

# Generation of X-Rays

(prepared by James R. Connolly, for EPS400-002, Introduction to X-Ray Powder Diffraction, Spring 2012)

## ***A Bit of History***

A good discussion of the early x-ray discoveries may be found in Chapter 1 of Moore and Reynolds (1997). I have borrowed freely from a variety of sources for this section. An online sketch of the early history of diffraction is at Cambridge University's Physics web site ([http://www-outreach.phy.cam.ac.uk/camphy/xraydiffraction/xraydiffraction\\_index.htm](http://www-outreach.phy.cam.ac.uk/camphy/xraydiffraction/xraydiffraction_index.htm)).

## **Röntgen's Discovery**

X-Rays were discovered by Wilhelm Röntgen in 1895, for which he was awarded the first Nobel Prize in Physics in 1901. The original records of Röntgen's experiments were lost, but a reconstruction of them in Moore and Reynolds (1997) is very interesting. Basically, he discovered that in an experiment with high-energy electrons, another type of radiation was being produced which had the following unique properties:

1. Travels in straight lines
2. Are exponentially absorbed in matter with the exponent proportional to the mass of the absorbing material
3. Darkens photographic plates
4. Make shadows of absorbing material on photosensitive paper

The 4<sup>th</sup> property led directly to the use of x-rays in medicine. In the years following Röntgen's discovery, much was learned about x-rays, and much of the wave vs. particle arguments about radiation which consumed science in the early part of the 20<sup>th</sup> century were rooted in studies of x-rays.

## **The Discovery of Diffraction**

Studies of x-rays revealed that they could be polarized, but could not be refracted, leading to controversy over whether x-rays were particles or waves. It was understood that if they were waves, the wavelength must be extremely small ( $10^{-10}$  meters or less). Max von Laue theorized that if x-rays could also be diffracted if the slits were small enough. Since it was understood that molecular spacings in crystalline materials were on the order of a tenth of a nanometer, he devised an experiment in which x-rays were allowed into a lead box containing a crystal, with sensitive film behind the crystal. When the films were developed there was a large central point from the incident x-rays, but also many smaller points in a regular pattern. These could only be due to the diffraction of the incident beam and the interference of many beams. By using a crystal as a diffraction grating, von Laue had proved the x-rays were not particles, but waves of light with very small wavelengths. He published his results in 1912.

Lawrence Bragg and his father W.H. Bragg used von Laue's discovery and, for monochromatic radiation, were able to show that diffraction could be treated geometrically like reflection. They derived Bragg's law, which allows diffraction to be treated in simple mathematical terms. The Bragg equation provides a simplified framework for diffraction that works for basic calculations:

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

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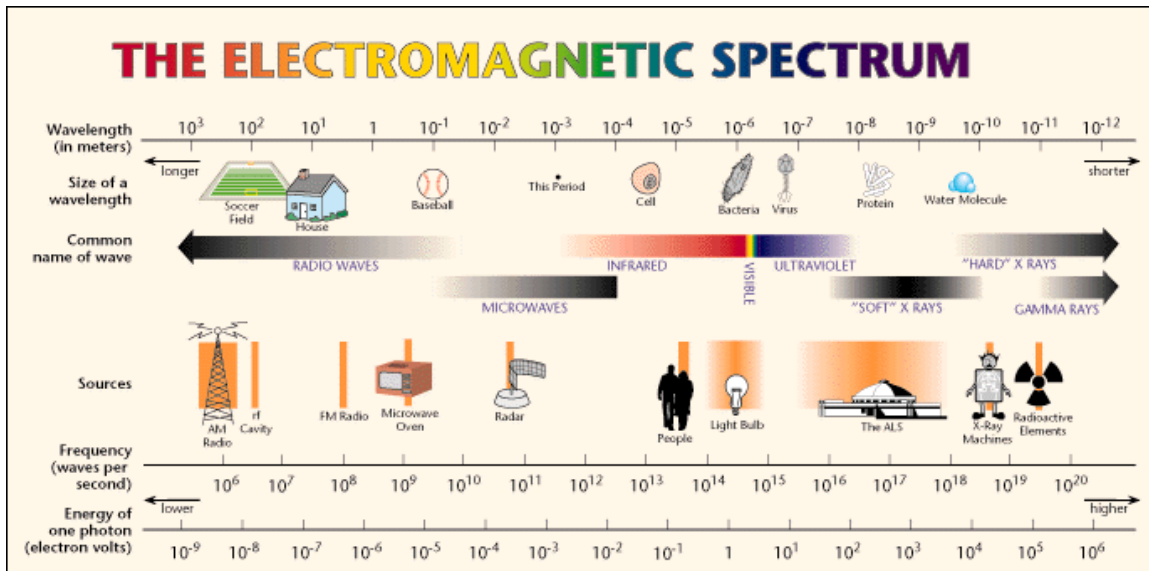
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where  $n$  is an integer  
 $\lambda$  is the wavelength of the X-radiation  
 $d$  is the interplanar spacing in the crystalline material and  
 $\theta$  is the diffraction angle

We will discuss the Laue and Bragg equations in more detail later in the first session on diffraction later in this class.

## The Electromagnetic Spectrum

X-Rays are part of the spectrum of electromagnetic radiation occupying the region between the ultraviolet and gamma rays. The full spectrum is shown graphically and in tabular format below:



## Spectrum of Electromagnetic Radiation

Region	Wavelength (Angstroms)	Wavelength (centimeters)	Frequency (Hz)	Energy (eV)
Radio	$> 10^9$	$> 10$	$< 3 \times 10^9$	$< 10^{-5}$
Microwave	$10^9 - 10^6$	$10 - 0.01$	$3 \times 10^9 - 3 \times 10^{12}$	$10^{-5} - 0.01$
Infrared	$10^6 - 7000$	$0.01 - 7 \times 10^{-5}$	$3 \times 10^{12} - 4.3 \times 10^{14}$	$0.01 - 2$
Visible	$7000 - 4000$	$7 \times 10^{-5} - 4 \times 10^{-5}$	$4.3 \times 10^{14} - 7.5 \times 10^{14}$	$2 - 3$
Ultraviolet	$4000 - 10$	$4 \times 10^{-5} - 10^{-7}$	$7.5 \times 10^{14} - 3 \times 10^{17}$	$3 - 10^3$

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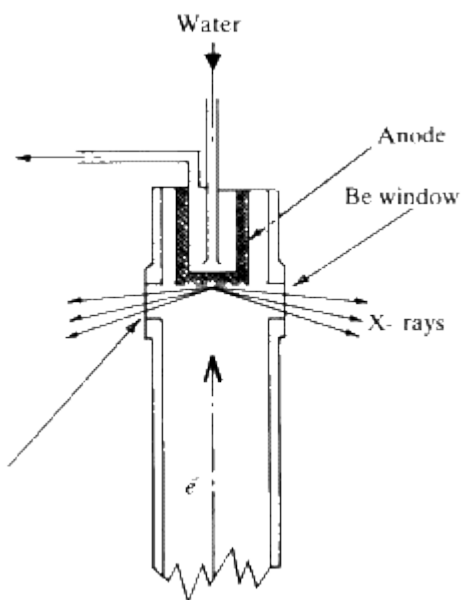
Region	Wavelength (Angstroms)	Wavelength (centimeters)	Frequency (Hz)	Energy (eV)
X-Rays	10 – 0.1	$10^{-7} - 10^{-9}$	$3 \times 10^{17} - 3 \times 10^{19}$	$10^3 - 10^5$
Gamma Rays	< 0.1	< $10^{-10}$	> $3 \times 10^{19}$	> $10^5$

A note on units: an angstrom (Å) is  $10^{-10}$  meters  
 a nanometer (nm) is  $10^{-9}$  meters  
 a micrometer (µm) or micron is  $10^{-6}$  meters  
 a millimeter (mm) is  $10^{-3}$  meters

The wavelengths of X-radiation commonly used for x-ray diffraction lie between 0.7 and 2.3 Å. This is very close to the interplanar spacings of most crystalline materials. The more penetrating radiation used for medical x-rays has a smaller wavelength.

### Generating X-rays for Powder Diffraction

To get an accurate picture of the structure of a crystalline material requires X-radiation that is as close to monochromatic as possible. The function of the x-ray tube and associated electronics is to produce a limited frequency range of high-intensity x-rays. Filters, monochromators, specially tuned detectors and software are then used to further refine the frequency of x-rays used in the analysis.



At left is a schematic of an x-ray tube similar to Cu tubes used in our laboratory. The (e<sup>-</sup>) arrow from the bottom indicates the direction of electrons generated from a tungsten filament (not in the diagram). The filament current is typically set in the range between 25 and 40 ma (milliamps).

The anode is a pure metal. Cu, Mo, Fe and Cr are in common use in XRD applications. In our lab we use a Cu tube. In one of the XRD systems in Chemistry, a Cr tube is used. Mo is in common use in metallurgical laboratories. The table below lists the common anodes in use in XRD and their particular advantages and disadvantages.

Cooling water (usually kept at about 20 °C) is circulated through the x-ray tube (and sprayed on the back side of the anode) to keep operating

temperatures low.

A high potential voltage (typically 30 to 40 KV) is maintained on the anode so that the generated electrons are accelerated and interact with it to generate x-rays. Electronics are usually designed so that the anode is maintained at ground and a high negative potential placed on the cathode (filament).

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Thin metal windows of a light metal (typically Be) that are effectively transparent to x-rays are used to allow x-rays generated to exit in the direction of the specimen and while maintaining the vacuum in the tube. X-ray tubes used for XRD typically have four Be windows to enable multiple ports for output. On our Scintag diffractometer (and most modern automated systems), only one window is used.

Common Anode Materials (abstracted from Jenkins and Snyder, 1996) are listed in the table below. Heading abbreviations used in the table are At. # (atomic number),  $K\alpha$  Å (characteristic  $K\alpha$  wavelength in Å), Char Min keV (characteristic minimum excitation potential in keV), and Opt kV (optimal operating kV for this anode type).

**Table 1: Characteristics of Common Anode materials**

Material	At. #	$K\alpha_1$ (Å)	$K\alpha_2$ (Å)	Char Min (keV)	Opt kV	Advantages (Disadvantages)
Cr	24	2.290	2.294	5.99	40	High resolution for large d-spacings, particularly organics (High attenuation in air)
Fe	26	1.936	1.940	7.11	40	Most useful for Fe-rich materials where Fe fluorescence is a problem (Strongly fluoresces Cr in specimens)
Co	27	1.789	1.793	~7.6	40	Useful for Fe-rich materials where Fe fluorescence is a problem
Cu	29	1.541	1.544	8.98	45	Best overall for most inorganic materials (Fluoresces Fe and Co $K\alpha$ and these elements in specimens can be problematic)
Mo	42	0.709	0.714	20.00	80	Short wavelength good for small unit cells, particularly metal alloys (Poor resolution of large d-spacings; optimal kV exceeds capabilities of most HV power supplies.)

### Generation of X-rays

Much of this section is abstracted from Jenkins and Snyder (1996), with parts from Moore and Reynolds (1997).

X-rays are short-wavelength, high-energy electromagnetic radiation, having the properties of both waves and particles. They can be described in terms of both photon energy ( $E$ ) or wavelength,  $\lambda$  (lambda – the distance between peaks) and frequency  $\nu$  (nu – the number of peaks passing a point in a unit of time). The relationships between these quantities are expressed in the following equations:

$$\nu = \frac{c}{\lambda} \quad (1)$$

and

$$E = h\nu \quad (2)$$

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where  $E$  is the energy of the electron flux in KeV

$h$  is Planck's constant ( $4.135 \times 10^{-15}$  eVs)

$c$  is the speed of light ( $3 \times 10^{18}$  Å/s)

$\lambda$  is the wavelength in Å

Substituting the first equation in the second yields:

$$E = \frac{hc}{\lambda} \quad (3)$$

which describes the energy of x-rays in terms of their wavelength. Substituting the values of the constants above in the equation yields the following relationship:

$$E = \frac{12.398}{\lambda} \quad (4)$$

Immediately apparent from this equation is that there is an inverse relationship between the energy and wavelength of x-rays.

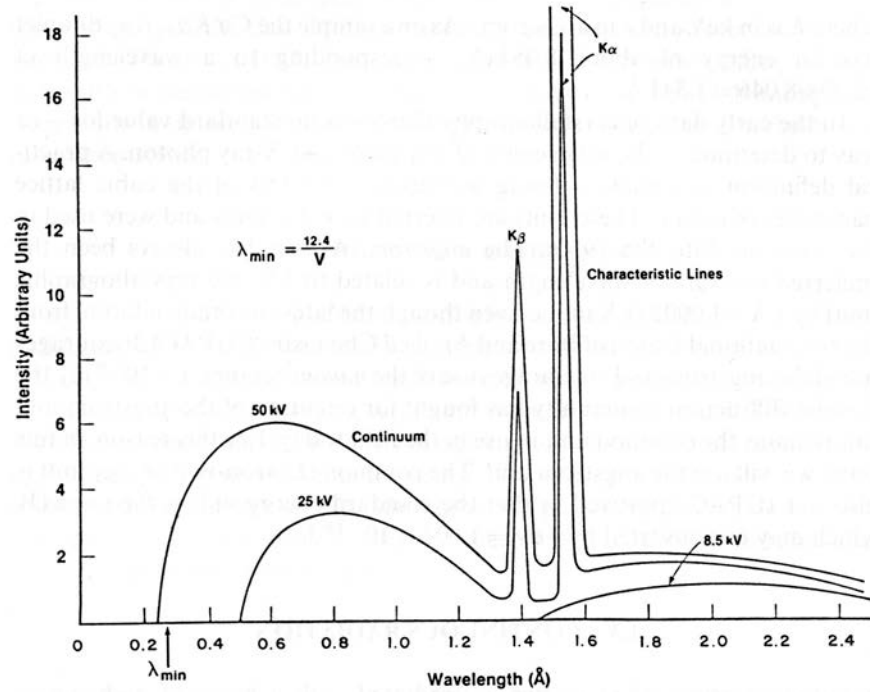
### Continuous Radiation

X-rays are produced whenever matter is irradiated with a beam of high-energy charged particles or photons. Interactions that occur between the beam (i.e., electrons) and target will result in a loss of energy. Since energy must be conserved, the energy loss results in the release of x-ray photons of energy (wavelength) equal to the energy loss (defined by Equation 4). This process generates a broad band of continuous radiation (a.k.a. bremsstrahlung or white radiation). This (along with the higher-intensity characteristic lines) is shown in the figure below (for a Cu target at a variety of kV) as the low-intensity spectrum.

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**Figure 1: Continuous and Characteristic Radiation for Copper**



Continuous and characteristic radiation for copper.

Note that for any given accelerating potential (8.5, 25 and 50 kV are shown below), there is a minimum wavelength of x-rays generated. The minimum wavelength at which the continuum begins is related to the maximum accelerating potential of the beam of electrons directed at the target,  $V$ , by the following equation (derived from Equation 4 above):

$$\lambda_{\min} = \frac{hc}{V} = \frac{12.398}{V} \quad (5)$$

Note from the shape of the curve that the continuum reaches a maximum intensity at a wavelength of about 1.5 to 2 times the  $\lambda_{\min}$ .

The sharp peaks labeled as characteristic lines on the diagram are not present at the continuum at 8.5 kV, but are present in the curve at 25 kV. The characteristic lines (discussed in the next section) only will appear when the critical excitation potential for the anode in use (listed in Table 1 above) is exceeded.

## Characteristic Radiation (and “Filtering”)

Successful analysis of powders by x-ray diffraction requires the Bragg diffraction condition to be met, which, in turn, requires high-intensity monochromatic radiation. In the standard powder diffractometer, this radiation is obtained by the generation of very high-intensity “characteristic” x-rays of known wavelength with an x-ray tube. The characteristic radiation (which consists of several wavelengths) along with the continuous spectrum is then “filtered” by means of an absorptive filter, a crystal monochromator, a wavelength-selective detector or a combination of these devices. Both of the generation of characteristic x-rays and the filtering processes will be addressed in this section.

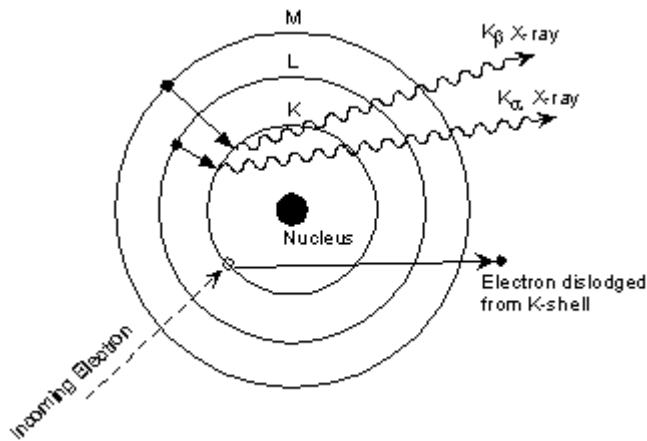
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## Generation of Characteristic X-rays

The simplified discussion below uses Cu as an example. The same process applies to all of the anodes listed in Table 1 above. The quantum physics related to the generation of x-rays is, in reality, much more complicated, but this simplified discussion works for our purposes. See Jenkins and Snyder (1997), Nuffield (1966) or any XRD text for a more rigorous discussion.

If a high-energy electron or photon interacts with electrons in the target anode atoms such that an inner orbital electron is displaced, an electron from a lower energy shell will “jump” to replace the displaced higher-energy electron. This results in the generation of an x-ray photon of quantum energy equal to that of the transition. The relationship between the energy of this x-ray and its wavelength is described by equation (3) above.



This is shown schematically in the diagram at left. An incoming electron displaces a K-shell electron. If an L-shell electron moves to replace it, a  $K\alpha$  x-ray is produced. If an M-shell electron moves to replace it, a  $K\beta$  x-ray is produced.

The selection rules of quantum transitions in the atom indicate what transitions will occur. These transitions are shown diagrammatically in the figure

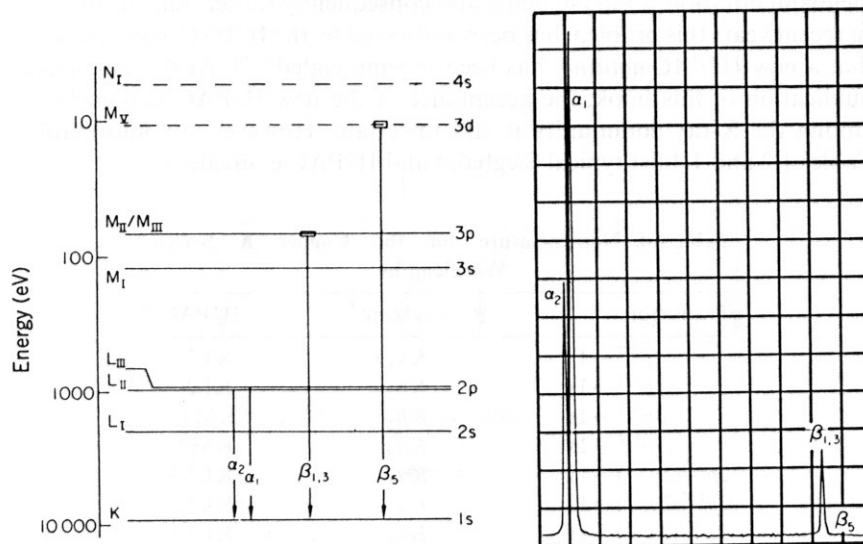
below. Two discrete energy “shifts” occur in the L- to K-shell levels transition, yielding  $K\alpha_1$  (8.045 keV, 1.5406Å) and  $K\alpha_2$  (8.025 keV, 1.5444Å) x-rays. The intensity of the resultant x-rays is related to the number of times the particular transition occurs. The  $K\alpha_1$  transition will occur almost exactly twice as frequently as the  $K\alpha_2$  transition, thus the resultant x-rays have twice the intensity.

Although three M to K transitions can occur in Cu, the energy difference between two of them is so small that  $K\beta_1$  and  $K\beta_2$  (8.903 keV, 1.3922Å) x-rays are effectively monochromatic, and the intensity of the  $K\beta_5$  transition is low enough to be ignored.

In the diagram below the energies of the various possible energy transitions are shown at left, and the resultant characteristic x-ray spectrum at right. On the chart at right, the y-axis is intensity and the x-axis is energy (increasing to right) or wavelength (increasing to left).

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The copper  $K\alpha$  spectrum.

***It is important to be clear about the distinction between wavelength (energy) and intensity.*** Since the  $K\beta$  ( $M \rightarrow K$ ) transition involves a larger energy transition, the resultant x-rays have higher energy (lower wavelength) than the  $K\alpha$  ( $L \rightarrow K$ ) x-rays.  $K\alpha$  transition, however, occurs much more frequently, thus the resultant x-rays have a much higher intensity.

Figure 1 (in the previous section) shows the characteristic and continuous spectrum for a Cu-anode x-ray tube at a variety of accelerating voltages. Note how much the intensity of the  $K\alpha$  and  $K\beta$  x-rays exceed that of the continuum at 25 keV, and that at 50 keV the  $K\alpha_1$  peak is way off the top of the intensity scale.

Each of the common anode elements will tend to have an optimum operating voltage in which the intensity of the characteristic  $K\alpha$  radiation will greatly exceed that of the continuum; these optimum values are listed for the common anode materials in Table 1.

The x-ray tube will produce several different characteristic-energy radiations plus the continuum. Techniques for making that “raw” radiation into the monochromatic radiation needed for powder diffraction are discussed in the next section.

## ***Making Monochromatic X-rays***

There are a number of ways that x-rays “out of the tube” can be modified such that the radiation detected after diffraction is only  $K\alpha$ . Some of these methods include:

- Use of a  $\beta$  filter
- Use of proportional detector and pulse height selection
- Use of a Si(Li) solid-state detector
- Use of a diffracted- or primary-beam monochromator

These methods will be discussed briefly below. Most texts discuss these methods. The treatment by Ron Jenkins in Bish and Post (1989) is very concise; Jenkins and Snyder (1997)

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discuss this material in more detail. The material in this section is abstracted from both sources.

Though the x-ray spectrum is more complicated, we are mostly concerned with the strong characteristic lines generated by our x-ray source. These lines are listed below for the common anodes:

**Table 2: Characteristic Wavelength values (in Å) for Common Anode Materials\***

Anode	$K\alpha_1$ (100)	$K\alpha_2$ (50)	$K\beta$ (15)
Cu	1.54060	1.54439	1.39222
Cr	2.28970	2.29361	2.08487
Fe	1.93604	1.93998	1.75661
Co	1.78897	1.79285	1.62079
Mo	0.70930	0.71359	0.63229

\* Relative intensities are shown in parentheses

There is no effective way to remove the  $K\alpha_2$  peak from the incident or diffracted beam. At low angles in the diffractogram, the  $K\alpha_1 - K\alpha_2$  doublet is poorly resolved and will be reflected as a slight asymmetry of the diffraction peak. At higher angles, the doublet will be resolved into two discrete peaks as the angular dispersion of the doublet increases. The known angular dispersion of the doublet and the intensity relationship (the  $\alpha_2$  peak is always about  $\frac{1}{2}$  the intensity of the  $\alpha_1$  peak) is used in analytical software (like MDI's Jade) to digitally filter the  $K\alpha_2$  peak from the resultant diffractogram.

## **$\beta$ Filters (and detector "tuning")**

Filtration is a process by which undesirable characteristic radiation is preferentially absorbed by a filtering material while most of the desired characteristic radiation is passed.

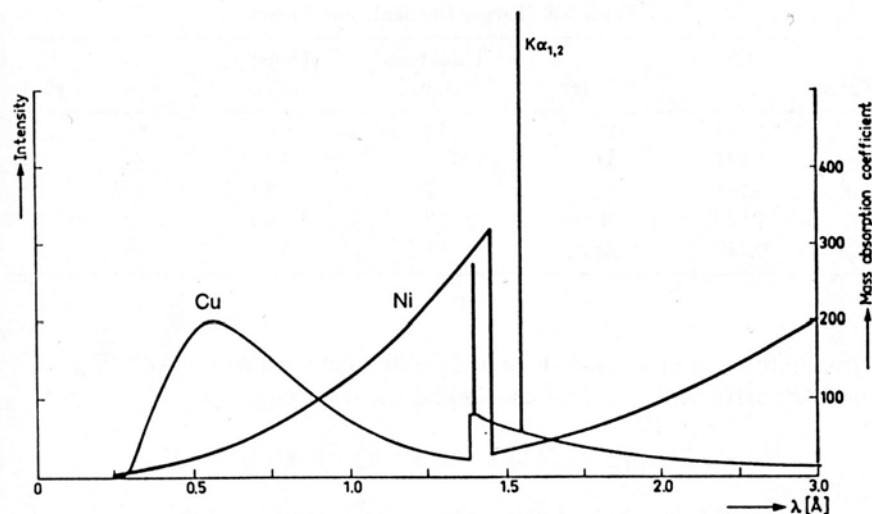
Absorption and absorption edges are discussed extensively in Jenkins and Snyder (1996) from which most of this discussion is abstracted.

All materials absorb x-rays, and several processes operate to produce the mass absorption response of a given material to x-rays. One type of absorption is fundamentally linear and dependent primarily to electron density (mass) of the absorber. Another type is photoelectric absorption, which is related to secondary excitation of orbital electrons in the absorber. This type of absorption is non-linear and varies significantly depending on the wavelength of the x-rays; the point in the wavelength spectrum to which absorption rises gradually and then drops abruptly is called an *absorption edge*. By selection of an element for a filter whose absorption edge lies between the characteristic  $K\beta$  and  $K\alpha$  wavelength, the  $K\beta$  radiation can be greatly attenuated while most of the  $K\alpha$  radiation is passed.

A Ni filter is used to filter Cu radiation. Below is the x-ray spectrum for a Copper target x-ray source overlain by the absorption curve for Ni. When used as a filter, the resultant intensity of the transmitted Cu radiation will be the Cu spectrum minus the Ni absorption curve.

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Use of the  $K\beta$ -filter. Reprinted from R. Jenkins and J. L. de Vries, *An Introduction to Powder Diffractometry*, p. 14, Fig. 12. Copyright © 1977, N. V. Philips, Eindhoven, The Netherlands.

The table below (from Jenkins and Snyder, 1997) lists the common anode (target) elements and their  $\beta$ -filtering elements. It is common practice in the industry to choose a filter thickness that yields a  $K\alpha:K\beta$  intensity ratio between 50:1 and 25:1.

Target	$K\alpha$ (Å)	$\beta$ -filter	Thickness ( $\mu\text{m}$ )	Density (g/cc)	% $K\alpha$ passed	% $K\beta$ passed
Cr	2.291	V	11	6.00	58	3
Fe	1.937	Mn	11	7.43	59	3
Co	1.791	Fe	12	7.87	57	3
Cu	1.542	Ni	15	8.90	52	2
Mo	0.710	Zr	81	6.50	44	1

With a diffractometer, if a filter is used, may be placed on the tube or detector side of the specimen, though the tube-side placement tends to be more common. For Debye-Scherrer powder cameras the filter will be between the source and the camera.

As will be noted from the diagram above, while the  $K\beta$  radiation is very effectively filtered, a significant amount of the high-energy portion of the continuous spectrum will be passed. In film work this can be a problem and lead to significant fogging of the film; this is one reason that generators used for powder camera work are commonly operated at lower than “optimum” accelerating voltages (i.e., 25 or 30 kV instead of 40 or 45 kV).

In the powder diffractometer, pulse height discrimination in the detector electronics can be used to limit the energy of x-rays seen (i.e., detected) by the detector, effectively removing the high-energy continuum and radiation produced by sample fluorescence. This method is

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used on our Scintag PAD V diffractometer in combination with a detector-side monochromator.

Some detector types of detectors (most commonly Si(Li) detectors<sup>1</sup>) allow very fine frequency discrimination or tuning such that it only “sees” a particular wavelength, usually only  $K\alpha$ . This eliminates the need for filters or monochromators and produces data with very high peak intensity and reduced background. The ability to tune the detector to a particular wavelength also allows the use of lower intensity but spectrally simpler  $K\beta$  radiation in experiments. Si(Li) detectors are in widespread use in large high-production industrial and commercial laboratories. Their disadvantages lie in their initial higher expense, requirements for special cooling while operating and higher dead time in relation to scintillation counters. The advantages and disadvantages of different types of detectors will be discussed later in this course.

## Monochromators

The most common way x-rays are filtered on diffractometers, particularly those using scintillation detectors, is by use of a monochromator in either the primary or diffracted beam. These devices make use of the fact that each component wavelength of a polychromatic beam of radiation directed at a single crystal of known orientation and d-spacing will be diffracted at a discrete angle (following the Bragg law). By choosing a crystal with the d-spacing to focus only the desired radiation (usually  $K\alpha$ ), undesired radiation of all other wavelengths is dispersed and does not enter the detector.

Whereas a filter selectively attenuates  $K\beta$ , a monochromator selectively *passes* the desired wavelength and attenuates everything else. High-level bremsstrahlung and other errant radiation from anode contamination (except that in the energy “band” passed by the monochromator) will not get to the detector. This allows the operation of the x-ray generator optimal accelerating voltages without significantly increasing background “noise” in the detected signal.

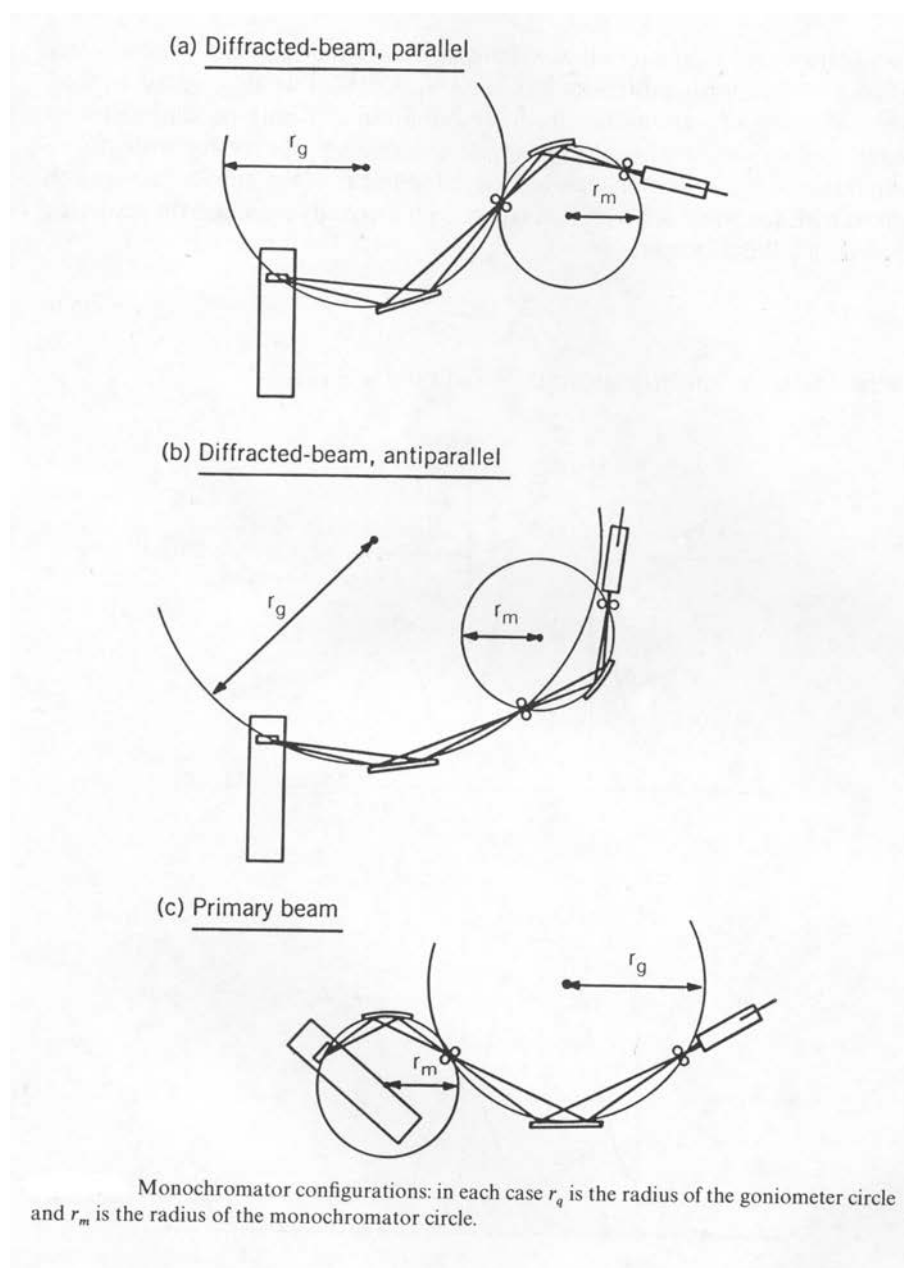
Possible geometric configurations of monochromators in the beam path are shown in the diagram below and include (a) diffracted beam parallel, (b) diffracted beam antiparallel, and (c) primary beam. In general the diffracted beam parallel configuration is most common; it passes a strong characteristic signal because of minimal path from monochromator to detector, and will remove radiation due to sample fluorescence. In the diagrams below (from Jenkins and Snyder, 1996)  $r_m$  is the radius of the monochromator circle and  $r_g$  is the radius of the diffractometer circle.

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<sup>1</sup> Si(Li) is shorthand for Lithium-drifted Silicon, commonly pronounced “silly”.

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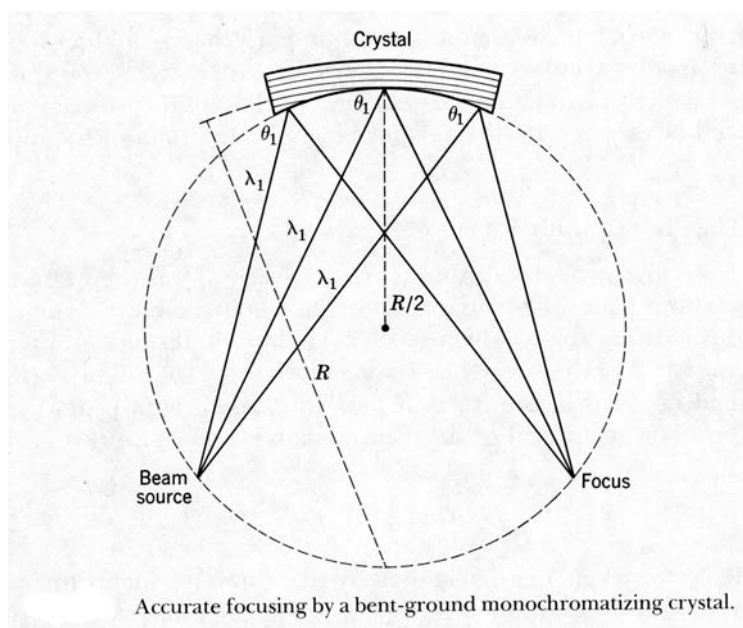
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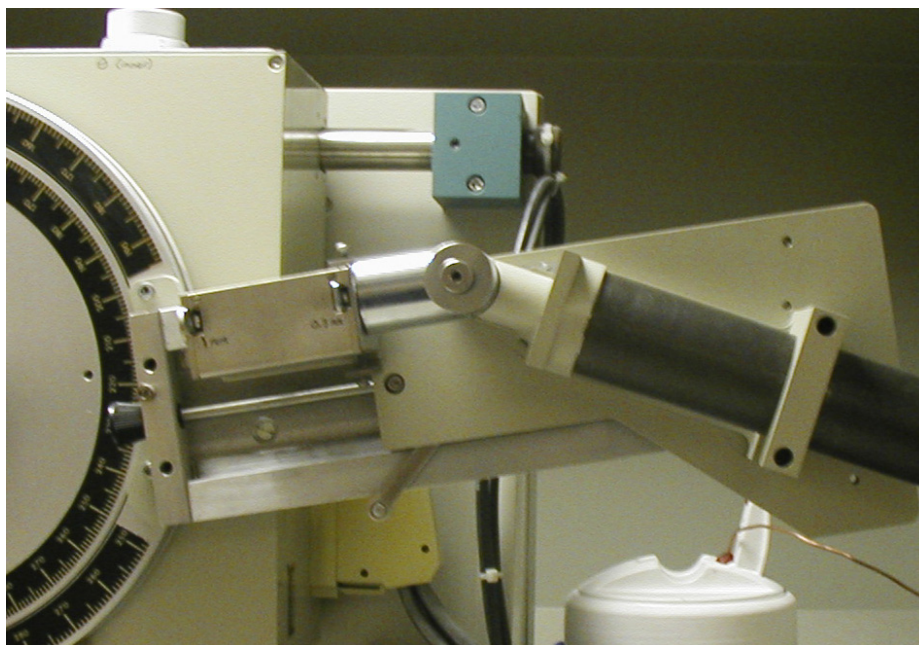
A planar crystal used in a monochromator will tend to diffract the selected wavelength over a very narrow range of incident angles, and will not be very effective in delivering a strong signal to the detector. For this reason, precisely deformed and machined synthetic curved crystals were developed. The **pyrolytic graphite curved crystal monochromator** is the most widely used crystal monochromator in use. The diagram below (from Klug and Alexander, 1974) shows how the monochromator is used to focus the beam. By varying the position of the crystal and the receiving slits within the monochromator assembly, the wavelength passed may be modified. In general, to maximize intensity of the diffracted signal, it is set to pass all  $K\alpha$  radiation, and to not pass  $K\beta$  radiation.

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Our Scintag PAD V system has a diffracted beam parallel monochromator utilizing a synthetic curved graphite crystal. The photograph below shows the receiving slits, monochromator and scintillation detector. Note that the monochromator radius is equal to the distance between the receiving slit (closest to the detector) and the center of the monochromator, and between the center of the monochromator and the detector.

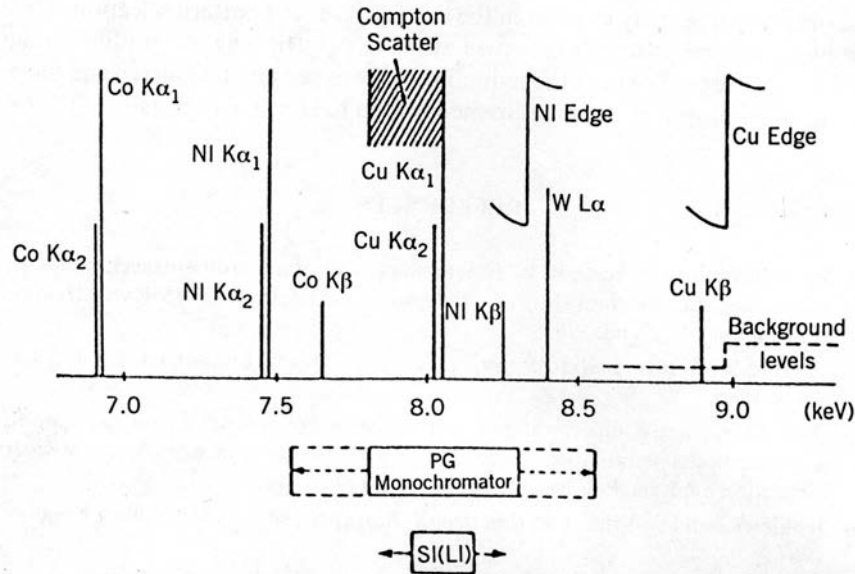


### Summary

The diagram below (Jenkins and Snyder, 1996) shows the spectral distribution of radiation around the characteristic Cu K-lines plotted as energy vs. intensity.

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Relative bandpass regions of various monochromatization devices. From R. Jenkins, Instrumentation. In *Modern Powder Diffraction* (D. L. Bish and J. E. Post, eds.), p. 37, Fig. 14. Mineralogical Society of America, Washington, DC, 1989. Reprinted by permission.

The characteristics of the primary methods of delivering monochromatic radiation to the diffractometer detector as shown graphically in the diagram are summarized below:

- A Si(Li) detector can be “tuned” to selectively detect only Cu K $\alpha$  radiation, and ignore virtually everything else.
- A pyrolytic graphite (PG) monochromator can similarly select Cu K $\alpha$ , however the window is somewhat wider than a Si(Li) detector and depending on settings and acceptance windows will also detect a few other wavelengths. The only possibly significant radiation passed is the tungsten (W) L $\alpha$  line; this line may be present as anode contamination in an “aged” Cu x-ray tube.
- A Ni filter will selectively attenuate Cu K $\beta$  radiation, but will pass almost everything else (including high-energy portions of the background spectrum).

Most diffractometer systems use a PG monochromator and use some type of pulse height discrimination to achieve effectively monochromatic x-rays.  $\beta$  filters are not in widespread use in modern diffractometers but are still used in some powder camera work. The extreme wavelength sensitivity of the Si(Li) detector permits the elimination of filters and monochromators and can produce data with extremely high signal to noise ratios and excellent wavelength resolution.