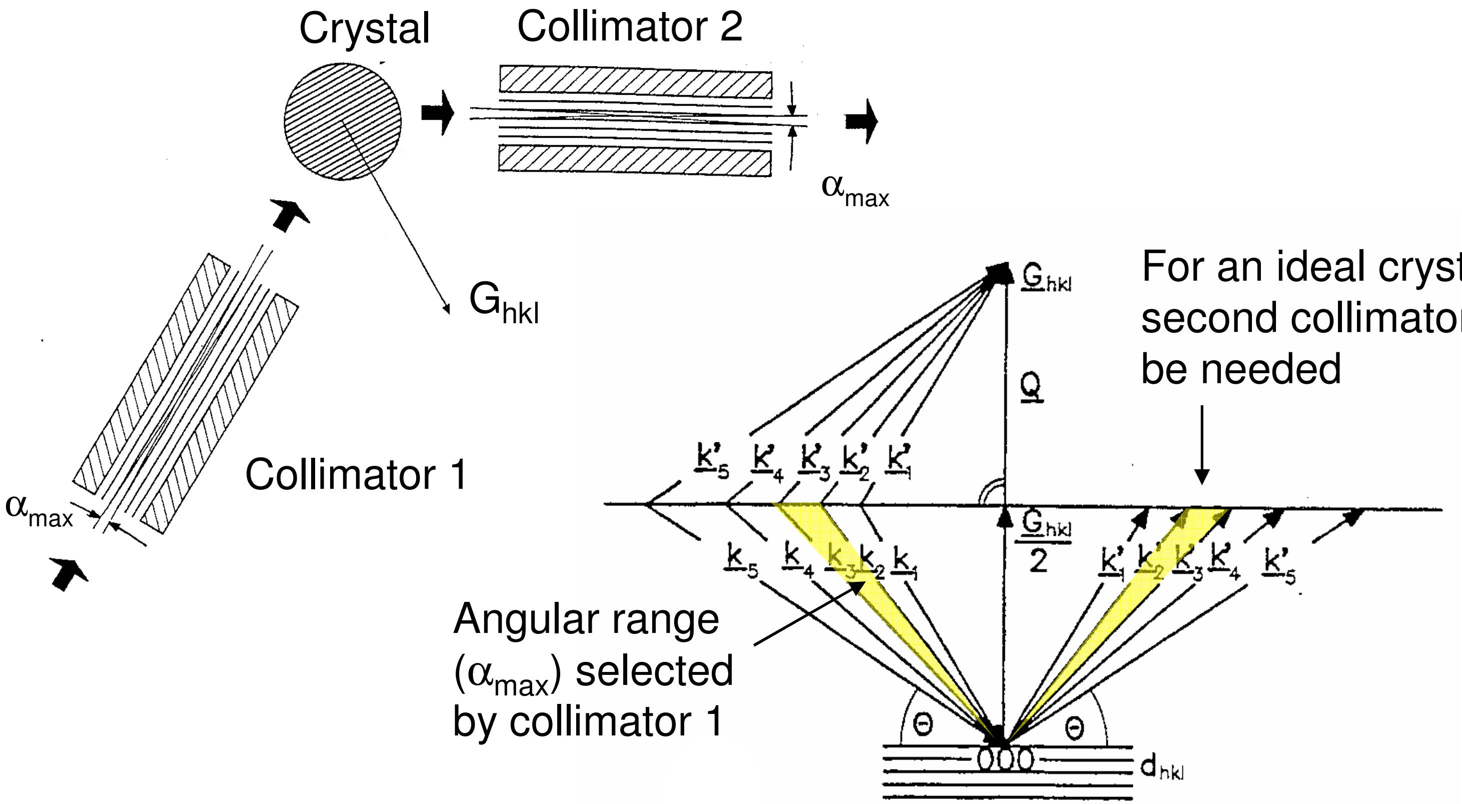
Space-time diagram



PSOs Affecting k_x, (k_y) and k_z (3)

Crystal monochromator systems

If a white beam impinges on a monocrystal, elastic Bragg scattering through an angle 2Θ occurs if the condition $\underline{Q}=\underline{G}_{hkl}$ is fulfilled. This means that, for a given d-spacing ($G_{hkl}=2\pi/d$) there is a unique relation between k_z and 2Θ, which must be selected by collimators in front of and behind the crystal.



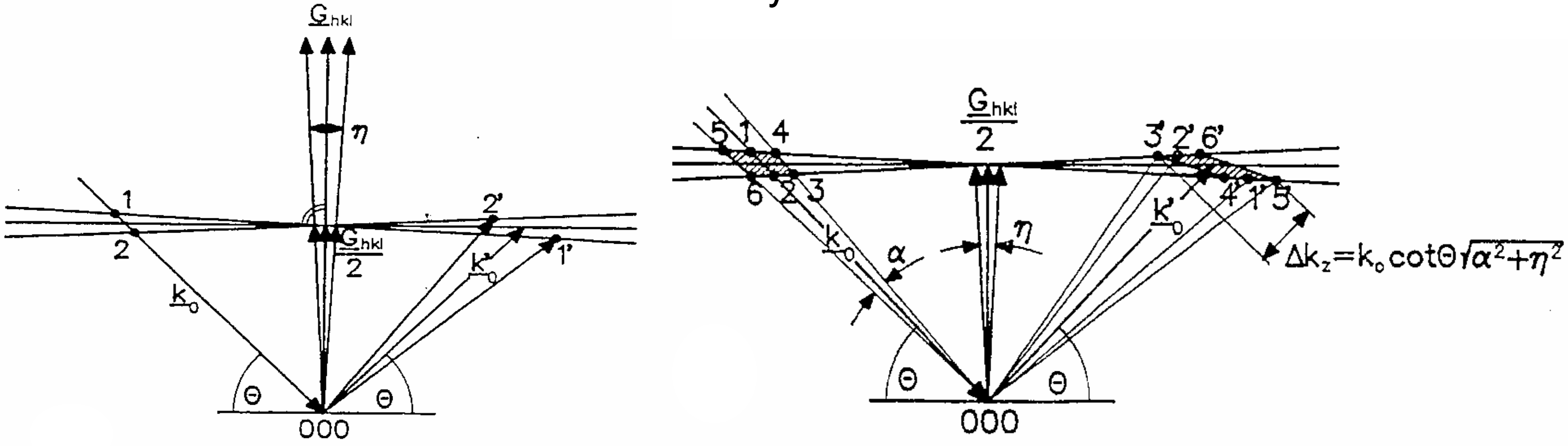
For an ideal crystal no second collimator would be needed

Angular range (α_{max}) selected by collimator 1

PSOs Affecting k_x, (k_y) and k_z (4)

Crystal monochromator systems (cntd.)

Real crystals have a “mosaic spread” η (angular uncertainty of G_{hkl}), which is desirable in order to obtain more intensity in the diffracted beam.



Effect of a mosaic spread η on the angular distribution of the diffracted beam in the case of an ideally collimated incident beam (left) and an incident beam with angular divergency α (right).

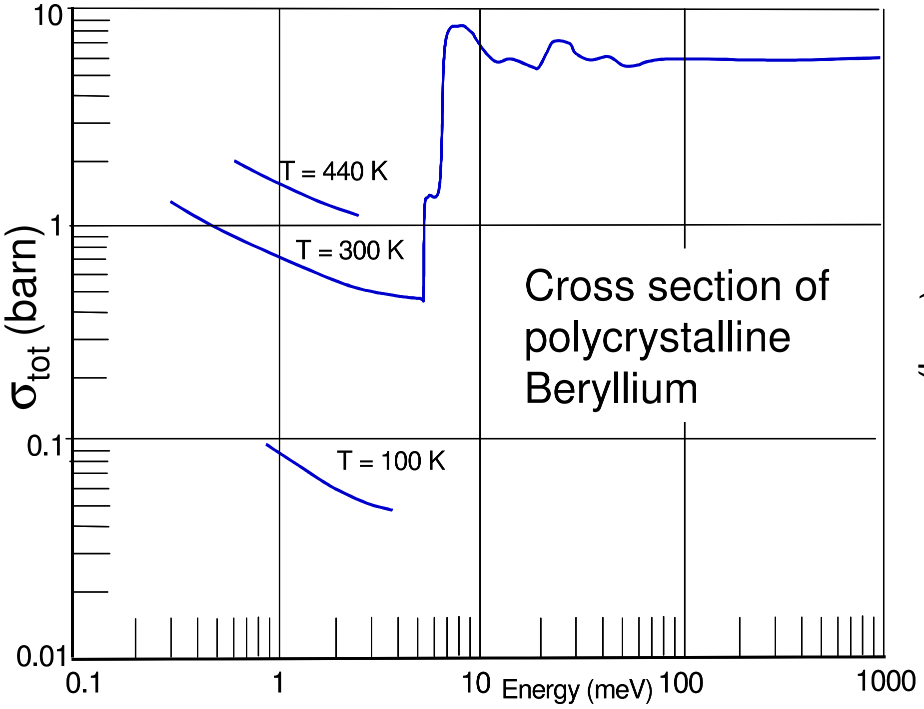
The total uncertainty in k_z of the diffracted beam is affected by η and α, as well as by the scattering angle 2Θ.

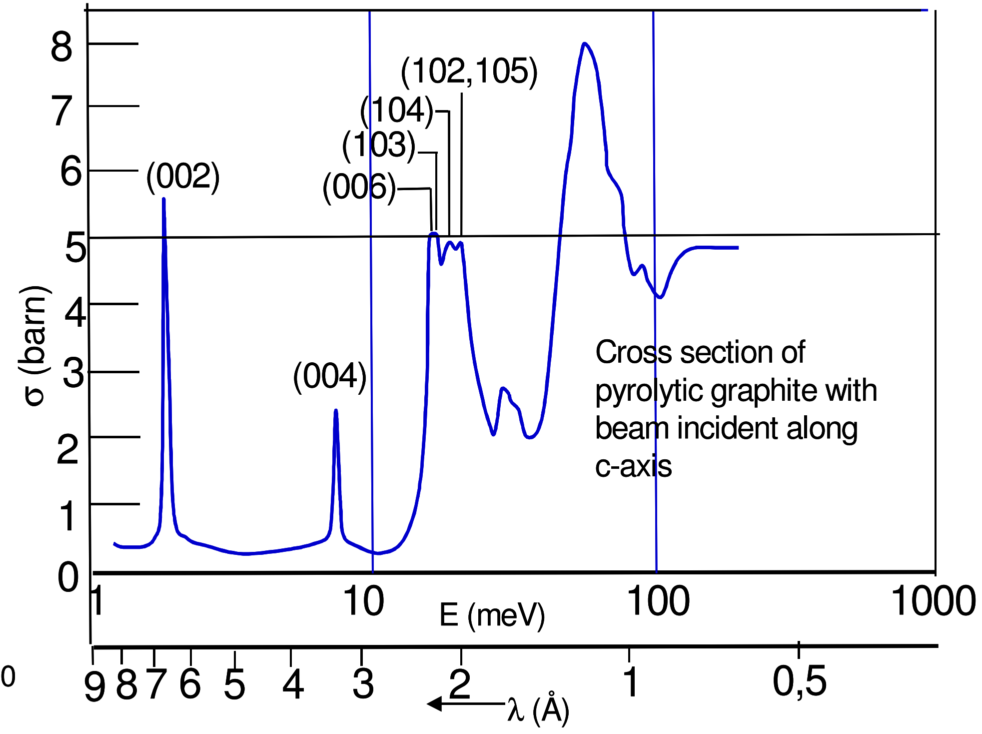
Similar diagrams can be used to judge the effect of moving crystals (phase space transformers)

PSOs Affecting k_z

Beam filters

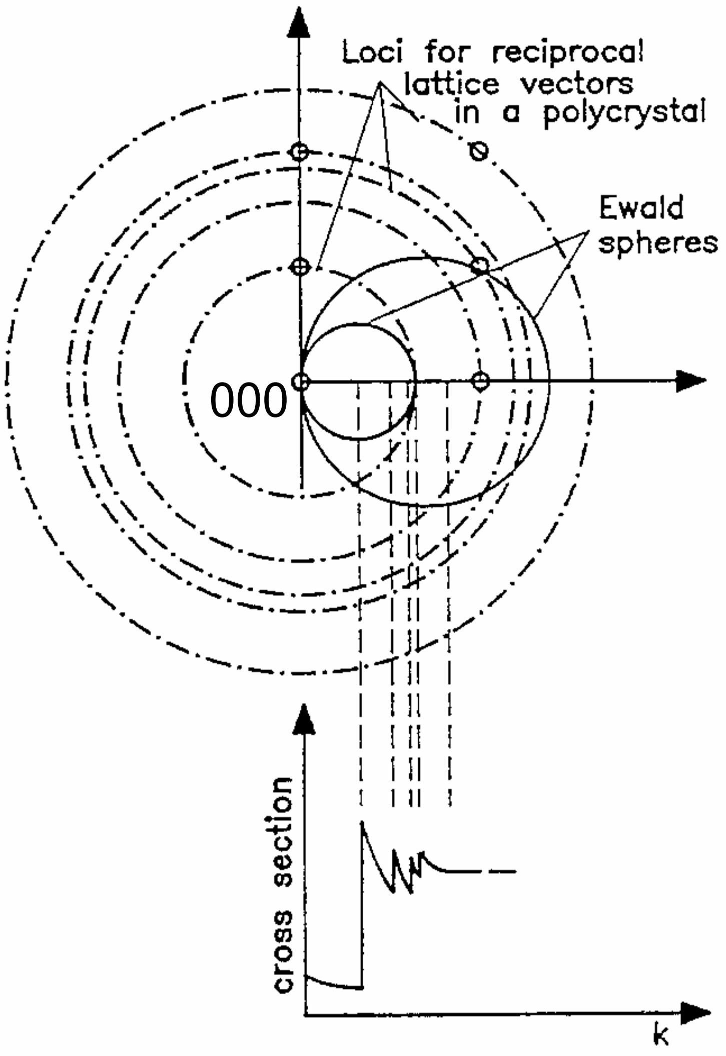
The Bragg condition $n\lambda=2d\sin\Theta$ or $\underline{Q}=\underline{G}_{hkl}$ means that, apart from the wavelength selected, also those corresponding to integer multiples (or fractions) of n res. (hkl) will be present in the beam. These can often be eliminated by filters. These are polycrystalline or semi-polycrystalline materials whose cross section has sharp edges or high bands for certain wavelengths.


Cross section of polycrystalline Beryllium


Cross section of pyrolytic graphite with beam incident along c-axis

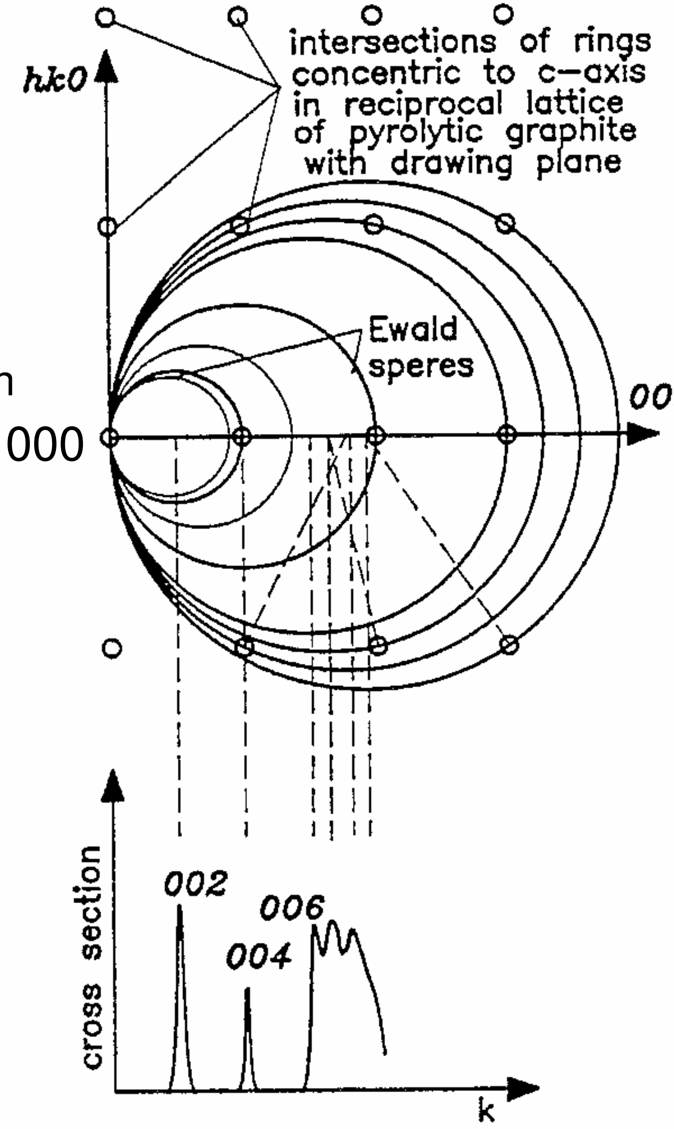
PSOs Affecting k_z

Beam filters (cntd.)



In a polycrystal the $\underline{G}_{hkl}$ lie on spheres around the origin of the reciprocal lattice (left). Pyrolytic graphite is a good single crystal along the c-axis but with random orientations perpendicular to it. This yields “rings” in the reciprocal lattice space with their midpoints on the c-axis (right).

Diffraction (beam attenuation) occurs whenever the Ewald sphere (of radius k_z) intersects the lattice spheres or rings



Summary on PSOs

- Phase space operators serve to select neutrons which fulfill the desired conditions in terms of their location at a given time and of their flight directions.
- They must, in general affect neutrons of different properties in different ways, in particular as far as spectral properties are concerned.
- Often they are fast moving devices (choppers, velocity selectors)
- This poses quite demanding requirements on the materials used in terms of their properties
  - nuclear (cross sections)
  - mechanical, magnetic
  - radiation effects
- Often combinations of different materials must be used.
- There are passive and active PSOs being used, which either select neutrons of the desired properties or (and) change them (e.g. moving crystals).

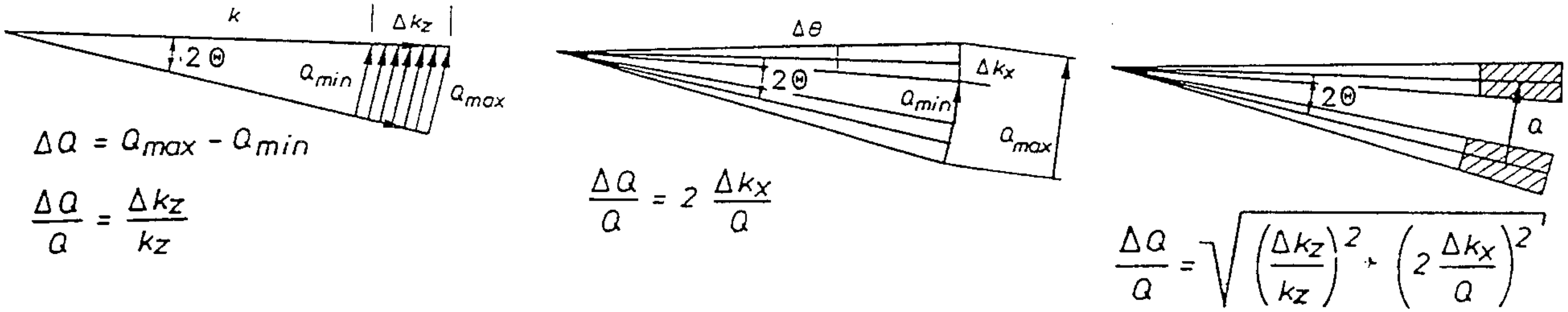
Part 4

Design Concepts for Neutron Scattering Instruments

Instruments for Elastic (Total) Scattering (1)

Small angle scattering ⇒investigation of large structures

Investigation of large structures requires measurement at small <u>Q</u>



$$\Delta Q = Q_{max} - Q_{min}$$

$$\frac{\Delta Q}{Q} = \frac{\Delta k_z}{k_z}$$

$$\frac{\Delta Q}{Q} = 2\,\frac{\Delta k_x}{Q}$$

$$\frac{\Delta Q}{Q} = \sqrt{\left(\frac{\Delta k_z}{k_z}\right)^2 + \left(2\,\frac{\Delta k_x}{Q}\right)^2}$$

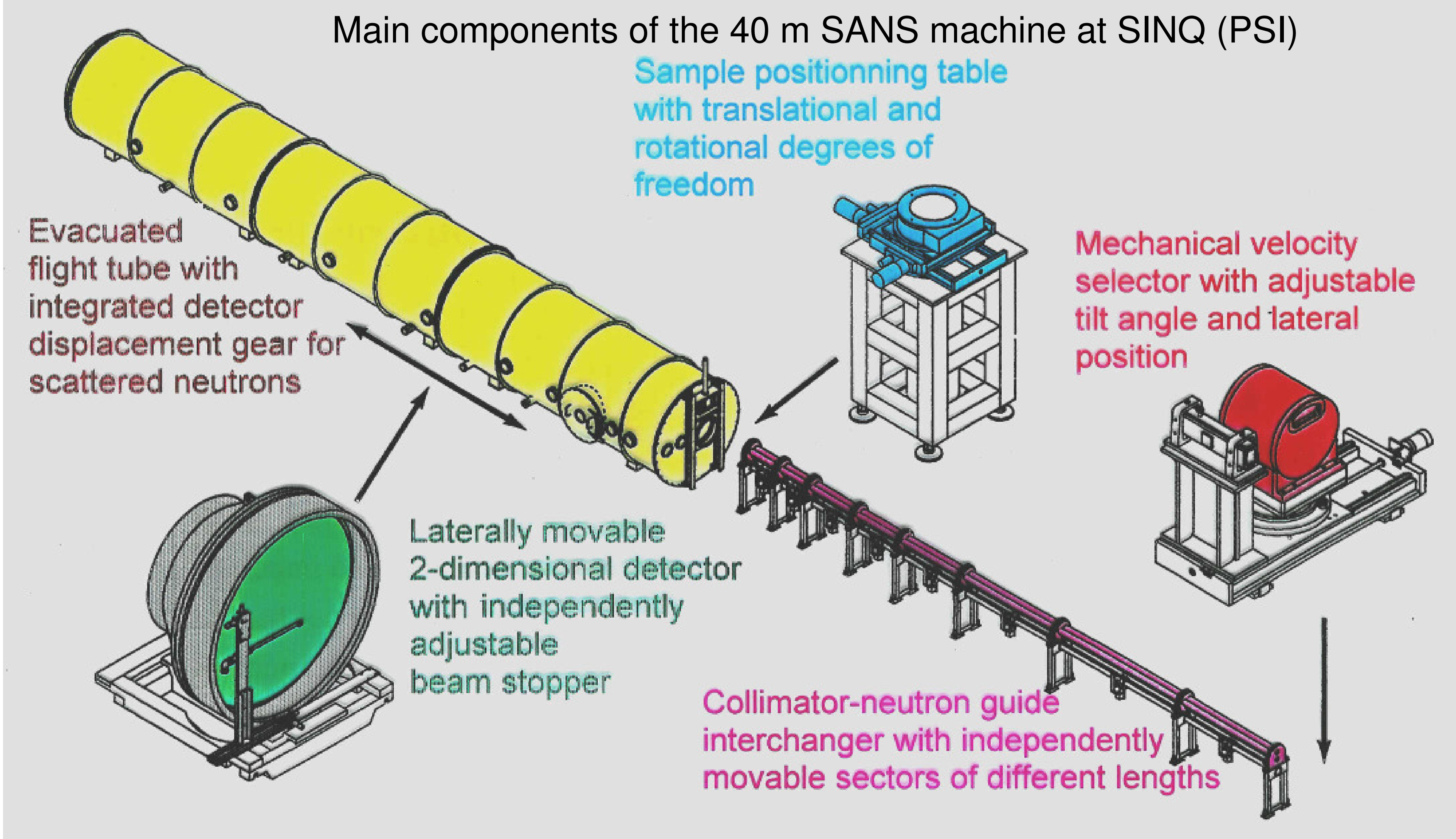
For small Θ $Q<<k_z$. The resolution therefore depends relatively weekly on Δk_z.

SANS can use thus relatively poor wavelength resolution but must have good angular collimation. Modern SANS-machines therefore have a high transmission mechanical velocity selector, 2-D position sensitive detectors, variable sample-to-detector distance and an interchangeable array of collimators to match the sample-to detector distance.

Instruments for Elastic (Total) Scattering (2)

Small angle scattering (cntd.)

Main components of the 40 m SANS machine at SINQ (PSI)



Sample positionning table with translational and rotational degrees of freedom

Mechanical velocity selector with adjustable tilt angle and lateral position

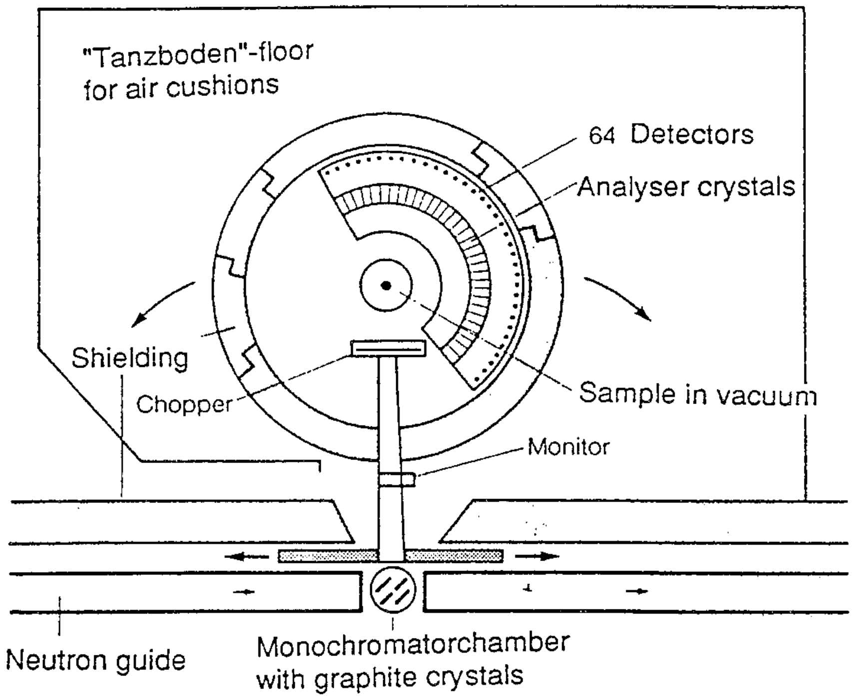
Evacuated flight tube with integrated detector displacement gear for scattered neutrons

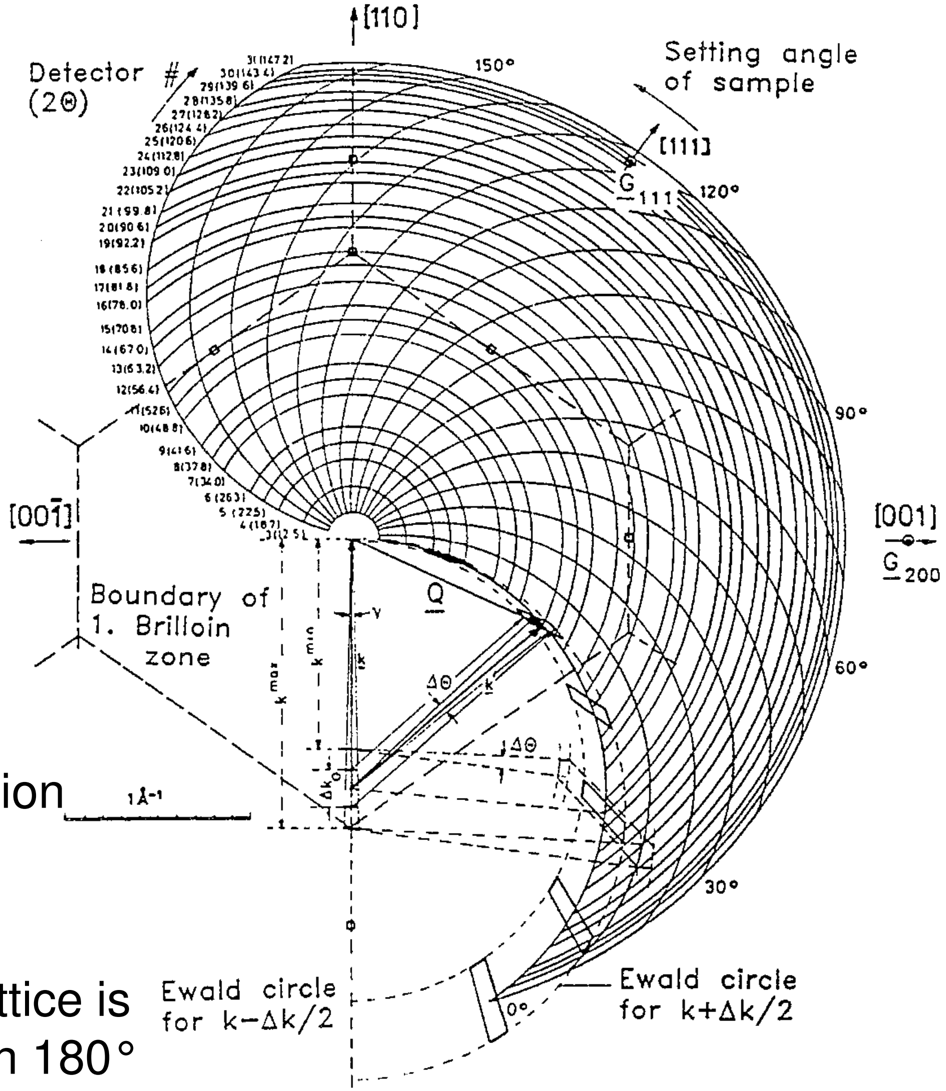
Laterally movable 2-dimensional detector with independently adjustable beam stopper

Collimator-neutron guide interchanger with independently movable sectors of different lengths

Instruments for Elastic (Total) Scattering (3)

Diffuse Elastic Neutron Scattering DENS



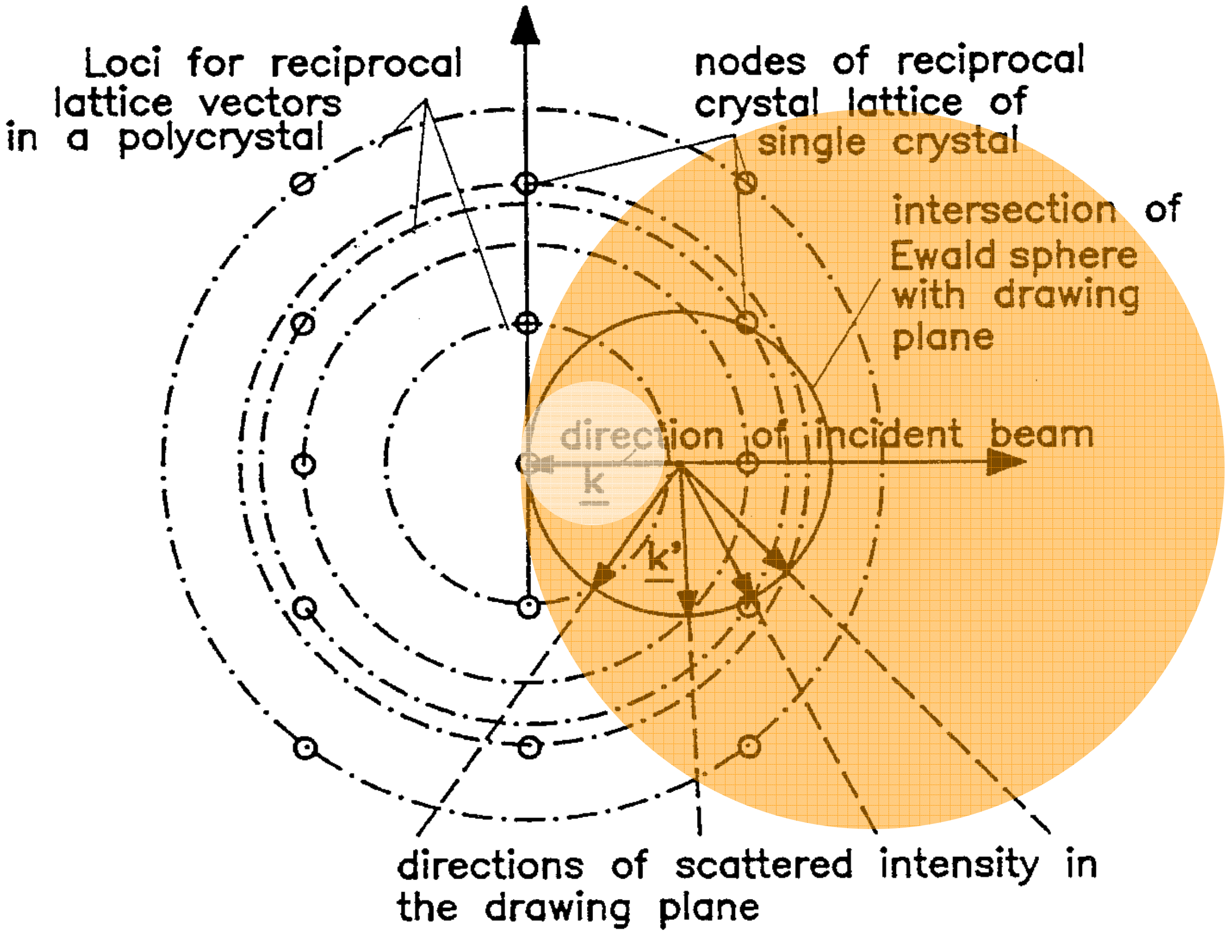


Since DENS is often very week, low resolution ToF discrimination is used to eliminate -or enable correction for- inelastic scattering.

A large area of the reciprocal lattice is covered by rotating the sample through 180° in suitable steps,

Instruments for Elastic (Total) Scattering (4)

Powder diffraction



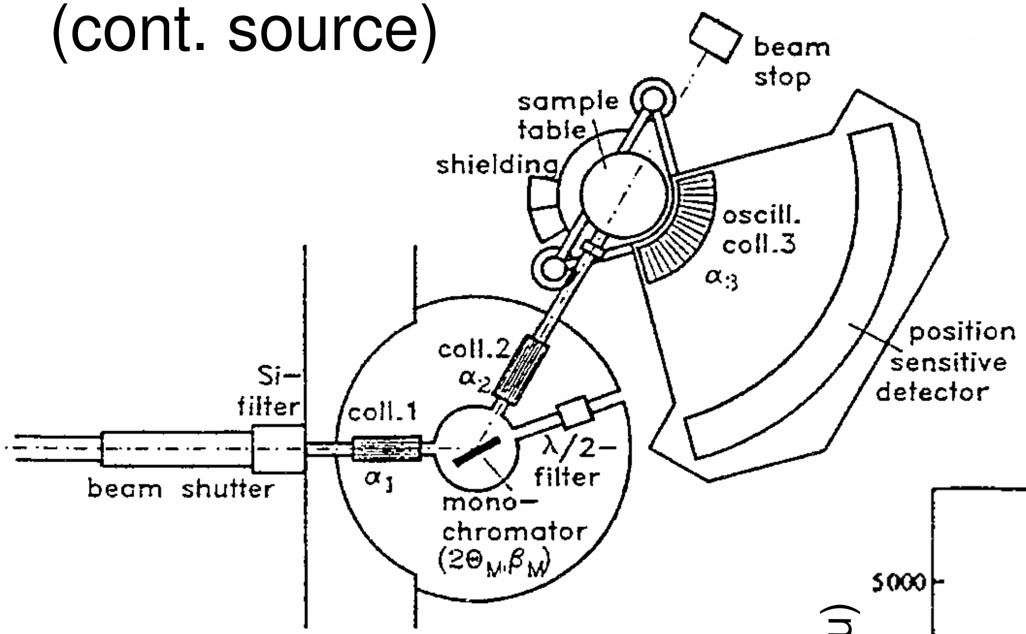
In a powder (polycrystal) all orientations of the $\underline{G}_{hkl}$ of its crystallites occur with (more or less) equal probability. Their endpoints therefore lie on concentric spheres around the origin of the reciprocal crystal lattice. For every value of k intensity will be recorded in a position sensitive detector, when k' lies on their intersection curves with the Ewald sphere.

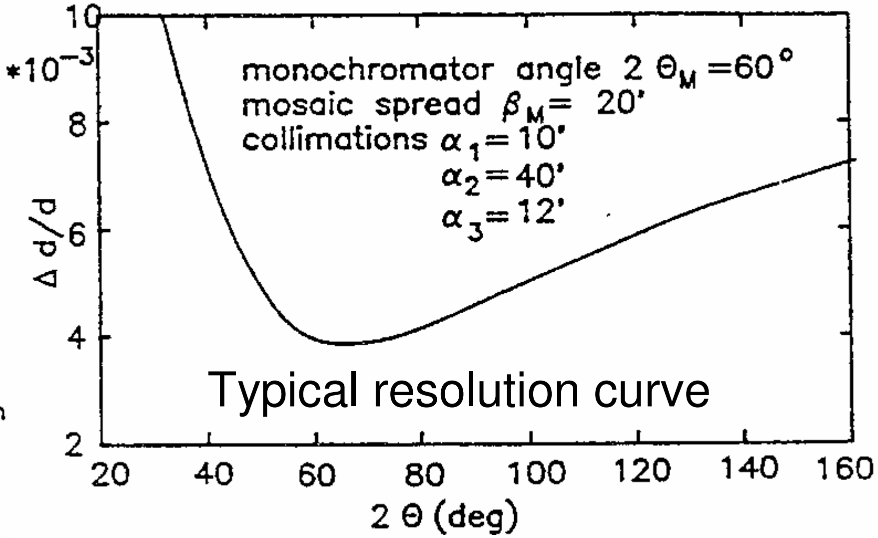
On pulsed sources (time of flight diffractometers) k_z decreases as a function of time after each pulse.

Instruments for Elastic (Total) Scattering (5)

High resolution powder diffraction

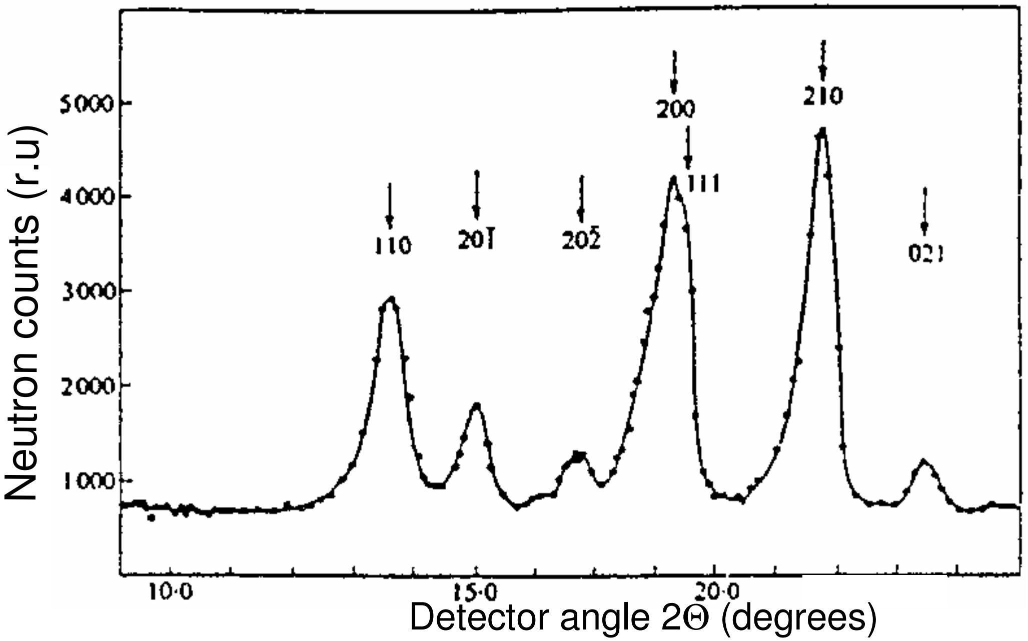
(cont. source)





Typical resolution curve

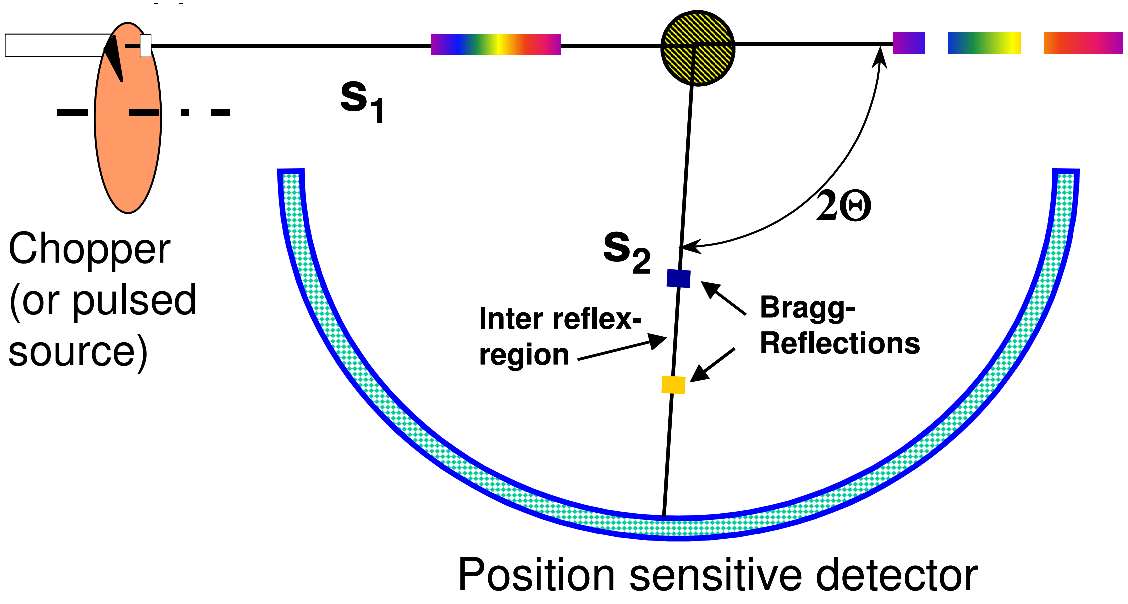
Narrow collimation is required for good resolution



Section of the diffraction pattern from powdered naphtalene recorded with neutrons of 1.1 Å

Instruments for Elastic (Total) Scattering (6)

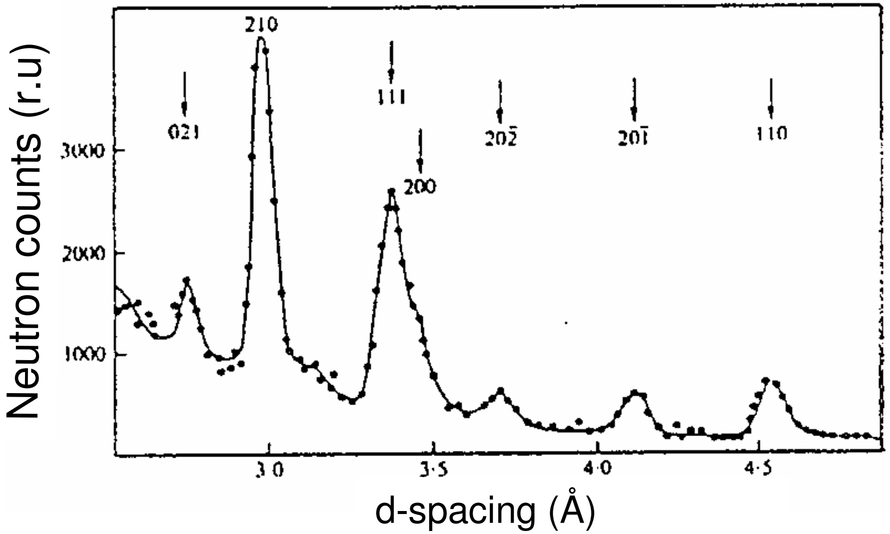
Time of flight diffractometer



In a ToF-diffractometer the flight time is directly proportional to the d-spacing for any given detector angle

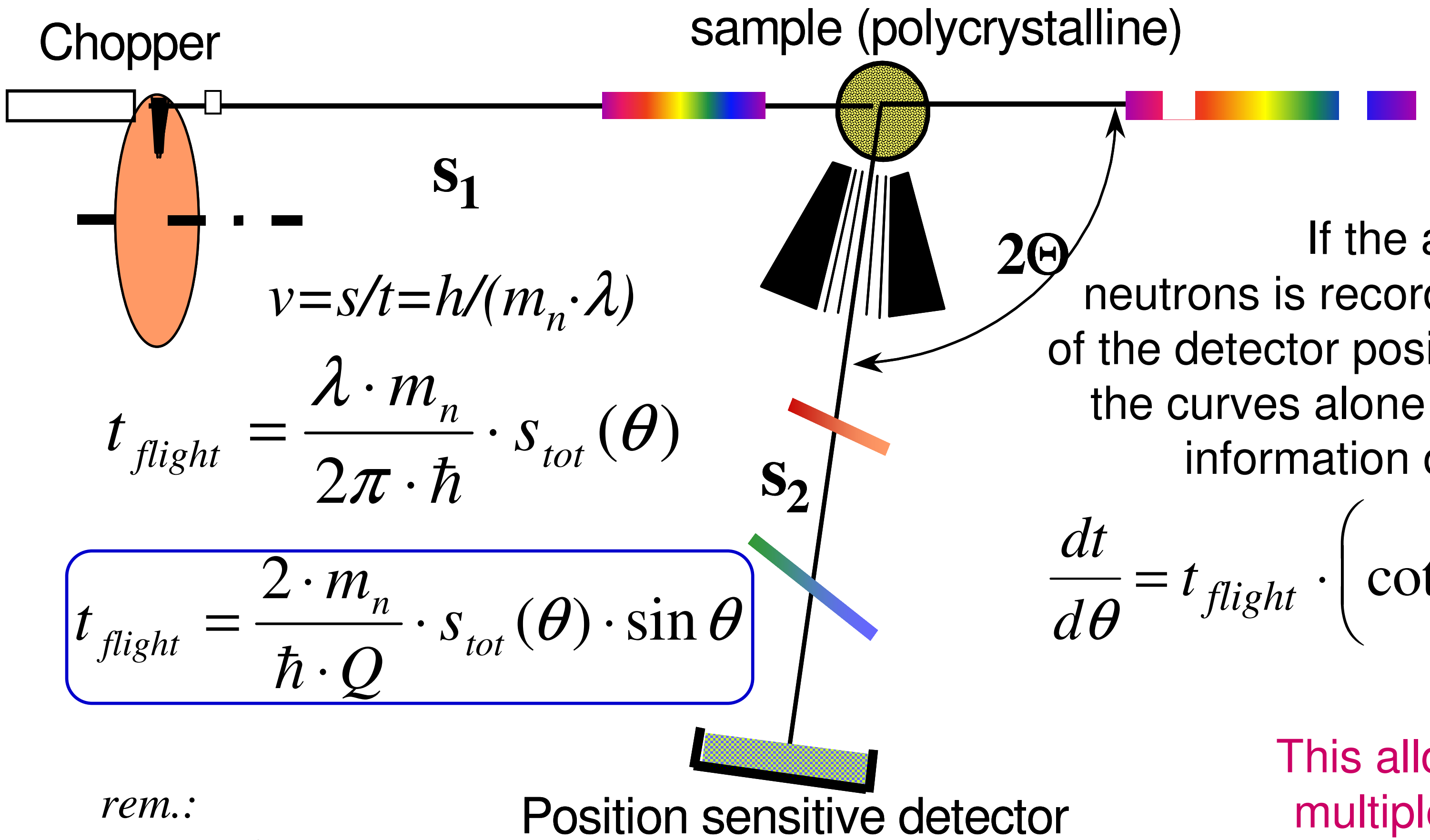
In a short pulsed source the velocity spread in a pulse is inversely proportional to the mean velocity ($\Delta v/v$ = const.) in the slowing down regime of the spectrum. This leads to a constant contribution to the resolution function.

Diffraction pattern from powdered naphtalene recorded for one detector angle with a ToF-diffractometer



Instruments for Elastic (Total) Scattering (7)

Time of flight diffractometer with a position sensitive detector



$$v = s/t = h/(m_n \cdot \lambda)$$

$$t_{flight} = \frac{\lambda \cdot m_n}{2\pi \cdot \hbar} \cdot s_{tot}(\theta)$$

$$\boxed{t_{flight} = \frac{2 \cdot m_n}{\hbar \cdot Q} \cdot s_{tot}(\theta) \cdot \sin\theta}$$

rem.:
$Q = 4\pi/(\lambda \cdot \sin\theta)$
in elastic scattering

If the arrival time of the neutrons is recorded as a function of the detector position, the slope of the curves alone already contains information on the flight time:

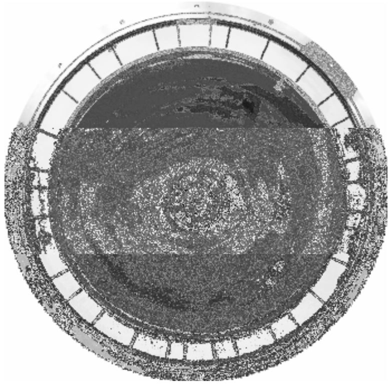
$$\frac{dt}{d\theta} = t_{flight} \cdot \left(\cot\theta + \frac{1}{s_{tot}} \cdot \frac{ds_{tot}}{d\theta} \right)$$

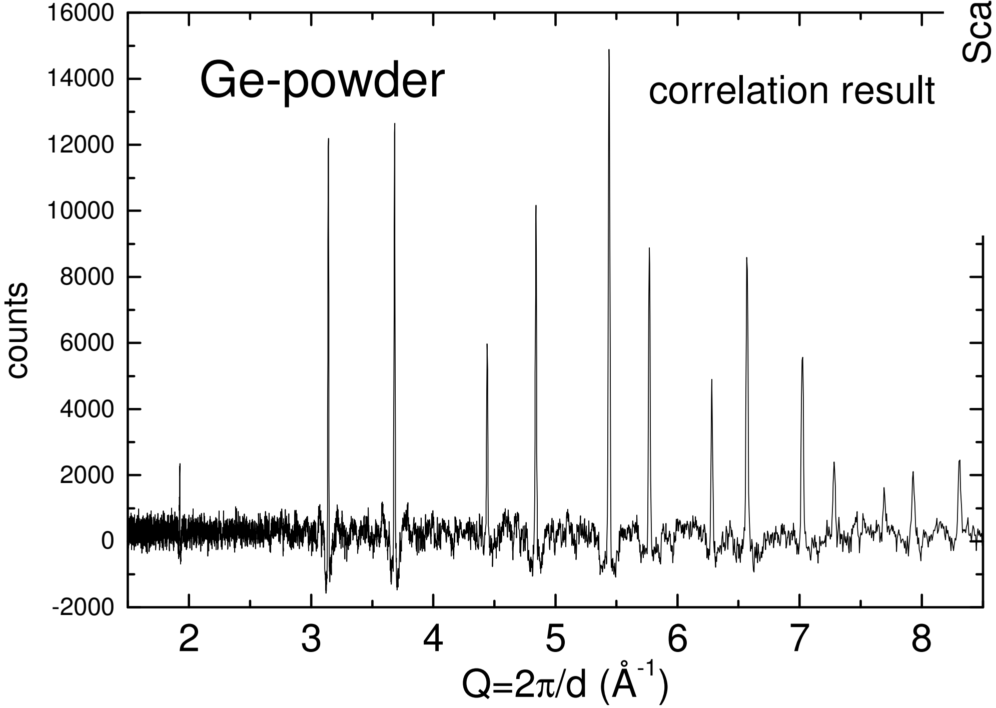
This allows to work with multiple frame overlap, enhancing significantly the overall count rate on continuous sources

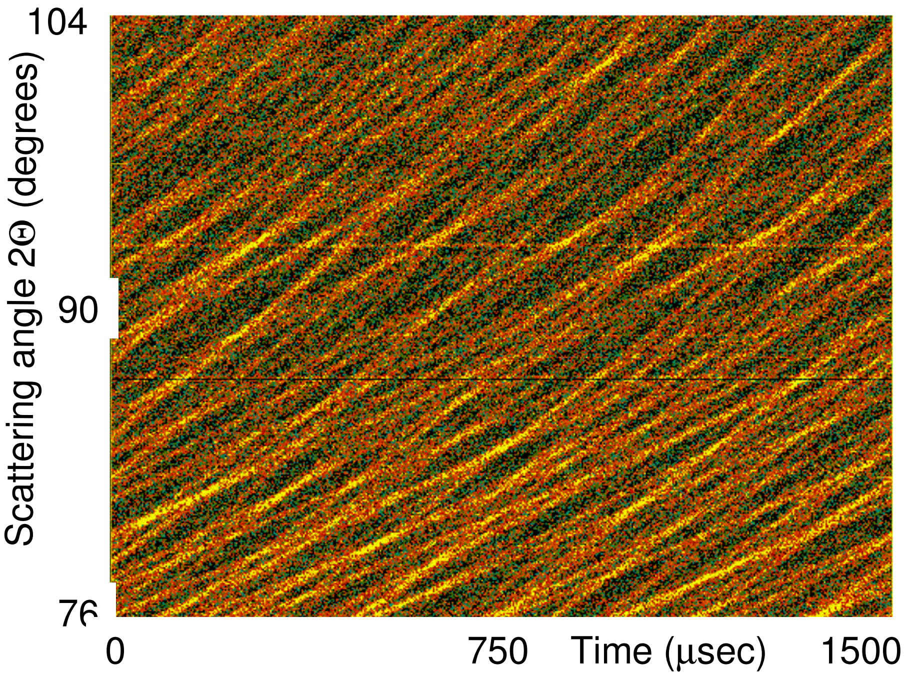
Instruments for Elastic (Total) Scattering (7)

Correlation ToF Diffractometer

Chopper with multiple (32) slits arranged in pseudo-random sequence







Since, after performing a correlation analysis of the pattern, the neutron ToF can be calculated from the slope it is possible to trace the slit opening they came from. With the known exact opening time of the slit and the arrival time of the neutron at the detector, **their exact flight time can be determined with the same accuracy as for a single slit chopper**.

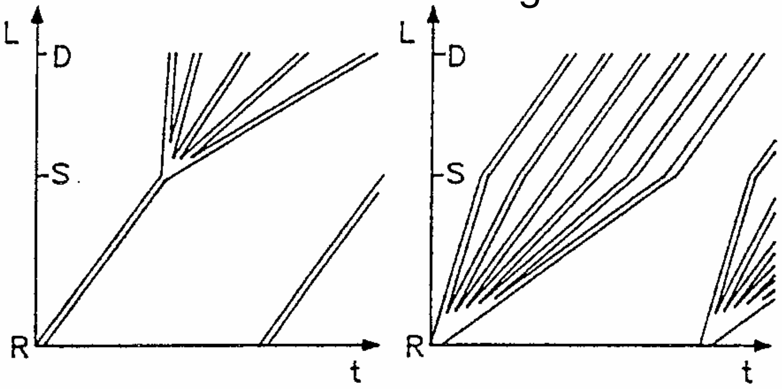
Instruments for Inelastic Scattering (1)

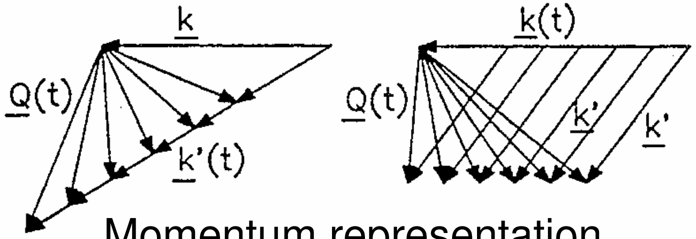
Direct and inverted time of flight methods

Direct ToF
incident energy fixed (monochromator)
outgoing energy scanned as function of time

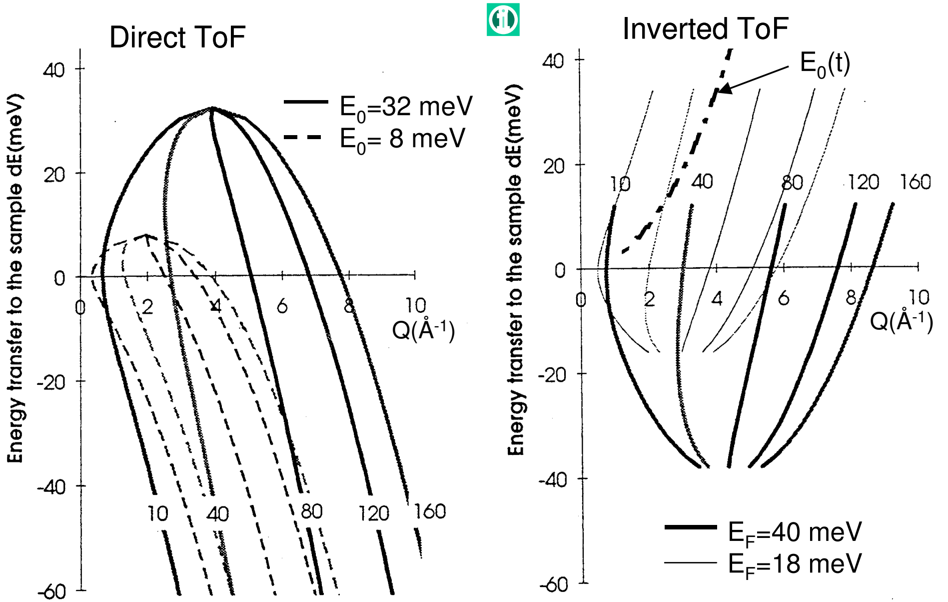
Inverted ToF
incident energy varies as function of time;
outgoing energy fixed (analyser crystals or filter)

Distance-time diagrams

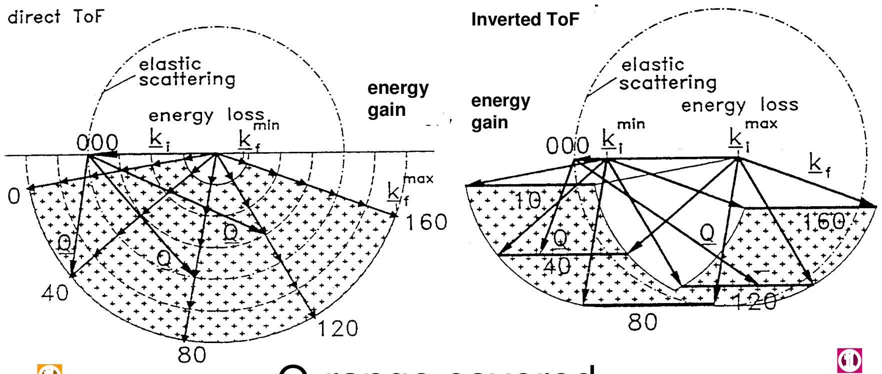




Momentum representation



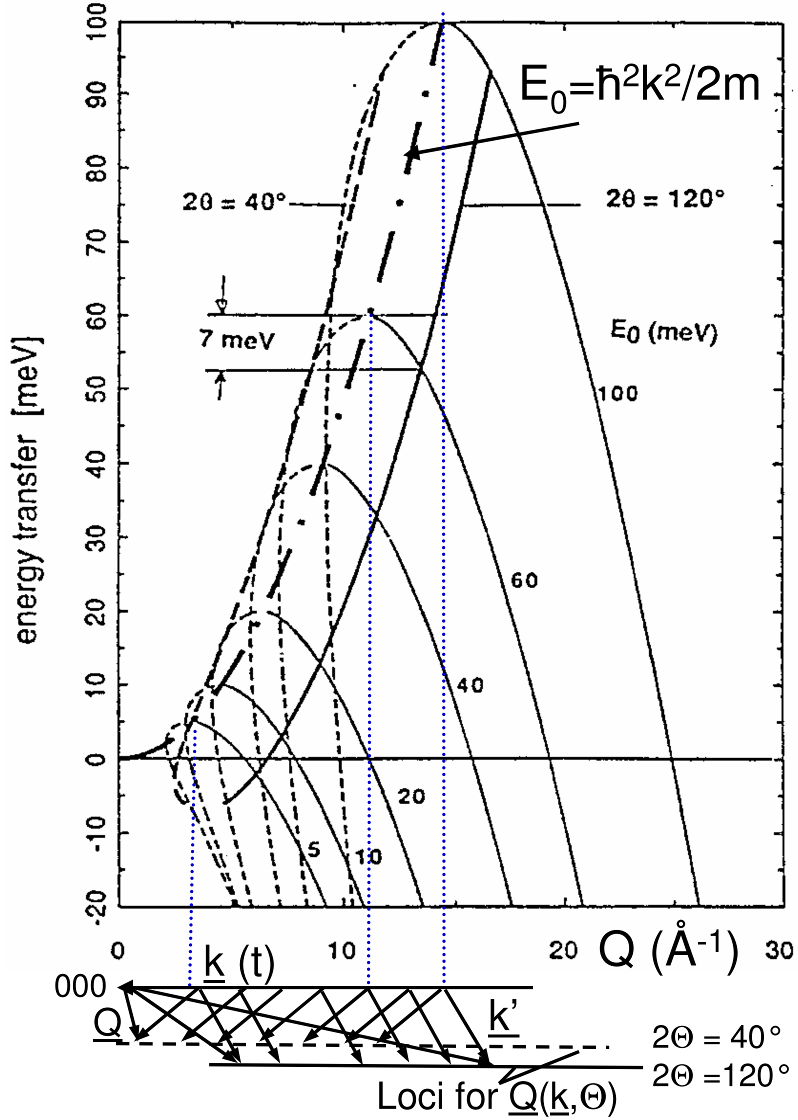
Kinematic diagrams for direct and inverted ToF for different scattering angles 2Θ



Q-range covered

Instruments for Inelastic Scattering (2)

Inverted ToF – the kinematic range



E_0 (k_0) decreases as a function of time.

The analyser energy E_F is constant at all times (e.g. 7 meV)

The energy transfer is E_0-E_F

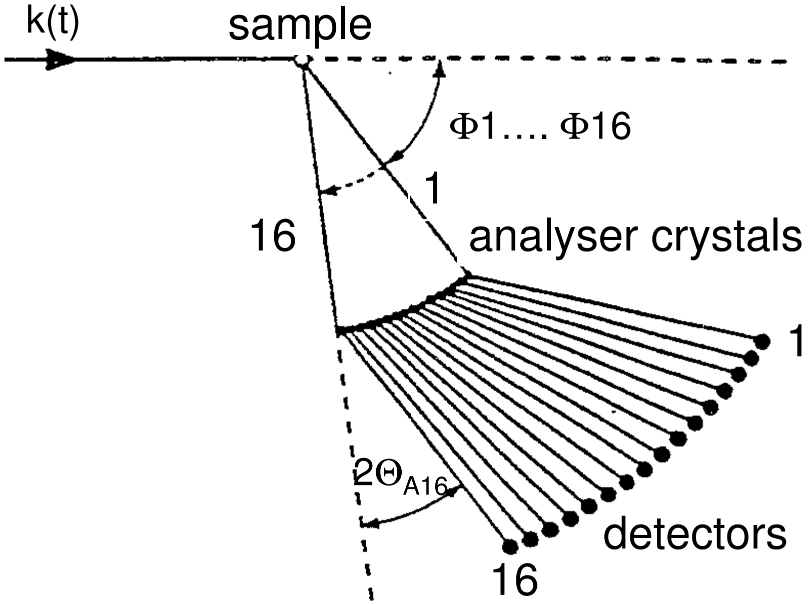
No neutrons with an energy gain greater than E_F can be measured.

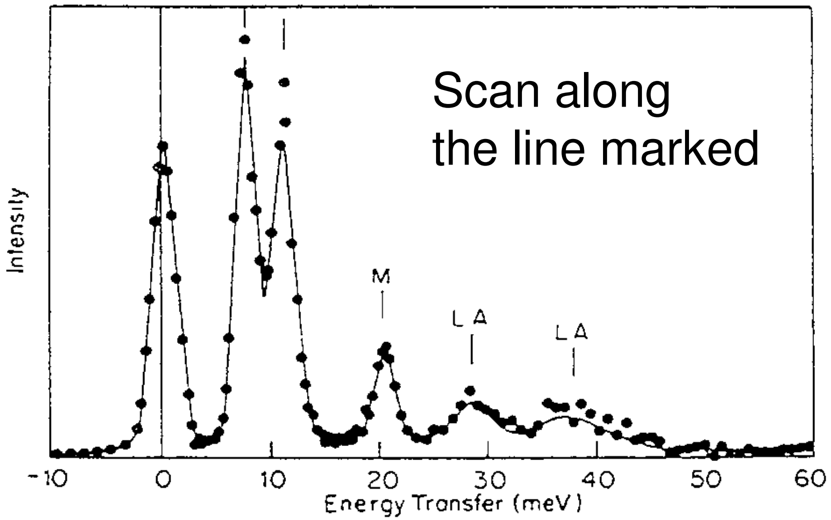
For any given scattering angle (40° and 120° shown) the locus for all scattered neutrons transmitted by the analyser is on the kinematic curves corresponding to E_0(t) minus E_F.

For any given scattering angle 2Θ all Q-vectors end in a plane parallel to the vertical plane through k

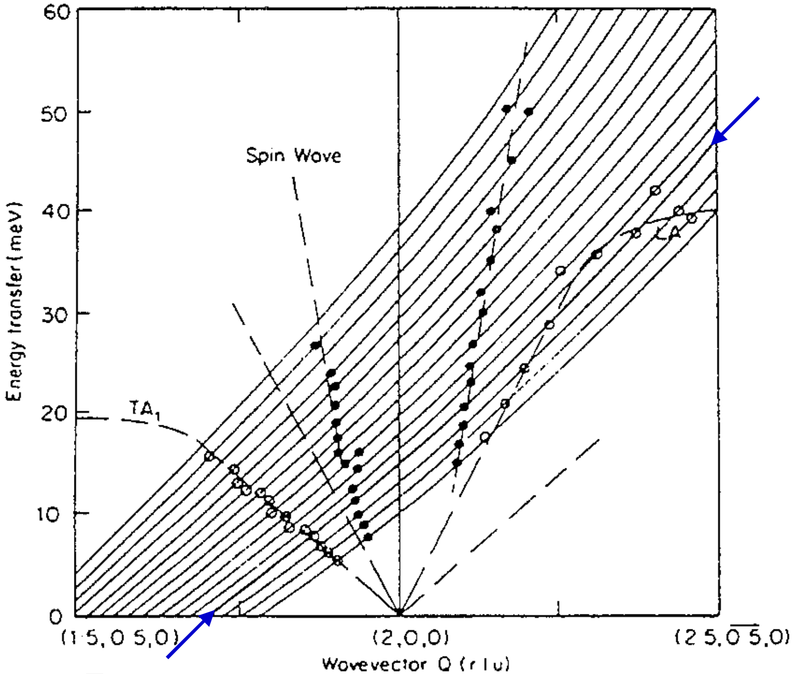
Instruments for Inelastic Scattering (3)

The PRISMA-instrument (ISIS)





Scan along the line marked



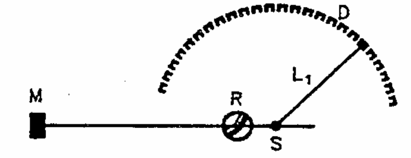
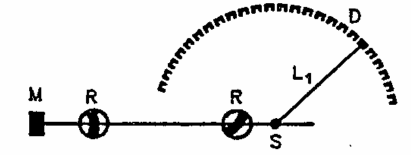
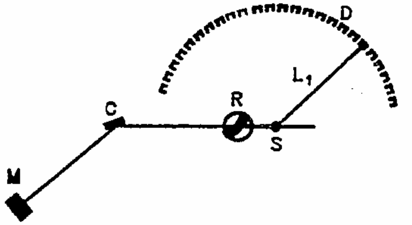
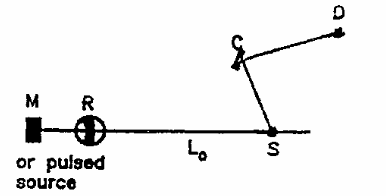
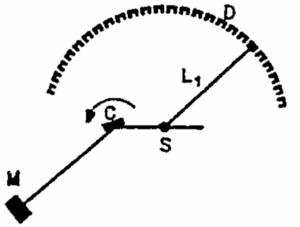
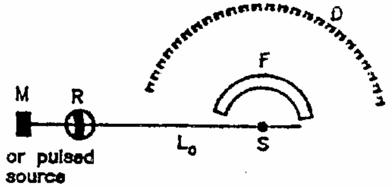
With the condition

$\sin\Phi_i/\sin\Theta_{Ai}$ = constant

all Q-vectors end in the same plane parallel to the vertical plane through <u>k</u>

Instruments for Inelastic Scattering (4)

Summary of Options for ToF Instruments

	direct TOF	inverted TOF
Fermi type chopper		
double chopper		
crystal plus chopper		
rotating crystal		
filter		

Fermi type chopper for simultaneous chopping and wavelength selection (coupled)

Double (multiple chopper system can give good energy and time resolution independently

Pulsing by chopper wavelength selection by crystal

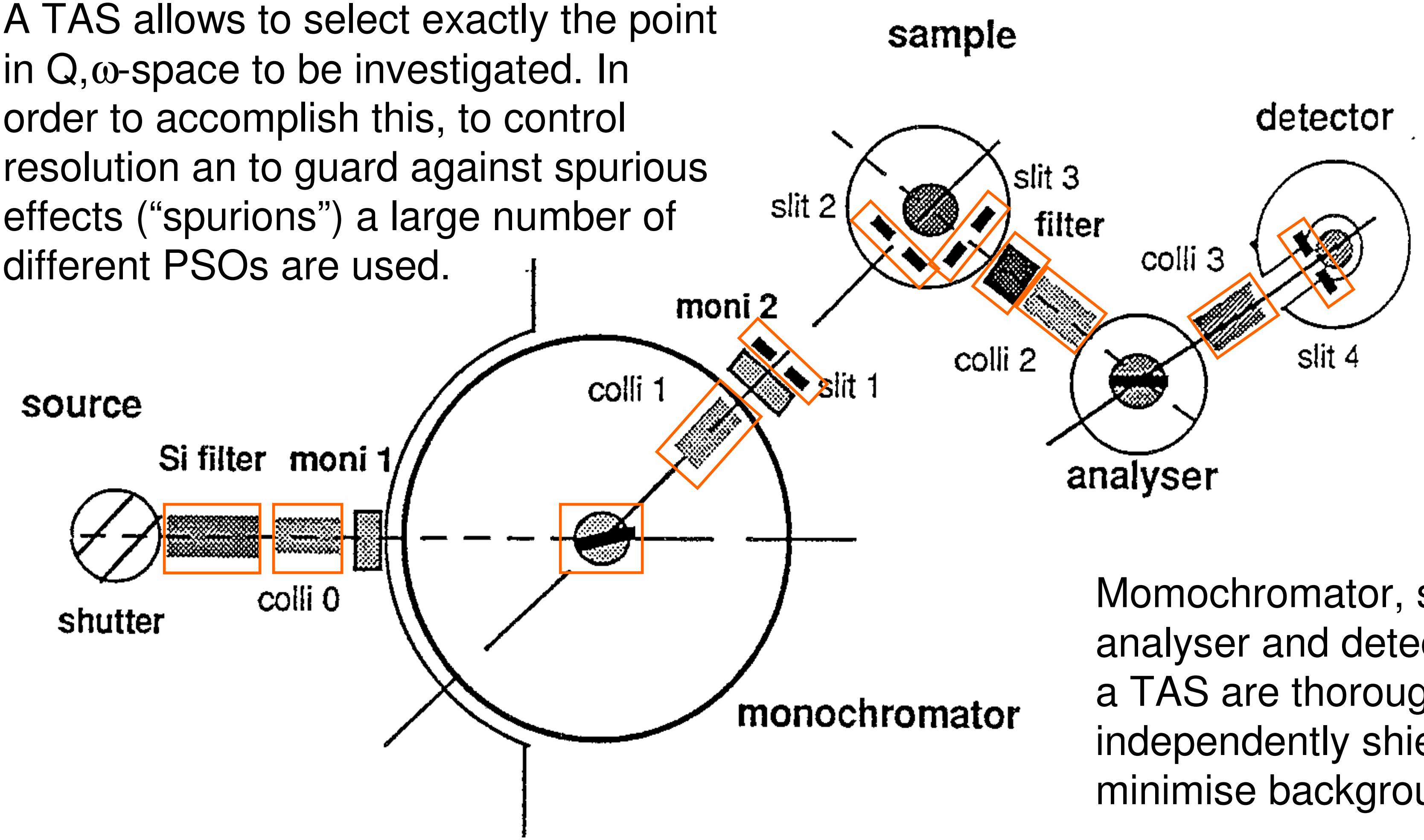
Rotating crystal acts ad monochromator and chopper simultaneously

Filter in the scattered beam suppresses all energies above the Bragg cutoff

Instruments for Inelastic Scattering (5)

The triple axis spectrometer (TAS) and its PSOs

A TAS allows to select exactly the point in Q,ω-space to be investigated. In order to accomplish this, to control resolution an to guard against spurious effects ("spurions") a large number of different PSOs are used.



Momochromator, sample, analyser and detector of a TAS are thoroughly and independently shielded to minimise background.

Instruments for Inelastic Scattering (6)

TAS (cntd.)

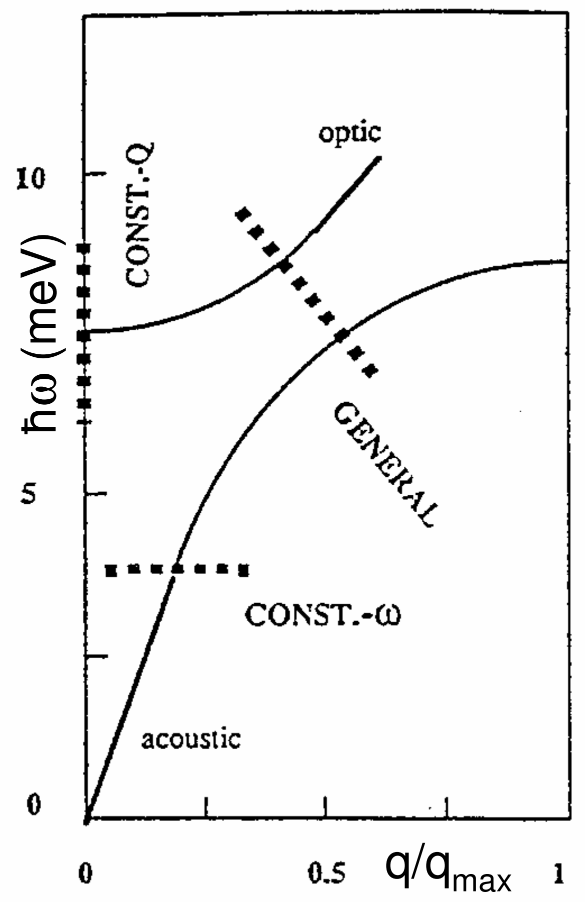
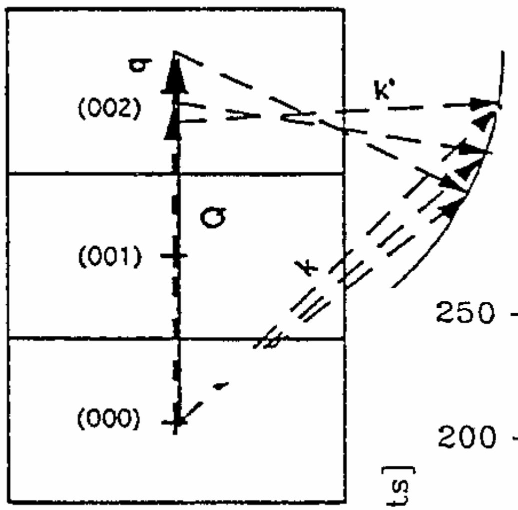
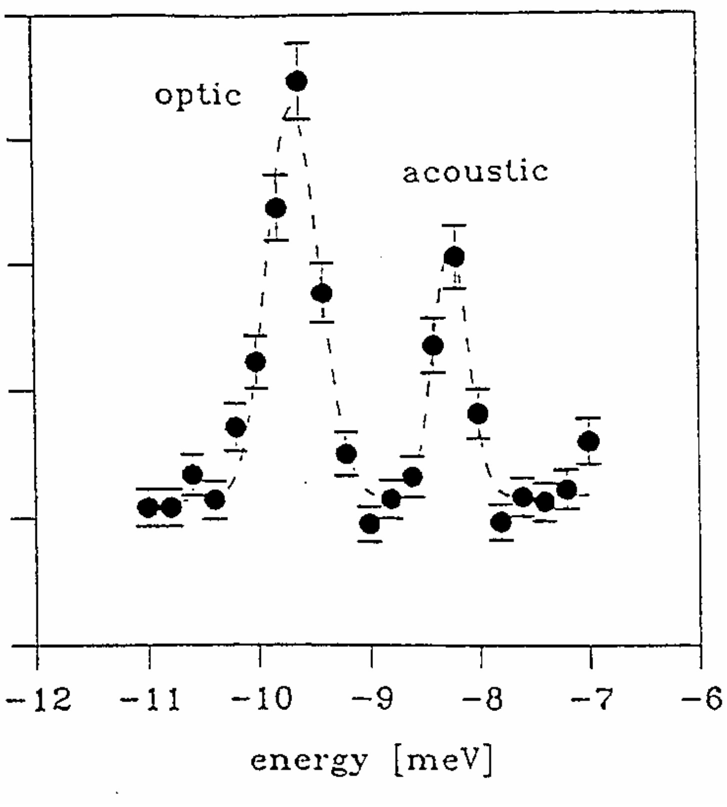


Illustration of a “constant q”, a “constant ω” and a “general” scan on a TAS



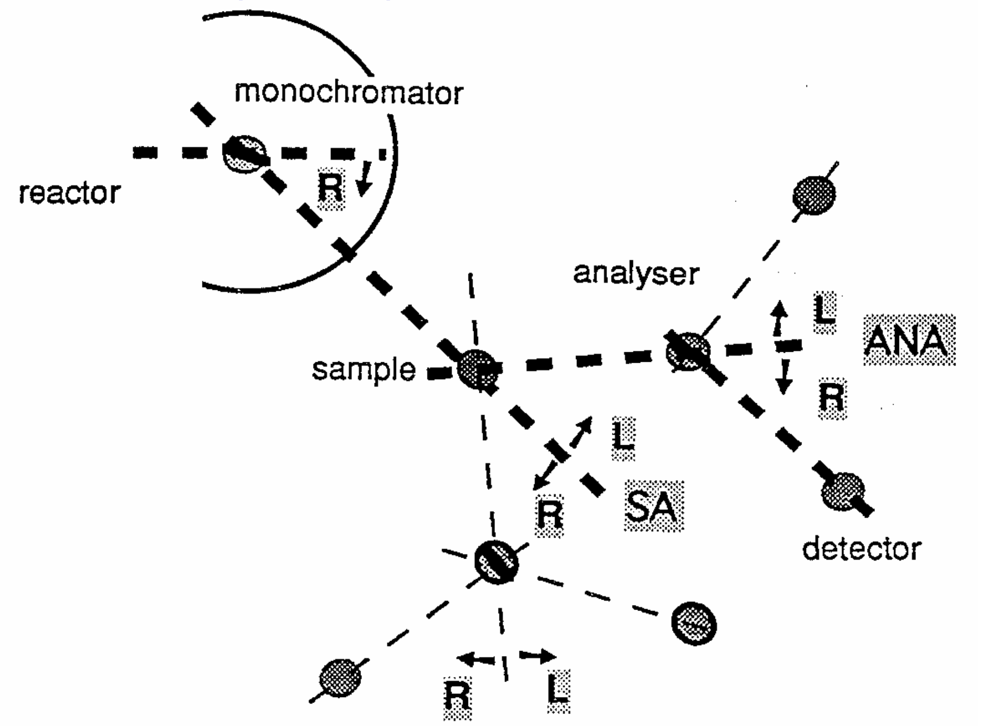
Momentum diagram for the “general” scan on the left



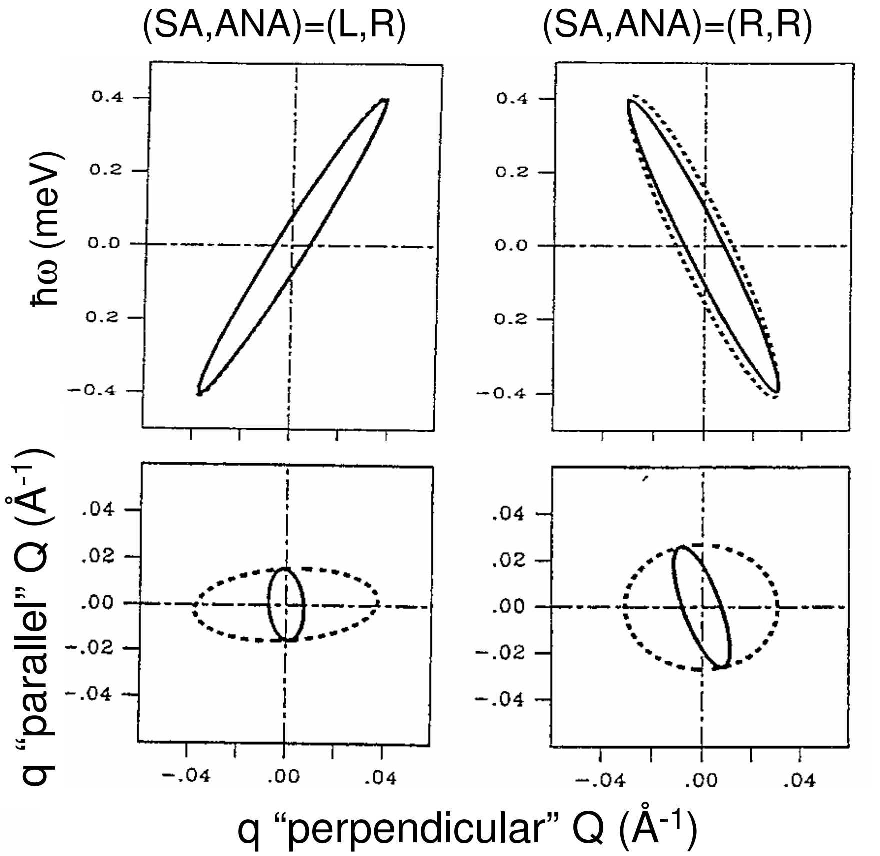
Intensity as a function of energy transfer in the “general” scan shown

Instruments for Inelastic Scattering (6)

TAS (cntd.)



Scattering configurations of a TAS.
L and R indicate “left” and “right”, as seen along the flight path of the neutron;
SA = L (sample axis) and ANA = R) (analyser axis) is the so called “W” configuration (MA = R) (monochromator).

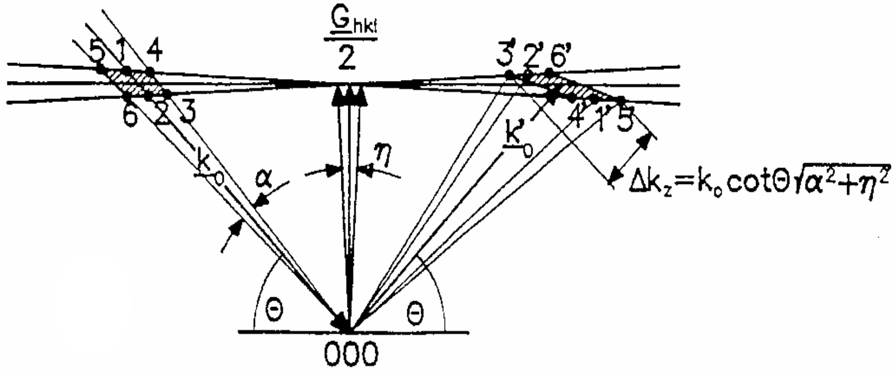


Examples of resolution ellipsoids for two different TAS-settings for $E_a=E_m=14.9$ meV, $Q=3.5$ Å^{-1}
solid: cut through ellipsoid at (0,0)
dashed: projection into plane

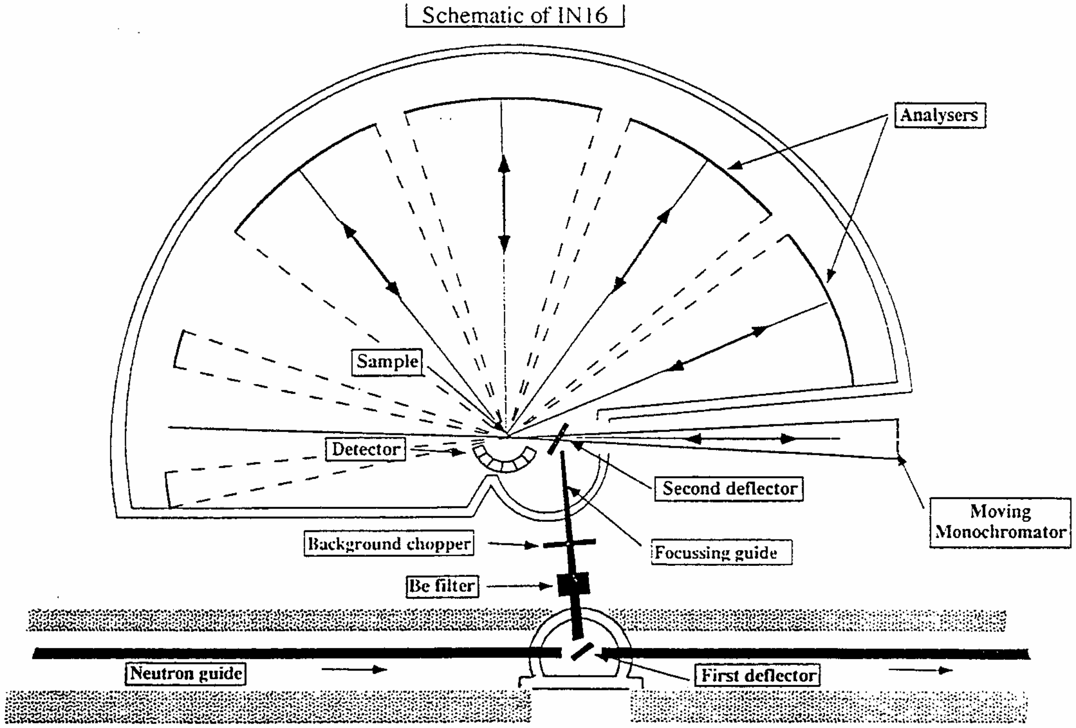
Instruments for Inelastic Scattering (7)

Back scattering

The back scattering spectrometer is essentially a TAS with its monochromator and analyser angles set to $2\Theta = 180°$, performing a k_i-scan (incident energy).



In this case the contributions from the mosaic spread and the beam divergency to Δk_z practically vanish ($\cot 90° = 0$).



k_i (v_i) is varied either by imposing a time dependent velocity from a moving monochromator or by changing its temperature (lattice spacing) as a function of time (cont. source), or by high resolution ToF (pulsed source).

With v_f fixed, intensity is recorded when $v_i + \hbar\omega/2m = v_f$

Summary on Instruments

- Neutron scattering instruments are generally very complex arrangements of PSOs, sample environment, detectors and control systems.
- They come in many different varieties because no single design can serve all the opportunities neutrons provide for science.
- Typically they can be classed according to continuous and pulsed operation, although there are hybrids.
- Certain classes of instruments can be best served by continuous sources, whereas for others intrinsically pulsed sources are preferable.
- In addition to thr time characteristics of the source, the spectral characteristics of the moderators are of prime importance for an integrated optimised design.