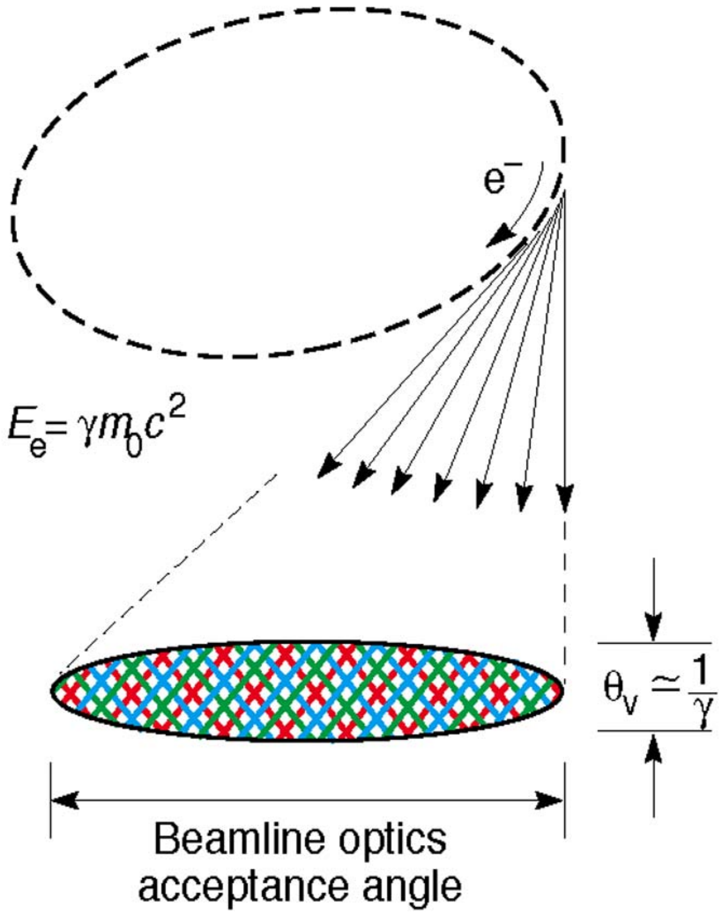
Basics of Synchrotron Radiation Beamlines and Detectors

- Basics of synchrotron radiation
- X-ray optics as they apply to EXAFS experiments
- Detectors

Important properties of Synchrotron Radiation

- Tunability
- High flux
- Collimation
- Polarization
- Time structure

Bending magnet radiation

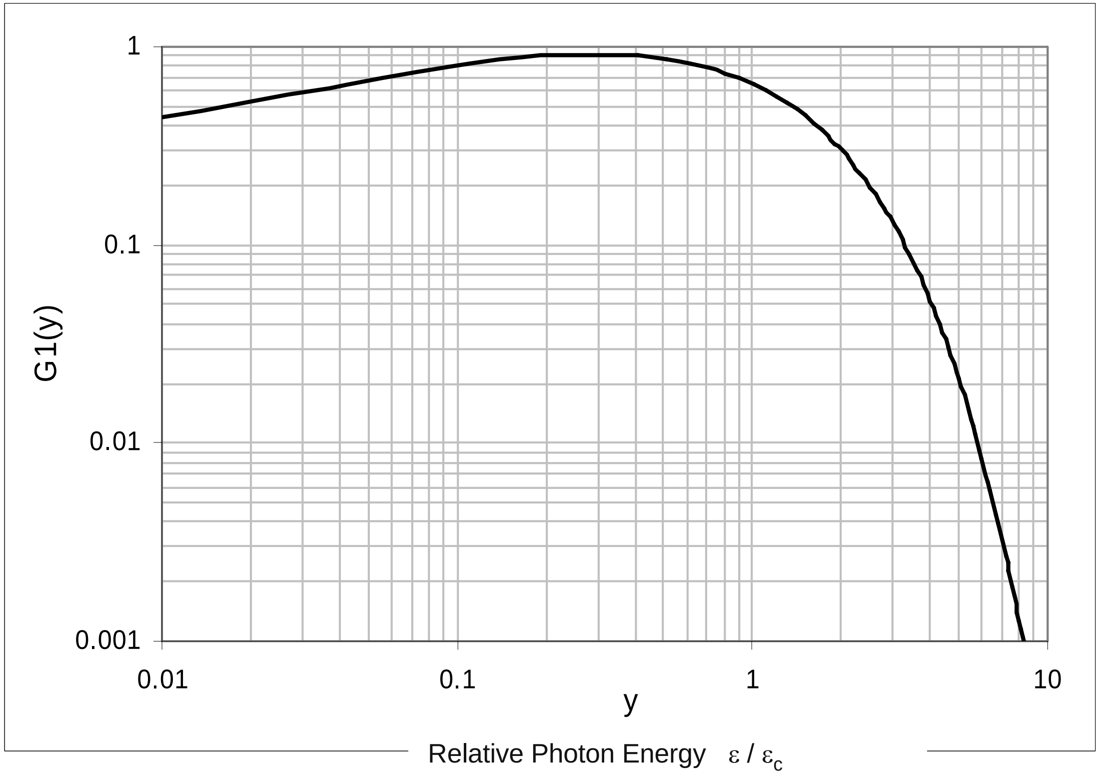


Emission limited to angle range $1/\gamma$.

γ= 1957E(GeV)

For APS: γ = 13699 or $1/\gamma$ = 73 μrad

Tunability – Bending magnet



Emitted Radiation has Characteristic Photon Energy

$$\varepsilon_c = 0.665\, B_\circ\, E^2$$

ε_c – Critical Photon Energy [keV]

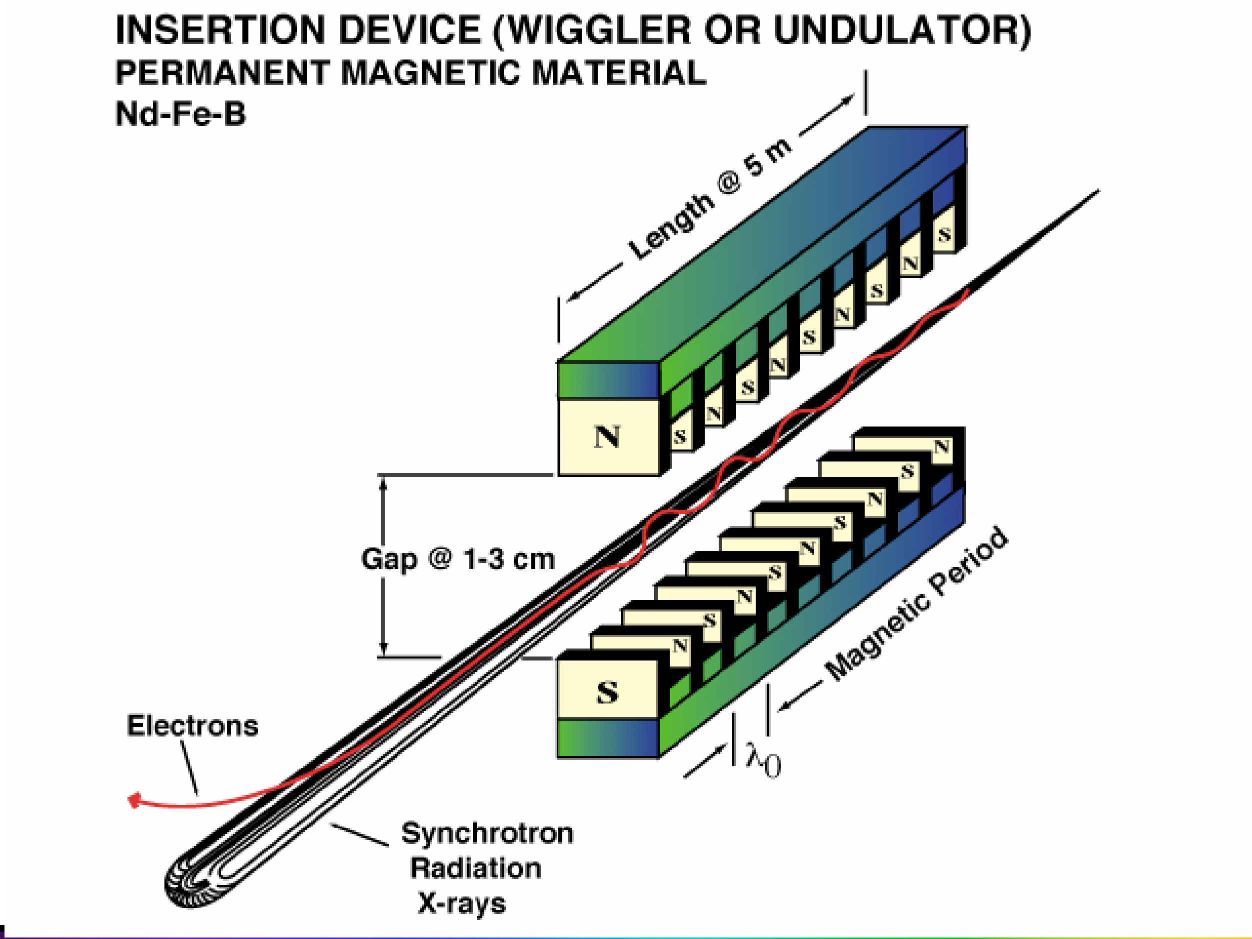
E – Electron Energy in [GeV]

B_o – Magnetic Field in [Tesla]

$$Flux / mrad / 0.1\% \, BW = 2.457 \times 10^{13}\, E\, I\, G_1(y)$$

Insertion device

Many bends to increase flux over single bend in bending magnet



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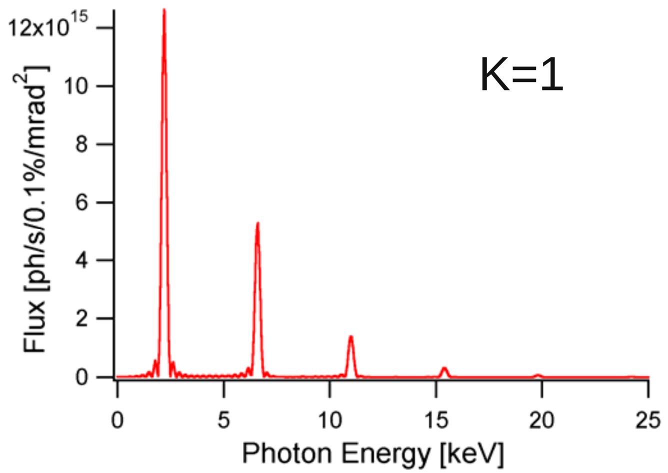
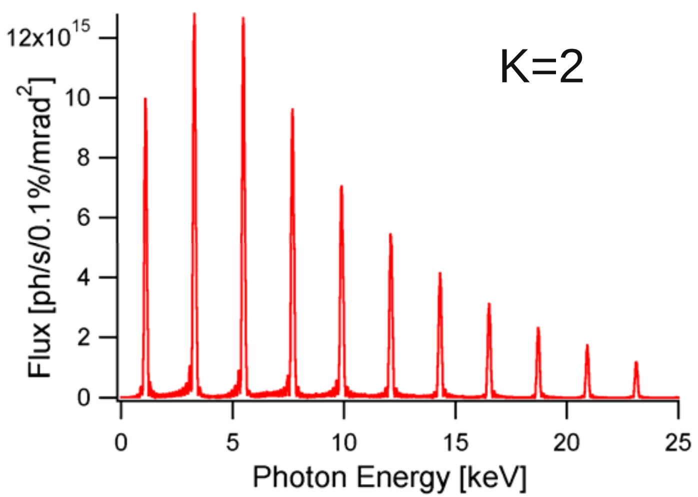
APS Undulator A

Tunability - Undulators





Undulator energy tuned by varying its K value – usually by tuning the magnetic gap which varies B

$K = 0.0934\ \lambda_u\ [mm]\ B_o\ [T]$

Undulator A tuning curve

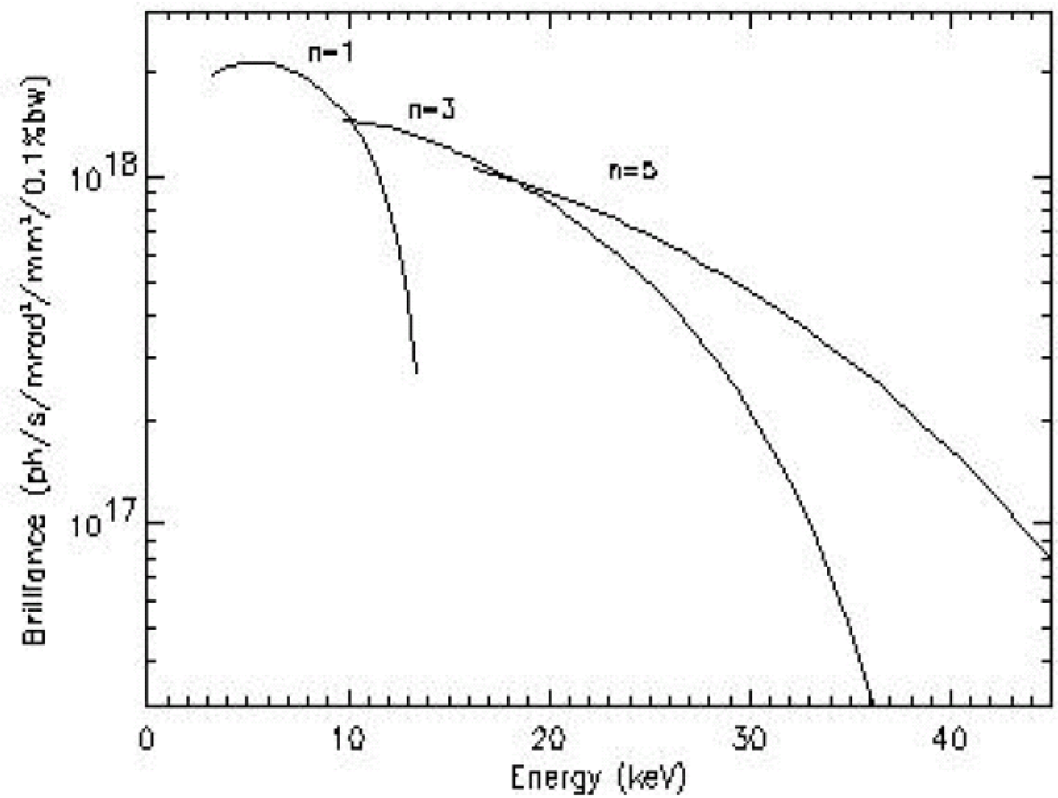


Figure 3. Tuning curves for the on-axis brilliance for the first three odd harmonics.

Each curve follows one of the harmonics as K (gap) is varied

Source characterization

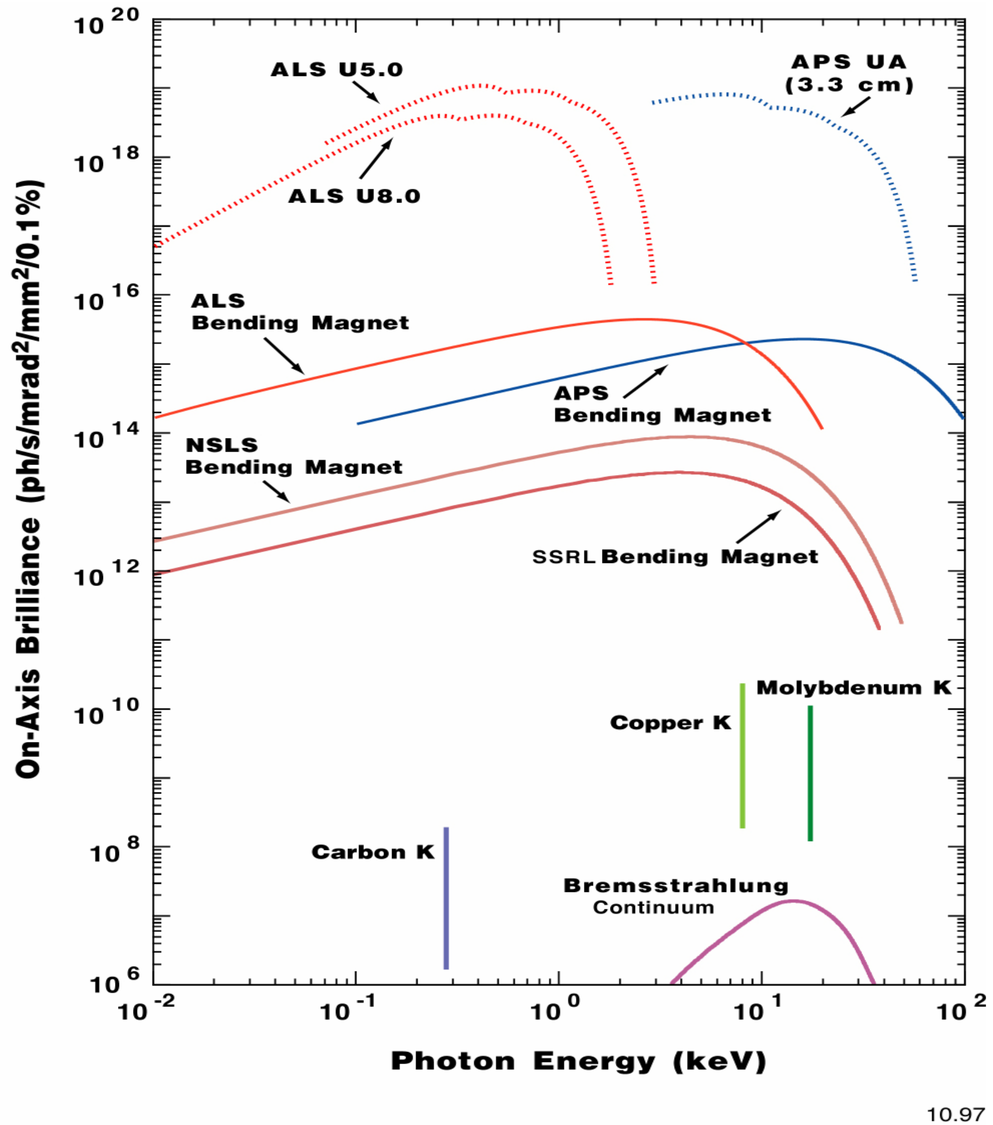
Flux – photons/sec/bandwidth
Bandwidth usually chosen as 0.1%
Most applications use 0.01-0.02%

Spectral Brilliance - flux/source size/source divergence
Photons/sec/0.1% bandwidth/mm^2/mrad^2

Liouville's theorem – brilliance is conserved
Optics can't improve brilliance of source

Higher Brilliance implies more flux on small samples

Comparison of source brilliance



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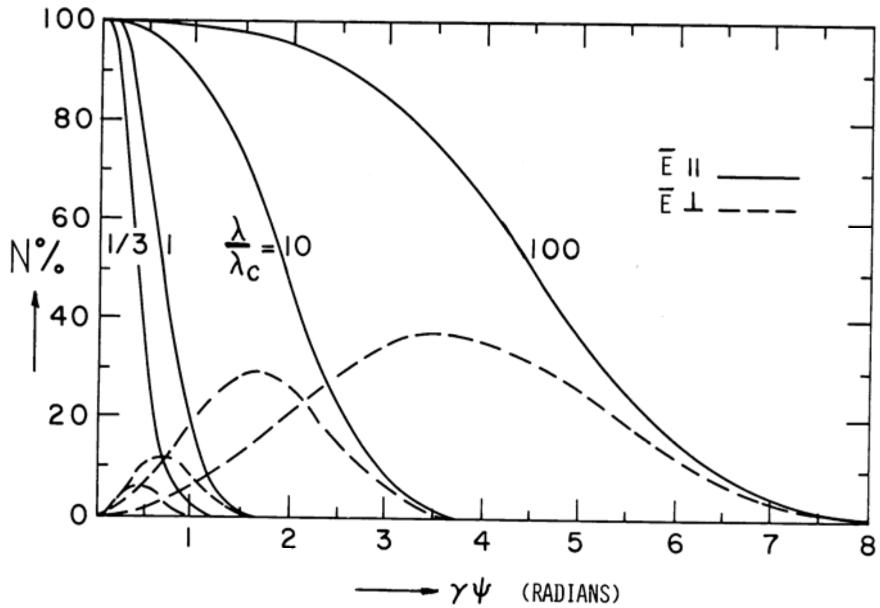
Polarization

Both BM and standard ID's primarily polarized in the horizontal
Fully polarized on axis

BM polarization



VERTICAL ANGULAR DISTRIBUTION OF PARALLEL AND PERPENDICULAR POLARIZATION COMPONENTS

Time structure

Storage rings store charge in discreet bunches

- Short pulses (100psec)
- 272 kHz circulation rate for an individual bunch at APS
- Many patterns possible (24, 324, 1296 bunches, hybrid fill with an isolated bunch)
- Generally not important, but can affect the deadtime of fast detectors

Current gradually decays

- Close shutters to refill
- Topoff : refill with shutters open

X-ray Optics

- Mirror Optics
  - Focusing and collimation (energy resolution)
  - Harmonic rejection
- Perfect crystals
  - Monochromatization
- Typical beamline setups

Mirror optics

Glancing angle optics needed for x-rays

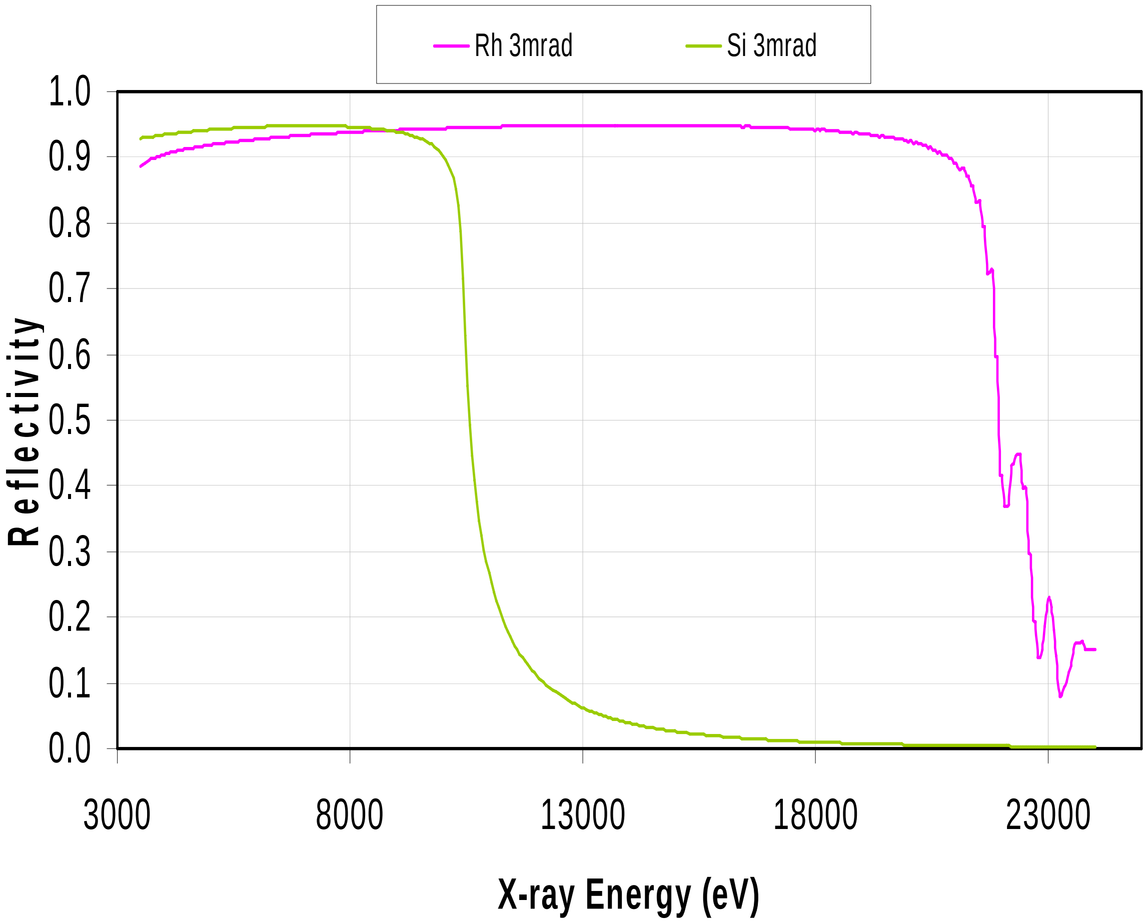
- For small enough angles reflectivity nearly 100%
- Achromatic for energies less than critical energy
- Ultra-smooth surfaces needed (0.5 nm roughness)
- Critical energy approximately linearly related to angle

  For example, for Rh,

$$E_c(\text{keV}) = 68/\text{angle}(\text{mrad})$$

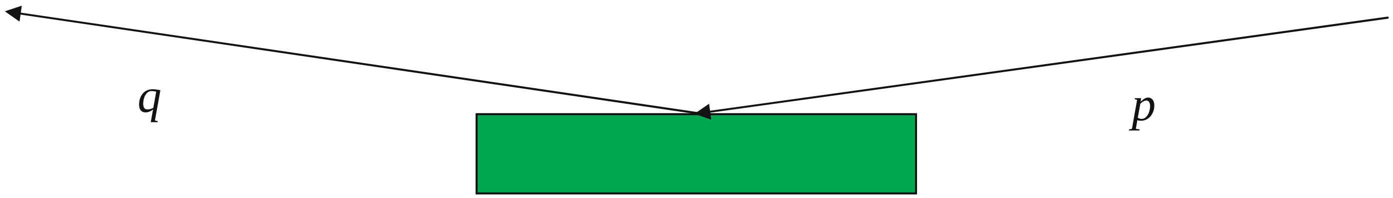
- Small angles mean mirrors need to be long

X-ray Reflectivity



Beamline mirrors

Collimating or focusing mirrors: typically ~1m long



Along beam: $$R_m = \frac{2pq}{(p+q)\sin\theta}$$ Typically km

Perpendicular to beam: $$R_s = \frac{2pq\sin\theta}{p+q}$$ Typically cm → limits collection angle

Magnification M=q/p

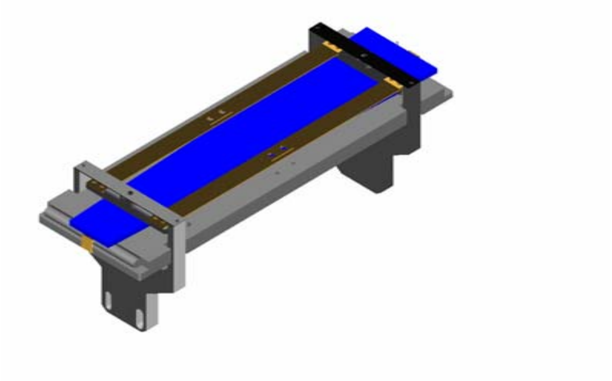
Toroidal (ellipsoidal) focusing mirror

Glancing angles result in long narrow cylindrical shapes

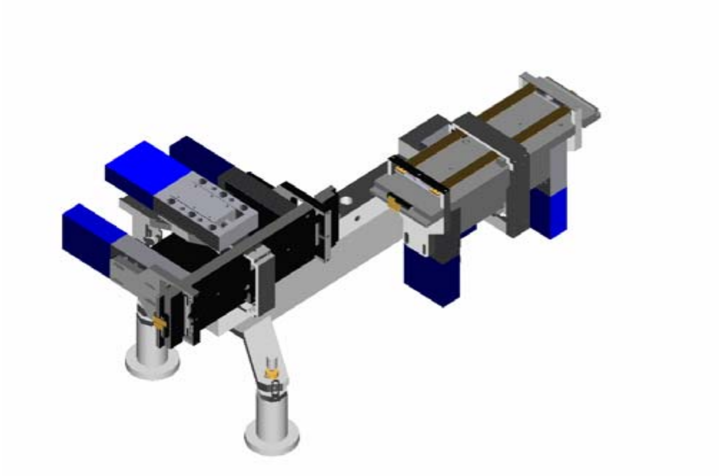
Often approximated by a cylinder bent along beam direction

Kirkpatrick-Baez (K-B) mirrors

Separately focus the horizontal and vertical using elliptical mirrors



Individual mirror



Assembled KB mirrors

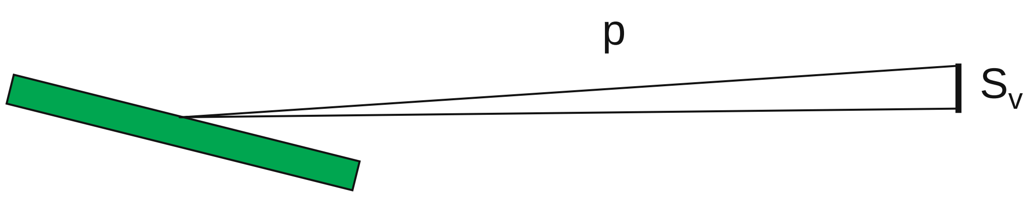
Collimating mirror

Need parabolic shape

$q \rightarrow$ infinity: $$R_m = \frac{2p}{\sin\theta}$$

Collimation limited by vertical source size:

$\Delta\theta = S_v/p$



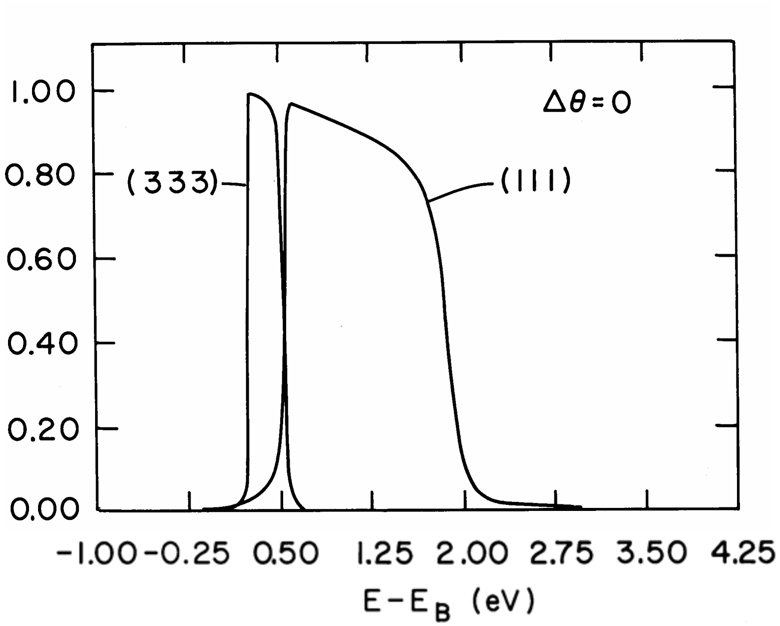
X-ray Optics – perfect crystals

- Bragg reflection and energy resolution
- Monochromators
- Detuning

Bragg reflection basics

- Bragg equation
  - Bragg equation $2d\sin(\theta) = n\lambda$, λ=12.4/E(keV)
  - Perfect crystal Si or diamond – reflectivity nearly 1 over finite range ΔE/E

Si 111 10 keV



Intrinsic Resolution of some common reflections

Reflection	ΔE/E
Si 111	1.3×10^{-4}
Si 220	5.6×10^{-5}
Si 311	2.7×10^{-5}
Diamond 111	6.0×10^{-5}

Energy Resolution

Depends on divergence and intrinsic resolution

From derivative of Bragg equation, divergence results in:

$$\Delta E/E = \cot(\theta)\Delta\theta$$

$\Delta\theta$ determined by slits or collimating mirror if present

Example: 1mm slit 30 m from source at 10 keV with Si 111

$\Delta\theta = 1/30000 = 3.3\text{x}10^{-5}$, θ=11.4 or $\cot(\theta) = 4.9$

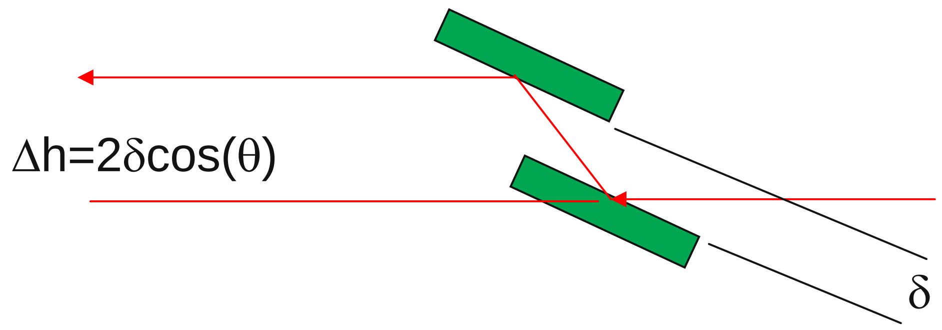
From divergence: $\Delta E/E = 3.3\text{x}10^{-5}(4.9) = 1.6 \text{ x } 10^{-4}$

Add divergence term and intrinsic term in quadrature to get the approximate final resolution:

$$\Delta E / E = \sqrt{(1.6x10^{-4})^2 + (1.3x10^{-4})^2} = 2.1x10^{-4} \qquad \text{(2.1 eV)}$$

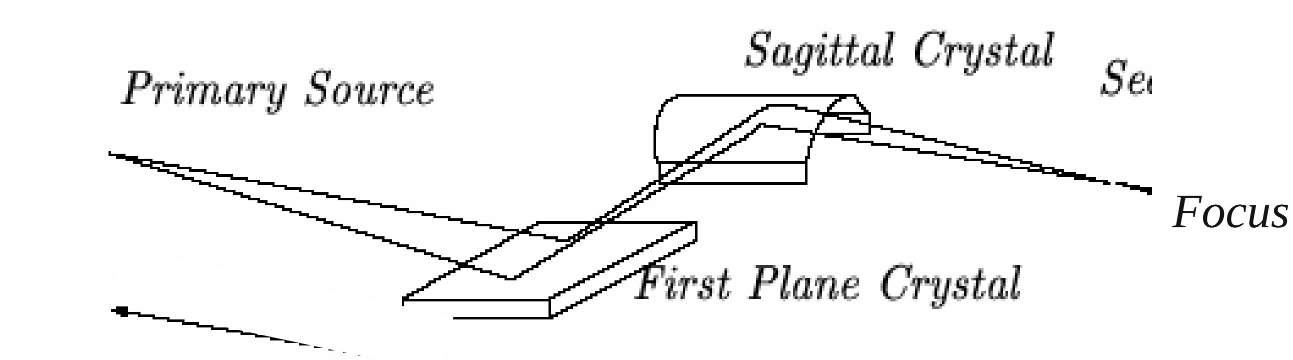
Double Crystal Monochromator

Use two crystals to minimize beam movement with angle change



For true fixed exit height need to change δ as angle changes

Horizontal focusing using sagittally bent crystal



Allows larger horizontal collection angle at higher energies

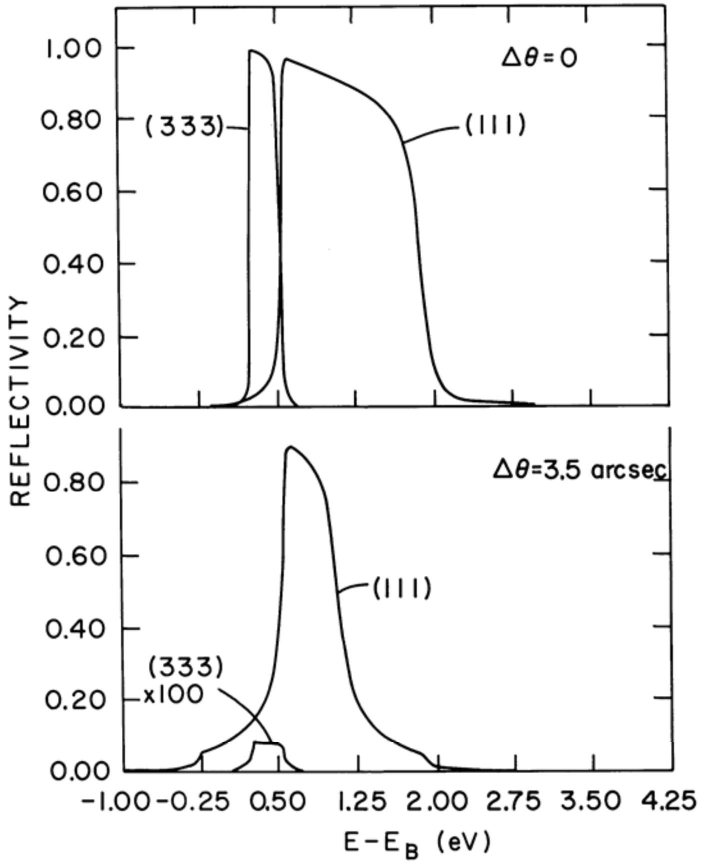
Crystal radius must change with angle

Anticlastic bending can be a problem – ribbed crystals

Detuning can be less effective at removing harmonics

Detuning

Detuning can be used to reduce the relative amount of harmonics in the beam



Note: detuning can also affect energy resolution

Some typical beamline layouts

- Monochromator only
  - Good for undulator, small beam size and divergence from source
- Monochromator with focusing mirror
- Collimating mirror – monochromator – focusing mirror
  - Typical setup for BM line, collimating mirror less useful at APS
- Collimating mirror – sagittal focusing mono – vertical focusing mirror
  - Can provide best flux from BM, may be difficult to optimize for spectroscopy

Detectors

- Signal to noise requirements
- Possible performance of ideal detectors
- Short description of some common detectors
  - Ion chambers
  - Multielement and deadtime issues
  - Filters and slits
  - Diffraction based detectors

S/N requirements

3 measurement regimes:

- Detection of element (imaging)
  - S/N > 10
  - 10^4 data points
- Near edge measurements
  - S/N > 100
  - 50-100 data points
- Extended fine structure (EXAFS)
  - S/N > 1000
  - 100-300 data points
- No background:
  - For EXAFS need >10^6 photons

$$S/N = \sqrt{\text{detected counts}}$$

Performance of Ideal Fluorescence Detector

- High flux beam provides > 10^{12} ph/sec
- For EXAFS need > 10^{6} signal counts/pt
- Fluorescence yield 20-50%

If the absorption from the element of interest is about 10^{-6} of the total, a spectrum can be acquired in a few seconds/pt.

Practical limitations

- Can't collect 4π
  - Good goal is 25% of 4π
- Fluorescence absorbed in sample
  - Negligible for surface or thin sample
  - Maybe factor of 5 for thick sample
- Radiation Damage

10^{-6} absorption still feasible in 1-2 hrs.

Example for Fe

- 10^{-6} absorption gives 3×10^{13} atoms/cm^2
  - small fraction of monolayer
- in solution:
  - 0.4 ppm by weight
  - 6 micromolar
- in Silicate mineral:
  - 5 ppm by weight

Current Detectors

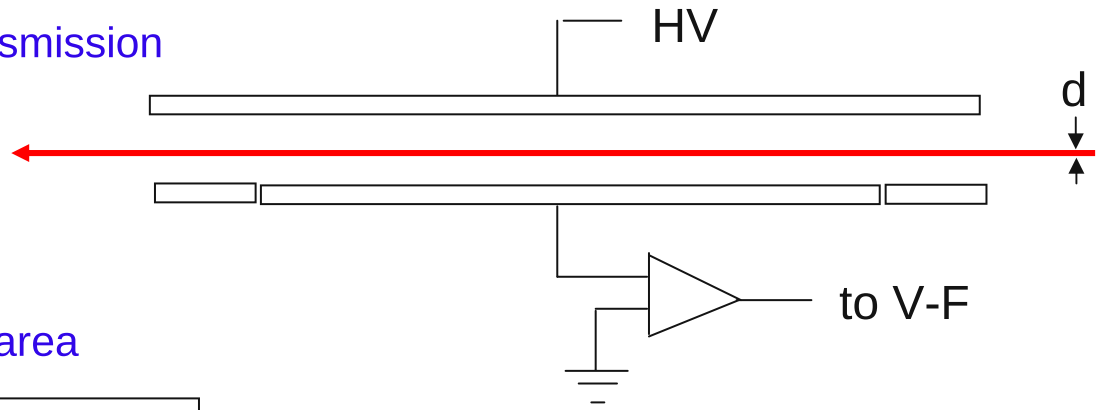
- Can be compared by effective count rate:

$$N_e = N_f \Big/ \sqrt{1 + N_b / N_f}$$

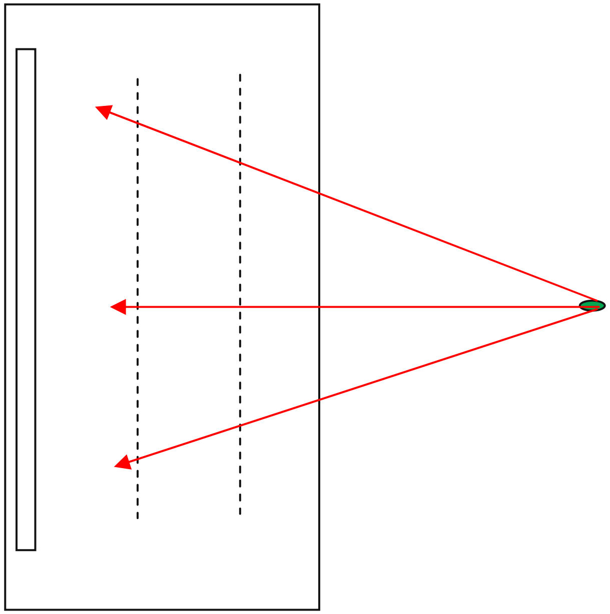
- Note: background scattering can be 1% of total absorption
- N_b can exceed 10^8 , ie $N_b/N_f \sim 100$
- Also need to consider total counting rate detector can accept

Ion chambers

transmission



Large area



Collection efficiency:

$$f = \frac{1}{1 + E^2/6} \qquad E = m\frac{d^2\sqrt{q}}{V}$$

m~30-40, V is volt/cm

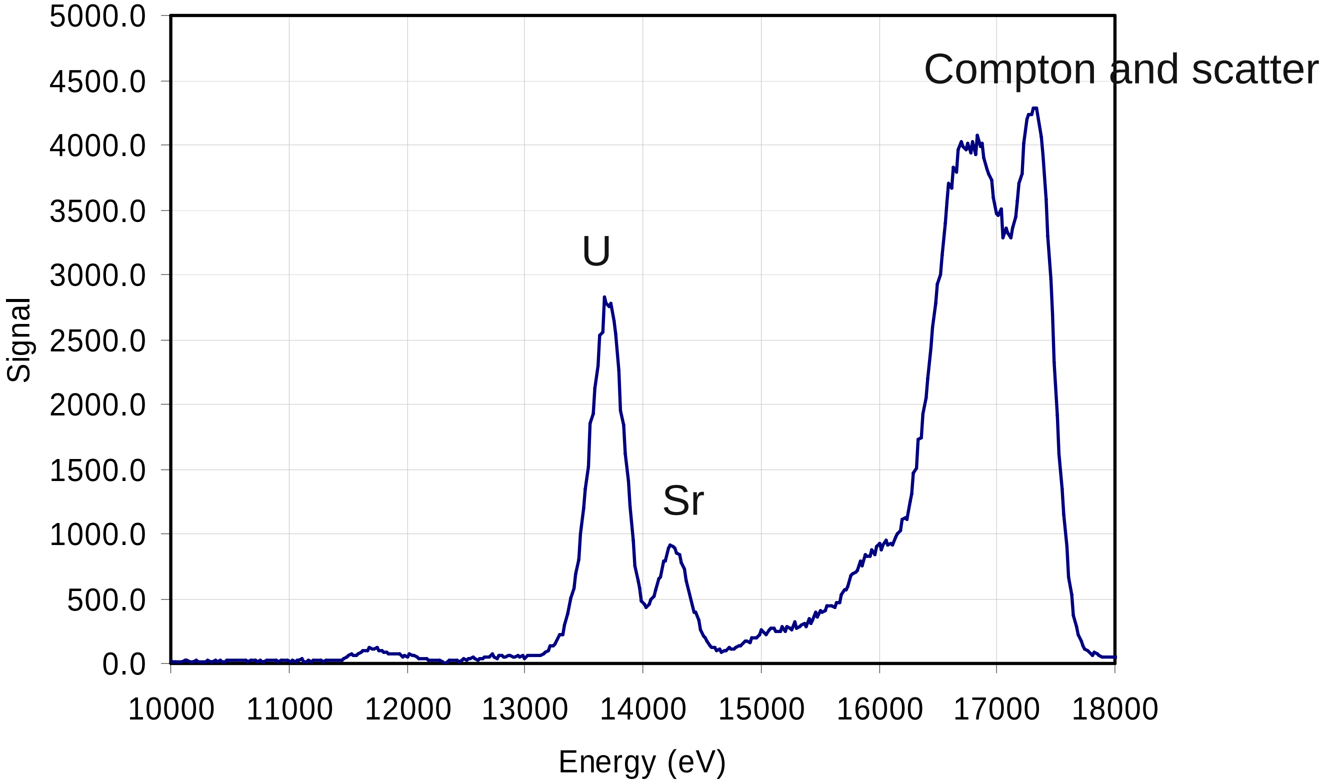
q is charge/cm^3

For linearity want E small
(V large and q small)

Multi-element solid state

- Resolution (fwhm) Ge: 200-300 eV, Si drift: 150-200 eV
- Individual element limited to few x 10^5
- Background or lower energy fluorescence lines can saturate countrate
- Standard arrays limited to about 30 elements (100 possible in monolithic arrays)

Typical spectrum- U contaminated Sediment



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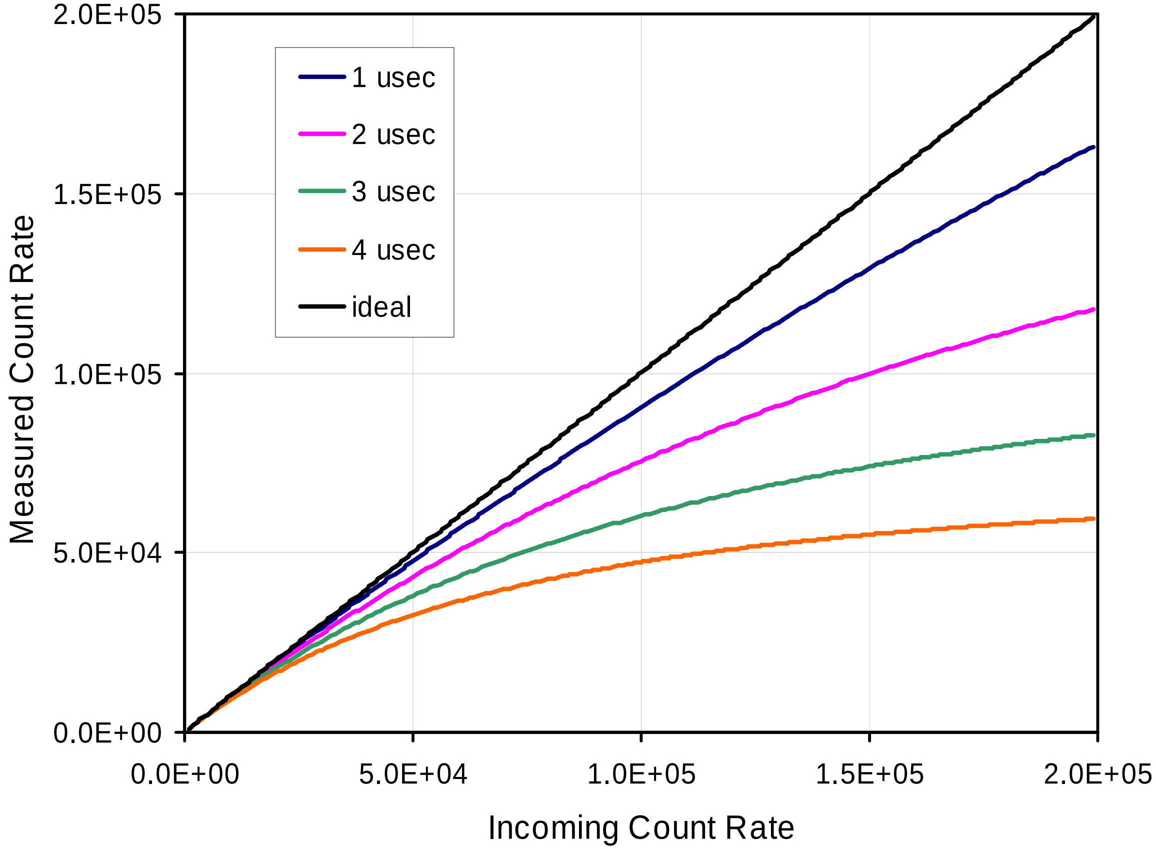
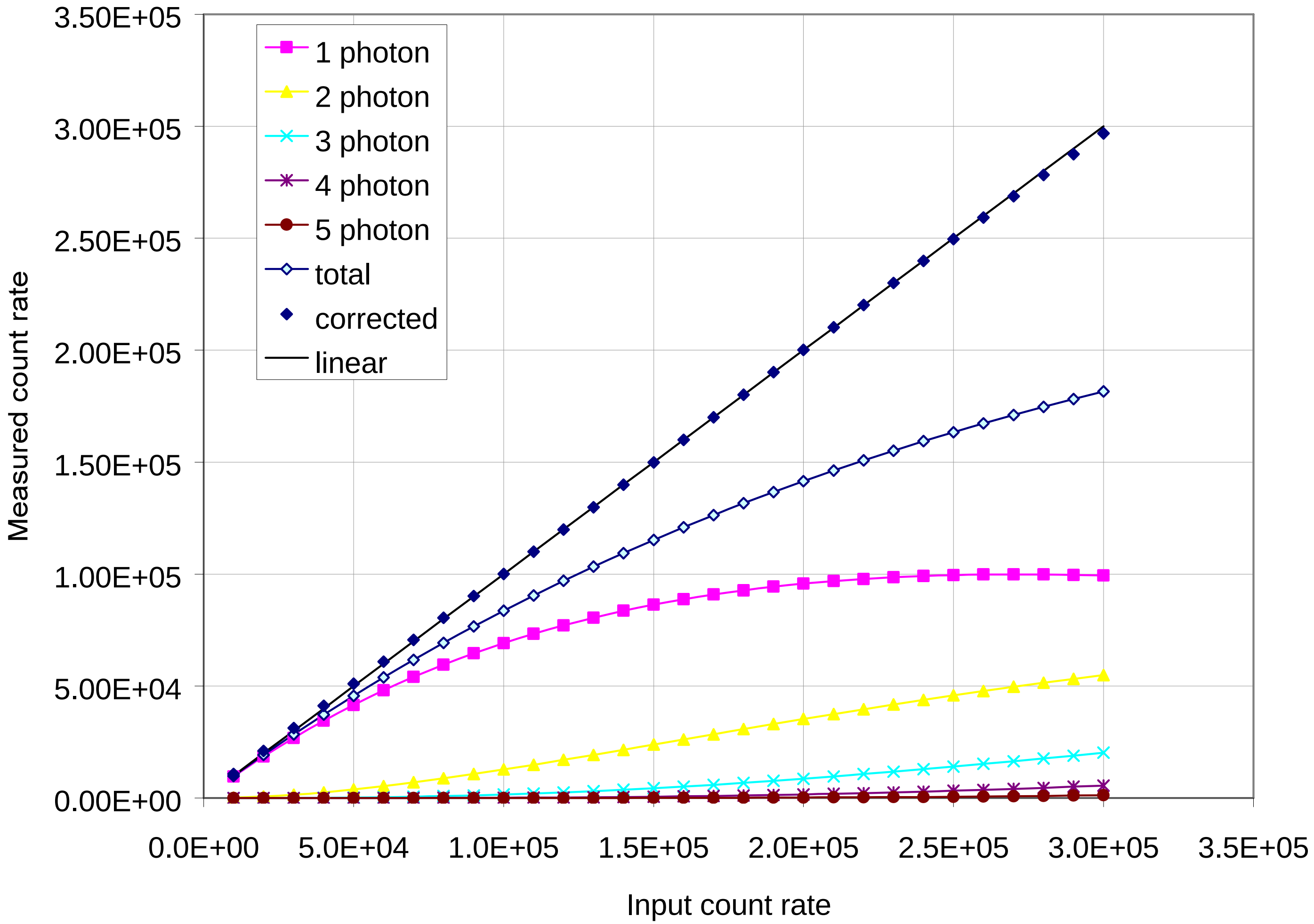
Dead time correction

Simple model:

MCR = ICR*Exp(-ICR*DT)

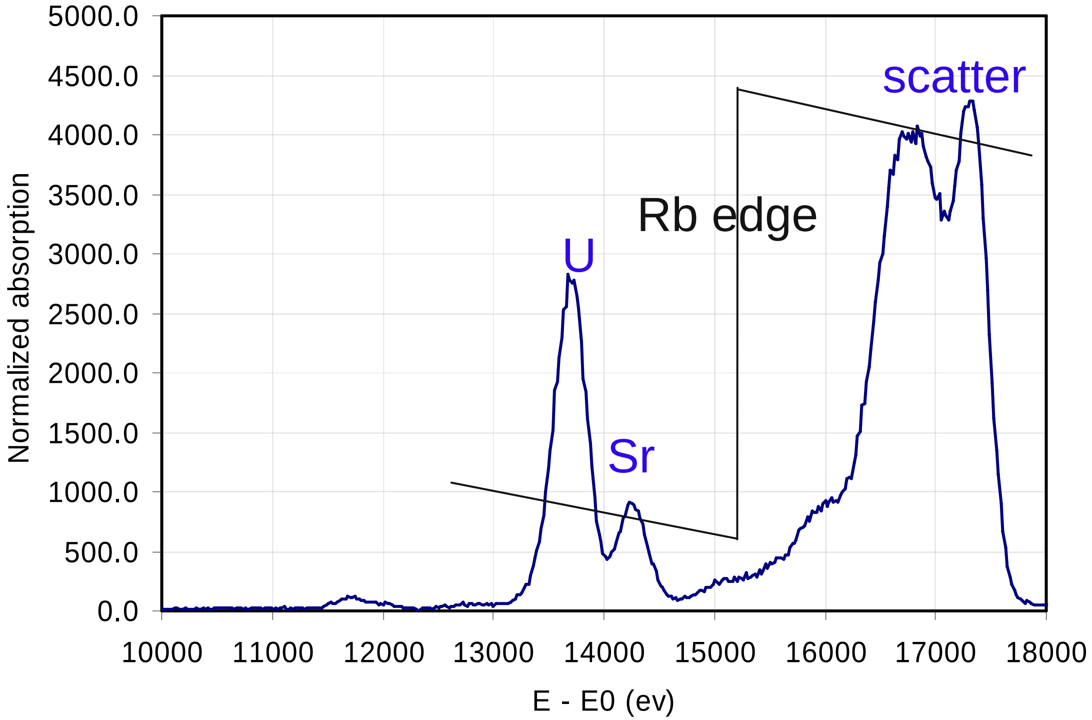


Pulsed source considerations



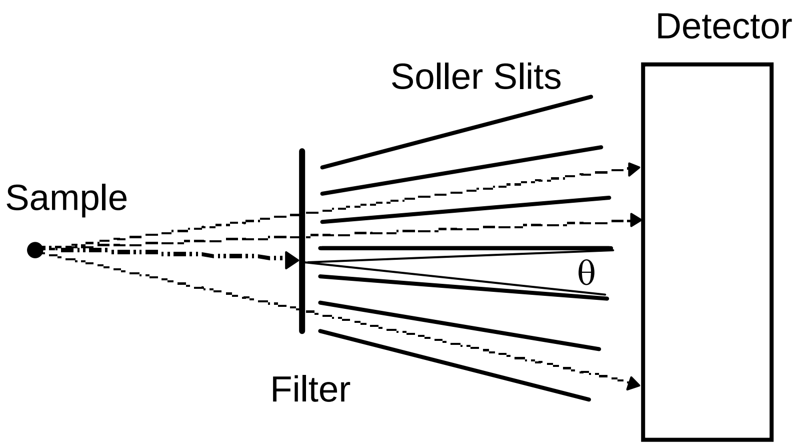
This is the single bunch case (272 kHz), for typical 24 bunch mode multiply the numbers by 24

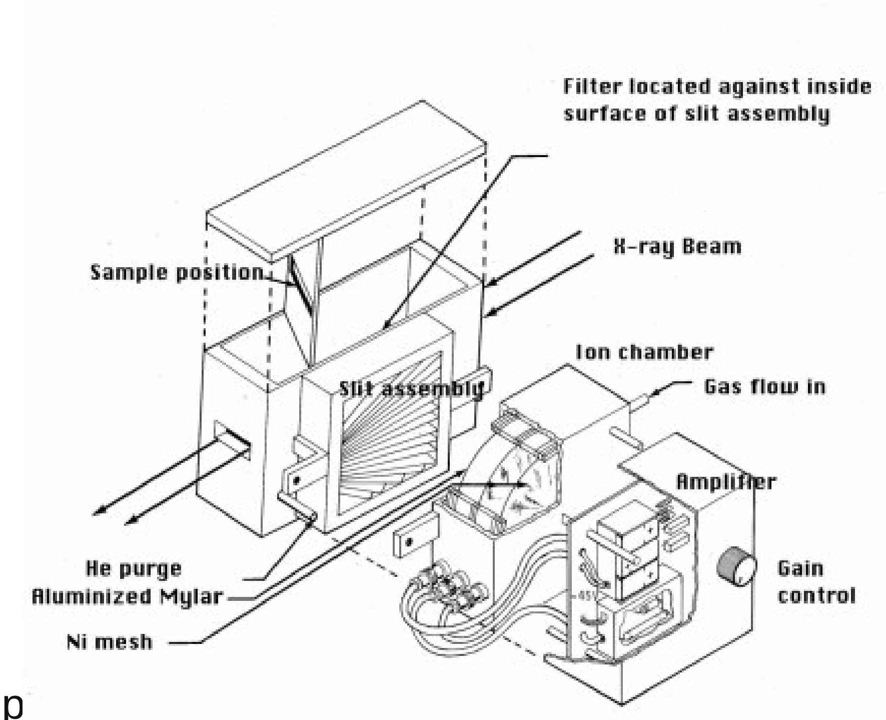
Filter can reduce the background in fluorescence measurement



Problem: Rb fluorescence can enter the detector

Filter-slits (Stern-Heald or Lytle detector)
see Stern and Heald, RSI 50, 1579 (1979)





- Large solid angle (large N_f)
- Unlimited count rate
- Moderate reduction in background – N_b still p
- Little rejection of lower energy fluorescence lines
- Near practical limits
- Works best for K edges above 4 keV

Diffraction based detectors

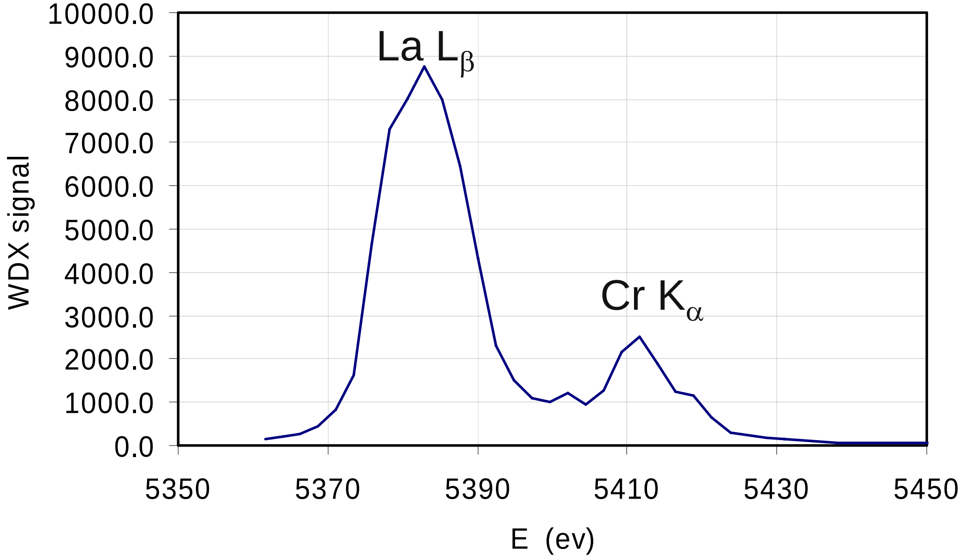
- Rowland circle, log-spiral (Bragg and Laue), multilayers
- Can have excellent resolution and background discrimination
- Unlimited count rates if integrating detectors used
- Usually require focused beam (0.1 mm)

WDX detector (Rowland circle)

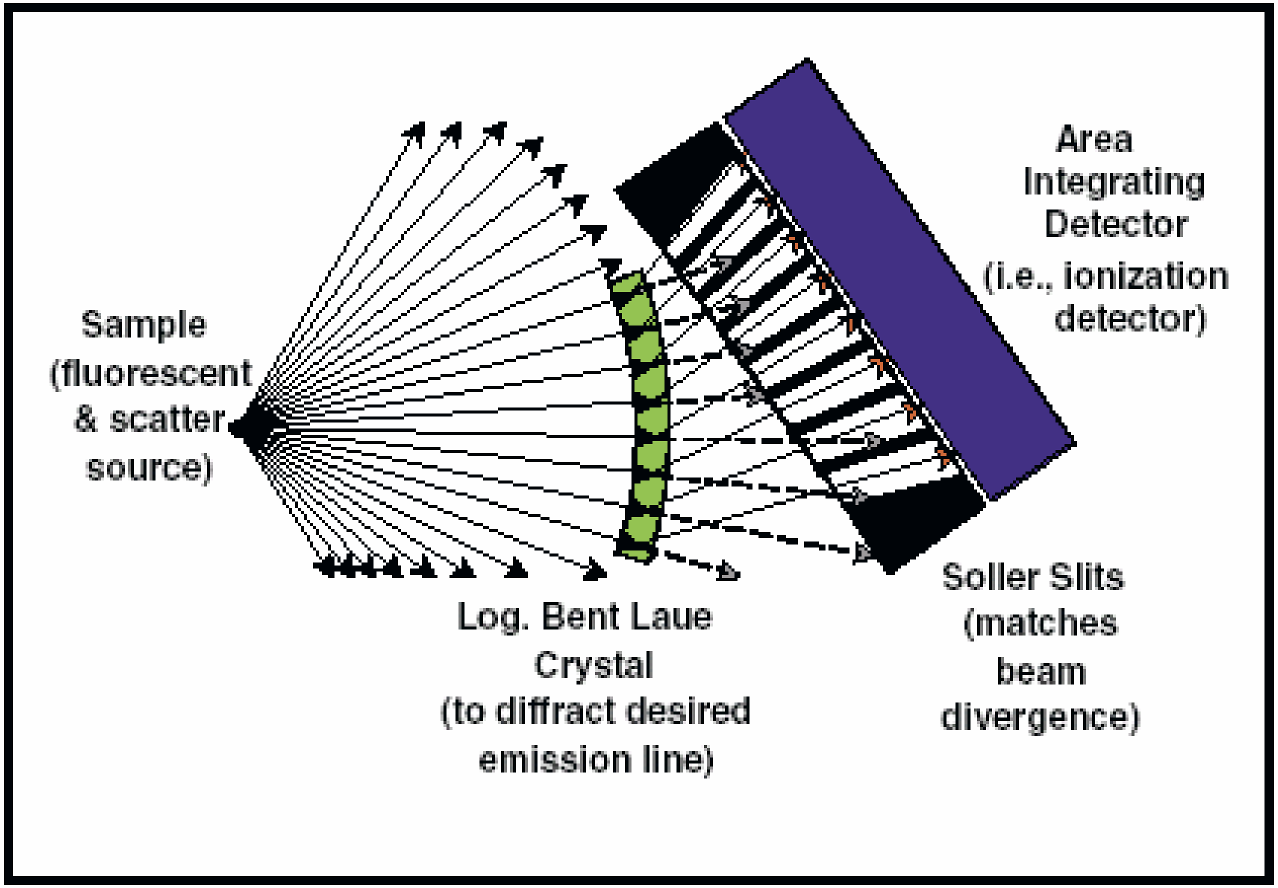
Very good energy resolution and background discrimination

Poor collection efficiency

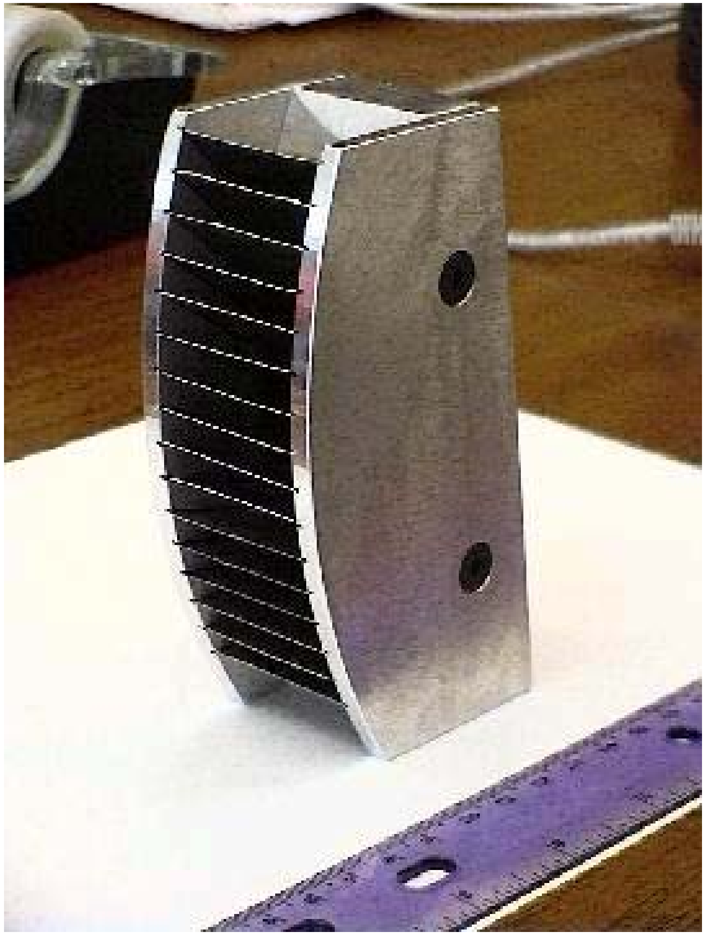
20 nm Cr doped TiO_2 on $LaAlO_3$



Log Spiral Laue detector

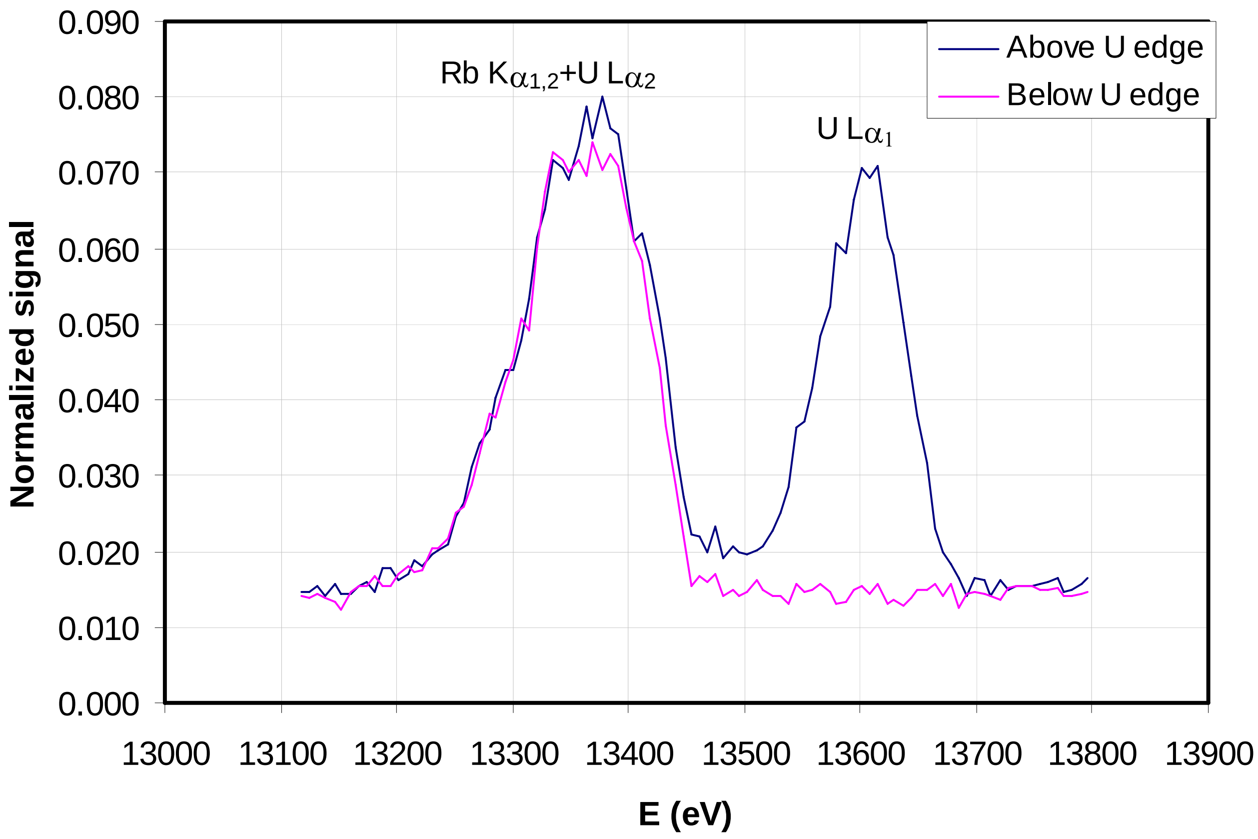


Log spiral detector (cont.)

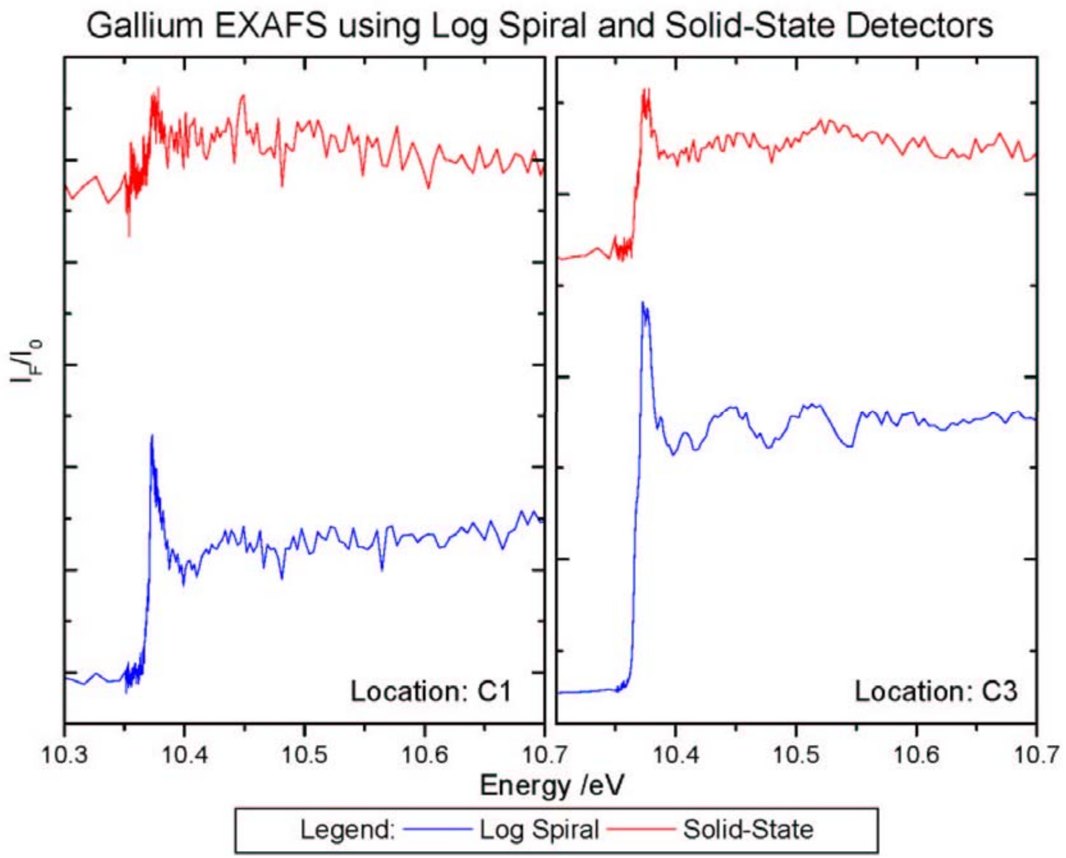


See: C. Karanfil, Z. Zhong, L.D. Chapman, R. Fischetti,G.B. Bunker, C.U. Segre, and B.A. Bunker, *SynchrotronRadiation Instrumentation, Eleventh U.S. NationalConference*, edited by P. Pianetta et al., Vol. **521**, pp. 178-182 (American Institute of Physics 2000).

Performance for detection of U



Detection of Ga in Nickel-Iron Meteorite
courtesy of Ron Cavell – Univ. of Alberta



Further development of both solid-state arrays and diffraction-based detectors warranted

- Solid state arrays:
  - More elements coming (100-400 under development)
  - Need to handle $>10^8$ hz
  - Preferable to keep resolution close to 200 eV
  - Si drift detectors look promising
- Diffraction based detectors:
  - Need to increase efficiency (multiple crystals)
  - Should strive for better resolution than solid state detectors
  - If above met, best bet for extreme diluteness
  - Need 0.1-0.2 mm high beam