DESIGNING AND BUILDING A NEUTRON INSTRUMENT
XIV School of Neutron Scattering Francesco Paolo Ricci (SoNS)
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Anatomy of a neutron scattering instrument

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D20 responsible

Anatomy of a neutron scattering instrument

- Neutron scattering experiments
  - Common object: one of the three neutron cross sections
    1. Total cross section $\sigma_{tot}(E_0)$ for incident energy E_0
       - Integrating over all angles and energies.
    2. Angle-dependent differential cross section $d\sigma/d\Omega(\mathbf{Q})$
       - Integration over energies
       - Quasi-elastic assumption: function of the scattering vector **Q**
    3. **Q**/E-dependent double differential cross section $d^2\sigma/d\Omega dE(\mathbf{Q},E)$
- Components
- Instruments
  1. Total cross section measurement
     1. Important in nuclear technology
     2. Defect properties of solids ← total-cross-section studies
  2. Diffractometers
  3. Spectrometers
  4. Nuclear and particle physics
- Anatomy of D20 via Monte-Carlo simulation and "back-on-the-envelope" analytics

Neutron instrument components

- Restricted number of distinct components

```
DEFINE INSTRUMENT D20 (...)
DECLARE
                %{ ... %}
INITIALIZE
                %{ ... %}
TRACE
COMPONENT Polychromatic_Beam = Arm() AT (0,0,0) ABSOLUTE
COMPONENT Thermal_Source = Source_adapt(...) AT (0,0,0) RELATIVE Polychromatic_Beam
COMPONENT Thermal_Neutrons = Convert_FlatE_2_Maxwell(T = SourceTemp, E0=Source_E0, dE=Source_dE) AT (0,0,0) RELATIVE Polychromatic_Beam
COMPONENT Circular_Source = Circular_slit(radius=0.1) AT (0,0,0.001) RELATIVE Polychromatic_Beam
COMPONENT Nose1 = Slit(...) AT (0,0,3.3400) RELATIVE Polychromatic_Beam
COMPONENT Nose2 = Slit(...) AT (0,0,3.3401) RELATIVE Polychromatic_Beam ROTATED (0,0,45) RELATIVE Polychromatic_Beam
COMPONENT Flux_Nose_1cm2 = Monitor (...) AT ( 0, 0, 3.3402) RELATIVE Polychromatic_Beam
COMPONENT Rotor1 = Slit(...) AT (0,0,3.720) RELATIVE Polychromatic_Beam
COMPONENT Rotor2 = Slit(...) AT (0,0,4.240) RELATIVE Polychromatic_Beam
COMPONENT Rotor3 = Slit(...) AT (0,0,4.7601) RELATIVE Polychromatic_Beam
COMPONENT Soller = Soller(...) AT (0, 0, 16.400) RELATIVE Polychromatic_Beam ROTATED (0, Soller_misalign, 0) RELATIVE Polychromatic_Beam
COMPONENT Flux_Soller_1cm2 = Monitor (...) AT ( 0, 0, 16.660) RELATIVE Polychromatic_Beam
COMPONENT Filter = Filter_Graphite(...) AT (0, 0, 16.700) RELATIVE Polychromatic_Beam
COMPONENT Monochromator_Plane = Arm() AT (0, 0, Mono_position) RELATIVE Polychromatic_Beam ROTATED (0, Mono_omega, 0) RELATIVE Polychromatic_Beam
COMPONENT Blade0 = Monochromator0(...) AT (0, mpos0, 0) RELATIVE Monochromator_Plane ROTATED (0, 0, mrotz0) RELATIVE Monochromator_Plane
COMPONENT Monochromatic_Beam = Arm() AT (0, 0, d_source_mono) RELATIVE Polychromatic_Beam ROTATED (0, Takeoff, 0) RELATIVE Polychromatic_Beam
COMPONENT Flux_Mono_1cm2 = Monitor (...) AT ( 0, 0,  0.398) RELATIVE Monochromatic_Beam
COMPONENT MSlit = Slit(...) AT (0,0,0.400) RELATIVE Monochromatic_Beam ROTATED (0,0,0) RELATIVE Monochromatic_Beam
COMPONENT Flux_MSlt_1cm2 = Monitor (...) AT ( 0, 0,  0.401) RELATIVE Monochromatic_Beam
COMPONENT SSlit = Slit(...) AT (0,0,2.800) RELATIVE Monochromatic_Beam ROTATED (0,0,0) RELATIVE Monochromatic_Beam
COMPONENT Sample_in = Powder_hollow (...) AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT ROC=ROC2(...) AT (0, PSD_y, Sample_z) RELATIVE Monochromatic_Beam
COMPONENT PSD_Entry = PSD_entry(...) AT (0, PSD_y, Sample_z) RELATIVE Monochromatic_Beam
COMPONENT PSD_D20 = PSD_curved(...) AT ( 0, PSD_y,  Sample_z) RELATIVE Monochromatic_Beam
FINALLY
                %{ ... %}
END
```

Neutron instrument components

- Source blocks
  - Important parameter of (reactor) source: thermal neutron flux
    - about 10^{14} $n \cdot cm^{-2} s^{-1}$ for a medium flux reactor,
      - referring to the number of all thermal neutrons, of all directions and all energies passing through one square centimetre per second
    - more detailed: flux per unit energy or wavelength
  - Further parameters: moderator area and spectral temperature.
- Flight paths
  - Neutron shielding
    - scatter, slow down and absorb fast neutrons
    - attenuate γ radiation
    - iron (scattering fast neutrons)
    - hydrogen in concrete, resin, or wax (slowing down the neutrons)
    - boron or cadmium (absorbing slow neutrons).

Neutron instrument components (cont`d)

- Collimators
  - Angular collimation within and outside the scattering plane
    - Two absorbing slits of given with, separated by given distance
      - triangular divergence distribution
    - Soller collimators define angles without restricting beam size
      - Many parallel, absorbing slits, e.g., gadolinium-oxide painted Mylar films
- Neutron guides
  - Leave this to the many experts in this room …
- Beam benders
    - Principles of Soller collimators and guide tubes combined
    - Stack of curved surfaces reflects (cold) neutrons

Neutron instrument components (cont`d)

- Absorbing filters
  - Cadmium to remove unwanted thermal neutrons below 500 meV
  - Thin gadolinium layers (high absorption < 100 meV) for slower neutrons
    - Time-of-flight (TOF) technique (pulsed neutrons):
      - remove slow neutrons that would be overtaken by faster neutrons from next pulse
- Scattering filters
  - Suppressing higher order harmonics (λ/n) of monochromators
    - HOPG (great for getting rid of ≈1.2 Å)
    - Saphire (getting rid of epithermal and hot neutrons)

Neutron instrument components (cont`d)[3]

- Crystal monochromators
  - Steady-state source instruments: large single crystals monochromators
    - Select neutrons of a particular wavelength
  - Wavelength: scattering angle & monochromator crystal-plane spacing
  - Resolution in wavelength depends on
    - Angular collimation before monochromator, α_0,
    - Collimation after monochromator, α_1,
    - Monochromator mosaic spread η_M (all in scattering plane)
    - Low angles: good resolution by good collimation
    - High angles, good resolution with relaxed collimation

$$\frac{\delta\lambda}{\lambda} = \cot\theta_M \sqrt{\frac{\alpha_0^2\eta_M^2 + \alpha_1^2\eta_M^2 + \alpha_0^2\alpha_1^2}{\alpha_0^2 + \alpha_1^2 + 4\eta_M^2}}$$

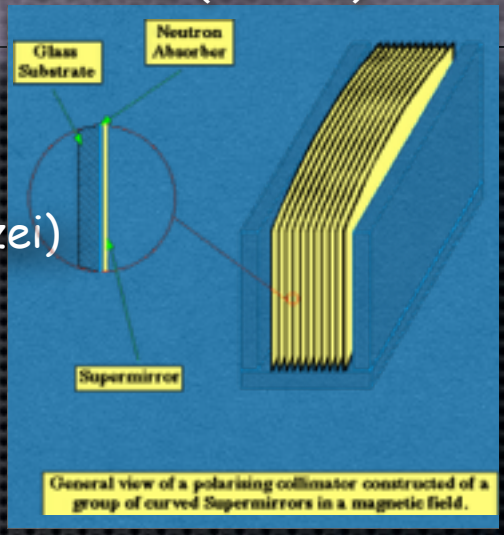
  - Neutron flux from monochromator:
    - Out-of-plane collimations β, crystal reflectivity P_M:
  - Given resolution: collimations comparable and mosaic spread large
    - Vertical collimation to permissible spread in scattering vector out-of-plane.
  - Pyrolitic graphite, silicon, germanium, copper at desired mosaic spread
    - Bent crystal or several smaller pieces mounted in a curved way: focussing

Neutron instrument components (cont`d)[4]

- Neutron choppers
  - Reactor sources: pulsed beams for TOF experiments
    - Fermi choppers, rotating at very high frequencies
- Neutron velocity choppers
  - Approximately monochromated beams without pulsing
    - rotating cylinder with set of helical grooves cut into its outer surface
    - spinning on its axis
  - Coarse monochromation of 5-20%, e.g., for small-angle scattering
- Scattering filters
- Resonance filters
- Sample magnetic fields
- Polarizing monochromators
  - Heusler alloy Cu_2MnAl (111) reflection
    - almost equal nuclear and magnetic scattering lengths
    - magnetised crystal: scattered neutron polarised

Neutron instrument components (cont`d)[5]

- Supermirrors: polarizing reflection
  - Total reflection from magnetic layers
  - Polarise cold neutrons without monochromatisation (Mezei)
- Polarising filters
  - Polarised ^{3}He gas: capture only of one polarisation direction
    - Protect the gas from any outer magnetic field
    - Sufficient thickness and pressure needed
- Polarised beam in guided field
  - Polarisation direction can be slowly rotated as well
- Spin flippers
  - Polarisation dependence of cross sections:
    - Spin flippers reverse rapidly the neutron spin.
  - Mezei flipper for monochromatic neutrons:
    - controlled field from a rectangular coil,
    - precesses the neutron spin by given angle
    - By changing field with time, a white, pulsed beam can be flipped



Neutron instrument components (cont`d)[6]

- Precession field
  - Length of flight path with horizontal magnetic field
    - Opportunity to precess by a defined angle
  - Crucial for the spin-echo technique

Neutron instrument components (cont` d)[2]

- Powder samples
  - A few cm^3 (or mm^3…) of polycrystalline (or amorphous) "powder" in (hopefully …) cylindrical can (sometimes double-walled)
    - vanadium can ideal (for diffraction, not for spectroscopy!)
      - adding no significant peaks to Bragg diffraction pattern of polycrystalline material
      - flat background contribution from incoherent scattering

Neutron instrument components (cont` d)[2]

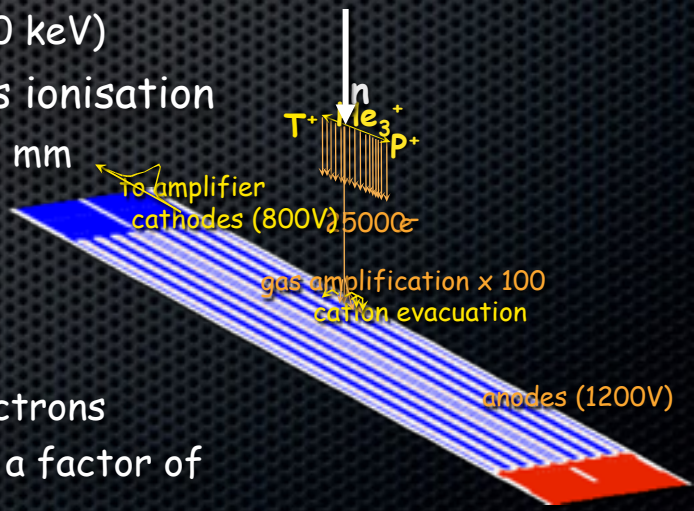
- Powder samples
  - A few cm^3 of polycrystalline or amorphous powder in cylindrical can
    - vanadium can ideal (for diffraction, not for spectroscopy!)
      - adding no significant peaks to Bragg diffraction pattern of polycrystalline material
      - flat background contribution from incoherent scattering
- Detectors

Neutron detection

- BF_3 or ^3He gas detectors
  - Single detectors or multidetector bank arrangements
  - Position sensitive detectors (PSD), 1- or 2-dimensional
    - Multi-wire or micro-strip gas chamber detectors
    - Resistive wire detectors
- Image plates and scintillation detectors

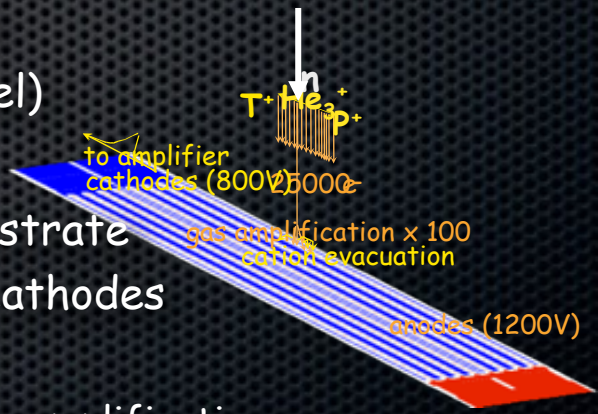
Neutron detection (cont` d)

- Gas chamber detector
  - Charged particles produced in the detection gas by neutron capture
    - ^{3}He captures thermal neutrons with high cross section
    - Produces tritium nucleus and proton as secondary particles,
  - Secondary particles moving randomly in opposite directions
    - Neutron's incident energy of some meV smaller than decay energy 770 keV
    - Distributed to both particles (T^+: 200 keV, p^+: 570 keV)
  - Slowed down on trajectory, loosing energy by gas ionisation
    - Mixture of 3 bar ^{3}He and 1 bar CF_4 : total trace 6 mm
    - creating about 25000 primary electrons
  - Primary electrons drift towards the anode
  - Strong field gradient close to anode
    - Acceleration of electrons, creating secondary electrons
    - Gas-amplification of primary electrons amplify by a factor of about 100
  - Electron avalanche creates a signal on anode
    - amplified and discriminated electronically



Neutron detection (cont` d)[2]

- Gas chamber detector
  - Neutrons: much stronger signal than other ionising particles (γ-rays)
    - Easily discriminated
  - Discriminated signals fed into the data acquisition system (DAS)
- Multi-wire gas chamber detector (MWGC)
  - Several anode wires in continuous gas chamber: PSD
  - One or more wires form one detector cell (or channel)
- Micro-strip gas chamber detector (MSGC)
  - Anodes: chromium layers sputtered upon a glass substrate
  - Detector cell of several parallel, close anodes and cathodes
  - Fast evacuation of gas-amplification cations
    - No positive charge density near the anodes, corrupting amplification
  - Several micro strip detector cells on one glass substrate plate: 1D-PSD
    - Several plates assembled in a single large gaschamber detector housing:
      - Huge, curved PSD without gaps in solid angle of detection
  - Each neutron must be counted only once!



Neutron instrument components (cont`d)[2]

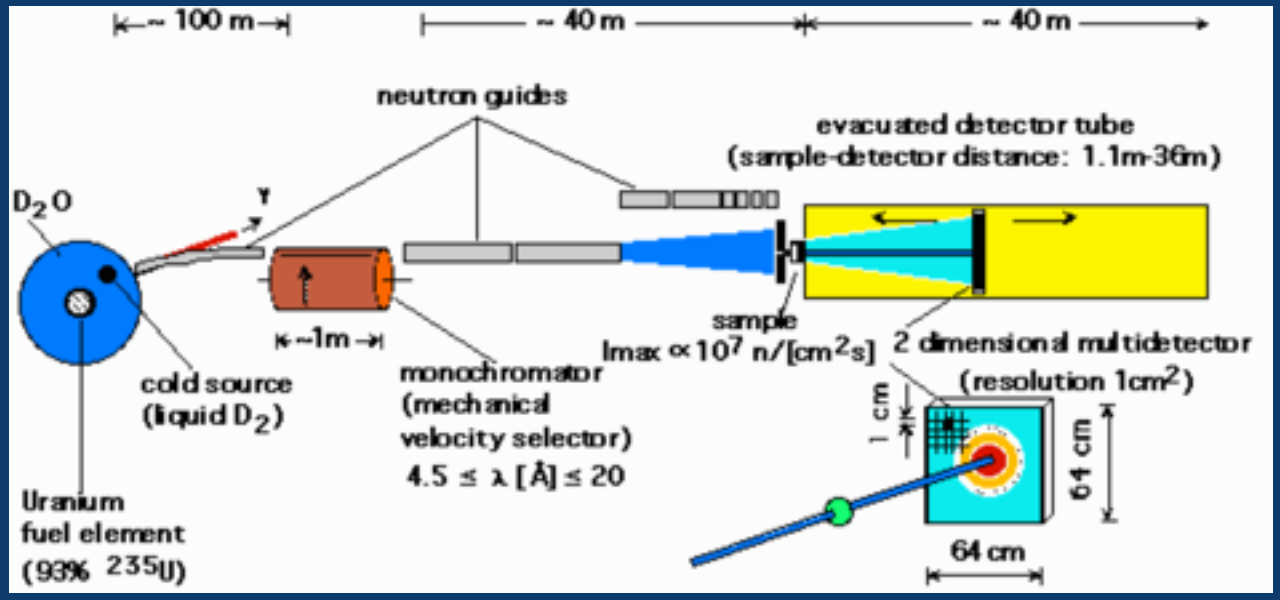
- Powder samples
  - A few cm^3 of polycrystalline or amorphous powder in cylindrical can
    - vanadium can ideal (for diffraction, **not** for spectroscopy!)
      - adding no significant peaks to Bragg diffraction pattern of polycrystalline material
      - flat background contribution from incoherent scattering
- Radial collimators
  - we'll come back to this later
- Detectors
- Time-of-flight analysers
  - Most instruments on pulsed sources
  - Chopper instruments on a steady-state source
  - Electronic device handles
    - several detector channels of multidetector or PSD
    - with sufficient large number (hundreds to thousands) of time channels.

Diffractometers

- Small angle neutron scattering
  - SANS
  - Reflectometry
- Two-axis neutron diffractometers
  - High resolution diffractometers (HRPD)
    - “Strain scanners”
  - High Intensity powder diffractometers
  - High-*Q* diffractometers
- Diffuse scattering diffractometers
- Single crystal diffractometers
  - Four-circle diffractometers
  - Laue diffractometer

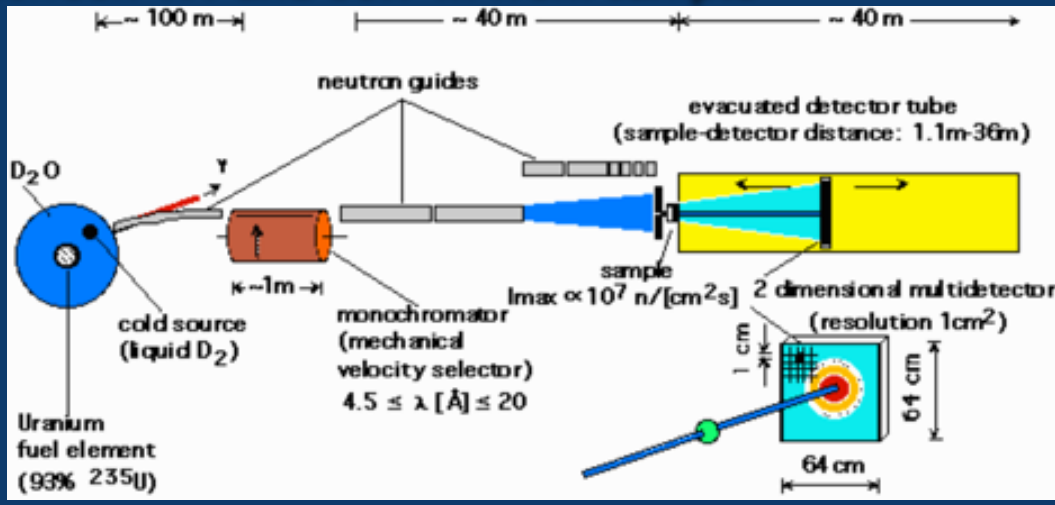
Small-angle scattering

- Low *Q* vectors from 0.005 to 0.5 $Å^{-1}$
- Not generally Bragg diffraction of crystalline materials
  - Except some, mostly biological applications
- Defects, precipitates, voids, macromolecules
  - Anything from 10 to 1000 Å
  - Contrast in scattering-length compared with its surroundings



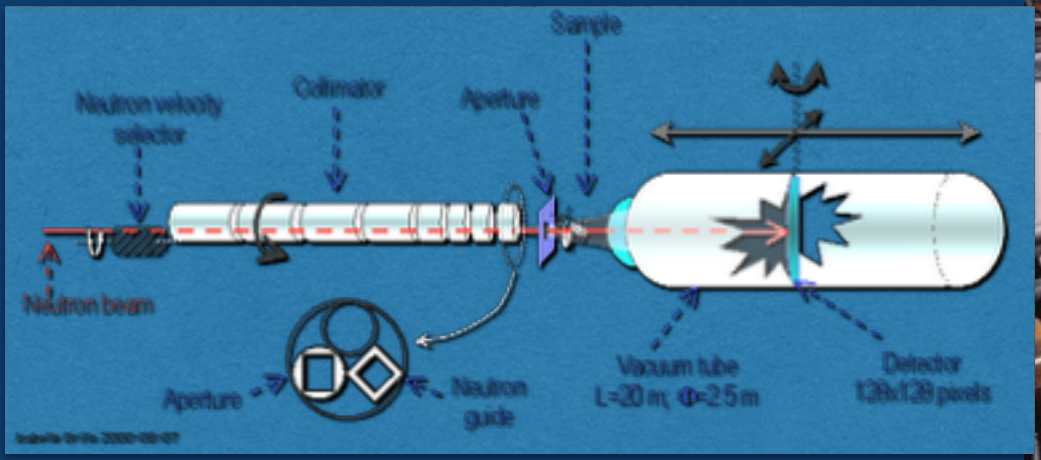
Small-angle scattering (cont`d)

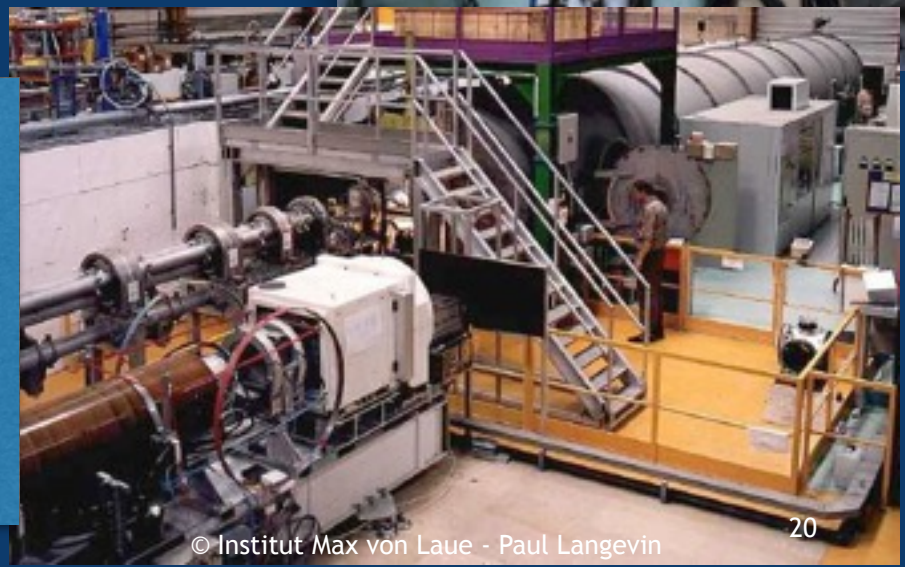
- D11: typical long, pinhole geometry SANS instrument
  - Neutrons from vertical cold source 100 m from first part of D11
    - 140 m from the sample position, detector in very low background
  - Monochromatisation by helical slot velocity selector
    - neutrons ±9% of mean wavelength, determined by rotation speed
  - Collimation by a series of moveable glass guides
  - Sample zone, situated 40 m down-stream from velocity selector
  - 64×64 cm ^{3}He multi-detector
    - moveable within tube between 1.1 and 36.7 m from sample
    - *Q*-range $5 \cdot 10^{-4}$ - 0.3 Å^{-1}.



Small-angle scattering (cont`d)[2]

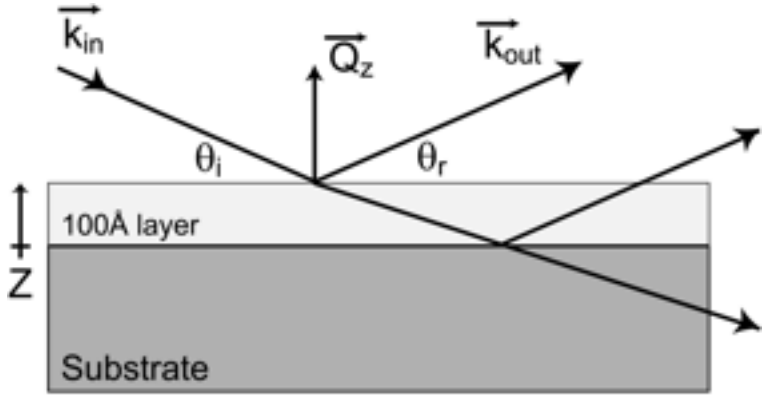
- D22: highest flux at sample from 4 to 40 Å
  - brilliant horizontal cold source
  - short rotor & high transmission of velocity selector
  - relatively large beam cross-section (55×40 mm)
- Maximal dynamic Q-range, Q_{max}/Q_{min} ratio: 50
- Reflectometers for surface studies:
  - D17, FIGARO



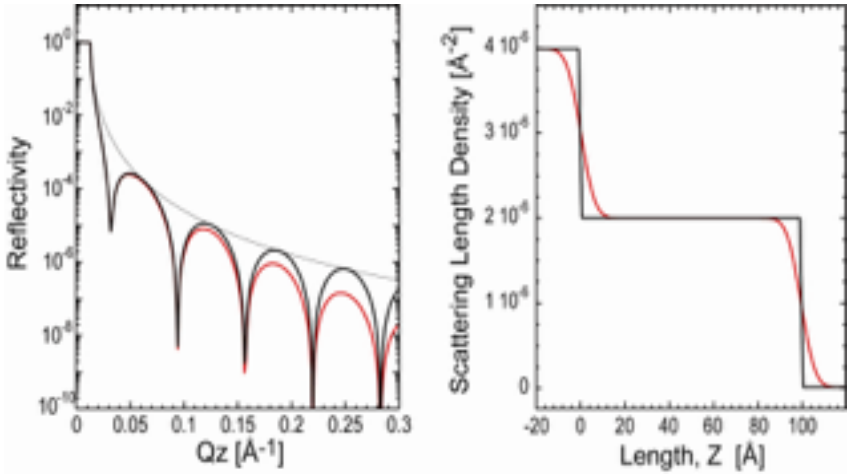


Neutron reflectivity (NR)…

…a technique to study interfaces and thin films



$$|\vec{Q}_z| = \frac{4\pi \sin(\theta)}{\lambda}$$



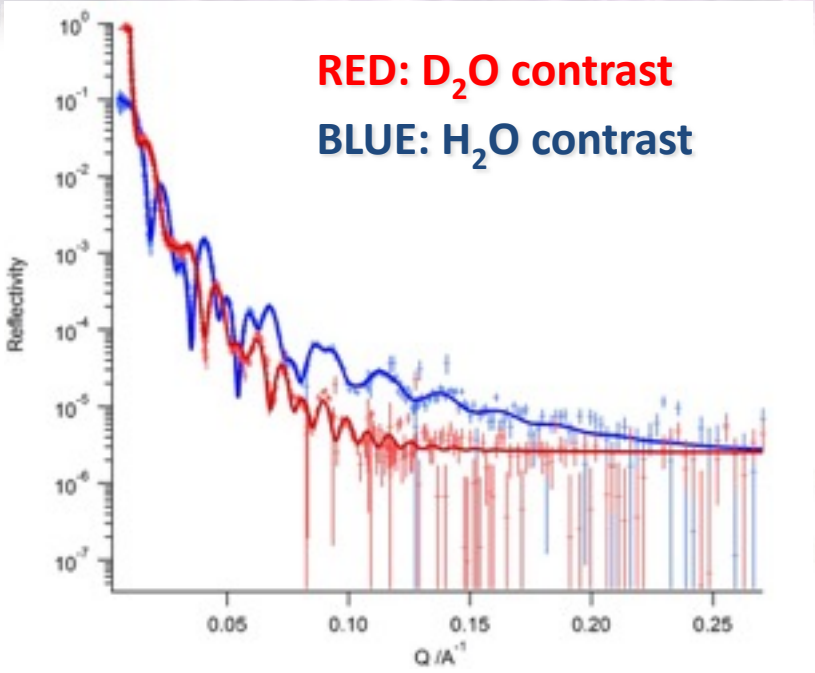
Neutrons strike a flat sample at small glancing angles

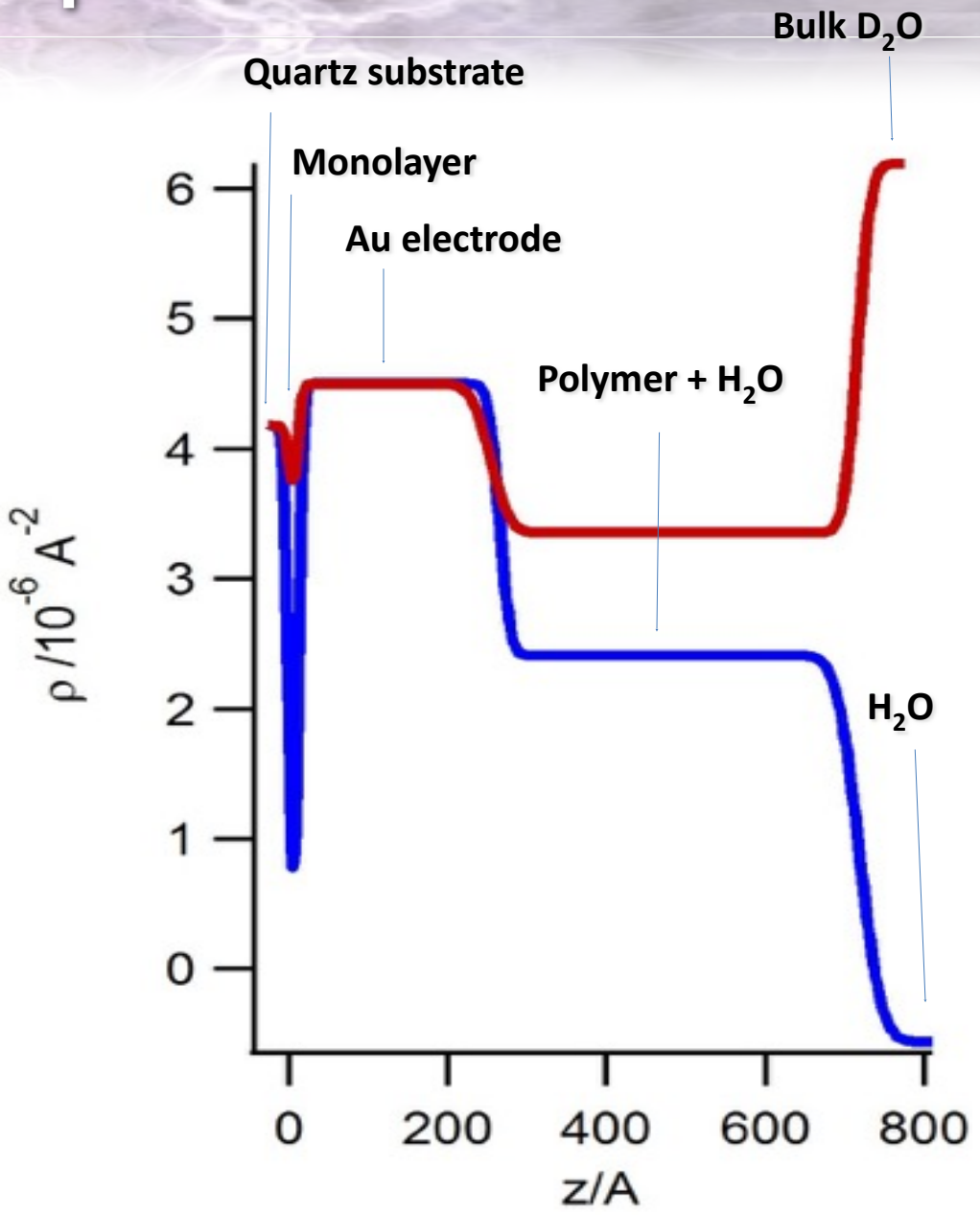
- Reflection and refraction from interfaces
- Interference between the various reflections

Provides out-of-plane structure

- Average density normal to the interface
- Layer thickness, density and roughness
- Ideal nano scale probe for structure & composition

An example of NR data





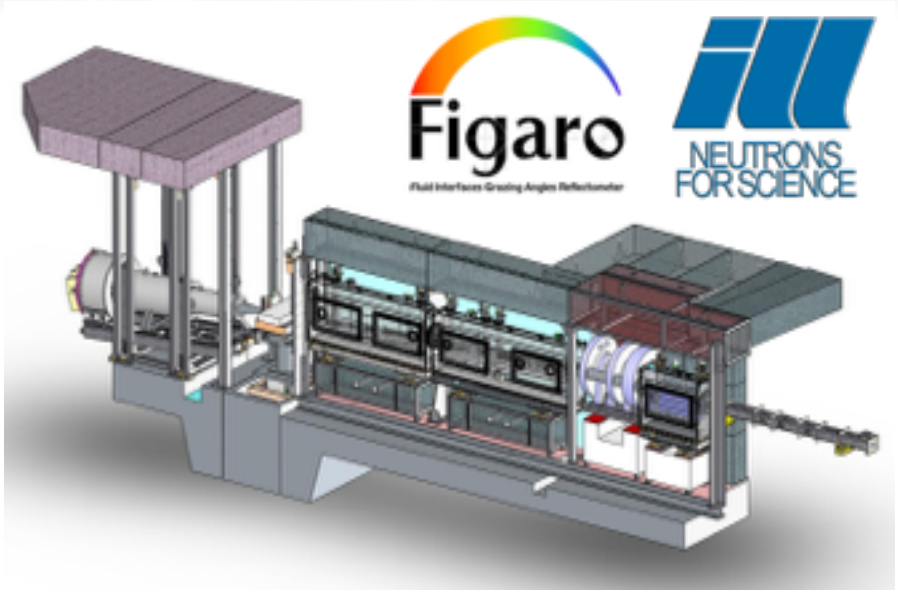
Electroactive polymer deposition on Au electrode

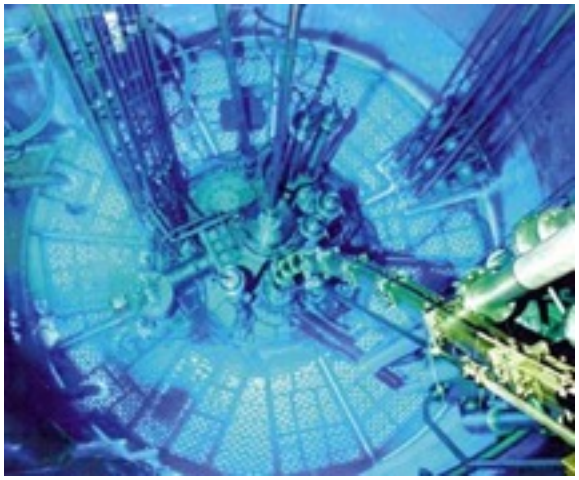
Multiple contrasts improve confidence in the model and quantify water content in the polymer film

Fluid Interfaces Grazing Angles ReflectOmeter

FIGARO stats:

- world-leading instrument from 2009
- white beam flux of 1.4 x 10^8 n mm^{-2} s^{-1}
- flux at sample: 4 x 10^2 n mm^{-2} s^{-1}
- Large beam size: 0.5-5 mm x 40 mm
- Resolution: 1-10% $\Delta Q/Q$
- Dynamic range of 6-7 orders in R





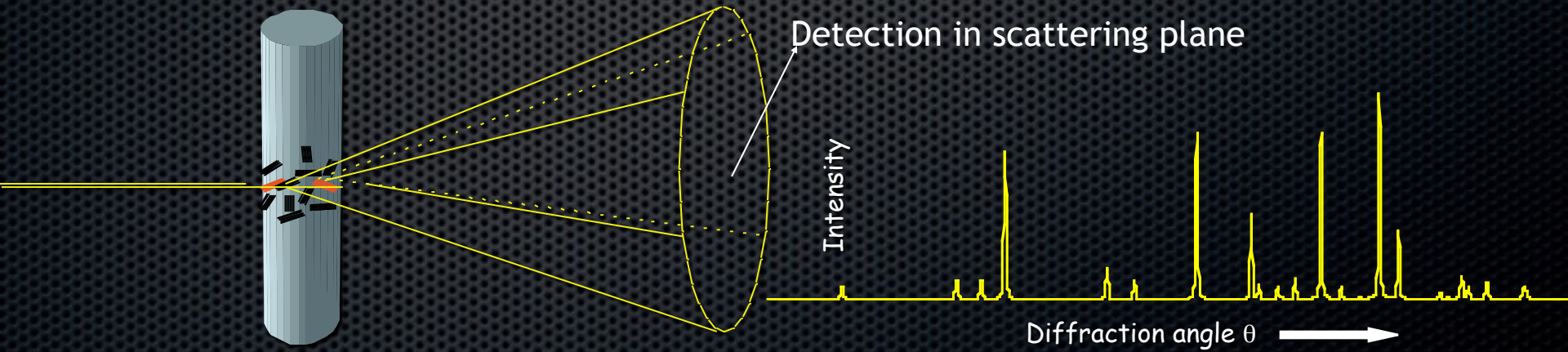
ILL reactor

FIGARO examples:

- polymer film structure
- nanoparticle interactions
- DNA & protein studies
- solvent drying in glues & paints
- rheology of polymer blends
- virus interactions with model membranes
- detergent & formulation optimization

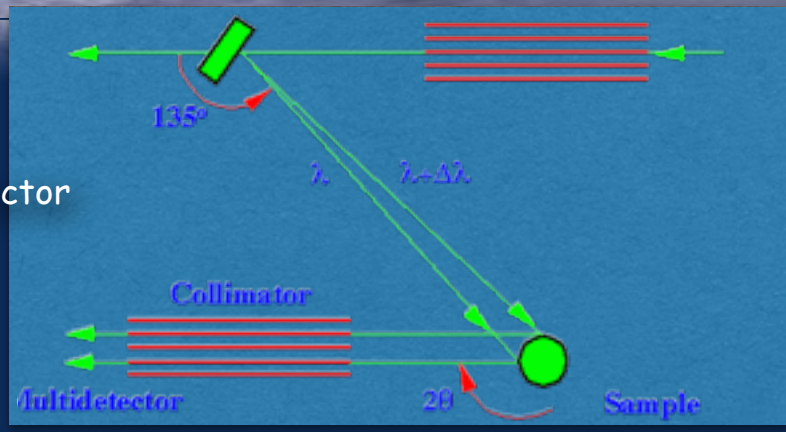
Two-axis powder diffractometers

- Intermediate Q range from 1 to 10 $Å^{-1}$
  - Powder samples for structural refinement
  - Needs adequate resolution:
    - Peaks closely spaced at higher the Q vector and for large unit cells
    - Resolution effectively determines size of refinable unit cell
    - Angular width of Bragg peak:
      - known function of collimations and monochromator mosaic



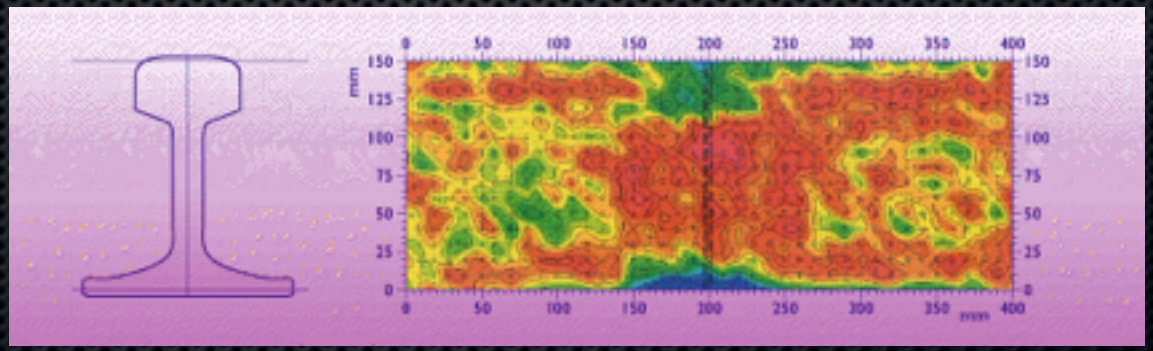
High resolution 2 axis diffractometers

- Common features (D2B at ILL):
  - high take-off angle from the monochromator
  - set of Soller collimators in front of scanning multidetector bank
- D2B: Very high take-off angle (135°) for monochromator
    - Ge monochromator with large mosaic spread of 20'
      - compensate for the corresponding intensity (Δλ/λ) loss
    - 300 mm high, focusing vertically onto about 50 mm
  - 200, now 600 mm high multidetector bank and Soller collimators
    - match this large incident vertical divergence
    - diffraction pattern : 50, now 25 steps of 0.05° in 2θ
    - 64, now 128 detectors spaced at 2.5° , now 1.25°
    - Scans take typically 30 minutes and repeated to improve statistics
  - Rietveld structure refinement of polycrystalline powder patterns
    - zeolites with absorbed molecules, superconductors, quasicrystals
  - Magnetism: high resolution at large d-spacings
    - higher wavelengths of 2.4 Å and 6 Å



High resolution 2 axis diffractometer

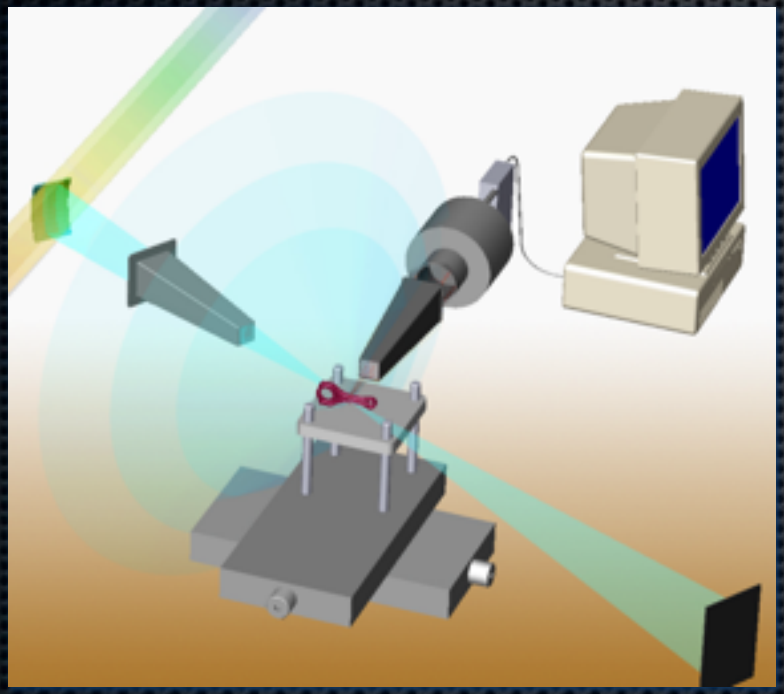
- Determination of residual stress (SALSA)
  - Shift of single Bragg peak: strain gauge
    - measured d-spacing represents compression or expansion
  - Stress tensor: strain measurements in three directions
    - calculate the stress tensor from strain, using Hooke's law
  - Small gauge volume defined
    - Incident slits and slits on the detector side
    - better, radial collimators instead
    - Rectangular shaped gauge volume:
      - Bragg peak: scattering angle 2θ close to 90°

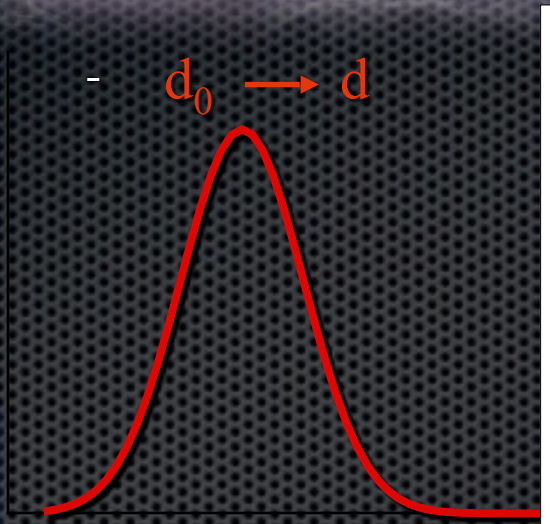


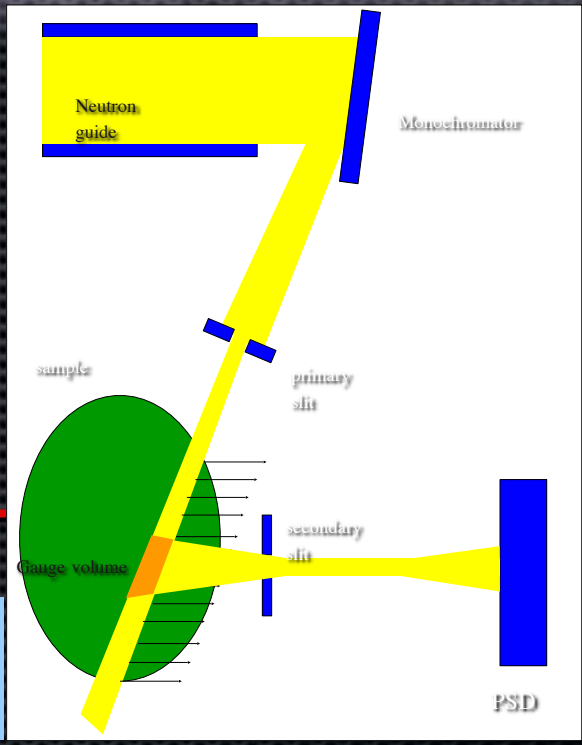
Neutron strain imaging

Diffraction from a (small) gauge - volume =>

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







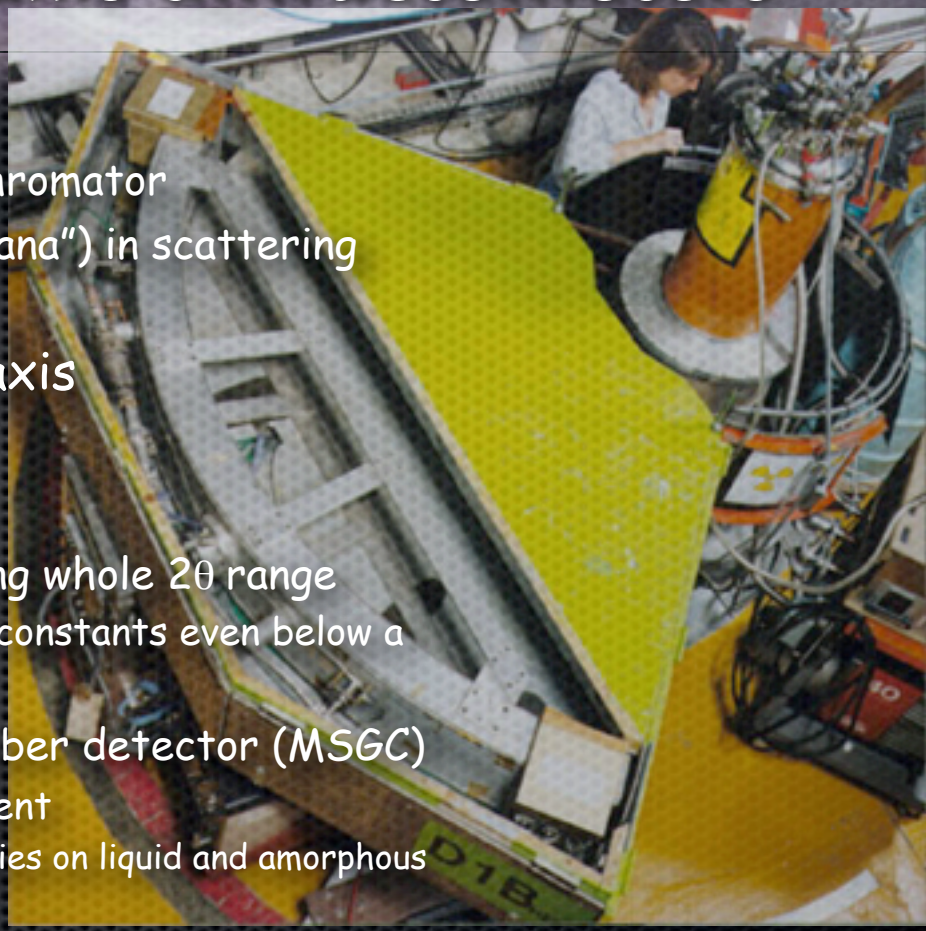
strain $\varepsilon = \dfrac{d - d_0}{d_0}$

Principal axes known:
3 measurements, Hooke's law

$$\sigma_x = \frac{E(1-\nu)}{(1-2\nu)(1+\nu)}\varepsilon_x + \frac{E\nu}{(1-2\nu)(1+\nu)}(\varepsilon_y + \varepsilon_z)$$

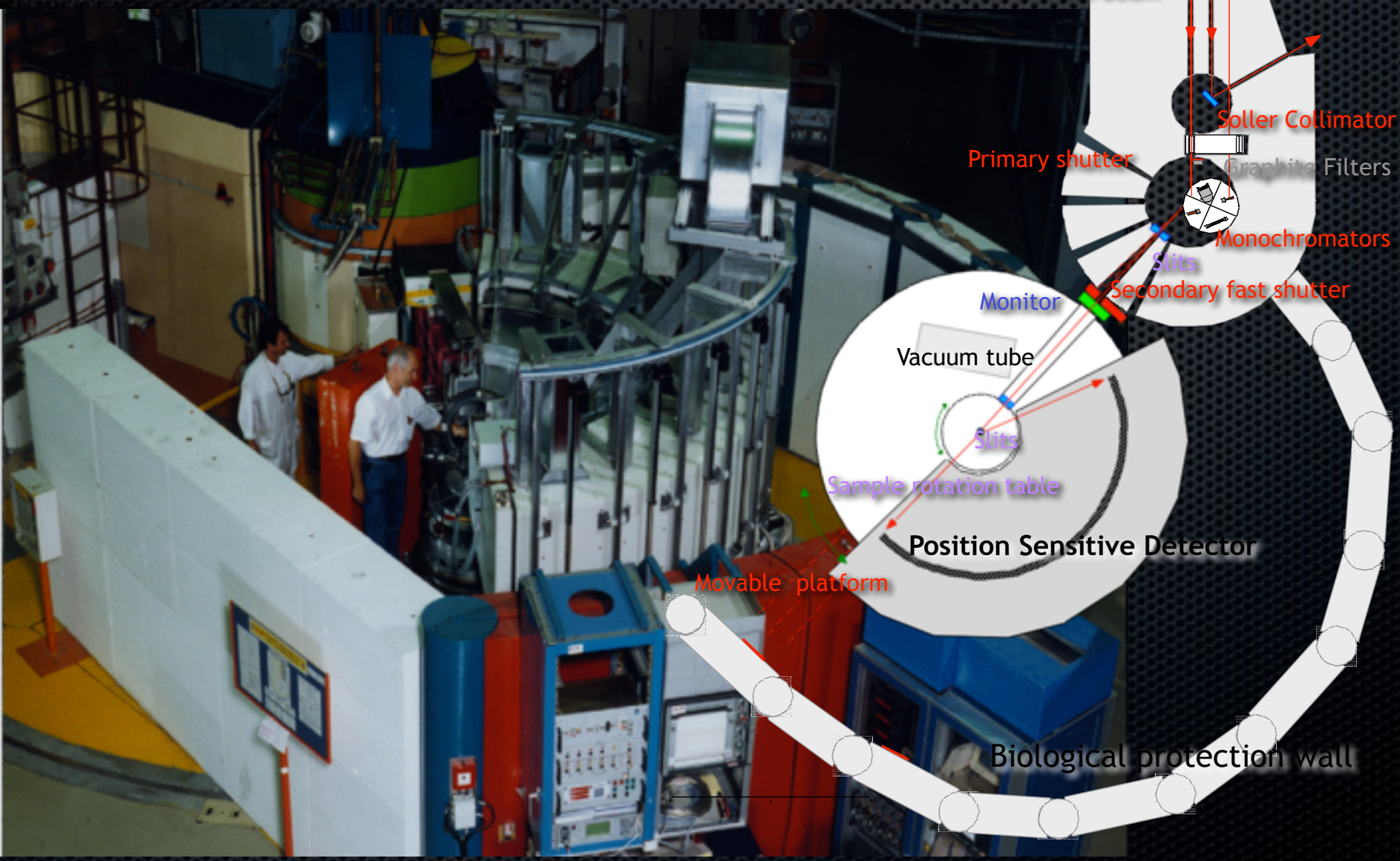
SALSA – Stress Analyzer for Large scaled engineering Applications

High intensity 2-axis diffractometers



- D1B and D20
  - lower take-off angle from the monochromator
  - large, 1-dimensional curved PSD ("banana") in scattering plane.
- D20: medium to high-resolution 2-axis diffractometer
  - Very high flux at the sample position
  - Stationary, curved linear PSD, covering whole 2θ range
    - in-situ diffraction studies with time constants even below a second
  - High stability of micro strip gas chamber detector (MSGC)
    - High precision in intensity measurement
      - differential measurements and studies on liquid and amorphous systems
  - Different monochromators and take-off angles
    - Optional incident beam Soller collimators and secondary slits
    - Large choice in Q, resolution, wavelength and flux
    - Various levels of complexity and rapidity of observed phenomenon.

High intensity 2-axis diffracto ers



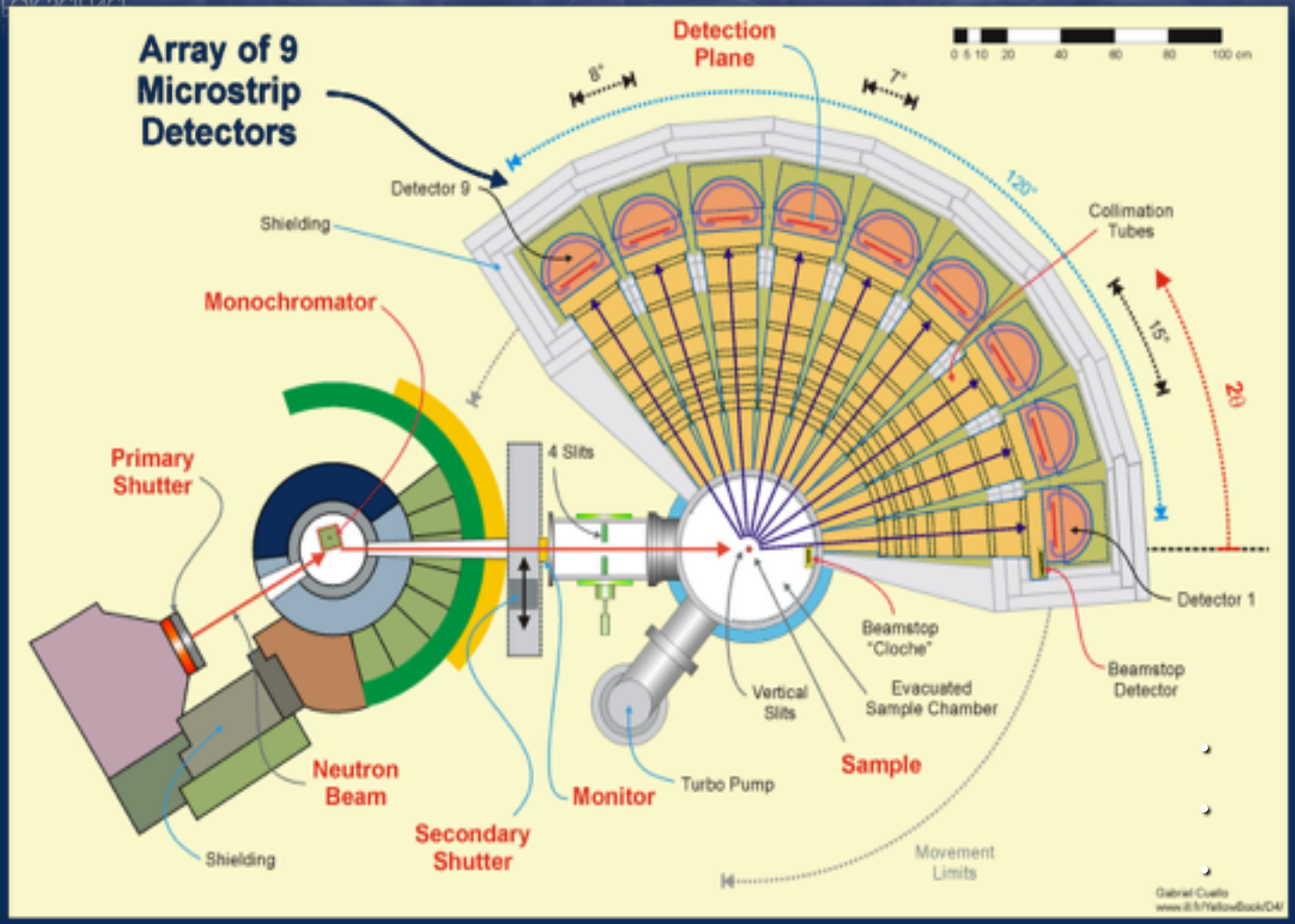
High intensity 2-axis diffractometers

- D20: High flux and stationary large PSD
  - Numerous short time measurements
    - Investigation of phase transitions
      - induced by temperature change ('thermodiffractometry')
      - or pressure
    - Kinetic studies ('one shot' experiments)
    - Large number of samples
    - Sample at many different orientations (texture analysis)
    - Very small samples
    - Stroboscopic acquisitions for fast repetitive phenomena
  - Very high precision of the measurement of diffracted intensity
    - disordered systems
    - physisorption

High-Q diffractometers

- Q ranges from 2 to 50 $Å^{-1}$
  - Determination of glass and liquid structure factors
  - Spatial resolution in radial distribution function about $2\pi/Q_{max}$
    - Good resolution demands a scan to highest Q values
  - Correction factors for inelastic effects (Placzek)
    - Scattering angles not too large
  - Therefore: hot source and low monochromator take-off angles
    - Intense short-wavelength beam
    - Angular scan can cover a large range, limited to $Q_{max} = 4\pi/\lambda_m$.
- D4C, dedicated high-Q diffractometer on hot source
  - bank of 9 small MSGC PSDs (64 cells, 8° each)
  - angular "scan" in only two steps.

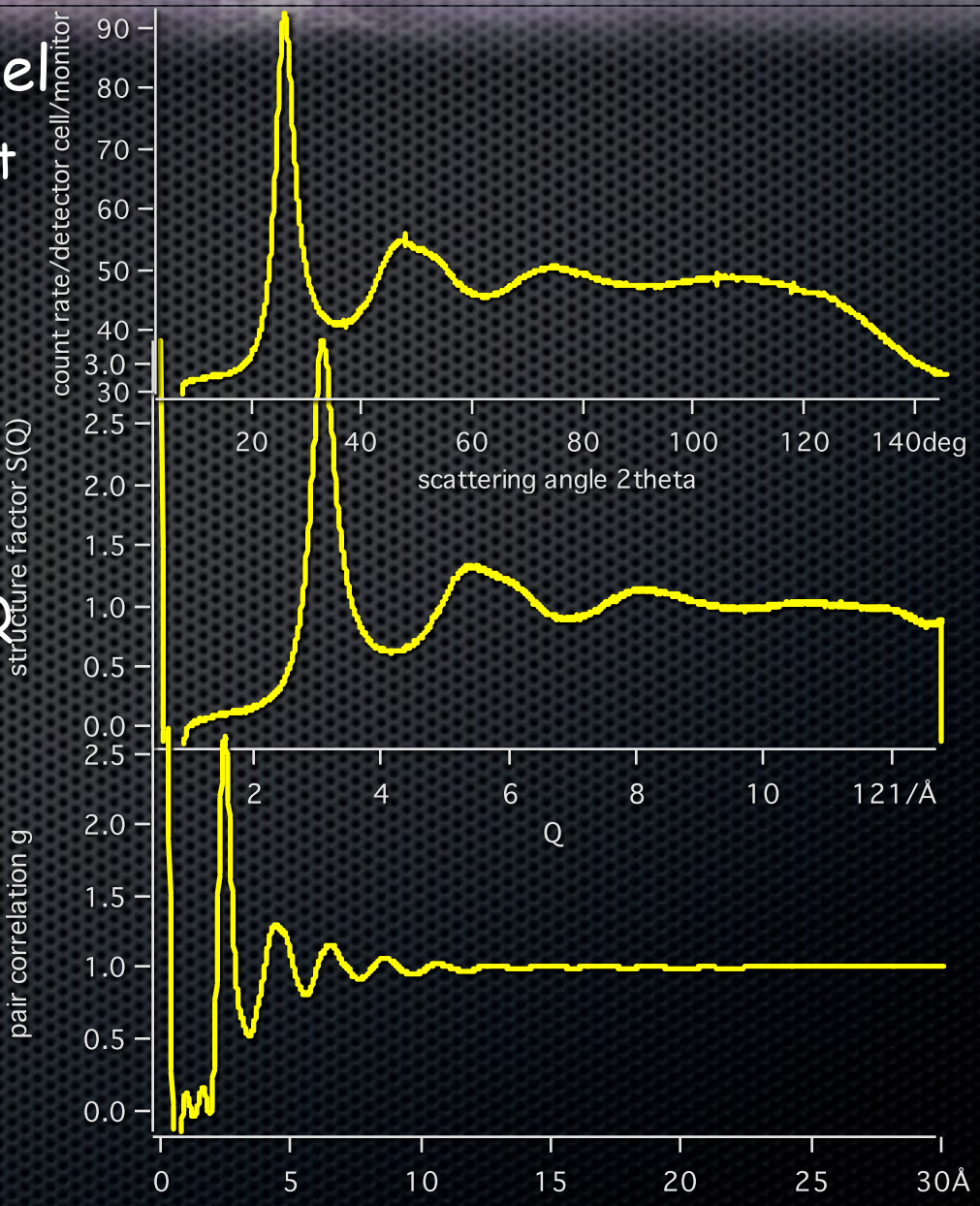
Liquids diffractometer D4C



- Large Q-range
- High stability
- High flux
- Very low background
- Simple corrections

High-Q diffractometers (cont` d)

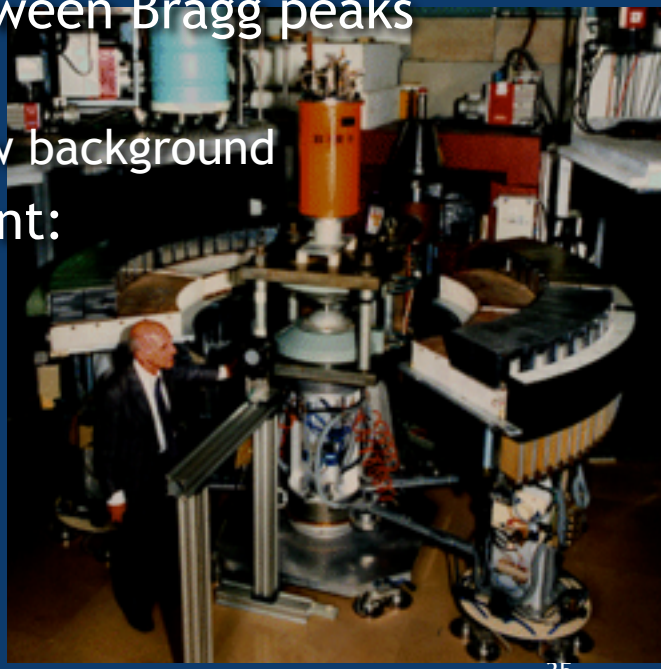
- Undercooled liquid Nickel
  - 290 K below melting point
- Diffraction pattern
  - D20, 0.94 Å
  - count rate vs. angle
- Structure Factor
  - vs. momentum transfer Q
- Pair Correlation
  - vs. distance in real space



- Dirk Holland-Moritz,
- Thomas Schenk,
- Virginie Simonet

Diffuse scattering diffractometers

- Single crystal-specimens
  - No need for high resolution in *Q* to resolve adjacent peaks
    - Peaks resolved by orientation of the crystal
  - Relaxed but precisely defined resolution required
    - for precise intensity measurements
- Defect structures or disorder in crystalline material:
  - smoothly varying or diffuse scattering between Bragg peaks
    - weak compared with Bragg scattering
    - relaxed collimation while maintaining a low background
  - Relaxed *Q* resolution of 5% usually sufficient:
    - diffuse scattering as function of Q
  - *Q* range from 0.5 to 5 $Å^{-1}$.

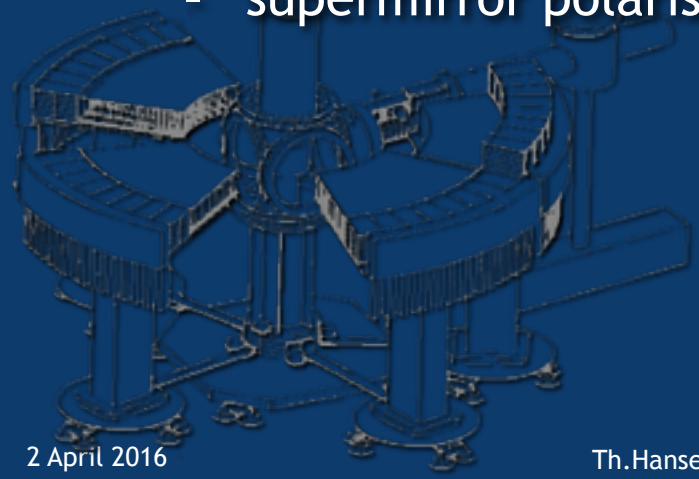


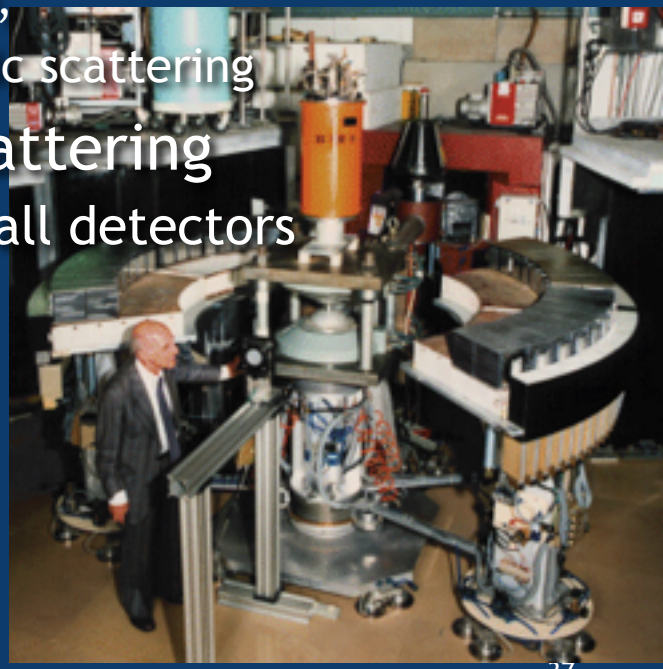
Diffuse scattering diffractometers

- Cold neutrons around 4 Å: relaxed angular collimation
- Neutron guide: low background
- Roughly monochromatic beam:
  - curved graphite monochromator array.
  - velocity selector on a white beam may be used
- Large area of detectors
  - Extending some degrees outside the scattering plane
- Simple chopper in front of sample, gating the counter
  - Only neutrons arriving in time window of elastic scattering
  - Eliminates thermal diffuse scattering from inelastic scattering
- Magnetic diffuse scattering separated from nuclear scattering
  - without polarized neutrons
  - magnetic field
    - rotate direction of sample magnetization parallel to scattering vector
    - remove all magnetic scattering

Diffuse scattering diffractometers

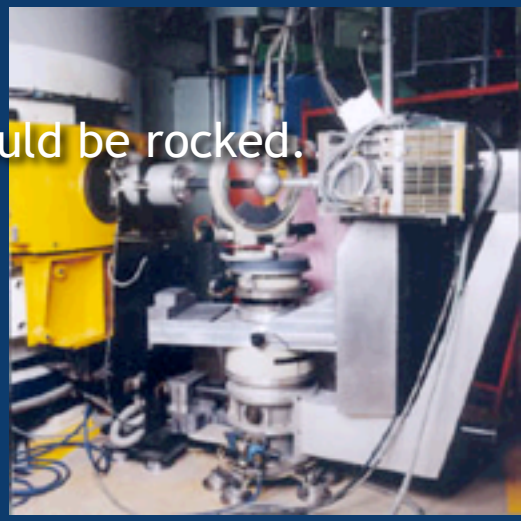
- Incident polarized beam, sample in external field
  - Polarization-dependent cross sections
  - Neutron spin flipper between the polariser and sample
  - Ratio of neutron counts with flipper on and off: “flipping ratio”
    - determines magnetic diffuse scattering
  - Pseudorandom correlation sequence applied to flipper,
    - followed by time of flight cross correlation,
    - chopper dispensed with for removing elastic scattering
- Full polarization analysis of diffuse scattering
  - supermirror polarisers before sample and all detectors





Four circle diffractometers

- Solution and refinement of crystal structures
  - Measurement of numerous reciprocal lattice point intensities
  - Accurate intensities: Scan over each reflection
- Four-cycle diffractometer
  - well collimated roughly monochromatic incident beam
  - Small sample bathed in the beam
  - Large detector(PSD) integrate neutrons scattered by the sample
  - For small mosaic spread of sample, wavelength spread of incident beam:
    - Spot in reciprocal space elongated parallel to **Q**
      - Integrated by performing a "$\theta/2\theta$" scan,
      - which also performs a parallel scan in reciprocal space
  - For samples with broad mosaic, the sample angle should be rocked.
- ILL: D19, D9, and D10



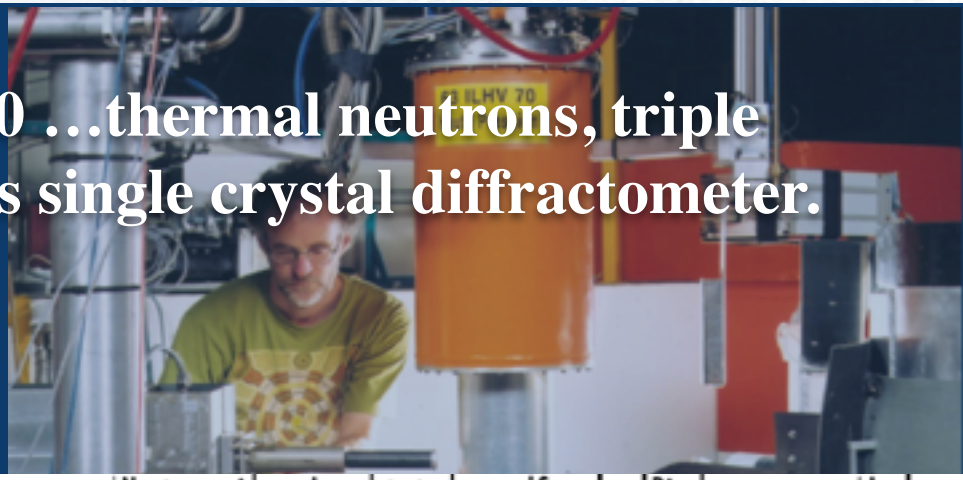
Single Xtal diffractometers at ILL

monochromator	
vertically focusing and continuously variable	
pyrolytic graphite	$1.8 < \lambda < 6$ Å
Cu (200)	$1.1 < \lambda < 3$ Å
incident energy range	$2.5 < E_o < 68$ meV
relative energy resolution $\delta E/E$	5×10^{-3}
absolute energy resolution δE	0.02 THz at $k_i = 1.55$ Å^{-1}
$\lambda/2$ contamination	$< 2 \times 10^{-4}$ at 1.26 Å with Cu monochromator and no analyser $< 10^{-5}$ at 1.26 Å with Cu monochromator and PG (008) analyser

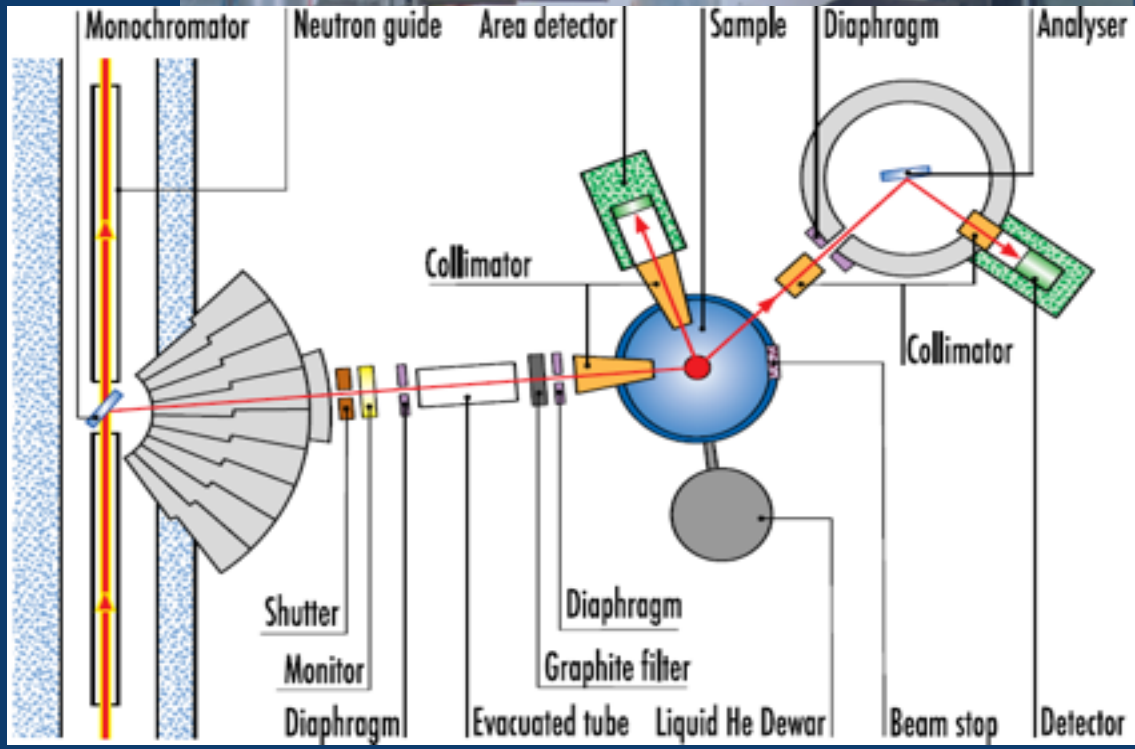
sample	
beam size	10×8 mm^2
flux	5×10 6cm^{-2} s^{-1} (= 2.36 Å; PG-monochromator and filter) 2×10 6cm^{-2} s^{-1} (= 1.26 Å; Cu-monochromator)

analyser
vertically focusing pyrolytic graphite

80x80 mm^2 area detector, or single ^{3}He detector	
background	< 3 cpm (without analyser) 0.5 cpm (with special detector shielding or with analyser)

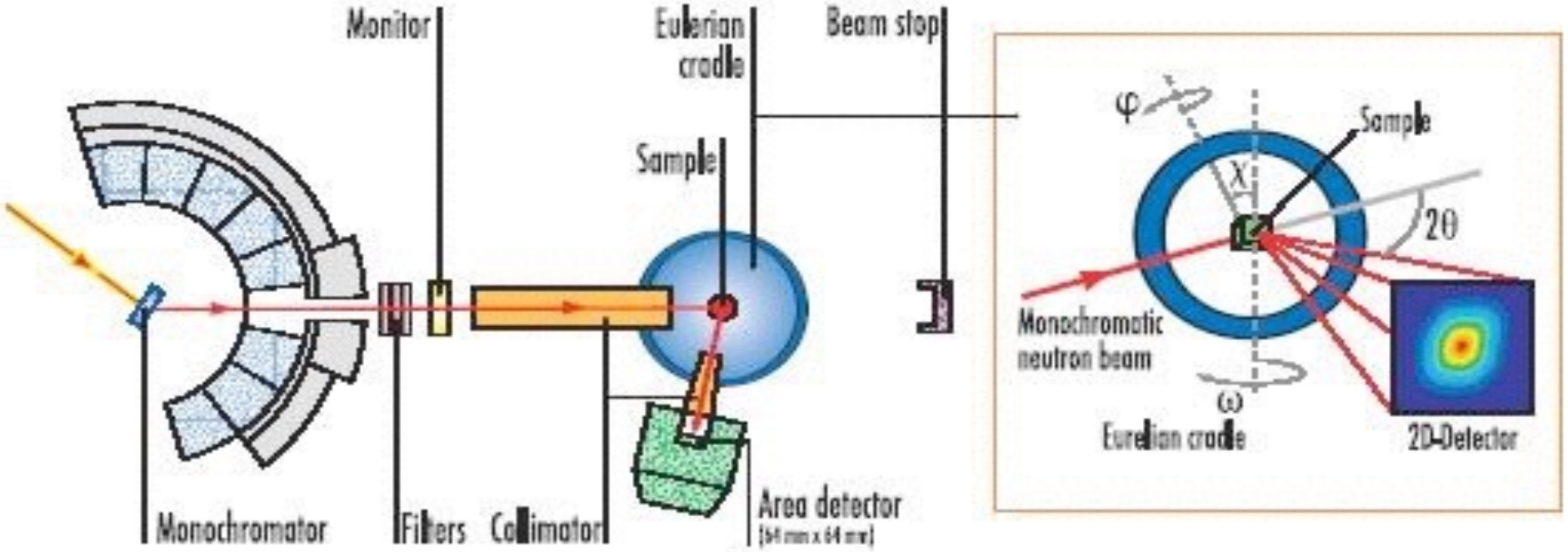


D10 …thermal neutrons, triple axis single crystal diffractometer.



Single Xtal diffractometers at ILL

D9 …hot neutrons, high Q single crystal diffractometer.



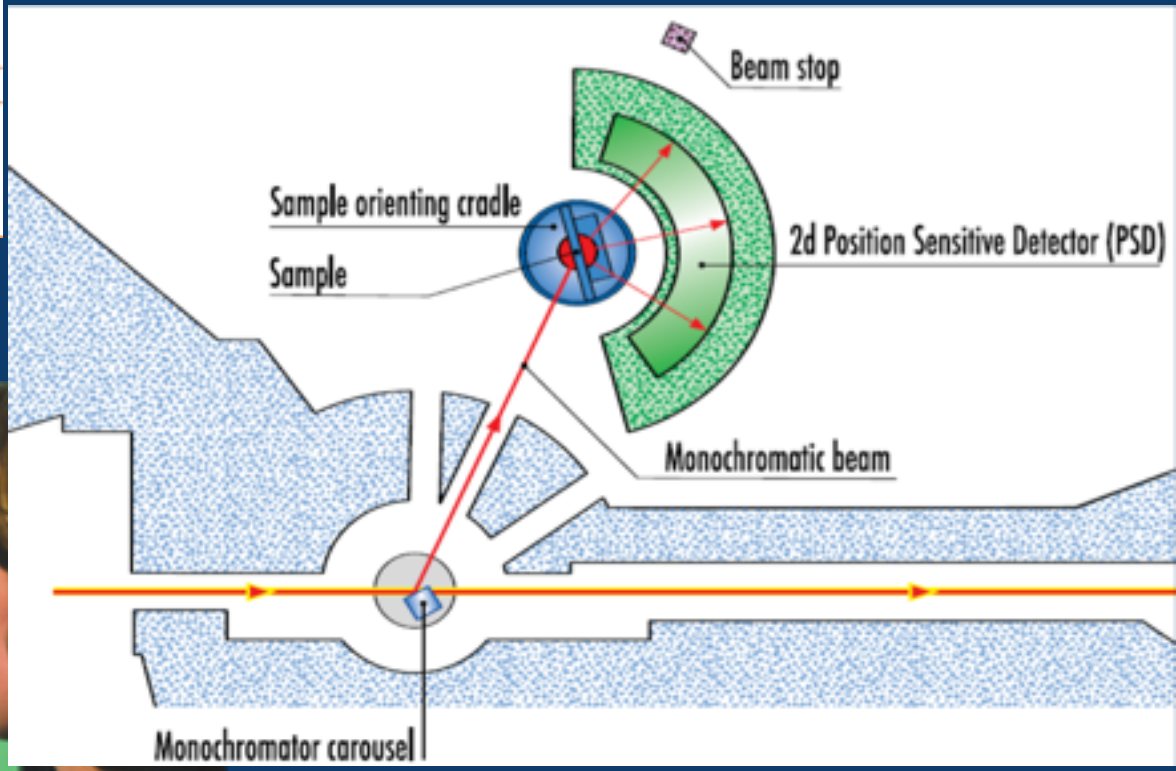
Single Xtal diffractometers at ILL

monochromators	
vertically focussed	graphite (002); germanium (11n), (335)
flat	Cu (220)
Bragg angle $2\theta_M$	42.7°, 70° or 90°
wavelength	$0.95 < \lambda/\text{Å} < 2.4$

sample	
max. beam size	10 mm
flux	$10^7 .. 10^8$ n cm^{-2} s^{-1}
angular range	$-60° < \gamma < 140°$ $-45° < \omega < 210°$ $+56° < \chi < 205°$ $-180° < \phi < 180°$

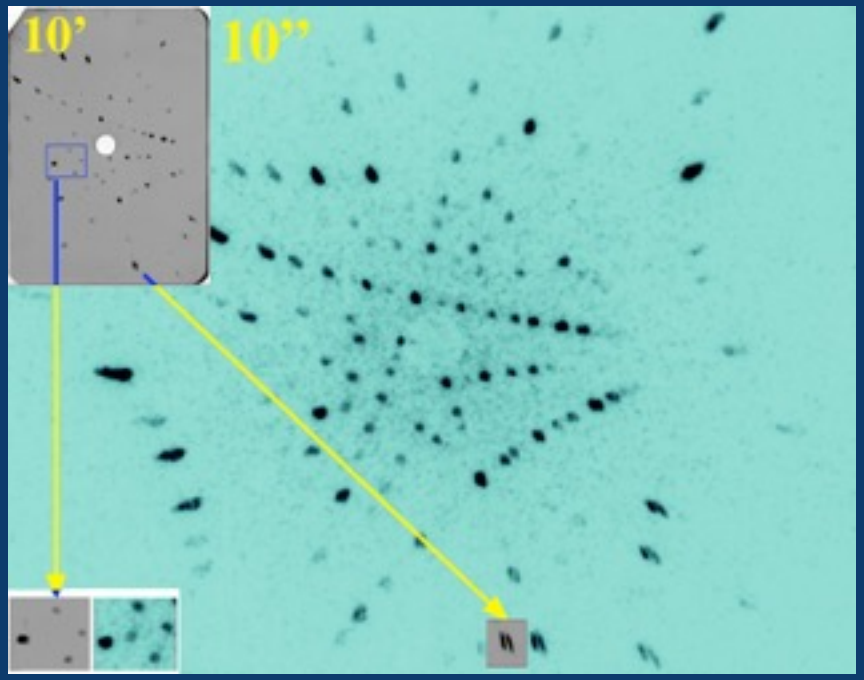
detector	
124°x30° position-sensitive detector detector gas: ^{3}He (5 atm) + CF_4 (1 atm)	
resolution for fixed sample-detector distance of 75 cm	0.23° x 0.23°

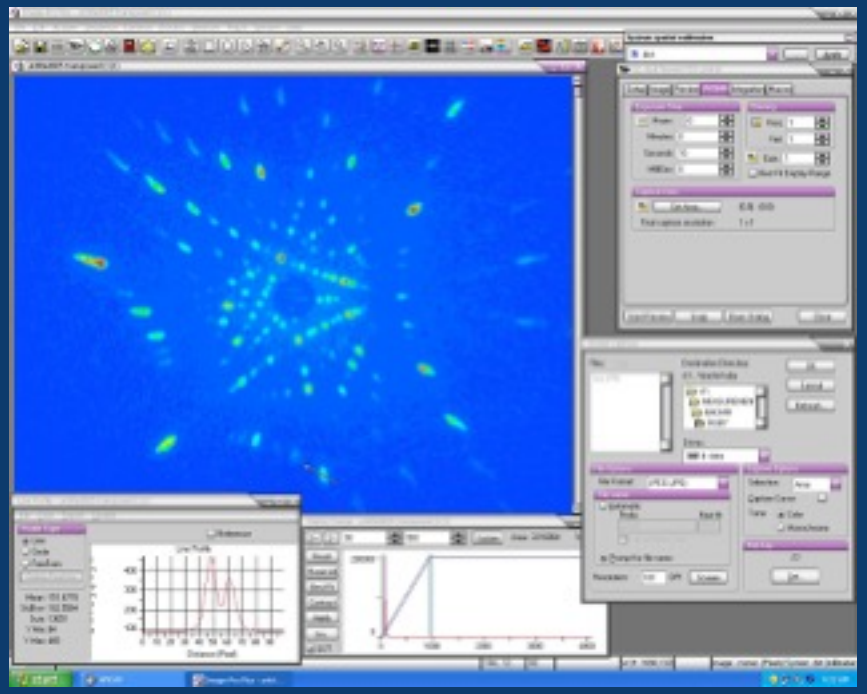
D19 …thermal neutrons, fibre, and big unit cell single crystal diffractometer.



Single crystal diffractometers

Laue and quasi-Laue diffractometers: VIVALDI, Orient Express, CYCLOPS





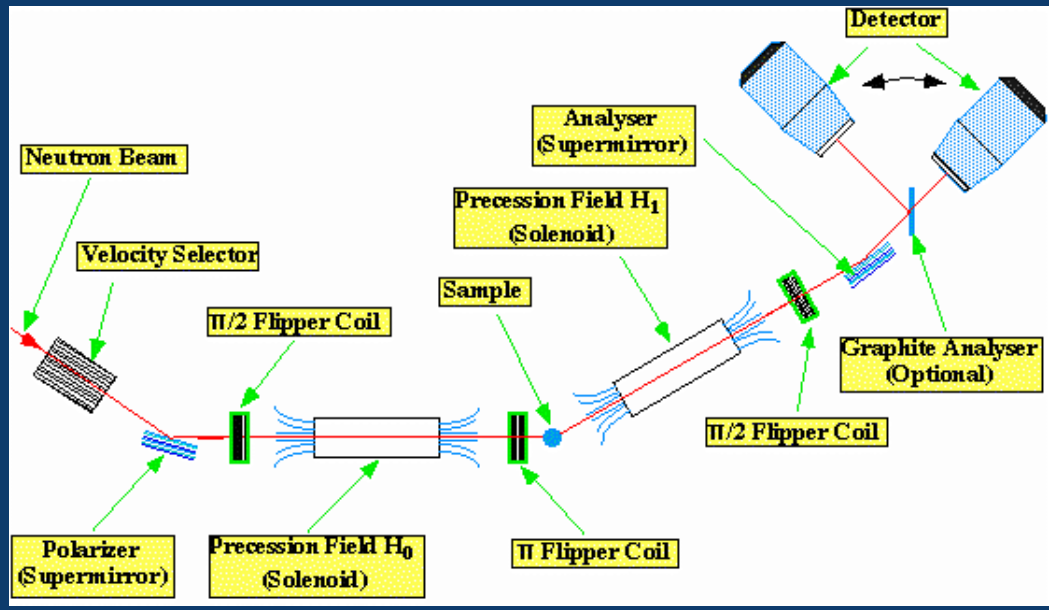
Laue diffraction pattern obtained from a ruby crystal in 10 s with OrientExpress compared to that obtained from the same crystal in 10 mn with Kodak film

Spectrometers

- "elastic scattering" used rather loosely in neutron scattering
  - often applied wrongly to the total scattering cross section
  - or the integral over all energies of the double differential cross section
  - This cross section measures spatial transform of instantaneous structure
- Energy analysis methods for elastic component of total scattering
  - $S(Q,\omega = 0)$
  - spatial transform of the time-independent structure
- Elastic scattering within an energy resolution $\delta\omega$
  - Structure averaged over a time $2\pi/\delta\omega$
- Quasi-elastic scattering
  - Scattering which merges continuously into elastic scattering
  - Samples where nuclei or spins are undergoing diffusion
  - giving rise to a characteristic Lorentzian distribution
  - Scattering at low energy transfers compared to the incident energy E_0

spin-echo spectrometer

- Quasi-elastic spectrometer
  - Energy transfers are measured directly
    - instead of difference between specified incident & scattered energy
  - Very high energy resolution, 10^{-6} meV, with good intensity
  - Cold neutrons roughly monochromatic and polarized
    - Polarisation direction moved horizontally by a $\pi/2$ flipper
  - Incident neutrons precess in horizontal precession field
    - Rotation angle
      - inversely proportional to incident velocity
    - Neutron polarisation reversed in π flipper
  - Scattered neutrons "unwound" in similar precession field



Back-scattering analyser methods

- Best intensity at given resolution at back scattering
  - Nearly exact back scattering in incident and scattered beams
    - from nearly perfect silicon crystals,
    - Resolutions up to 0.3 μeV
  - Silicon (002) reflection gives 2.074 meV neutrons in back scattering.
- On reactors: how to scan the incident energy?
  - Velocity changes induced by a moving monochromator
    - Doppler effect
    - Pyrolitic graphite crystal reflects back-scattered beam to sample
    - Large analyser crystals arrays back-scatter from sample to detectors
    - Chopper removes neutrons scattered directly from sample

TOF spectrometers

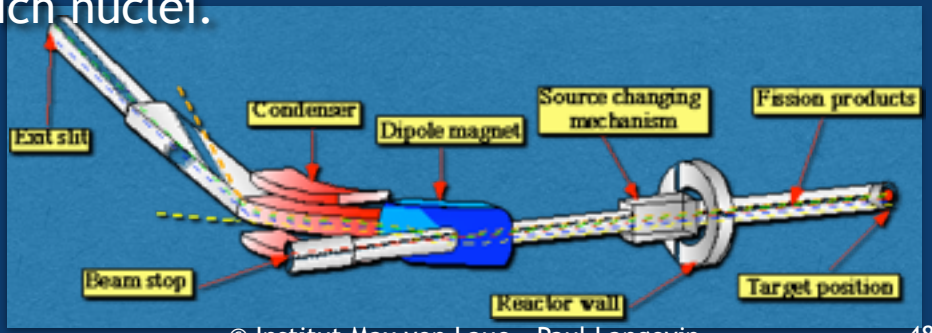
- Vibrational spectroscopy
  - magnetic equivalent, crystalfield spectroscopy
  - polycrystalline samples
  - good energy resolution required, *Q* resolution not
  - absolute value of *Q* remains important
  - 1st-order vibrational scattering
    - intensity increases as *Q* attenuates by Debye-Waller factor
  - Higher-order multiphonon scattering
    - increases at still higher powers of *Q*.
- TOF spectrometers for low Q
  - merge into those for quasi-elastic scattering
  - can go to higher-energy transfers as well.
  - even useful at reactors: at ILL: IN4, IN5 and IN6

triple axis spectrometer

- Single crystal inelastic instruments
  - close degree of definition of scattering vector **Q**, and energy
  - degree in precision depends
    - on the size of reciprocal lattice,
    - on the rate of change of normal mode energy with wave vector.
- Triple axis spectrometer
  - air-cushion ported
  - View a chosen point in (**Q**,ω) space
    - one degree of freedom left to choose
    - e.g., any incident energy $E_{0,}$ - constant-*Q* scan in three ways:
      - constant E_0,
      - constant E_1,
      - constant scattering angle.

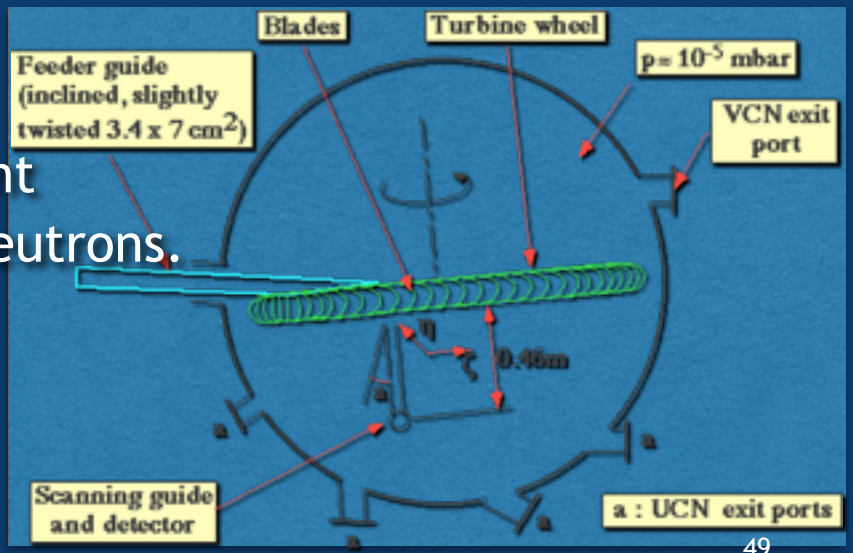
Instruments for nuclear physics

- Lohengrin (PN1)
  - Recoil mass separator for unslowed fission products
  - Fission products from fissile isotopes source
    - placed in beam tube near reactor core
  - Fission products selected
    - Magnetic and an electric sector field
      - Deflections perpendicular to each other
  - Freely recoiling fission products analysed according to
    - Energy over ionic charge (E/q)
    - Mass over ionic charge (A/q)
  - Main directions of research
    - Fission process
    - Spectroscopy of very neutron rich nuclei.



Instruments for particle physics

- Ultracold neutron facility PF2
  - density of 50 cm-3 of ultracold neutrons (UCN)
    - with speeds less than 6 m/s
  - UCN produced at top end of a vertical guide
    - neutrons with speeds of 50 m/s
    - converted by Steyerl turbine into UCN with about 5 m/s
  - Then horizontal guides to several experiments in parallel
  - Output for very cold neutrons (VCN) with wavelengths of 100 Å
  - UCN: option for storage in traps
  - Experiments
    - Neutron lifetime
    - Neutron electric dipole moment
    - "anomalous losses" of stored neutrons.



Anatomy of an instrument by Monte-Carlo simulation

High-Intensity

2-Axis Neutron Diffractometer D20

D20

Versatility

4 monochromators

15 takeoff angles

Soller collimators, slits

resolution
Q range
intensity

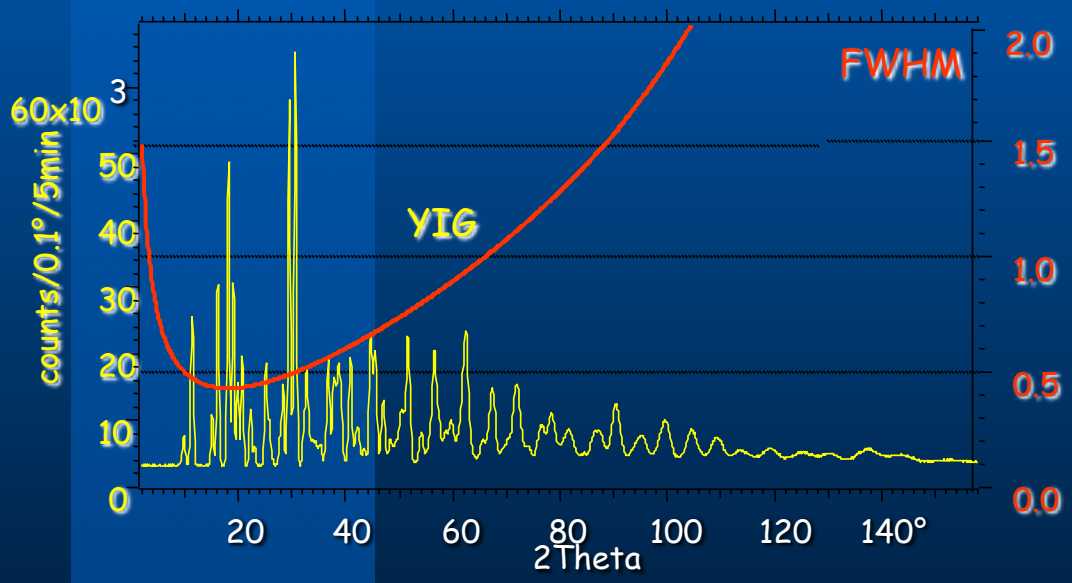
- Crystallography
- Kinetics
- Magnetism
- Disordered systems

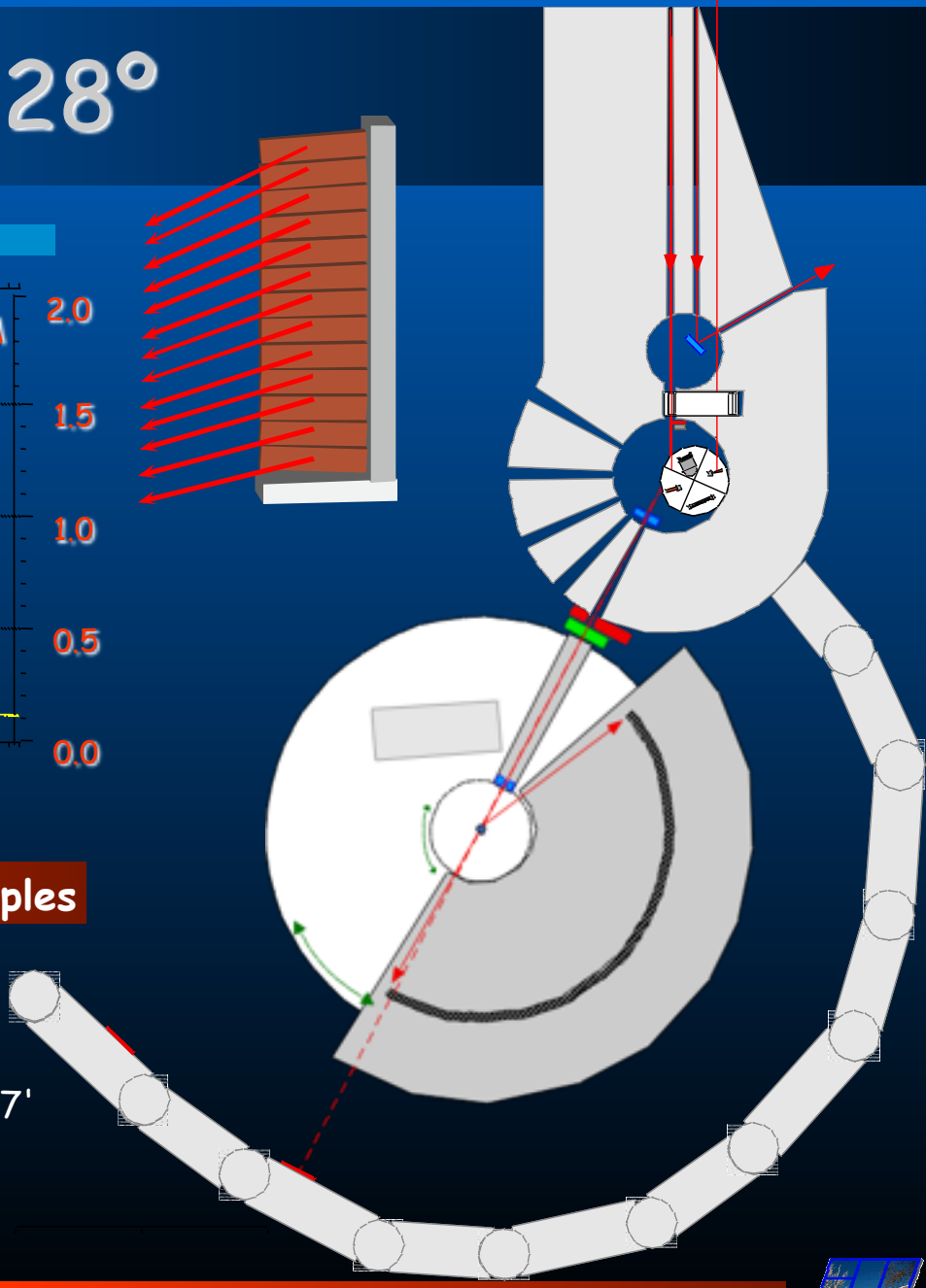
High Intensity

Very high flux at sample:
Position in the reactor

Detecting maximum of neutrons:
Large PSD, 160°, definition 0.1°

Cu (200), takeoff 28°





Disordered systems or absorbing powder samples

Transmission

Fixed focussing

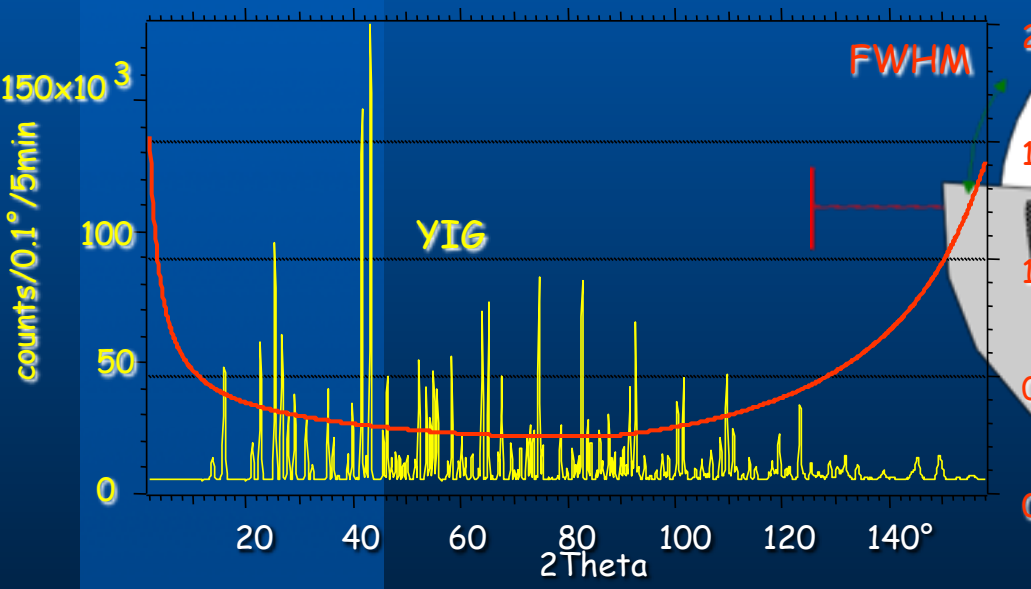
Takeoff angle 28°

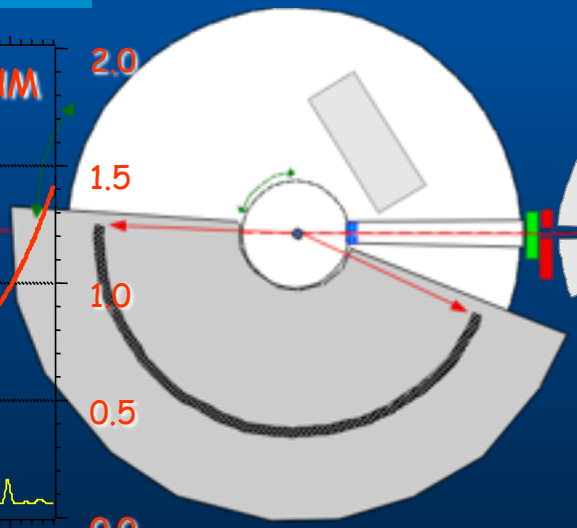
Wavelength 0.874306 Å

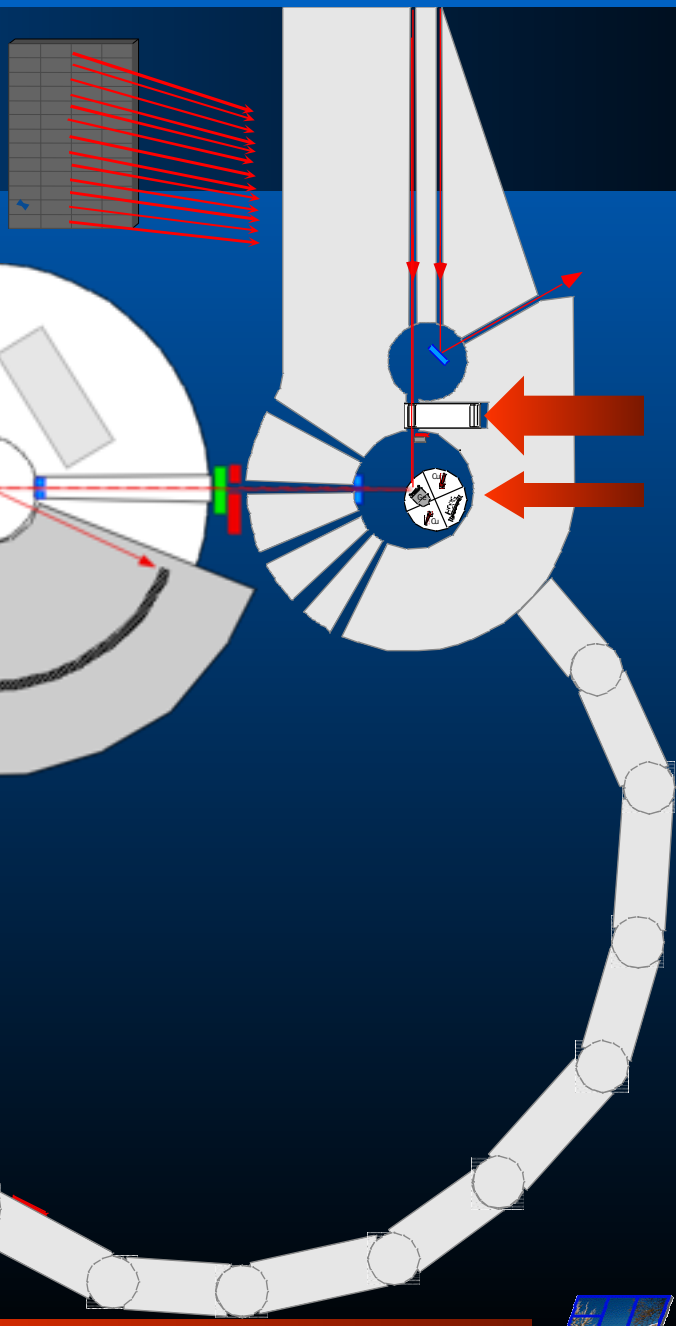
Horizontal incident divergence 27'

0.25/Å < Q < 14.11/Å

Ge(335), takeoff 90°







High Resolution → D2B

Reflection

Variable focussing

Takeoff angle 90°

Wavelength 1.2185Å

Horizontal incident divergence 10'

0.09/Å < Q < 5.16/Å

Why doing Monte-Carlo simulation?

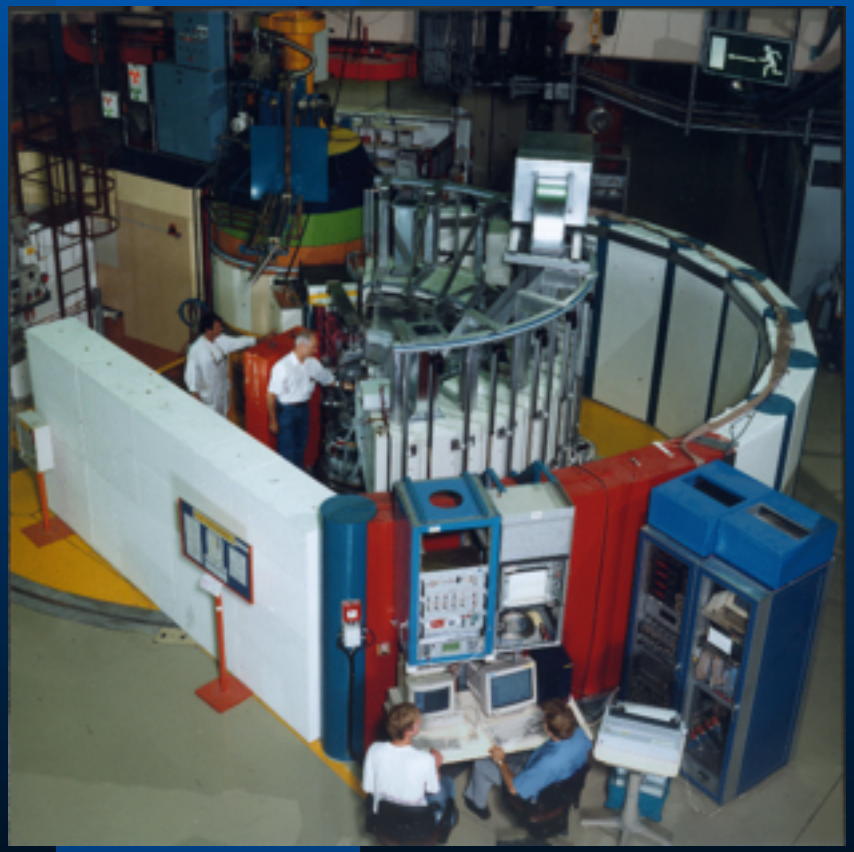
- analytical approach impossible or difficult
- monitoring physically inaccessible neutrons and properties

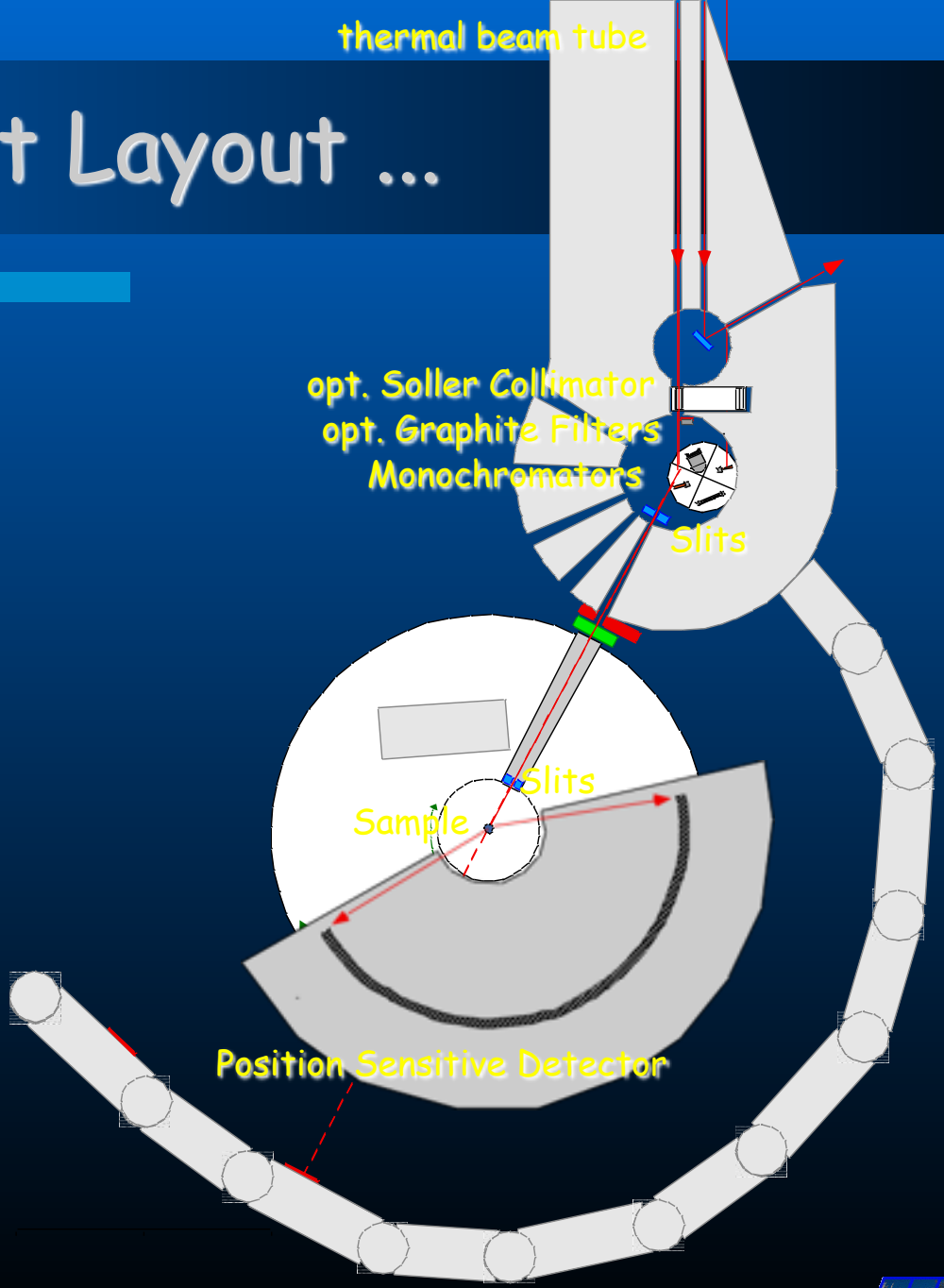
- development of components
- understanding measured data
- de-convolution of sample and instrument contribution
- choice of optimum configuration
- preparation of beam-time request
- estimation of measuring time

McStas neutron ray-tracing

- **Risø National Laboratory, Denmark**
  - K. Lefmann, K. Nielsen, Neutron News **10/3** (1999) p. 20.
  - http://neutron.risoe.dk/mcstas
- **Meta-Language**
  - Instrument composed of sequential components
  - Translation in ANSI-C by McStas
  - Co-ordinate transformation managed by McStas
- **Component Library ...**
  - ... and pre-defined functions

From Instrument Layout ...

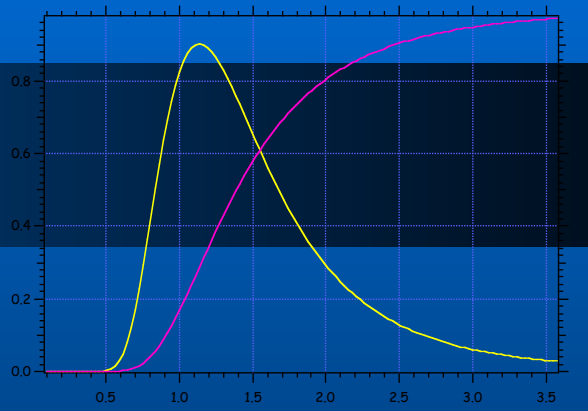




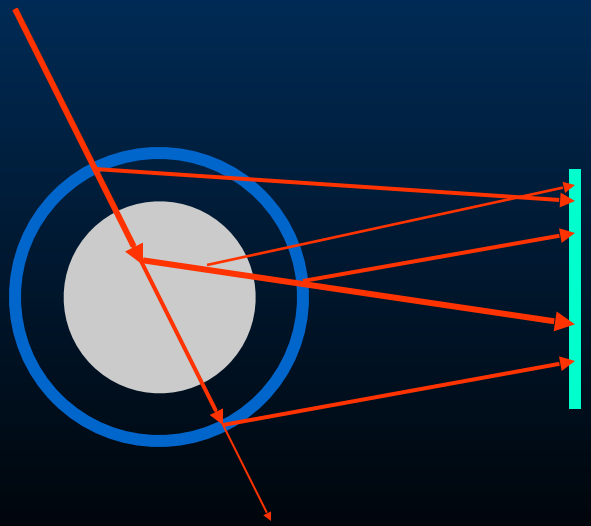
… to McStas Instrument Definition

```
DEFINE INSTRUMENT D20 (Source_alpha, Source_beta, Source_N_E, Source_N_xpos, Source_N_xdiv, Source_Lmin, Source_Lmax, Source_flux,Soller_collim, Soller_misalign,
DECLARE
INITIALIZE
TRACE
COMPONENT Polychromatic_Beam = Arm() AT (0,0,0) ABSOLUTE
COMPONENT Thermal_Source = Source_adapt(xmin=-0.1,xmax=0.1,ymin=-0.1,ymax=0.1, dist = Mono_position, xw = 0.095, yh = 0.300, E0= Source_E0, dE = Source_dE,
        AT (0,0,0) RELATIVE Polychromatic_Beam
COMPONENT Thermal_Neutrons  Convert_FlatE_2_Maxwell(T = SourceTemp, E0=Source_E0, dE=Source_dE)
        AT (0,0,0) RELATIVE Polychromatic_Beam
COMPONENT Filter = Filter_Graphite(xmin = -0.16, xmax = 0.16, ymin = -0.16, ymax = 0.16, len = Filter_d)
        AT (0, 0, 16.700) RELATIVE Polychromatic_Beam
COMPONENT Monochromator_Plane = Arm()
        AT (0, 0, Mono_position) RELATIVE Polychromatic_Beam ROTATED (0, Mono_omega, 0) RELATIVE Polychromatic_Beam
COMPONENT Blade0 = Monochromator0(xmin = Mono_xmin, xmax = Mono_xmax, zmin = Mono_zmin, zmax = Mono_zmax, ymin = Mono_ymin, ymax = Mono_ymax,mosh =
        AT (0, mpos0, 0) RELATIVE Monochromator_Plane  ROTATED (0, 0, mrotz0) RELATIVE Monochromator_Plane
COMPONENT Monochromatic_Beam = Arm()
        AT (0, 0, d_source_mono) RELATIVE Polychromatic_Beam ROTATED (0, Takeoff, 0) RELATIVE Polychromatic_Beam
COMPONENT MSlit = Slit(xmin = SML, xmax=SMR , ymin = SMB, ymax = SMT)
        AT (0,0,0.400) RELATIVE Monochromatic_Beam ROTATED (0,0,0) RELATIVE Monochromatic_Beam
COMPONENT SSlit = Slit(xmin = SSL, xmax = SSR, ymin = SSB, ymax = SST)
        AT (0,0,2.800) RELATIVE Monochromatic_Beam ROTATED (0,0,0) RELATIVE Monochromatic_Beam
COMPONENT pCan_in = Powder_prep_i (radius_o = Can_ro, radius_i=Can_ri, h = Can_h, pack = Can_pack, Vc=Can_Vc, sigma_a = Can_sigma_abs, q = Can_Q, j = Can_mul
        AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT pSample_in = Powder_prep_i (radius_o = Sample_ro, radius_i=Sample_ri, h = Sample_h, pack = Sample_pack, Vc=Sample_Vc, sigma_a = sigma_abs, q = Sample_
        AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT pCan_out = Powder_prep_o (radius_o = Can_ro, radius_i=Can_ri, h = Can_h, pack = Can_pack, Vc=Can_Vc, sigma_a = Can_sigma_abs, q = Can_Q, j = Can_mult, F2
        AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT Can_in = Powder_hollow (radius_o = Can_ro, radius_i=Can_ri, h = Can_h, pack = Can_pack, Vc=Can_Vc, sigma_a = Can_sigma_abs, q = Can_Q, j = Can_mult, F2 =
        AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT Sample_in = Powder_hollow (radius_o = Sample_ro, radius_i=Sample_ri, h = Sample_h, pack = Sample_pack, Vc=Sample_Vc, sigma_a = sigma_abs, q = Sample_Q, j
        AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT Can_out = Powder_hollow (radius_o = Can_ro, radius_i=nCan_ri, h = Can_h, pack = Can_pack, Vc=Can_Vc, sigma_a = Can_sigma_abs, q = Can_Q, j = Can_mult,
        AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT PSD_D20 = PSD_curved(radius = PSD_r, height = PSD_h, nd = PSD_cells, pitch=PSD_pitch,gap=PSD_gap,filename=psd_filename, sign = Diffractometer_sign, tt0
        AT ( 0, 0,  Sample_z) RELATIVE Monochromatic_Beam
COMPONENT scattered = Adapt_check(source_comp=Thermal_Source)
        AT ( 0, 0,  End_Position) RELATIVE Monochromatic_Beam
FINALLY
END
```

McStas Components

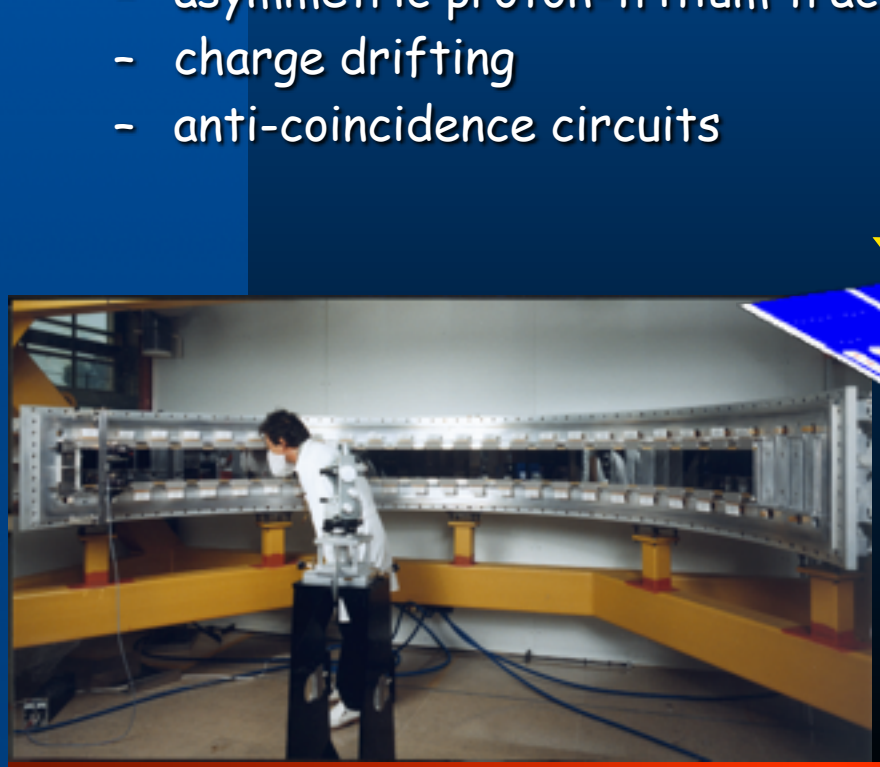


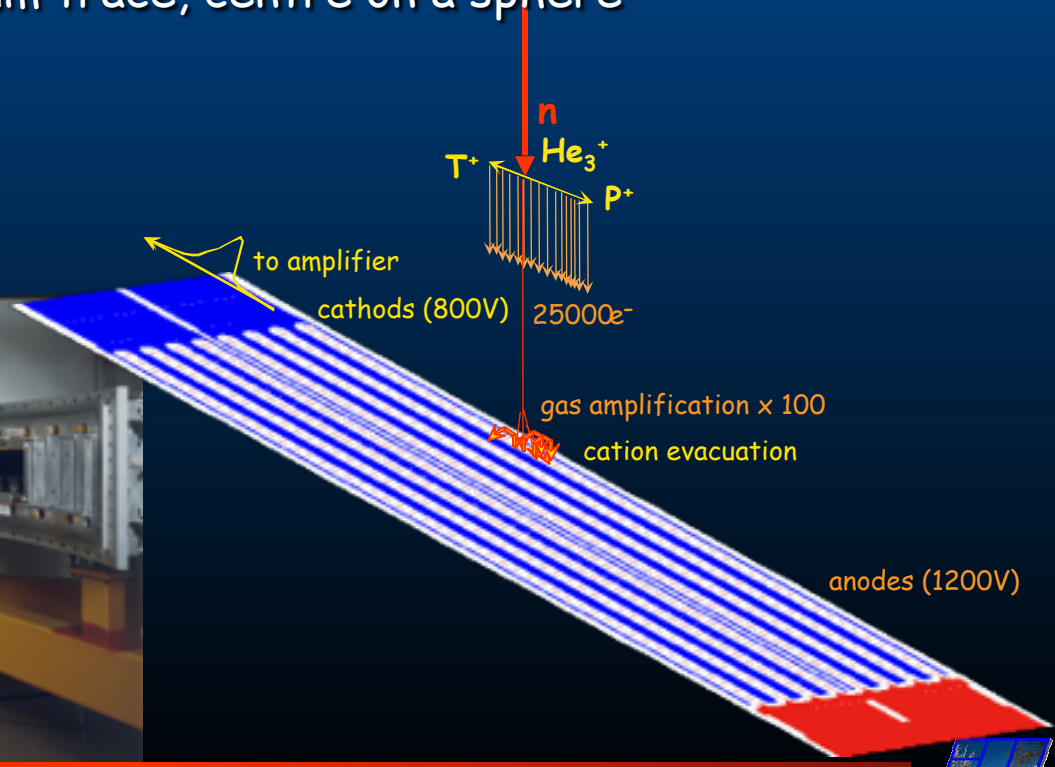
- **Source**
  - Converter from flat to Maxwell distribution (thermal neutrons)
- **Monochromator**
  - finite thickness (not just flat)
  - multiple scattering -> reflectivity from crystallography
  - optional transmission geometry (small take-off)
- **Powder sample**
  - several Bragg peaks
  - incoherent scattering
  - scattering from container
  - multiple scattering



McStas Components: PSD

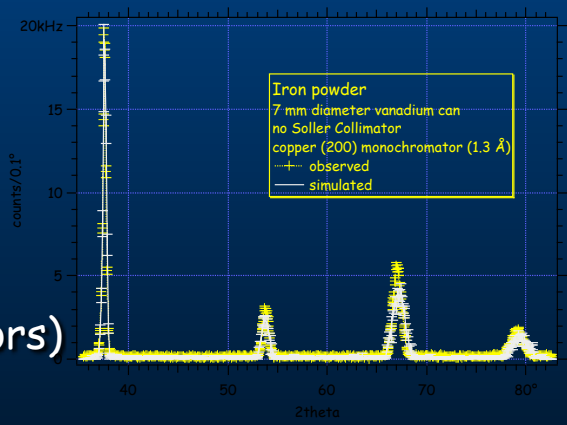
- **Curved micro-strip gas chamber position sensitive detector**
  - Curvature
  - Penetration depth in detection gap and efficiency
  - asymmetric proton-tritium trace, centre on a sphere
  - charge drifting
  - anti-coincidence circuits





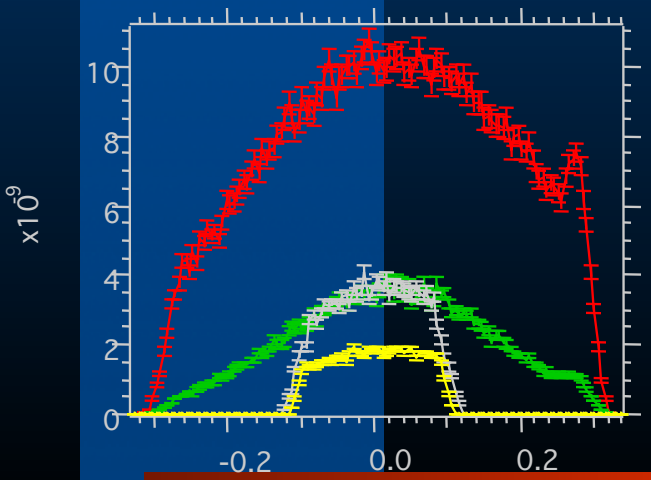
Validation: quantitative simulation

- **Idea**
  - first: successful **quantitative** simulation of real data
  - then: extrapolation to new configurations or instruments
- **Several parameters not well known**
  - incident neutron spectrum and flux
  - misalignment of optical components
    - Collimators, monochromator, sample, PSD
  - monochromator mosaic (horizontal/vertical)
    - not always precisely known (older monochromators)
    - distribution over blade/crystal surface
  - guessing by simulation of different known configurations

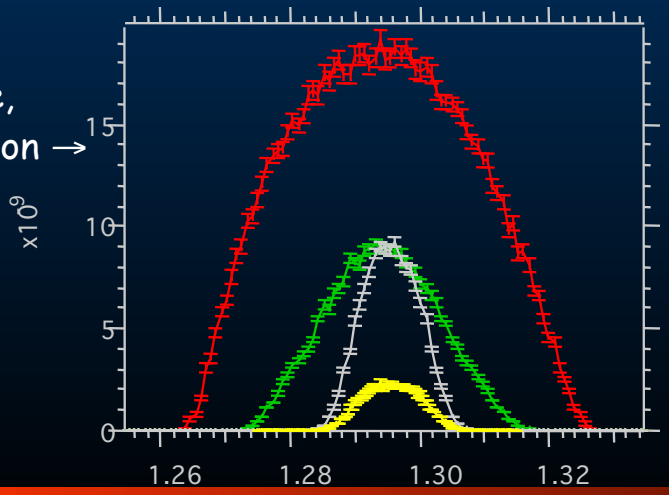


Understanding the neutron beam

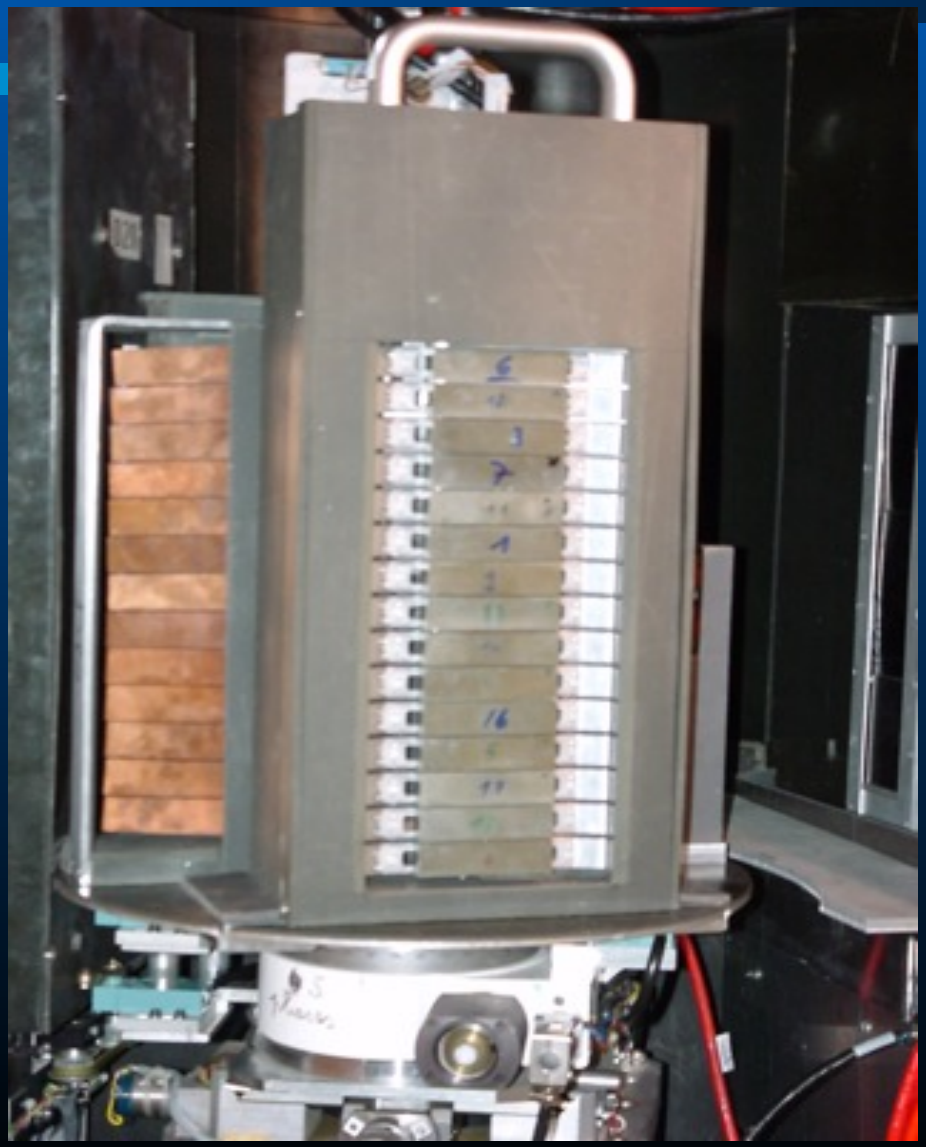
- **Insight into inaccessible parameters**
  - 2D-Intensity profile at sample position
    - Optimisation of shielding and slit setting for sample environment
  - Divergence and wavelength distribution
  - 3D-Mapping of scattering position in sample



← horizontal divergence,
wavelength distribution →
at sample position, for
- full beam
- 10' Soller collimator ...
- ... & secondary slits ...
- ... & smaller sample

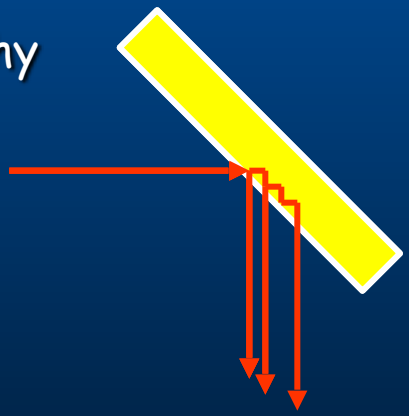


Building a new monochromator



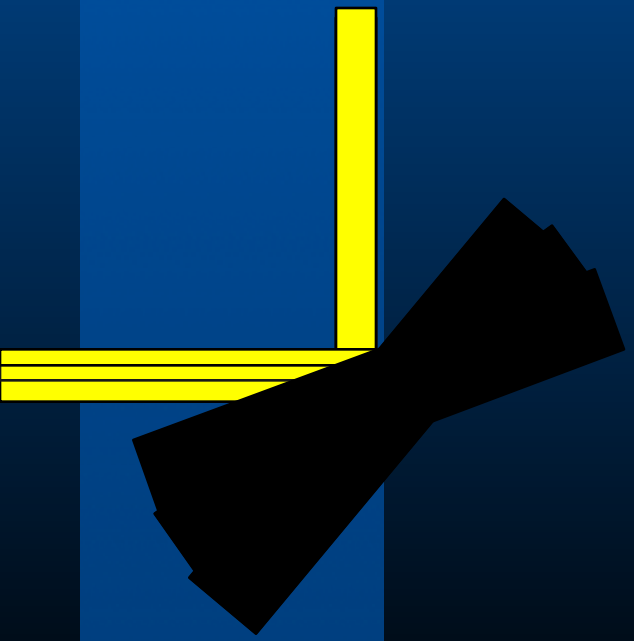
McStas Components

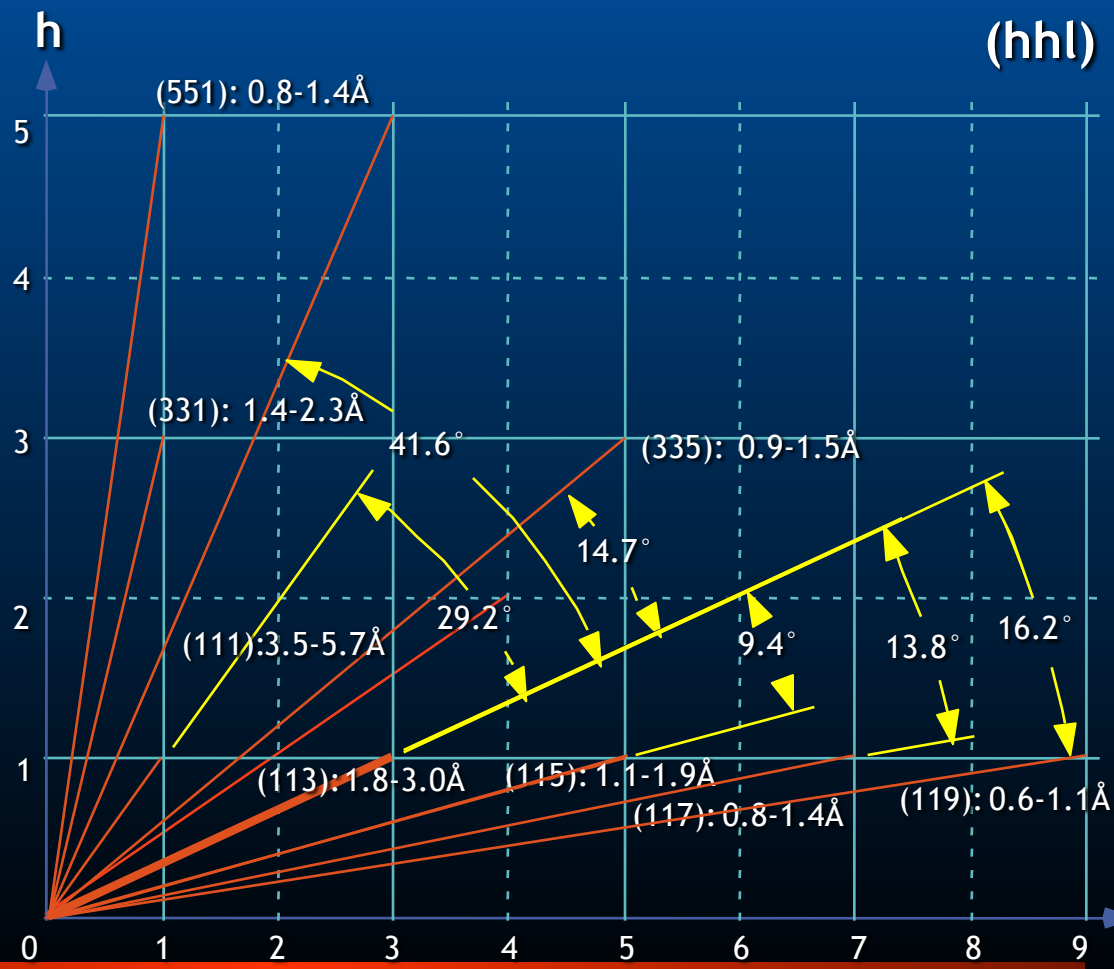
- **Monochromator**
  - finite thickness (not just flat)
  - multiple scattering -> reflectivity from crystallography
  - optional transmission geometry (small take-off)



Accessible wavelengths

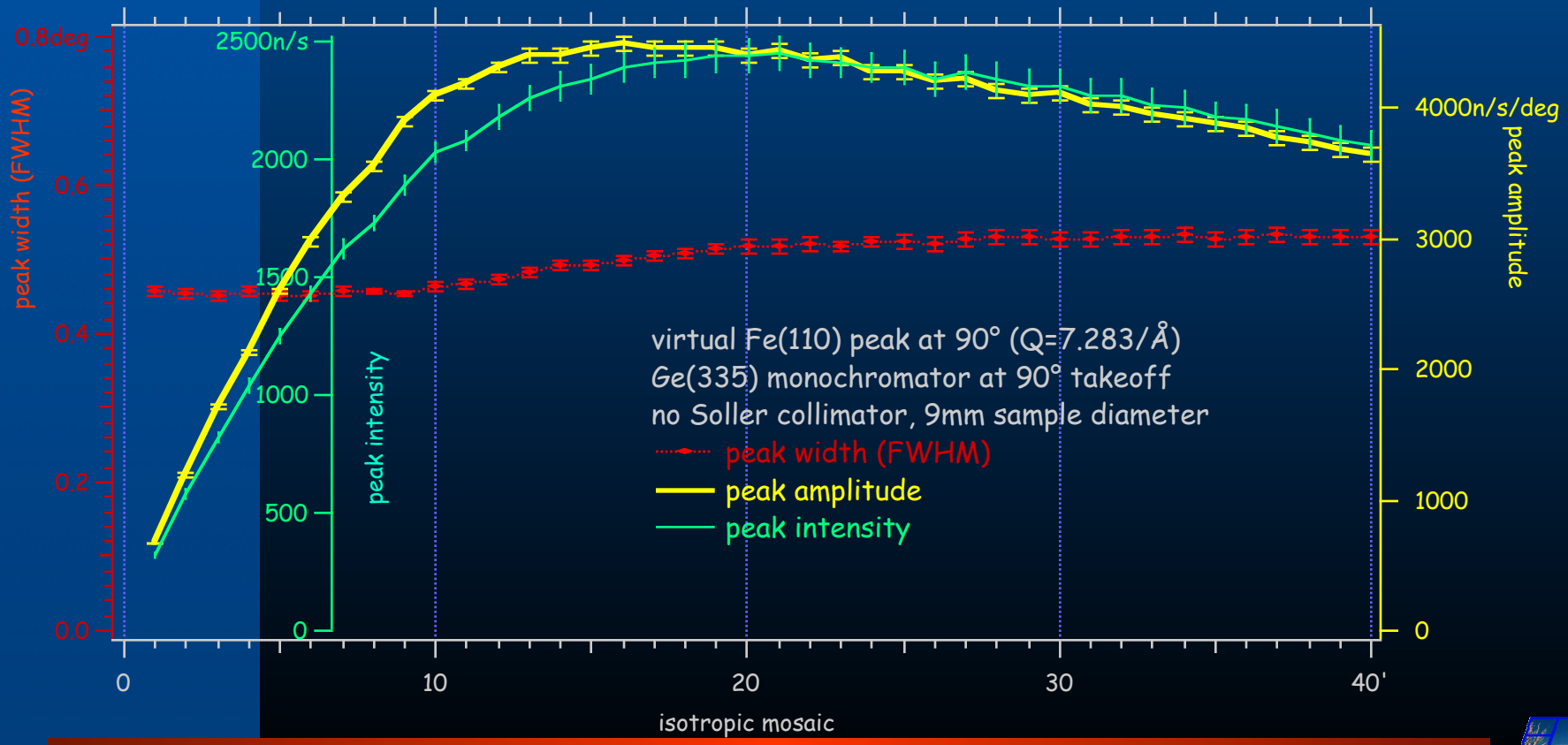
- Accessible reflections to provide a choice of wavelengths
  - good flux (Fankuchen), (nearly) no contamination λ/3:
    - 1.1-1.9Å @ 122°
    - 0.8-1.8Å @ 63°





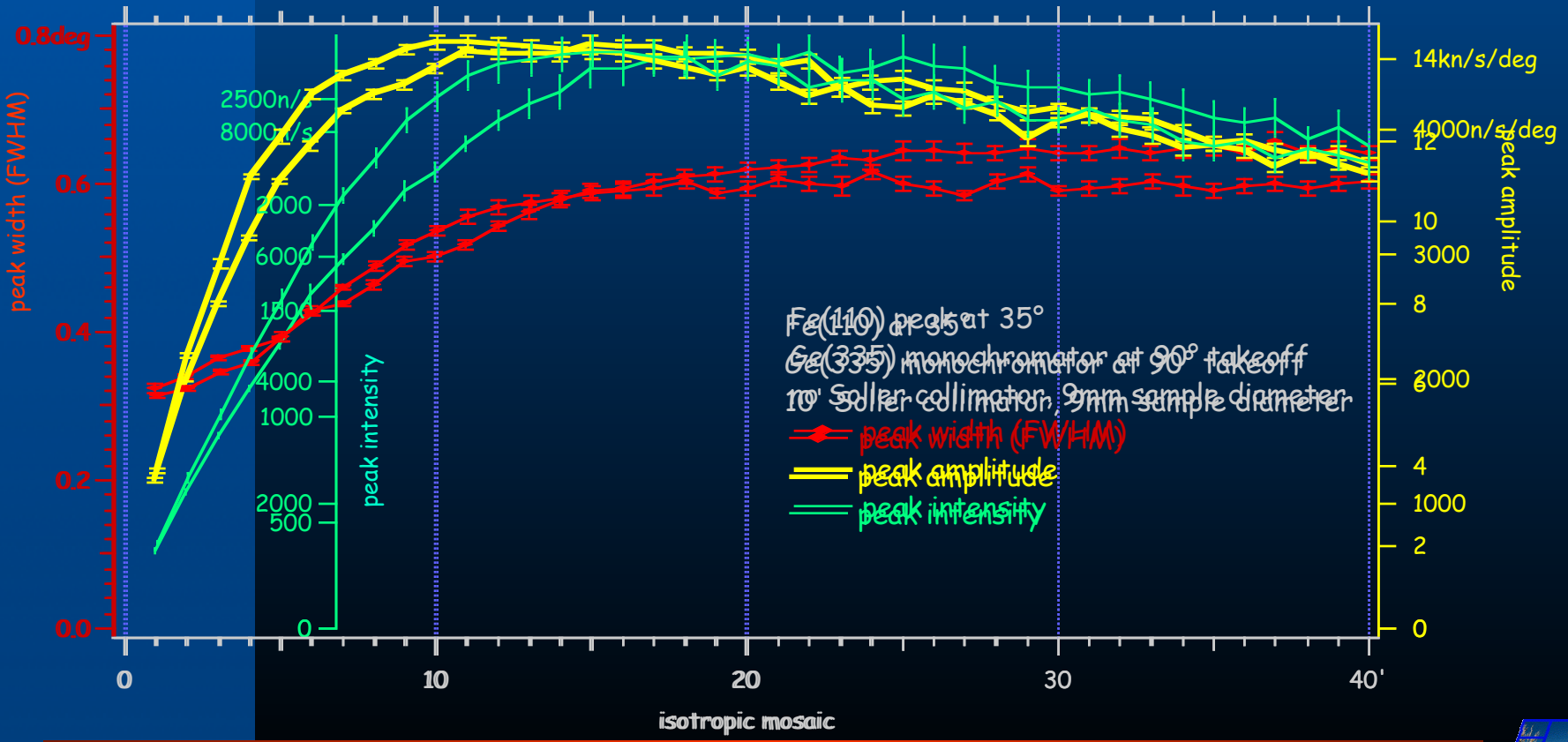
Monochromator mosaic - Cagliotti

- **parallel incident beam, diffraction angle = takeoff angle**
  - 20′/20′ fine for a new Germanium monochromator



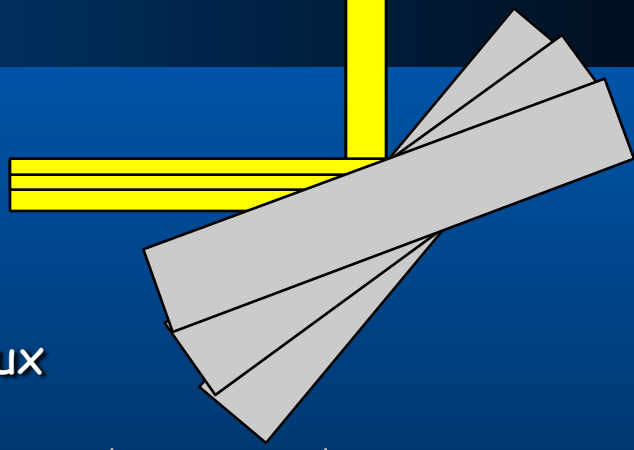
Monochromator mosaic - simulation

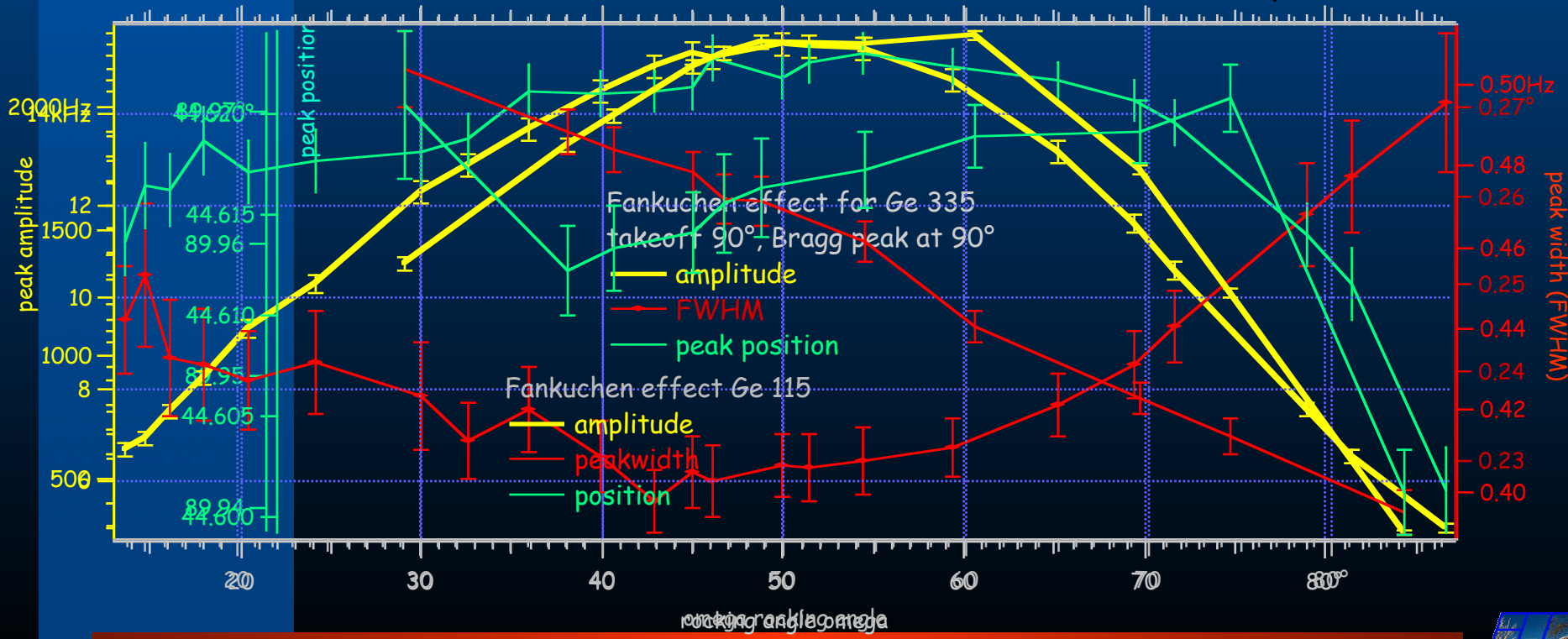
- ... leaving ideal conditions:
  - 10'/10' reasonable, if amplitude regarded



Fankuchen effect

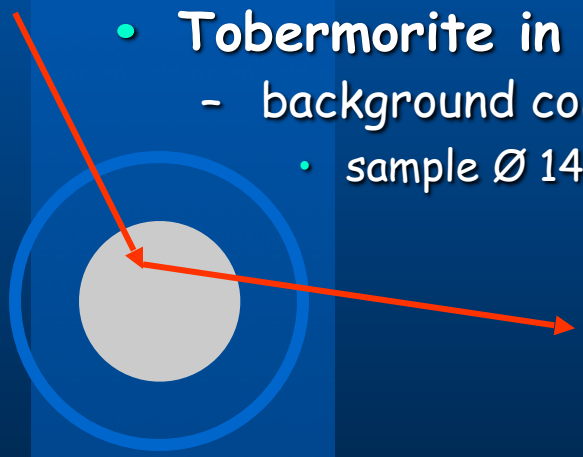
- **Fankuchen effect**
  - monochromator reflections ≠ cut plane
  - Quantitative evaluation of resolution and flux

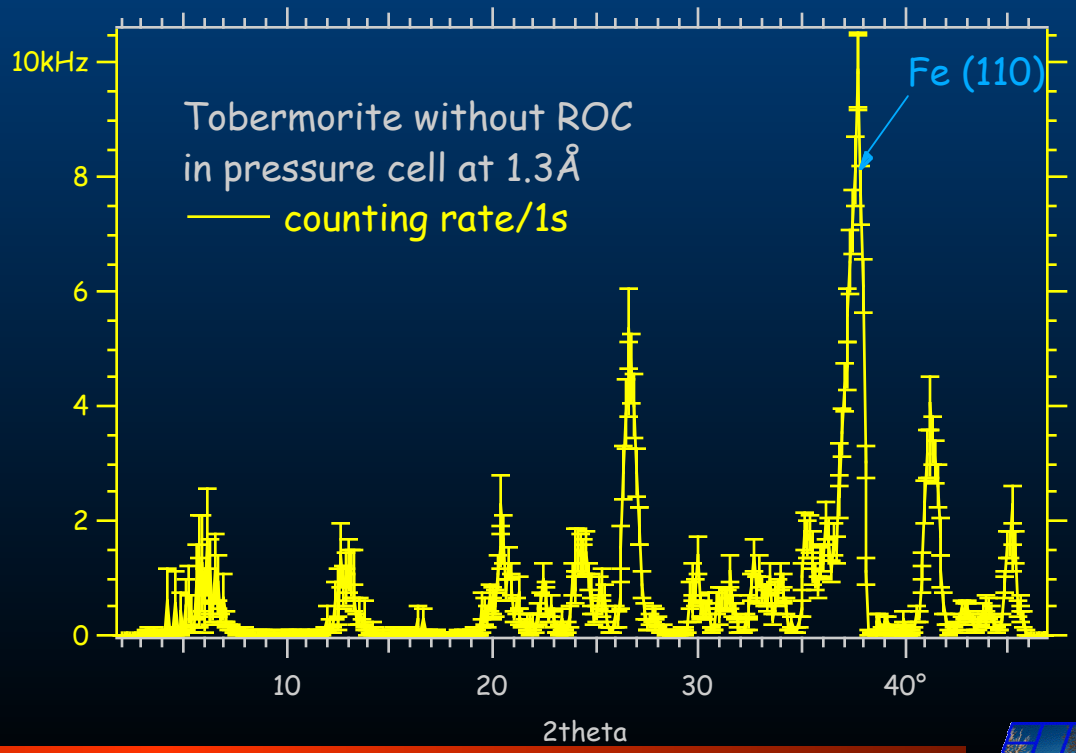




Hydrothermal reaction cell

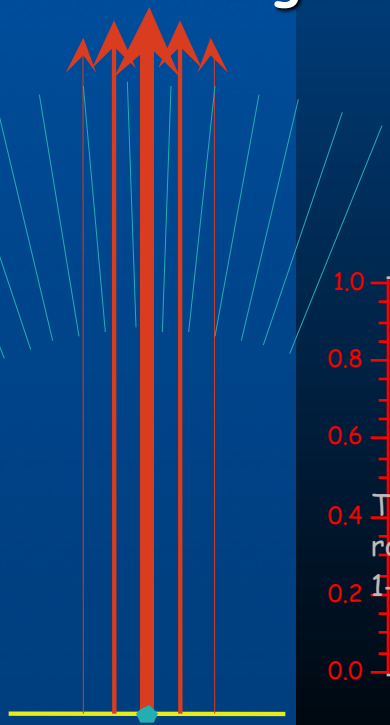
- **Tobermorite in steel reaction cell**
  - background contribution of reaction cell
    - sample Ø 14mm, cell Ø 28mm, 1mm thick

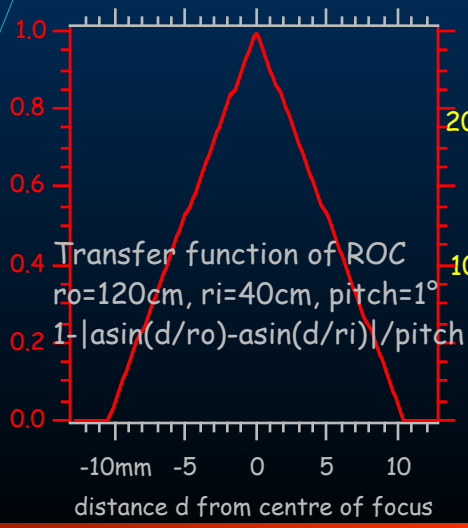


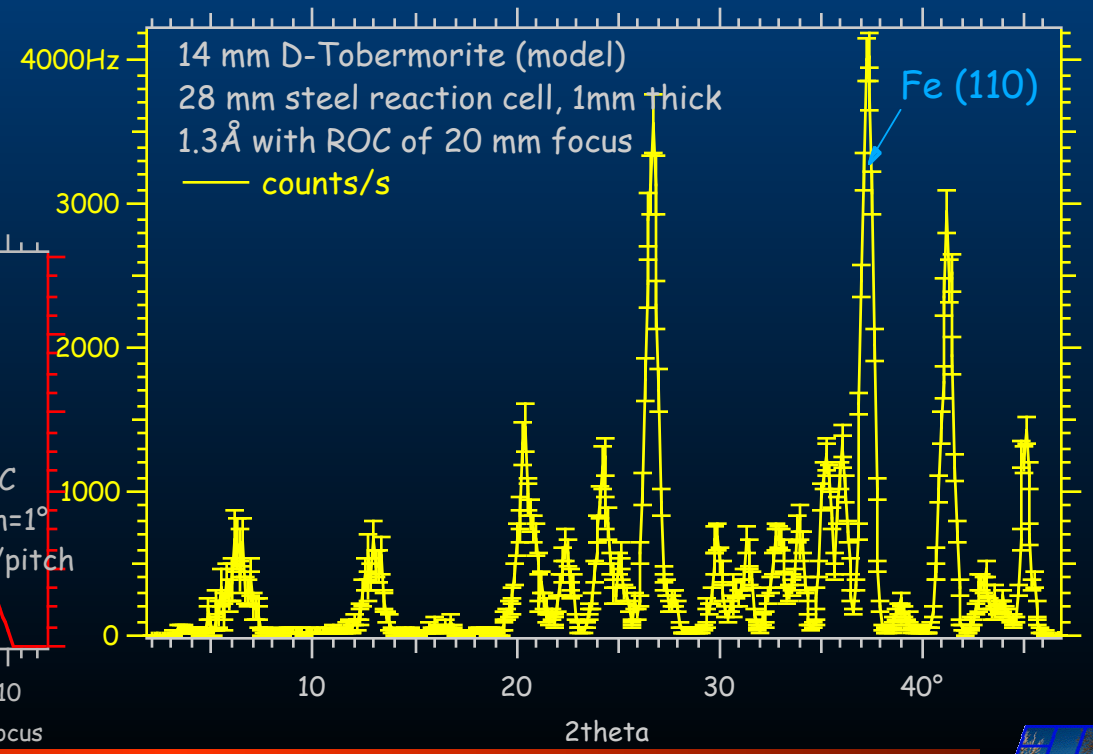


Radial Oscillating Collimator ROC

- No/less background contribution from sample environment
- Gain in resolution
- Triangular transfer function

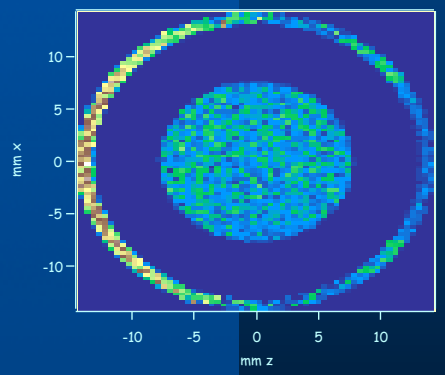


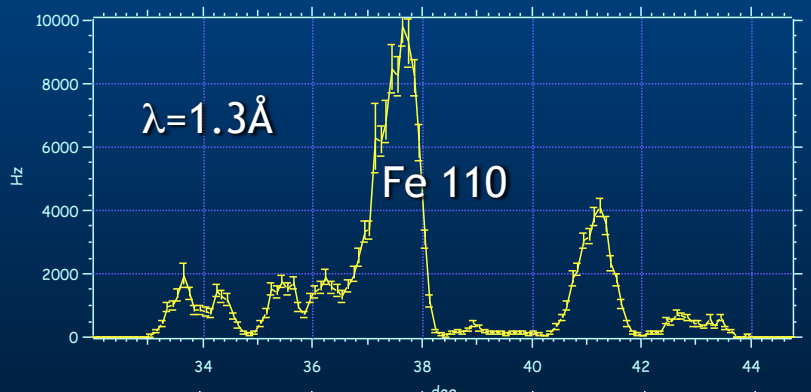


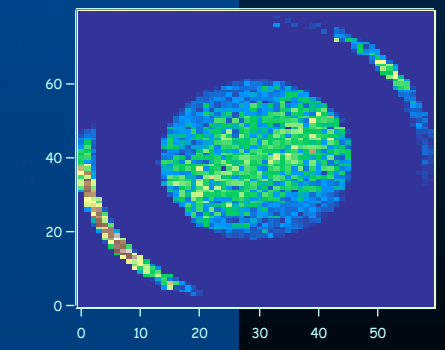


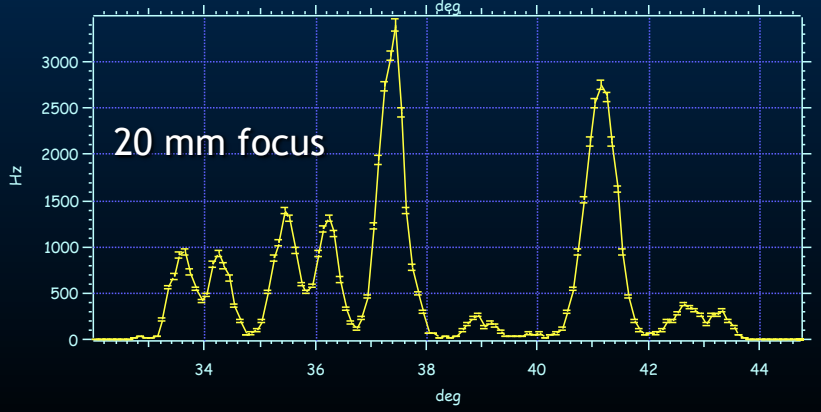
ROC and hydrothermal reactors

- Hydrothermal hardening of concrete
  - Fast formation of tobermorite (main phase)
  - Quantitative phase analysis (QPA) in situ
  - ROC would here reduce background





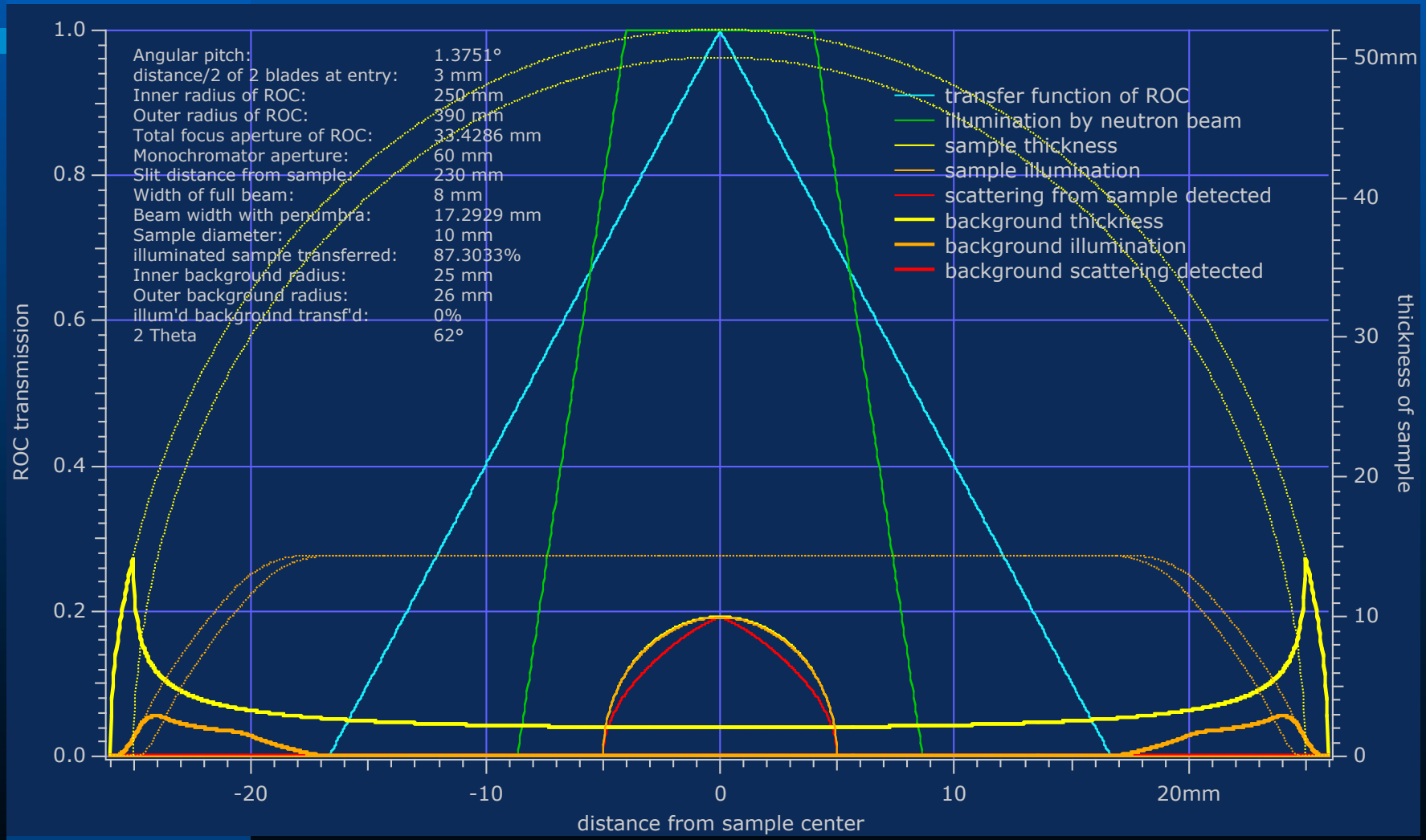




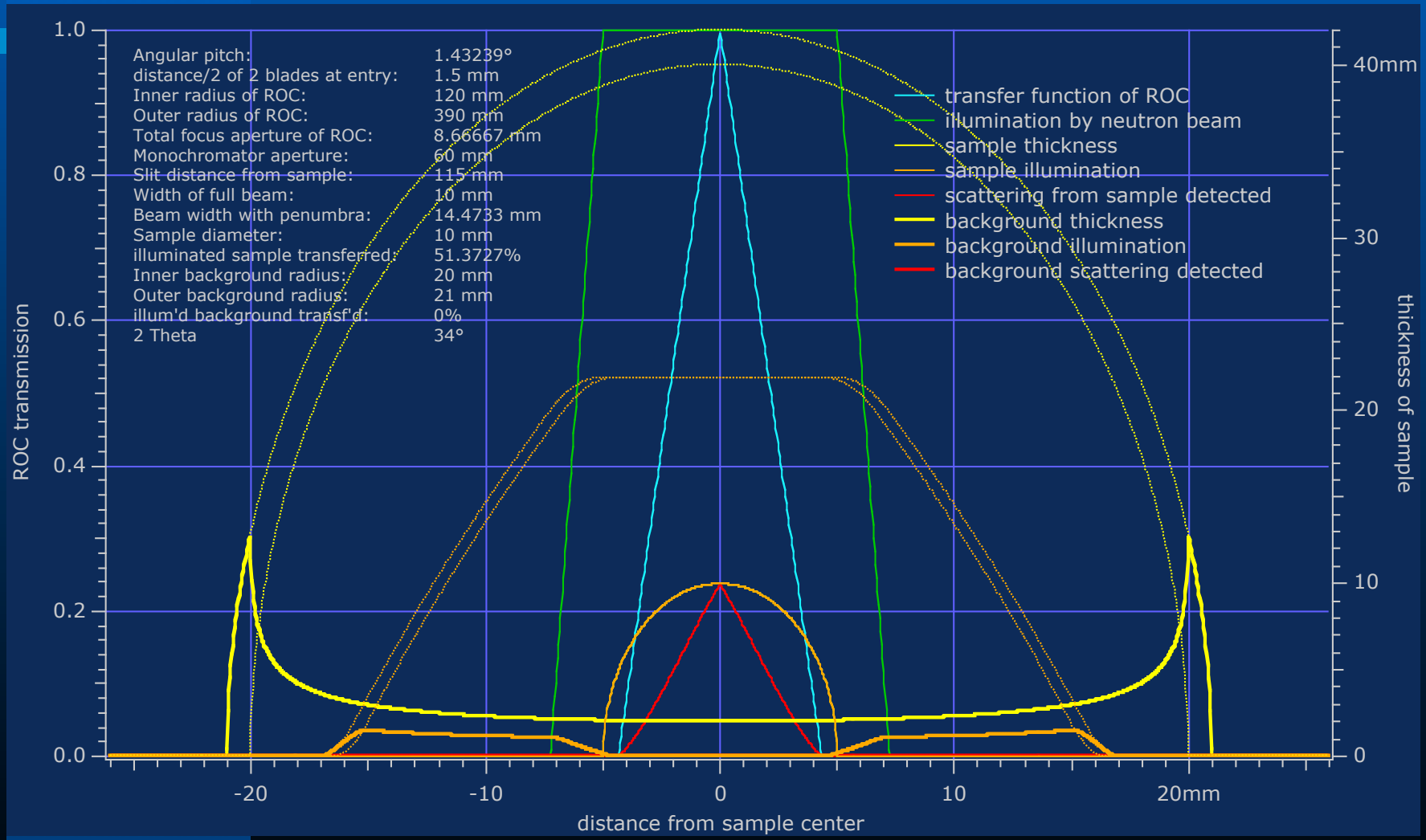
Conception of a ROC for D20

- Geometrical constraints
  - Sample environments
    - Cryomagnets, cryostats, furnaces
    - Pressure cells, autoclaves
- Shielding needs
  - Maximum sample diameter
  - Inner diameter of background contributor
  - Wavelengths and diffraction angles
- Two ROCs
  - Large calorimeter, large sample, large wavelength, large vessel
  - Small heating element, smaller wavelength, smaller vessel
    - Non-complete background suppression in pressure cells as well

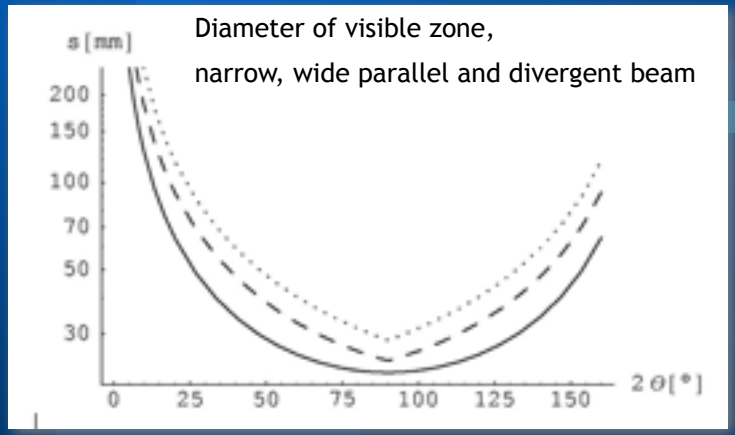
Conception of ROCs for D20



Conception of ROCs for D20

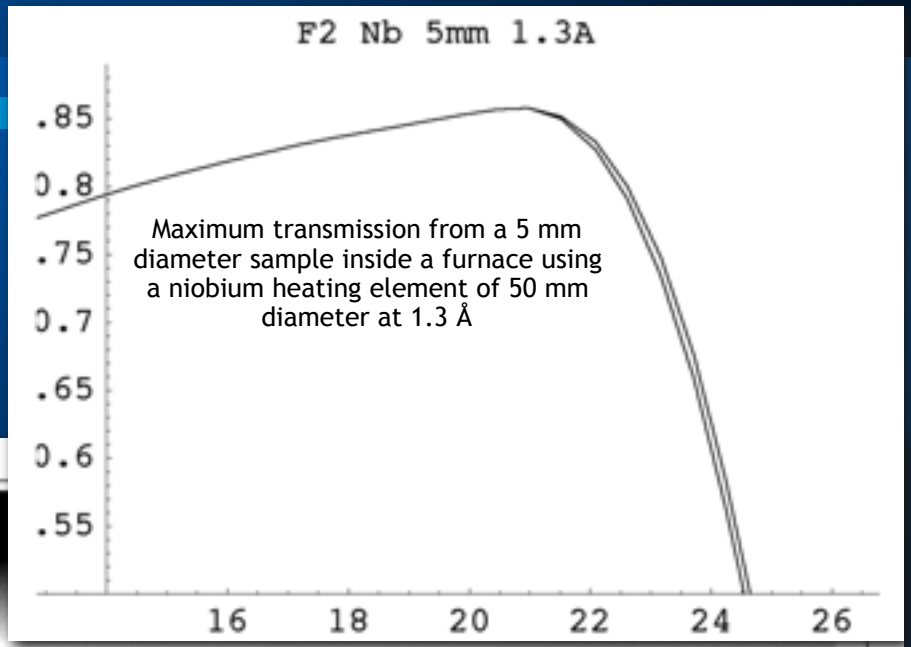


Visible sample environment

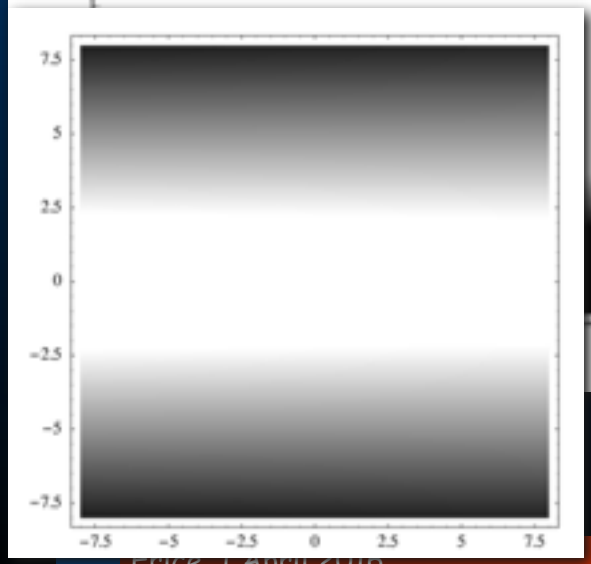


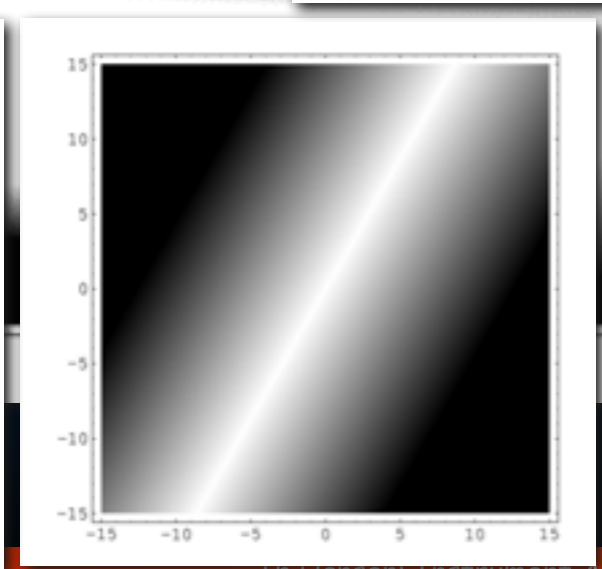
Diameter of visible zone,

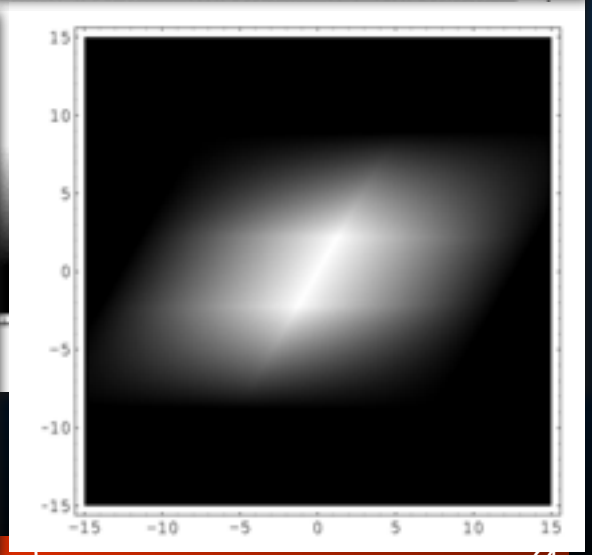
narrow, wide parallel and divergent beam



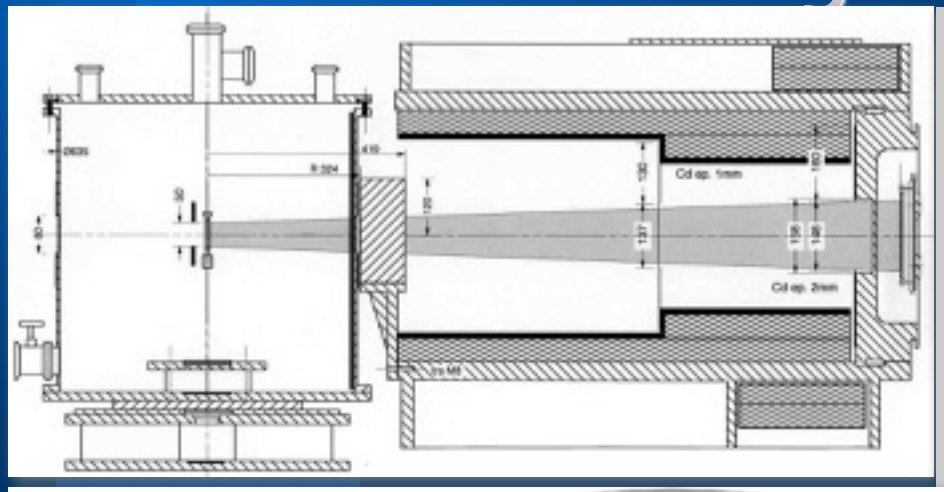
Maximum transmission from a 5 mm diameter sample inside a furnace using a niobium heating element of 50 mm diameter at 1.3 Å

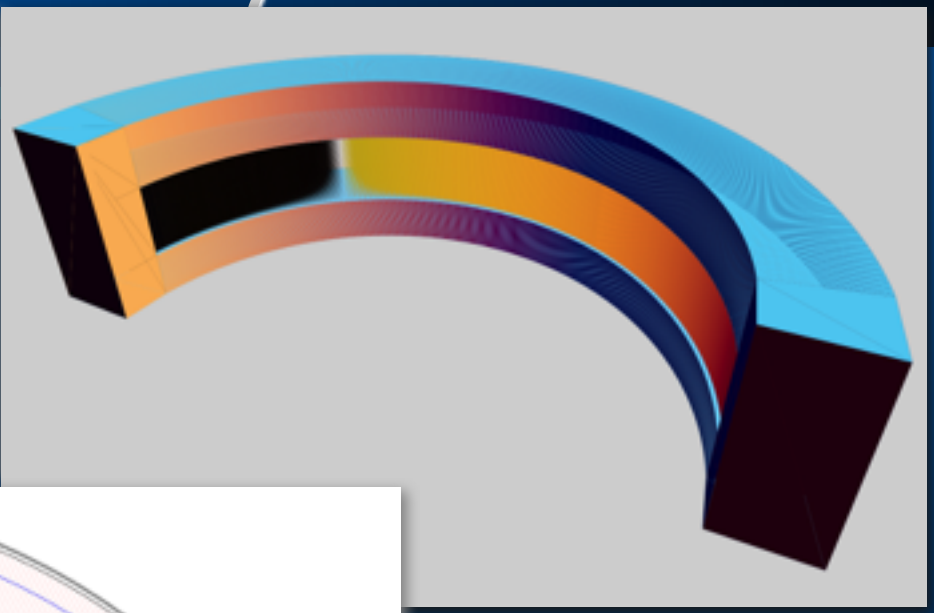


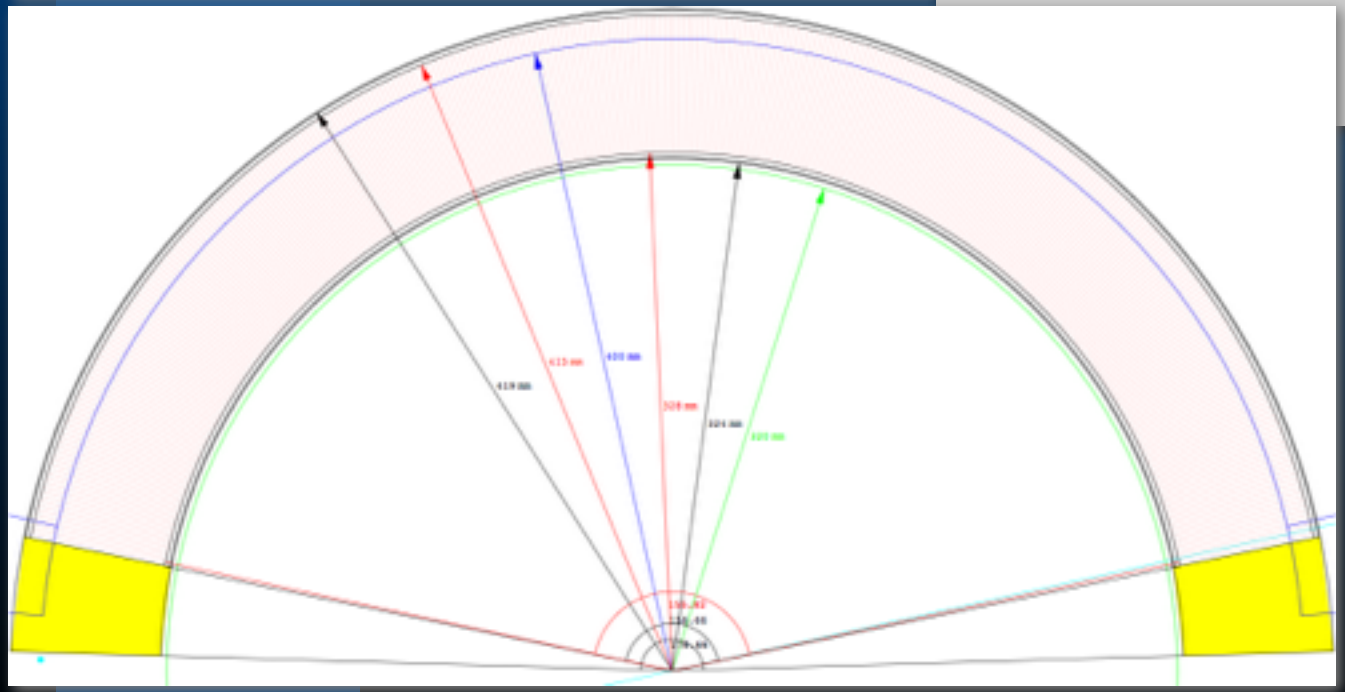




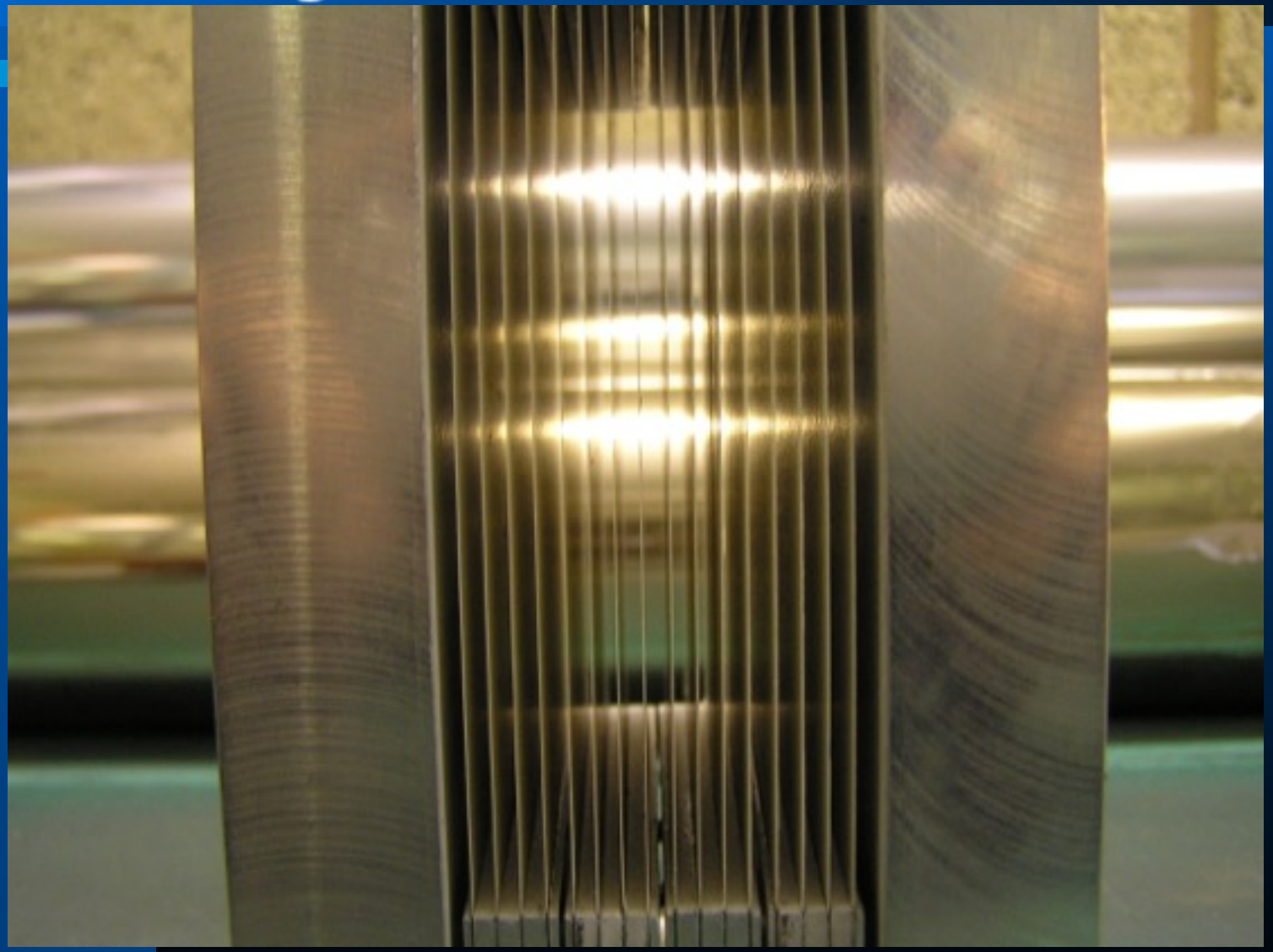
The D20 ROC: geometry



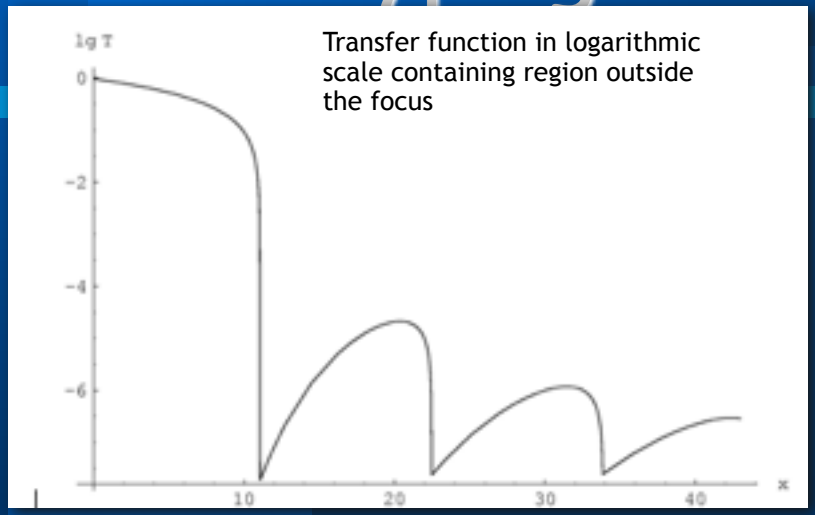




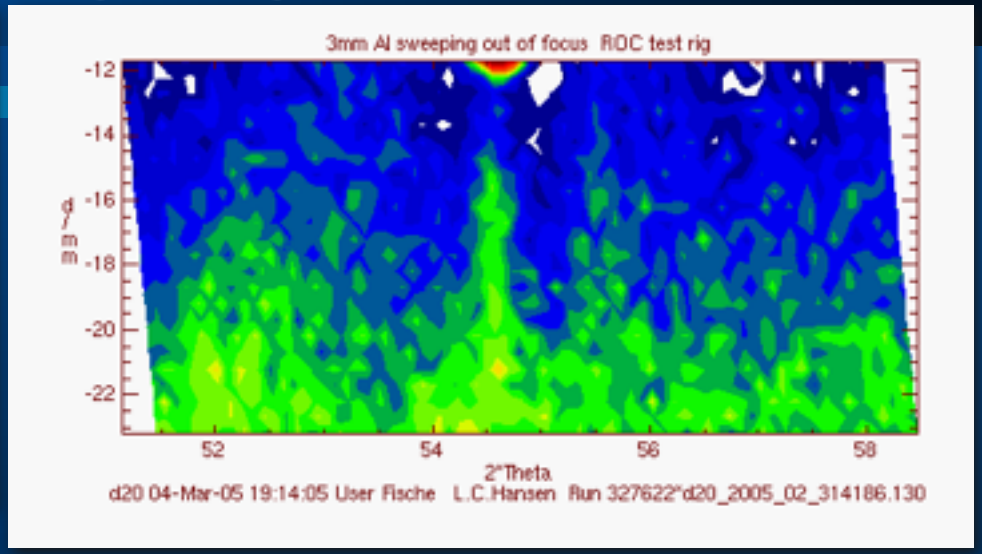
A test rig

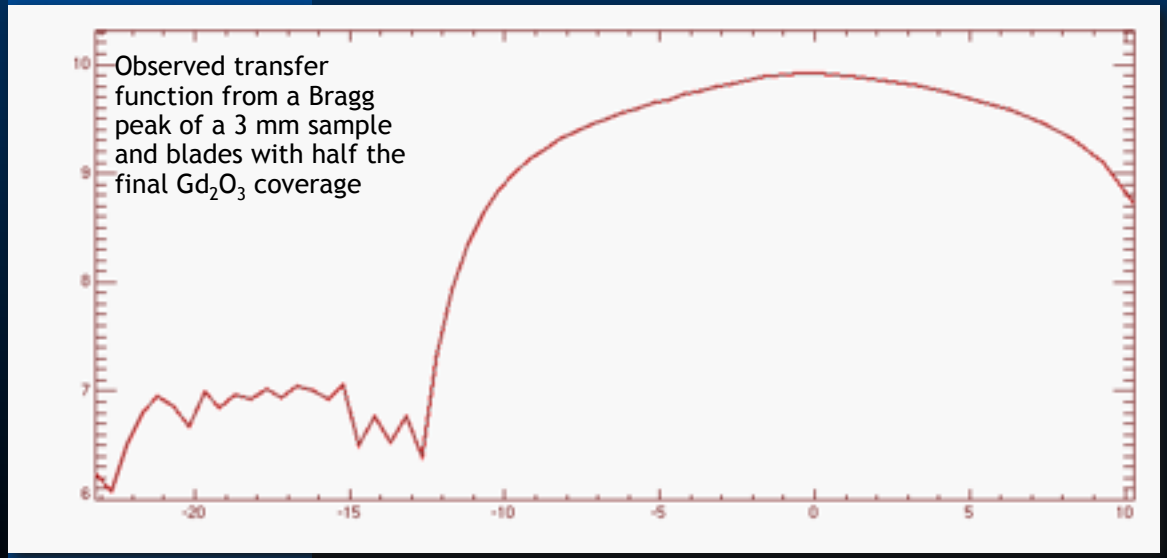


Prototyping: transfer function



Transfer function in logarithmic scale containing region outside the focus

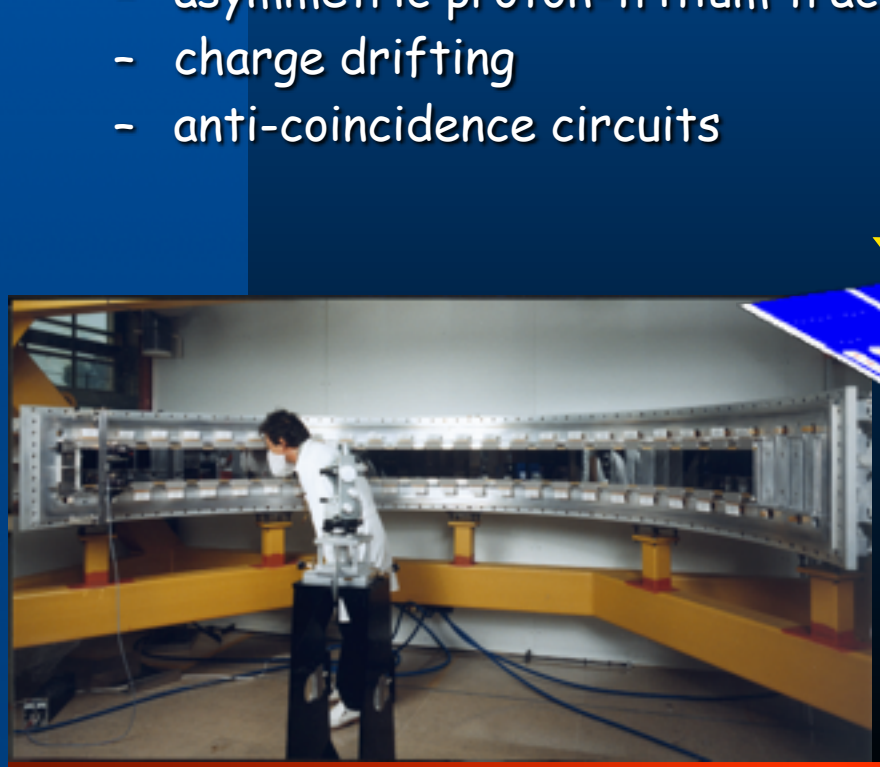


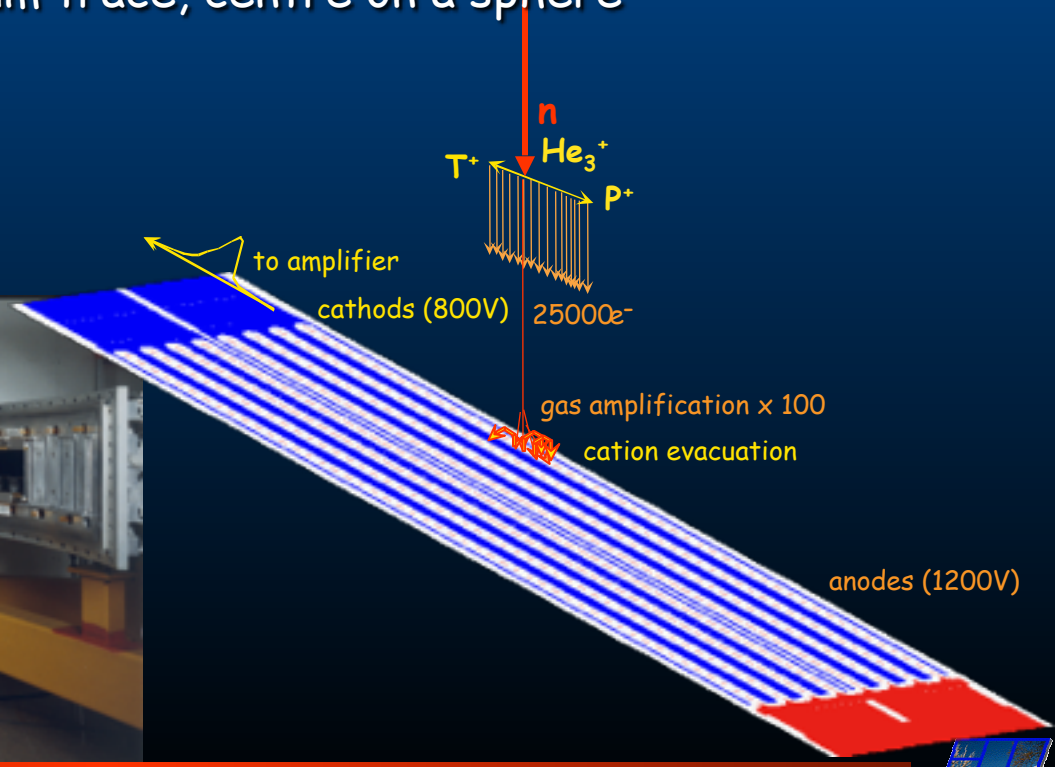


Observed transfer function from a Bragg peak of a 3 mm sample and blades with half the final Gd_2O_3 coverage

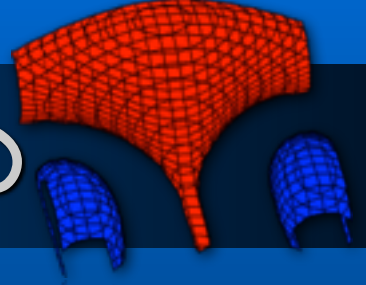
McStas Components: PSD

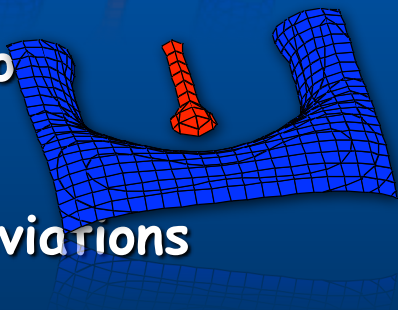
- **Curved micro-strip gas chamber position sensitive detector**
  - Curvature
  - Penetration depth in detection gap and efficiency
  - asymmetric proton-tritium trace, centre on a sphere
  - charge drifting
  - anti-coincidence circuits



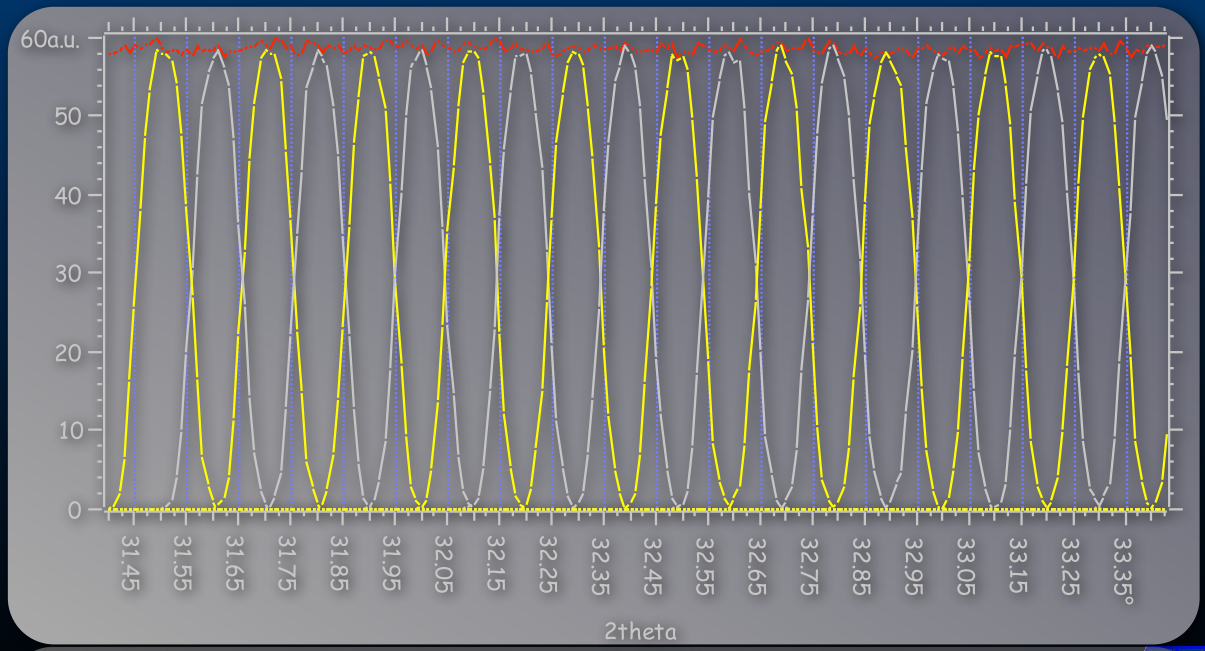


Micro-strip gas chamber PSD

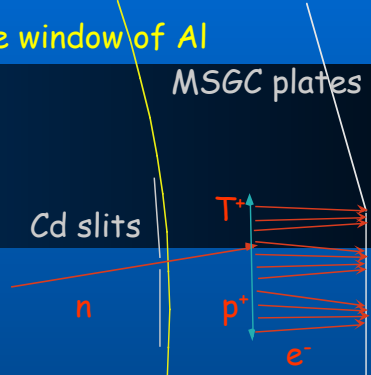




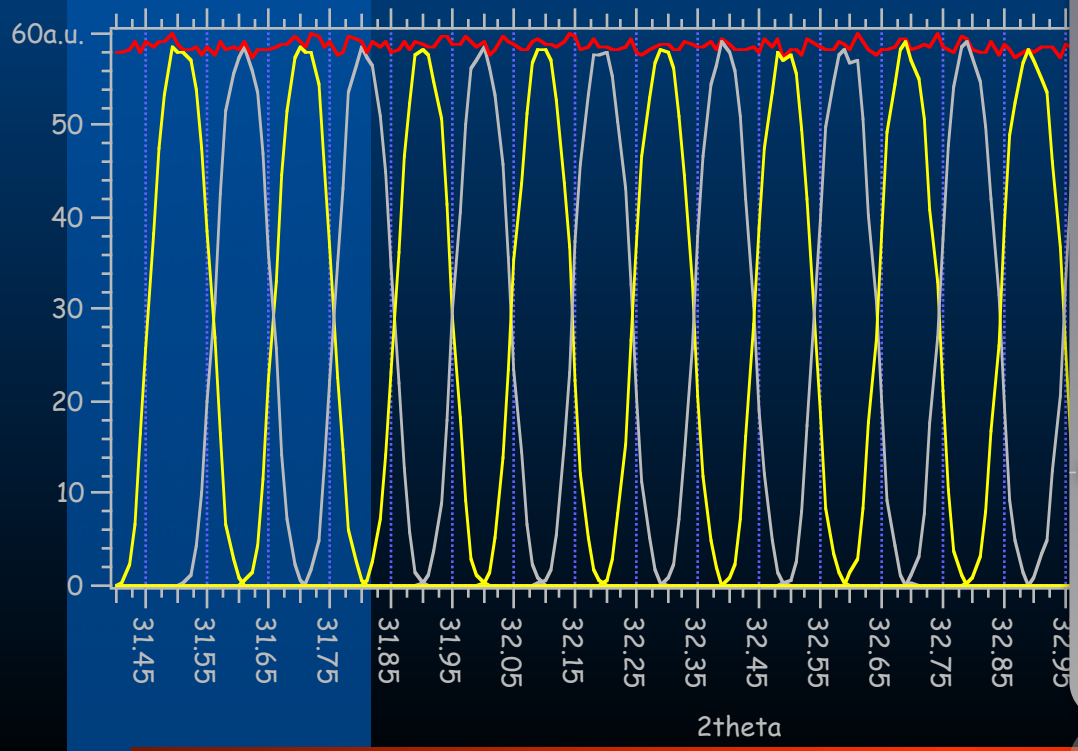
- Computing of electrical field in three dimensio
  - Validation by reproduction of detector scan
  - Polygonality of micro-strip plates: position deviations
- Angle and efficiency calibration

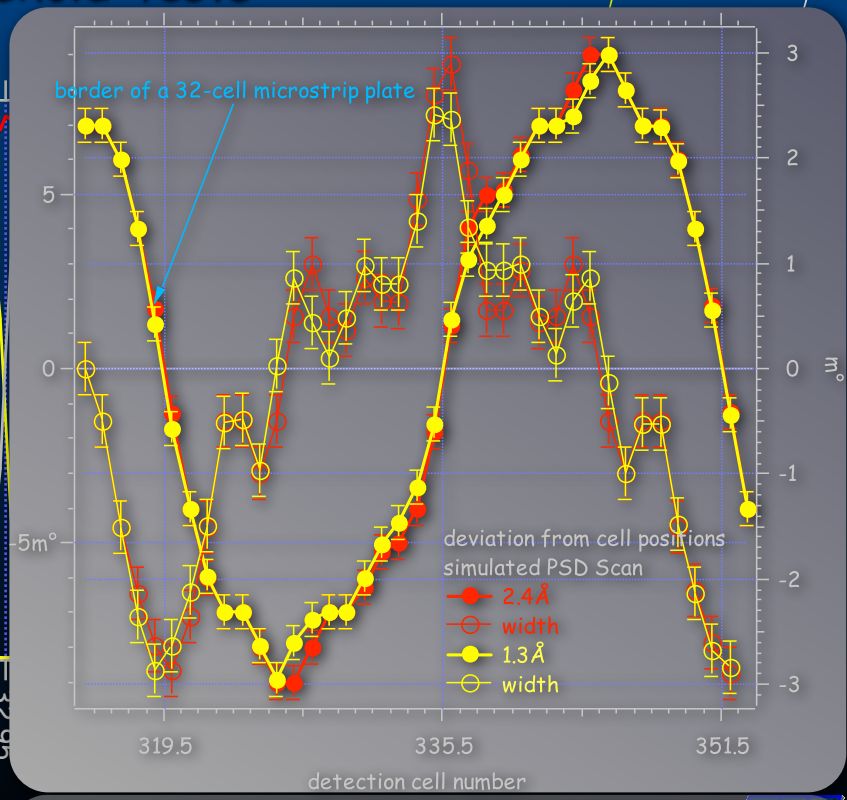


PSD response



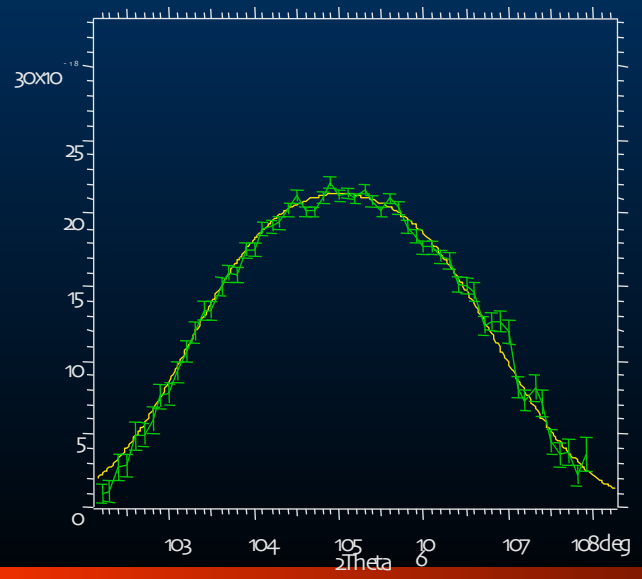
- **PSD response to scan with narrow beam**
  - response curve of each cell, periodic deviations (polygonality)
  - anti-coincidence circuits and threshold 'tests'





Understanding peak-shapes

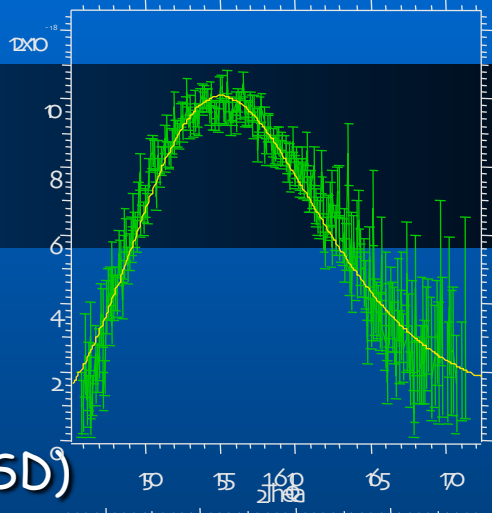
- **"Strange" peak-shapes**
  - Especially under highest flux conditions (no Soller collimator)
    - "Difficult" shapes at high angles
  - Simulations show same observed profile
    - -> intrinsic to the method
  - Convolution of Gauss and rectangle describes quite well

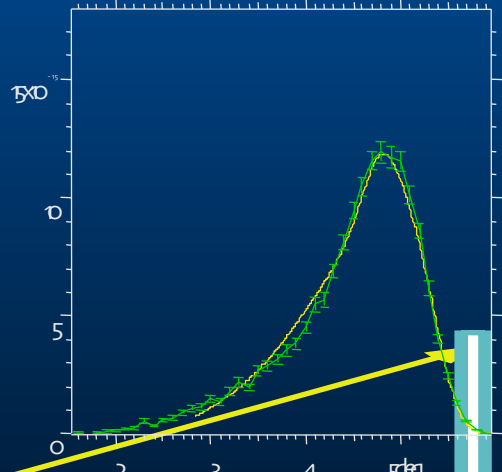


The better peak-shapes are described,
the better the effective resolution

Understanding peak-shapes

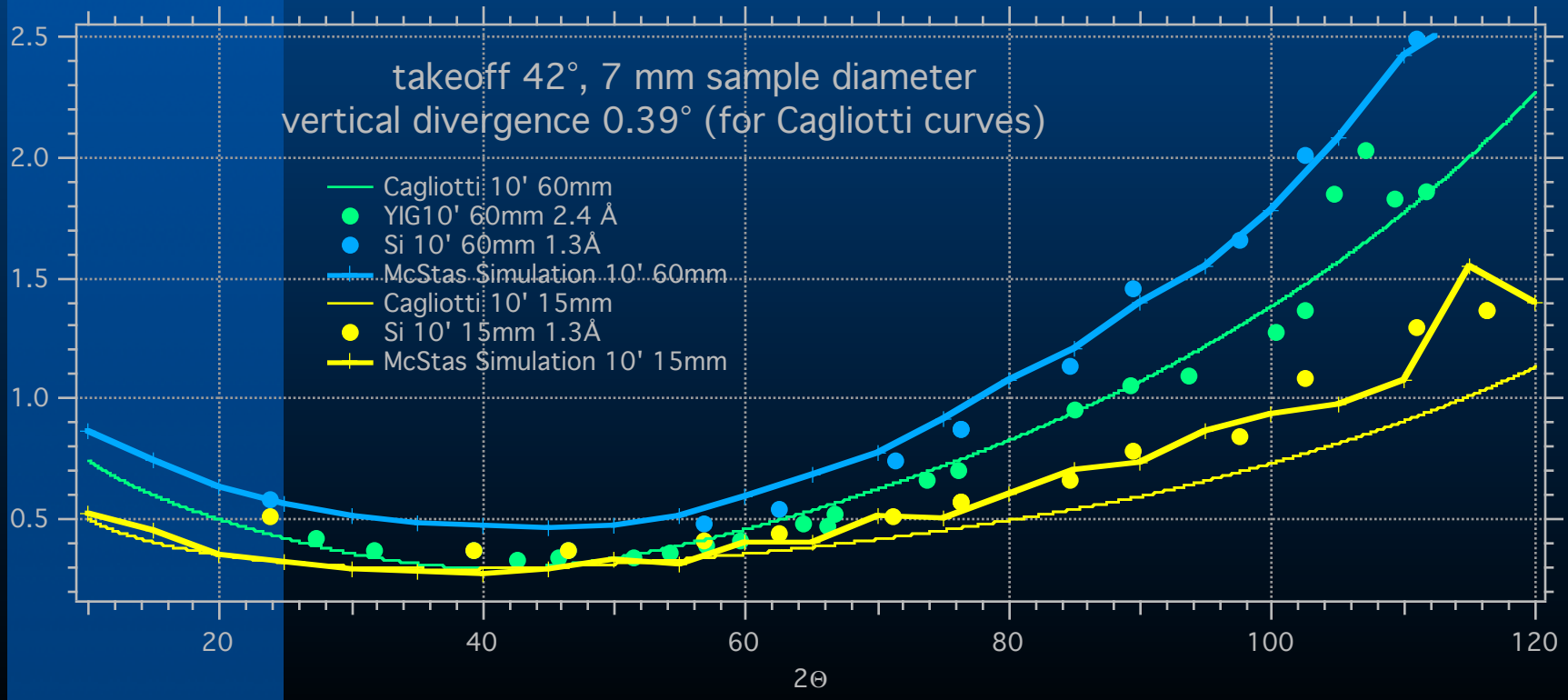
- **Asymmetry**
  - "Umbrella" effect (Debye-Scherrer cones -> 1D PSD)
  - High angles (Bragg law)
  - Vertical focussing (inhomogeneous illumination)





Instrumental Resolution function

- **Reproducing non-Cagliotti resolution curves**
  - especially copper monochromator in transmission
- **Prediction of future germanium monochromator resolution**



Results as presented by McPlot