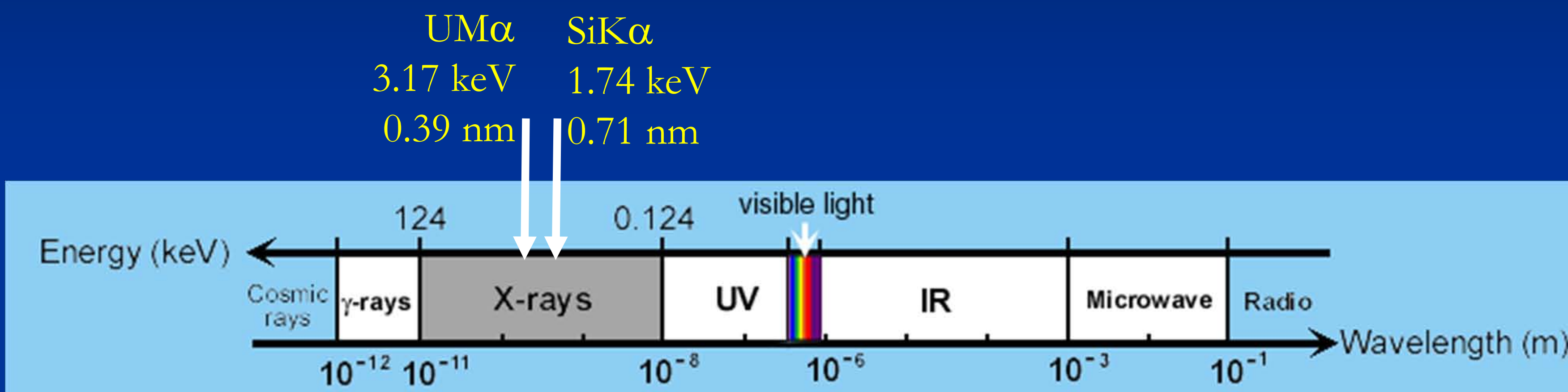
Understanding X-rays: The electromagnetic spectrum



$E = h\nu$ $\qquad$ h : Planck's constant = **$6.626\text{x}10^{-34}$** Joule.sec

= **$6.626\text{x}10^{-34}/1.6021\text{x}10^{-16}$** keV.sec

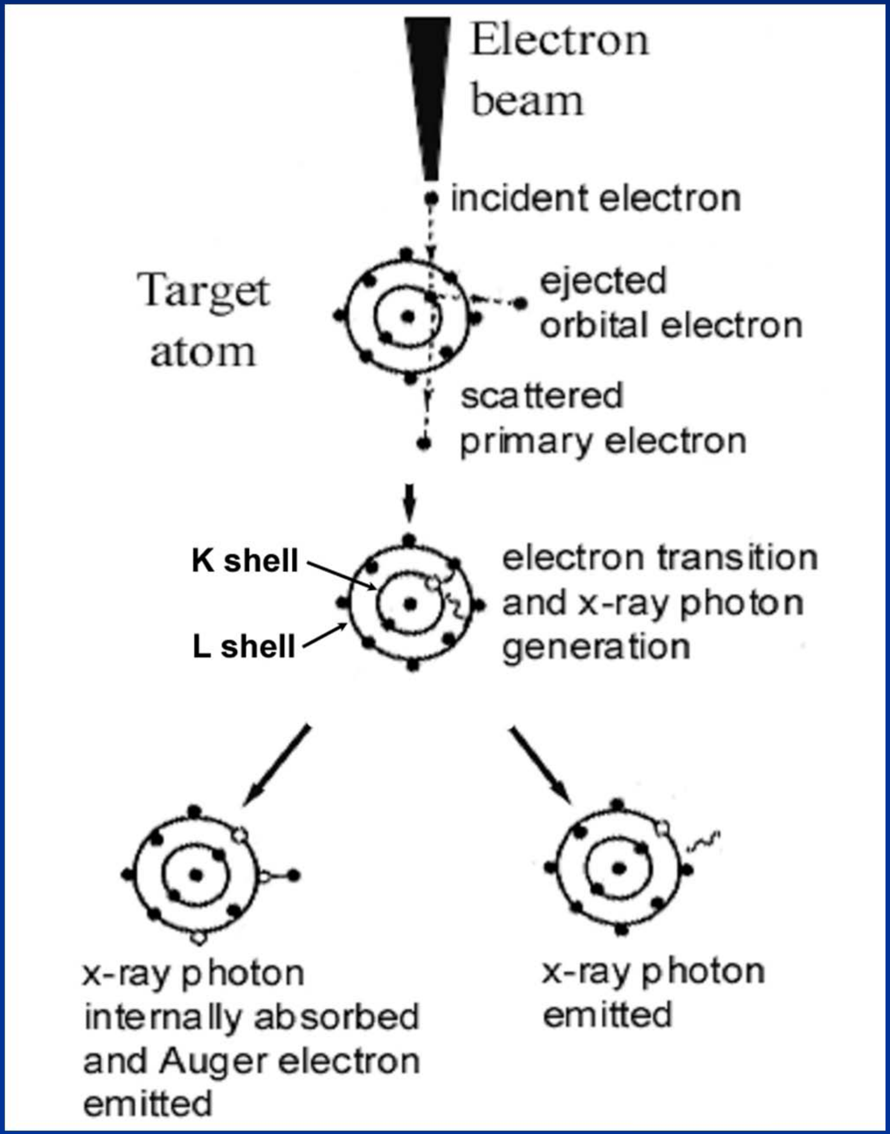
$= h\,(c/\lambda)$ $\qquad$ ν : frequency

c : speed of light in vacuum = **$2.99793\text{x}10^{17}$** nm/sec

λ : wavelength

λ (nm) = hc/E = **1.2398**/E (keV)

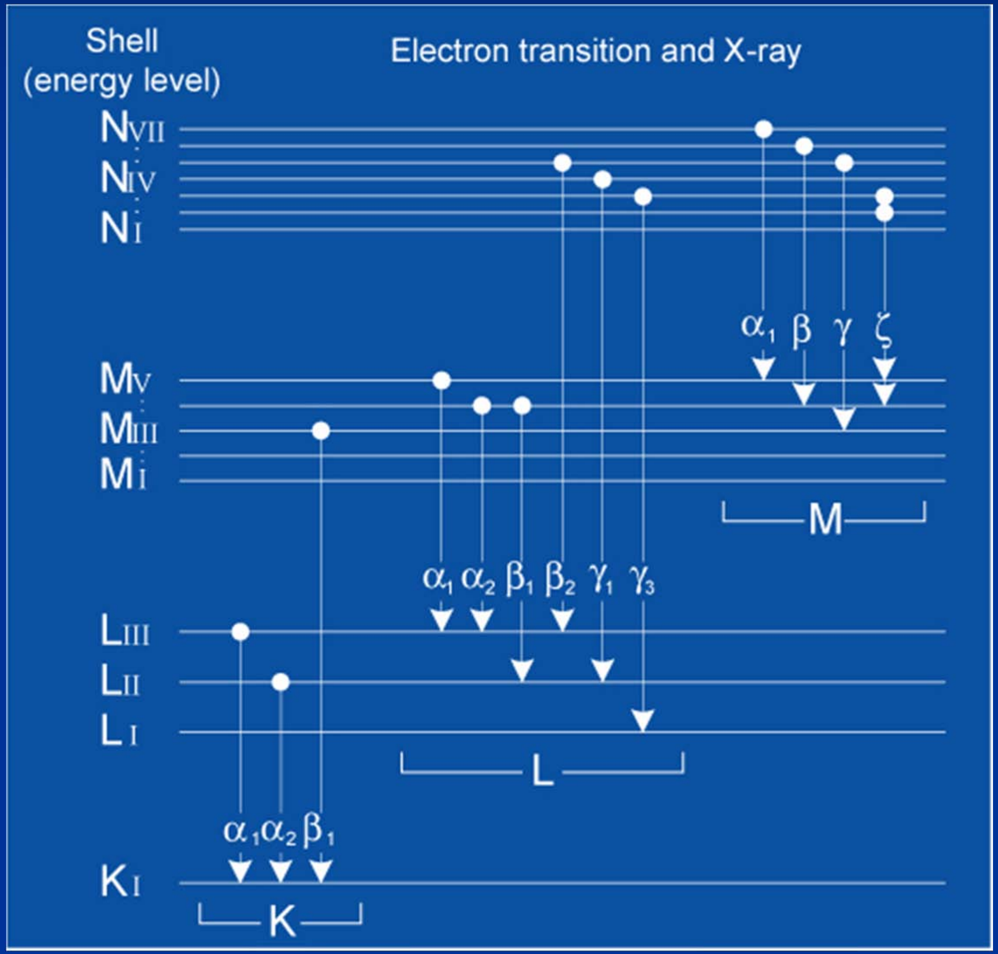
Characteristic X-ray generation



Inner-shell ionization

X-ray nomenclature

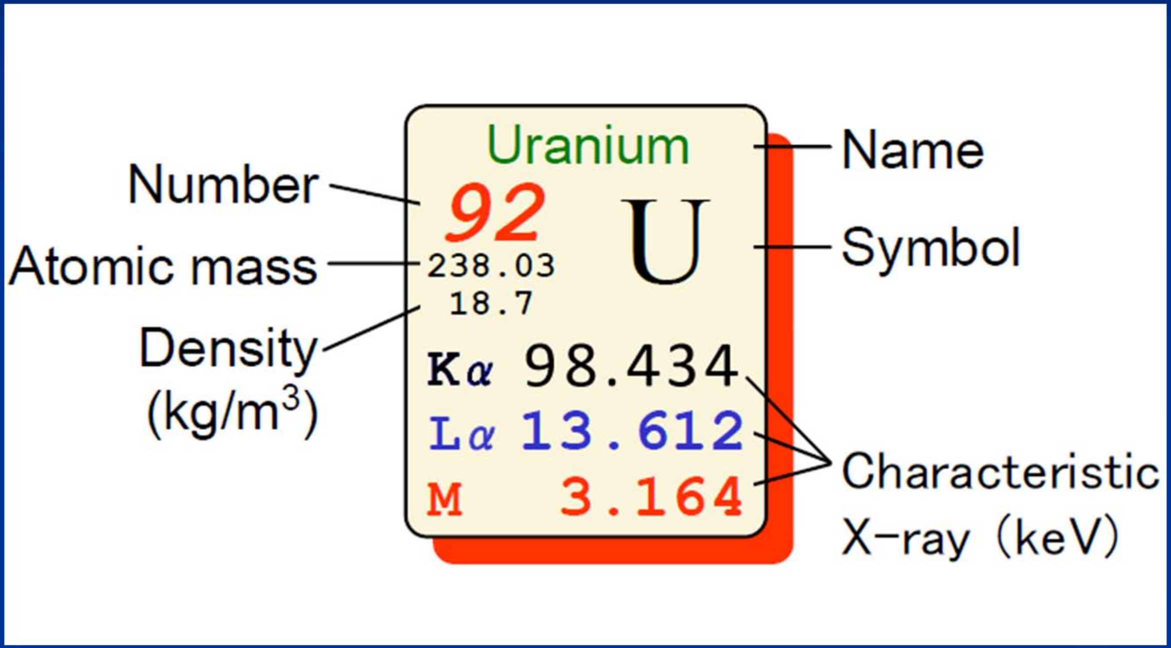
X-ray	electron transition
Kα	L to K-shell
Kβ	M to K-shell
Lα	M to L-shell
Mα	N to M-shell



X-ray energies

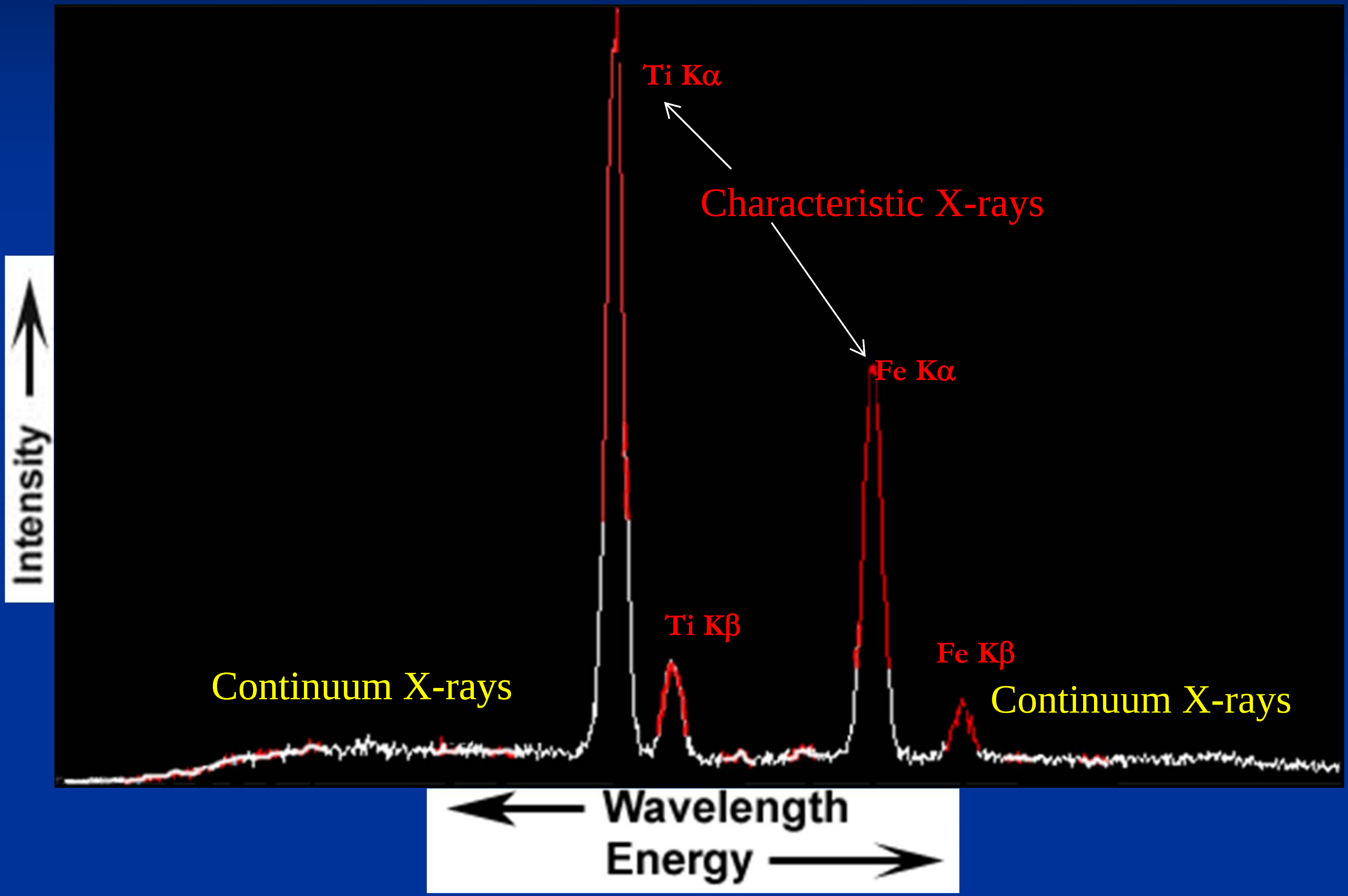
X-ray	electron transition	X-ray energy
$K\alpha$	L_{II+III} to K_I	$E_{K\alpha} = E_{c(K_I)} - E_{c(L_{II+III})}$
$K\beta$	M_{III} to K_I	$E_{K\beta} = E_{c(K_I)} - E_{c(M_{III})}$
$L\alpha$	M_{IV+V} to L_{III}	$E_{L\alpha} = E_{c(L_{III})} - E_{c(M_{IV+V})}$
$M\alpha$	N_{VII} to M_V	$E_{M\alpha} = E_{c(M_V)} - E_{c(N_{VII})}$

X-ray energy and Critical excitation energy



$$E_{c(K)} = E_{K\alpha} + E_{c(L)}$$
$$= E_{K\alpha} + E_{L\alpha} + E_{c(M)}$$
$$= E_{K\alpha} + E_{L\alpha} + E_{M\alpha} + E_{c(N)}$$
$$\approx E_{K\alpha} + E_{L\alpha} + E_{M\alpha}$$

The X-ray spectrum



Continuum X-rays are generated by deceleration of beam electrons in the Coulombic field of outer shells of target atoms. Maximum energy = electron beam energy

Condition for Ionization: Overvoltage

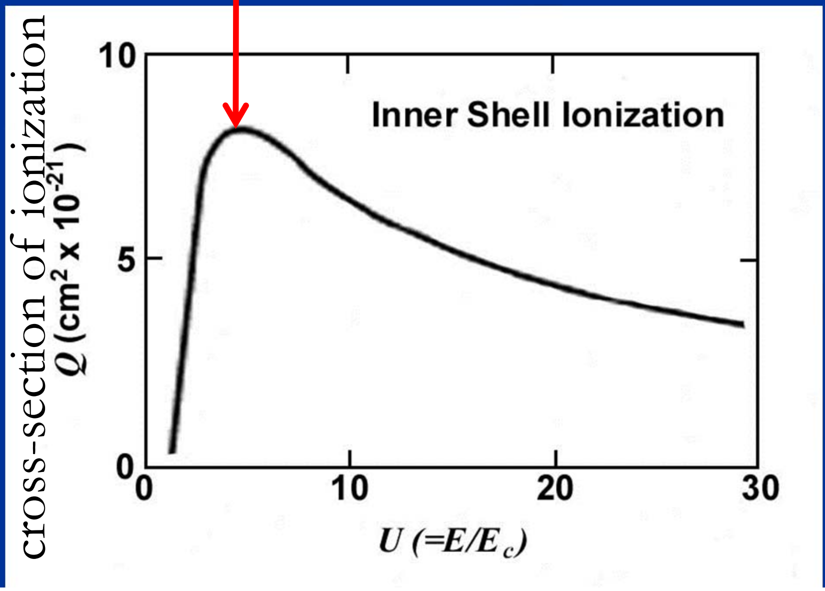
$$U = E/E_c$$

$$> 1$$

E : electron beam energy *(~10-20 keV)*

E_c : critical excitation energy *of the shell*

Best analytical condition, U≈5

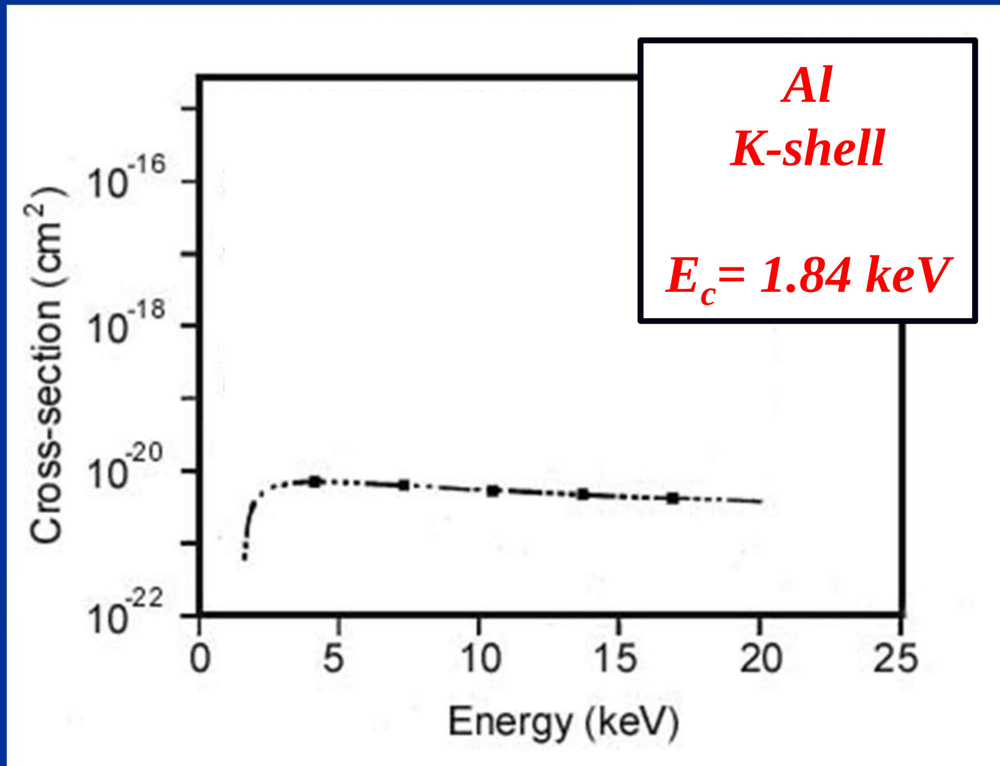


Cross-section of ionization

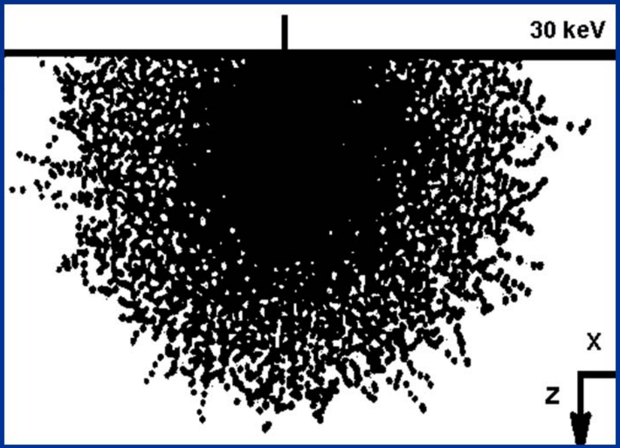
$$Q = 6.51\text{x}10^{-20}\,[(n_s b_s)/(UE_c^{\,2})]\,\ln(c_s U)$$

n_s : *number of electrons in the shell*

b_s, c_s : *constants*



X-ray production volume and maximum depth



$$R_{\text{X-ray}} = 0.064\left(\boldsymbol{E}^{1.68} - \boldsymbol{E}_{\boldsymbol{c}}^{1.68}\right)\frac{1}{\rho}$$

(Anderson-Hasler range)

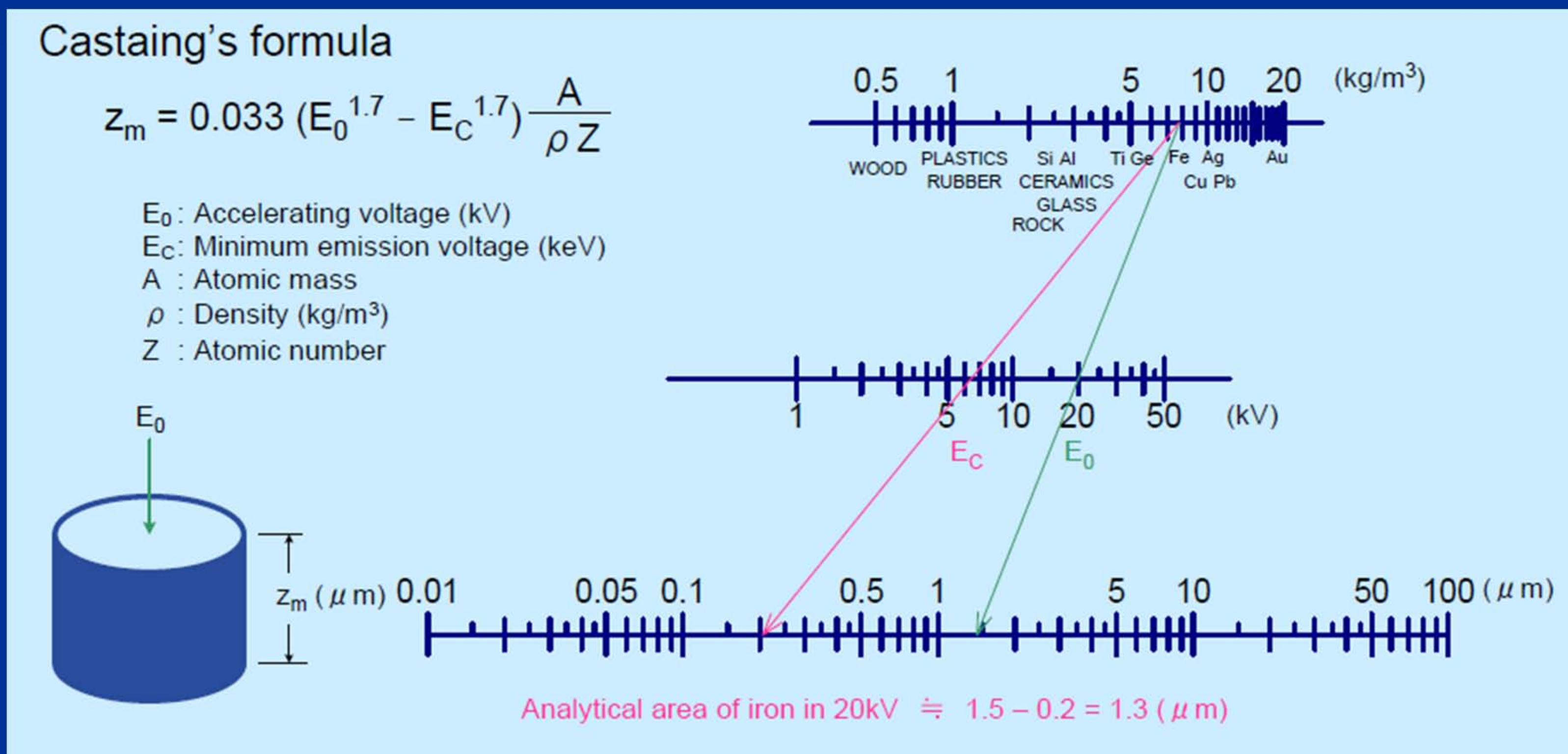
- Always smaller than electron range

  recall analogous expression for electron range:

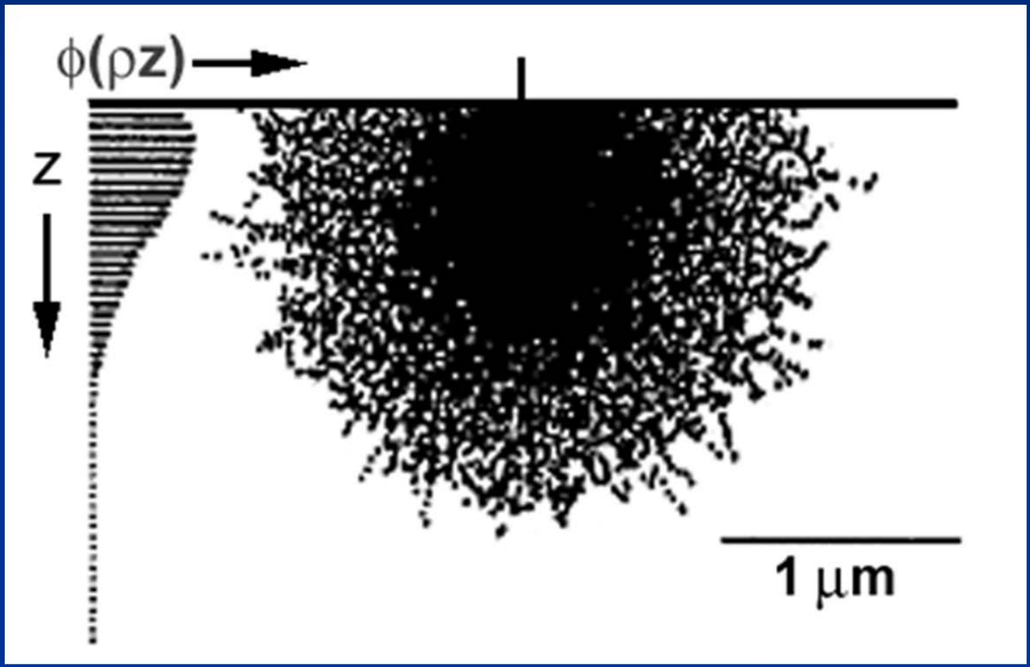
$$\boldsymbol{R}_{\text{electron}} = \left(\frac{0.0276\,\boldsymbol{A}}{\boldsymbol{Z}^{0.889}}\right)\boldsymbol{E}^{1.67}\frac{1}{\rho}$$

- Depends on energy of ionized shell, $\boldsymbol{E_c}$
- Increases with electron beam energy, $\boldsymbol{E}$

X-ray production volume: Castaing's formula



X-ray depth-distribution: the $\phi(\rho z)$ function

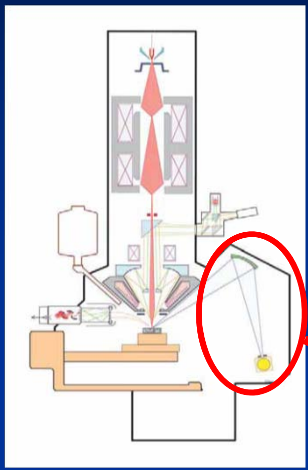


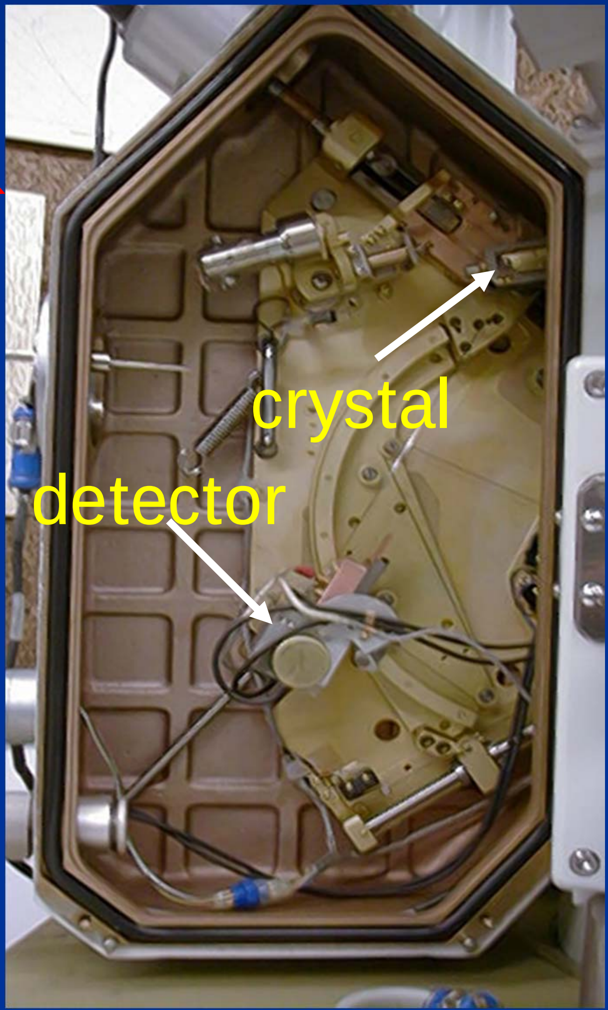
$\phi(\Delta\rho z)$ = intensity from a freestanding layer of thickness ‘z’

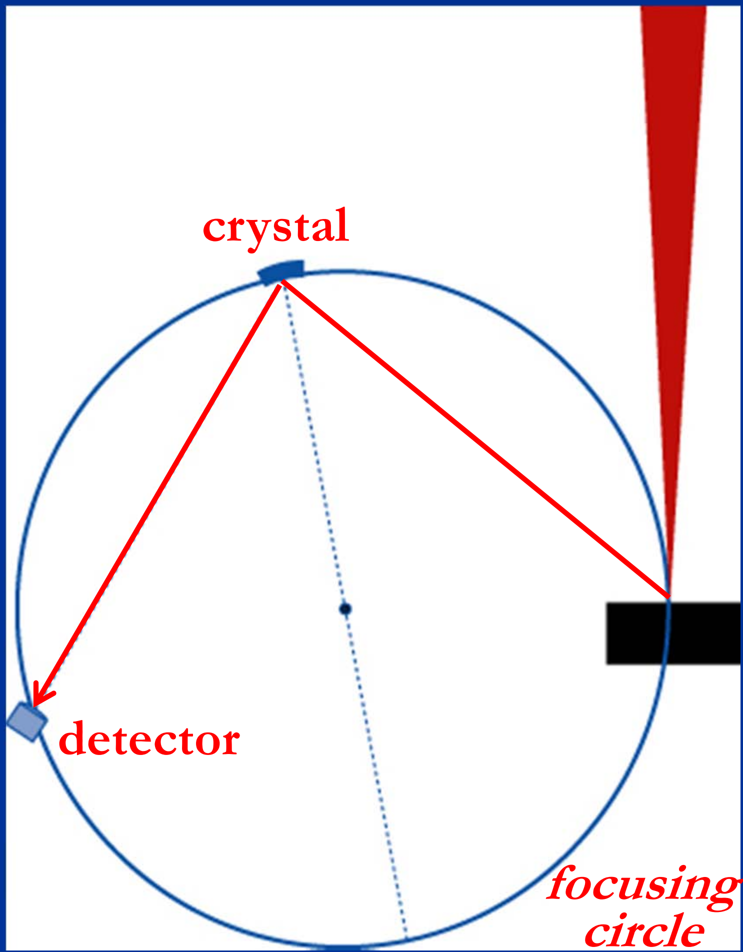
$\phi(\rho z)$ at depth z = intensity from depth ‘z’ divided by $\phi(\Delta\rho z)$

where, ρ = density, and z = depth

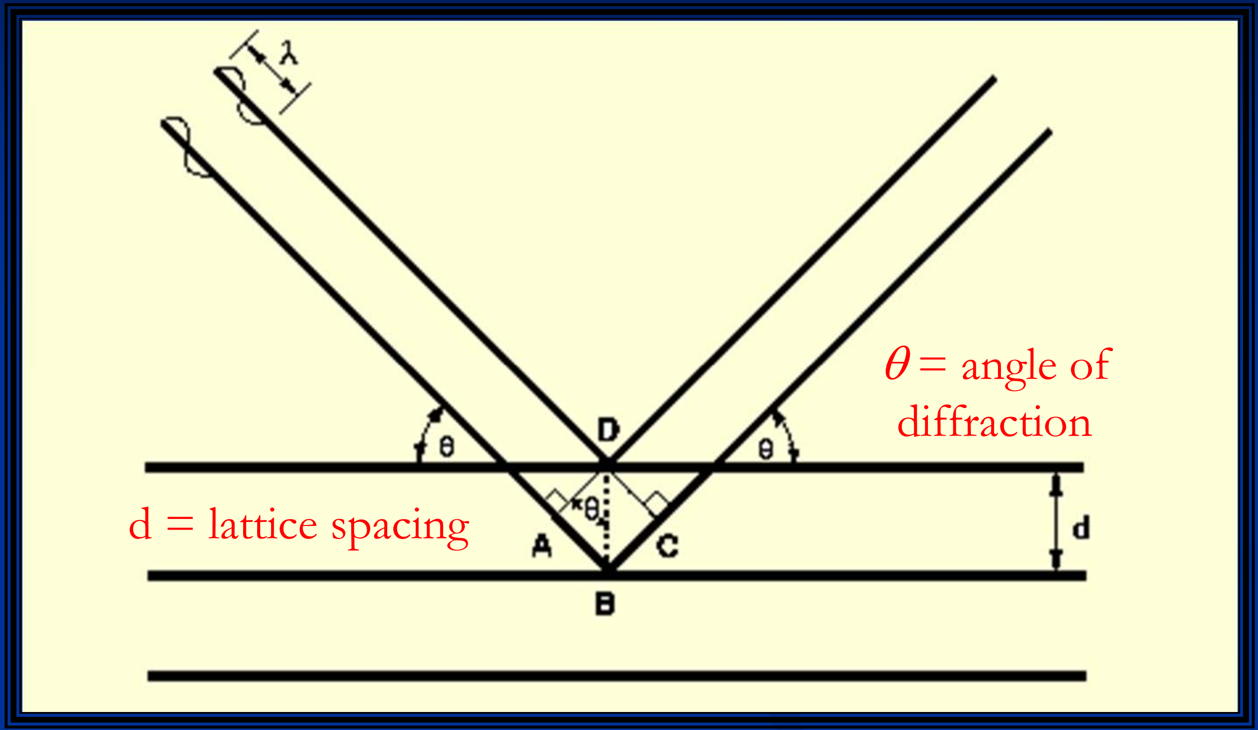
Wavelength Dispersive Spectrometer (WDS)







Bragg's Law

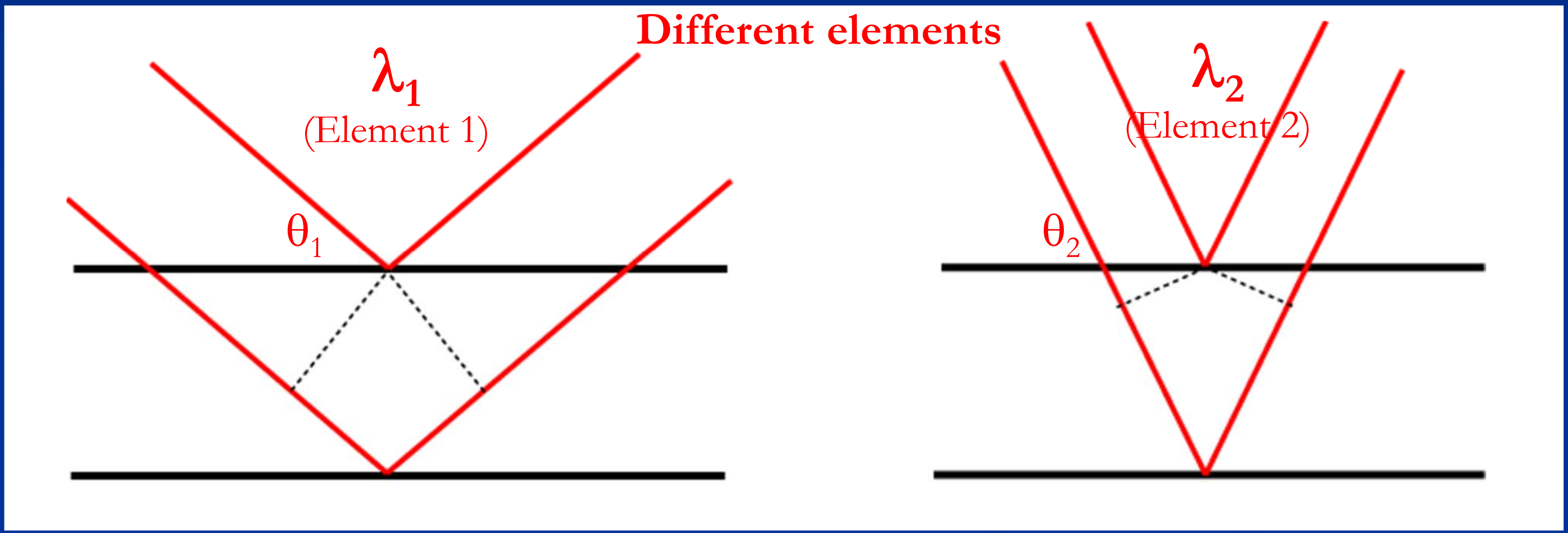


$$n\lambda = 2d\sin\theta$$

= path length ABC

n = order of reflection (any integer)

Diffraction angle

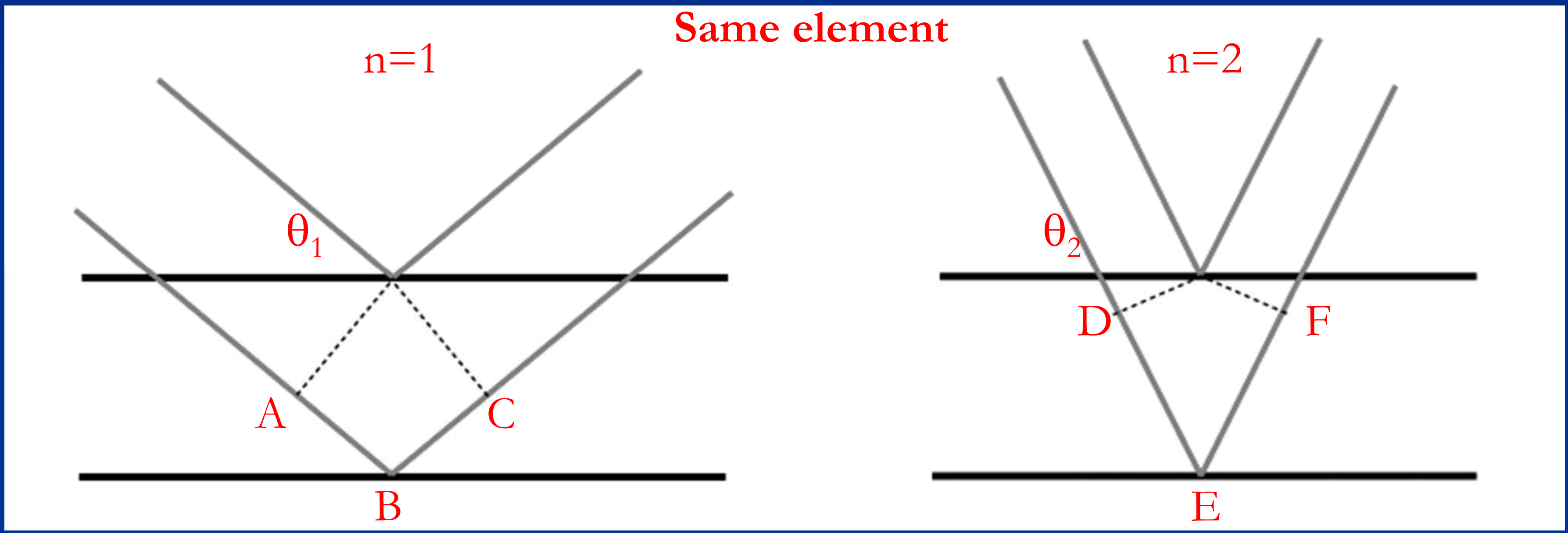


$n\lambda_1 = 2d \sin\theta_1$

$n\lambda_2 = 2d \sin\theta_2$

Diffraction angle changes with wavelength being diffracted (for the same order of reflection, n)

First and second order diffractions



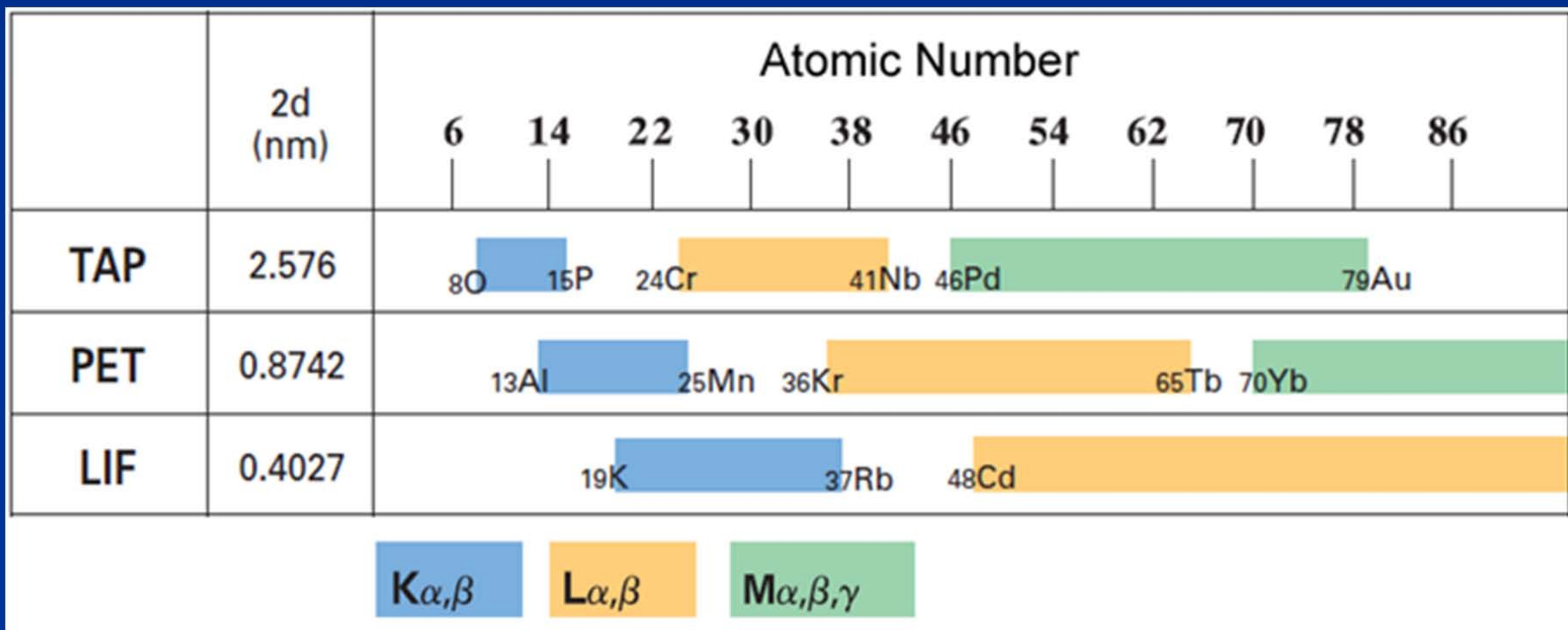
$1\lambda = 2d \sin\theta_1$
=ABC

$2\lambda = 2d \sin\theta_2$
=DEF

path DEF = 2* path ABC

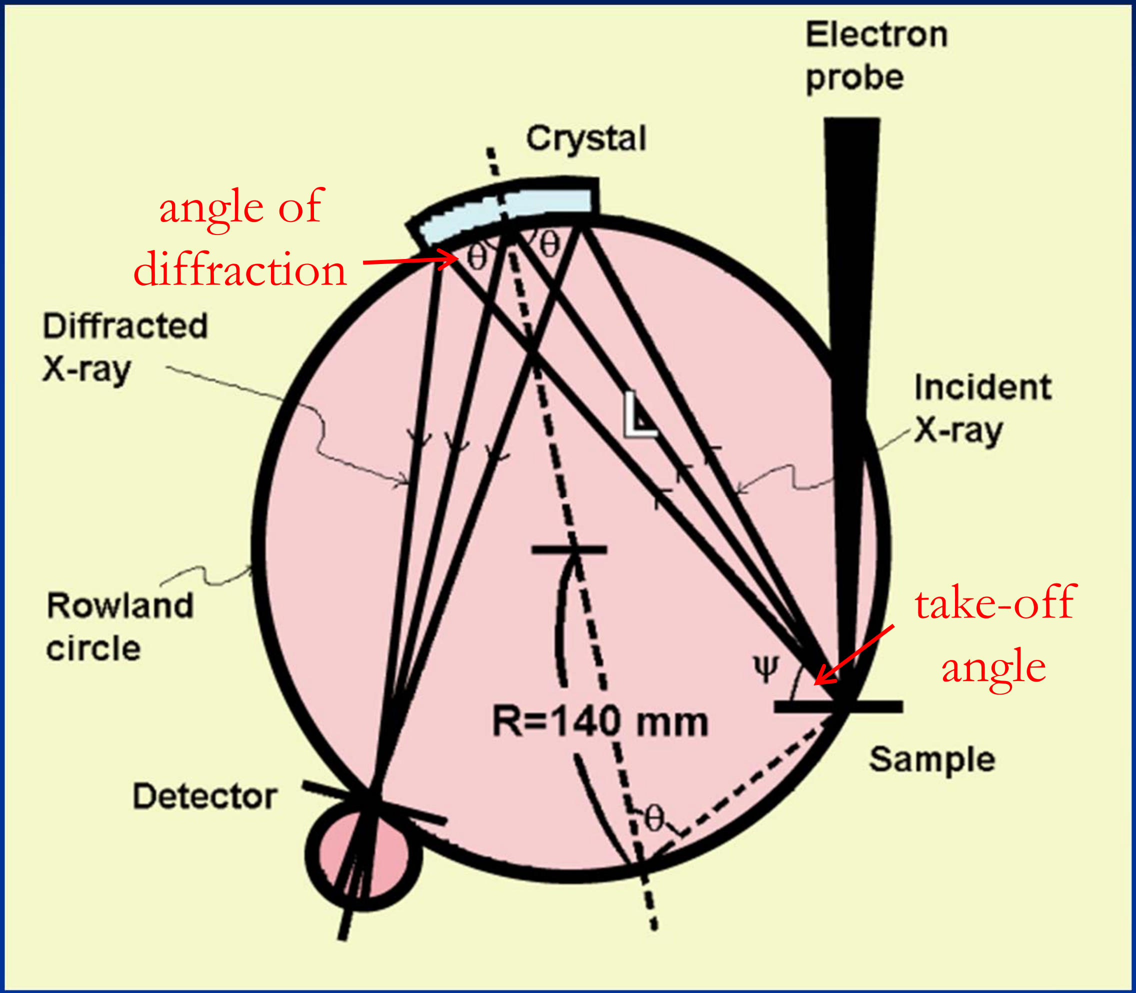
Same wavelength is being diffracted at different diffraction angles

Analyzing crystals with different "d" spacings



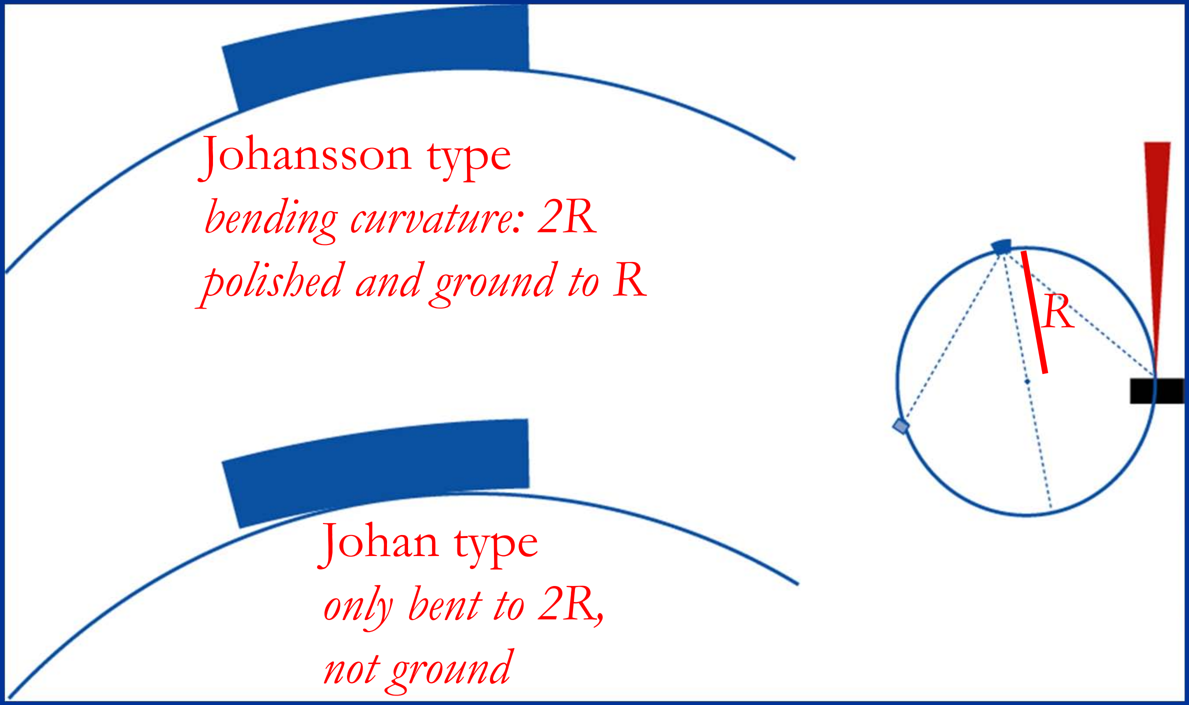
	2d (nm)	Be	B	C	N	O	F
LDE1	Approx.6			• •••	◎	◎	◎
LDEB	Approx.14.5	◎	○				
LDE2H	Approx.10		◎	◎			

WDS: Focusing geometry



$$L = n\lambda . R/d$$

Curved diffracting crystals

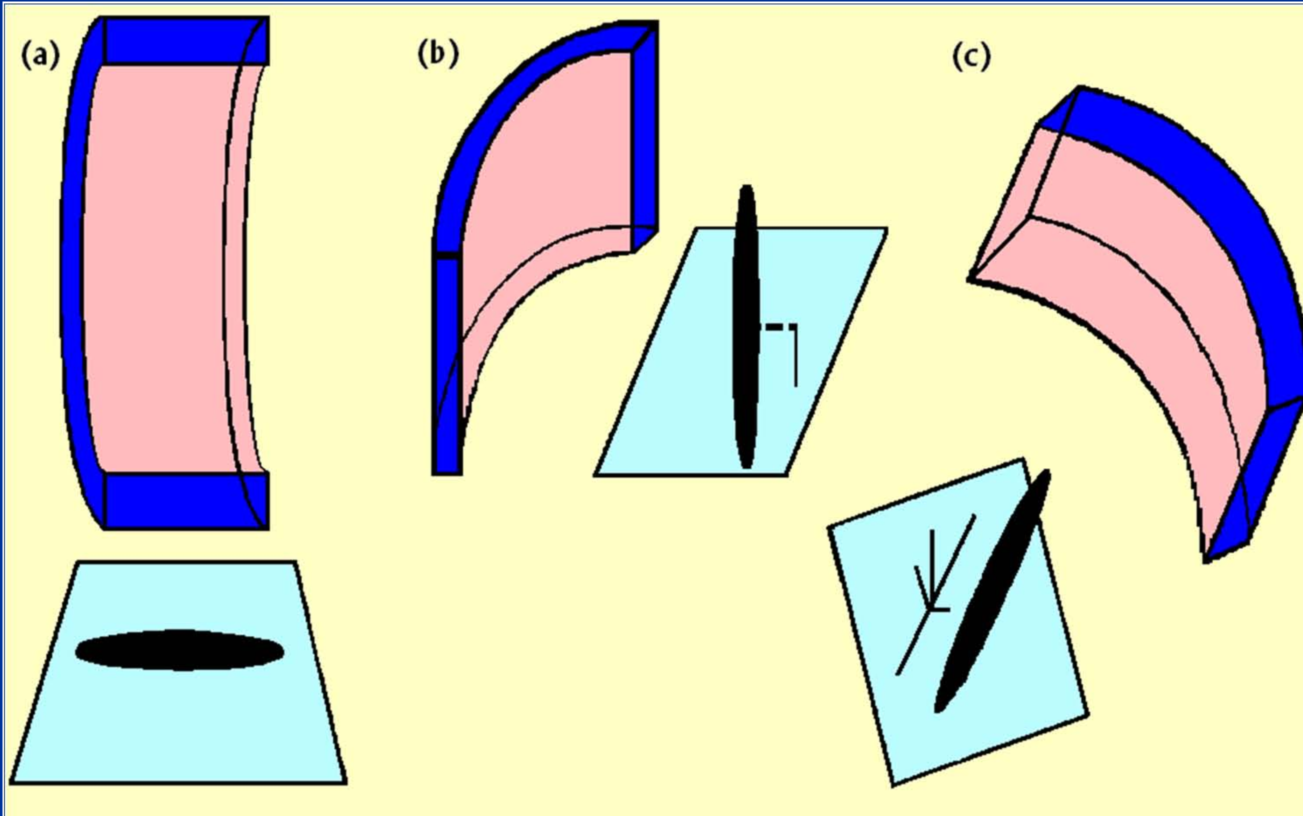


FWHM of fully focusing Johansson-type crystal ~10 eV

Some defocusing in Johan-type, but resolution is not compromised

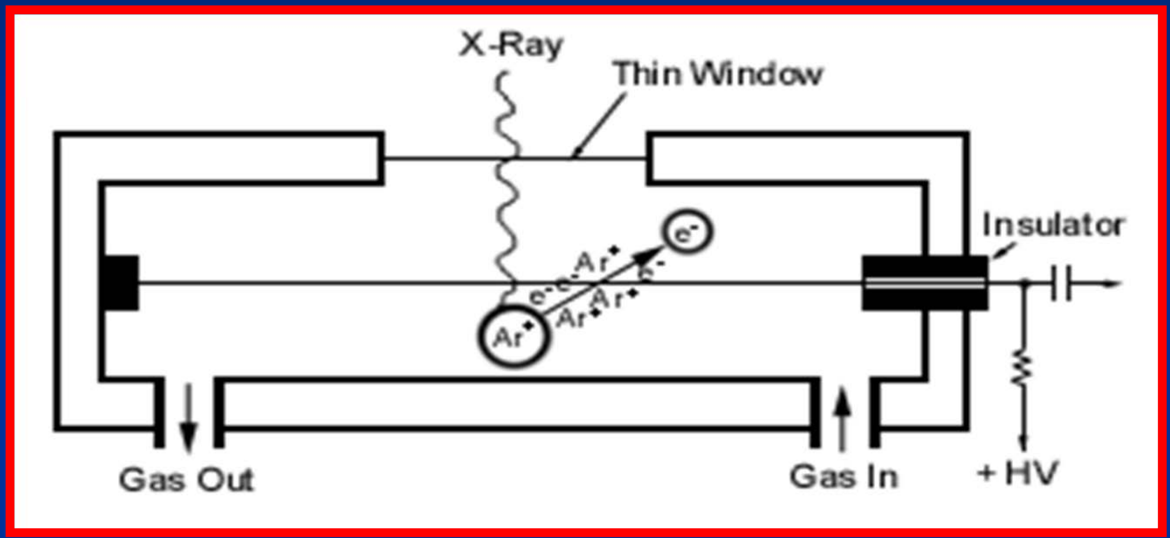
Crystal orientations

Vertical, horizontal and tilted spectrometers



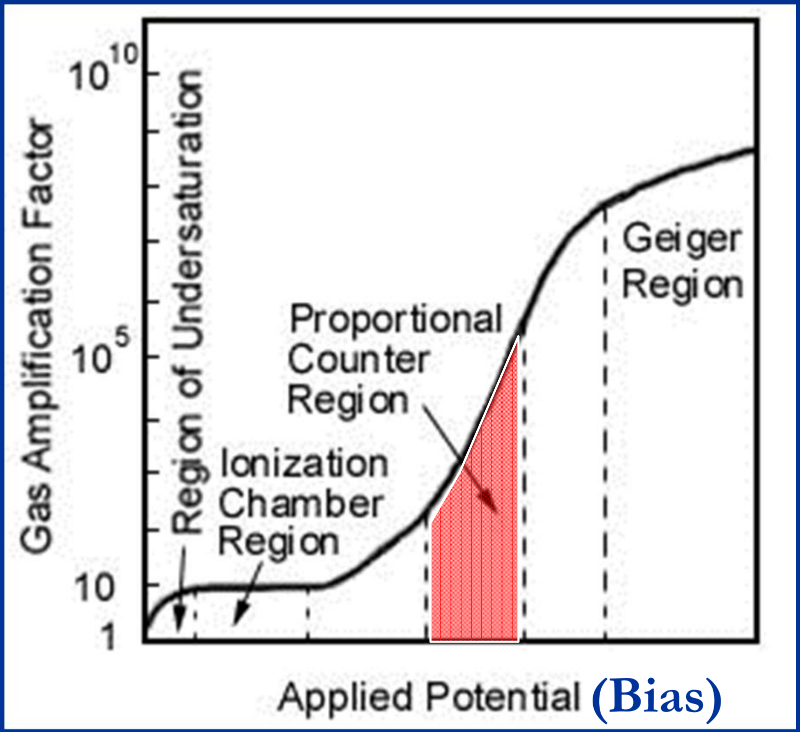
Position of the focus ellipsoid for a WDS spectrometer in the (a) vertical, (b) horizontal, and (c) inclined geometries.

WDS: X-ray detector
(proportional counter)



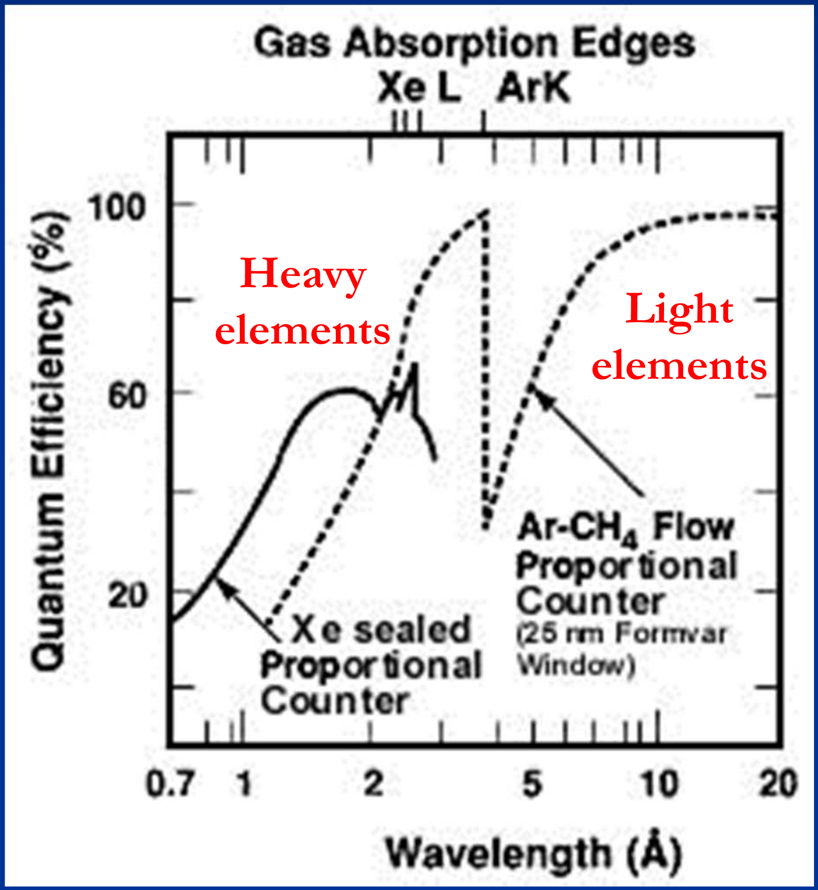
- *Tungsten collection wire set at 1-3 kV bias*
- *Flow counter: 90% Ar +10% CH_4 (P-10); poly-propylene window*
- *Sealed counter: Xe; Be window*

Amplification in proportional counter



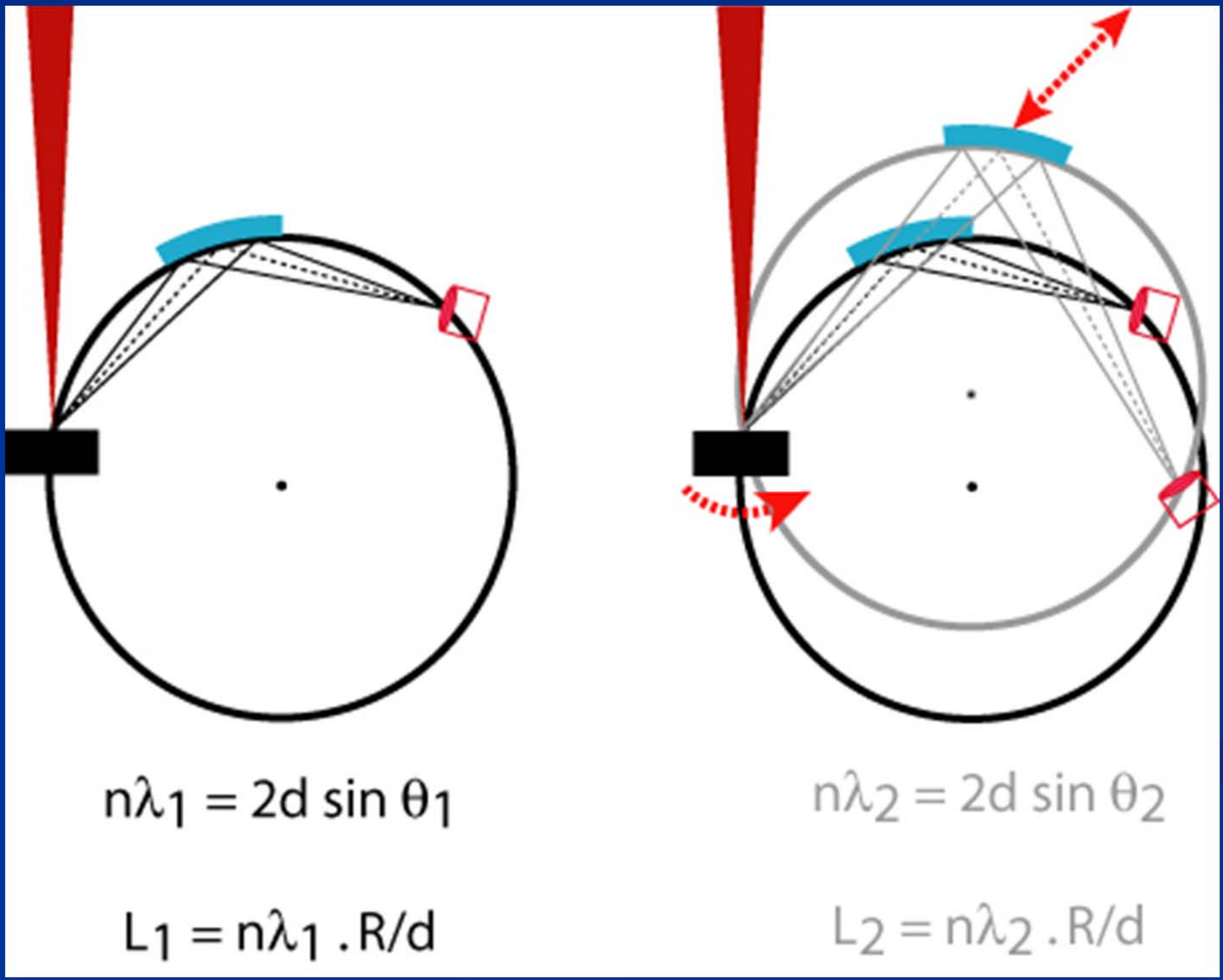
- *Collection wire bias range (applied potential): 1-3 kV*
- *Bias is set so that amplification is in the proportional region*

Counting efficiency of gas in proportional counter

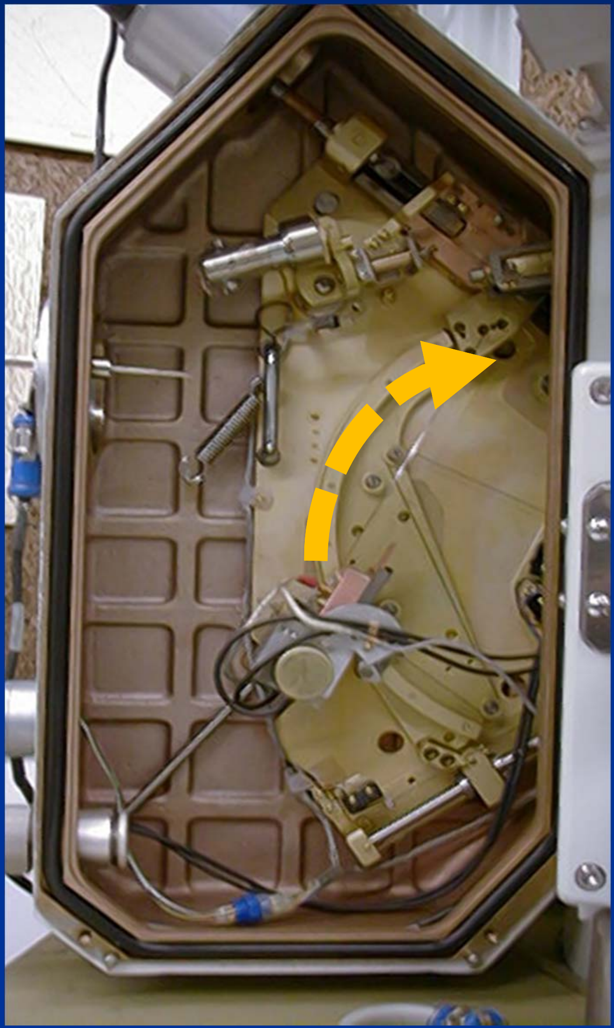


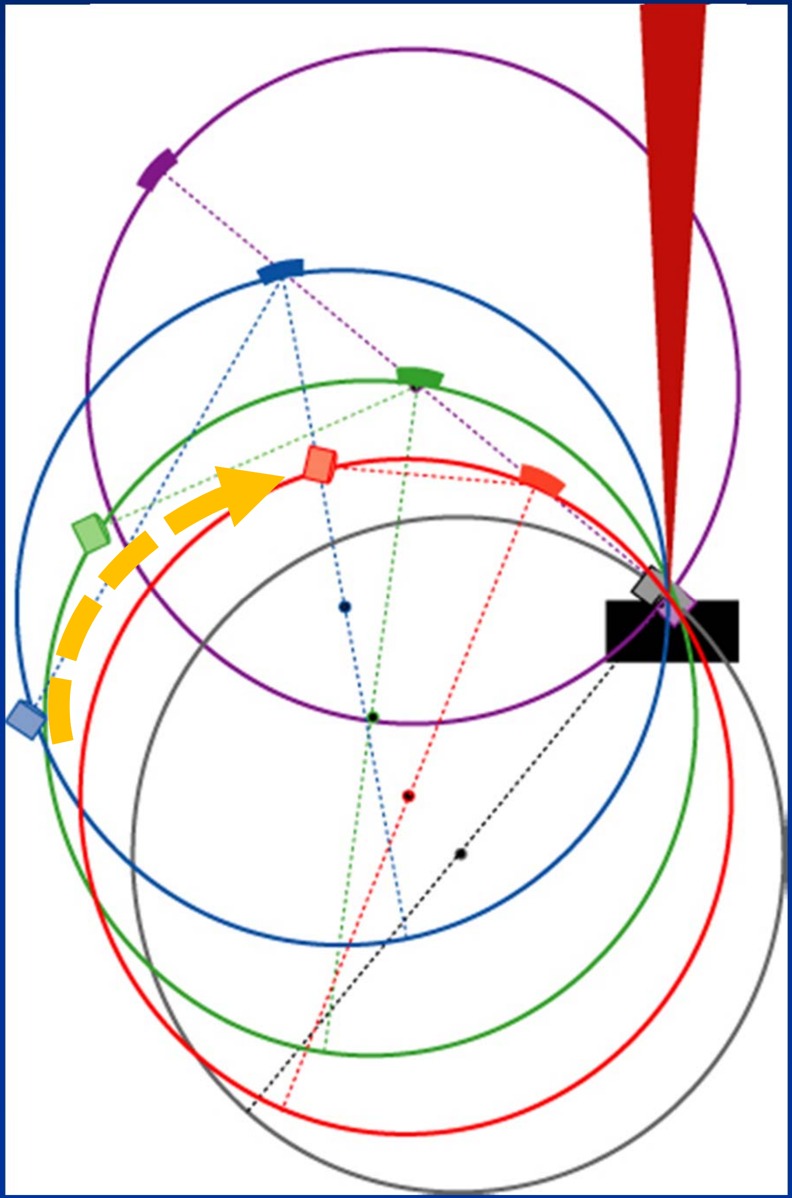
- Gas used for long wavelengths: 90% Ar +10% CH_4 (P-10)
- Gas used for short wavelengths: Xe

WDS: changing the angle of diffraction



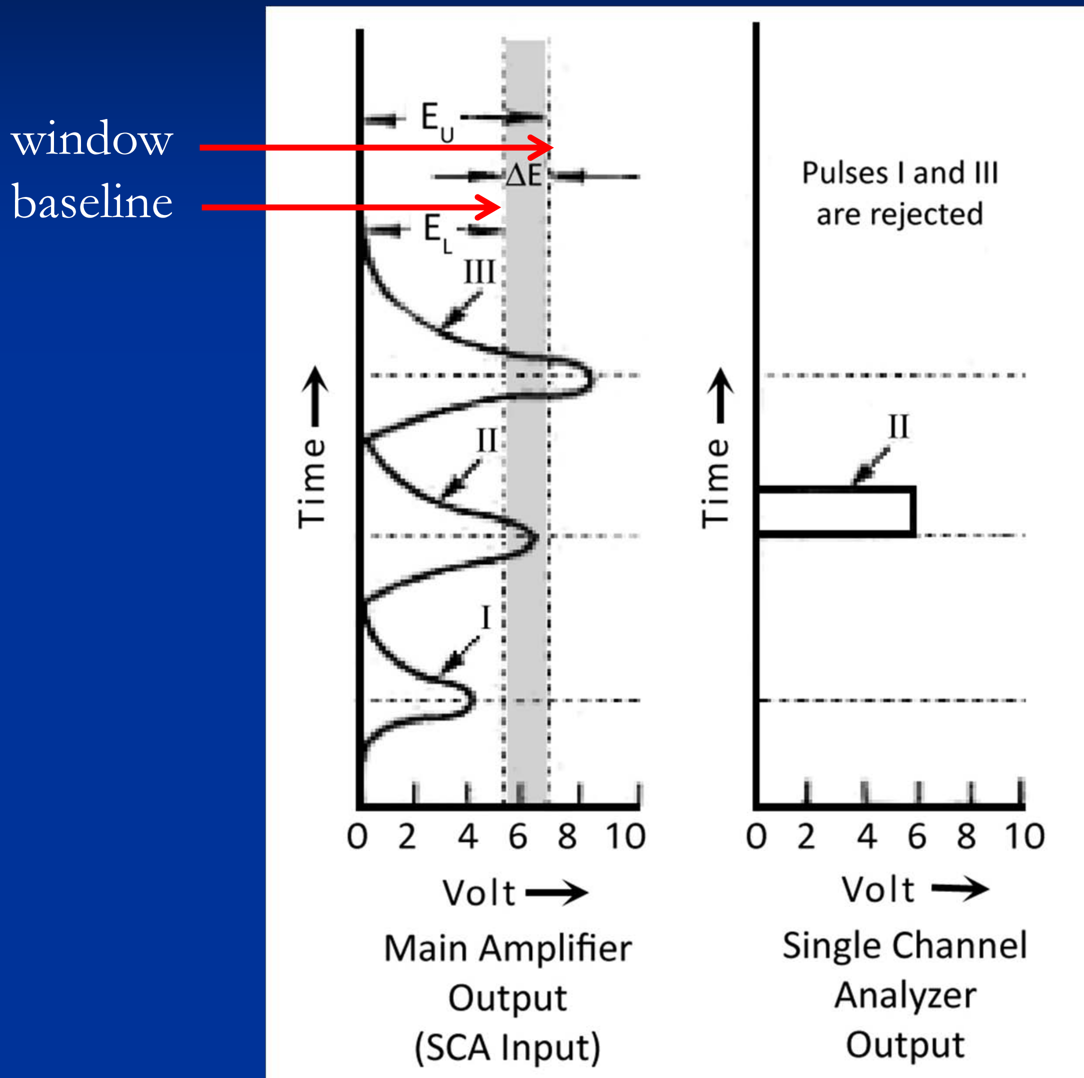
Theoretical and actual limits of spectrometer movement





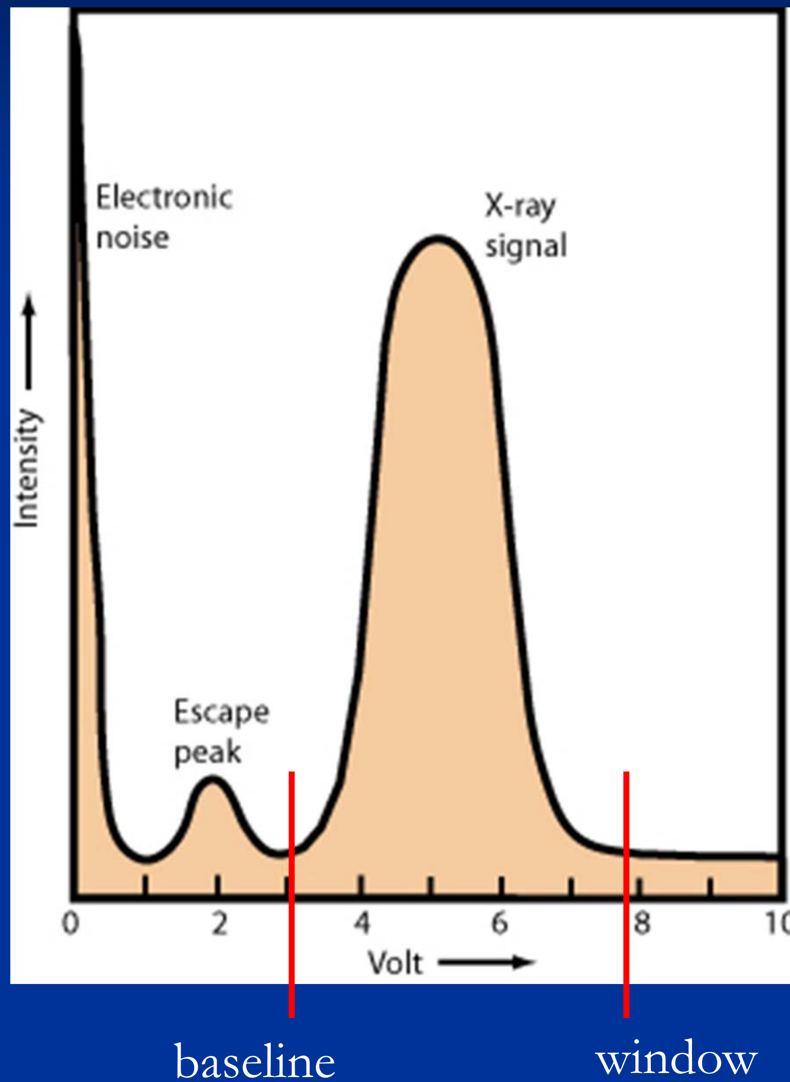
2R ≤ L ≤ 0

WDS signal processing



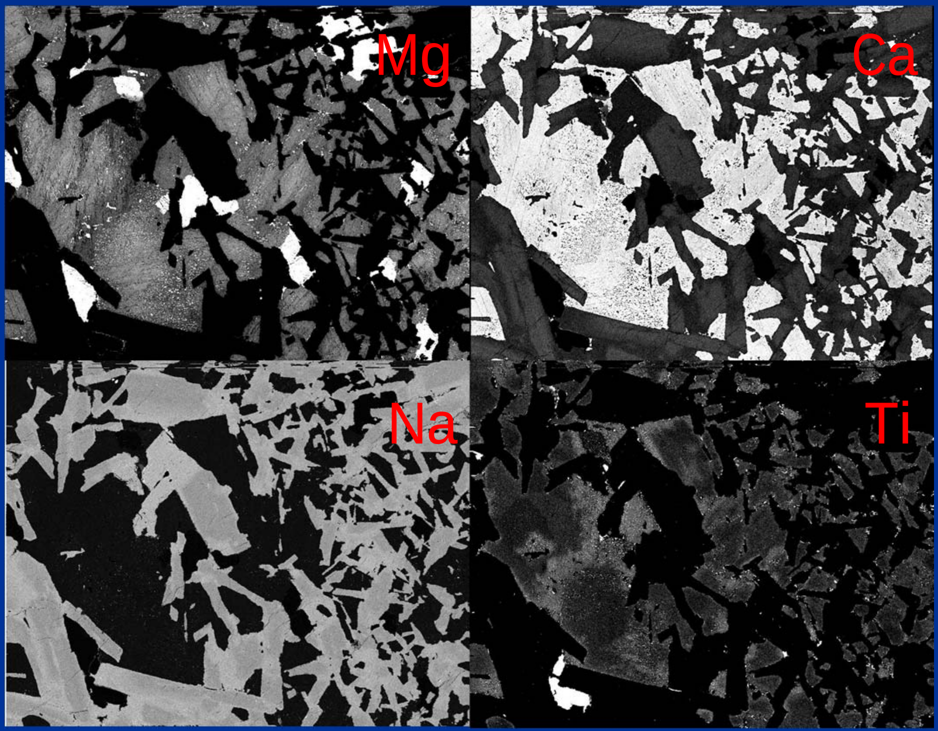
Single channel analyzer (SCA)

Pulse Height Analysis (PHA)



Single Channel Analyzer (SCA) scan

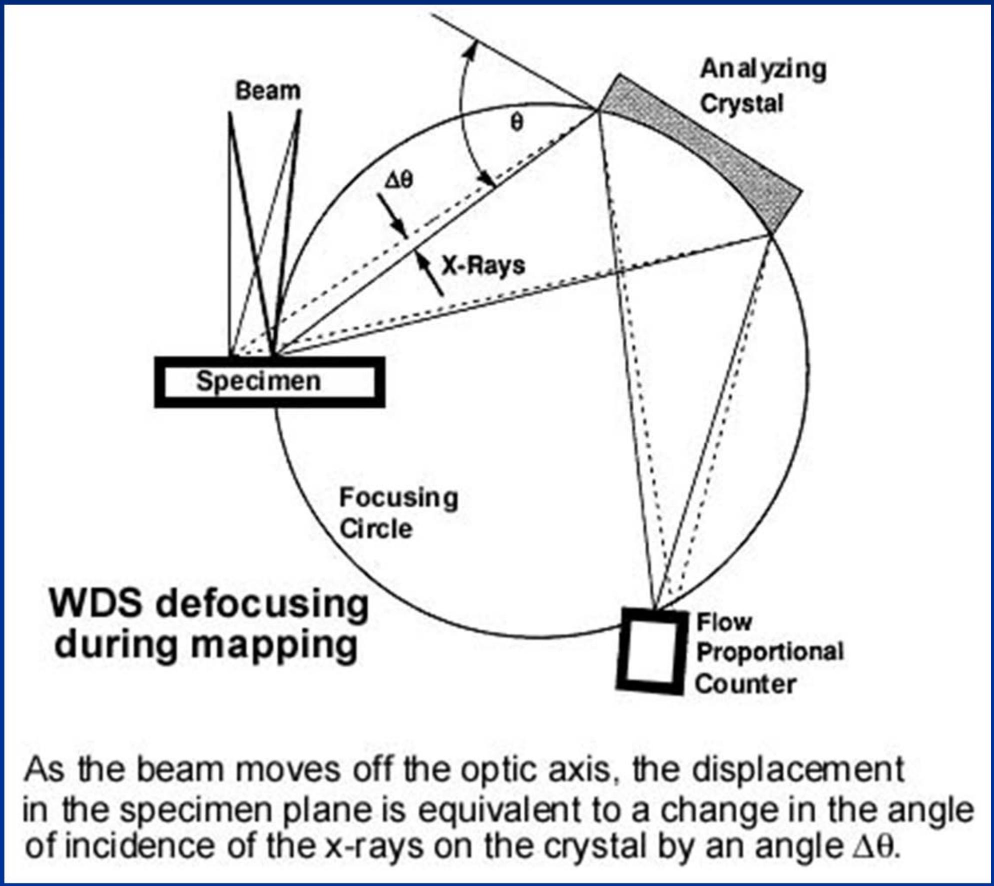
Imaging with X-rays: compositional mapping

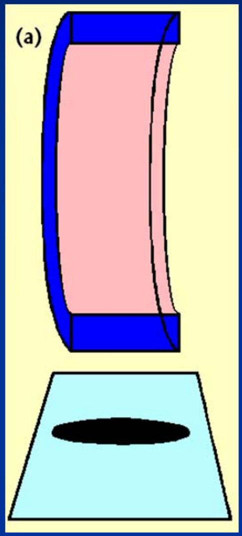


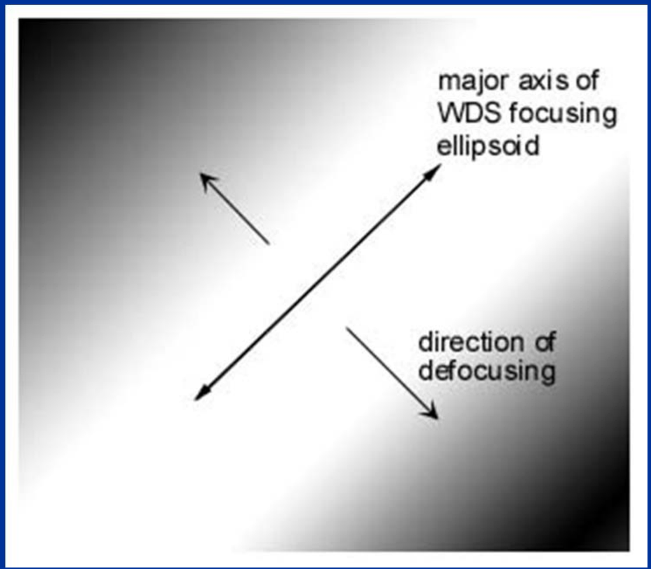
- **Beam-rastered image:** *electron beam rasters over the area to be imaged*
- **Stage-rastered image:** *electron beam is stationary, stage moves*

- **Number of point measurements: image resolution**
- **Signal: beam current and dwell time/point**

Defocusing in beam-rastered WDS X-ray maps

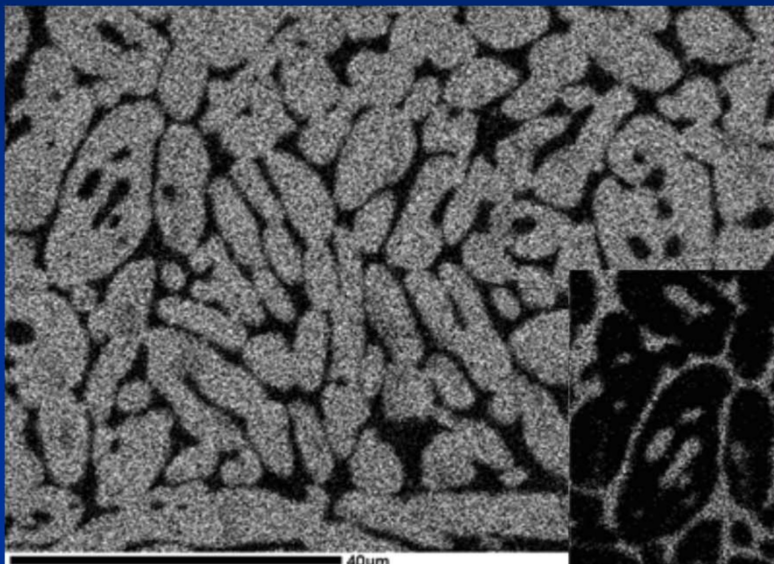




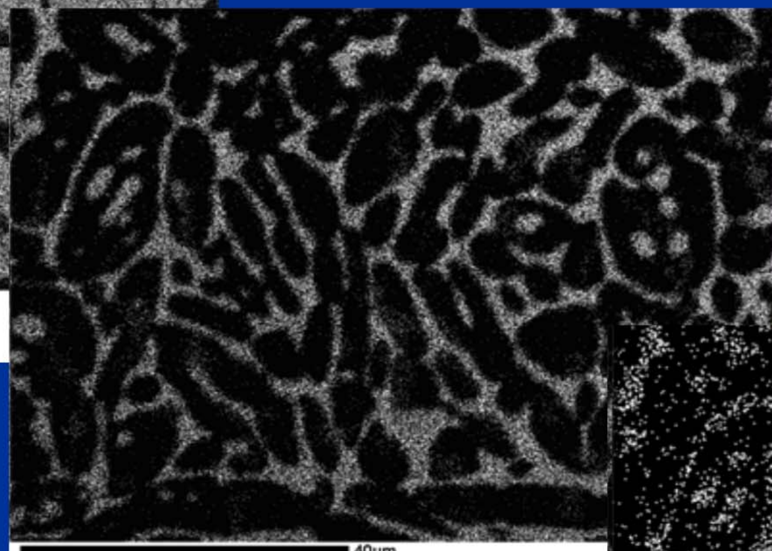


X-ray image artifact: background

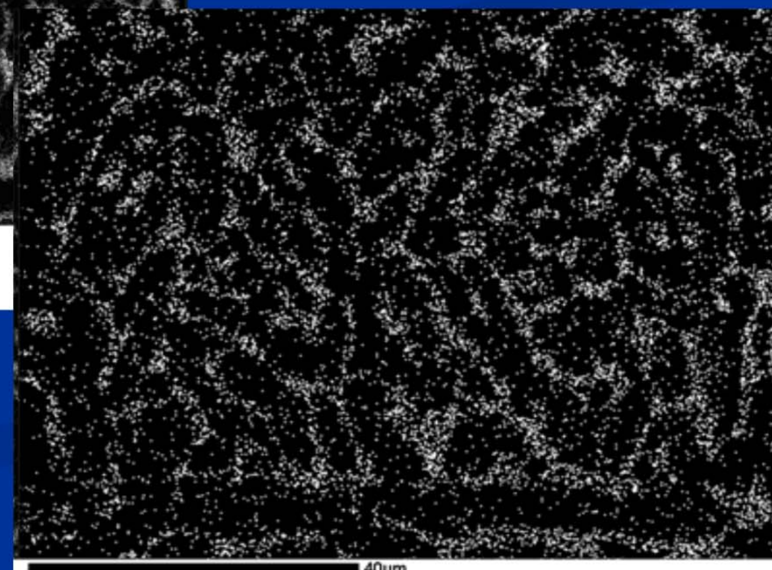
Zn-Sn composite



Zn x-ray image



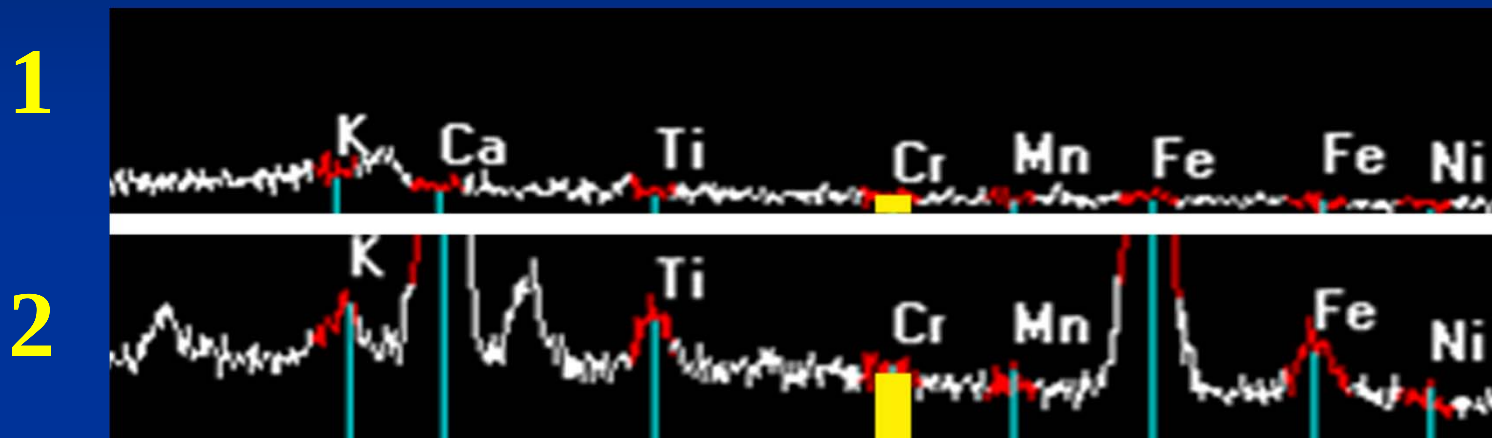
Sn x-ray image



Ca x-ray image

Continuum X-rays: background artifact

A composite made of 2 materials is being mapped:



Neither material contains Cr

But background counts for Cr :

in 1 in 2

Therefore, if a Cr X-ray map is acquired, material 2 will show higher Cr than material 1