

X-ray Detectors II

Film, Image Plates & Phosphors

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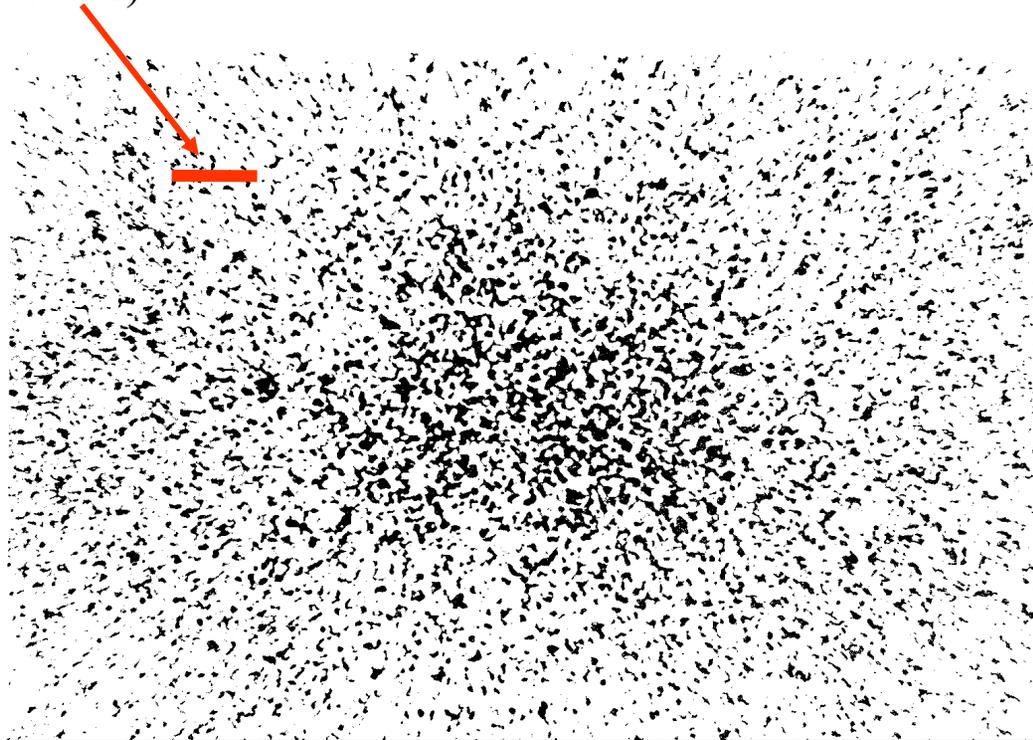
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Integrating Detectors: X-ray Film

X-ray film is actually a photon counter, in that each absorbed x-ray renders one grain of film developable. The individual grains are visible in this magnification of a protein Bragg spot. (Scale bar = 100 microns)



Magnified single Bragg protein diffraction spot on x-ray film. From PhD Thesis of J. Milch, 1974, Physics Dept, Princeton University, Princeton, NJ



The practical difficulty of using film as a photon counter is in counting the grains. For this reason, it is always used as an **integrator**, where the determination of cumulative darkened film grains is made by measuring the optical density of the developed film.

Advantages:

- Film has excellent resolution.
- Inexpensive.
- Very long exposures are possible with negligible “dark current” accumulation.

Disadvantages:

- Film has a high noise of about 10^7 “fog” grains per cm^2 . These are nearly Poisson distributed, so the noise is $\sqrt{10^7} \approx 3000$. The stopping power of x-ray film for 8 keV x-rays is about 0.6. So one needs about $3000/0.6=5000$ x-rays/ cm^2 just to match the fluctuation in the fog, e.g., $S/N=1$. Hence, film is an insensitive detector. It is advantageous to place film close to the specimen to concentrate the signal over as small an area as practical.
- Film has relatively low stopping power.
- Film ages and the ultimate signal it is sensitive to the exact development procedure.
- The optical density of the film is linear with the dose only over about a factor of 100. Basically, film is non-linear when the density of developed grains is high enough that they start shadowing one another.
- Film requires a slow, messy “off-line” development process and a complex optical density scanner, making its use very cumbersome.



X-ray Film

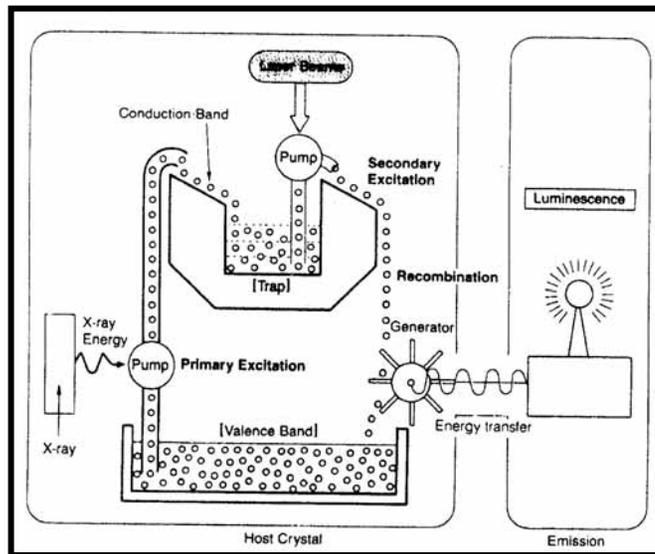
In summary, film is most useful for measurements that take a very long time, which involve a relatively high signal dose into a small area (small, sharp reflections), and where qualitative info is needed. An example where film is very useful is x-ray topography, where there are lots of photons and micron resolution is needed. The only problem is that x-ray film is going extinct.



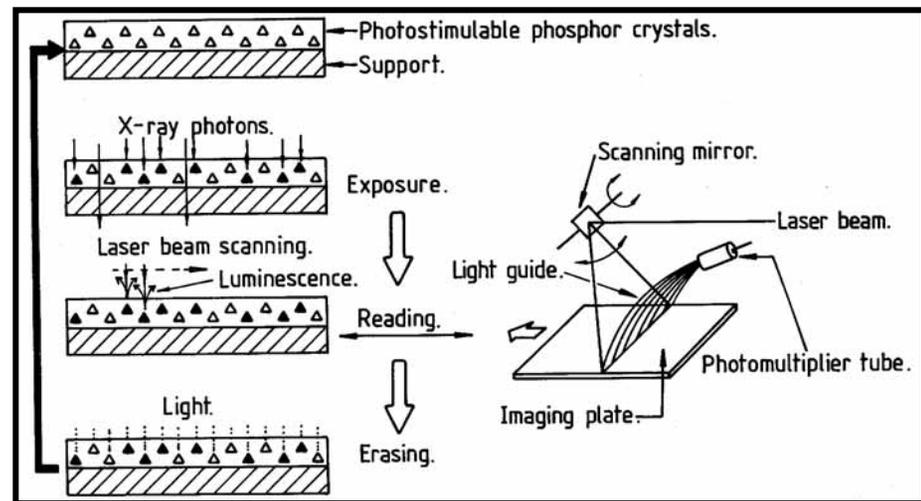
Image Plates = Storage Phosphors

Certain materials, notably BaFBr:Eu^{+2} , have energy levels below the conduction band which can be populated when x-rays de-excite in the material. These levels cannot de-excite to lower levels and are sufficiently below the conduction band that thermal excitation to the conduction band is very unlikely. Photoexcitation, e.g., by a red laser, into the conduction band then allow de-excitation with the emission of a blue photon. This is photostimulated luminescence. A phototube covered with a filter to only pass blue photons then can sense the magnitude of photostimulated luminescence without sensing scattered red laser photons.

An intense flood of light is used to totally photostimulate the plate, thereby erasing it.



Amemiya (1997) *Meth. Enzymology* 276:233



Miyahara (1986) *Solid State Phys (Japan)* 21 : 172.



Comparison: Film, Image Plates, I-CCD

1. Use 100 mCi Fe-55 source for absolute stability.
2. Observe that 1 Optical Density (OD) in film absorbs 10% of Fe-55 x-rays by silver absorption.
3. Use precise, electro-formed metal mask of grid of 300 micron holes to make a photographic mask in normal photographic film.
4. Set x-ray contrast by OD of spots and OD of surround. This allows slight ($< 20\%$) Fe-55 contrast.



From: Eikenberry et al (1991), X-ray detectors: comparison of film, storage phosphors and CCD detectors. In *Photoelectronic Image Devices, 1991*, E.L. Morgan, ed. (Inst. of Physics Conf.

Series No. 121, Inst. of Physics, Bristol, UK, 1992, pp. 273-280).

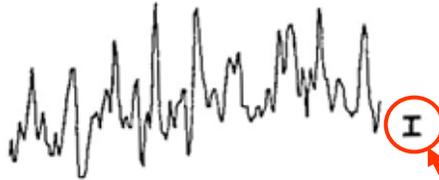


Comparison: Film, Image Plates, I-CCD

	Film	IP	I-CCD	Film	IP	I-CCD
Exposure, s	200	200	200	500	1800	500
Contrast, %	11	11	11	6	6	6
Signal, x-ray/spot	2,000	2,000	2,000	2,900	10,000	2,900
Spot dia, microns	300	300	300	300	300	300
Bkgnd, x-ray/spot	18,000	18,000	18,000	48,000	190,000	48,000
Gain, ADU/x-ray	0.022	0.93	2.1	0.022	2.1	2.1
System noise, x-ray/pix	130	3	2.7	120	11	2.9
Measured signal accuracy, %	59	94	25	64	33	52
Poisson limiting accuracy, %	24	23	17	28	15	30
DQE	0.15	0.06	0.42	0.19	0.20	0.33

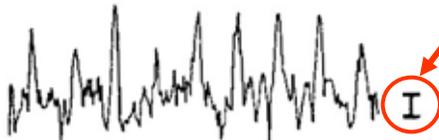


Comparison: Film, Image Plates, I-CCD

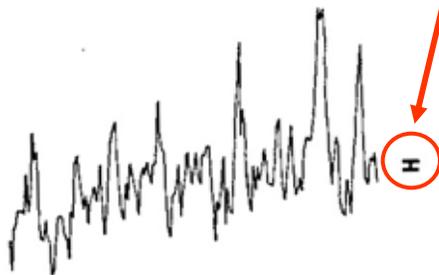


Film, 6% contrast, 500s, smoothed

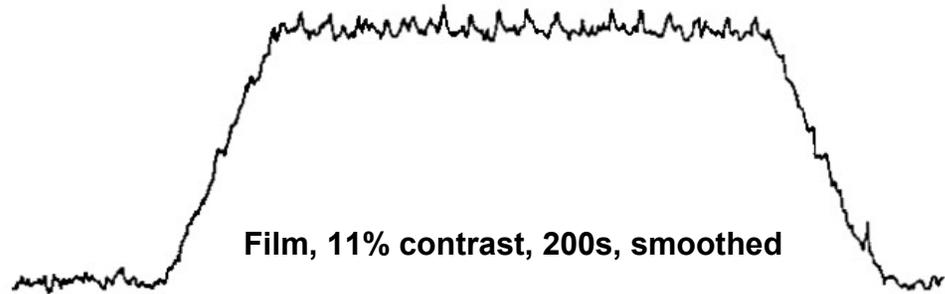
Incident quantum statistics of pedestal



I-CCD, 6% contrast, 500s



IP, 6% contrast, 1800s



Film, 11% contrast, 200s, smoothed

From: Eikenberry et al (1991), X-ray detectors: comparison of film, storage phosphors and CCD detectors. In *Photoelectronic Image Devices, 1991*, E.L. Morgan, ed. (Inst. of Physics Conf. Series No. 121, Inst. of Physics, Bristol, UK, 1992, pp. 273-280).



Image Plates

IPs have good stopping power to the Br K edge (13.47 keV). The linearity of response vs. x-ray energy needs to be calibrated above for higher energies. This is important, for example, in Laue protein crystallography.

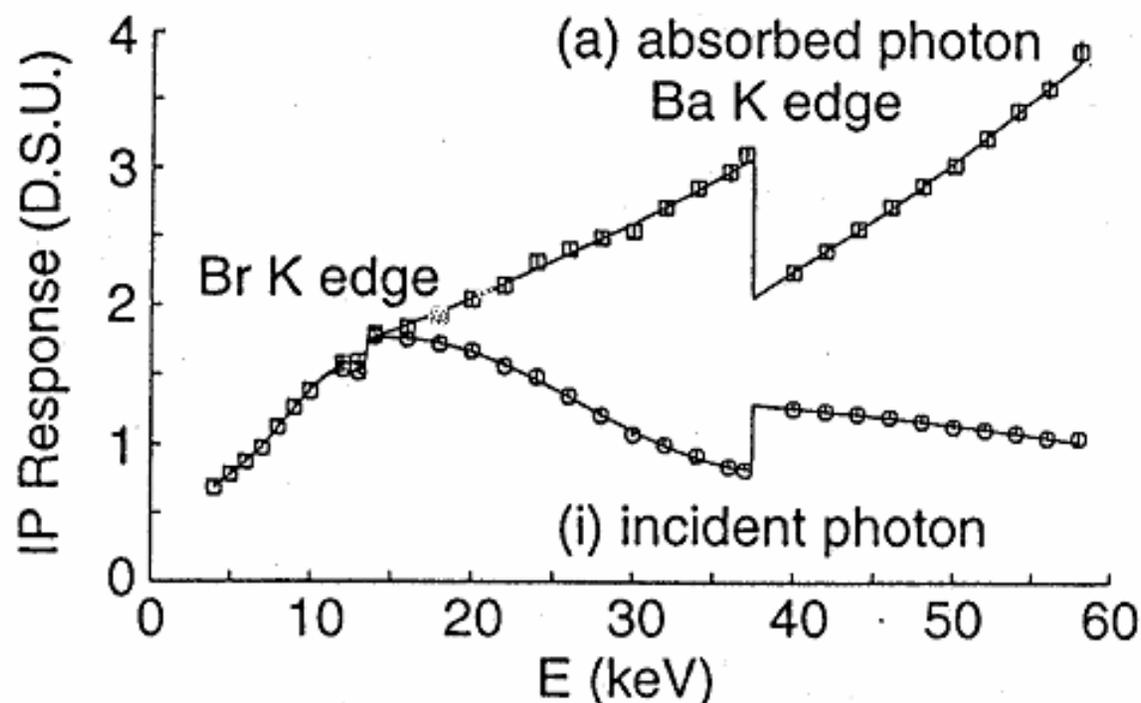


FIG. 7. Dependence of the IP response as a function of the energy of an X-ray photon. (i) is the IP response per incident X-ray photon, and (a) the IP response per absorbed X-ray photon. The unit of the ordinate corresponds to the background noise level of the IP scanner.



Image Plates

IPs have a very wide inherent dynamic range because of the very large number of photostimulatable centers per unit phosphor volume. However, the practical dynamic range is always limited by the dynamic range of the photosensing system, which runs from 8 to 14 bits in most systems. A wider working range of, say 1% accuracy over 6 decades of intensity can be obtained by rereading the IP.

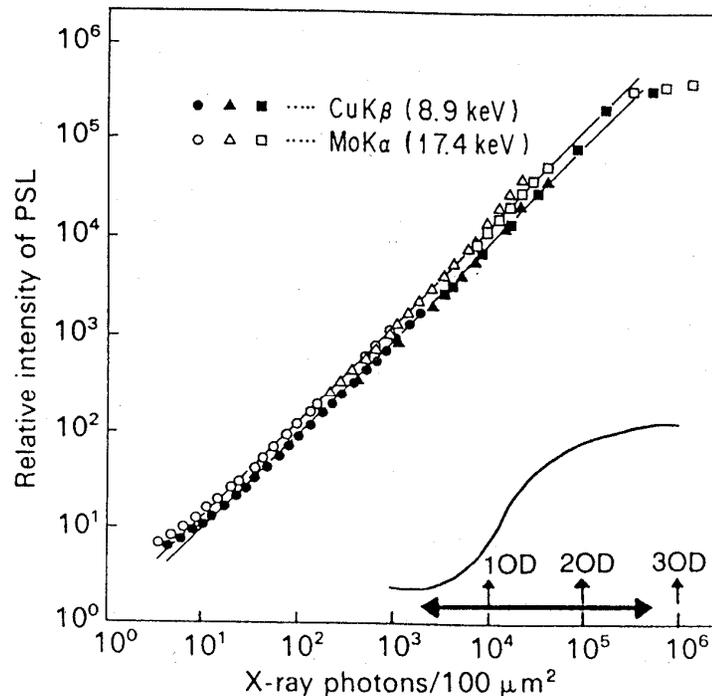


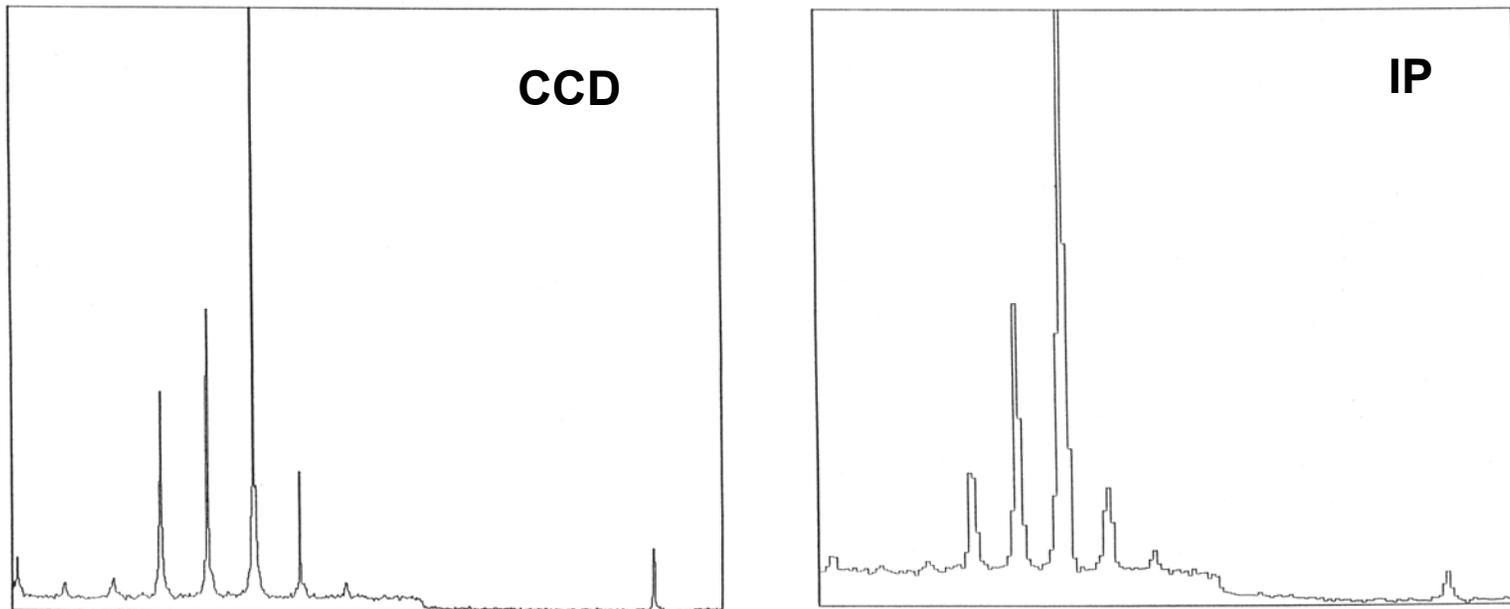
FIG. 6. Measured dynamic range of the photostimulated luminescence of the imaging plate with two different X-ray energies. The dynamic range of typical high-sensitivity X-ray film is also shown. OD, Optical density.

Amemiya (1997) *Meth. Enzymology* 276:233



Image Plates

IPs suffer from many systematic effects which ultimately limit the real accuracy and real dynamic range, as may be seen from this experiment:



GaAs multilayer Laue Diffraction

From: Eikenberry et al (1991), X-ray detectors: comparison of film, storage phosphors and CCD detectors. In *Photoelectronic Image Devices, 1991*, E.L. Morgan, ed. (Inst. of Physics Conf. Series No. 121, Inst. of Physics, Bristol, UK, 1992, pp. 273-280).



Image Plate Summary

IPs are good detectors for measurement to $\sim 1\%$ accuracy over a wide span.

Advantages:

- Wide working span, especially if plates are multiply scanned.
- Available in large areas, many pixels across.
- Good sensitivity for most experiments, though hardly quantum limited.
- Affordable.
- Good stopping power.

Disadvantages:

- IP readers are slow and have many moving parts, leading to calibration drift.
- Single photon sensitivity is usually not possible.
- The real accuracy rarely exceeds 1%.
- IPs are difficult to erase and exhibit systematics, so background subtraction is difficult.
- The resolution of the IP is limited. Many systems have flare in low-level PSF.

A good, if optimistic overview is Amemiya (1997) *Meth. Enzymology* **276**:233. Reality checks are in Templer et al (1991) *Nucl. Instr. Meth. Phys. Res.* **A300**:357; Harrison et al (1991) *Nucl. Instr. Meth. Phys. Res.* **A310**:220; Hall et al (1992) *Rev. Sci. Instrum.* **63**:697.



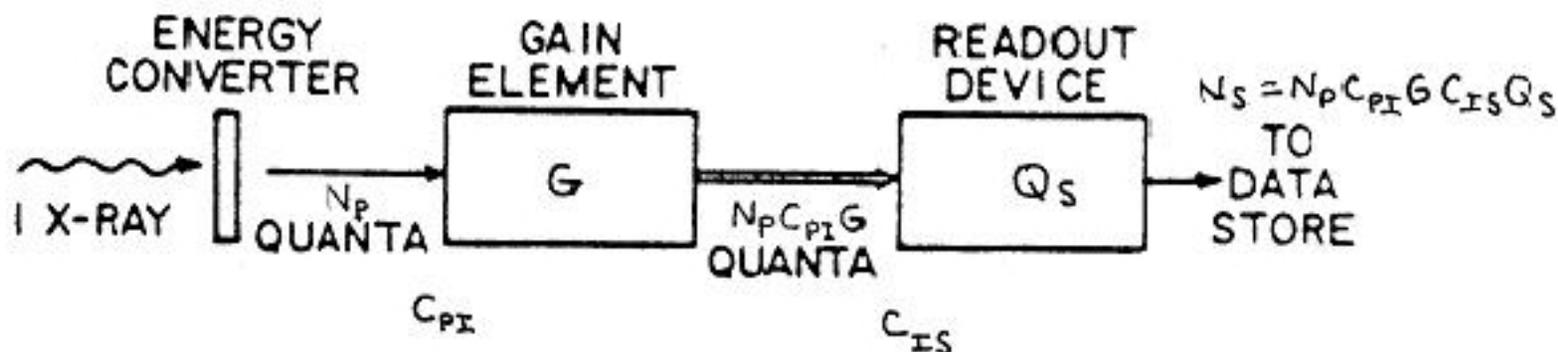
CCD Detector Introduction to Phosphors

Note: In 1999 I was asked to prepare a personal, historical perspective of CCD detectors as part of an American Crystallographic Association symposium – see Gruner, *Trans Amer, Cryst. Assoc.* **34**:11. It gives a sense of the time and difficulty needed to develop a new detector technology, as well as an opinion of the sociology of the field.

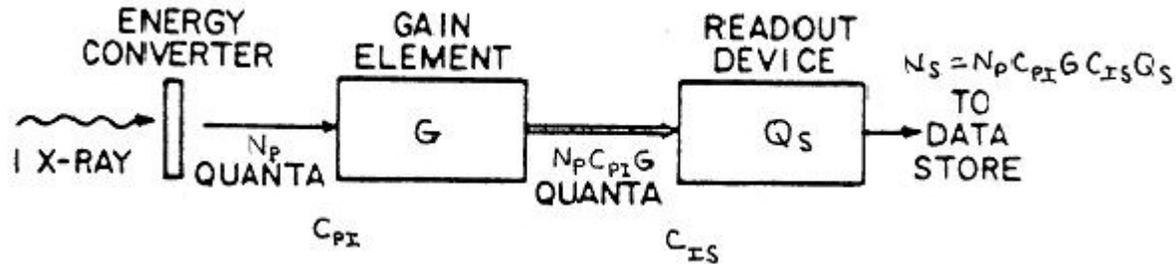
For a review of CCD area x-ray detectors, see Gruner et al, *Rev Sci Instr* 73 (2002) 2815-2842.

The Quantum Optical Chain

In area detector made up of electro-optical elements typically consists of a chain of elements which convert the x-rays to more readily manipulated quanta, and a possible gain element to increase the number of quanta/x-ray to better compete with noise in the final image sensing element:



Quantum Detector Chain



For example, the energy converter may be a thin, phosphor screen, the luminescence of which is amplified by an image intensifier viewed by a CCD image sensor. Here,

N_P = the number of quanta/x-ray

C_{PI} = fraction of N_P conveyed to the gain element

G = gain

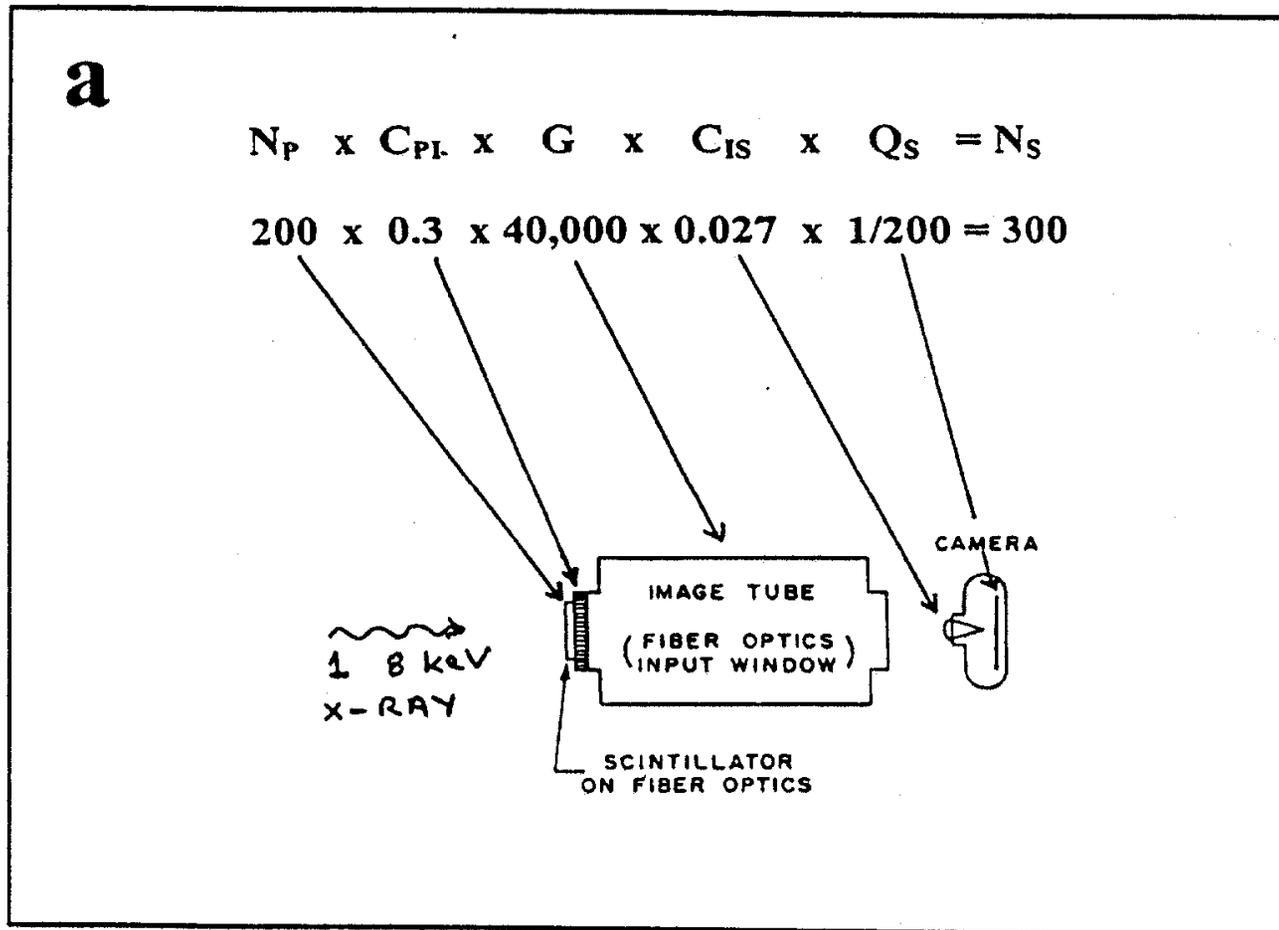
C_{IS} = fraction of amplified quanta conveyed to image sensor of quantum efficiency Q_S .

This works for practically any x-ray detector, even if one or another element is absent. So, for example, CCDs are directly x-ray sensitive. In this case, $C_{PI} = G = C_{IS} = 1$.



Quantum Detector Chain

Let's try this with an x-ray detector made at Princeton University by George Reynolds in 1968:



Quantum Detector Chain

George found that each x-ray led to 300 developed film grains, as opposed to 1 grain/x-ray in x-ray film. He verified that the detector could record diffraction patterns about 300 times faster than x-ray film.

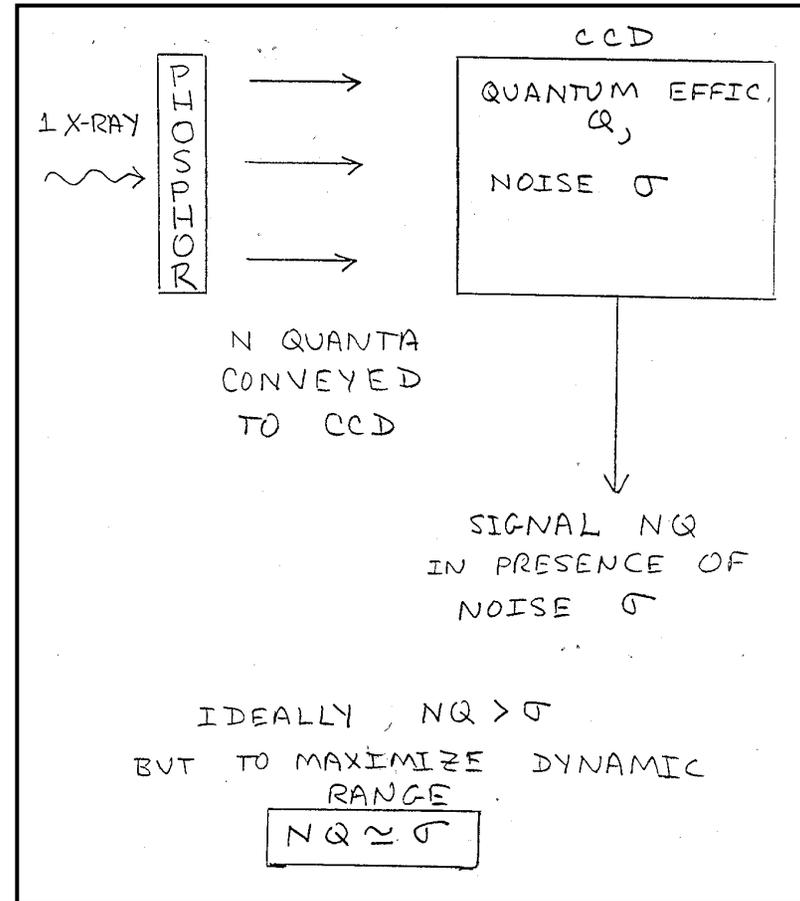
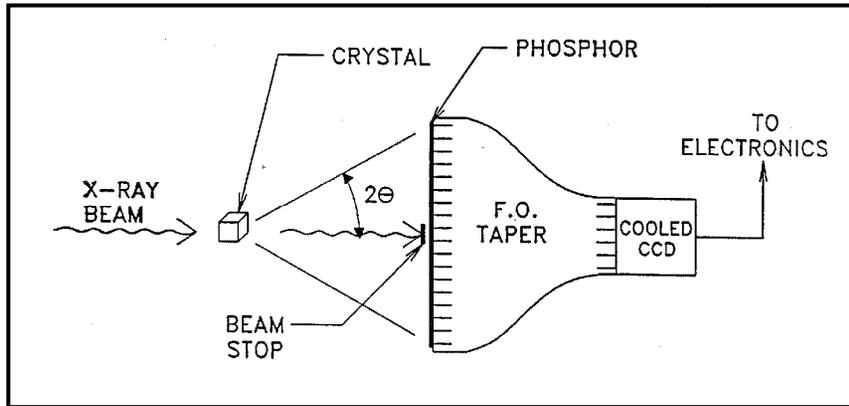
The history of x-ray detectors based on electro-optical sensors (e.g., TV cameras and CCDs) is a progression of detectors in which the various essential elements of phosphor screen, optical coupling techniques, image intensifiers and sensors were progressively replaced with better components as better components became available. The purpose of the gain element was to pump up the signal per x-ray to exceed the noise in the sensor.

CCD detectors matured when CCDs and coupling methods became good enough that the gain element could be eliminated while still preserving near unity DQE. At that point, the many detector configurations which had been tried collapsed into the canonical design which now accounts for most CCD detectors, namely, a thin phosphor screen coupled to a tapered fiber optic bundle which is, in turn, coupled to a cooled, low-noise CCD.

The goal is typically to maximize the dynamic range while preserving near-photon limited (DQE=1) imaging.



Quantum Detector Chain for CCDs



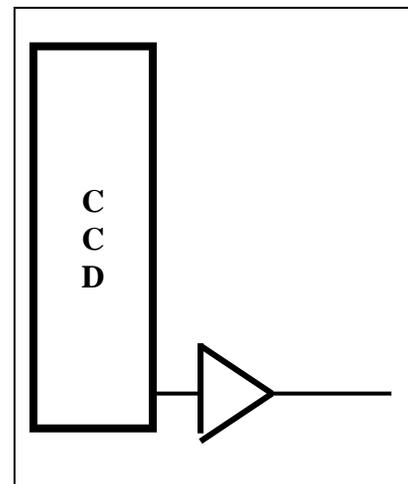
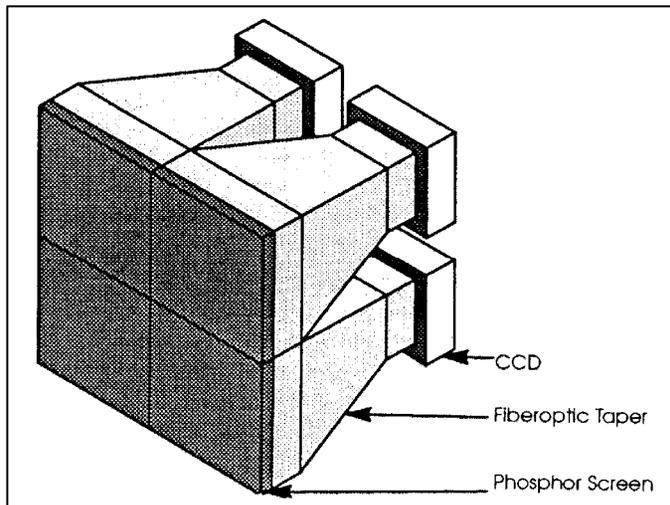
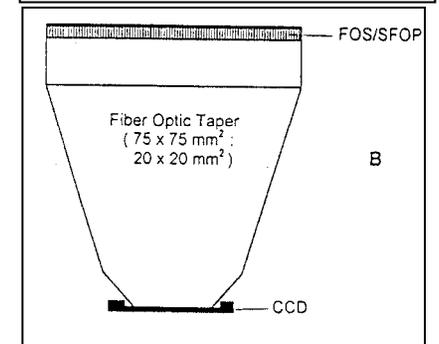
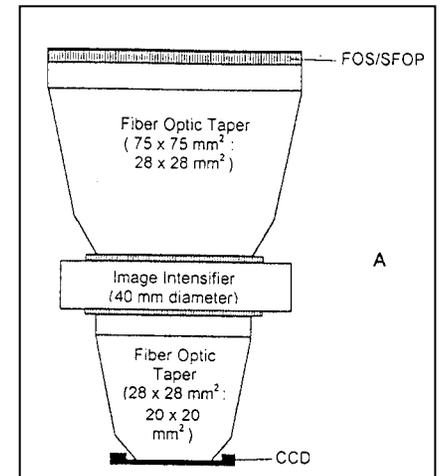
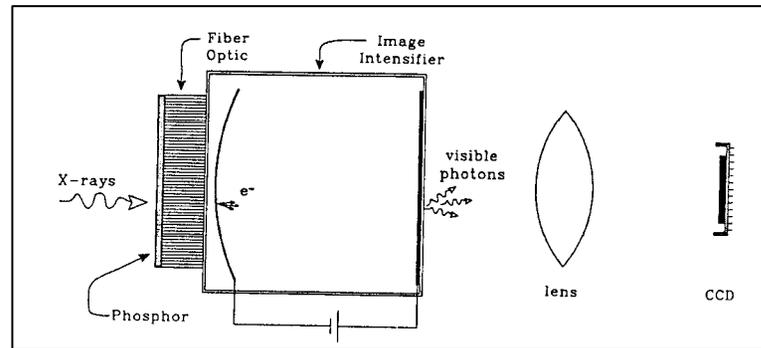
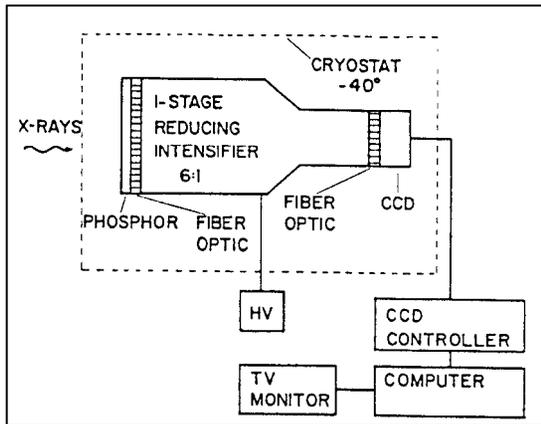
Desirable Detector Characteristics

	Film	Wire	IP	CCD*
Robust, Stable, Convenient	-	-	-	+
High DQE at Low Dose	-	+	+	+
Adequate Area	+	+	+	+
Intensity Linearity	-	-	+	+
Dose Linearity	-	+	+	+
Adequate Spatial Resolution	+	-	+	+
Geometric Linearity	+	-	+	+
Stopping Power	-	-	+	+/-
High Count-rate	+	-	+	+
Read-out Time	-	+	-	+/-
Flat-field Response	+	+	+	+
Very Long Integrations	+	+	+	-
Wide Span	-	+	+	+/-
Low Cost	+	-	-	-

* Phosphor-coupled CCD detectors.



Many CCD Detector Configurations are Possible



It Comes Down to Details

Performance depends on the details of the essential parts of the CCD system:

- Phosphor
- Fiber Optics
- CCD
- Control Electronics
- Calibrations
- Software

Let's start with phosphors

Important Characteristics:

1. Efficient conversion of x-ray energy to light.
2. Spectral match to FO-taper & CCD.
3. Prompt light emission w/low persistence.
4. Linear w/dose & intensity.
5. High stopping power.
6. Robust & stable.
7. High spatial resolution.

No single phosphor has best of all of these.



Efficiency

Fraction of stopped x-ray energy emitted as light: $n = hv_e/E$, where

h = Planck's constant

v_e = averaged frequency of luminescent radiation

E = average energy required to create an electron-hole pair. (Varies from about 3 times the band gap for NaI and CsI to ~ 7 for CaWO_4 ; depends on band gap, high frequency & static dielectric constants and frequency of longitudinal optical vibration mode. High efficiencies result if the optical vibrational modes are of low frequency and the emission energy is close to the band gap energy.)

Phosphor	Theoretical	Measured
CaWO_4	8%	6.5%
ZnS : Ag	25%	20%
$\text{Gd}_2\text{O}_2\text{S} : \text{Tb}$	13%	15%
(Zn,Cd)Se	30%	19%

Sources: Robbins, Bruker-Axe

Spectral matching determines overall efficiency.

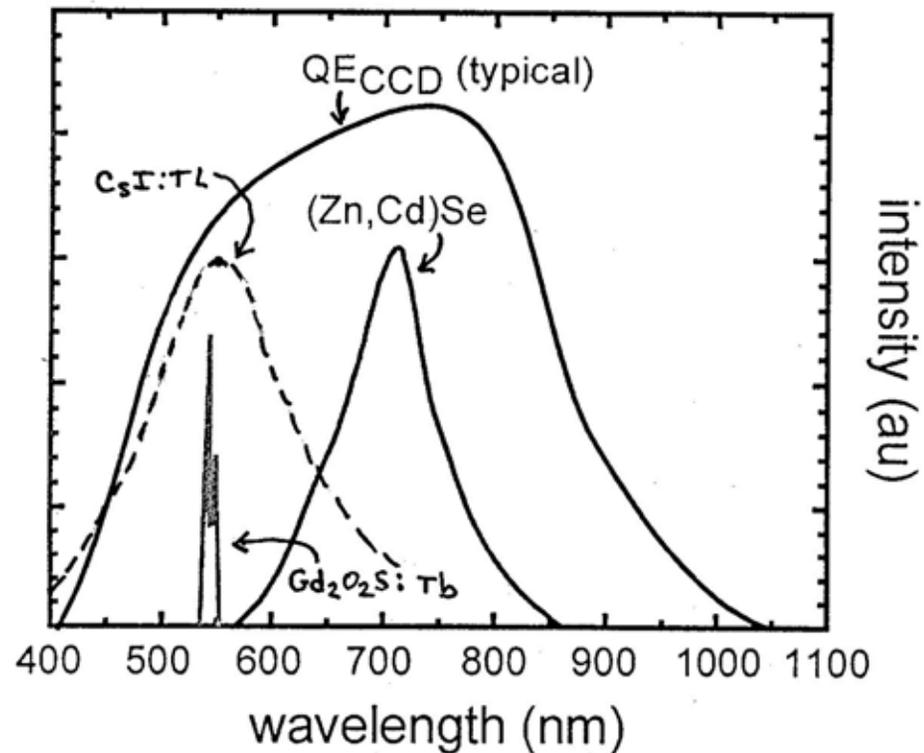
- Fiber optics are about 5% more efficient in the red than the blue because of the wavelength dependence of the indices of refraction of the fiber parts.
- The quantum efficiency of CCDs typically peaks at 30 – 40% in the red. The losses are mostly due to absorption in the CCD gate over-structures in normal front-illuminated CCDs. If special back-illuminated CCDs (or new Kodak chips) are used, QE's can rise to 80%, even towards the blue.



Efficiency

For normal front illuminated CCDs, a typical QE curve and phosphor emission curves are below. The curves aren't scaled to one another; ignore the relative heights:

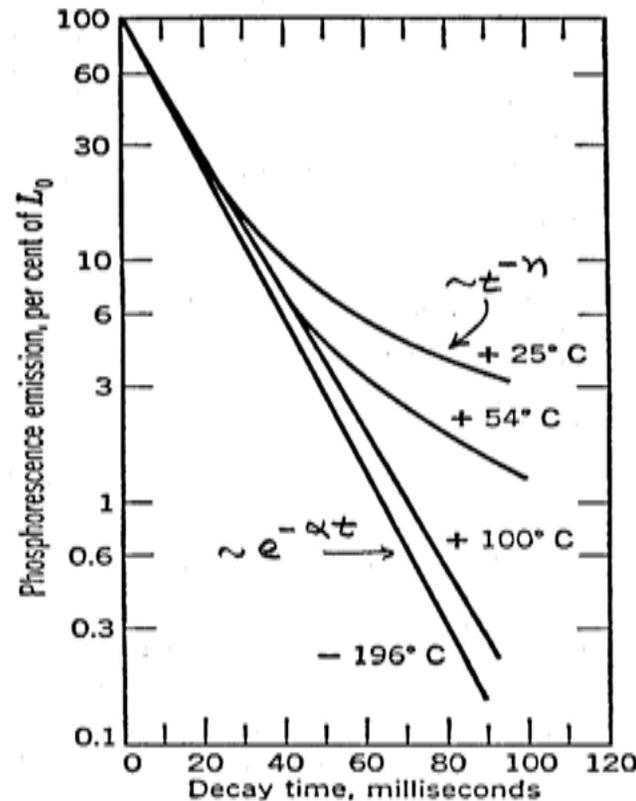
Spectral matching of phosphor emission to CCD sensitivity $\text{Gd}_2\text{O}_2\text{S:Tb} < \text{CsI:Tl} < (\text{Zn, Cd})\text{Se}$



Phosphor Persistence and Lag

The decay time of the light emission is the biggest weakness of most phosphors. Ideally, the light should decay exponentially over many decades of intensity with a very rapid time constant. Typically, the decay constant is msec and the decay goes from exponential to algebraic after only a few decades. Algebraic decays can go on for minutes! The behavior is very complex:

RbhdI – Zn₂SiO₄:Mn (0.3)

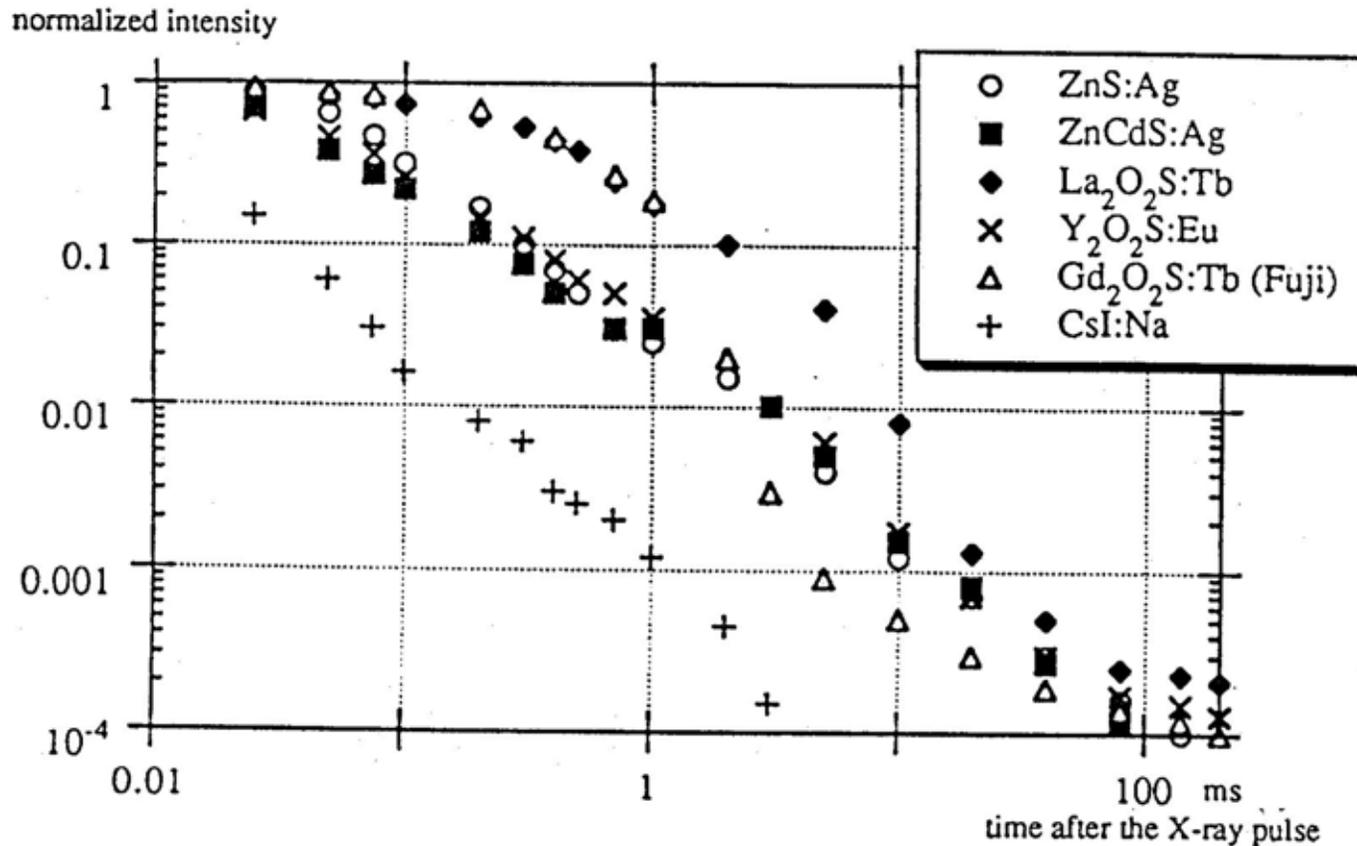


H.W. Leverenz, An Introduction to the Luminescence of Solids (Wiley, NY, 1950)



Phosphor Persistence and Lag

J-P Moy characterized the decay of several phosphors for the ESRF:

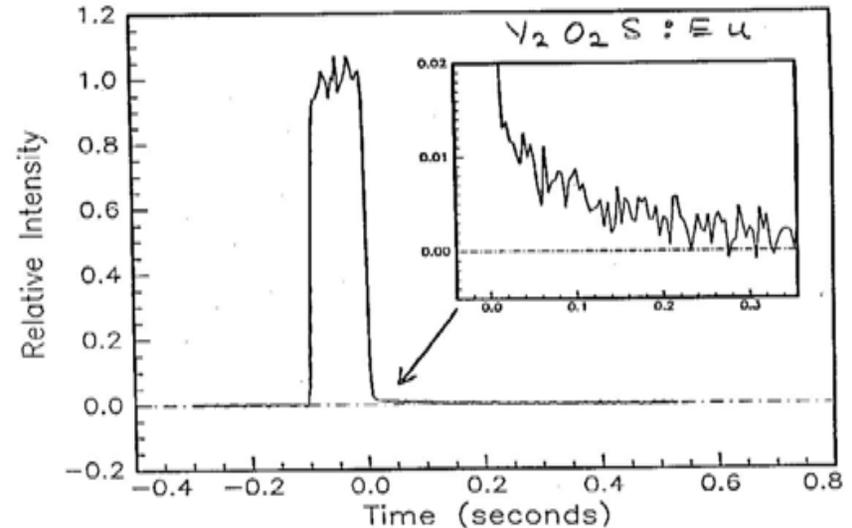
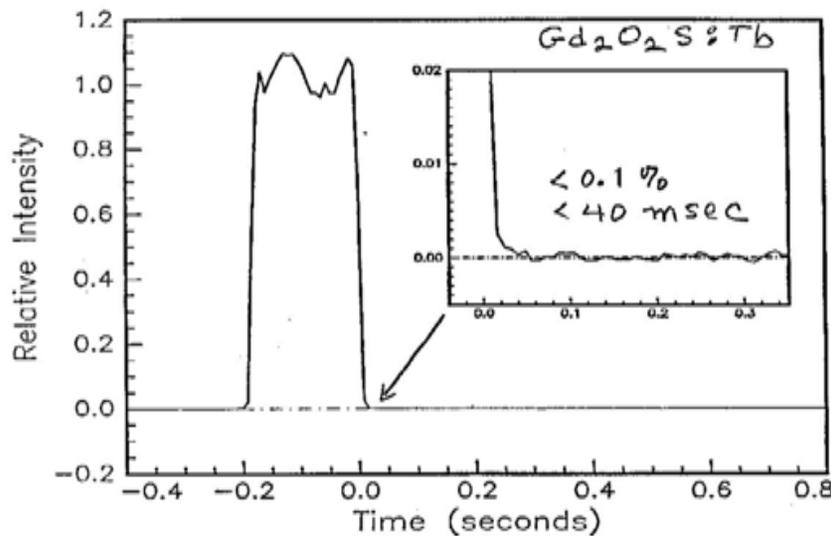


J-P Moy, Time Response of Phosphors, ESRF, 1992



Phosphor Persistence and Lag

Characterizing the time decay of a phosphor over many decades of intensity is difficult. One way is to put on an intense x-ray spot, rapidly gate it off and graph the fall of intensity across the spot vs. time. A CCD detector set up for 1-dimensional line shifting (discussed below) is ideal for this purpose. See also, Gruner et al, 1993, *Proc. SPIE* 2009: 98; Shepherd et al, 1995, *Proc SPIE* 2519 : 24.



Stopping Power

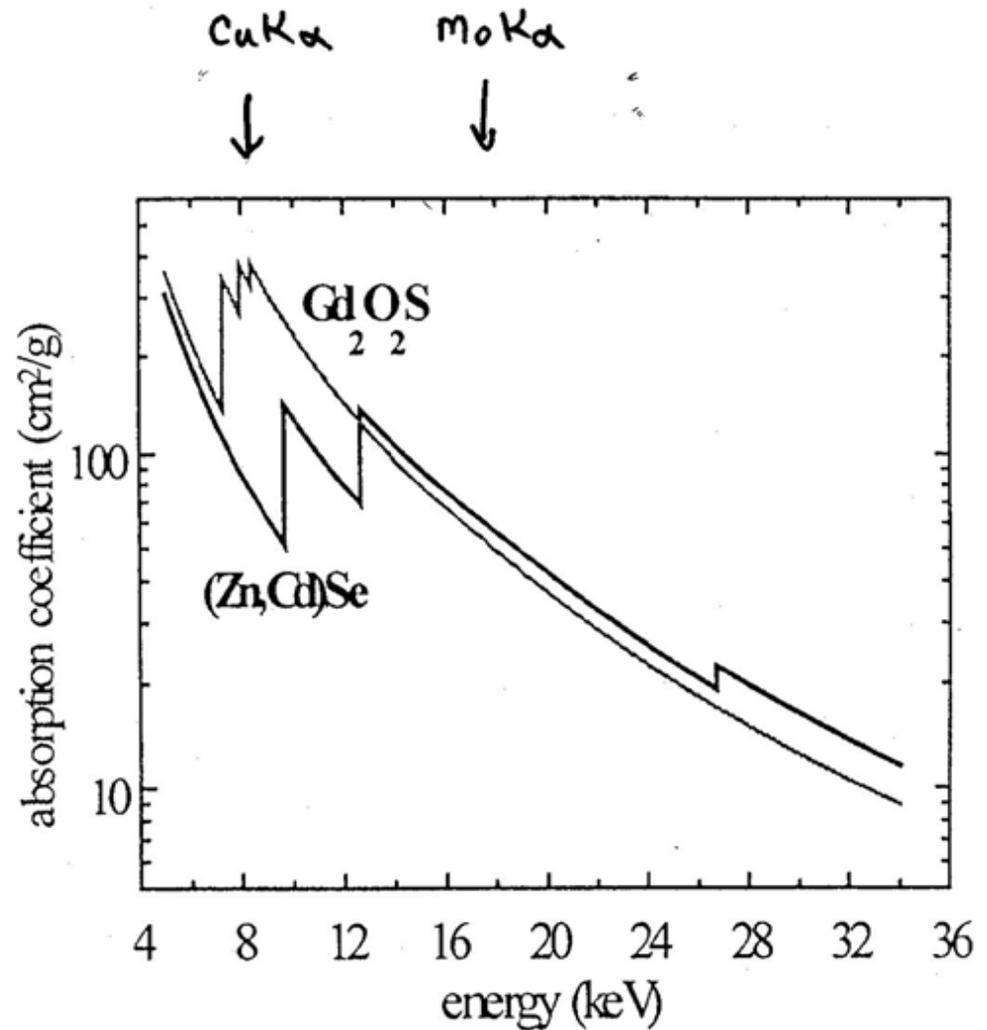
Stopping power is another major consideration.

@ CuK α :

(Zn,Cd)Se < CsI:Tl < Gd₂O₂S:Tb

@ MoK α :

All about equal in stopping pwr



Phosphor Summary

The following table summarizes the characteristics of several popular x-ray phosphors.

	CsI:Tl	Gd ₂ O ₂ S:Tb	La ₂ O ₂ S:Eu	(Zn,Cd)Se
Robustness	Hydroscopic	Stable	Stable	
Stopping Power	high	high	medium	med. – Cu high – Mo
Efficiency	high	high	high	very high
Speed	fast	slow	slow	fast
Persistence	low	low	medium	low
Spectrum	green	green	red	red

Note: CsI:Tl can be grown into columns; other phosphors are settled powders.

