

Data Collection Strategy

8th X-ray Scattering School

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ENERGY

Office of Science



SLAC NATIONAL
ACCELERATOR
LABORATORY

- Before
- Geometry, detectors, resolution...
- Data collection strategy.



Science



What is the scientific question?

How will probing the structure help?

- Is there an alternative method?
 - TEM
 - EXAFS
 - Neutron diffraction
 - BET porosity measurements
- Can data from a sealed tube diffractometer give at least part of the answer?

If answer to either of the question above is even a hesitant yes.

Do those measurements first

- They will help you do a better and easier synchrotron experiment.
- They will complement your synchrotron data and will simplify data analysis and interpretation.

- Structure completely unknown
 - Single Crystal
 - Polycrystalline → Rietveld method
 - Disordered → PDF
- Partially known
 - Site occupancy → full pattern refinement, AXS/REXS
- Interested in noncrystallographic features
 - Particle size → small angle scattering, peak shape analysis
 - Texture → 2D diffraction
 - Strain → 2D diffraction, Q space resolution



Science: Structural Transition

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- Impulse driven
 - Phase transition
 - Strain
- Time Dependent
 - Chemical Reaction
 - Relaxation processes
- In-situ rigs and reactors
 - Laser pump-probe
- Will the scattering from the reactor windows interfere?

Sample

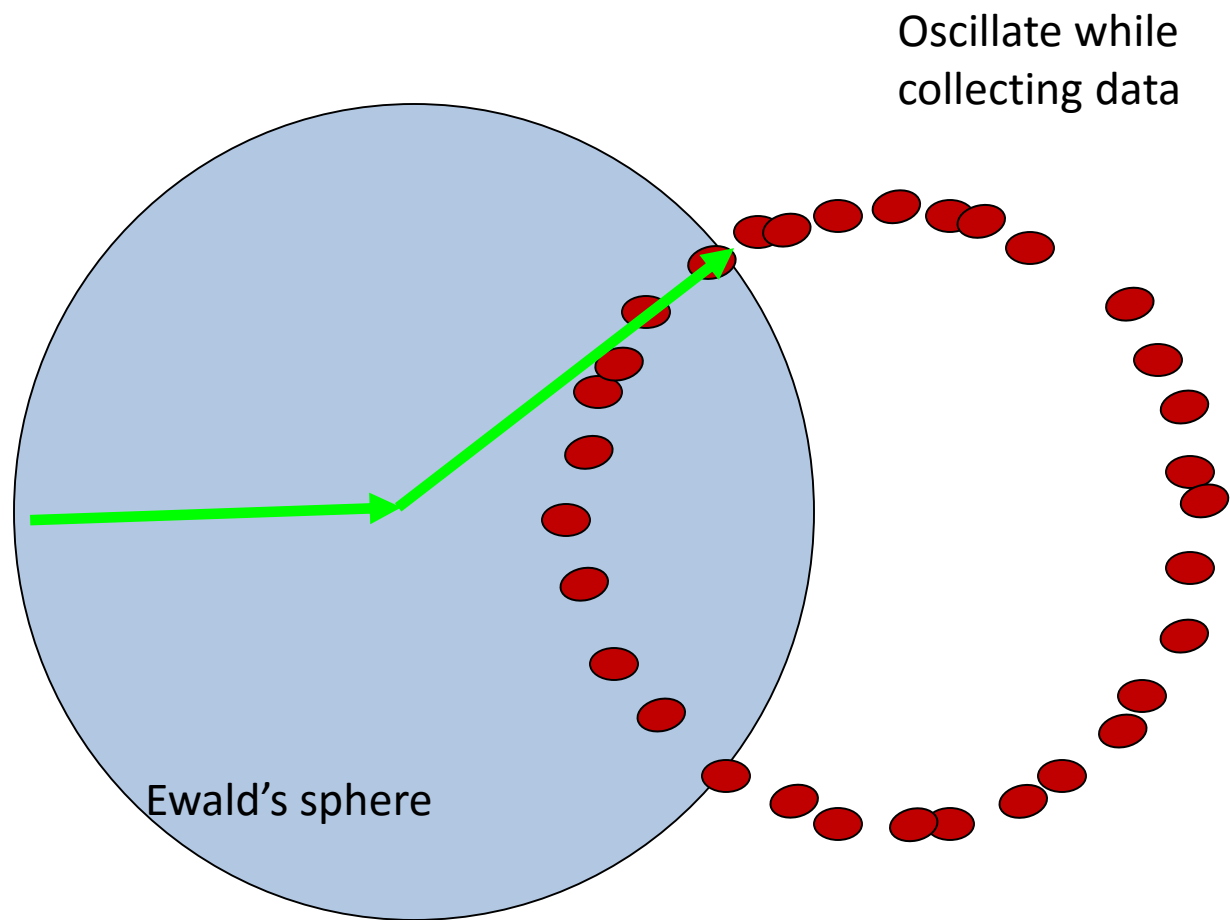


What does the sample look like?



- Bulk
 - powder or solid/pressed pellet
 - Would preferred orientation matter?
 - finely grind AND spin if possible
- Transmission or Reflection:
 - Refl → blocks bottom half of the pattern
 - Refl sample should be infinitely thick if possible
 - Trans samples are ideally $\sim \mu$
- Wet?
- Does it need containment window?

Powder Average and Rocking

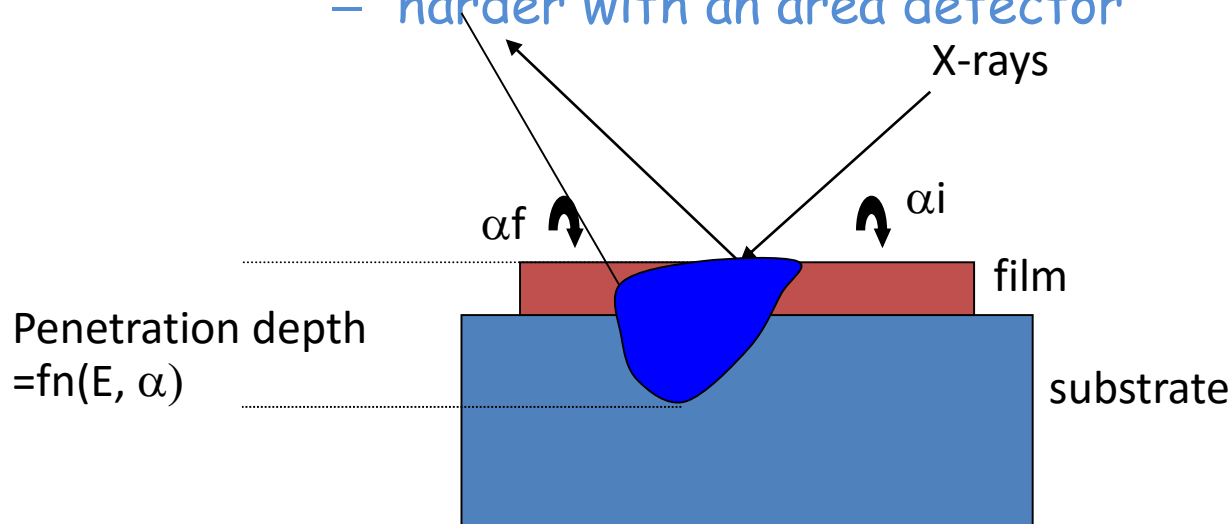


What Does the Sample look like?

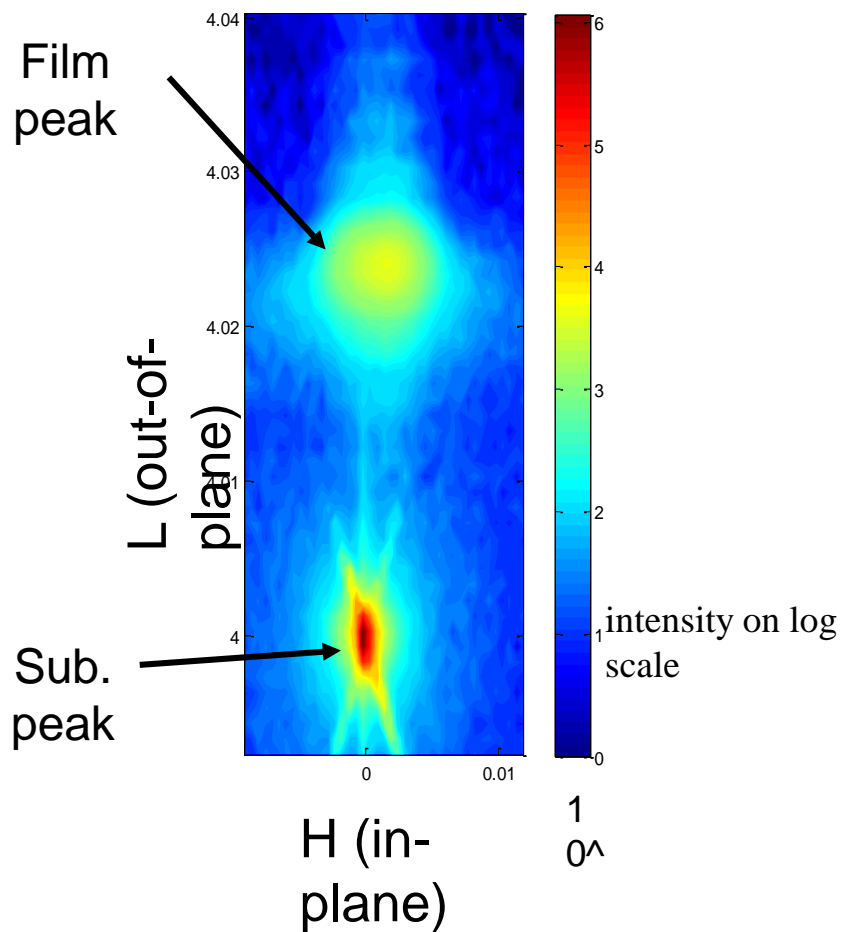
- Thin Film

- Will the scattering from the substrate interfere?

- Weak - as it is not in diffracting condition (e.g., single crystal substrates)
- Can it be avoided by placement of the detector?
 - harder with an area detector

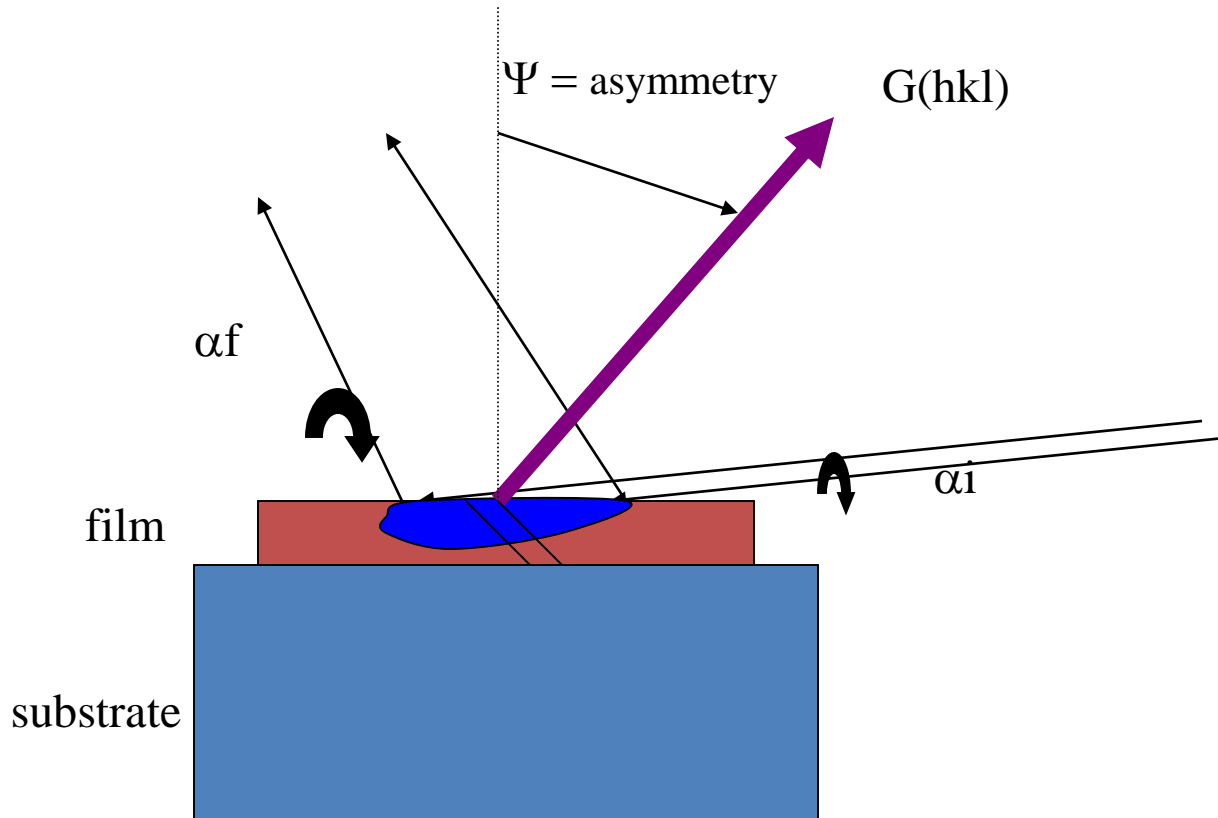


Thin Film



Thin Film: Substrate interference

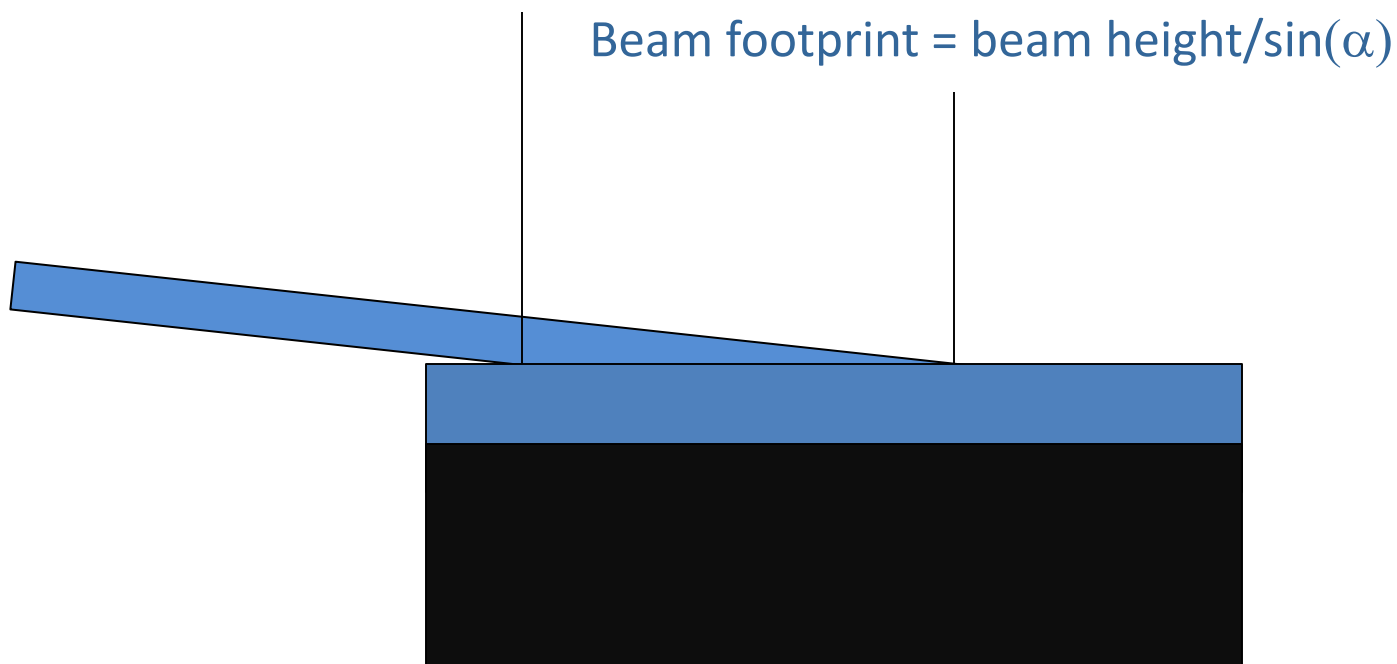
Grazing Incidence Geometry



Grazing Incidence : Beam footprint

SSRL

SLAC



$\alpha \sim 0.5^\circ$,

for a 100 micron beam \rightarrow beam footprint ~ 10 mm

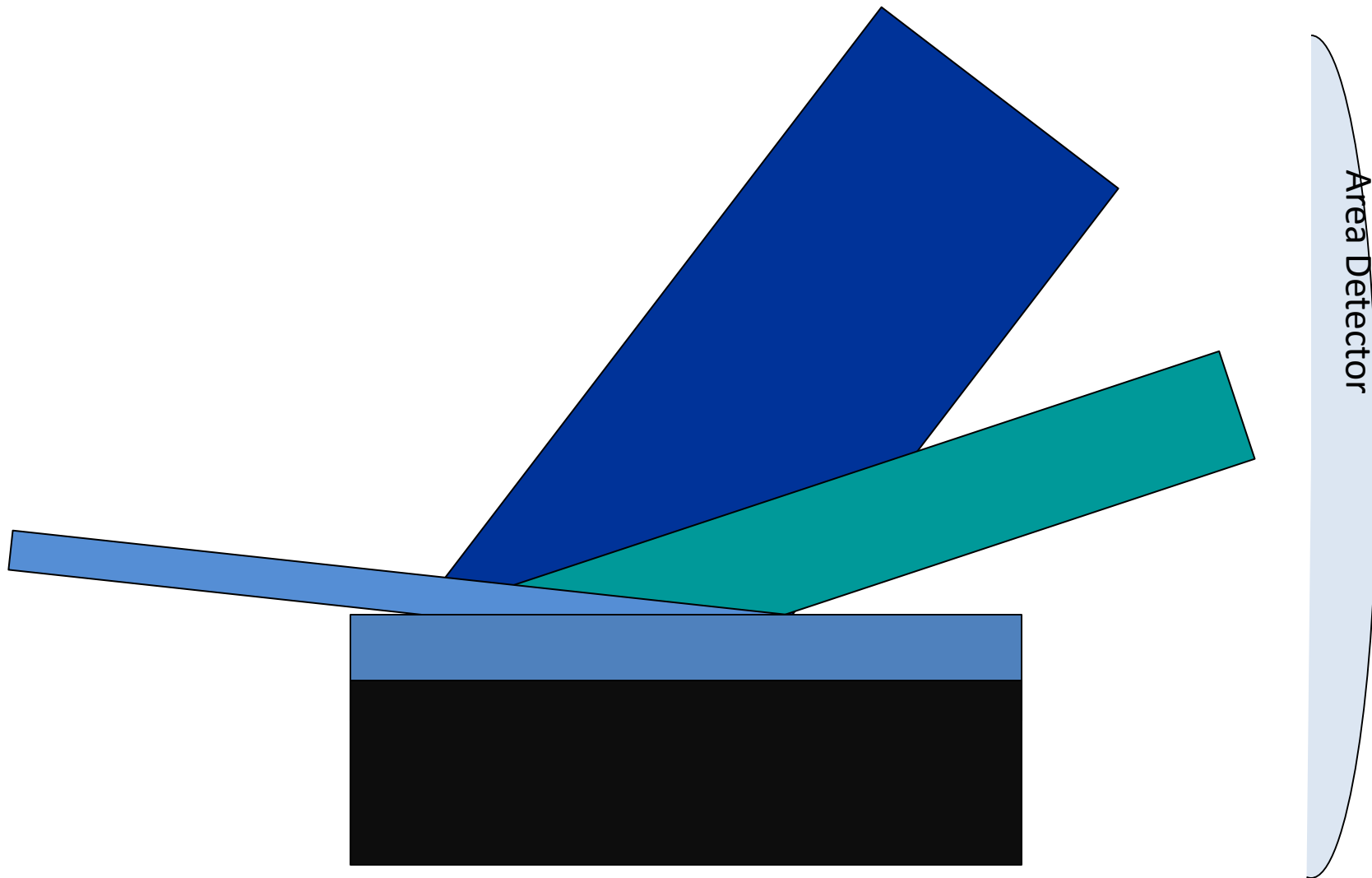
GIXRD requires long (~ 20 mm) and flat samples



SSRI-

Grazing Incidence: Peak widths

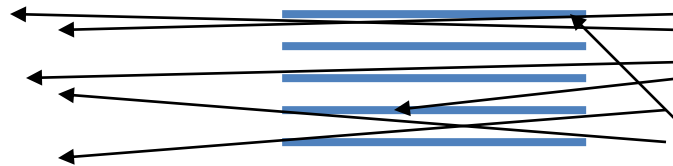
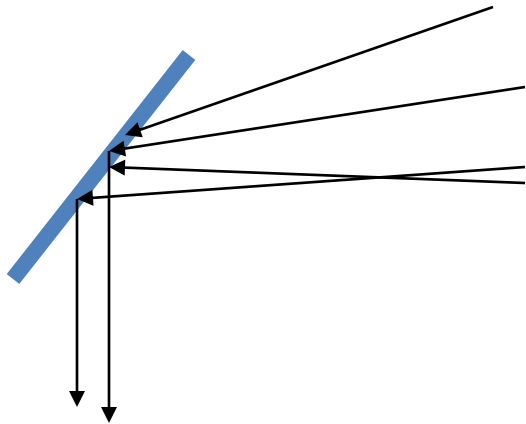
SLAC



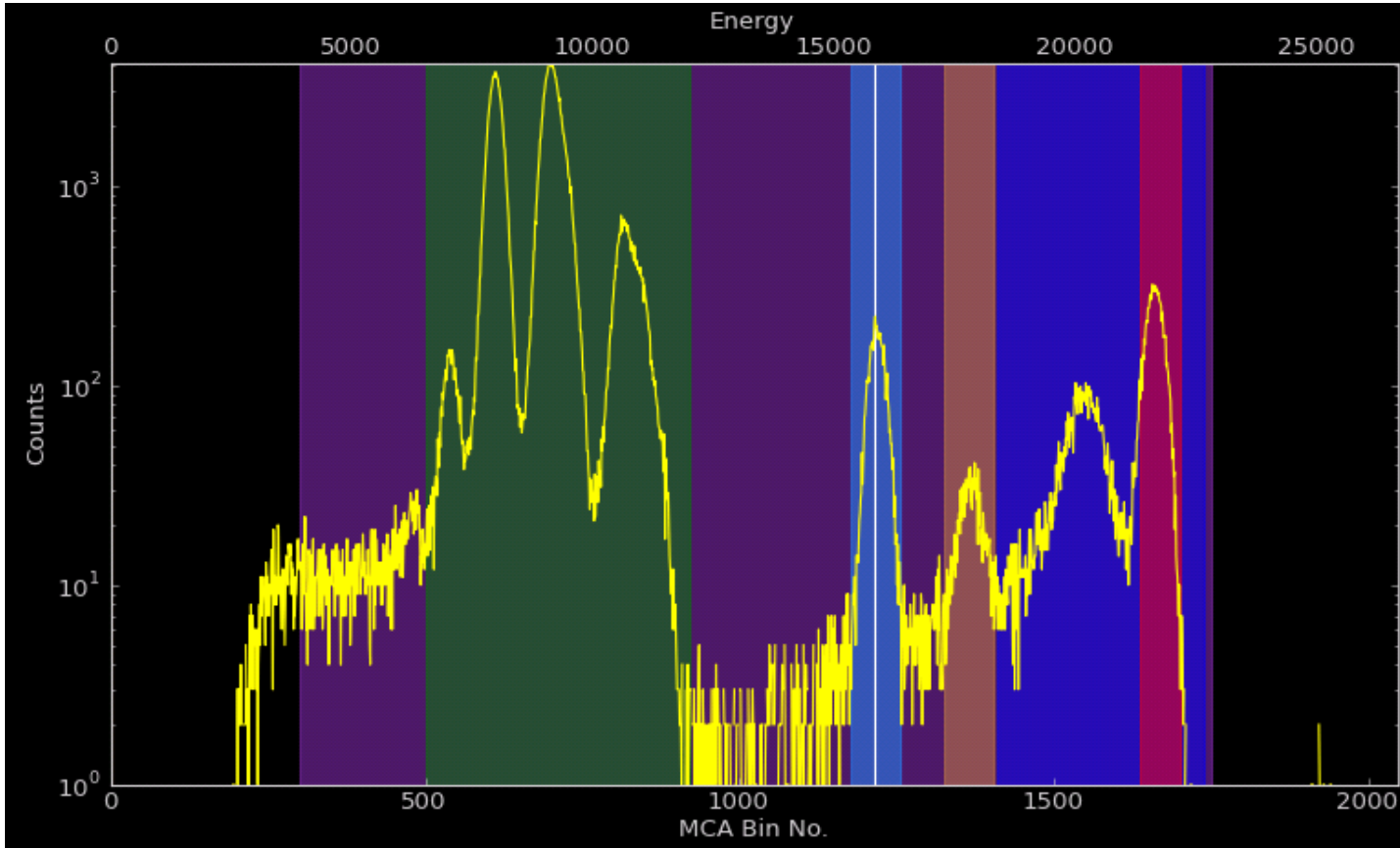
Is parallel beam detection required?



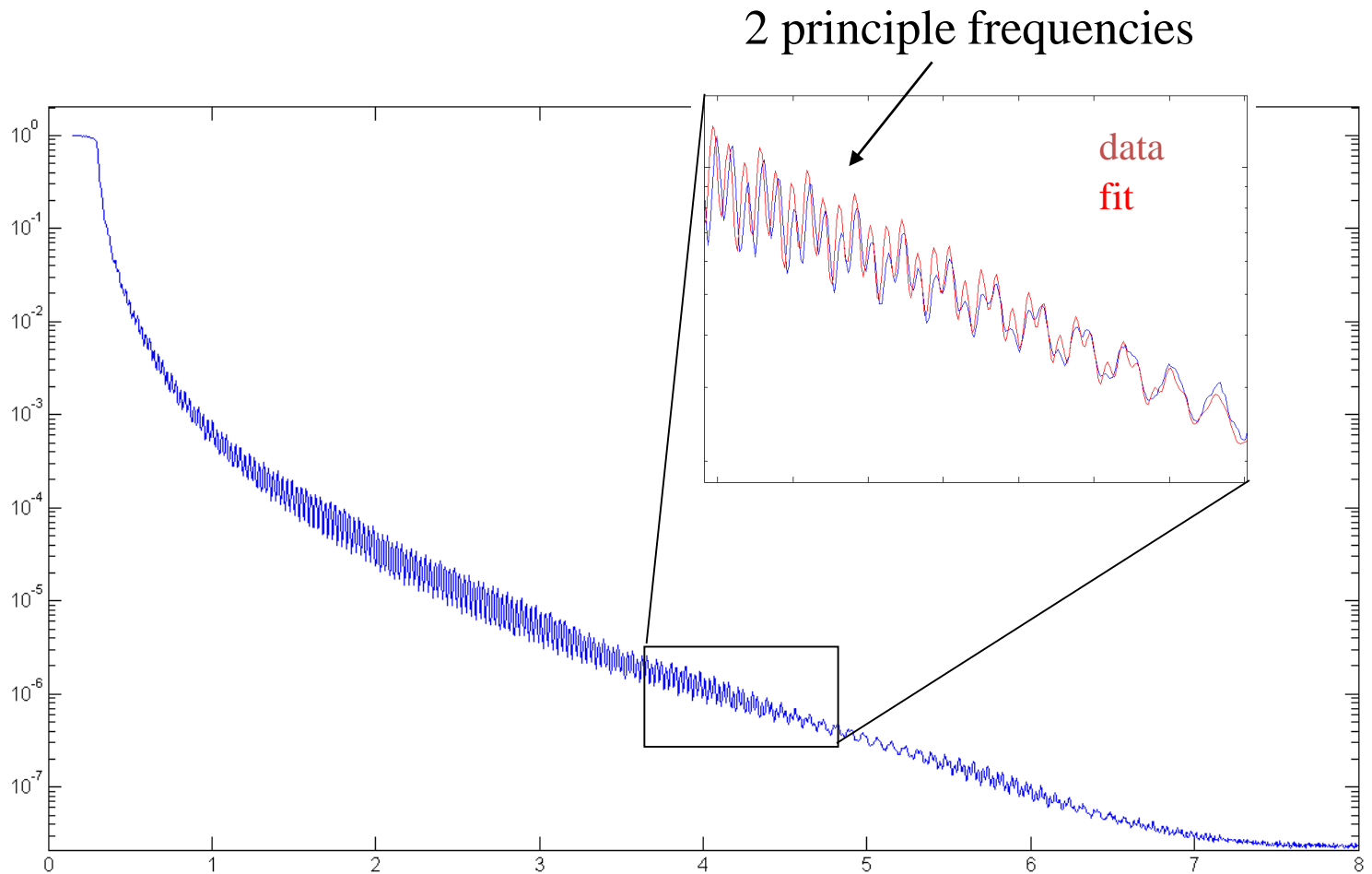
- Crystal Analyzer
 - Perfect crystal (Si or Ge)
- Soller Slits



Is Energy Discrimination Necessary?



How much dynamic range needed?





How much angular resolution needed?



Combine Detectors?

- Area detector for lower resolution, quick measurements?
- High resolution point detector for details?

Optimal scan range and resolution

Instrumental Resolution & peak width

$$W_m^2 = W_s^2 + W_i^2$$

Measured sample instrumental

Ideally $W_i < 0.5 W_s$

But not too small as improvement in instrumental resolution comes at the cost of intensity

OK for nanocrystals

Area Detector

1mRad Soller Slits

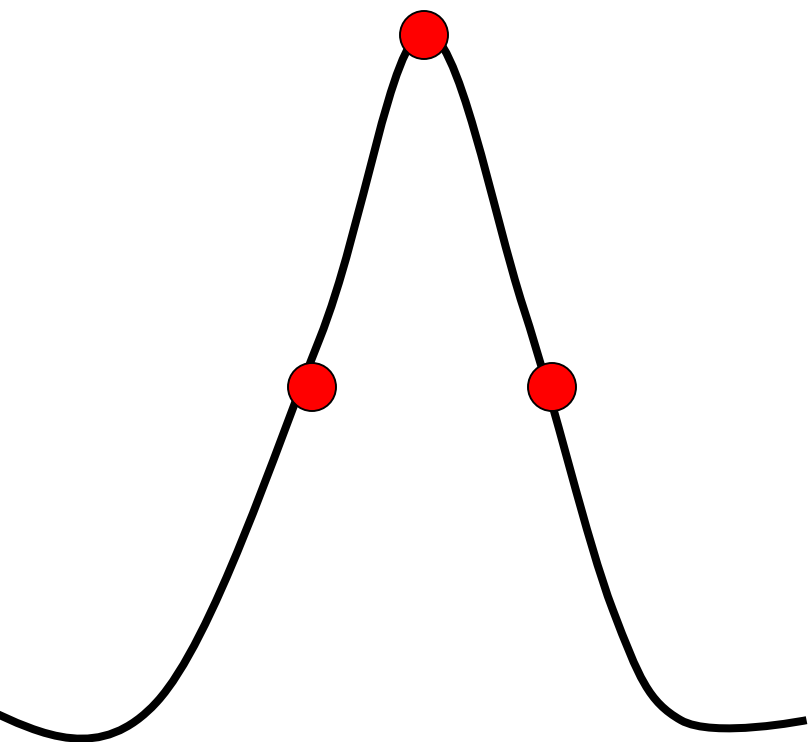
Crystal Analyzer

Resolution
Increased



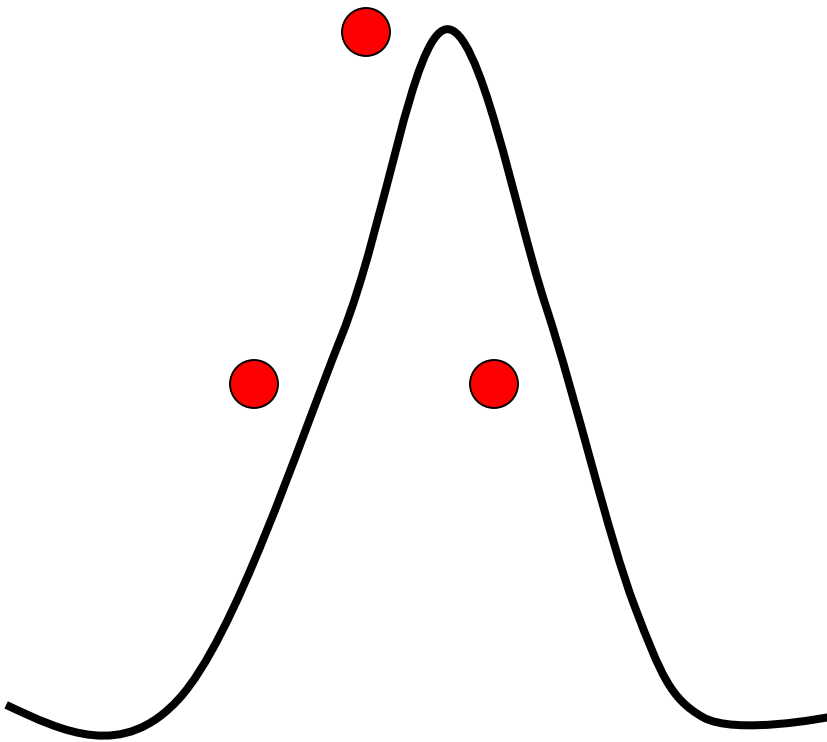
Optimal Scan

Step Size



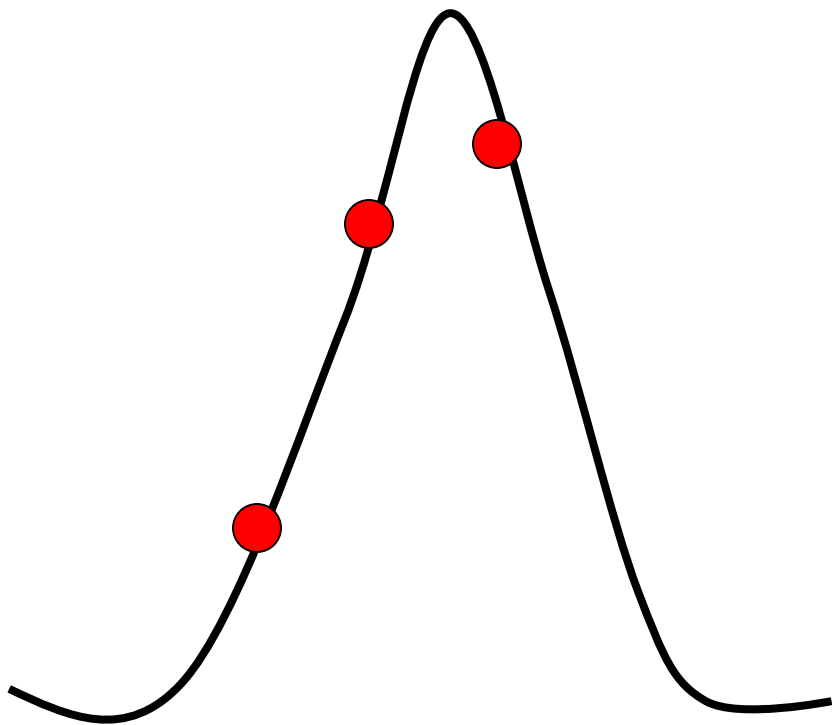
Optimal Scan

Step Size



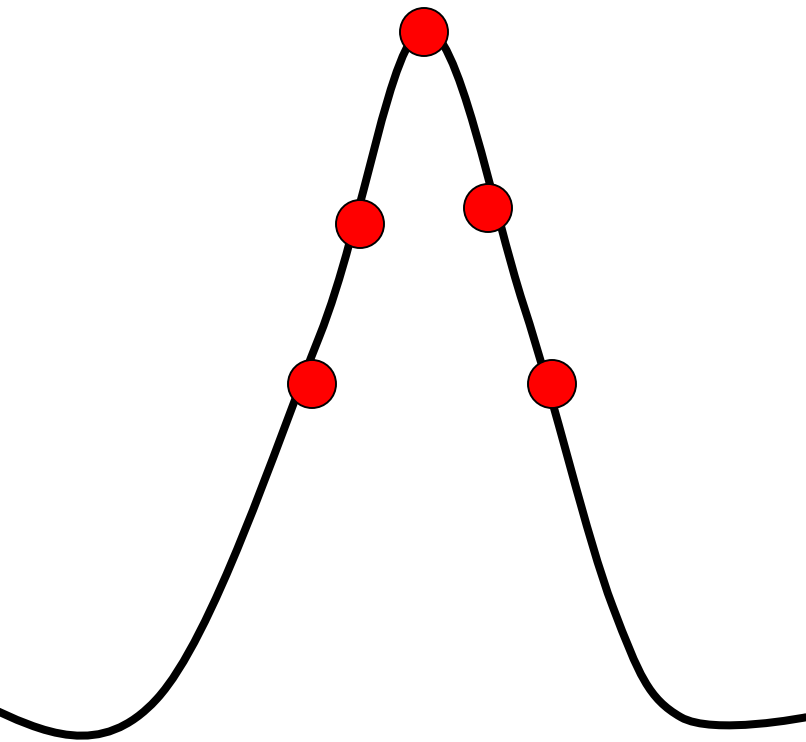
Optimal Scan

Step Size



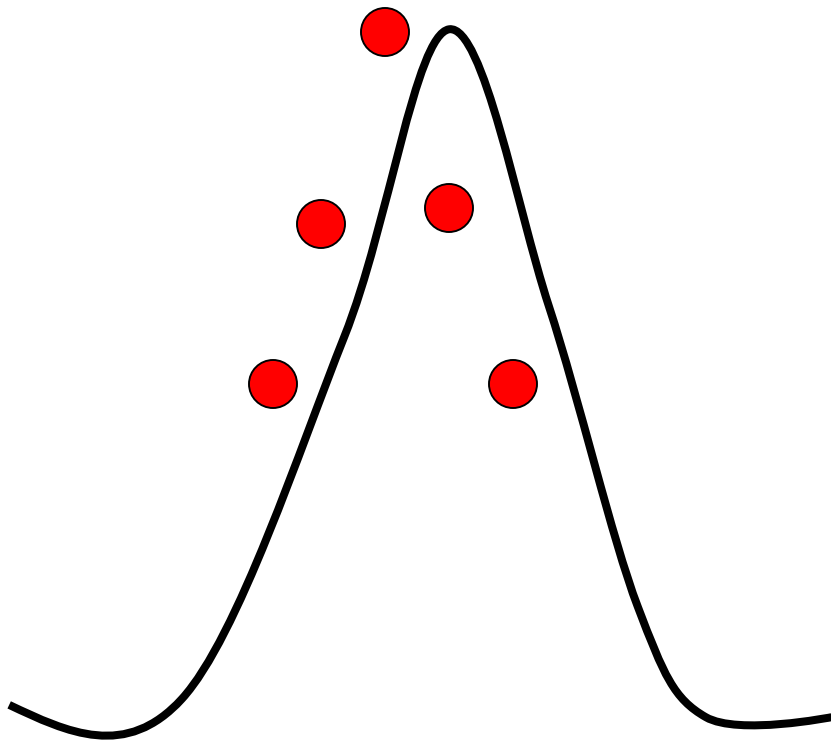
Optimal Scan

Step Size



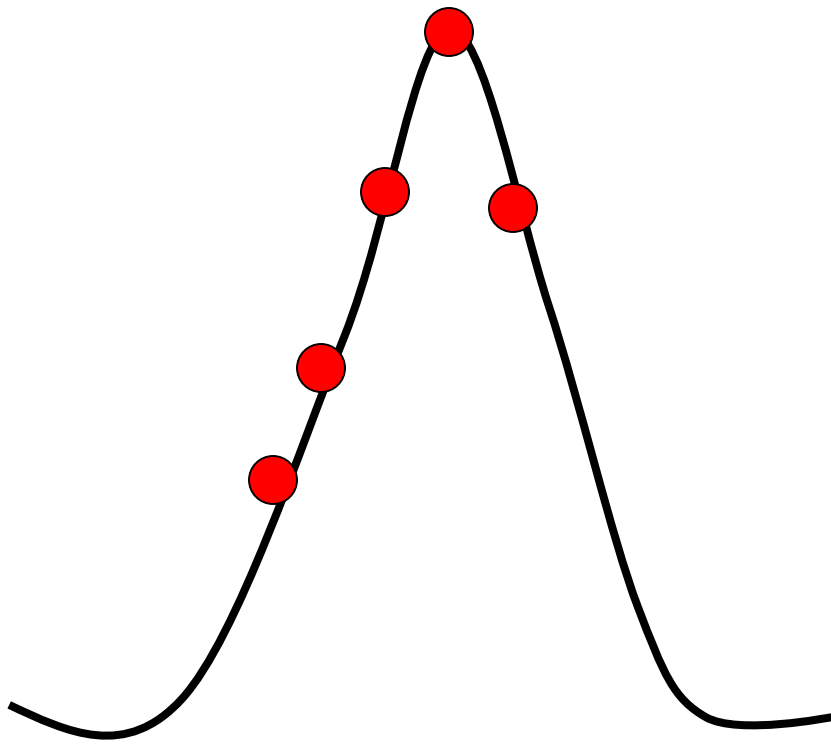
Optimal Scan

Step Size

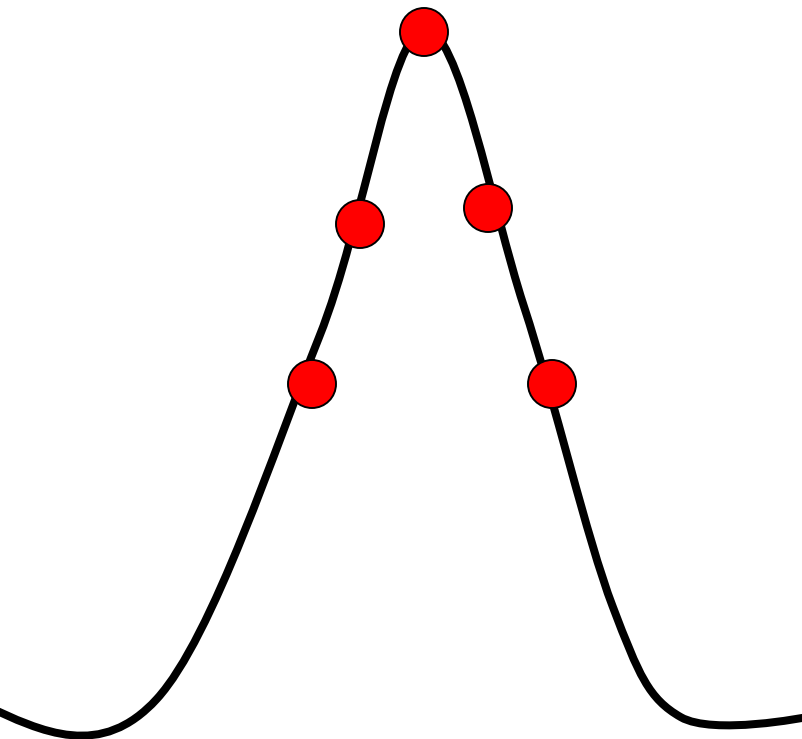


Optimal Scan

Step Size



Step Size



Minimum of 5 points in FWHM

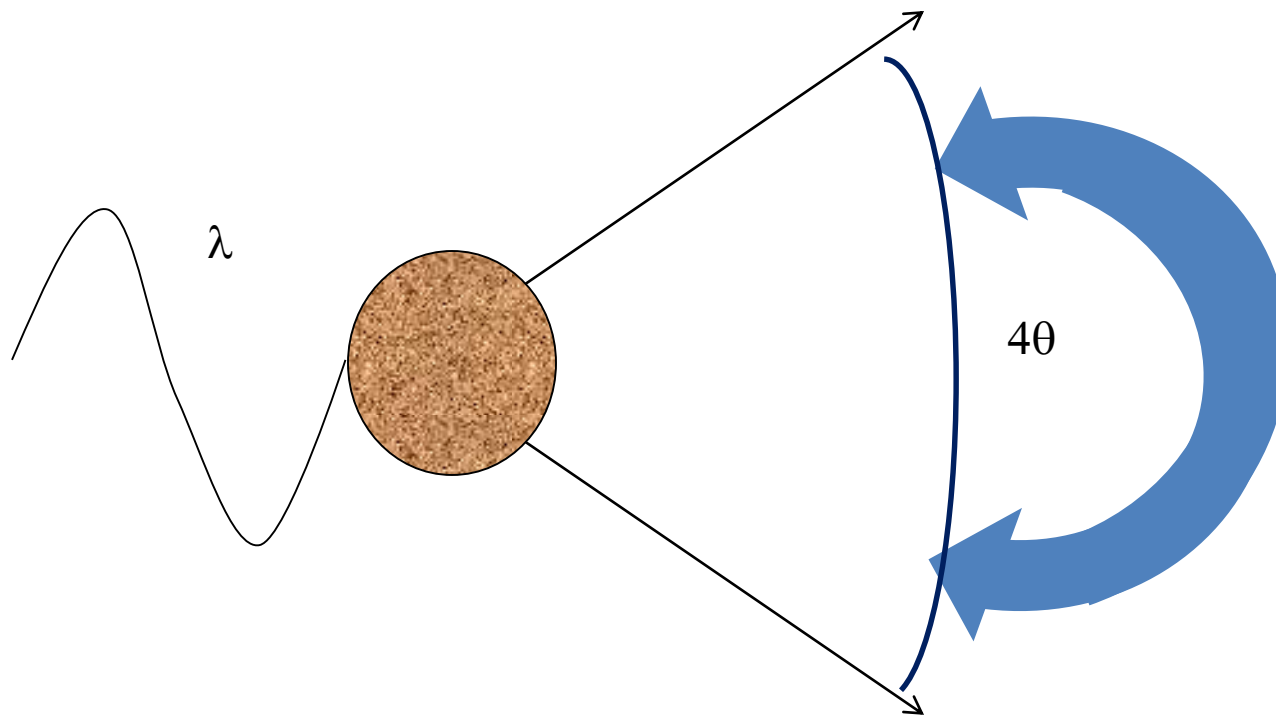
But probably not more than 10

Keep the dwell time so that Each Sweep is not too long
~ 2 -6 hr.

If better statistic reqd
Repeat sweeps and add.

- Averages out some of the time dependent drifts
- If unexpected beam dumps still have usable data
- Allows better control on statistics

Diffraction Pattern



Diffraction Pattern:

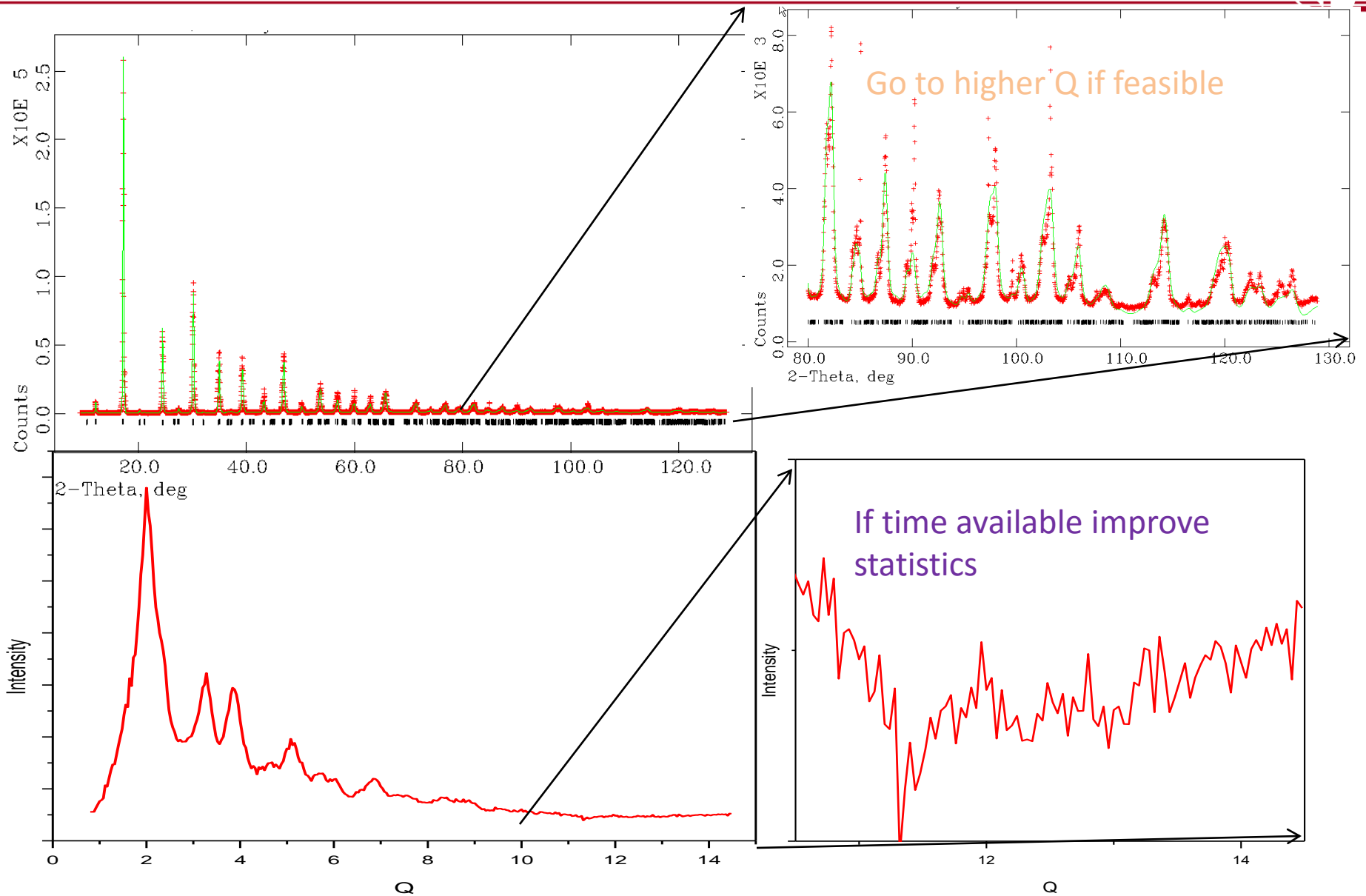
Contains all the contrast relevant information at the resolution of

$$\lambda/2\sin(\theta) \sim 2\pi/Q_{\max}$$

$$Q = 4\pi \sin(\theta) E/hc$$



Best use of time



- Before the experiment
 - Q Range
 - Q resolution
 - X-ray Energy
 - Fluo bkg, Resonance scattering
 - Diffractometer and sample geometry -
 - 2 circle, 4 circle, GI, transmission
 - Detectors
- During Data collection
 - Scan parameters (step size, dwell time.)
 - Beam damage?
 - Dehydrate, photo- redox, reacts with the environment



Questions?