

The 2022 IARIA Annual Congress on Frontiers in Science, Technology, Services,
and Applications

IARIA Congress 2022

July 24, 2022 to July 28, 2022 - Nice, Saint-Laurent-du-Var, France

Advances in Sensors and X-ray Spectroscopy for Agricultural Soil Analyses



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Abstract

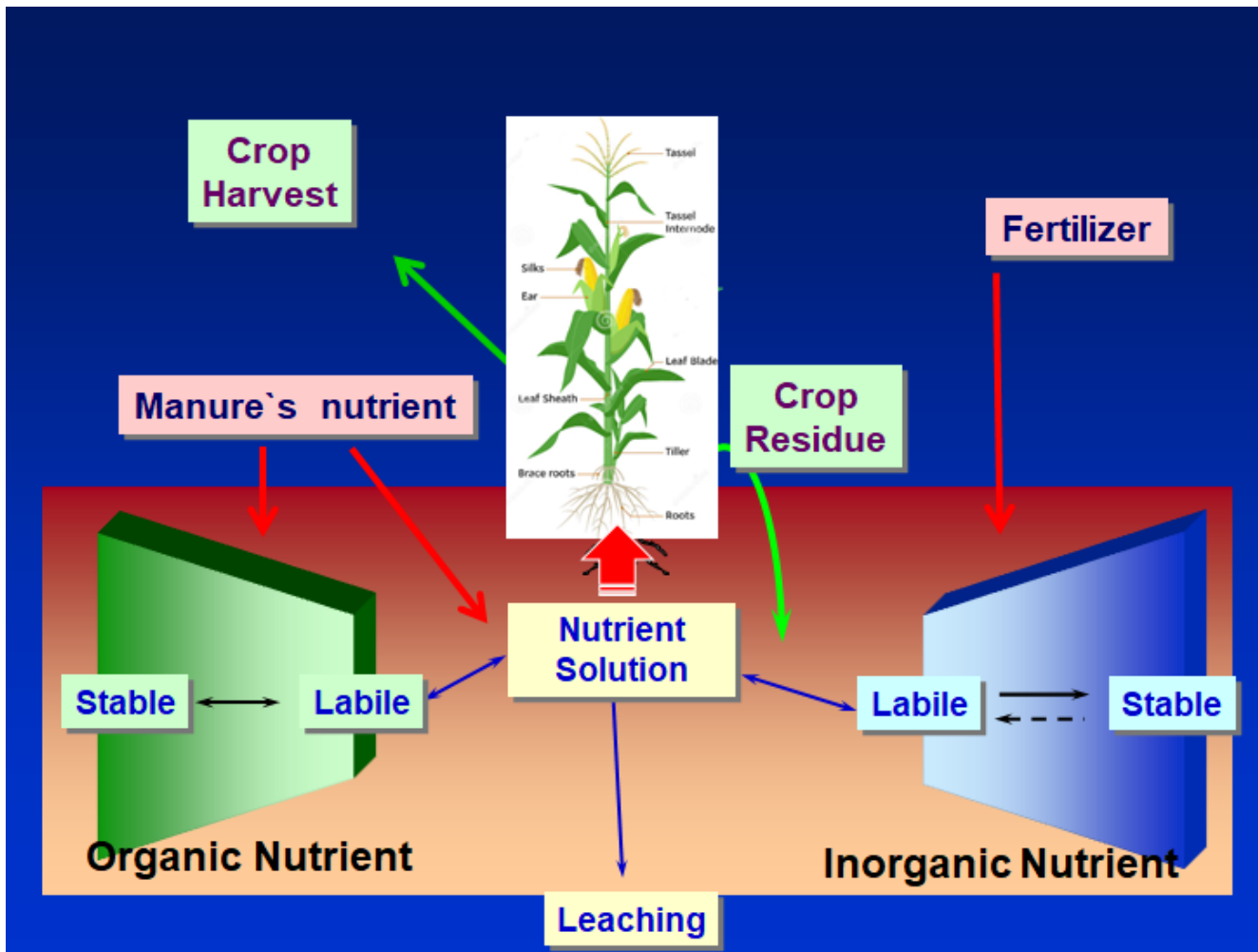
This paper presents a study regarding to the sensors development and use for X-ray spectroscopy. In fact, not only a novel discussion on sensors since the X-rays discovery has been included but also prospective about future is presented. The X-ray based-spectrometry is an analytical technique for determination of elemental composition of different materials. For agricultural soils, either soft or hard X-ray spectroscopies have been shown opportunities to improve agronomic competitiveness and agroecosystems sustainability. This review in X-ray sensors have considered their use in both the X-ray fluorescence and the particle induced X-ray emission technique, i.e., highlighting new materials, accuracy, resolution, efficiency, energy response, and related methods.

Essential nutrients for agricultural production

Element	Chemical symbol	Atomic weight	Ionic forms Absorbed by plants	Approximate dry concentration
<i>Macronutrients</i>				
Nitrogen	N	14.01	NO_3^- , NH_4^+	4.0 %
Phosphorus	P	30.98	PO_4^{3-} , HPO_4^{2-} , H_2PO_4^-	0.5 %
Potassium	K	39.10	K^+	4.0 %
Magnesium	Mg	24.32	Mg^{2+}	0.5 %
Sulfur	S	32.07	SO_4^{2-}	0.5 %
Calcium	Ca	40.08	Ca^{2+}	1.0 %
<i>Micronutrients</i>				
Iron	Fe	55.85	Fe^{2+} , Fe^{3+}	200 ppm
Manganese	Mn	54.94	Mn^{2+}	200 ppm
Zinc	Zn	65.38	Zn^{2+}	30 ppm
Copper	Cu	63.54	Cu_2^+	10 ppm
Boron	B	10.82	BO_3^{2-} , $\text{B}_4\text{O}_7^{2-}$	60 ppm
Molybdenum	Mo	95.95	MoO_4^{2-}	2 ppm
Chlorine	Cl	35.46	Cl^-	3000 ppm
<i>Essential But Not Applied</i>				
Carbon	C	12.01	CO_2	40 %
Hydrogen	H	1.01	H_2O	6 %
Oxygen	O	16.00	O_2 , H_2O	40 %

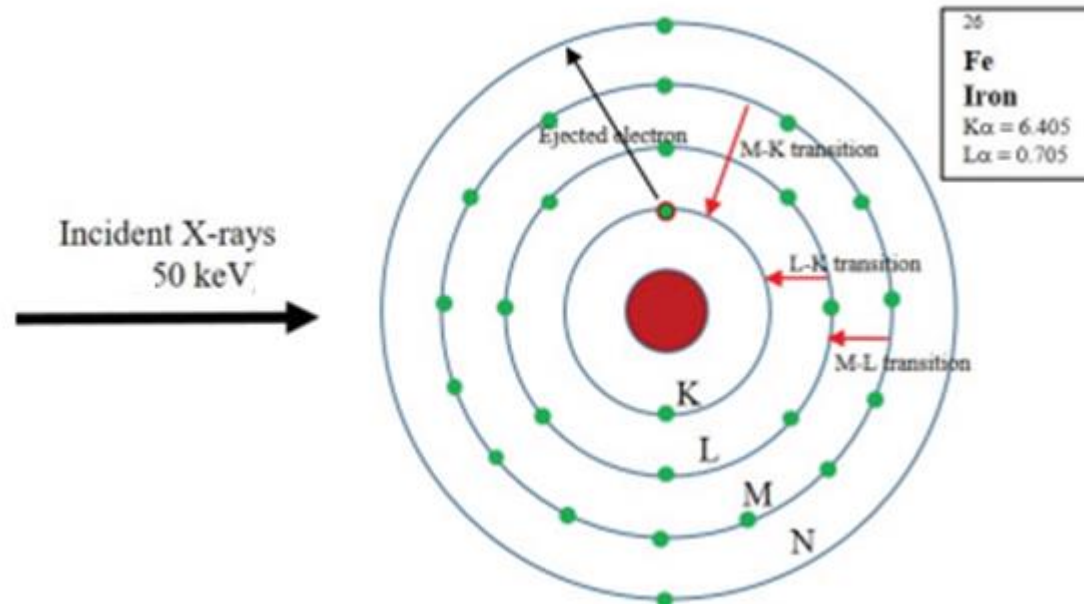
Plant tissues also contain other elements (Na, Se, Co, Si, Rb, Sr, F, I) which are not needed for the normal growth and development.

Nutrient Cycle in the Soil-Plant-Atmosphere systems



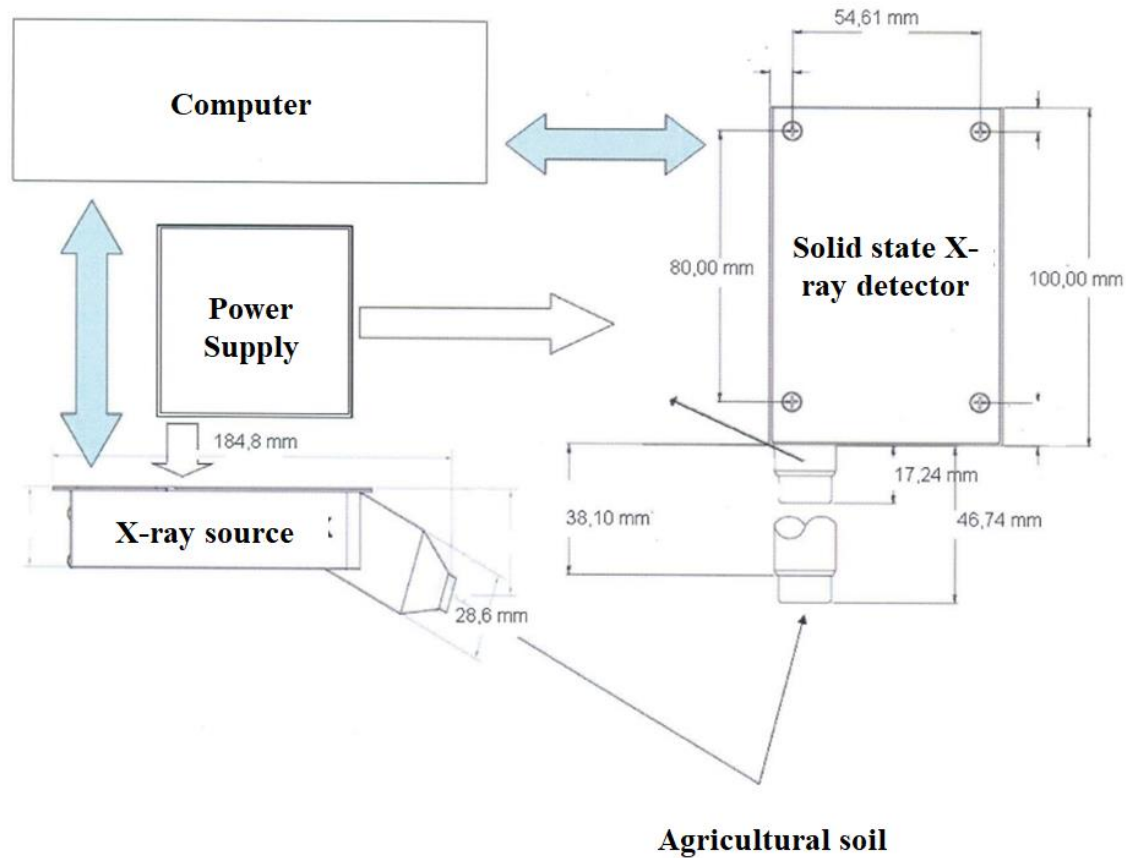
X-ray spectroscopy

Excitation of electrons (example for Fe) when subjected to high energy X-rays

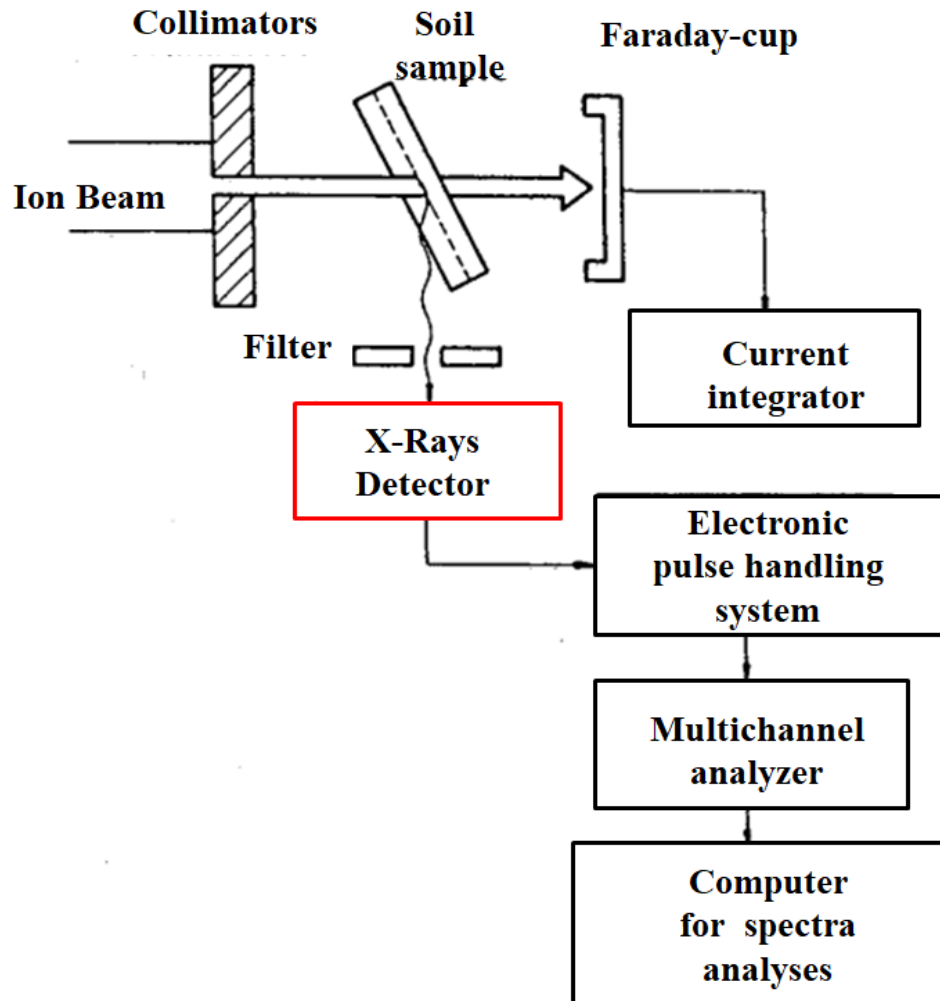


X-ray spectroscopy is a technique that detects and measures photons of light that have wavelengths in the X-ray portion of the electromagnetic spectrum. There are different X-ray spectrometer's configurations and associated methods that can be used for several disciplines and fields of application.

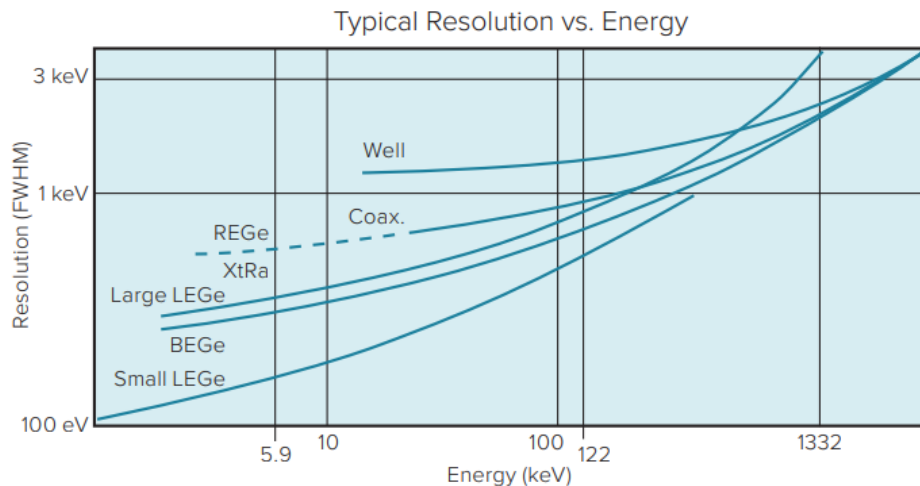
Typical XRF Spectrometer



Typical PIXE Spectrometer



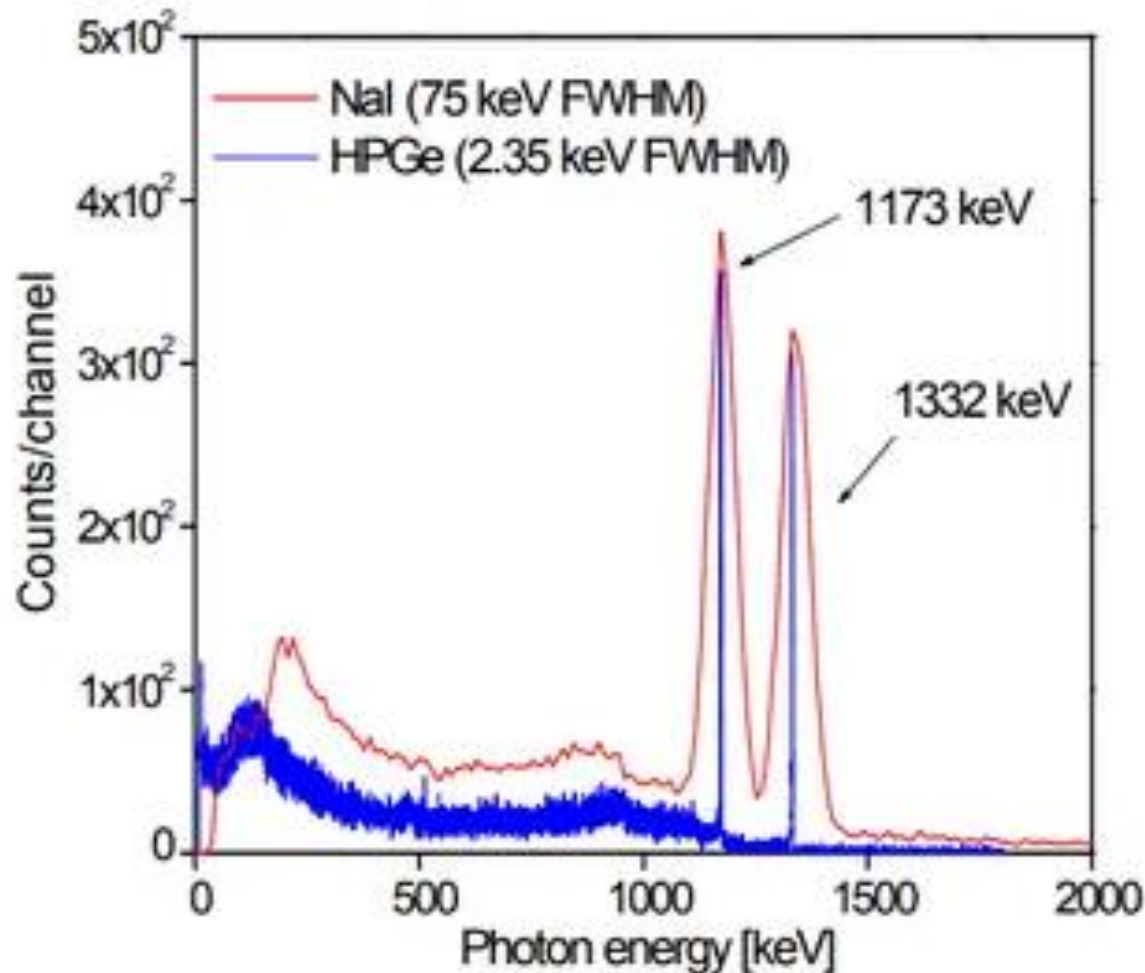
Germanium Detectors



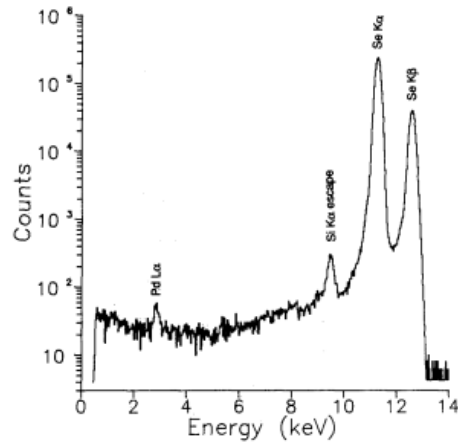
Germanium detectors are semiconductor diodes having a PIN structure in which the intrinsic (I) region is sensitive to ionizing radiation, particularly X rays and gamma rays. Under reverse bias, an electric field extends across the intrinsic or depleted region. When photons interact with the material within the depleted volume of a detector, charge carriers are produced and are swept by the electric field to the P and N electrodes. This charge, which is in proportion to the energy deposited in the detector by the incoming photon, is converted into a voltage pulse by an integral charge sensitive preamplifier.

(Source: Mirion Technologies - Canberra)

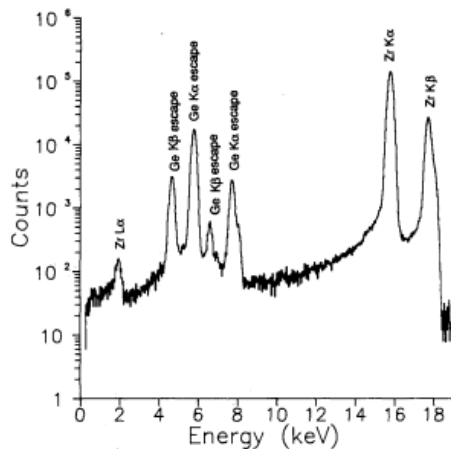
Comparison of NaI(Tl) and HPGe spectra for ^{60}Co



Comparison of Ge and Si(Li) Detectors (in the 2 - 20 keV range)

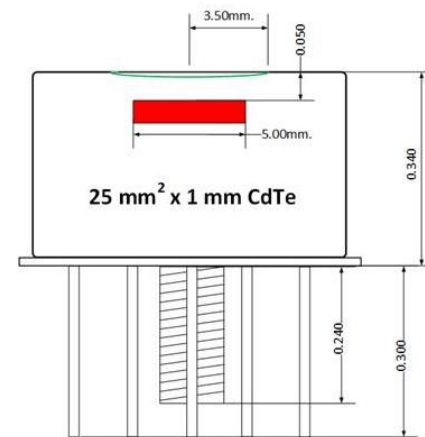
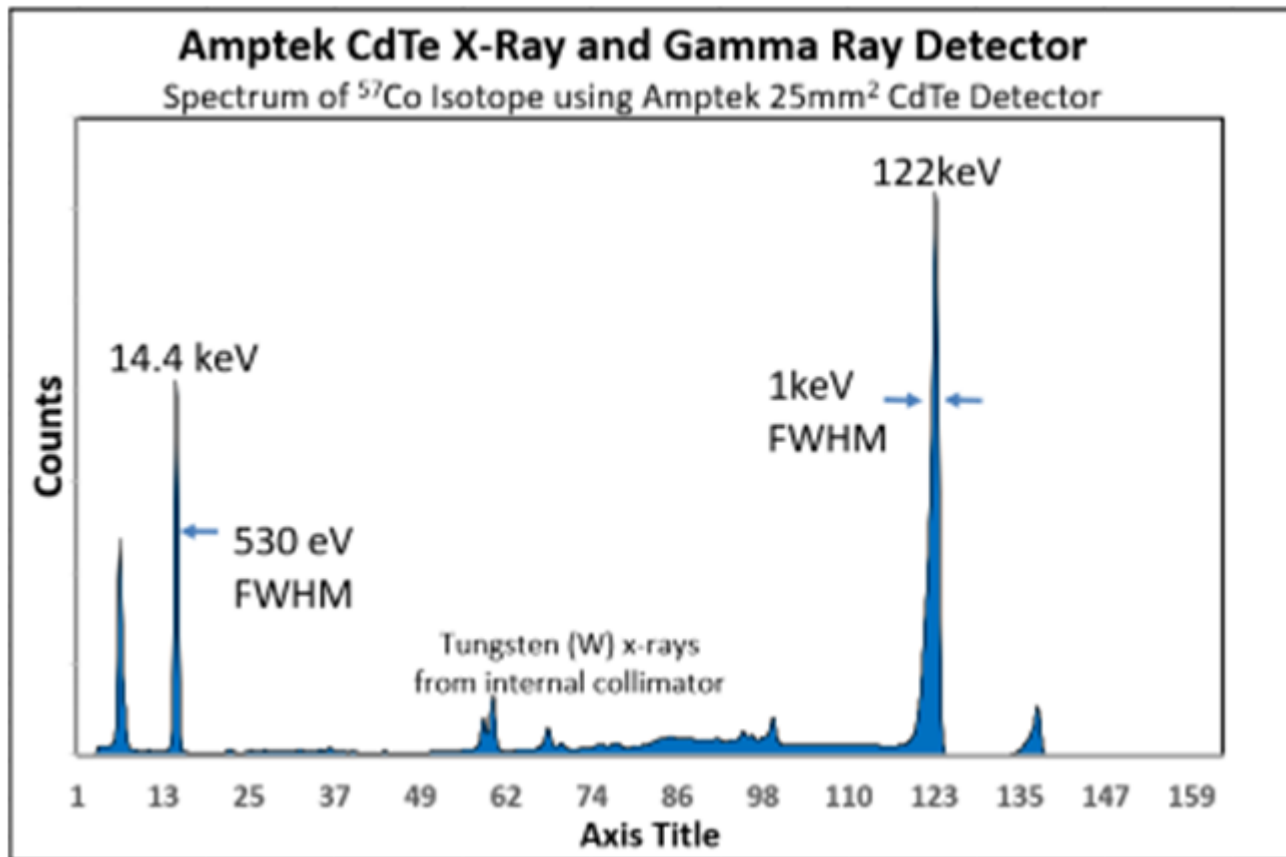


X-ray spectrum from a Se metal foil using a Si(Li) detector. The Pd L photopeak is from the Pd surface barrier contact on the detector.



X-ray spectrum from a Zr metal foil using an HPGe detector.

CdTe X-Ray Detector

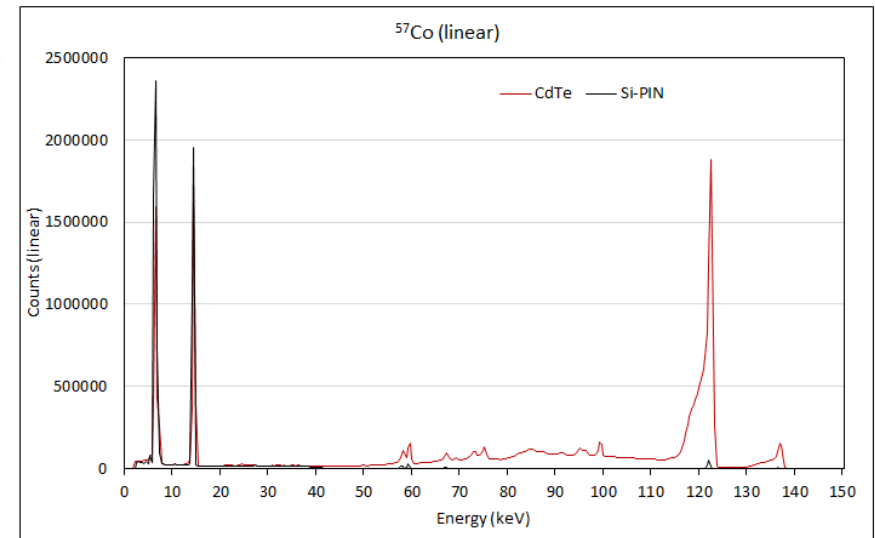
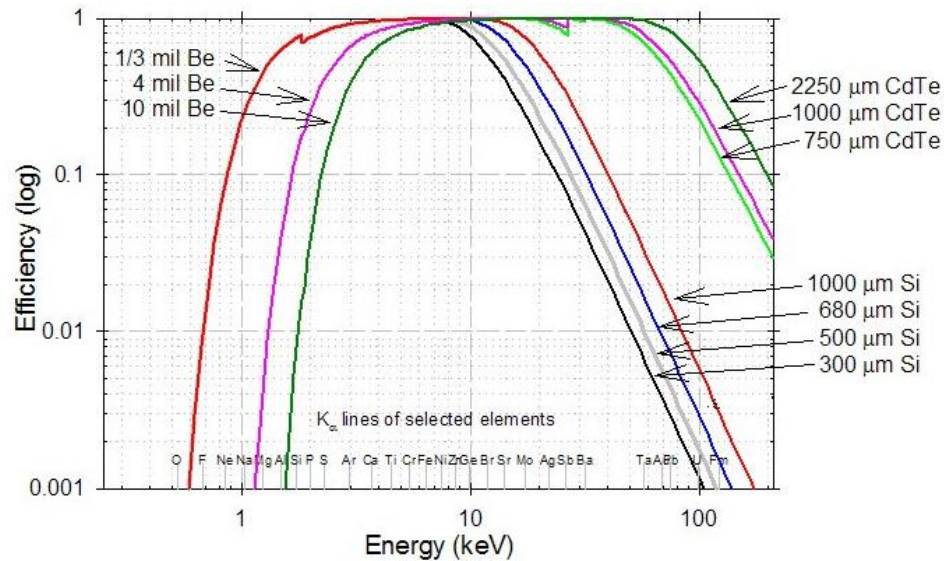


^{57}Co Spectrum
take with the
Amptek XR-100
CdTe detector

Source: Amptek Catalog

The CdTe detector is a thermoelectrically cooled X-ray detector. The high stopping power of CdTe makes excellent for applications requiring high detection efficiency at energies up to 100 keV.

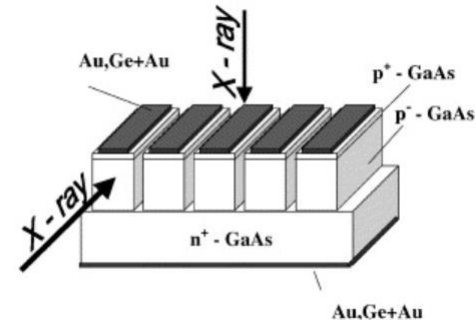
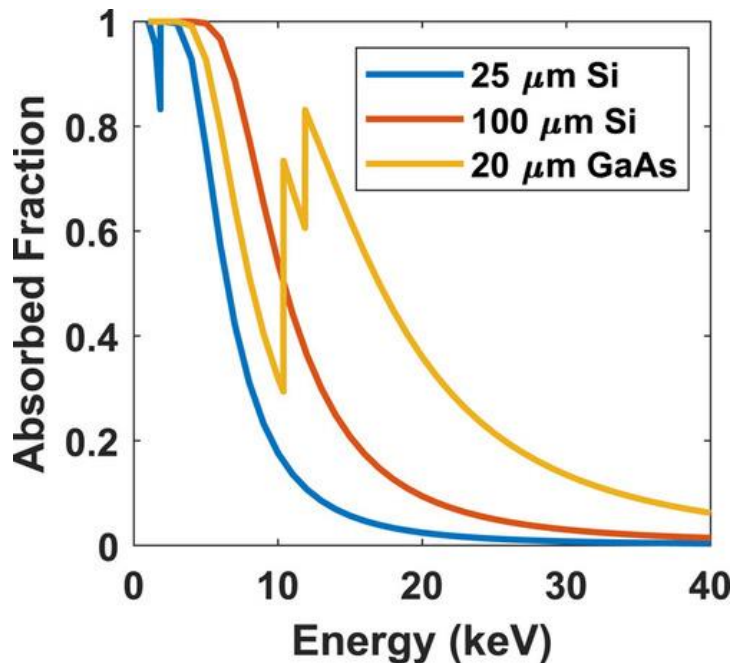
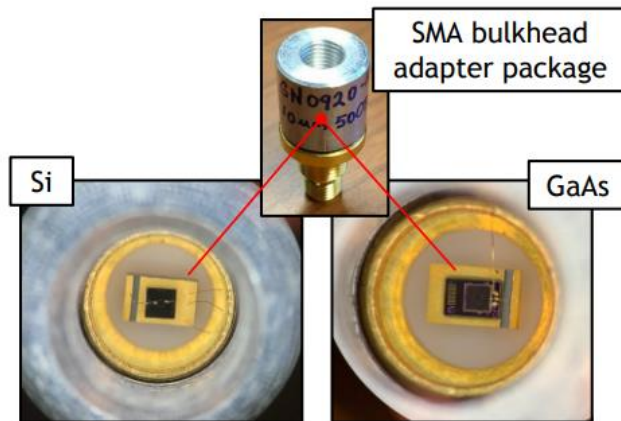
Si-PIN versus CdTe Efficiency: Probability of a photon undergoing any interaction



The photoelectric effect is dominant at low energies but at higher energies above about 40 keV the photons undergo Compton scattering, depositing less than the full energy in the detector.

(Source: Amptek Catalog)

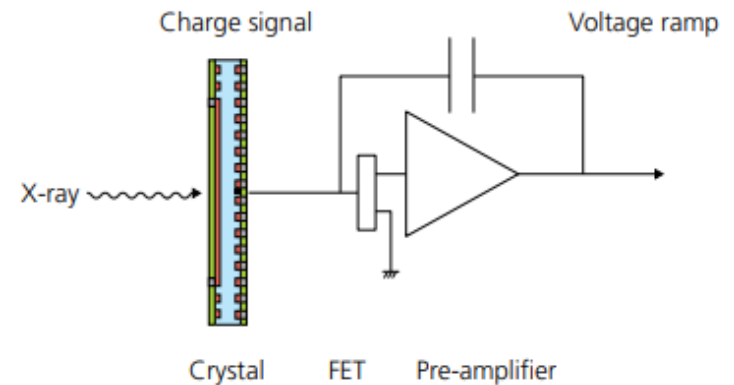
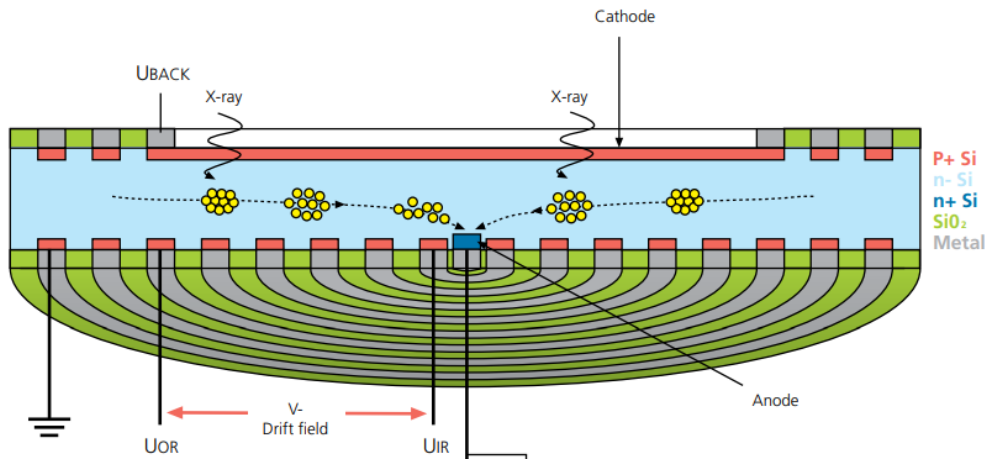
GaAs X-ray detectors



Absorbed fraction of x-rays in selected detector materials and thicknesses. Absorption in Si drops rapidly above 10 keV, leading to rapidly diminishing returns in absorption in exchange for increased temporal impulse response. Above ~ 11 keV, 20 μm of GaAs provides significantly greater absorption than 100 μm Si.

Sources: Looker et al, Review of Scientific Instruments 90, 113505, 2019; Ayzenshtat et al, Nuclear Instruments and Methods, vol. 46, Issue 1, 21 June 2001.

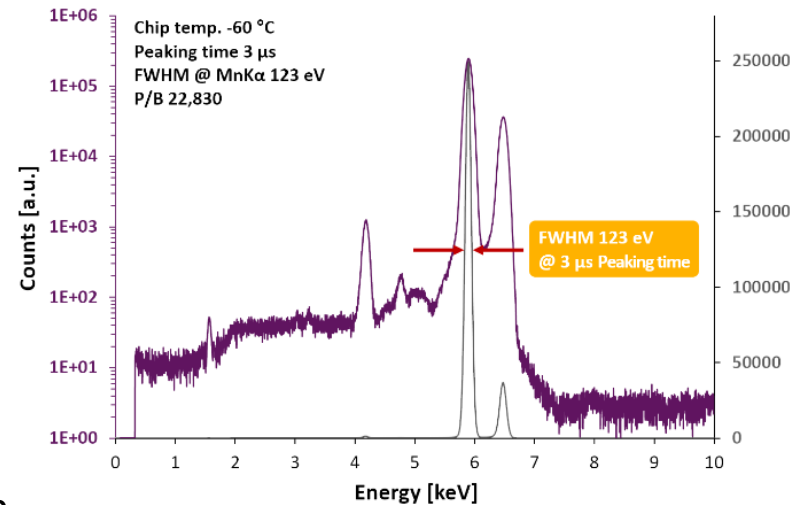
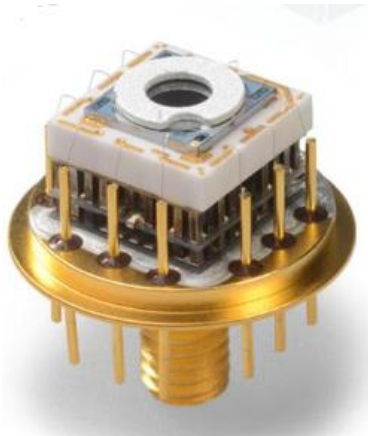
Silicon Drift Detector (SDD)



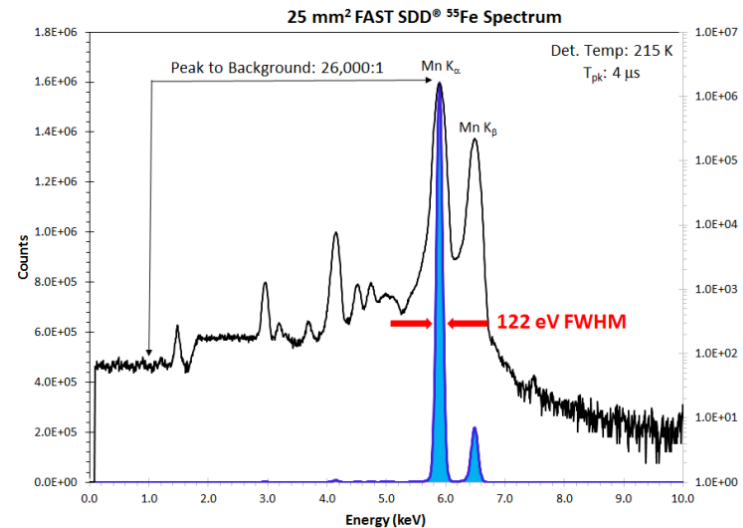
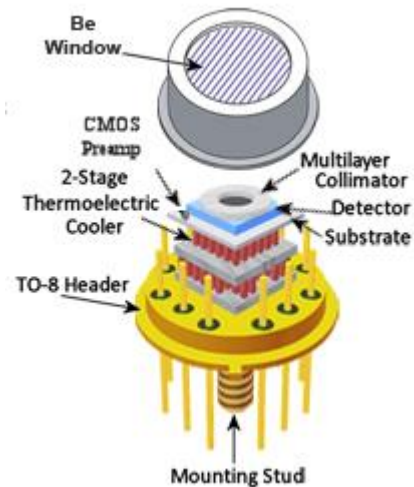
The SDD sensor is fabricated from high purity silicon with a large area contact on the entrance side facing the incoming X-rays. On the opposite side there is a central, small anode contact, which is surrounded by a number of concentric drift electrodes.

When a bias is applied to the SDD detector chip and the detector is exposed to X-rays, it converts each X-ray detected into an electron cloud with a charge that is proportional to the characteristic energy of that X-ray.

SDD and Energy resolution (FWHM)

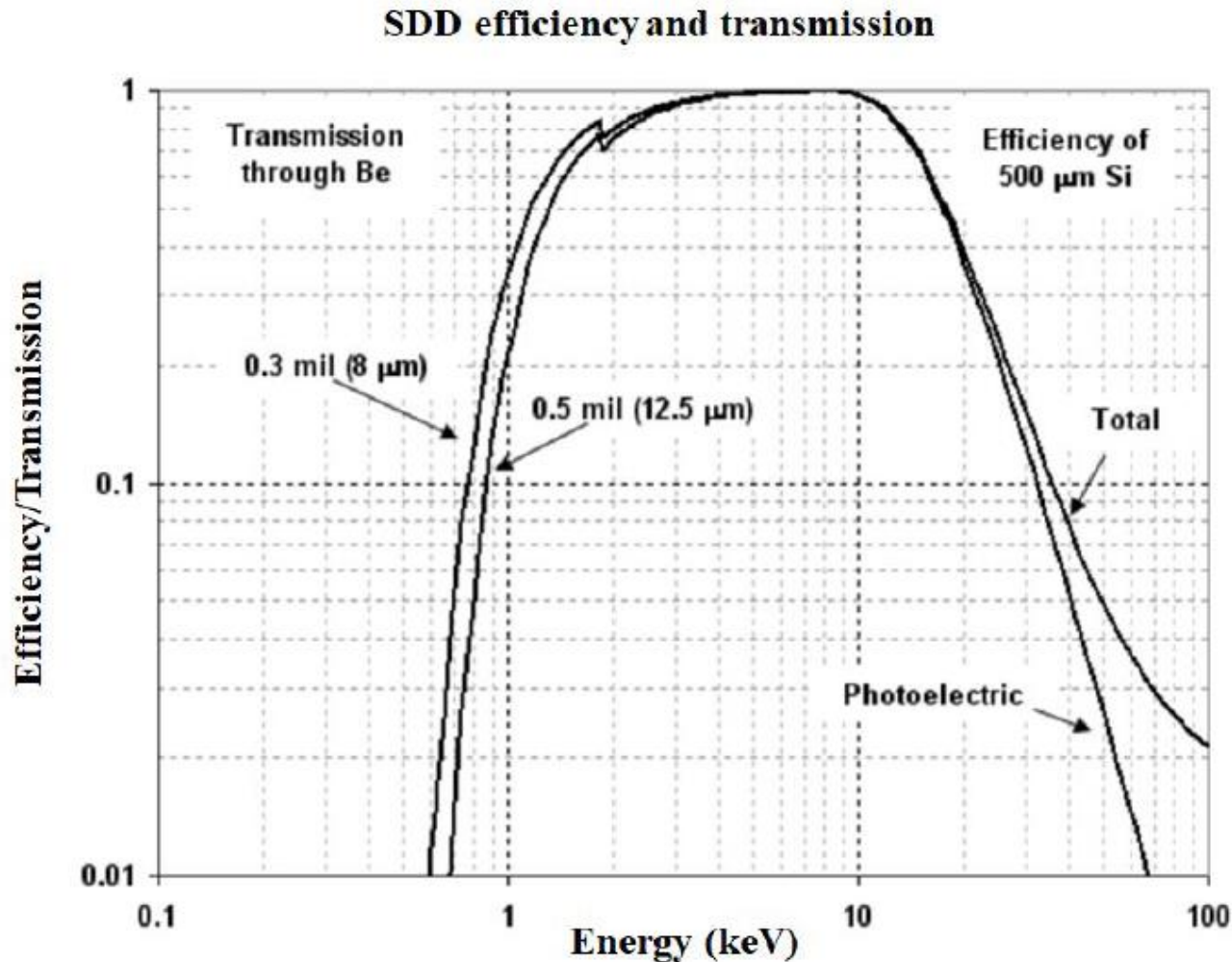


Source: REITER Catalog

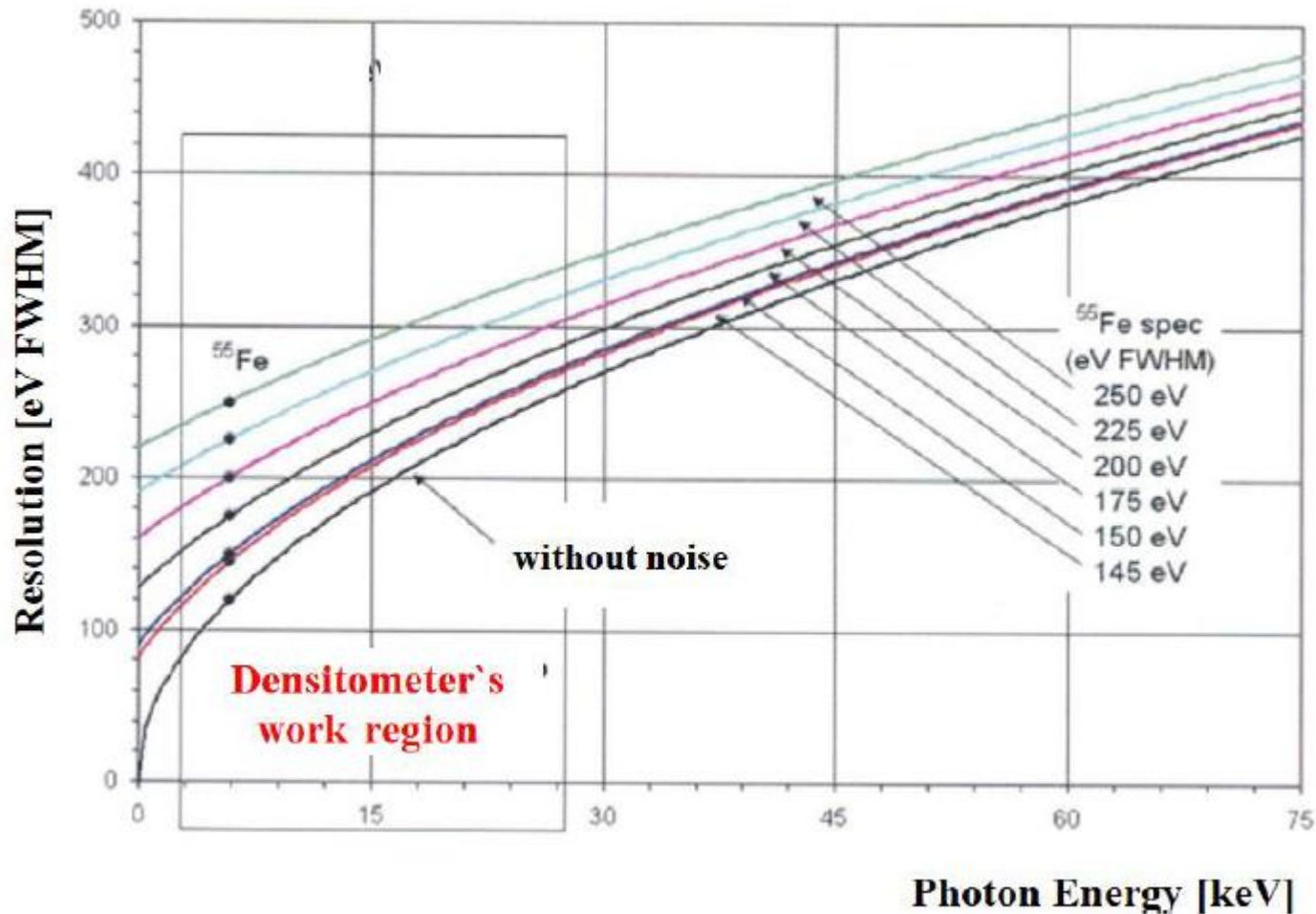


Source: Amptek Catalog

Efficiency versus energy for a typical SDD



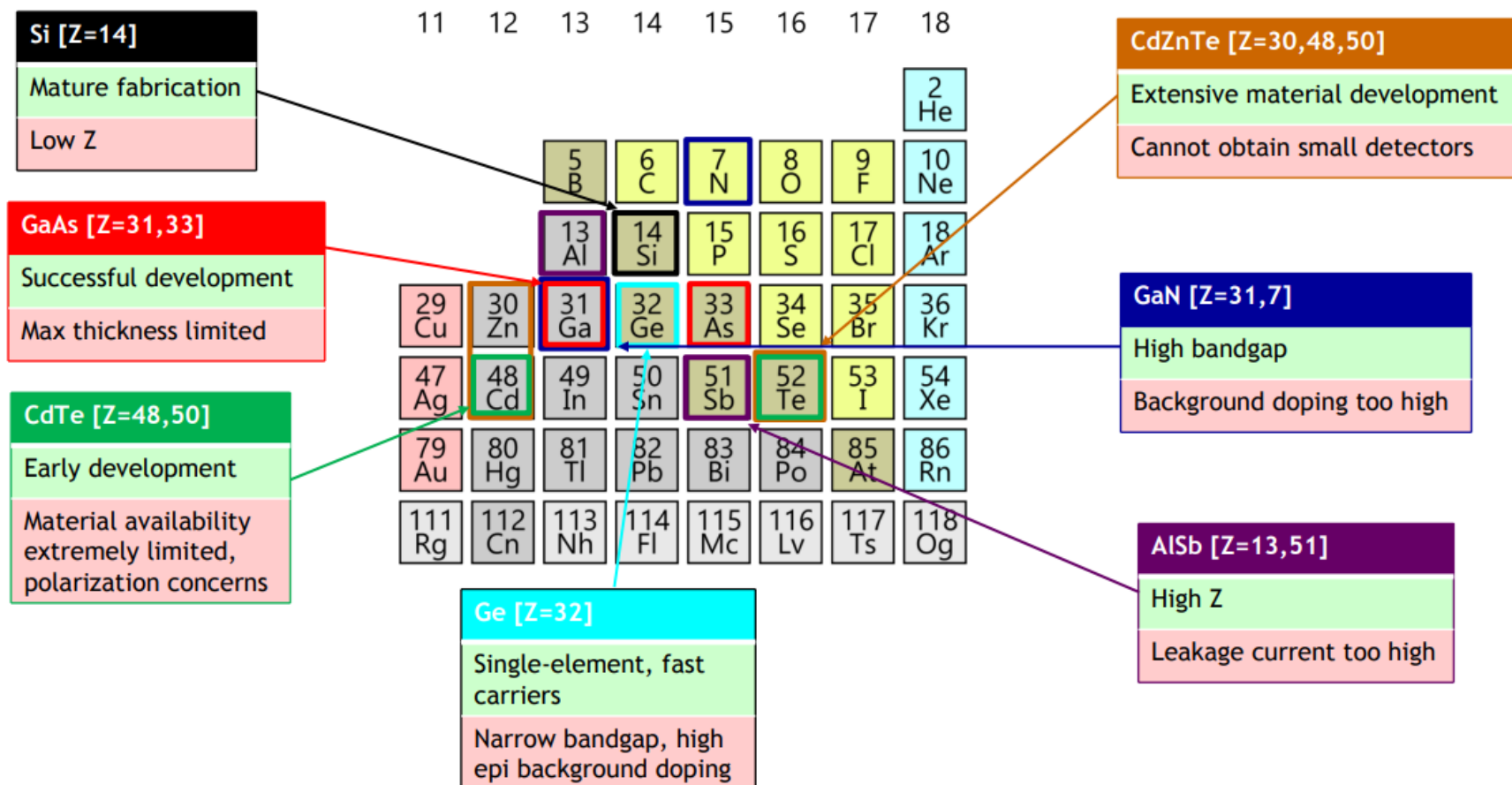
A solid state sensor working region evaluation based on 145 eV resolutions



Opportunities for new sensors for X-ray spectrometry

- There are some prospective opportunities to the future is related to the use of conductive polymers for X-ray sensors.
- The conductive polymers can present numerous advantages such as high sensitivity, short response time, room temperature operation, and the possibility of tuning both chemical and physical properties by using different substituents.
- The conductive polymers used for sensors mainly consist, for instance, of polyaniline, polypyrrole and poly (3,4-ethylenedioxythiophene), among others.

Research on X-ray sensors should continue...



Main Conclusions

- Despite different detectors are widely used in X-ray spectrometry, there are still challenges in relation of needs for improvements for both soft and hard x-ray detection.
- Several studies have been performed in the last decade, all of them looking forward to new possibilities for advanced X-ray detection based on new materials and intelligent electronics for signal processing, and other decision-making computational support related developments.
- The concepts of physics and the analysis tools available or developed by various branches of knowledge and engineering have allowed advances use of XRF and PIXE in agricultural sciences.
- The main challenges are linked to the integration and interpretation of the results at different scales also the instrumentation effort is the development of on-the-go and portable X-ray sensors-based spectrometers. These could allow not only the measurements of stationary elemental concentration values, but also dynamic studies in relation to soil nutrients availability and uptake by plants. In addition, to help in real-time the soil fertilization in variable rate based on the use of precision agriculture concepts.

Acknowledgments

