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QUB XRD Course

Focusing Optics

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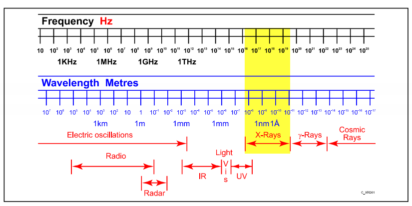
What are X-rays?



X-rays: electromagnetic radiation with a wavelength from 0.1 Å to 100 Å (0.01 nm to about 10 nm).

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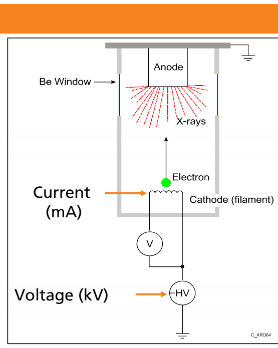
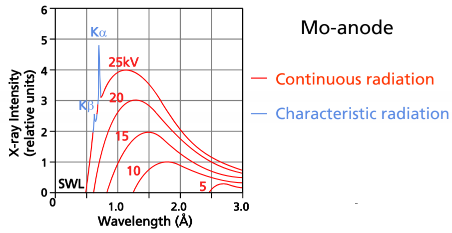
Generation of X-rays

- Electrons are emitted by a hot filament
- High voltage accelerates electrons
- Electrons bombard anode material at high speed
- Kinetic energy of electrons largely transferred into heat and X-ray radiation



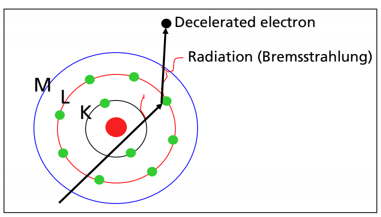
Generation of X-rays



Spectrum of X-rays depending on applied voltage and anode material. Characteristic radiation is used for experiments.

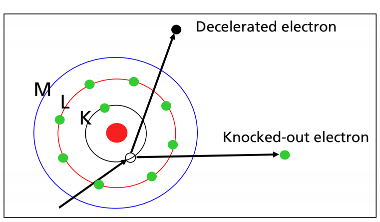
Generation of X-rays

<u>Continuous radiation:</u> caused by deceleration of electrons when passing the positively charged nuclei in the anode or when colliding with electrons of the anode atoms.



Generation of X-rays

<u>Characteristic radiation:</u> When an atom is bombarded with sufficiently high energy electrons ($E > E_c$) electrons can be knocked out from their shell.



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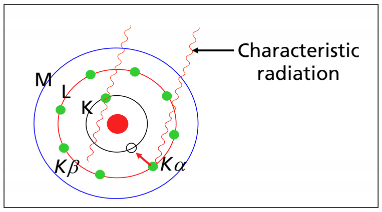
Generation of X-rays

Characteristic radiation: An electron from a higher shell takes the place of the knocked-out electron. The energy difference between both shells is released in the form of X-ray radiation of a specific wavelength.



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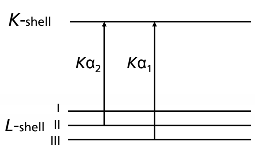
Generation of X-rays

$K\alpha_1$ and $K\alpha_2$ radiation:

$K\alpha$ radiation comprises two wavelengths: $K\alpha_1$ and $K\alpha_2$.

The wavelengths correspond to the transitions from the L-shell to the K-shell. The L-shell has three energy levels from which level I is empty.



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X-ray Operation Conditions

Intensity of characteristic radiation:

$I(\lambda) = K \cdot mA \cdot (kV - V_c)^n$

I = intensity
K = constant
V_c = critical voltage for characteristic radiation
n = 1.6

In general: kV = **3 to 5 x V_c**

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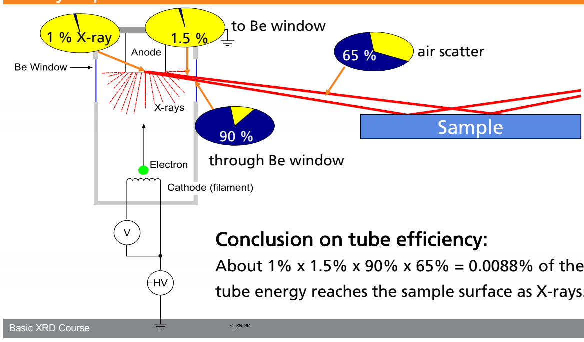
X-ray Operation Conditions



Conclusion on tube efficiency:

About 1% x 1.5% x 90% x 65% = 0.0088% of the tube energy reaches the sample surface as X-rays.

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X-ray Operation Conditions

Anode material = choice of wavelength

Element	Symbol	$K_\alpha\lambda$ [nm]	Application
Chromium	Cr	0.2291	large unit cells (clays, organic materials, zeolites), steel (residual stress)
Iron	Fe	0.1937	matrix effects of Fe and Cr
Cobalt	Co	0.1791	ferro materials
Copper	Cu	0.1542	standard tube
Molybdenum	Mo	0.0710	single crystal, small unit cells (metals)
Silver	Ag	0.0561	high absorbing materials
Tungsten	W	0.0211	Laue camera (continuum needed)

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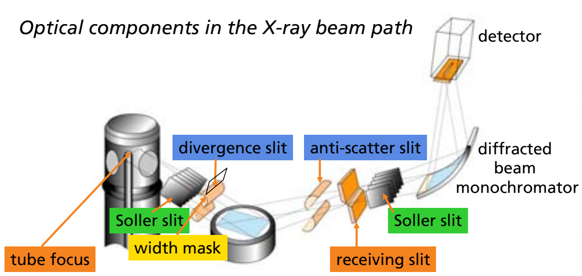
Classical Powder Diffractometer

Optical components in the X-ray beam path



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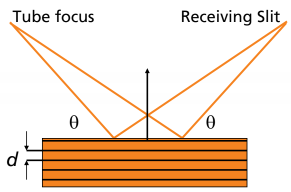
Classical Powder Diffractometer

- Sample surface normal bisects incident and diffracted beams
- Diffracted beams focus at the same distance as the tube focus in receiving slit
- Optimal resolution



$$\lambda = 2\,d\,\sin(\theta)$$

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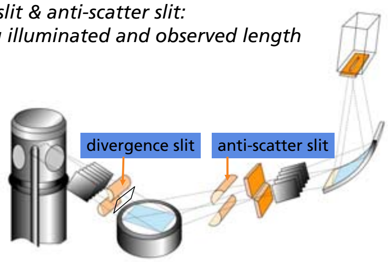
Divergence Slit & Anti-scatter Slit

Divergence slit & anti-scatter slit: determining illuminated and observed length



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Divergence Slit & Anti-scatter Slit

- Divergence slit determines the irradiated length on the sample
  - *major effect on intensities*
  - *minor effect on resolution*
- Anti-scatter slit determines observed length on the sample
  - *used to reduce background*
  - *major effect on the peak-to-background ratio*
  - *ideally the observed area equals the illuminated area*

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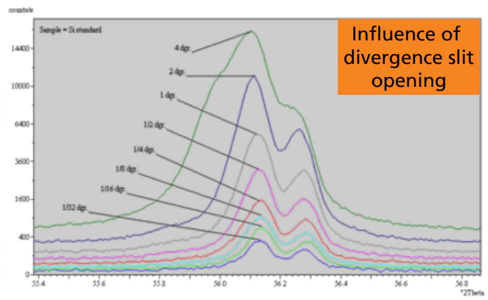
Divergence Slit & Anti-scatter Slit



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Divergence Slit & Anti-scatter Slit

- The size of the divergence slit depends on the angular range (starting angle) and on the size of the sample.
- A smaller divergence slit gives a smaller illumination on the sample and less diffracted intensity.

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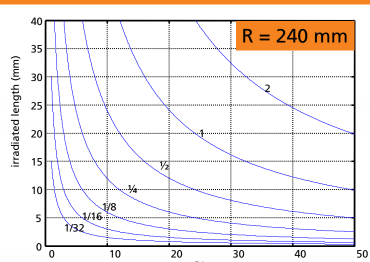
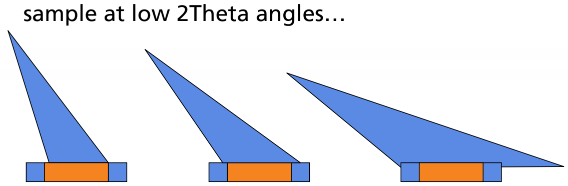
Divergence Slit & Anti-scatter Slit



Divergence and irradiated length

If You Want to Gain Intensity….

- Make sure that you never over-irradiate the sample at low 2Theta angles…



… it will give a very high background!

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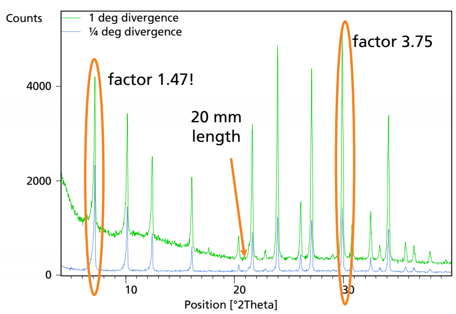
Divergence Slit Size and Peak Intensities



Hint:
you can observe the irradiated length on the status bar of the X'Pert Data Collector!

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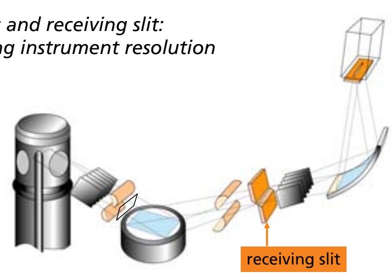
Receiving Slit

Tube focus and receiving slit: determining instrument resolution



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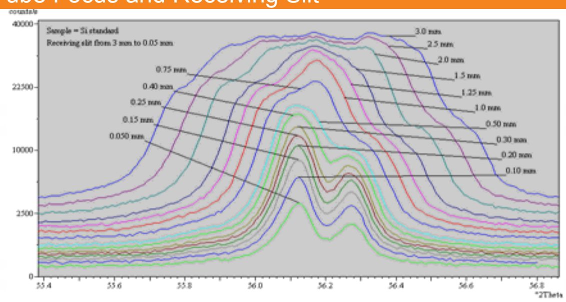
Tube Focus and Receiving Slit



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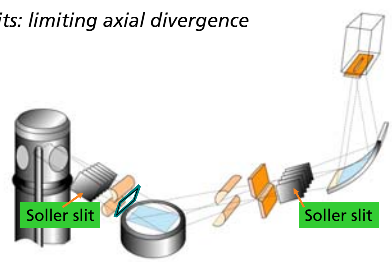
Soller Slits

Soller slits: limiting axial divergence



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Soller Slits

- Soller slits consist of large numbers of parallel plates in the plane of diffraction.
- Soller slits limit the spread of the incident and diffracted X-ray beam out of the plane of diffraction: 0.01, 0.02, 0.04 and 0.08 rad.
  - *large effect on intensities*
  - *moderate effect on resolution (low/high 2θ)*
- It is good practice to place similar Soller slits in the incident and diffracted beam.

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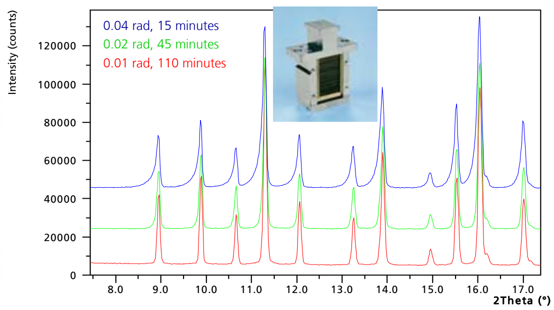
Instrument geometry influence - Sollers



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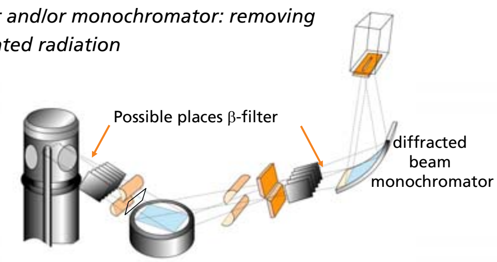
β-filter & Monochromator

β-filter and/or monochromator: removing unwanted radiation



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β-filter & Monochromator

- The β-filter and the diffracted beam monochromator remove unwanted wavelengths like the Kβ-line and continuous radiation.
- The β-filter selectively absorbs radiation.
- Monochromators select radiation by means of diffraction.

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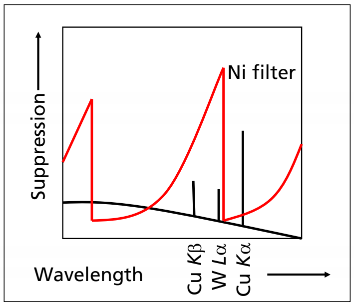
β-filter & Monochromator

Use filter material with absorption edge in between Kα and Kβ line.

For example:
Cu-radiation

Ni filter
- Intensity Cu Kα : 50 %
- Intensity Cu Kβ : 1%



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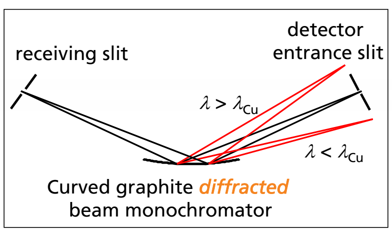
β-filter & Monochromator

Only X-ray waves with correct wavelength pass the entrance slit of the detector.

Others do not focus in the entrance slit of the detector.



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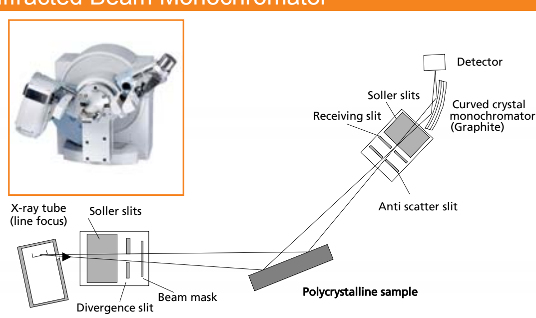
Diffracted Beam Monochromator



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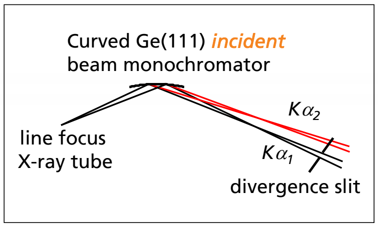
β-filter & Monochromator

The symmetrically cut curved Ge(111) monochromator in combination with the divergence slit filters out the $K\alpha_2$ component leaving a beam with $K\alpha_1$ radiation only.



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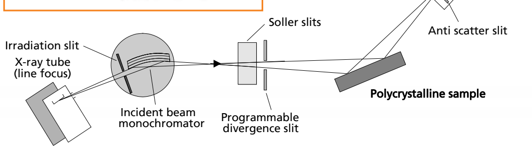
Incident Beam Monochromator



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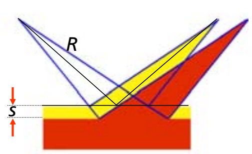
Sample Stages

Displacement error:

Sample stage and/or sample above or below diffraction plane: peaks are displaced from original position by

$$\Delta 2\theta = \frac{2s\cos\theta}{R}$$



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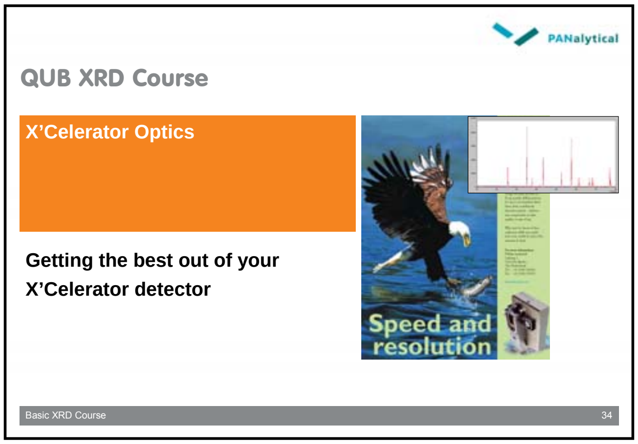
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X'Celerator Optics
Getting the best out of your
X'Celerator detector
Speed and resolution
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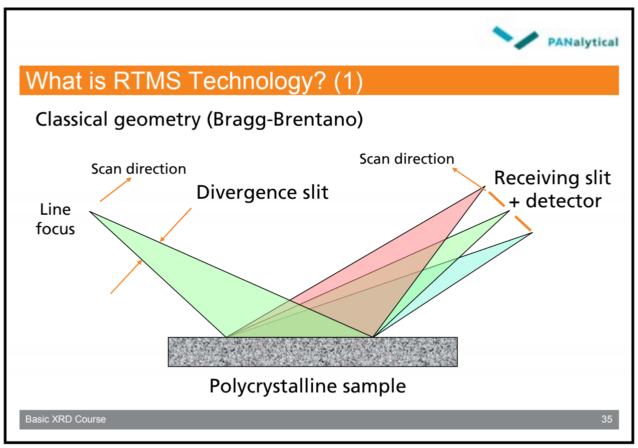
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What is RTMS Technology? (1)
Classical geometry (Bragg-Brentano)
Scan direction
Divergence slit
Scan direction
Receiving slit
+ detector
Line
focus
Polycrystalline sample
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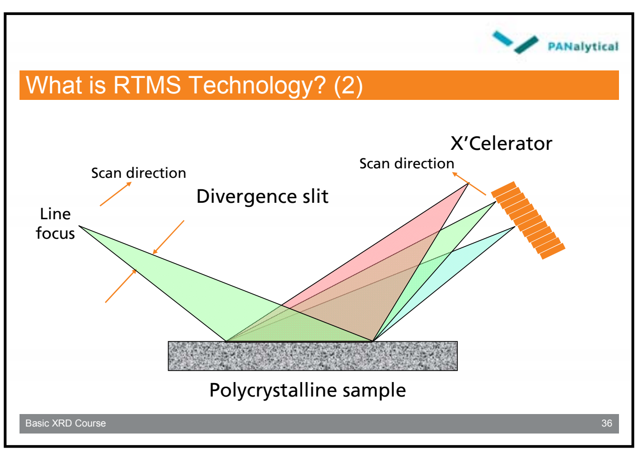
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What is RTMS Technology? (2)
X'Celerator
Scan direction
Scan direction
Divergence slit
Line
focus
Polycrystalline sample
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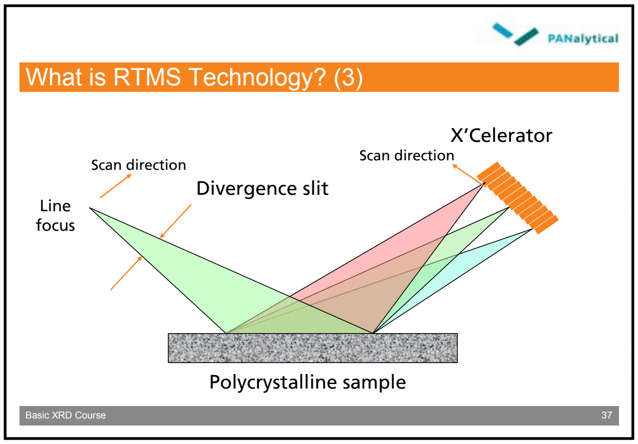
PANalytical
What is RTMS Technology? (3)
X'Celerator
Scan direction
Scan direction
Divergence slit
Line focus
Polycrystalline sample
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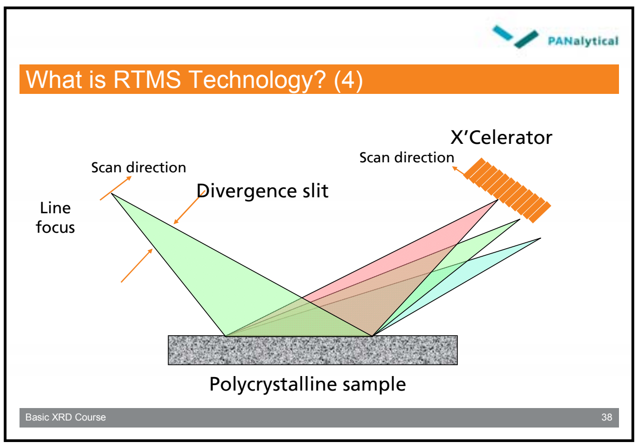
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What is RTMS Technology? (4)
X'Celerator
Scan direction
Scan direction
Divergence slit
Line focus
Polycrystalline sample
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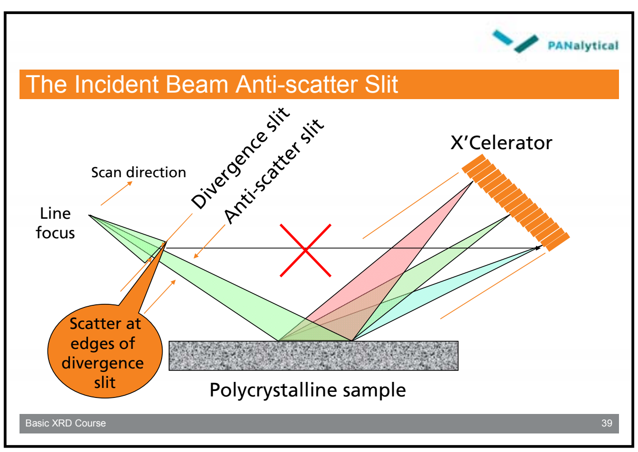
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The Incident Beam Anti-scatter Slit
Divergence slit
Anti-scatter slit
X'Celerator
Scan direction
Line focus
Scatter at edges of divergence slit
Polycrystalline sample
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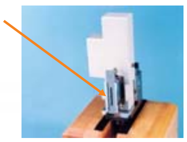
The Incident Beam Anti-scatter Slit

Divergence slit size	Anti-scatter slit size
1/32°	1/16°
1/16°	1/8°
1/8°	¼°
¼°	½°
½°	1°
1°	2°
2°	4°
4°	-



- So: 1° divergence slit ⇒ 2° inc. beam anti-scatter slit

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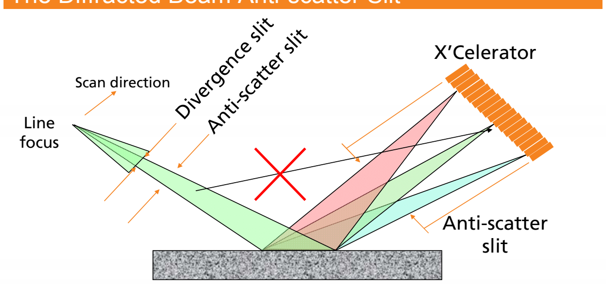
The Diffracted Beam Anti-scatter Slit



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The Programmable Anti-scatter Slit

- Two modes of operation:
  - *constant observed length*
  - *constant observed angle*
- Can be changed at will
- Actual opening is also a function of the active length of the X'Celerator: important when working at low angles!

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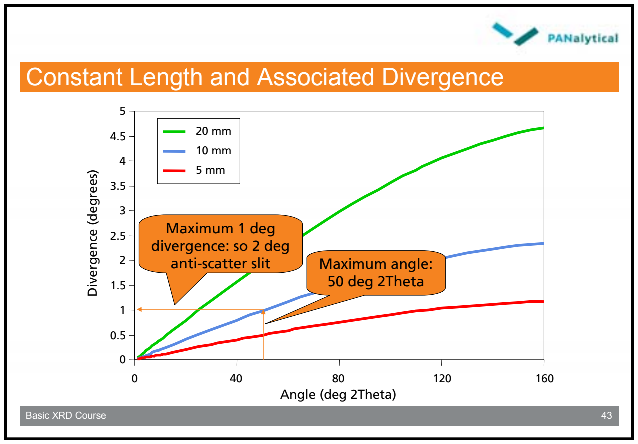
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Constant Length and Associated Divergence
20 mm
10 mm
5 mm
Maximum 1 deg divergence: so 2 deg anti-scatter slit
Maximum angle: 50 deg 2Theta
Divergence (degrees)
Angle (deg 2Theta)
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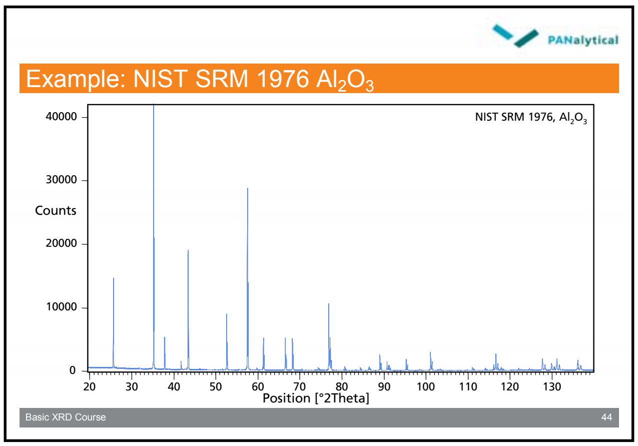
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Example: NIST SRM 1976 Al_2O_3
NIST SRM 1976, Al_2O_3
Counts
Position [°2Theta]
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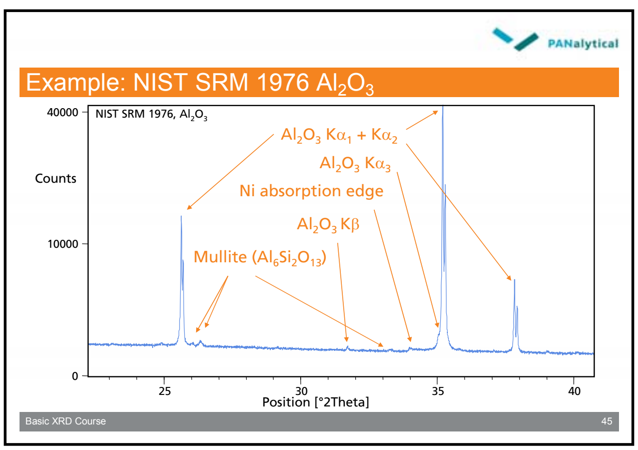
PANalytical
Example: NIST SRM 1976 Al_2O_3
NIST SRM 1976, Al_2O_3
Al_2O_3 $K\alpha_1 + K\alpha_2$
Al_2O_3 $K\alpha_3$
Ni absorption edge
Al_2O_3 $K\beta$
Mullite ($Al_6Si_2O_{13}$)
Counts
Position [°2Theta]
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Sample Fluorescence

- X-rays excite the atoms in the sample
- As a result of this, characteristic X-radiation is emitted
- If this emitted radiation is close to the Cu Kα, it is detected!

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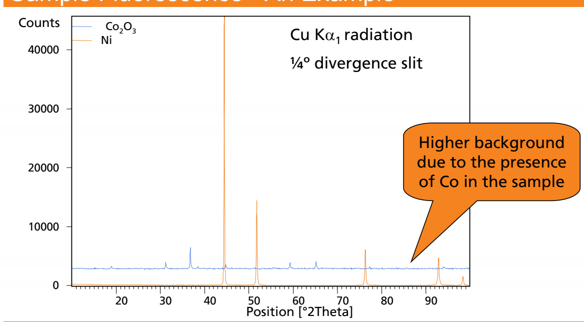
Sample Fluorescence - An Example



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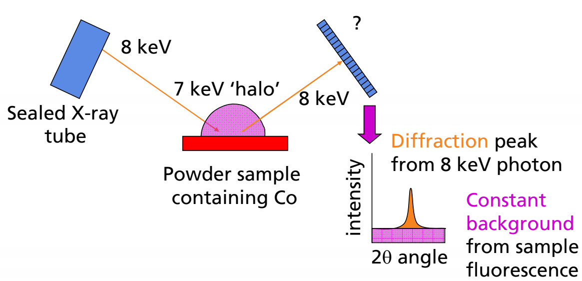
Sample Fluorescence (1)



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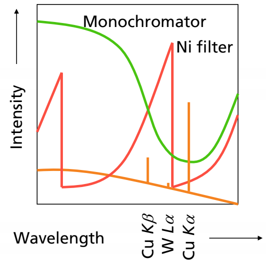
The Monochromator

- Crystal which acts as band pass filter



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Rules of Thumb - Monochromators

- Monochromators help to reduce the range of problematic elements

Cu, inc. beam mono	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As
Cu, dif. beam mono	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As
Co, inc. beam mono	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As
Co, dif. beam mono	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As

low — high

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Rules of Thumb - Beta Filters

- β-filters: more background, but least reduction of Kα
- For best results, the β-filter should be placed in the diffracted beam
  - *exceptions: Ni with Cu radiation, Fe with Co(!)*
- For not-too-complex patterns, Mo radiation is a valuable alternative

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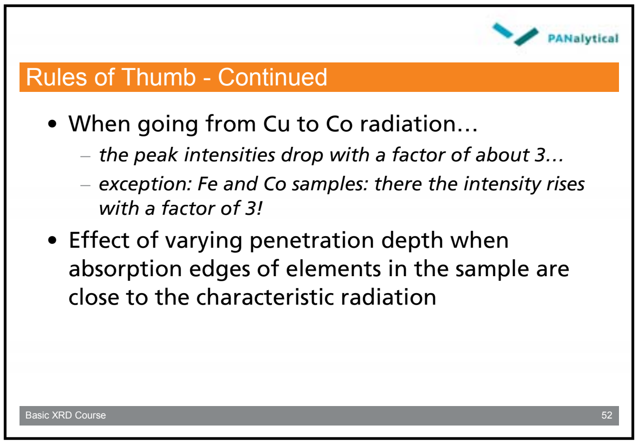
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Rules of Thumb - Continued
When going from Cu to Co radiation…
the peak intensities drop with a factor of about 3…
exception: Fe and Co samples: there the intensity rises with a factor of 3!
Effect of varying penetration depth when absorption edges of elements in the sample are close to the characteristic radiation
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