Lesson 2
Diffractometers

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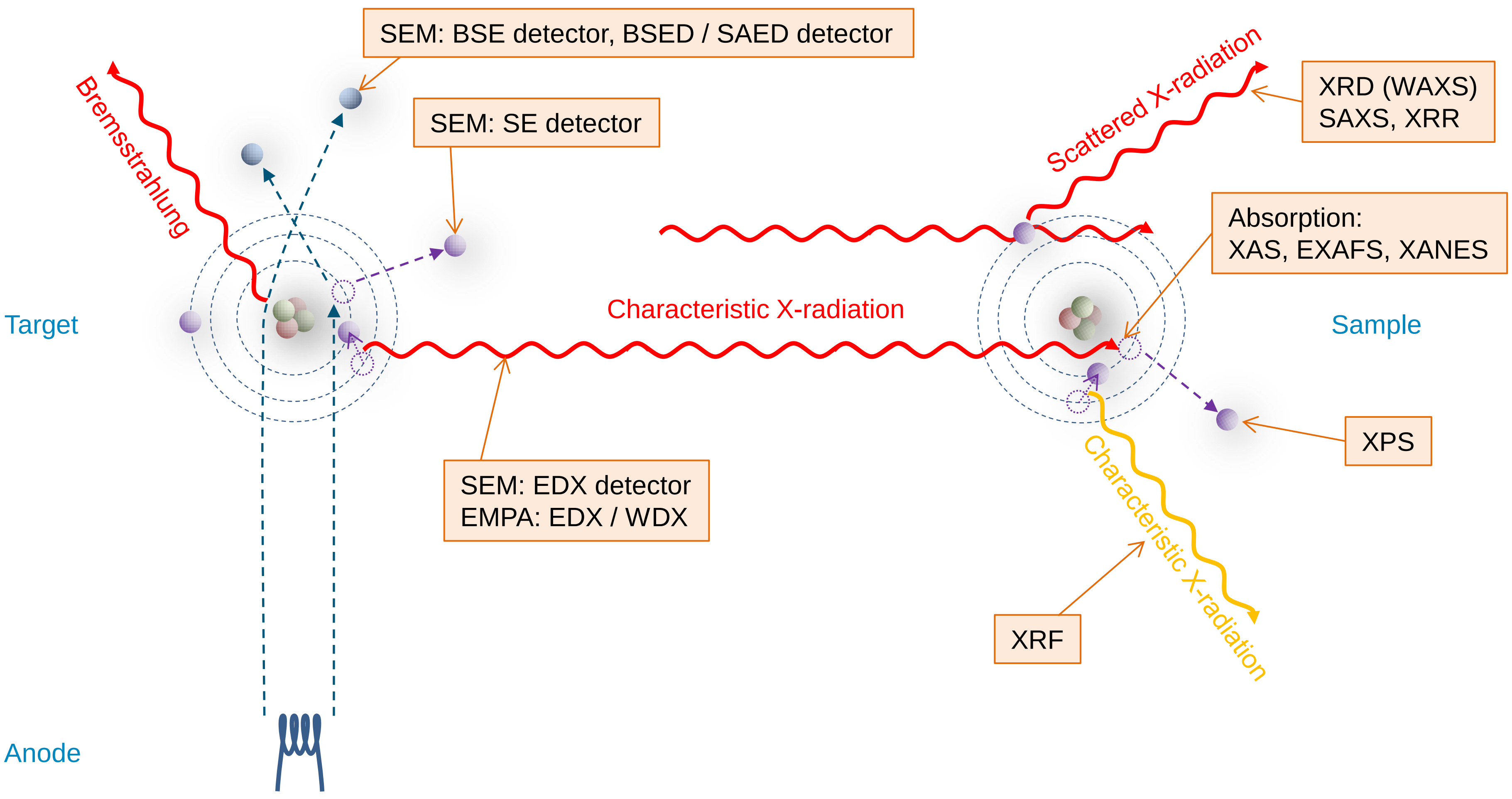
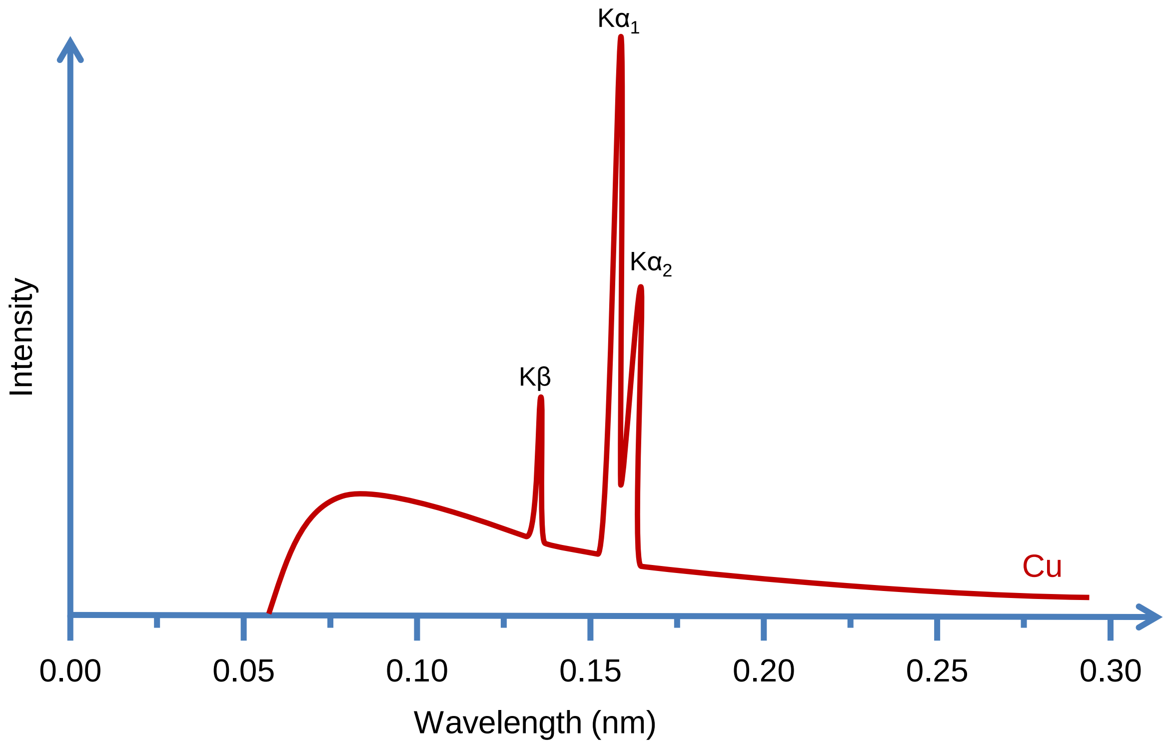
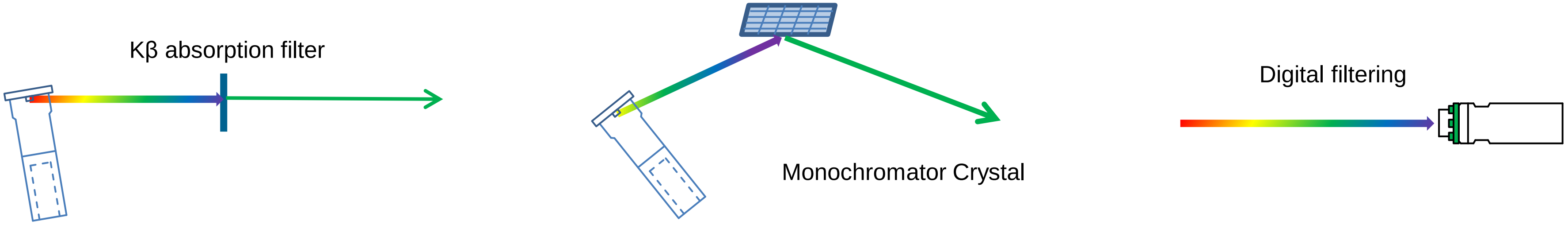
Repetition: Generation of X-rays / Diffraction

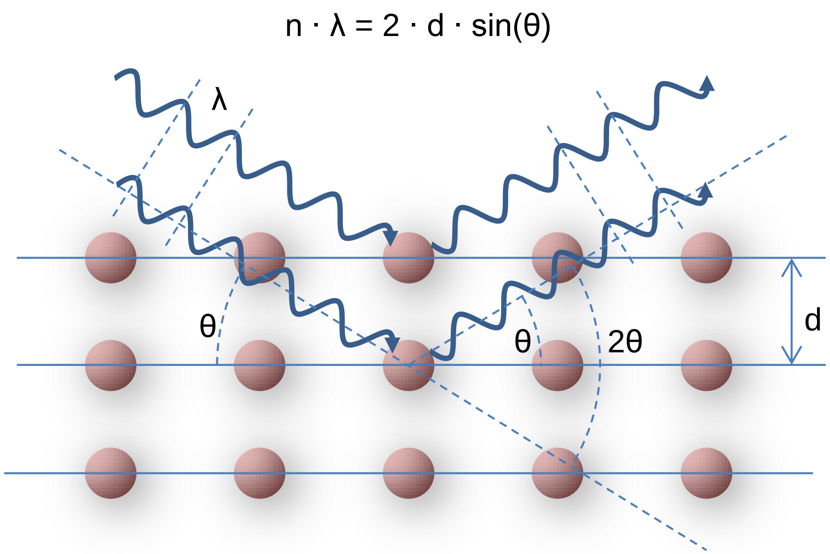


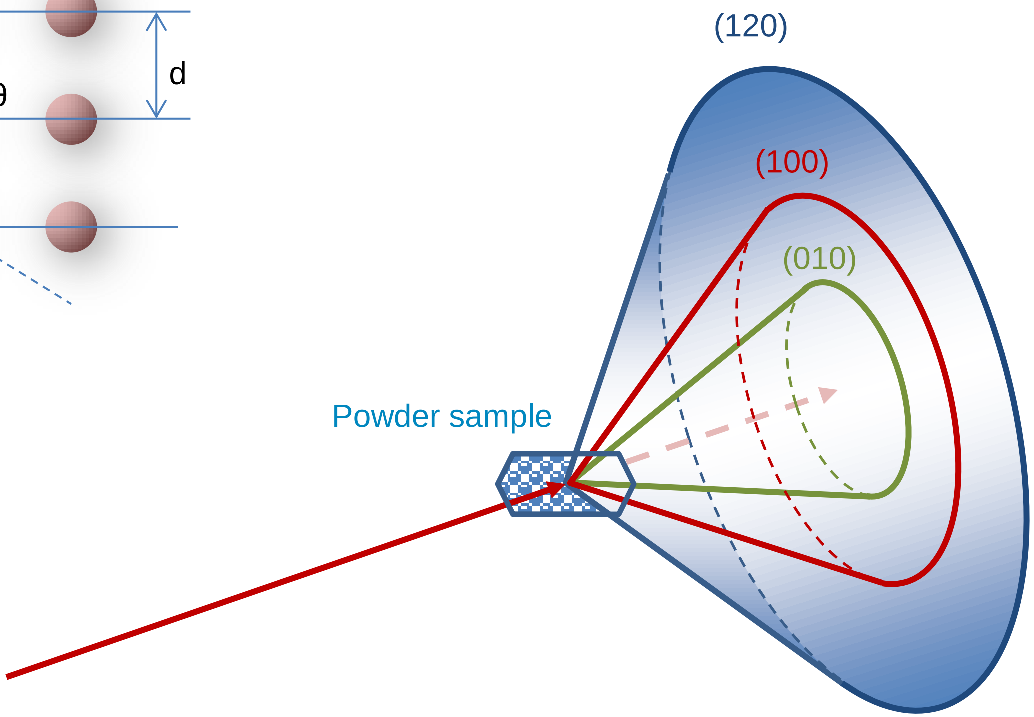
Repetition: Generation of X-rays



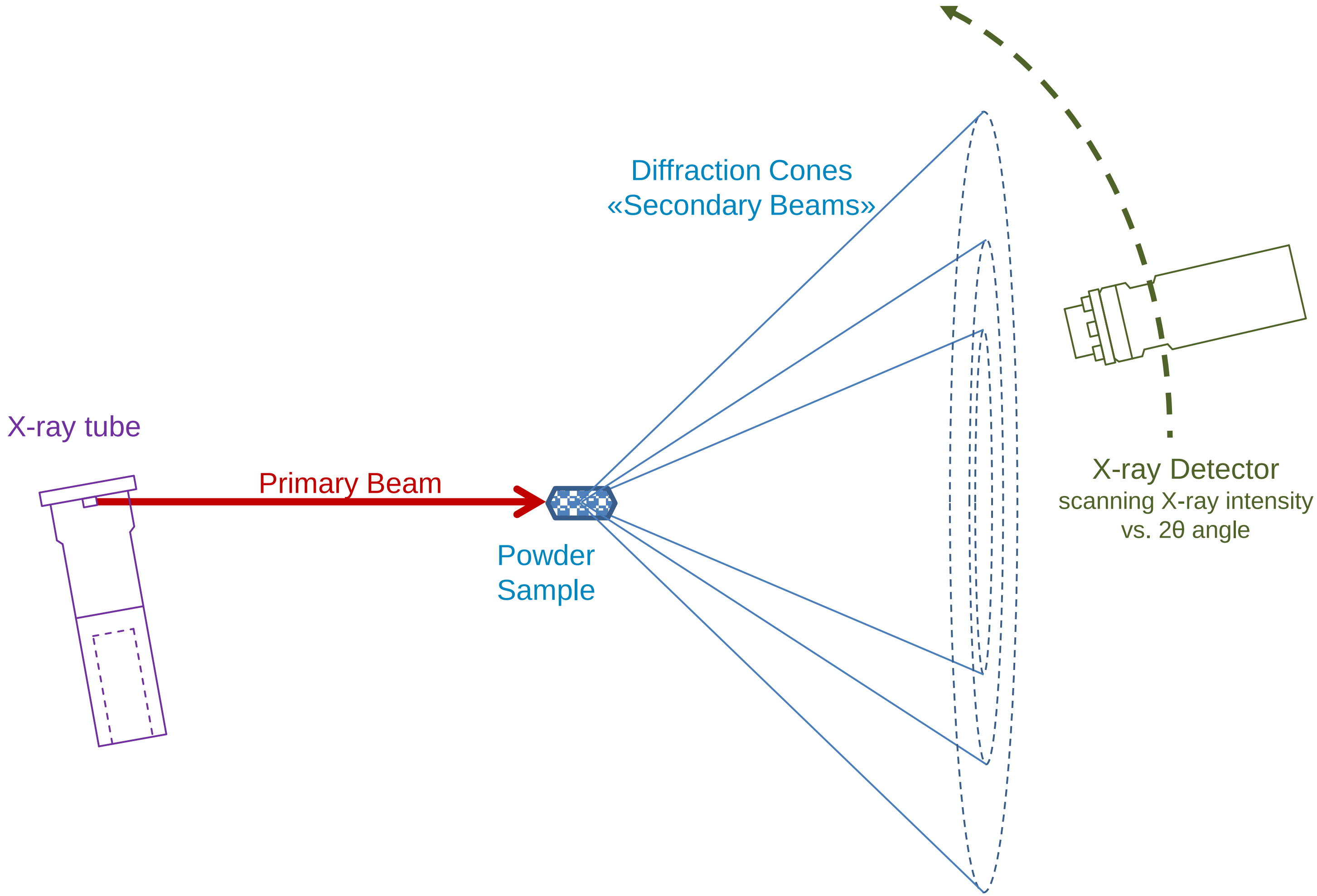


Repetition: Powder Diffraction





Repetition: Powder Diffractometer

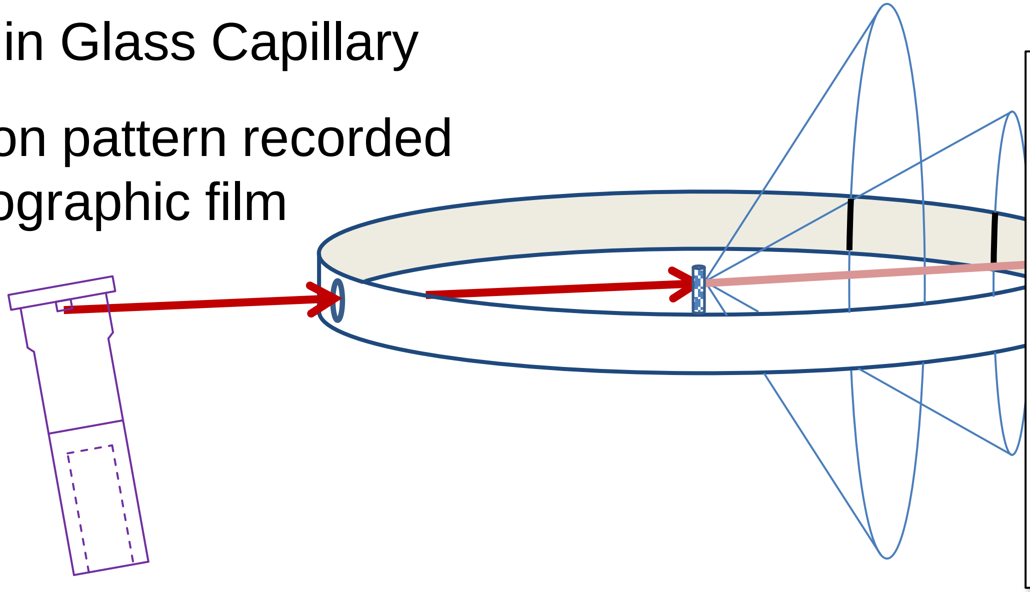


Analogue Cameras

Debye-Scherrer Camera:

Powder in Glass Capillary

Diffraction pattern recorded on photographic film



Various alternative setups:

Gandolfi …

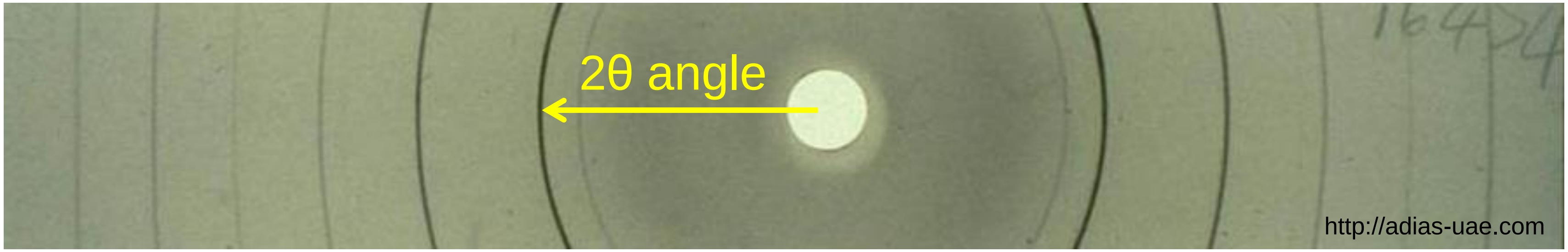
Guinier …

Straumanis …

Bradley …

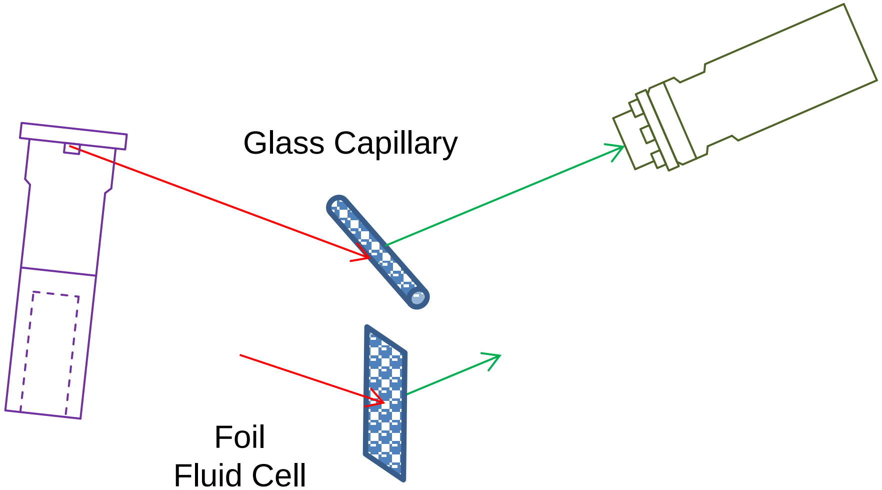
Seemann-Bohlin …

…Camera



Digital Diffractometers

Transmission Geometry

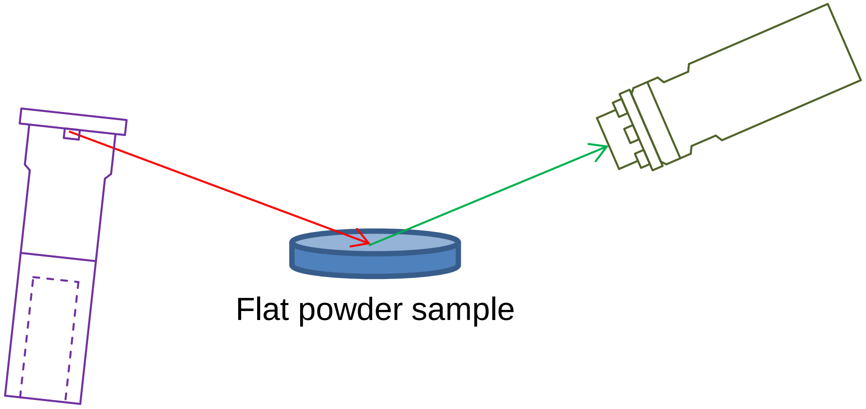


Capillaries are ideal for:

- Light atoms (Polymers, Pharmaceuticals)
- Small amounts
- Hazardous materials
- Air-sensitive materials

Use characteristic radiation with low absorption coefficient

Reflective Geometry



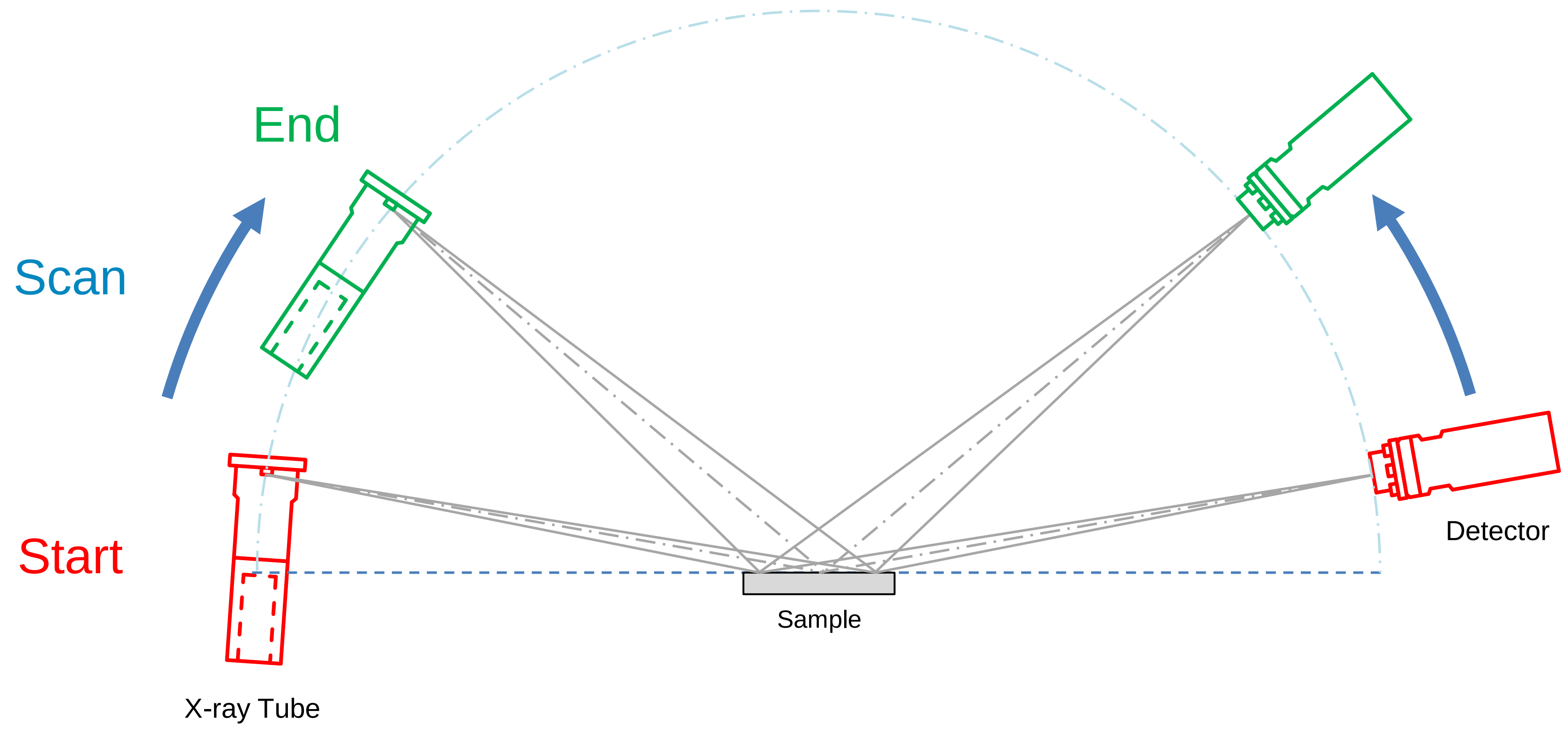
Reflective Geometry is ideal for:

- Absorbing materials (Ceramics, Metals)
- Thin films
- Texture analysis

Use characteristic radiation with high absorption coefficient

Bragg-Brentano Parafocusing Diffractometer

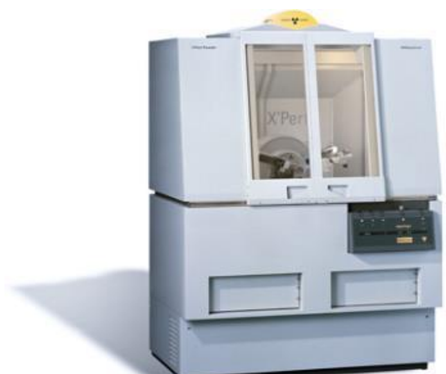
Tube and Detector move symmetrically



Instruments

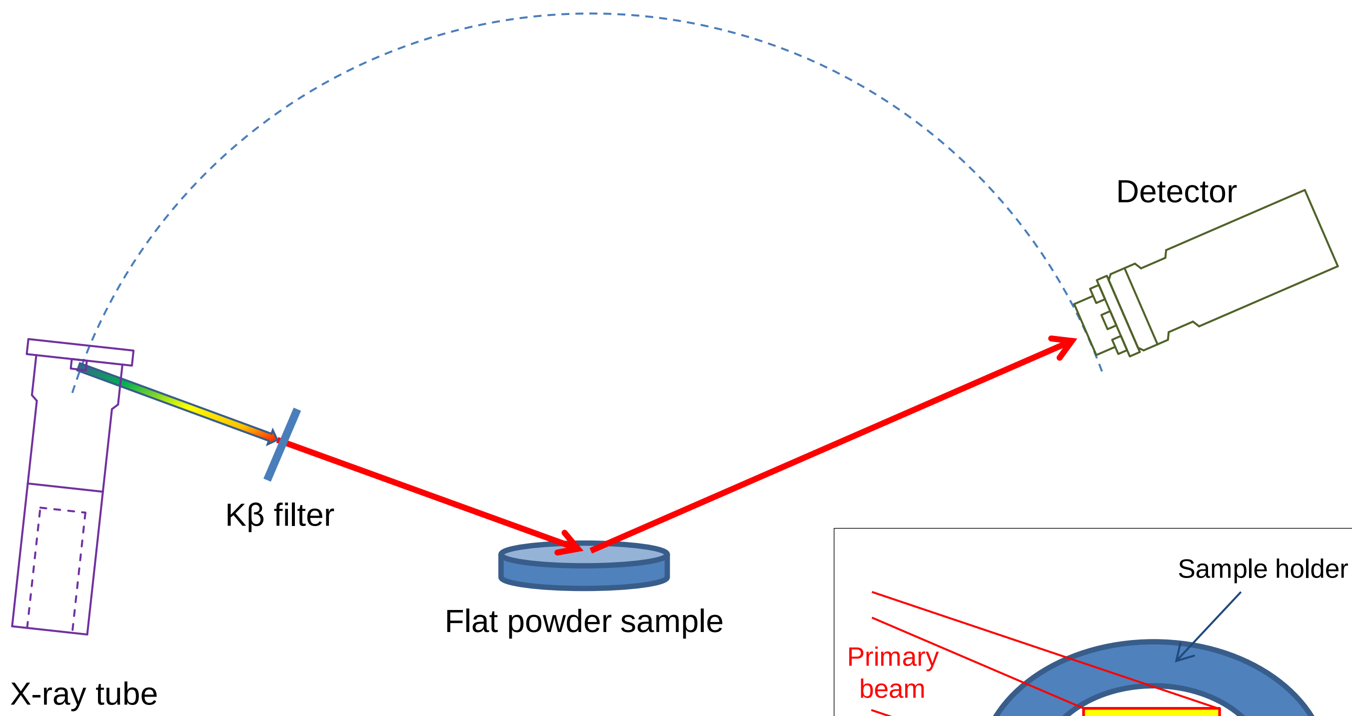
Lab	Instrument	Monochromator	Configuration
RMS Foundation	Bruker D8	Energy dispersive Detector	Bragg-Brentano (Reflection) Debye-Scherrer (Capillary)
Uni Bern	Panalytical X'Pert	Ni-Filter	Bragg-Brentano (Reflection)
Uni Bern	Panalytical CubiX	Graphite Monochromator	Bragg-Brentano (Reflection)

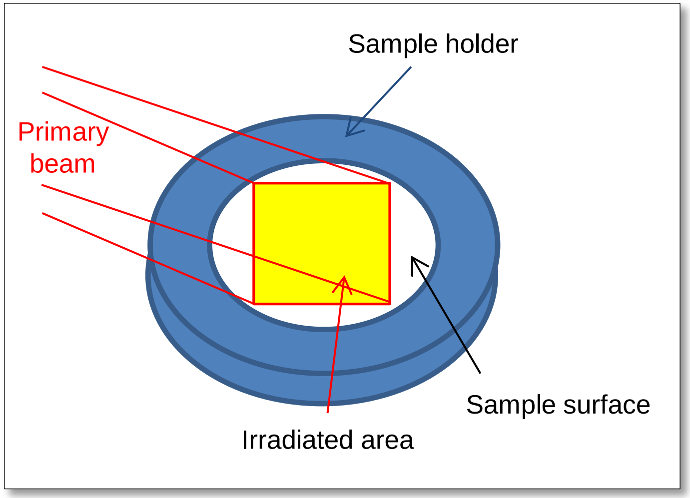

Bruker D8


Panalytical X'Pert


Panalytical CubiX

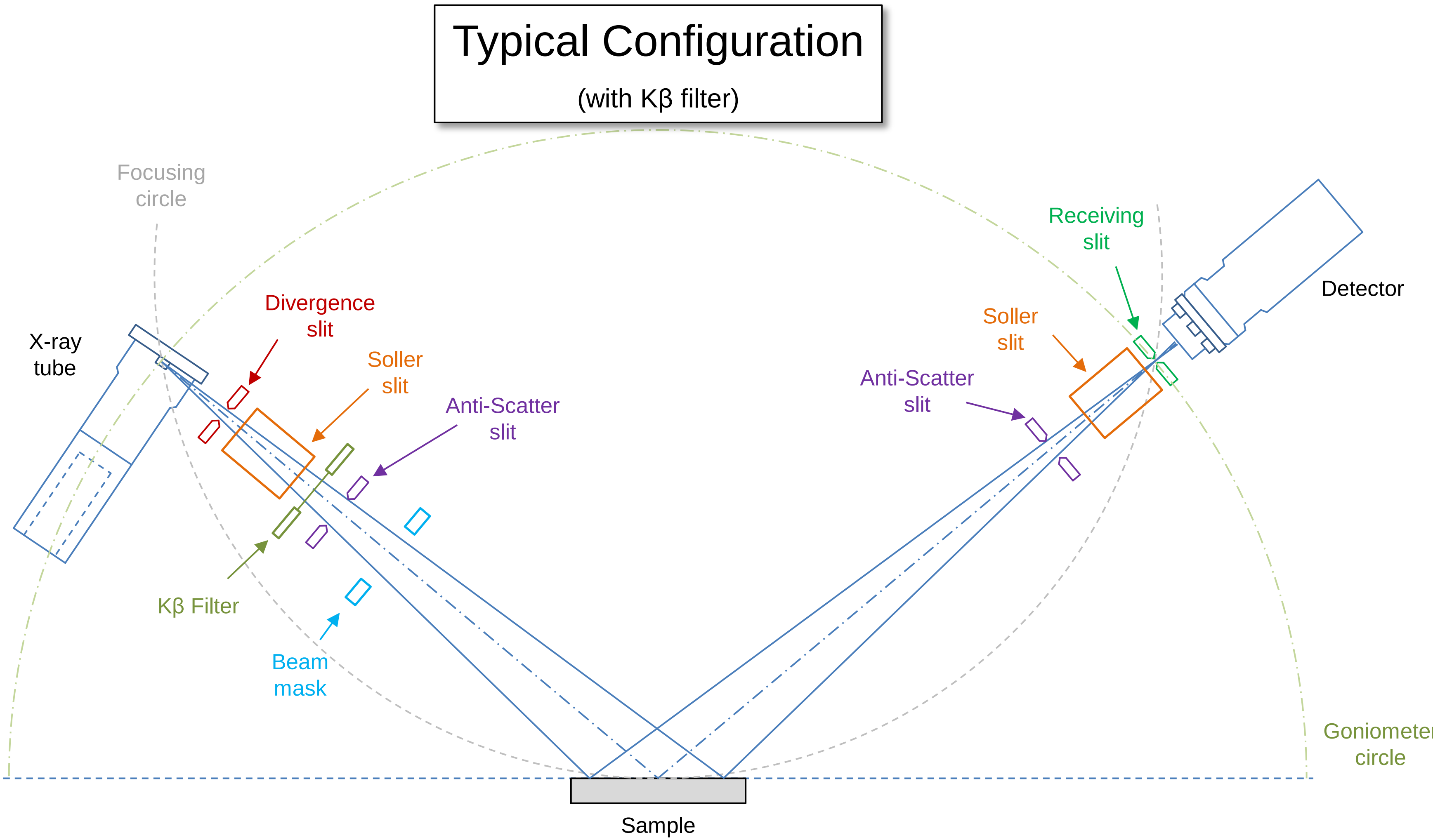
Bragg-Brentano Diffractometer





More optical elements are required to control the beam pattern.

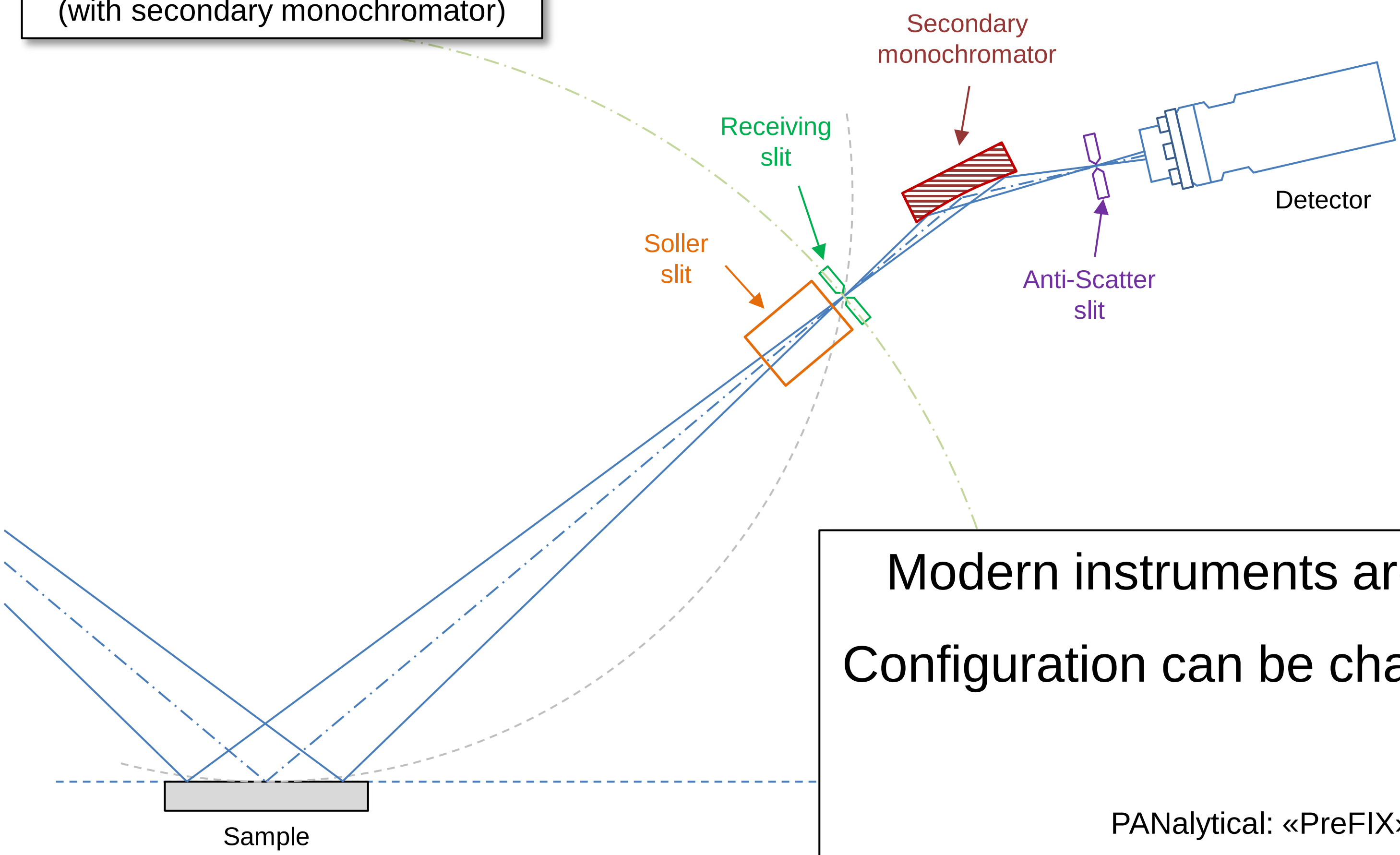
Bragg-Brentano Parafocusing Diffractometer



Bragg-Brentano Parafocusing Diffractometer

Typical Configuration

(with secondary monochromator)



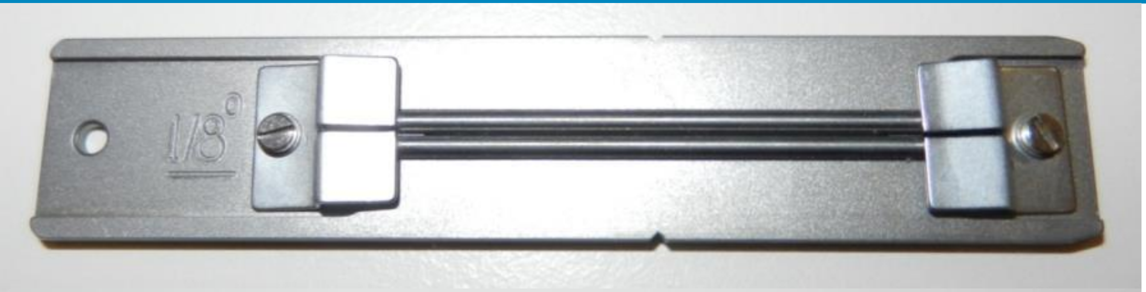
Modern instruments are modular.

Configuration can be changed easily.

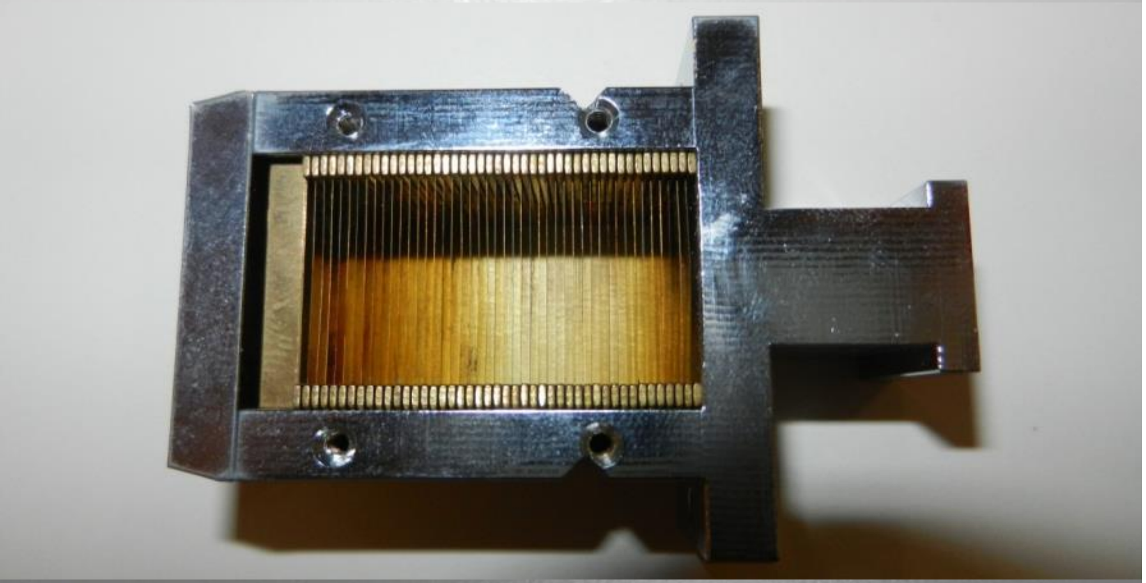
PANalytical: «PreFIX»

Bruker: «SNAP-LOCK»

Beam Divergence



Divergence Slit

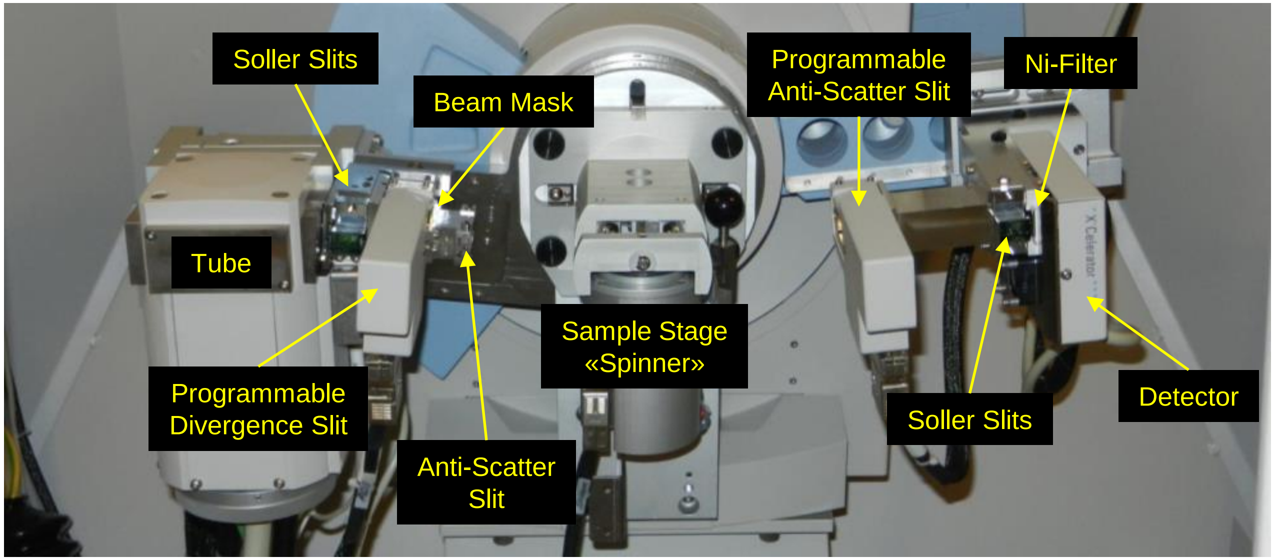


Soller Slit

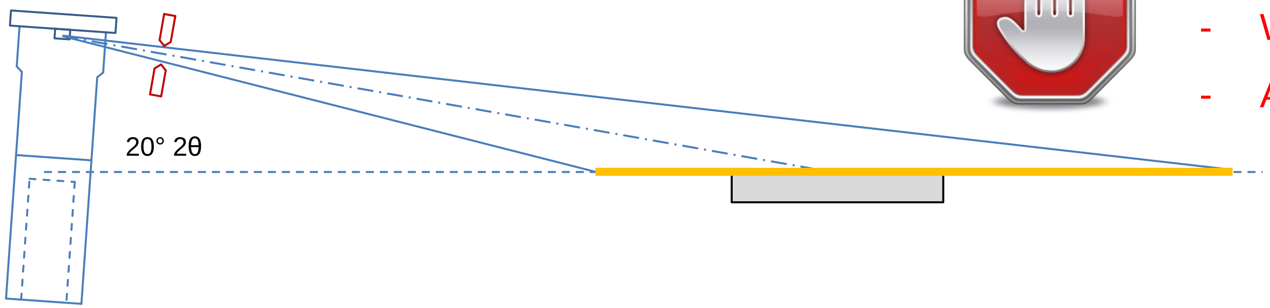
Beam Masks

Instrument Configuration

- Many optical elements = many options to optimize data quality
- How to find the best configuration?

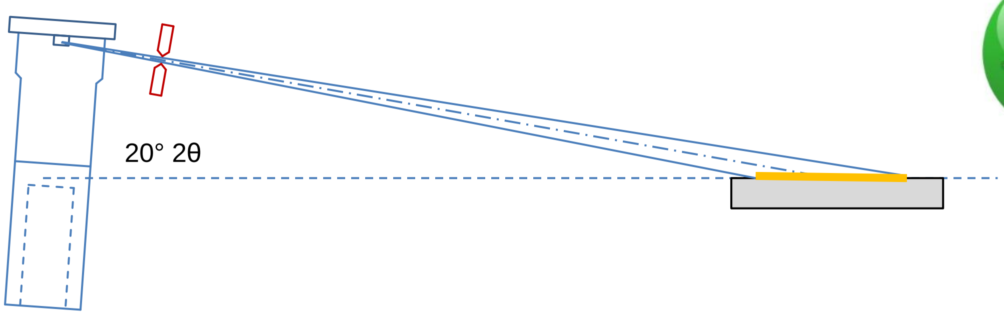


Optimum Settings: Divergence Slit

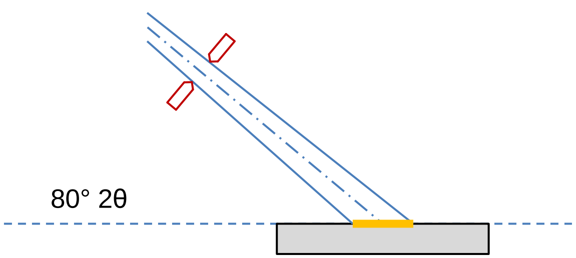


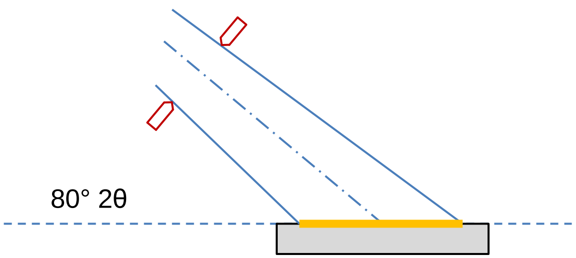
Beam overflow!

- Wrong peak intensities
- Artifact signal from sample holder



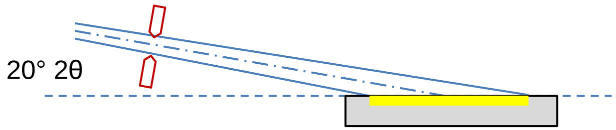
Reduced beam divergence angle

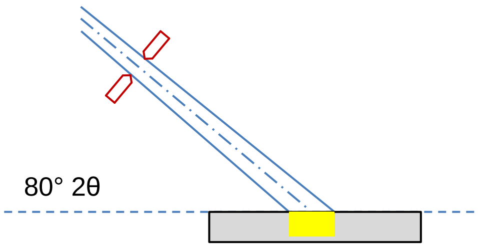




Optimum Settings: Divergence Slit

Fixed divergence slit:





Low incident angle:

- Low penetration depth
- Large illuminated area

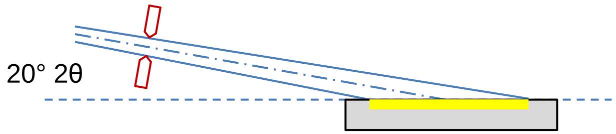
High incident angle:

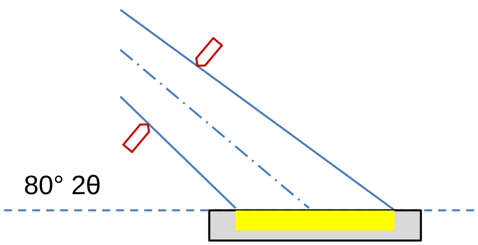
- Deep penetration depth
- Small illuminated area

Irradiated **Volume** is constant

Constant intensity of diffraction pattern

Variable divergence slit:





Low incident angle:

- Narrow divergence slit
- Low penetration depth

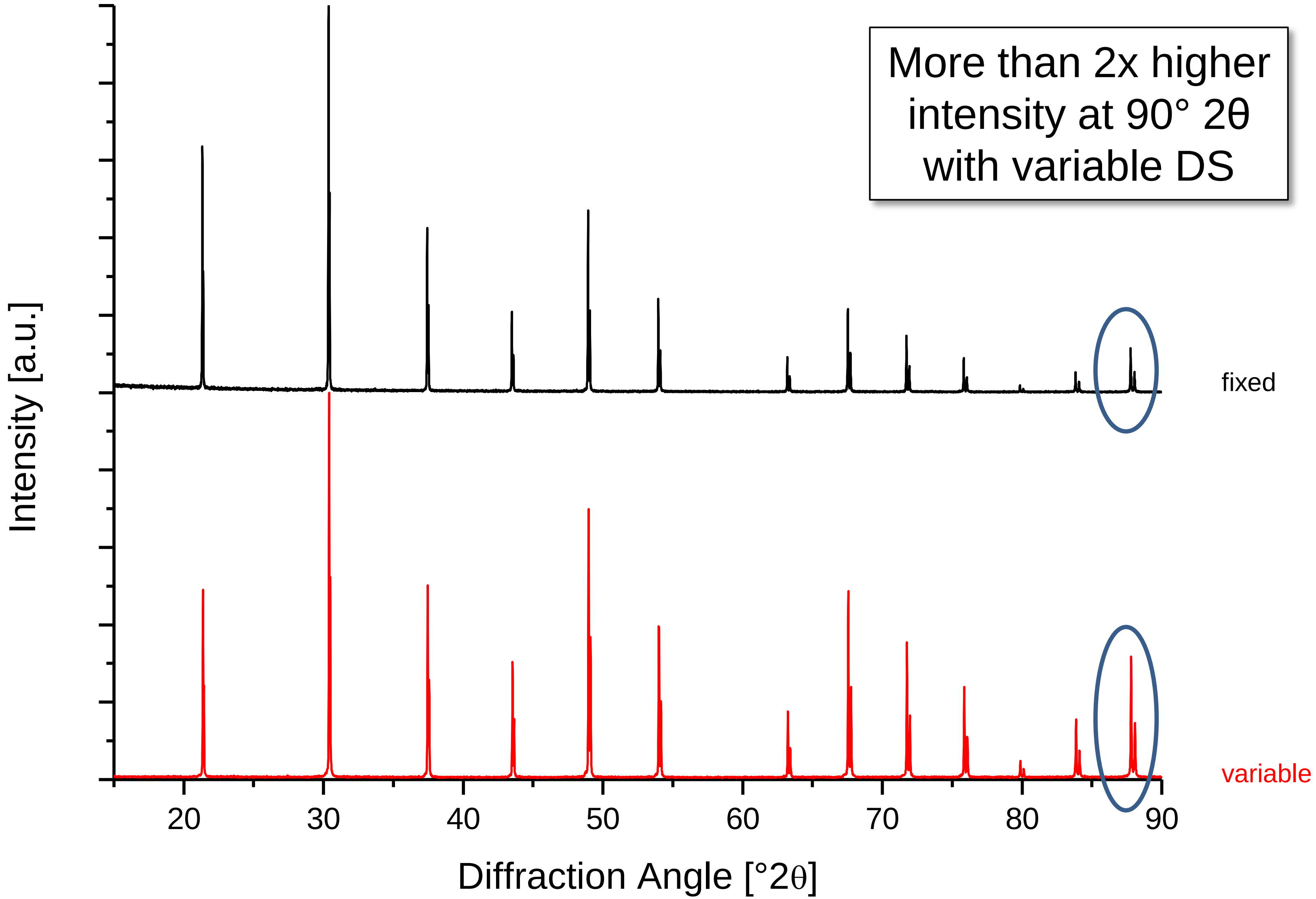
High incident angle:

- Wide divergence slit
- Deep penetration depth

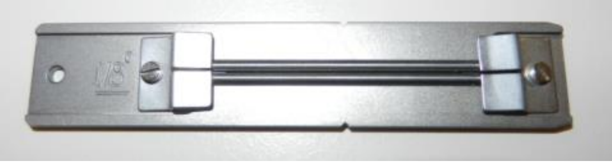
Irradiated **Area** is constant

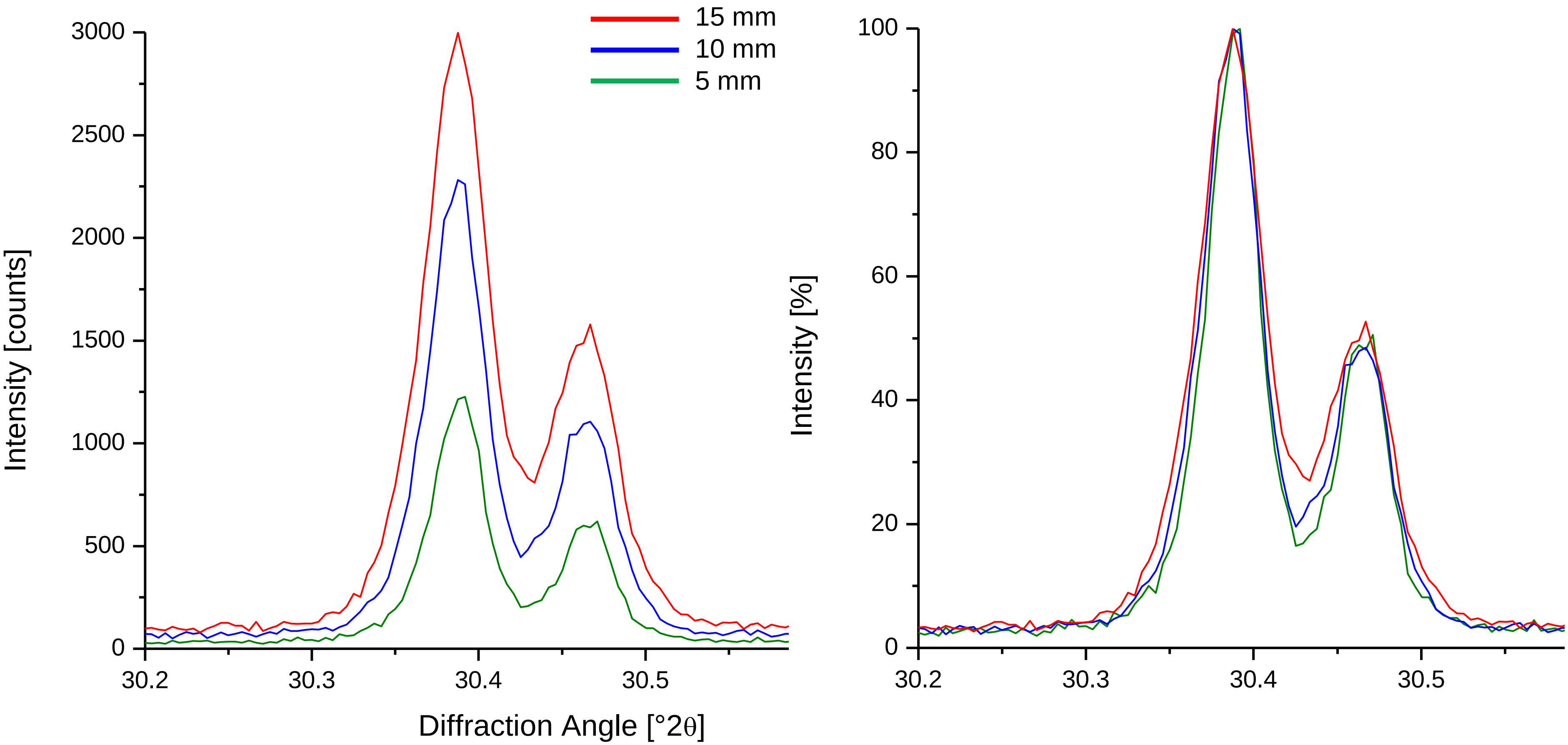
Higher diffracted intensity at high 2θ angle

Fixed vs. Variable Divergence Slit



Divergence Slit: Irradiated Length

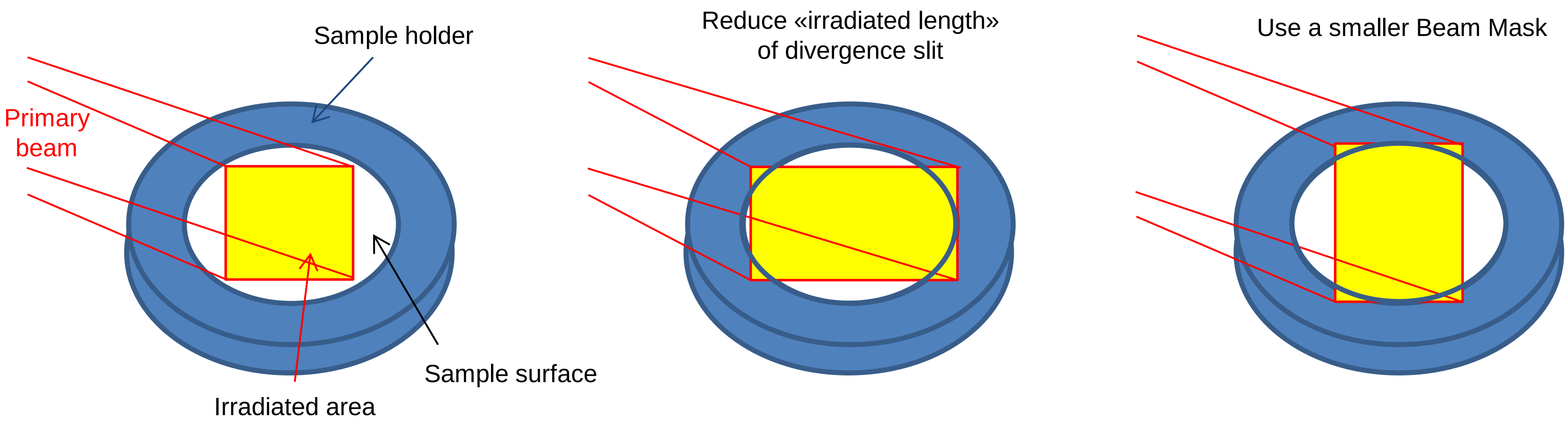




Soller Slits: 0.02 rad, Beam Mask: 10mm

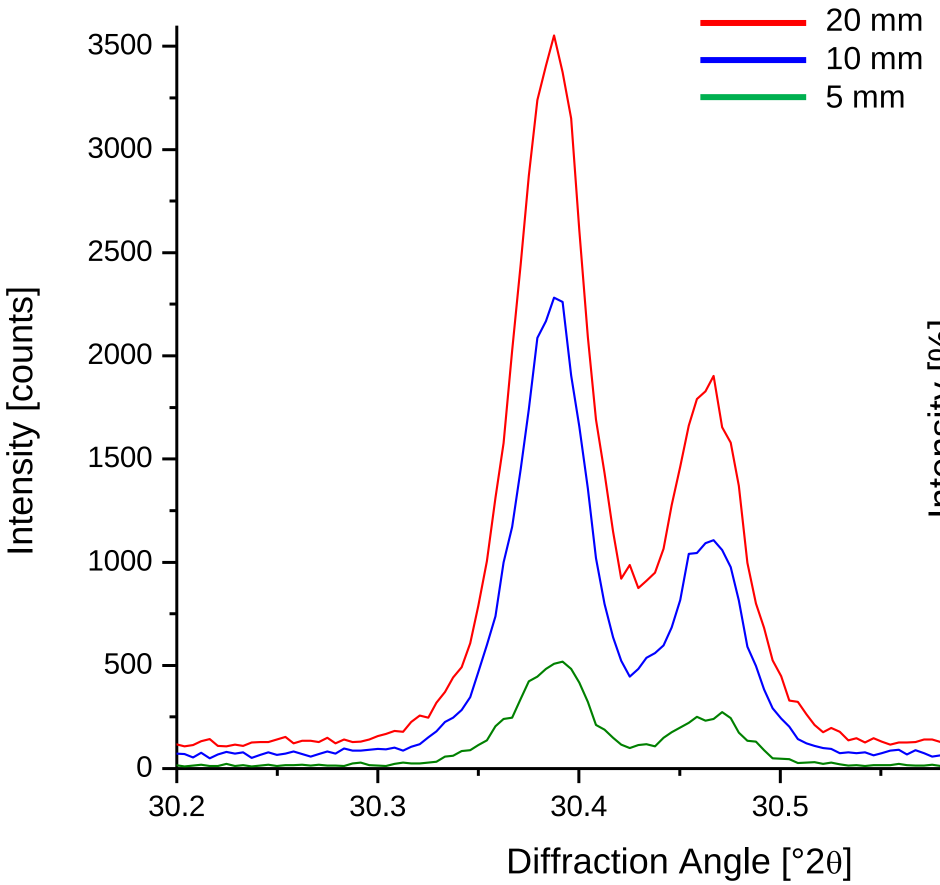
Optimum Settings: Divergence Slit

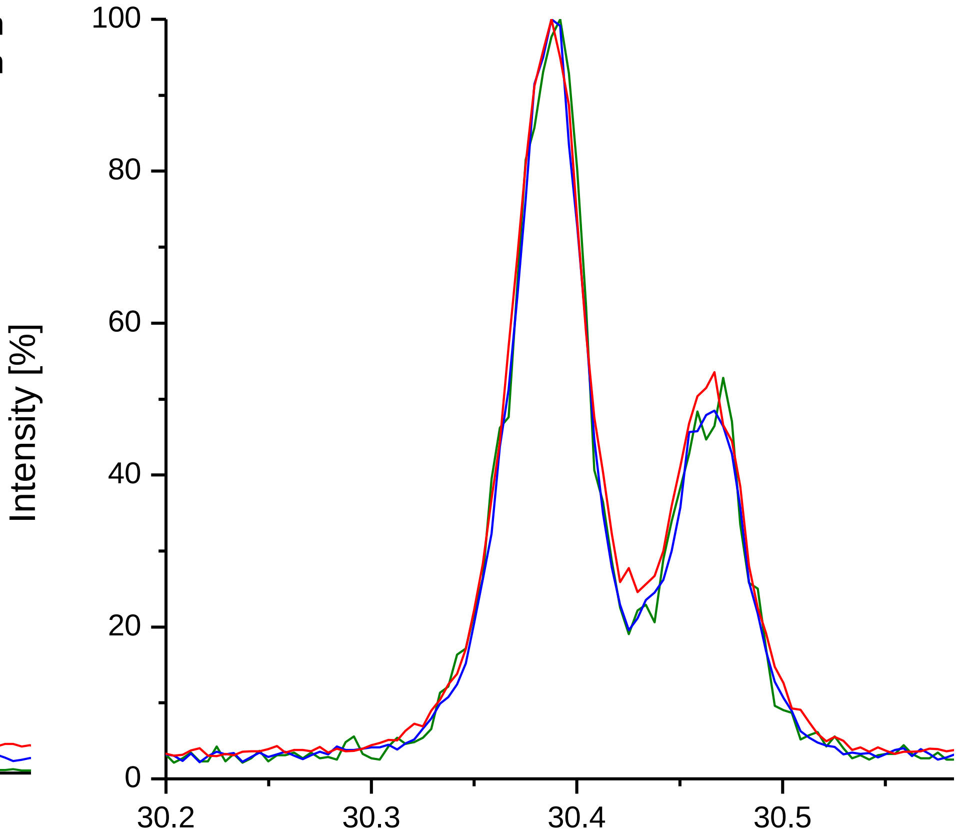
Correct! | Wrong! | Wrong!



Beam Mask

Beam Mask



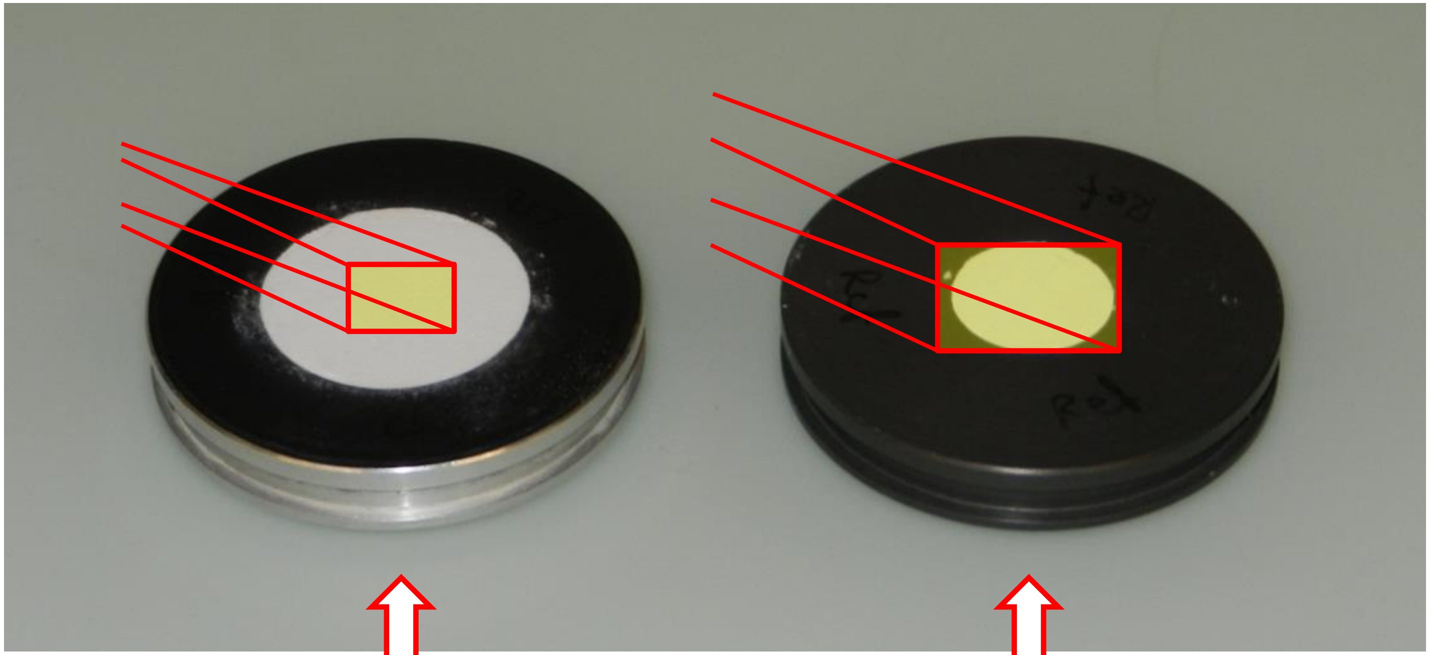


Soller Slits: 0.02 rad, Irradiated Length: 10mm

Optimum Settings: Divergence Slit

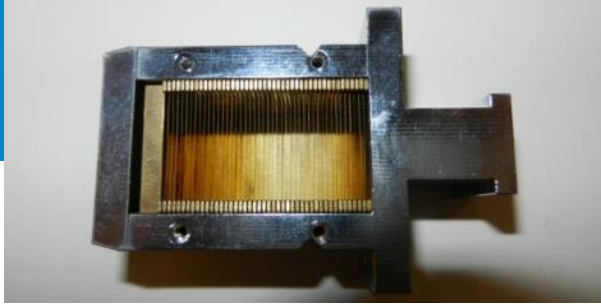
Using sample holders of various sizes?

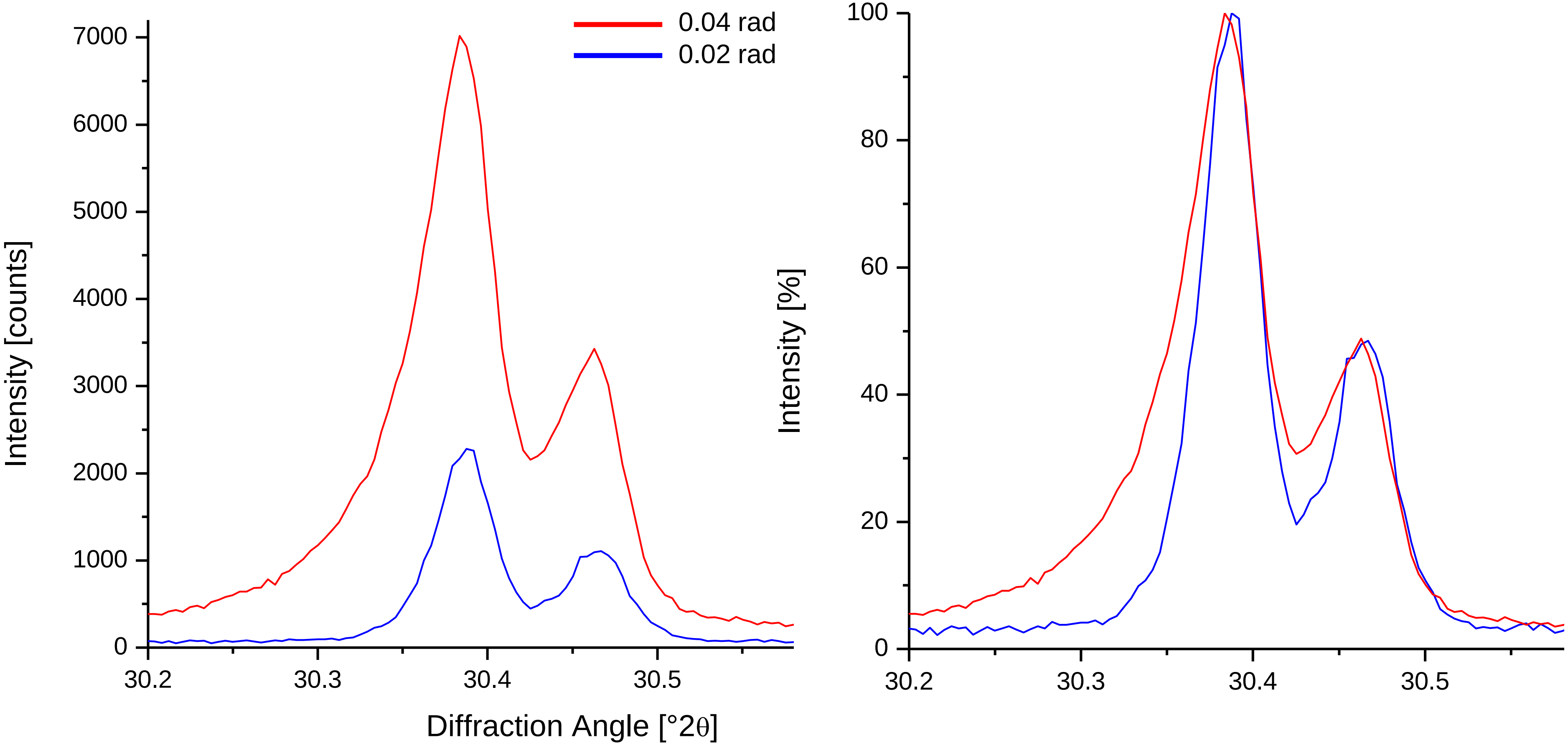
⇨ Match your Divergence Slit and Beam Mask!



Or else: Waste of intensity or Beam spill-over

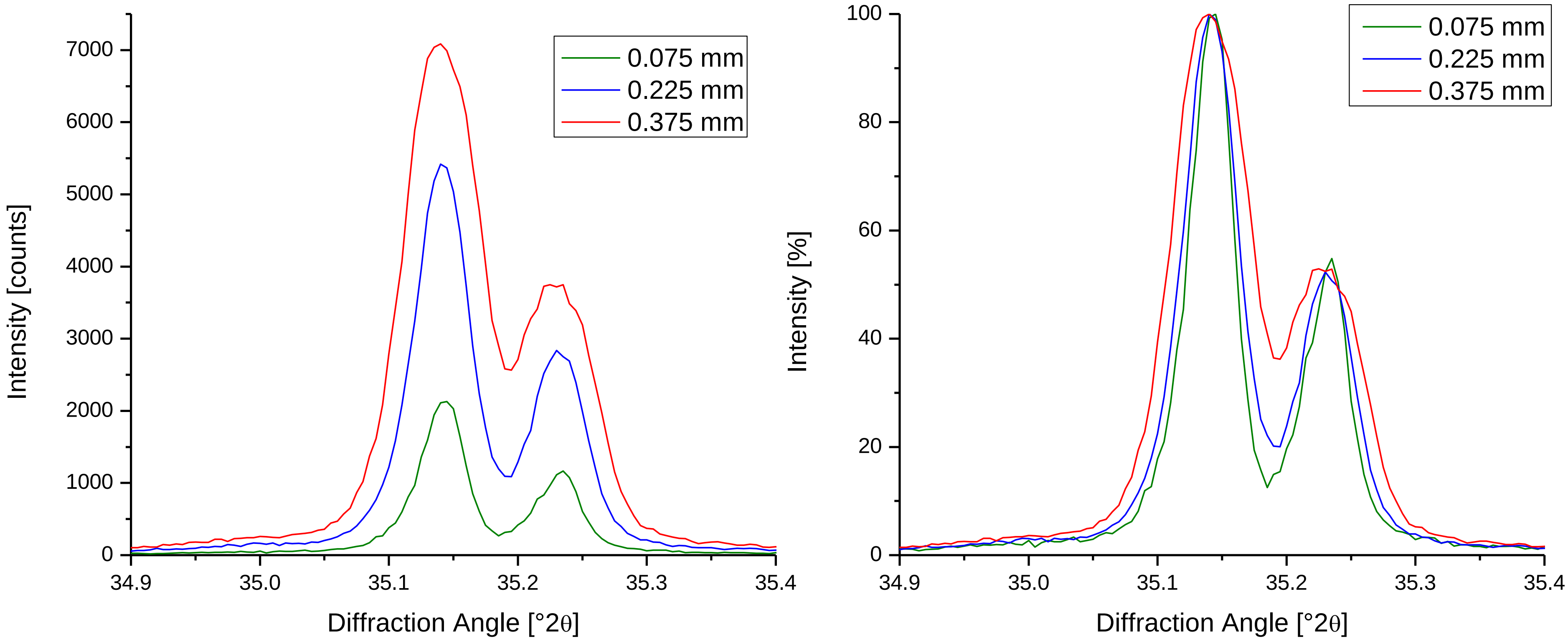
Soller Slits / Collimators





In primary & secondary beam, Beam Mask: 10mm, Irradiated Length: 10mm

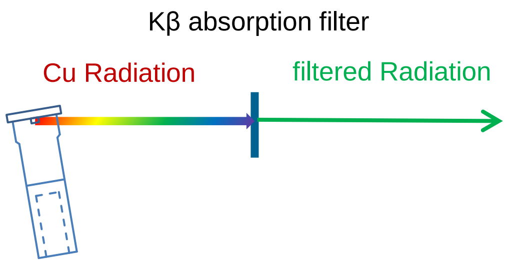
Receiving Slit / Detector Slit

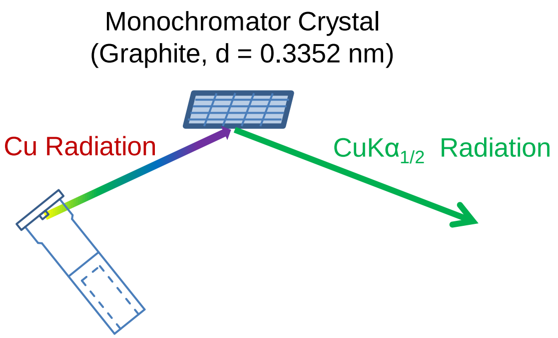


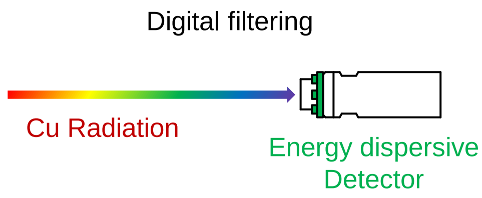
Al_2O_3, 15 mm irradiated length, 2.5° soller slit

Summary: Monochromators

Optical Element	Effect on Spectrum	Effect on Intensity
Kβ Filter	Reduces Kβ peaks	Moderate loss
Graphite Monochromator	Eliminates Kβ peaks Eliminates Fluorescence	Strong loss
Multi-bounce Monochromator	Eliminates Kβ and $K\alpha_2$ Eliminates Fluorescence	Massive loss (mostly used on Synchrotrons)
Energy dispersive Detector	Reduces Kβ peaks Eliminates Fluorescence	No loss



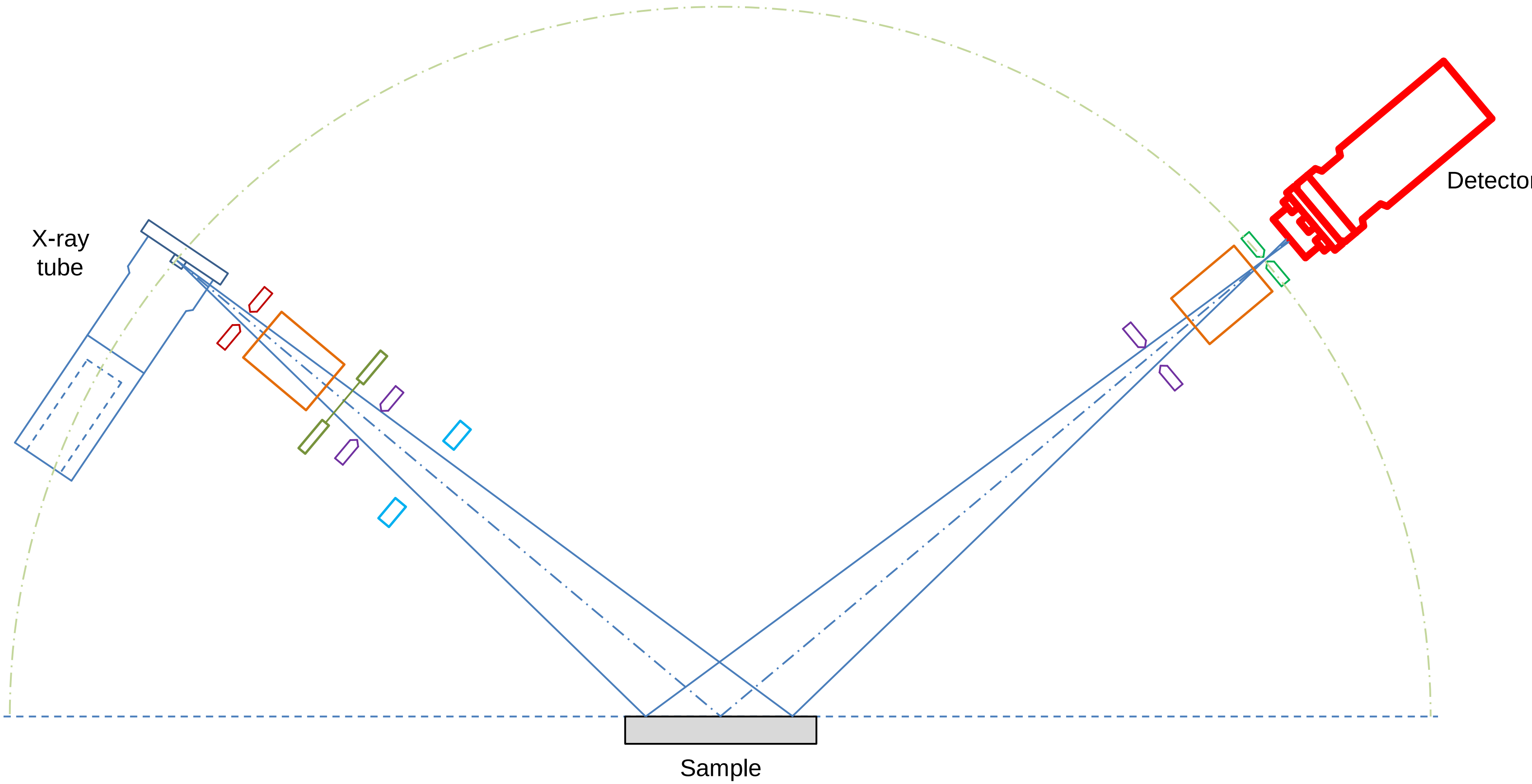




Summary: Optical Elements

Optical Element	Effect	Too Small	Too Large
Divergence Slit	Adjusts beam length on the sample	Loss of intensity	Beam spills over sample
Soller Slit	Reduces peak asymmetry	Loss of intensity, Better resolution	More asymmetry, Less resolution
Anti-Scatter Slit	Reduces background signal	Loss of intensity	High background
Beam Mask	Adjusts beam width on the sample	Loss of intensity	Beam spills over sample
Receiving Slit	Adjusts peak width / resolution	Loss of intensity Better resolution	Loss of resolution Higher intensity
Kβ Filter	Reduces Kβ peaks	-	-
Graphite Monochromator	Eliminates Kβ peaks	-	-

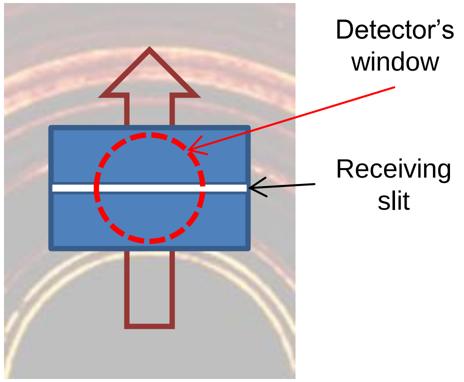
Bragg-Brentano Parafocusing Diffractometer



Detectors

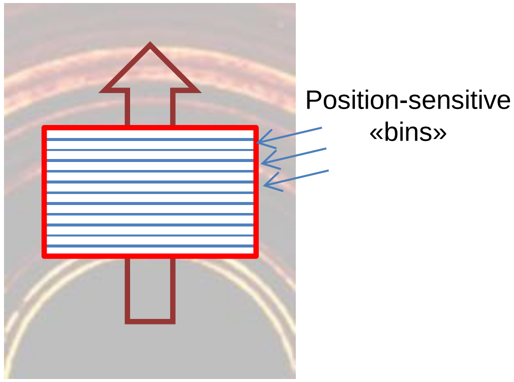
Detector Type

Point Detector (0D)



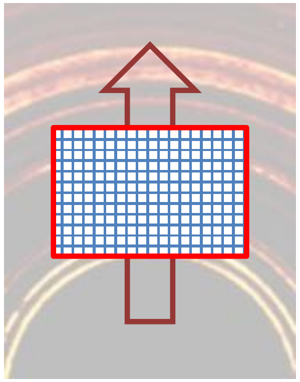
Receiving slit determines active height

Linear Detector (1D)



Linear array of solid state detectors

Area Detector (2D)



2D array of solid state detectors

Example

Point Detector (0D)	Linear Detector (1D)	Area Detector (2D)
Scintillation counter (various) SOL-XE (Bruker) XFlash (Bruker)	X'Celerator (PANalytical) PIXcel^{1D} (PANalytical) LynxEye (Bruker) LynxEye XE (Bruker) Våntec-1 (Bruker) D/teX Ultra (Rigaku)	PIXcel^{3D} (PANalytical) Våntec-500 (Bruker)

Key Features

Point Detector (0D)	Linear Detector (1D)	Area Detector (2D)
SOL-XE: Energy dispersive XFlash: Combines XRD + XRF	Fast LynxEye XE: Energy dispersive	Fast 2D image of Debye rings

Instruments

Lab	Instrument	Monochr.	Detector
RMS Foundation	Bruker D8	Energy dispersive Detector	1D LynxEye XE
Uni Bern	Panalytical X'Pert	Ni-Filter	1D X'Celerator
Uni Bern	Panalytical CubiX	Graphite	0D Scintillation Counter

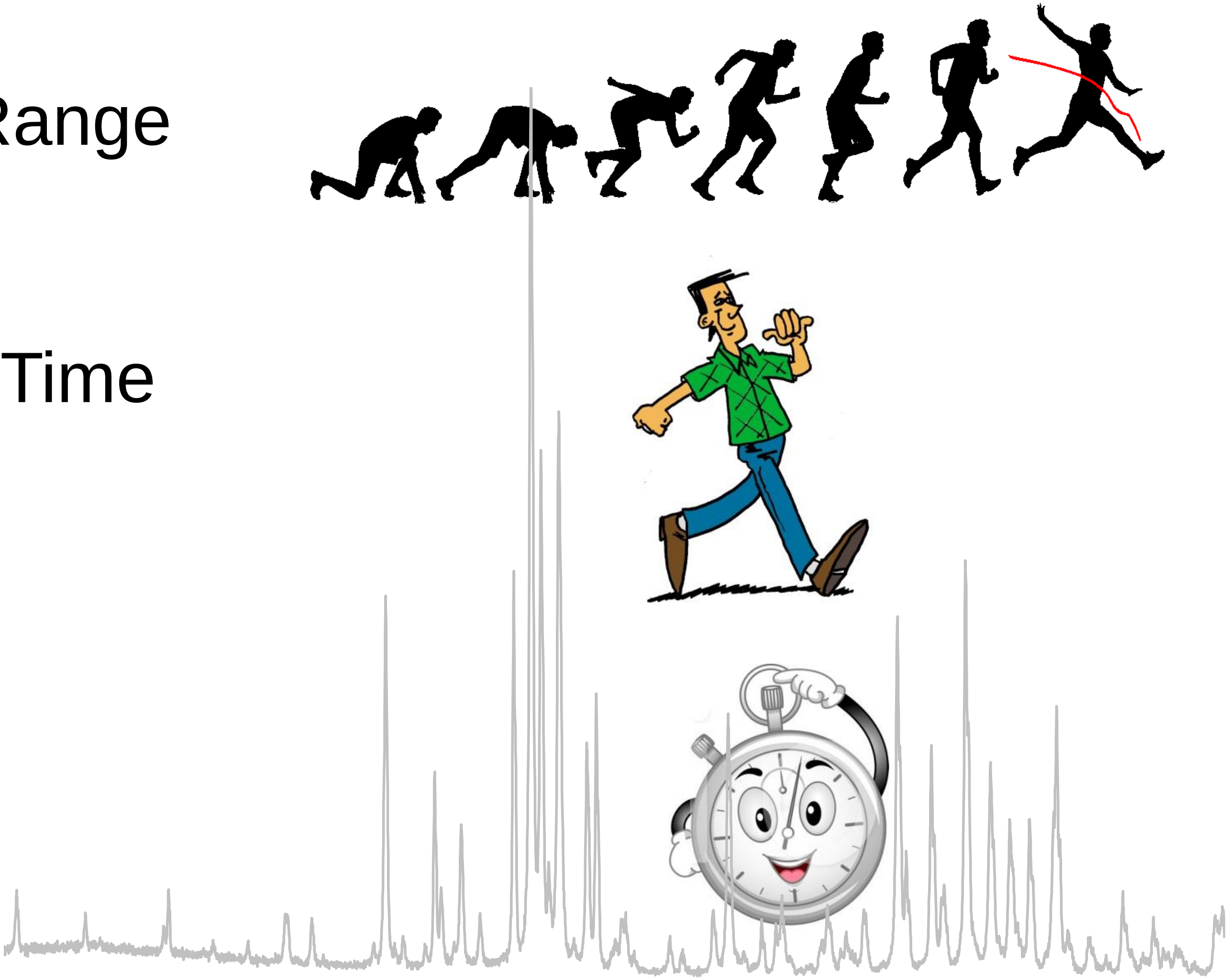
Bruker D8

Panalytical X'Pert

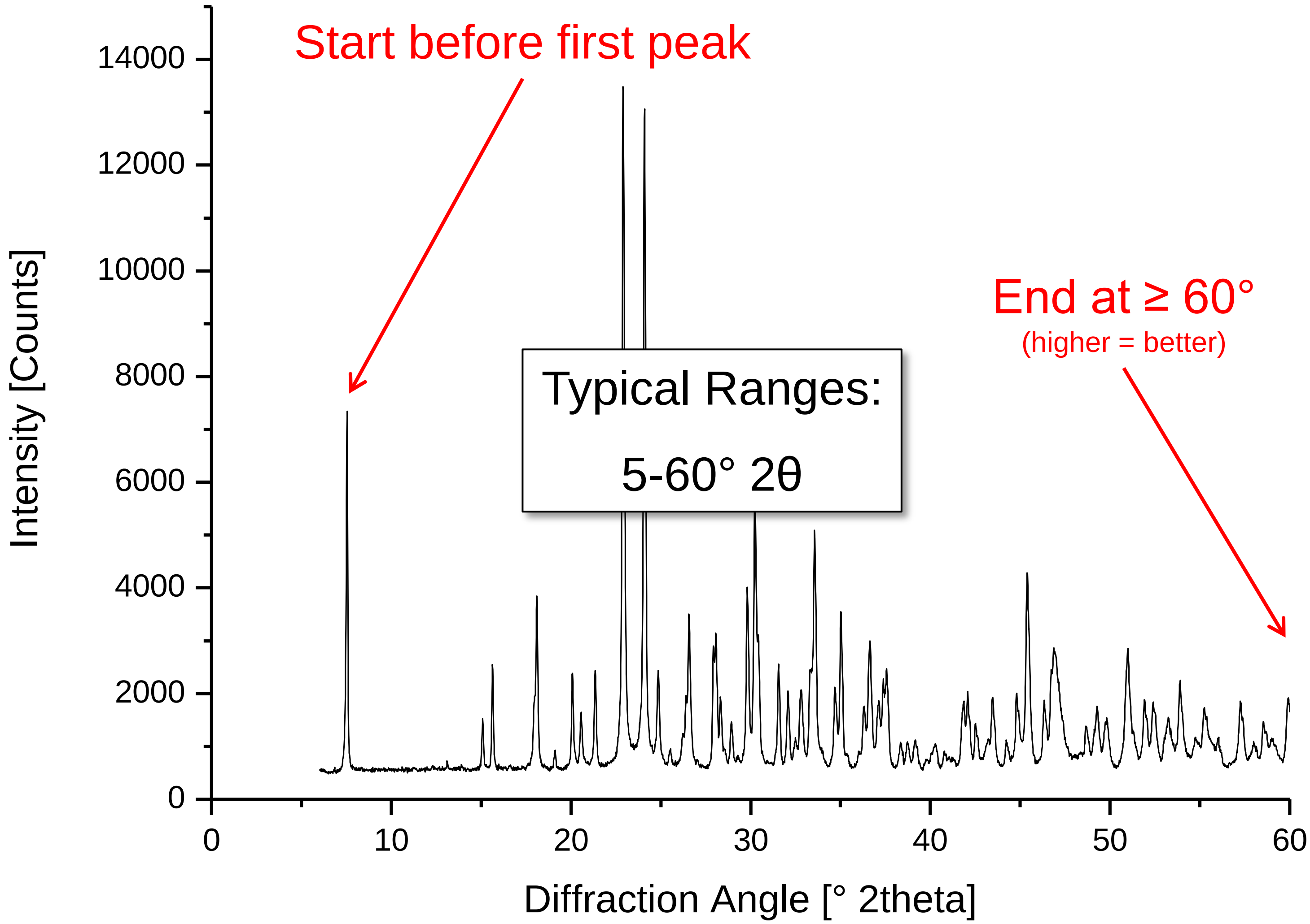
Panalytical CubiX

Measurement parameters

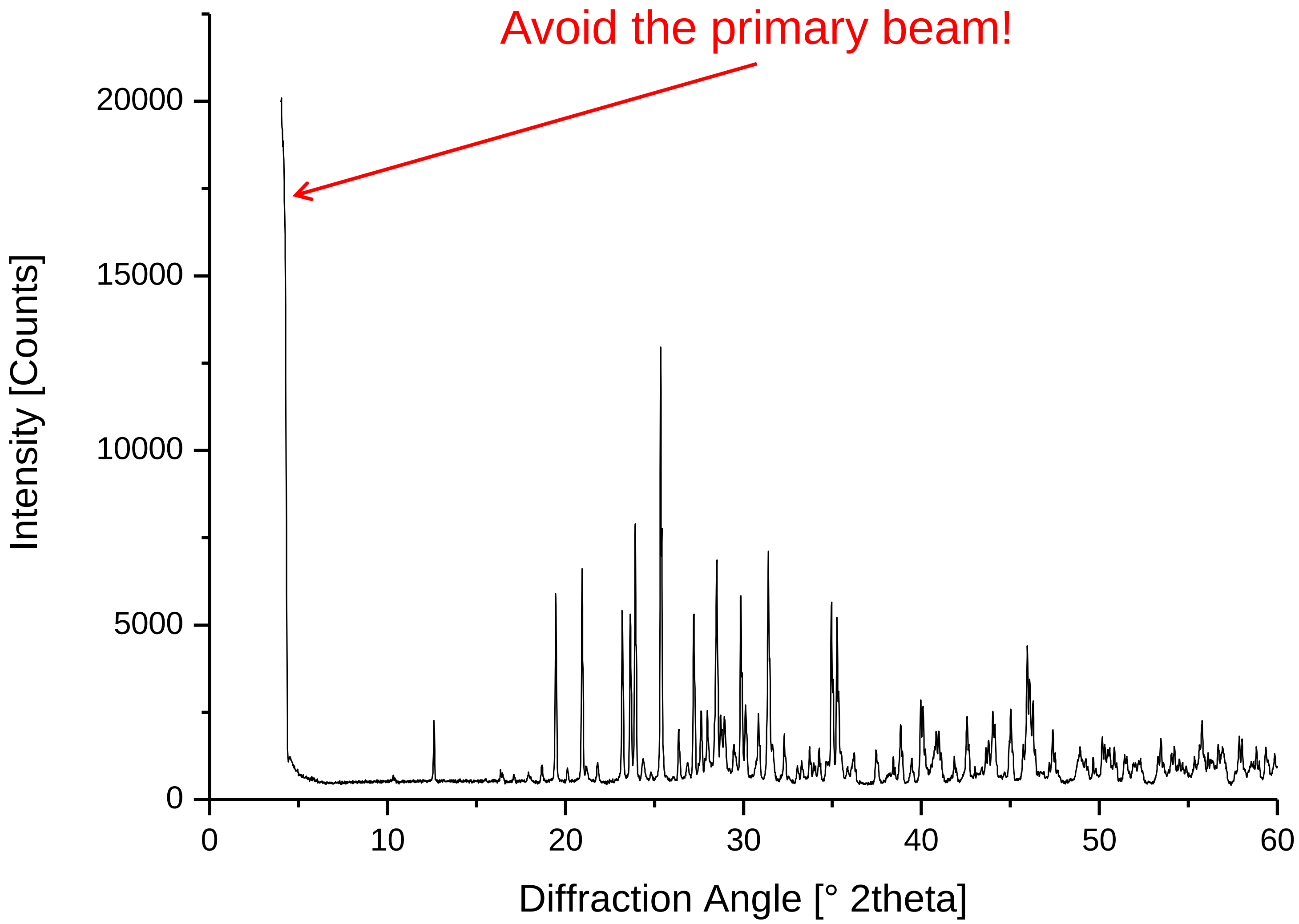
- Angular Range
- Step Size
- Counting Time



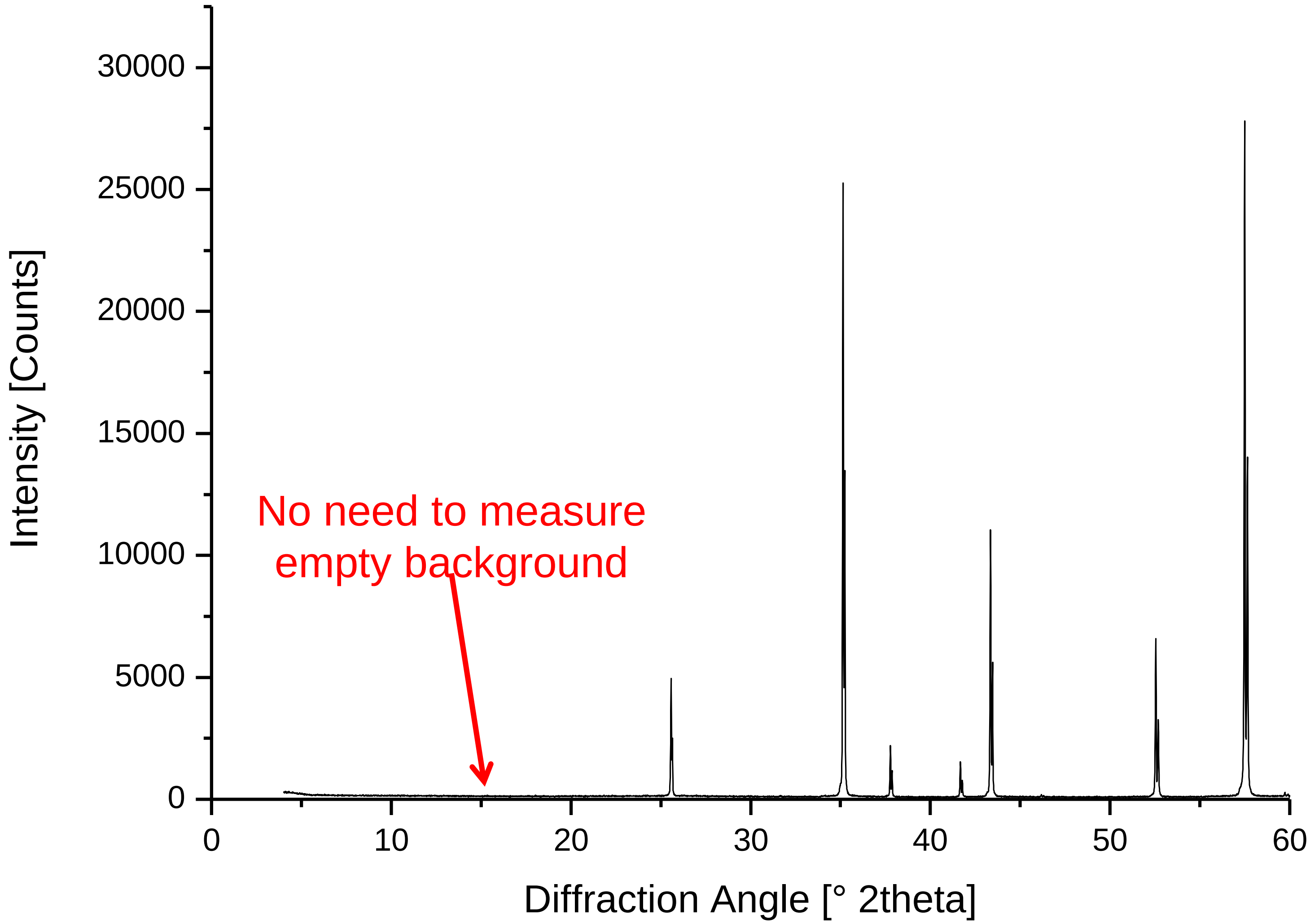
Angular Range



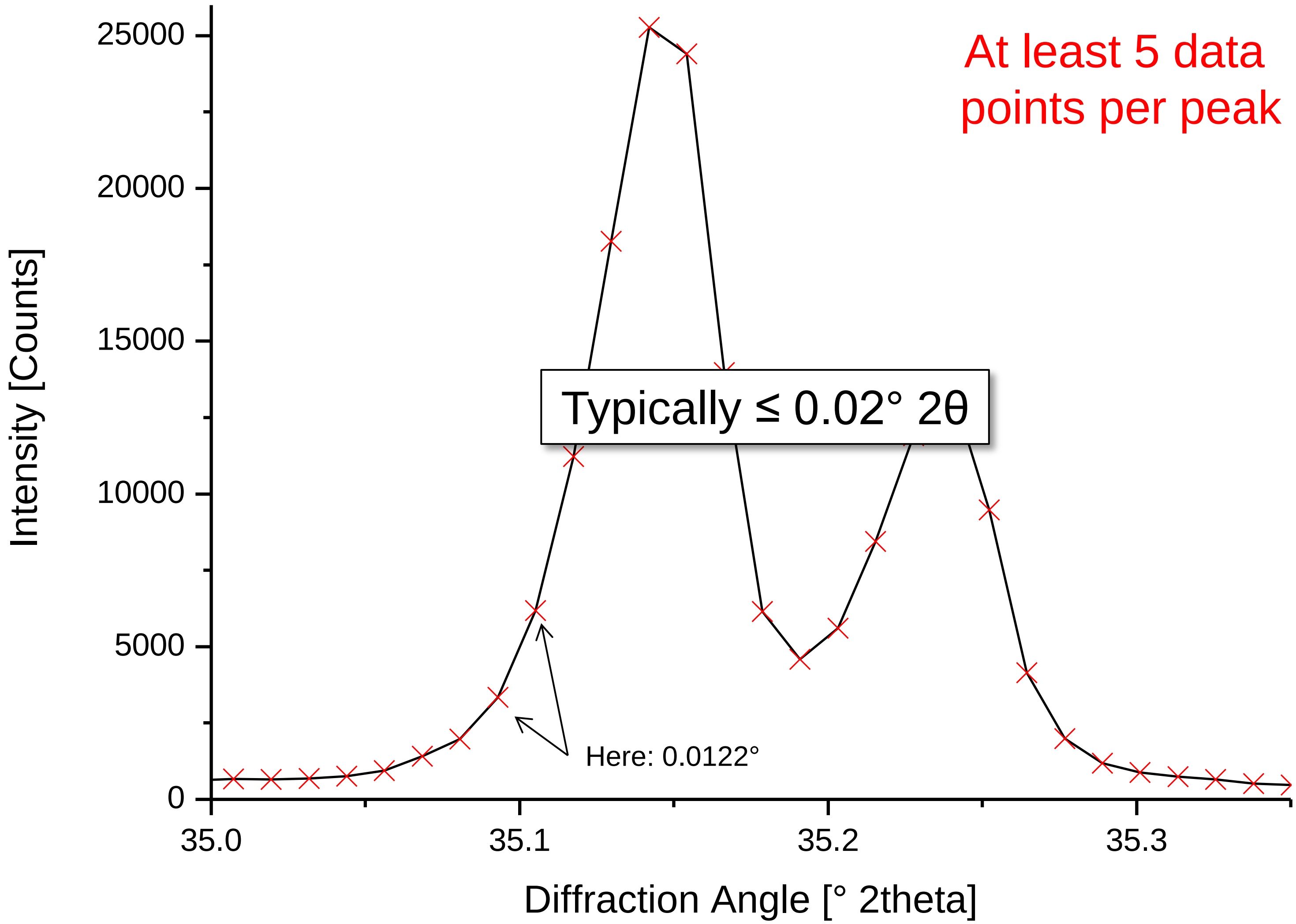
Angular Range



Angular Range



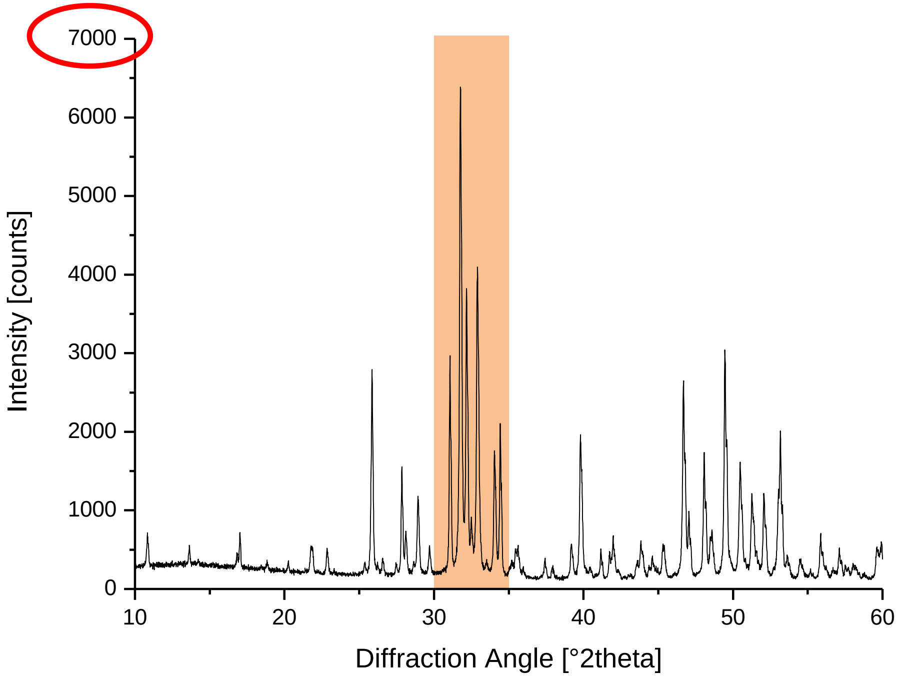
Step Size



Intensity [Counts]
25000
20000
15000
10000
5000
0
35.0
35.1
35.2
35.3
Diffraction Angle [° 2theta]
At least 5 data points per peak
Typically ≤ 0.02° 2θ
Here: 0.0122°

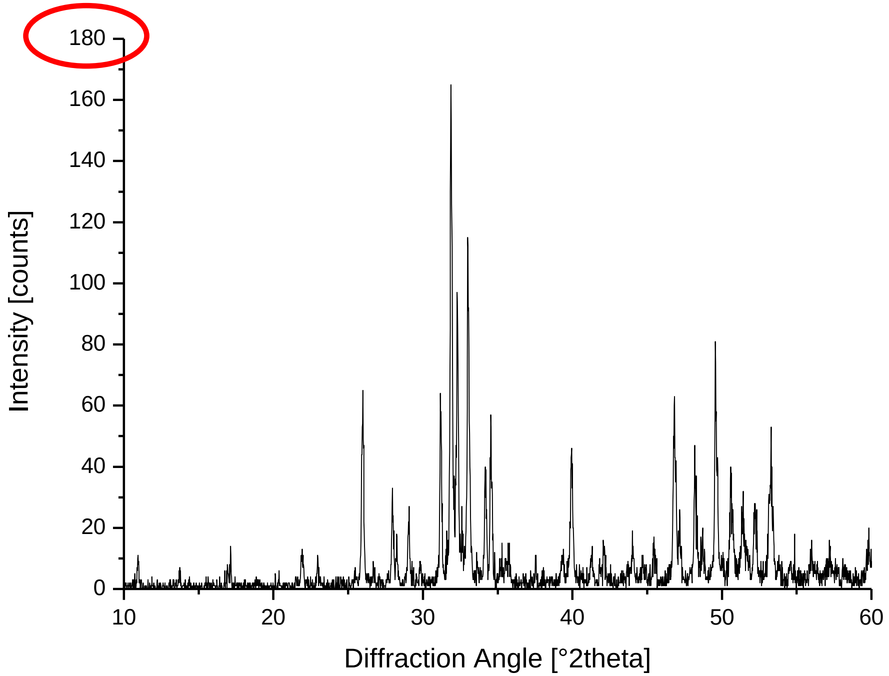
Time per Step

1D Energy dispersive Detector



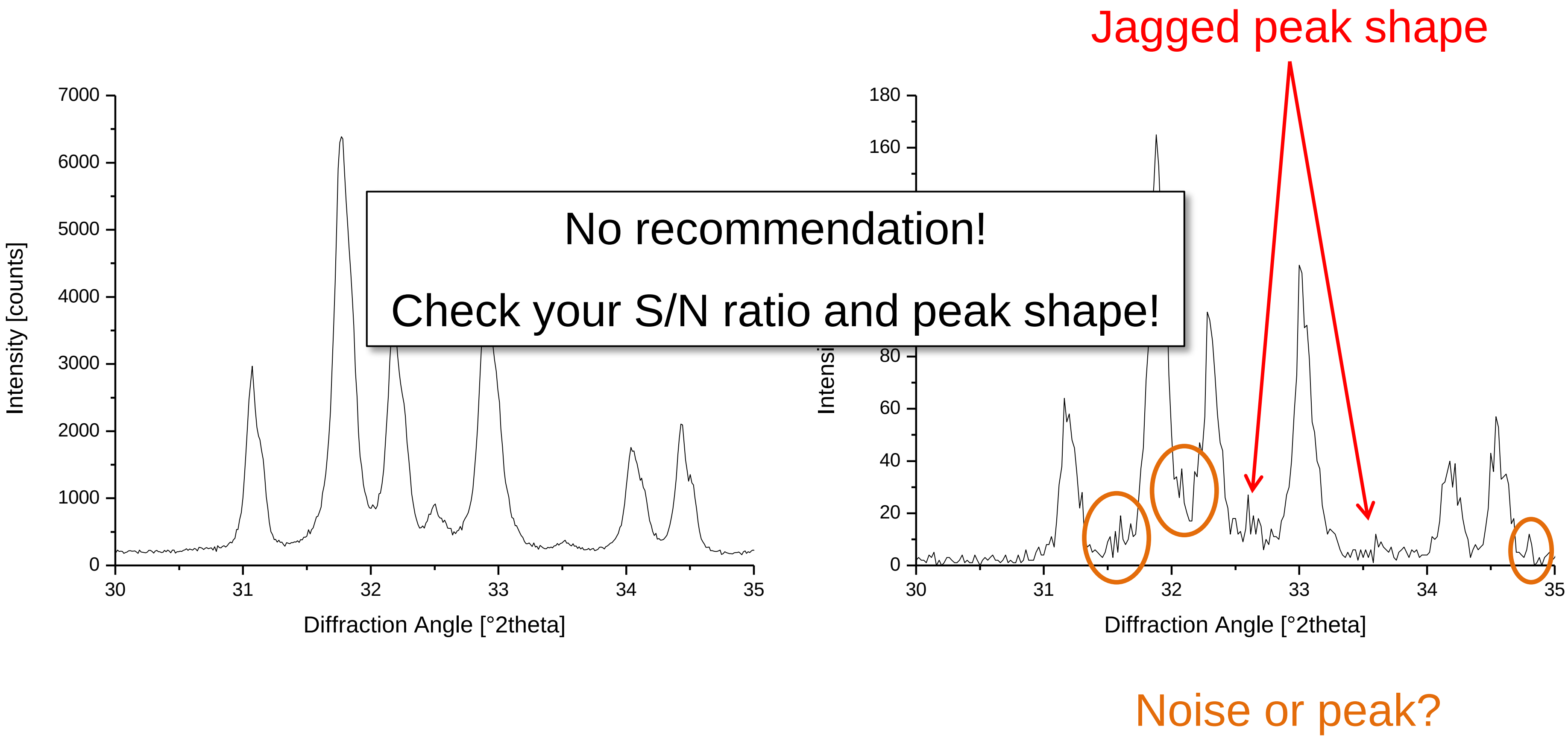
12.5 min

0D Detector



12.5 min

Examples

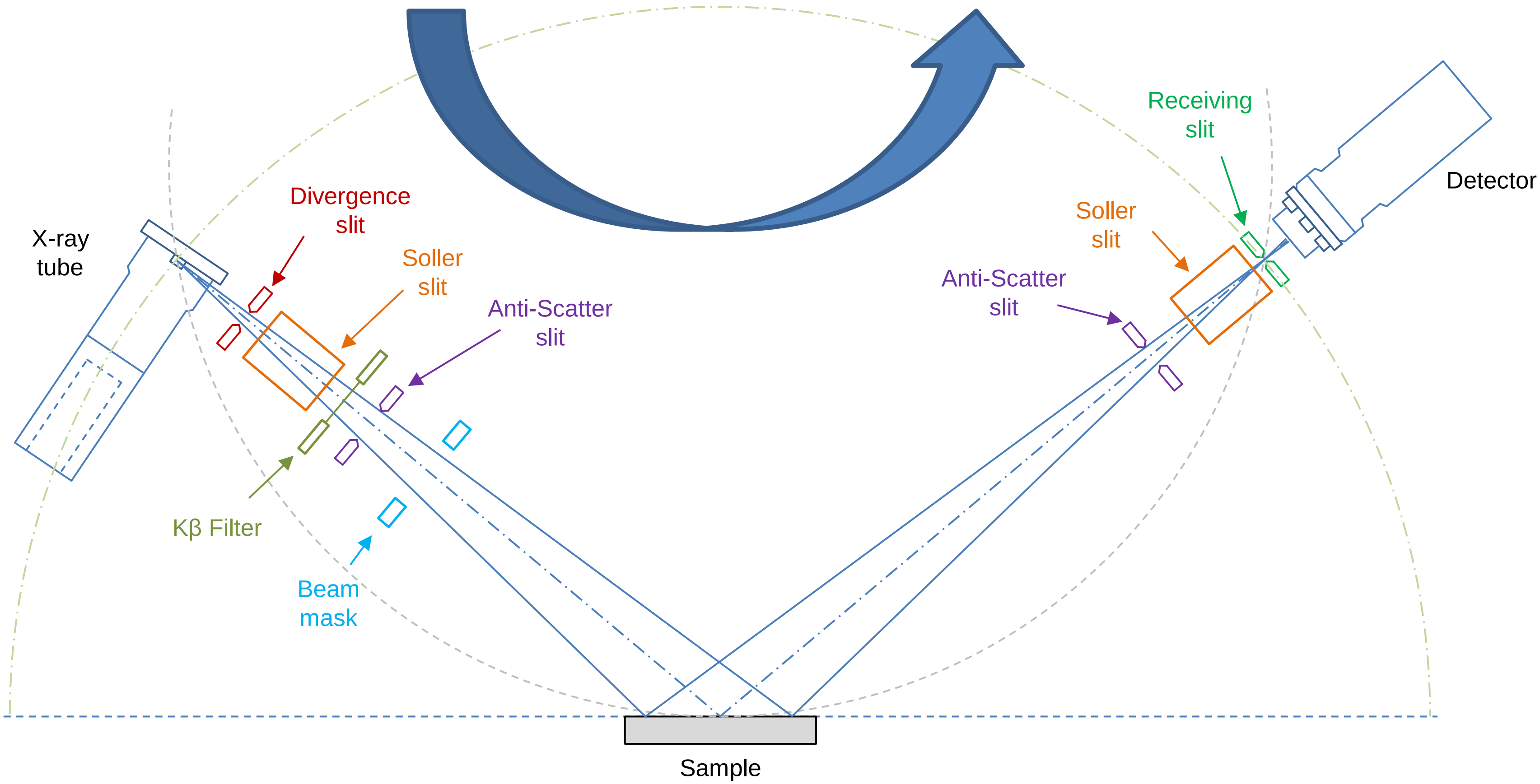


No recommendation!

Check your S/N ratio and peak shape!

Data Quality Checklist

Check beam paths Tube → Sample → Detector



Data Quality Checklist

For linear detector with Kβ filter

		Optical Element	Ideal setup
Incident beam path	✓	Divergence Slit	Automatic Max irr. length w/o beam overflow
Incident beam path	✓	Soller Slit	Installed Small opening
Incident beam path	✓	Mask	Installed (if available) Max irr. width w/o beam overflow
Incident beam path	✓	Anti-scatter slit	Identical to divergence slit
	✓	Sample	Spinning
Diffracted beam path	✓	Anti-scatter slit	Wide open
Diffracted beam path	✓	Soller slit	Installed Small opening
Diffracted beam path	✓	Additional slits	Wide open
Diffracted beam path	✓	Kβ filter	Installed