Analytical Methods for Materials

Lesson 20

Components in an X-ray Diffractometer

Suggested Reading

Chapter 6 in Cullity & Stock

Chapter 2 in Brandon & Kaplan

Geometry of the X-ray Diffractometer

- Generically, diffractometers consist of:
  - X-ray source
  - X-ray detector
  - Specimen to be examined
  - Other things
    - Monochromators
    - Filters
    - Slits
    - Etc…

D.J. Dyson, X-ray and Electron Diffraction Studies in Materials Science, Maney Publishing, London (2004)

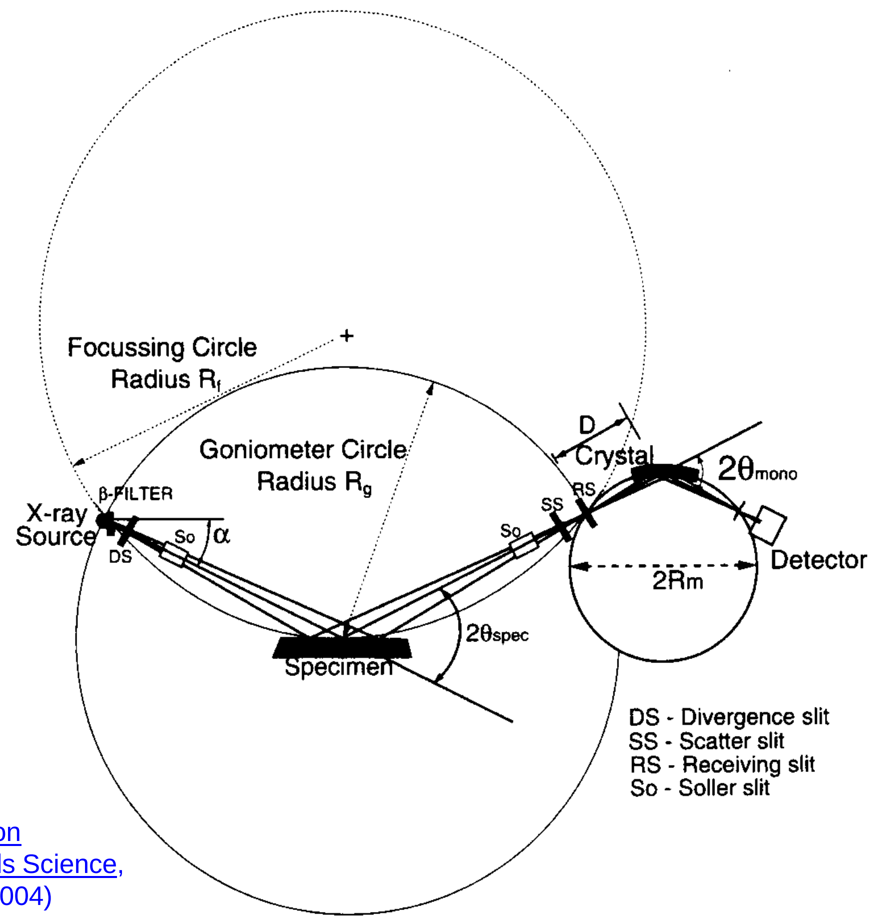
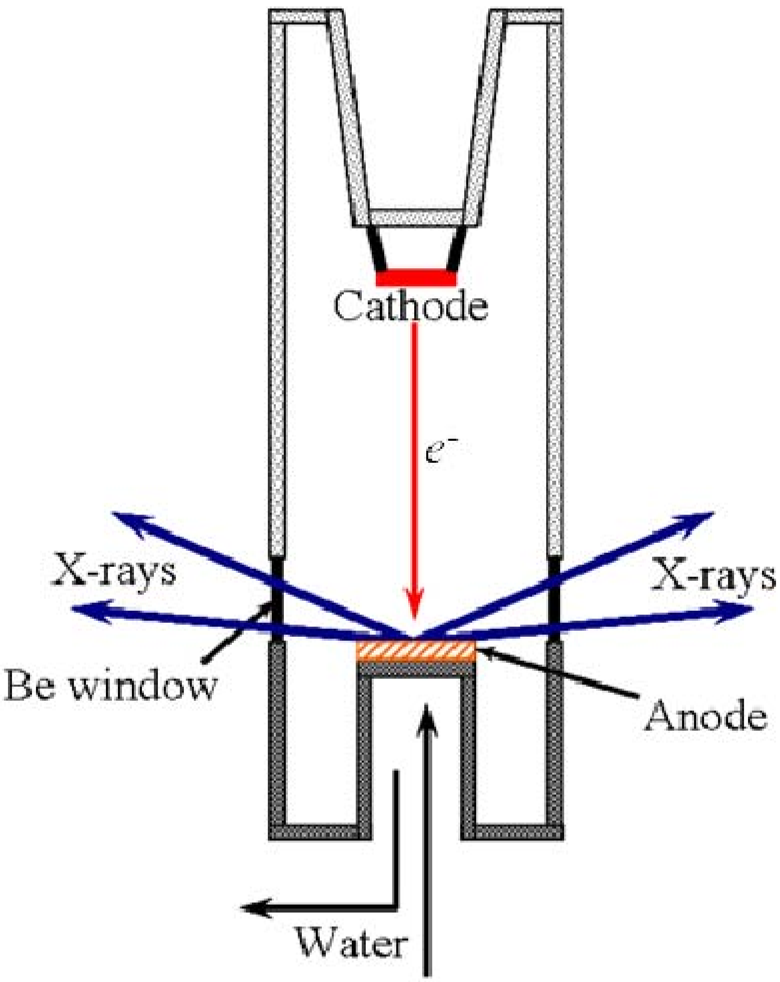
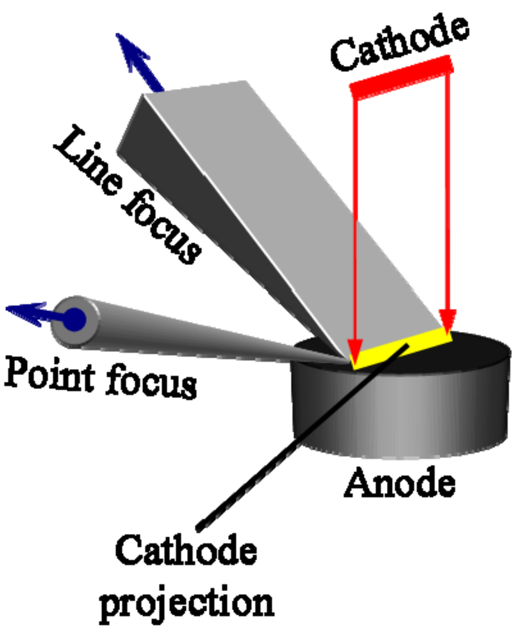


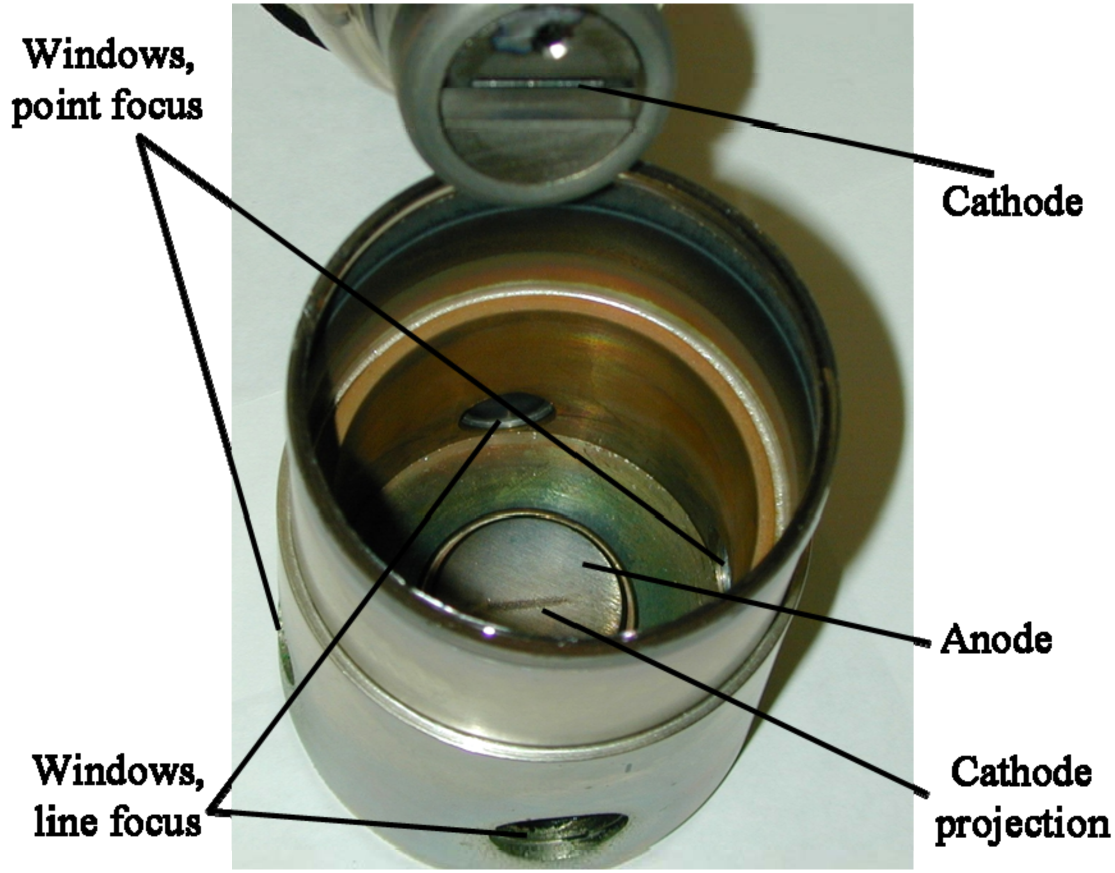
Fig. 5.5 The geometry of the diffractometer arrangement: DS is the divergence slit, SS is the scatter slit, RS is the receiving slit, So is the soller slit.

X-ray tube



Adapted from Vitalij K. Pecharsky and Peter Y. Zavalij, Fundamentals of Powder Diffraction and Structural Characterization of Materials, Kluwer Academic Publishers, 1999.





X-ray tubes will have line focus and point focus windows

Soller Slits

Divergence Slits

- Closely spaced parallel metal planes parallel to the plane of the diffractometer circle that collimate the incident beam. They are usually constructed of a high atomic number element to maximize absorption.

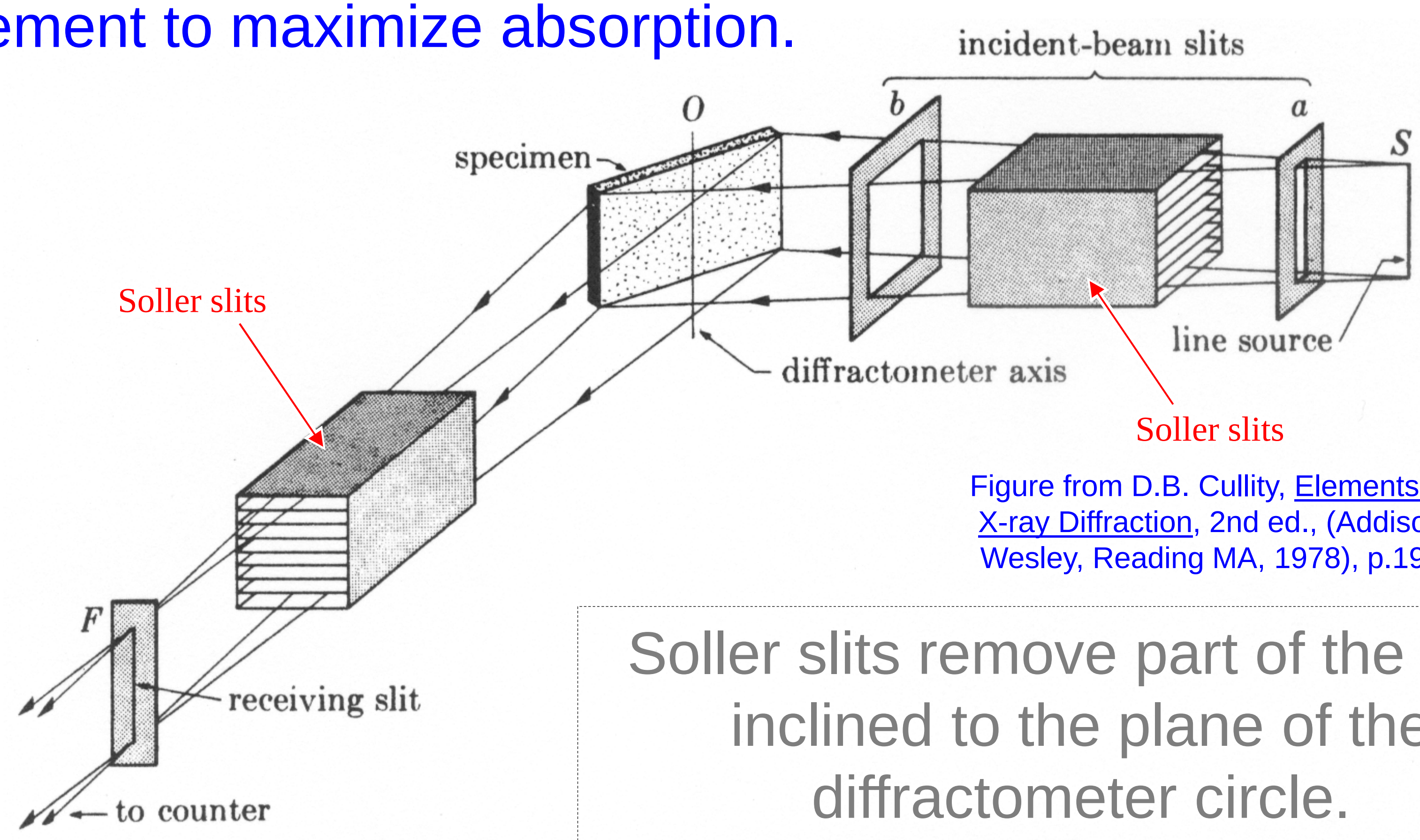
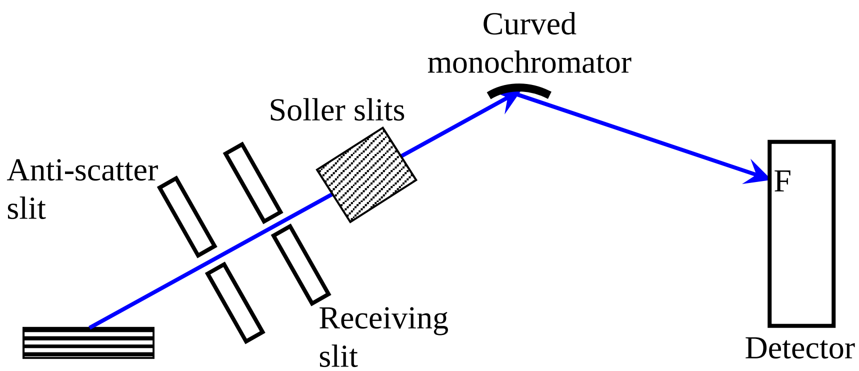


Figure from D.B. Cullity, Elements of X-ray Diffraction, 2nd ed., (Addison Wesley, Reading MA, 1978), p.199

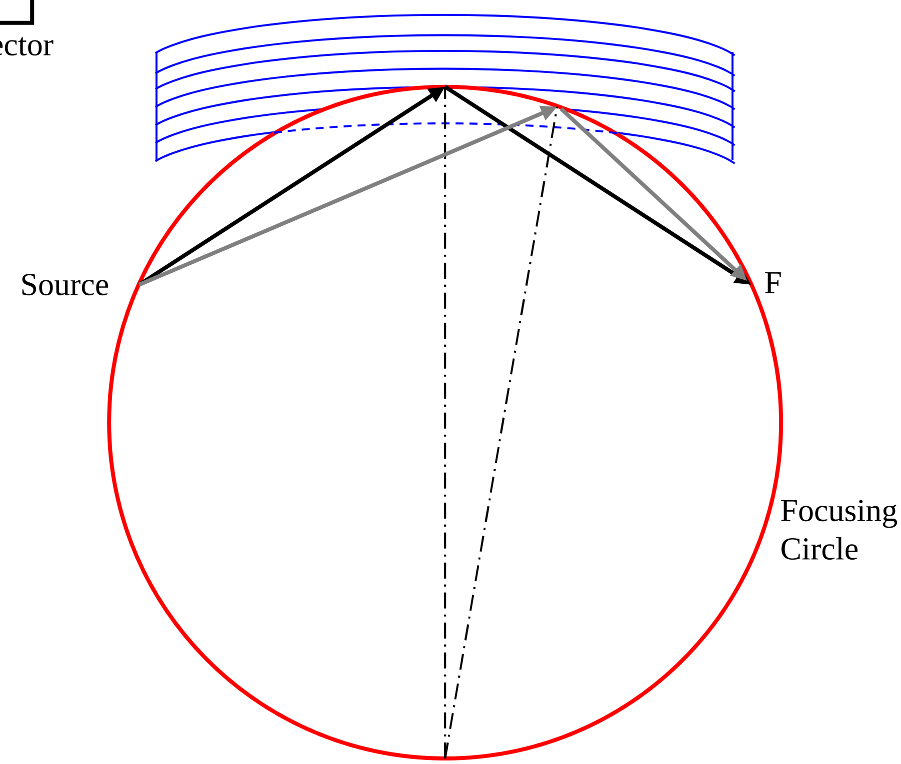
Soller slits remove part of the rays inclined to the plane of the diffractometer circle.

Monochromator



A device in neutron and X-ray optics to select a defined wavelength of the radiation for further purpose

They re-focus the diffracted beam just as a parabolic mirror is used to focus light.



Eliminates Fluorescence!

X-ray Detectors

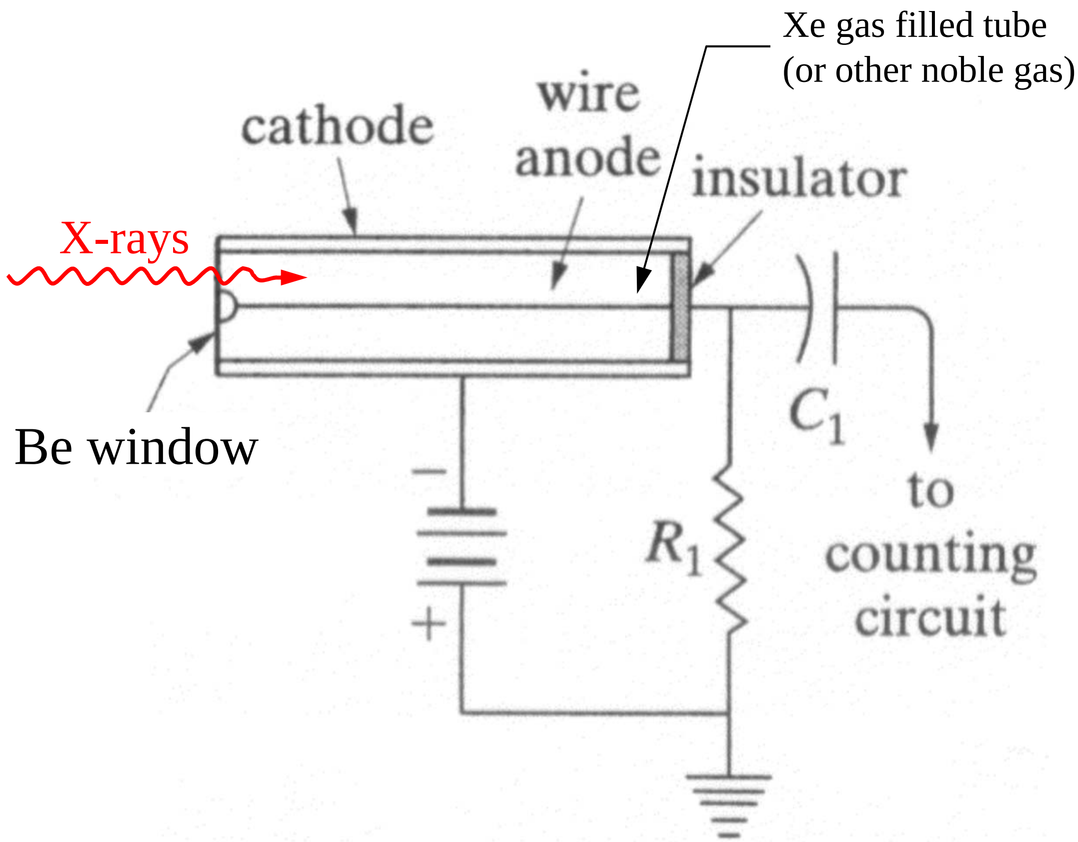
Table i7.1 Properties of different detector types.

	Proportional counter	**Scintillation counter**	**Solid-state detector**
Working principle	Electron–ion pair cascade	Conversion to light and electrons and electron multi-plication	Generation and separation of electron–hole pairs
Transducing medium	Xe	NaI:Tl	Si, Ge
Noise rate (cps)	1	0.1–10	<0.1
Maximum count rate (cps)	10^5–10^6	10^5–10^6	10^4
Resolution $\Delta E/E$ (%)	18–20	40–50	depends on type
Costs	Low	Medium	High

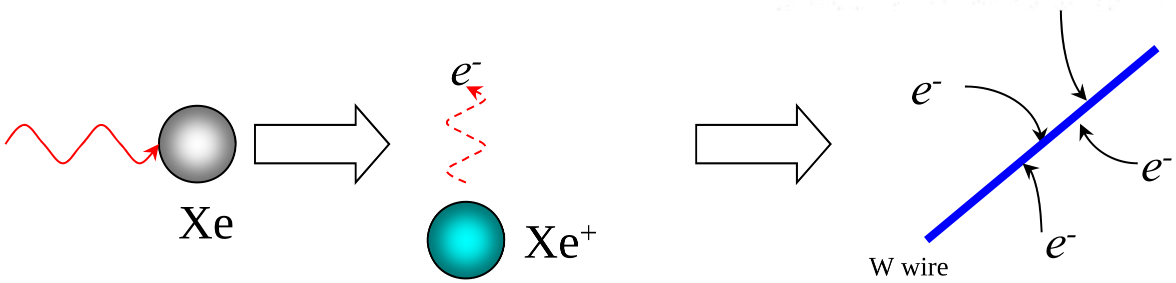
From M. Birkholz, Thin Film Analysis by X-ray Scattering, Wiley-VCH, Weinheim, 2006, p. 254

Proportional Detector

- Most common type of detector in powder diffraction.



Adapted from B.D. Cullity and S.R. Stock, *Elements of X-ray Diffraction, 3rd Edition*, Prentice-Hall, Upper Saddle River, NJ (2001) page 203.



The way proportional detectors work

- X-rays enter the tube and are absorbed by gas atoms.
  - Results in the emission of a photoelectron (i.e., an electron produced by ionization of an atom by a photon).

- Released electrons are attracted to the W wire, resulting in a charge pulse.
  - The charge the collects on the W wire is “proportional” to the energy of the incident x-ray photon. This allows us to distinguish between photons with different E and λ.

Scintillation Detector

- Uses a NaI single crystal doped with Thallium ions (Tl^{+}) attached to a photocathode and photo-multiplier tube.

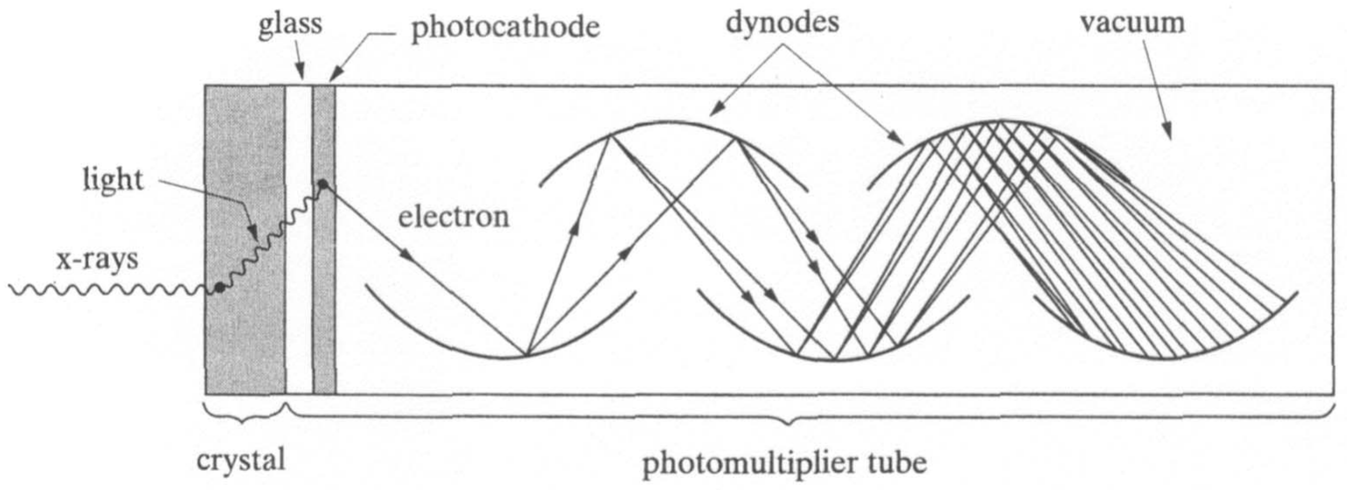
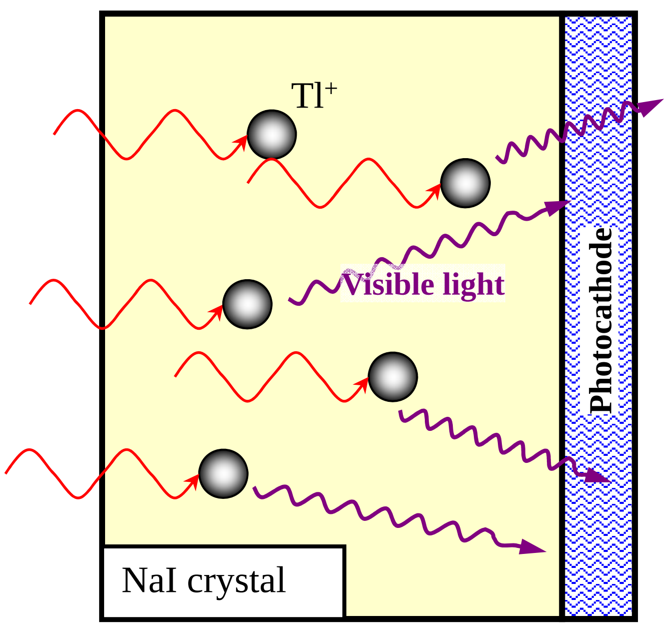


Figure 6-21 Scintillation detector (schematic). Electrical connections not shown.



Adapted from B.D. Cullity and S.R. Stock, *Elements of X-ray Diffraction, 3rd Edition*, Prentice-Hall, Upper Saddle River, NJ (2001) page 207.

The way scintillation detectors work

1. Incident x-rays cause the crystal to fluoresce in the violet part of the EM spectrum.

2. A flash of light (*scintillation*) occurs for every X-ray photon absorbed.

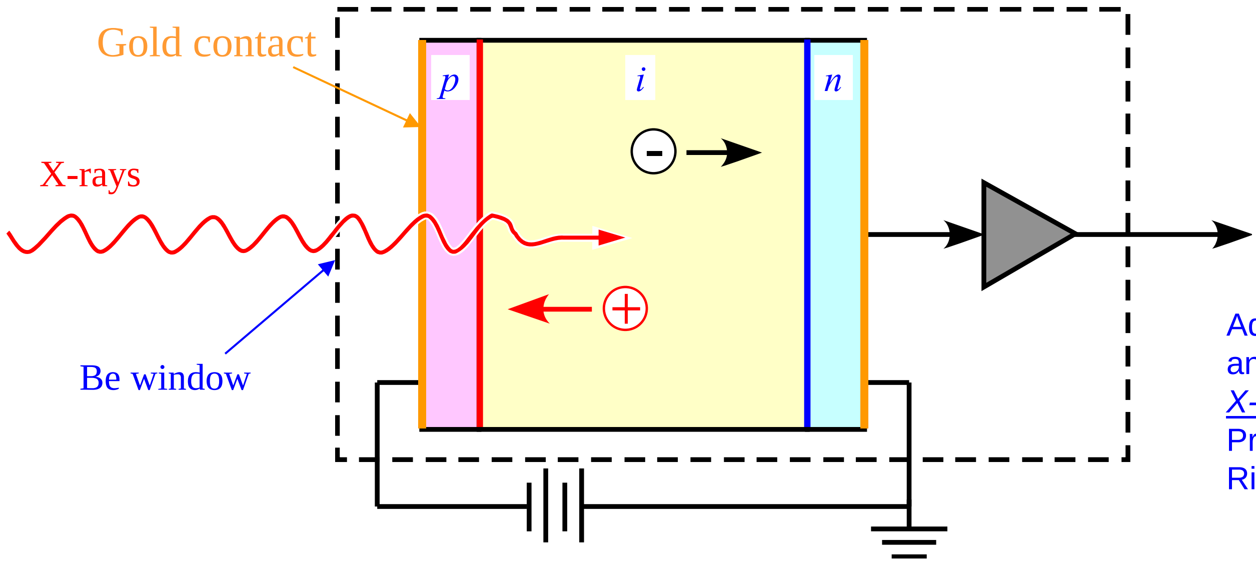
3. Light is measured with a photomultiplier attached to the photocathode.

4. Amount of light emitted is proportional to the X-ray intensity.

5. The magnitude of the light pulses is proportional to the energy of the X-rays.

Has lower resolution than proportional or solid state detectors.

Solid State Detector



Adapted from B.D. Cullity and S.R. Stock, *Elements of X-ray Diffraction, 3rd Edition*, Prentice-Hall, Upper Saddle River, NJ (2001) page 210.

Schematic of a Si(Li) detector and a field effect transistor (FET) preamplifier. Both are in a cooled evacuated space. X-rays enter through a beryllium window. The detector is operated at around 1000 V.

- Based on the PIN diode
- Allows separation of CuK_{α} and CuK_{β}
- Eliminates the need for a β-filter or a monochromator to select K_{α} wavelengths.
- If it's advantageous one can record the XRD pattern using K_{β} radiation as opposed to K_{α}.
- Lower background signal which leads to improved signal to noise ratios.

The way solid state detectors work

- X-rays excite electrons from the valence band into the conduction band creating an electron hole pair.
- Application of a reverse bias potential causes the electrons and holes to separate, which allows a charge pulse of electrons to be measured.
- The number of electrons or holes is directly proportional to the energy of the x-ray photon.
- Solid state detectors offer the highest levels of efficiency and the highest resolution.