XRF Instrumentation

Introduction to spectrometer

AMPTEK, INC.
14 Deangelo Drive, Bedford, MA 01730
Ph: +1 781 275 2242 Fax: +1 781 275 3470
sales@amptek.com   www.amptek.com

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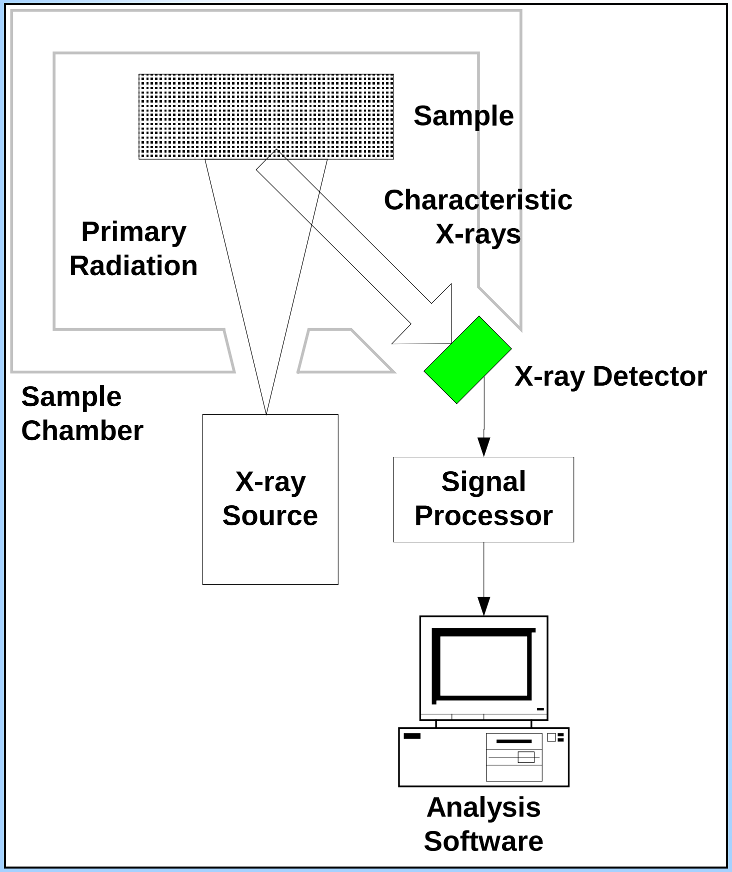
Instrument



- Excitation source
  - X-ray tube or radioisotope
- Spectrometer
  - X-ray detector
  - Signal processing electronics
- Software
  - Spectrum correction and processing software
- Other
  - Radiation shielding
  - Sample fixture

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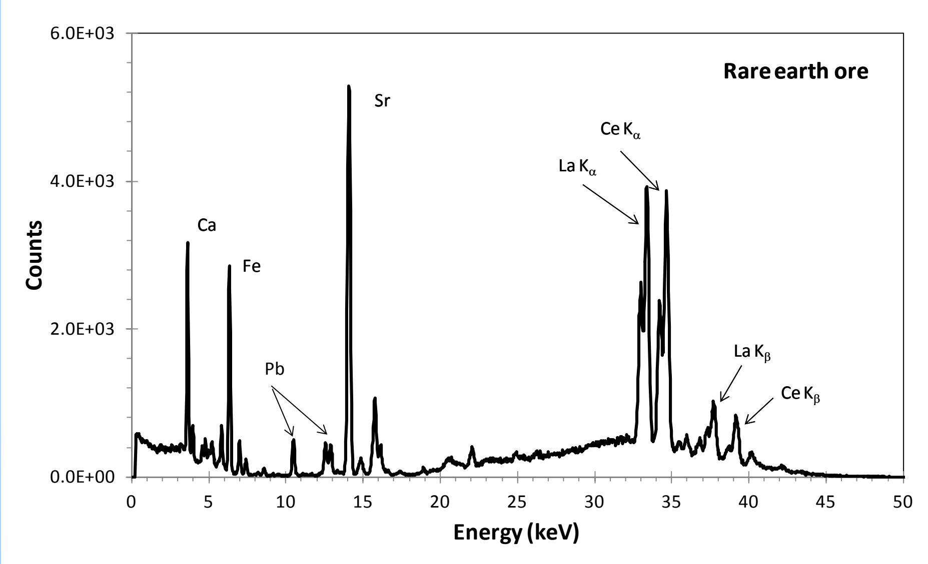
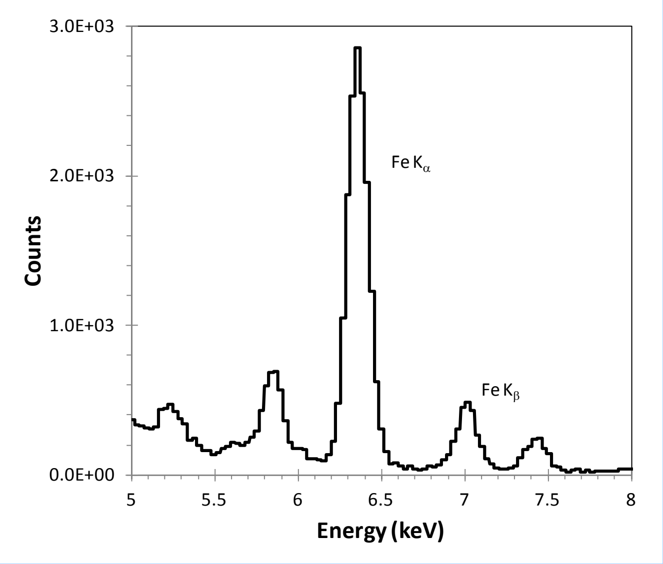
Spectrometer

What is the purpose of the spectrometer?

- Measures energy deposited by each X-ray interacting in detector
- Outputs the spectrum, a histogram showing the number N_i of X-rays in each energy channel, between E_i and $E_i+\delta E$



Typical spectrum



Region expanded to show channels in histogram

Spectrometer

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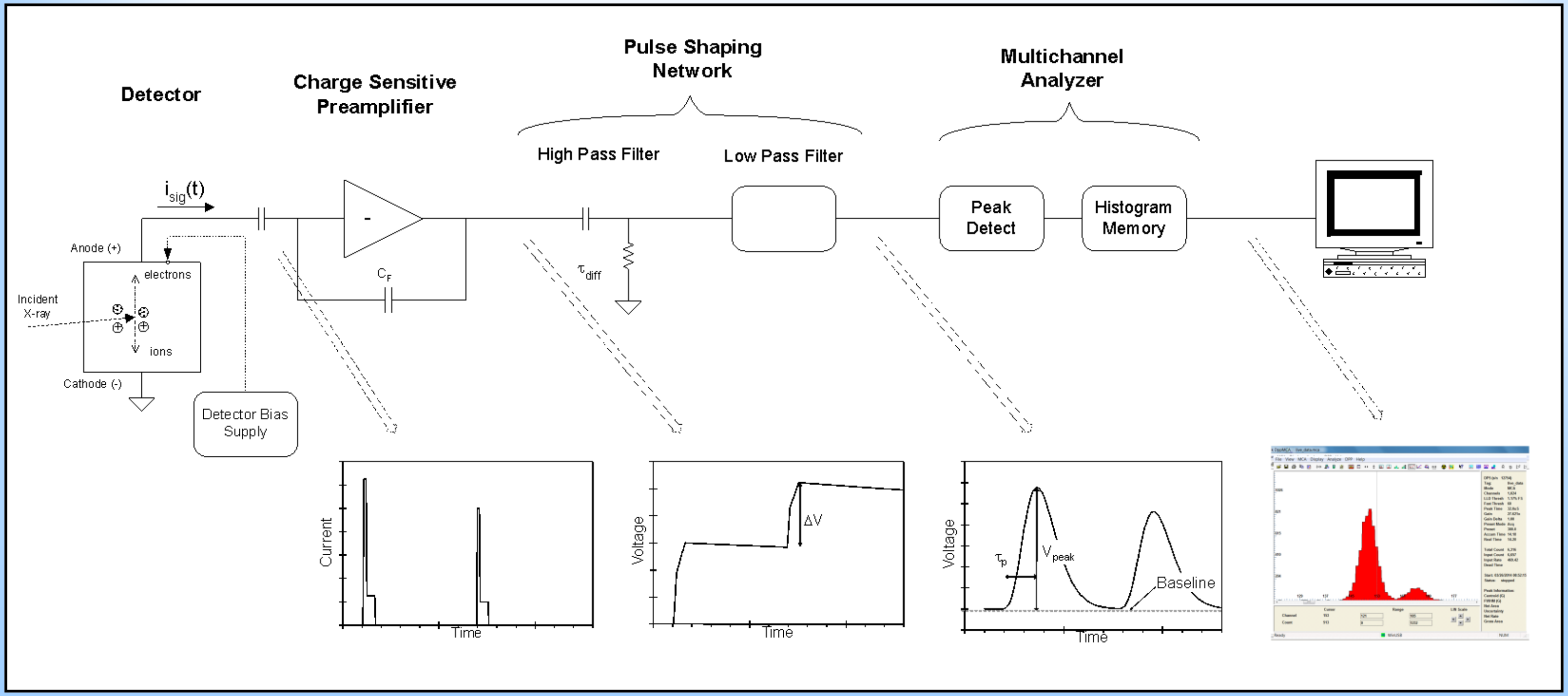
What does the spectrometer include?

- Detector, signal processing electronics, multichannel analyzer



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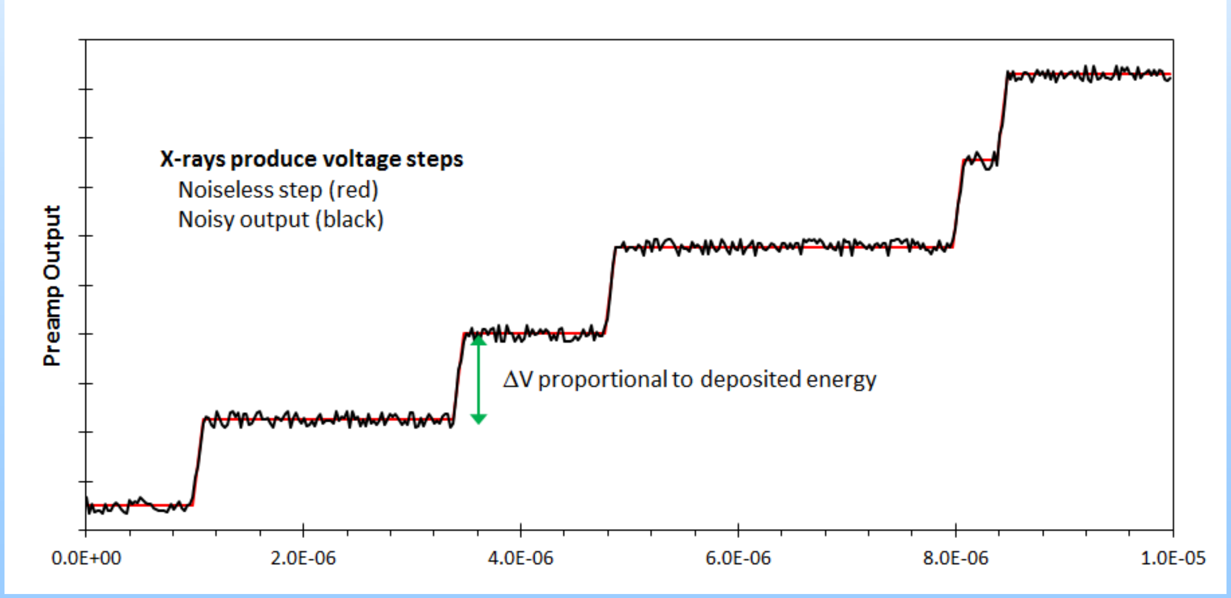
Detector

- Converts energy of each X-ray into a current pulse

Preamplifier

- Converts current pulses into voltage steps where $\Delta V \sim E_{X\text{-ray}}$
- Superimposed on noise (thermal noise, shot noise, etc)
- Pulse timing is random



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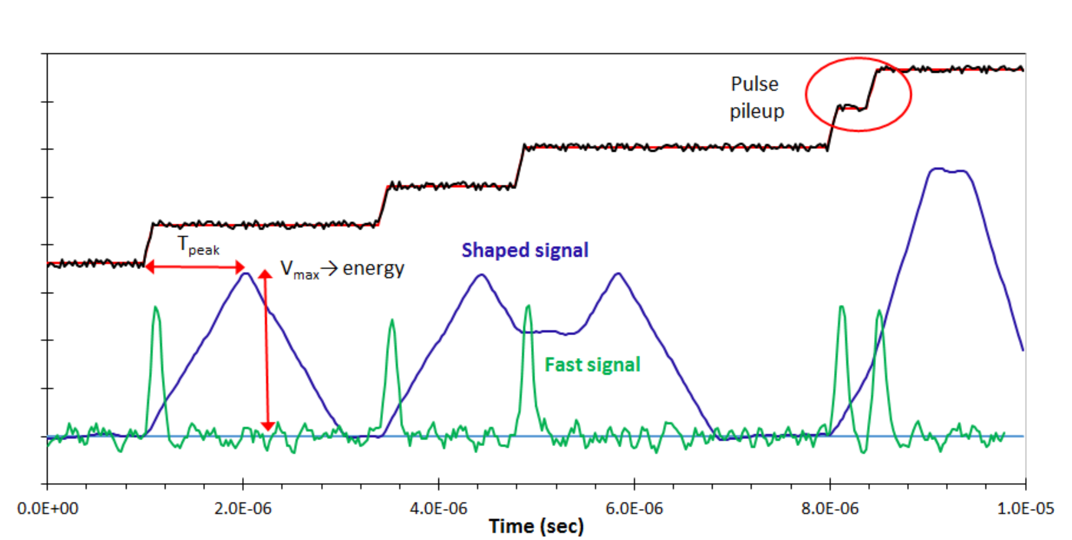
Pulse shaping (a.k.a. pulse processing)

- Removes baseline from voltage steps and applies gain
- Applies a noise filter (i.e. averages over time T_{peak})
- Detects overlapping or piled up pulses, counts input pulses

Multichannel analyzer (MCA)

- Measures energy from pulse height for each X-ray
- Integrates results to produce spectrum as a histogram



Longer T_{peak} →
1) More average, less noise
2) More pileup & deadtime

X-Ray Detector

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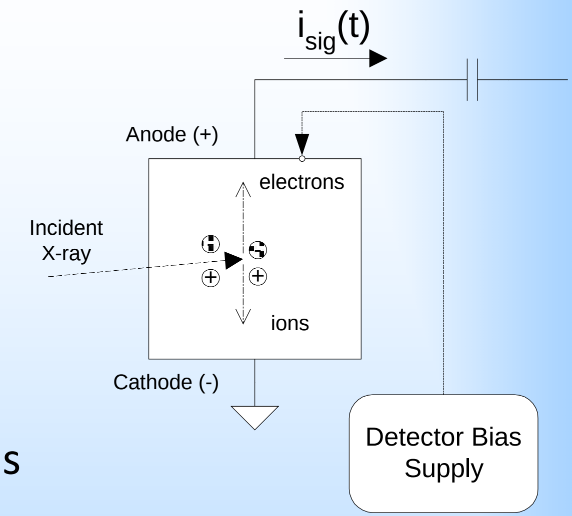
How does the X-ray detector work?



- Non-conducting material between two biased electrodes
- X-ray interaction ionizes material, producing free charge
  - X-ray ejects a photoelectron from an atom in detector; it loses energy by ionizing other atoms in the detector material
  - Number of free charges is proportional to energy deposited: in silicon, 3.6 eV (on average) produces one electron-hole pair
- Charges move toward electrodes, producing current pulse $i_{sig}(t)$
- Energy deposited is proportional to integral of $i_{sig}(t)$

Detector

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What is an ideal X-ray detector?

- Excellent energy resolution
  - Low intrinsic broadening, low electronic noise
- High efficiency
  - Large area, thick active depth, thin dead layers
- High count rates
  - Short signal processing time
- Practical
  - Compact, low cost, rugged, ….

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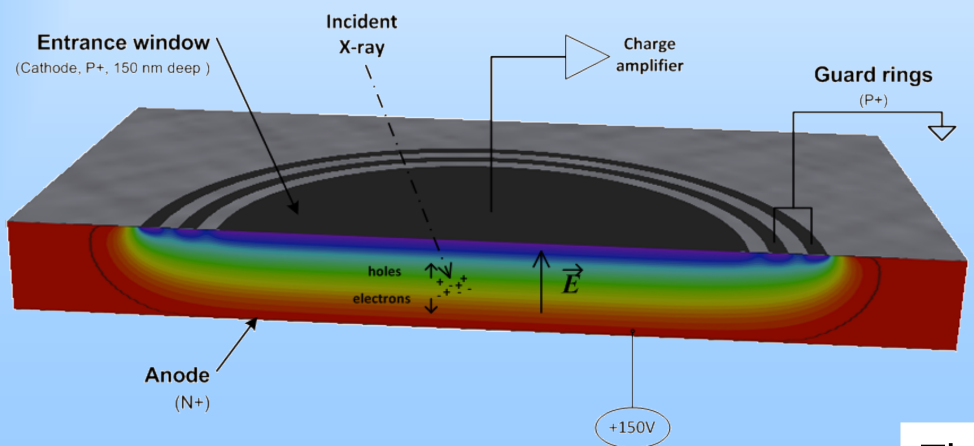
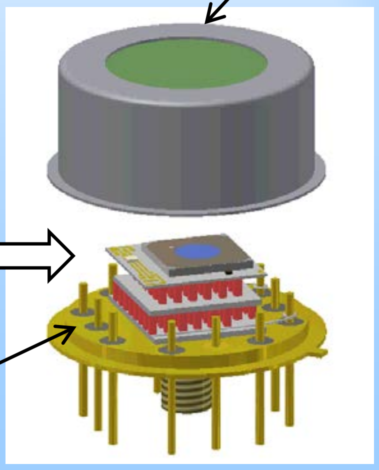
Detector

SiPIN diode

- Planar silicon diode, 0.5 mm thick, 6 to 25 mm^2 active area
- Mounted on thermoelectric cooler to reduce noise
- Resolution 145 to 240 eV at Mn K_α
- Count rates 1,000 to 10,000 cps
- Energy range best from 2 to 30 keV
- Lowest cost, compact, but moderate performance





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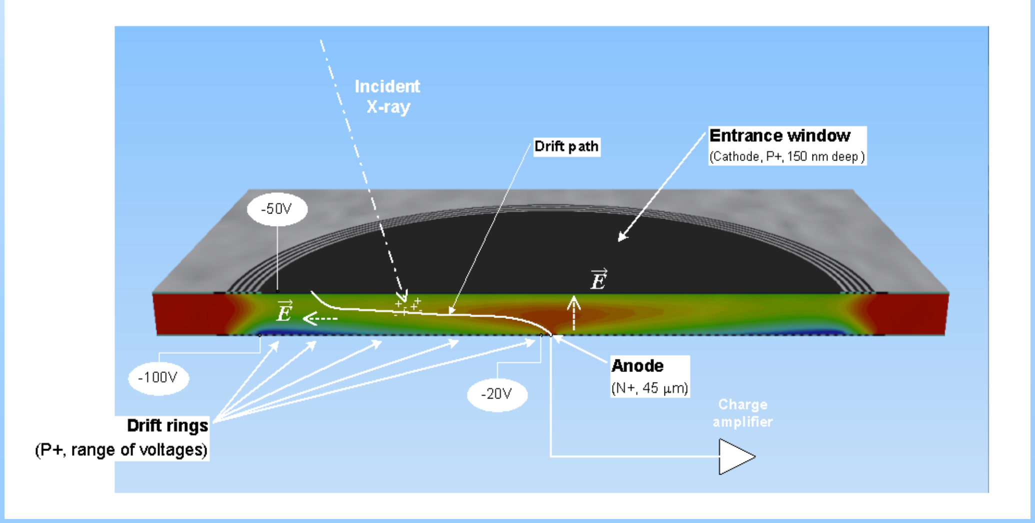
Detector

SDD (Silicon drift detector)

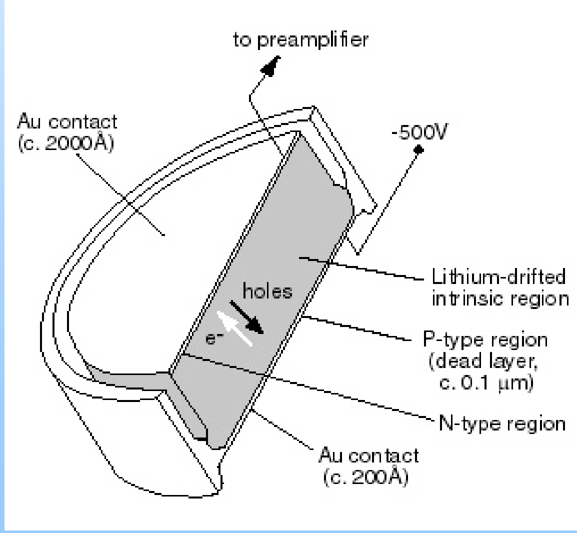
- Also a silicon diode but electrode structure reduces capacitance, improving noise and allowing higher rates. Used with newer FETs
- Up to 70 mm^2 area, thickness up to 1 mm
- Also mounted on thermoelectric cooler to reduce noise
- Resolution 123 eV at Mn K_α (theoretical limit is 119 eV)
- Count rates up to 1,000,000 cps
- Energy down to 50 eV
- Small, compact, rugged
- ***Highest performance for most applications***



Detector

Si(Li)

- Special silicon diode. Available in very large areas and thicknesses
- Requires cryogenic (LN2) cooling so limited to labs
- Resolution 123 eV at Mn K_α (theoretical limit is 119 eV)
- Count rates up to 20,000 cps
- Energy range 250 eV to 50 keV
- Historically, was the highest performance but now replaced by SDDs (higher count rates, no LN2)



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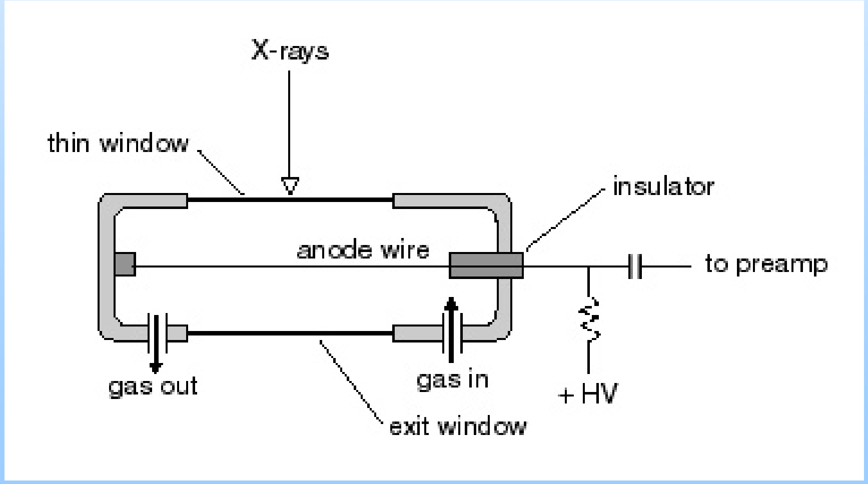
Other semiconductors

- Found in specialized applications, particularly at higher energies
- HPGe (high purity germanium)
  - Higher atomic number, bigger volume gives excellent performance for higher energy X-rays.
  - But require LN2 cooling and is not common
- CdTe
  - Higher atomic number gives much better stopping power.
  - Wide bandgap means it does not need to be cooled as much
  - Exhibits charge collection issues which lead to imperfect spectra
  - Found in portable instruments measuring X-rays from 30 to 100 keV

Proportional counters

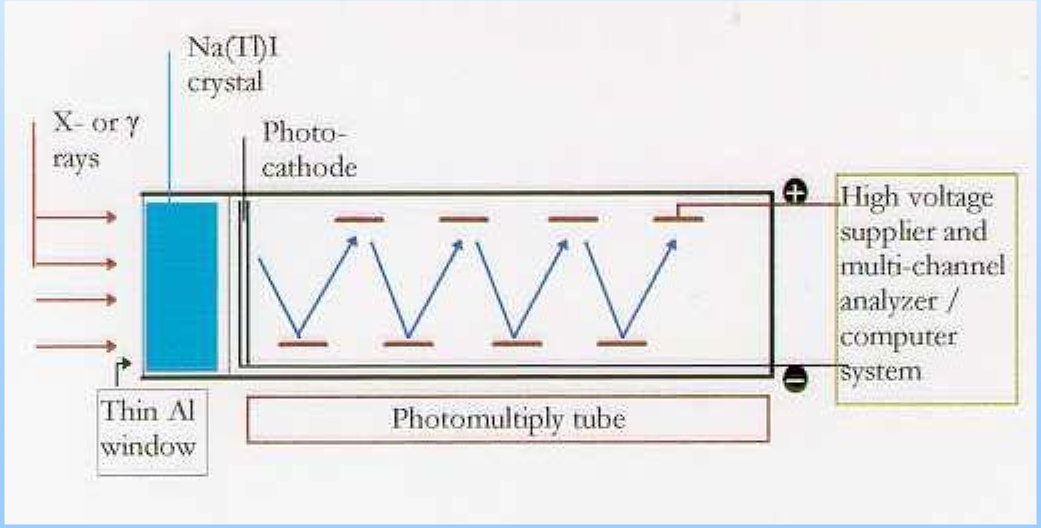
- Gas filled detector (Ne, Ar, Xe) operated at high enough bias to get avalanche multiplication of initial charge
- Can have very large area (many cm_2)
- Resolution poor (500 to 1000 eV at Mn K_{α})
- Count rates high (to 50,000 for EDXRF, 1 Mcps for WDS)
- Historically common but being replaced by SDD in many uses



Detector

Scintillation counters

- X-ray produces light pulses, generated charge at photocathode, multiplied by avalanche gain in PMT
- Can have very large area (many cm_2) and quite thick
- Resolution poor (>1000 eV at Mn K_α)
- Count rates high (to 50,000 for EDXRF, 1 Mcps for WDS)



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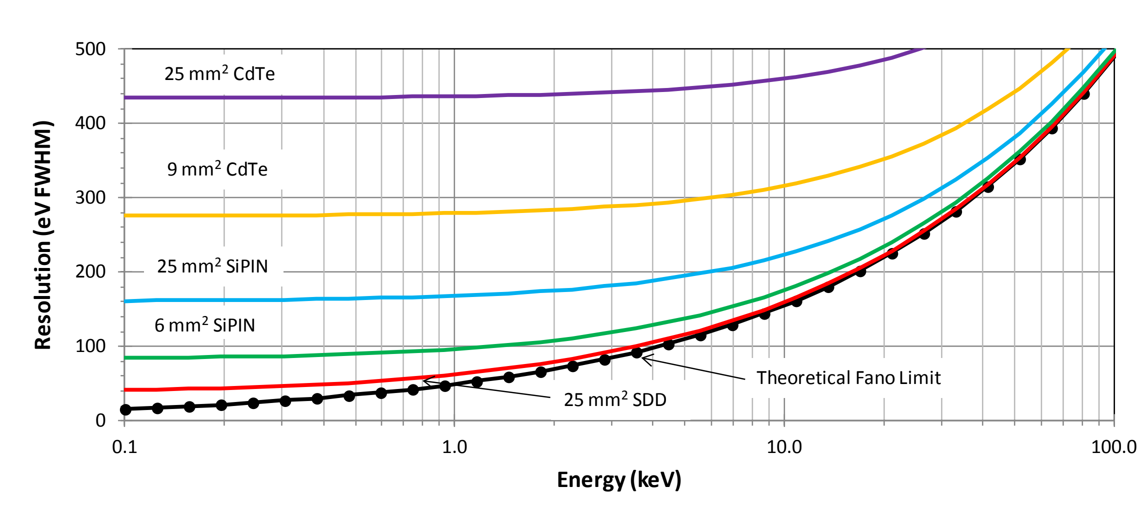
Detector - Advanced

What determines energy resolution?

- Intrinsic resolution
  - Due to quantum fluctuations in producing initial charge pulse
  - Theoretical limit to ionization detectors.
  - Proportional to square root of energy
- Electronic noise
  - From detector leakage current, FET thermal noise, other sources
  - Depends on detailed design of detector and FET, operating conditions, etc
  - Depends on shaping amplifier time constant



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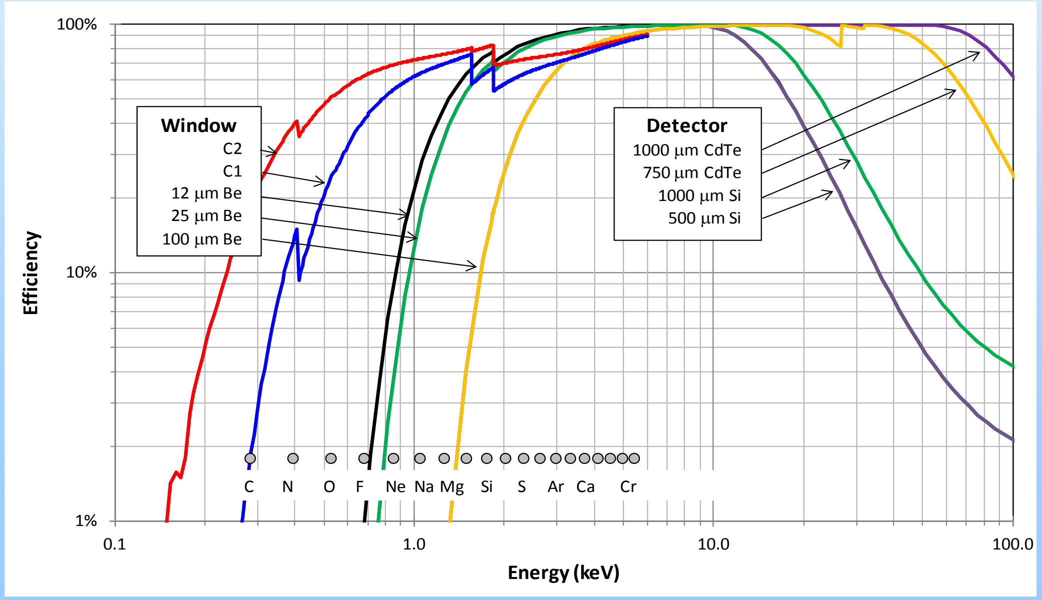
What determines energy range?

- Detector material and thickness
  - In 0.5 mm silicon, 60% of X-rays at 20 keV pass through without interacting
- Window material and thickness
  - A "window" is needed to stop light and keep vacuum inside package (for cooling)
  - Low energy X-rays are stopped by the window
  - Be, most common is good down to 2 keV. Below this, Amptek's C windows are good



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What Amptek detector should I choose?

Silicon drift detectors (SDD)

- **Best energy resolution, highest count rates, and lowest X-ray energies (<2 keV)**
- Fast SDD®: 125 eV FWHM, up to 2×10^6 cps, 25 mm^2 area, P/B 20,000:1
  - FastSDD® with C2 window for energies down to 50 eV
- Standard SDD: 125 eV FWHM, up to 5×10^5 cps, 25 mm^2 area, P/B 20,000:1

SiPIN detectors

- **For moderate energy resolution and count rate where cost is critical**
- Area of 6, 13, or 25 mm^2. Resolution 145 to 200 eV FWHM, up to 10^4 cps

CdTe detectors

- **Recommended for X-ray energies >20-30 keV**
- Efficient up to the U K_α lines (100 keV)
- 25 mm^2 area, 0.75 or 1 mm thick, up to 10^5 cps

AMP TEK®

Amptek Detectors

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Available in many packaging configurations

- Handheld or tabletop instrument?
- Lower cost or higher performance?
- Just getting started, or established OEM?

See www.amptek.com for details

Signal Processing Electronics

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Signal Processor

How does the signal processor work?

- Applies high pass filter to remove baseline between steps
- Applies gain to the signal in the right range
- Applies low pass filter (or average) to reduce noise
- Acquire pulse height (proportional to energy)
- Produces histogram with output spectrum
- Auxiliary functions
  - Detects piled up or overlapping pulses and removes from spectrum
  - Corrects count rate for pulses lost due to overlap or other effects
  - May include other features to eliminate artifacts in the spectrum

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What do I need to know about signal processors?

- End user needs to select the signal processor
  - Most systems today use digital pulse processors rather than the traditional analog pulse shaping circuits
  - Digital processors (a) have both lower noise and higher count rates, (b) have much more flexibility in configuration, to optimize for different applications, and (c) have better stability and reproducibility
- End user should understand the key configuration parameters

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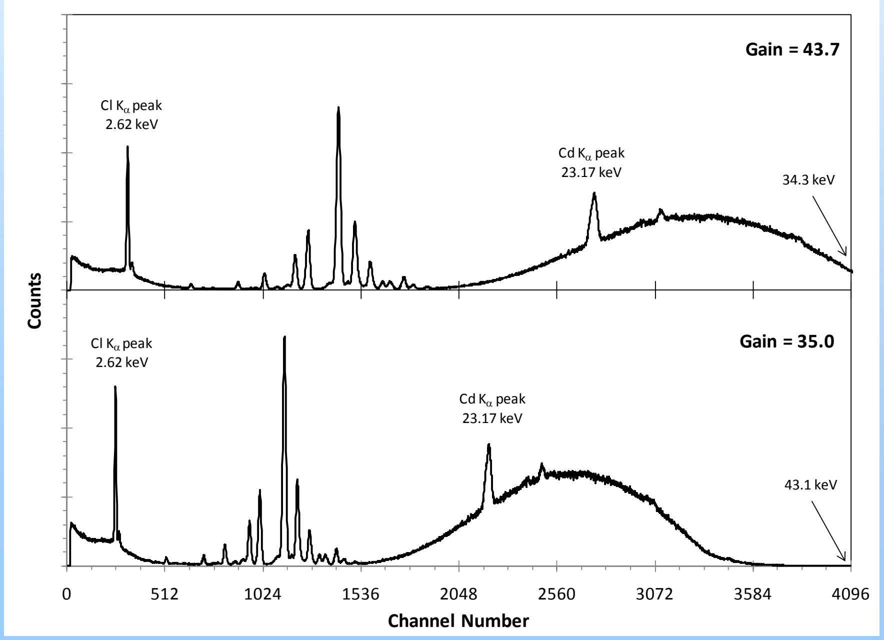
Signal Processor - Gain

Why does the gain matter?

- Gain corresponds to (a) energy calibration (eV/channel) and (b) full scale energy
  - The signal processor works on "MCA channels" ; the gain determines what energy corresponds to the channels
  - If the gain is not right, the photopeaks are in the wrong channels and then huge errors occur (if Fe X-rays occur where software expect Cr, answer will be wrong)

Plot shows same spectra but at two gains.

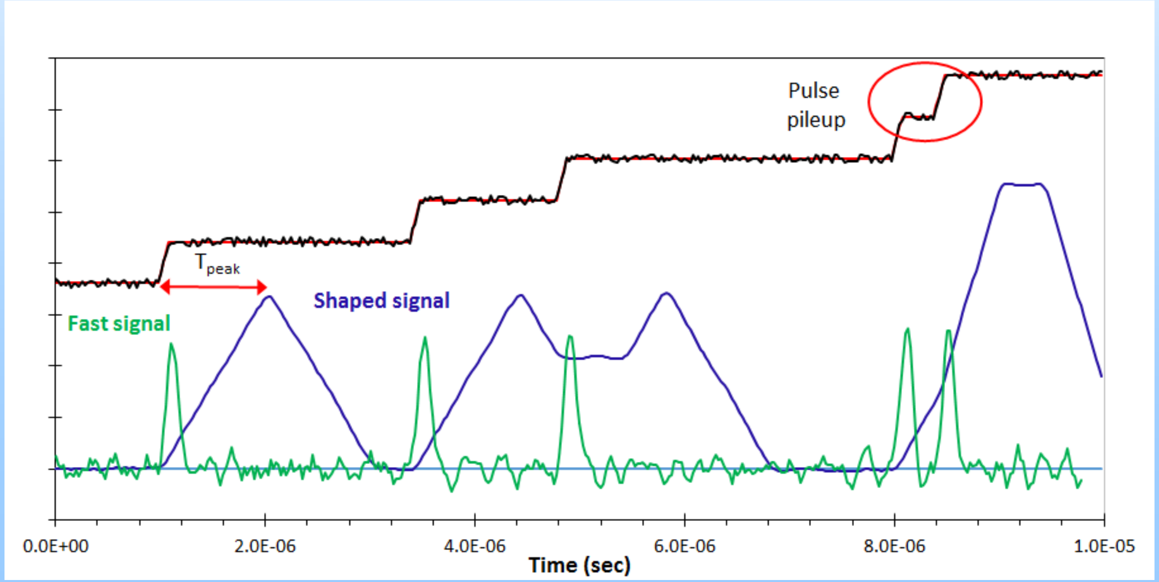
Lower gain → higher full scale energy



Signal Processor - T_{peak}

Why does T_{peak} matter?

- Primary factor in trade-off between resolution and count rate
  - Long peaking time → filters noise better → better resolution
  - Long peaking time → longer dead time per pulse → lower count rates
  - In plot below blue (green) trace has long (short) T_{peak}. Green trace (fast signal) separates overlapped pulses but has more noise so worse resolution



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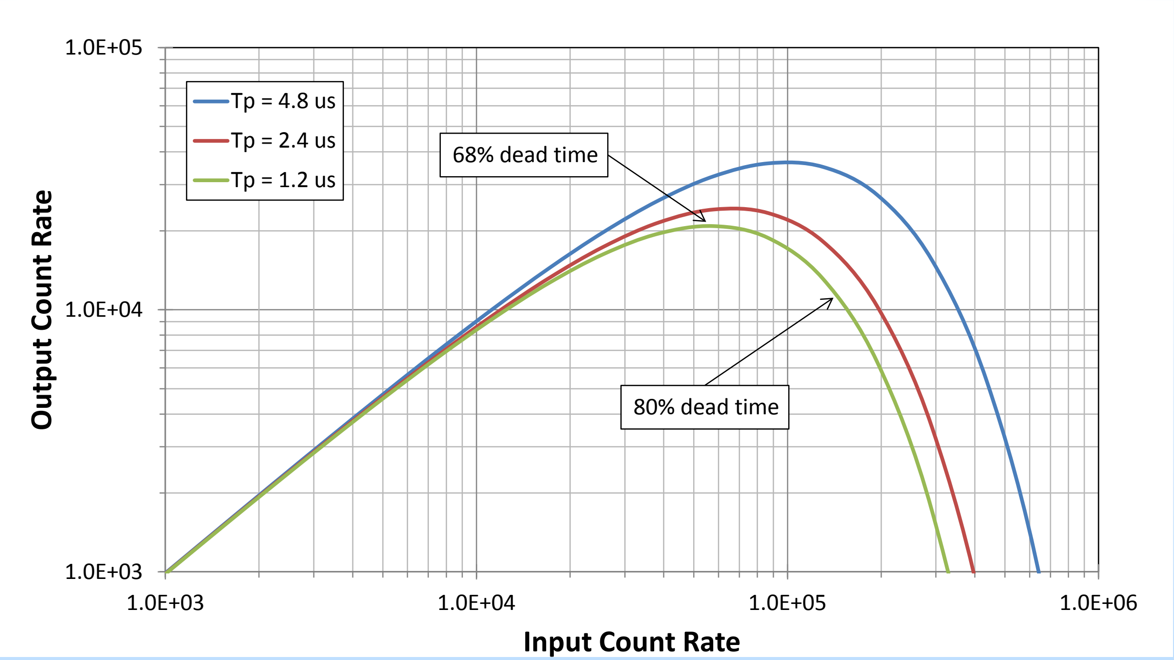
Signal Processor - T_{peak}



- For any T_{peak}, there is a curve of R_{out} vs R_{in} due to dead time losses
  - Curve is independent of detector, due only to pulse duration
  - It does vary with signal processing electronics
- Output rate peaks at 68% dead time→ Never operate above this
  - At 80% dead time, output rate doubles if you DECREASE input count rate x2
  - At 68%, dead time correction is a factor of 3 → subject to errors

Signal Processor - T_{peak}

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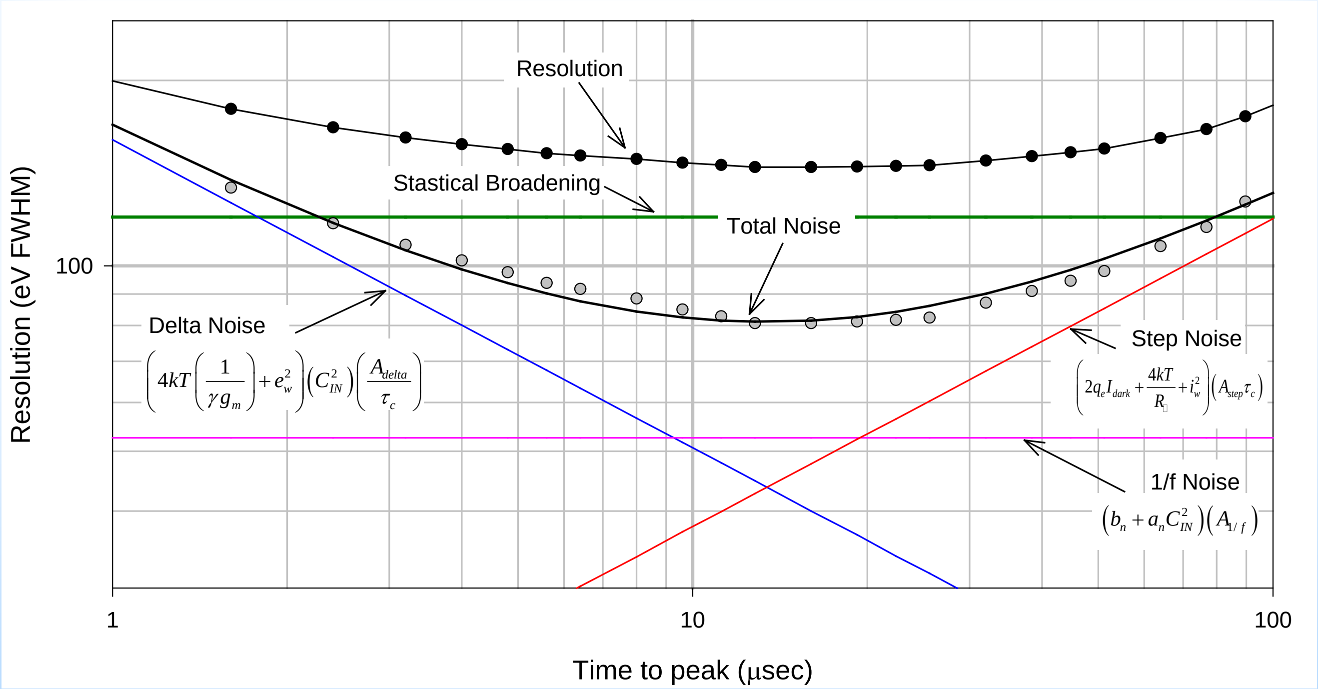

- Noise depends strongly on peaking time (filter noise bandwidth)
  - Every detector and processor produces a curve with this general shape
  - Noise also depends on the signal processor design
- Noise depends strongly on detector type and temperature
- Noise is less important at high energy (intrinsic resolution)

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Are there other parameters that matter?

- In a digital processor, there are many parameters and most of them have some effect on the spectrum!
- Amptek's DPPMCA HELP file and online FAQ discuss many
- Most important ones in Amptek DPPs:
  - Fast and slow channels
  - Thresholds
  - Flat top
  - Number of channels
  - Detector power controls:

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What Amptek processor should I choose?

DPP Family

- All of Amptek's signal processors are based on the latest generation of digital pulse processing technology (the DP5 core).

X123

- Compact, complete system (detector, preamp, processor, and power supplies)

DP5 board

- Board level solution for OEMs needing to integrate spectrometer in a compact instrument

PX5

- Bench size signal processor, most suitable for laboratory systems

Custom

- Amptek can prepare custom designs for manufacturer's with specific constraints

Detector compatibility

- Most often used with SiPIN and SDDs but these are available for use with scintillators, proportional counters, Si(Li) and HPGe detectors, etc

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X-123

- Includes:
  - X-Ray Detector & Preamplifier
  - Digital Pulse Processor and MCA
  - Power Supply and PC Interface
- Available with SDD, FAST SDD, Si-PIN and CdTe detectors
- Optional extensions up to 9” (23 cm) for use in vacuum

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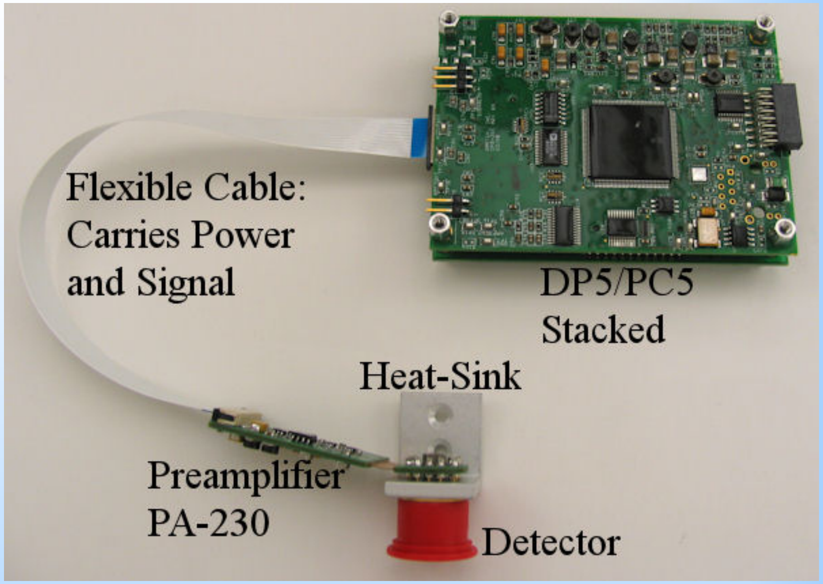
DP5 & PC5

- Board level solution
- For use with AXR Detectors
- Provides OEM customers with mounting options as they design in Amptek detectors & electronics
- Includes DPPMCA software and SDK to help OEM interface to their system



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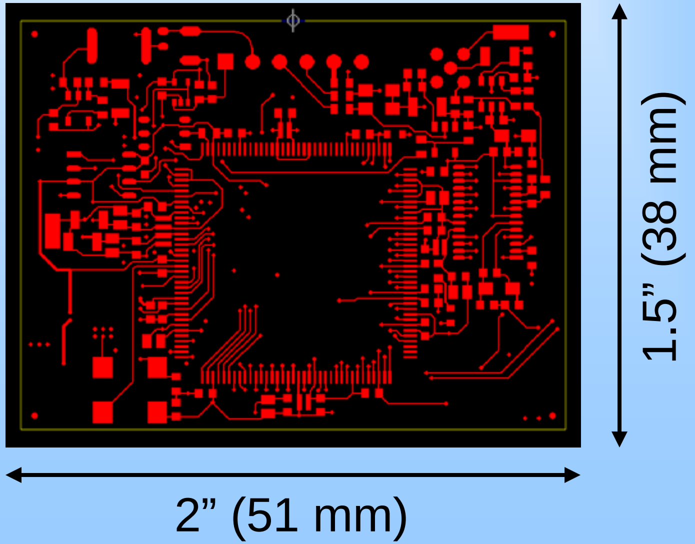
Amptek Processors

PX5

- Enhanced DPP, MCA & Power Supply
- For XR-100 or other detector types
- Primarily for lab use

Custom

- Design DPPs for specific OEMs
- Tailor size, footprint, power, capabilities



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Spectrometer performance

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Performance

Why do energy resolution and count rate matter?

This presentation has discussed these often:

- Why do they matter?
- When do they matter?
- Which is most important?

Do other performance parameters matter?

– Yes. But which ones matter most depends on the application.

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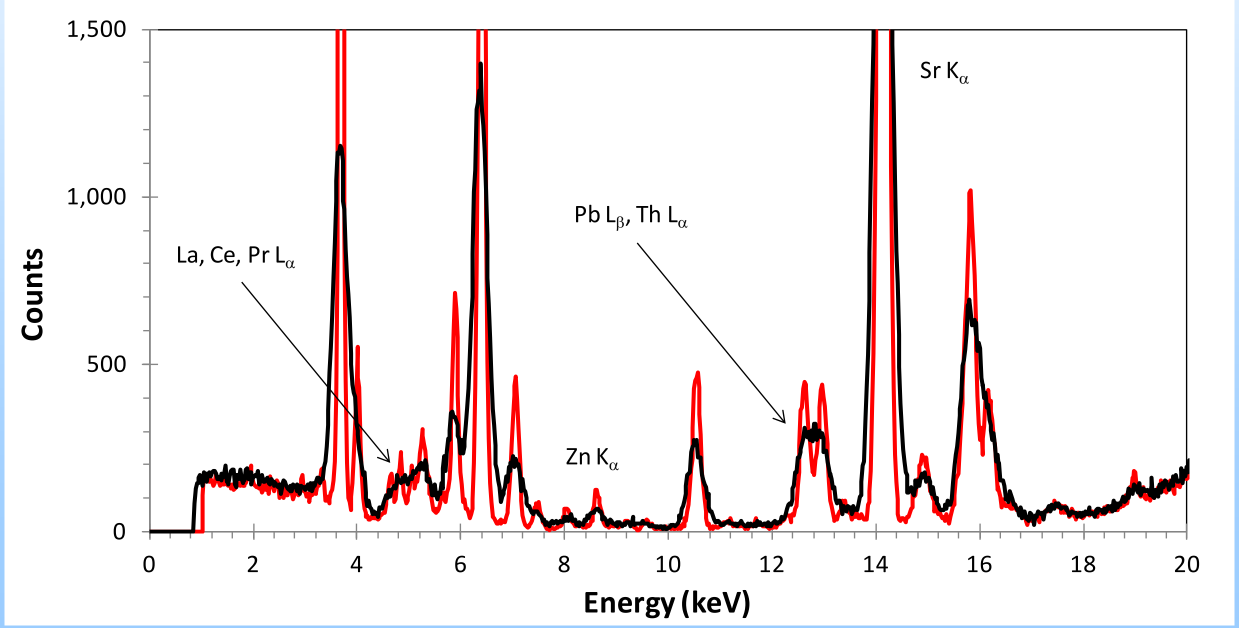
Why does resolution matter?

- Separate closely space peaks (e.g. Pb and Th L lines below)
- Improve signal to background (e.g. Zn K_{α} below)
- Less important for strong, separate lines (e.g. Sr K_{α} below)



Performance

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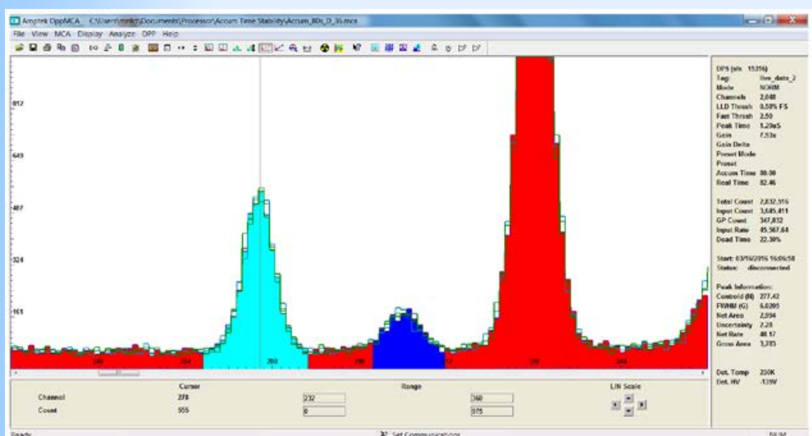
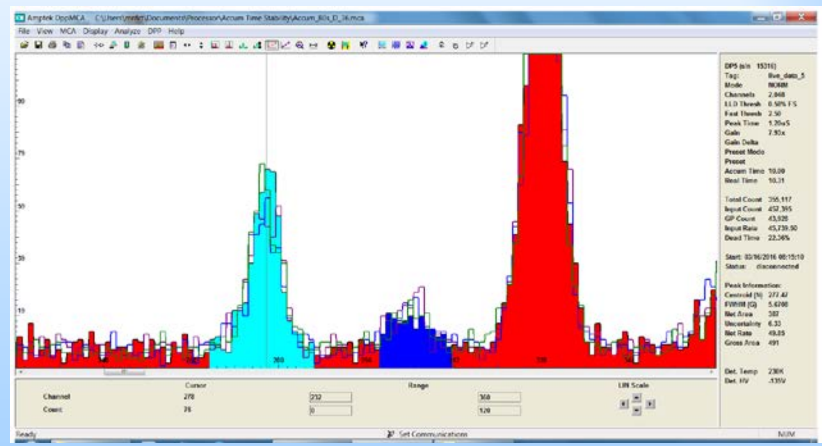
Why does count rate matter?

- Precision of measurement improves as $\sqrt{(N_{X\text{-rays}})}$
- 1% precision → 10,000 X-rays   0.1% precison → 1,000,000 X-rays
- A good measurement means either high rate or long measurement time





Spectra on left taken for 8x longer than on right. On the right, there is more variability in counts in light blue peak, and dark blue is hard to distinguish from background

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Performance

Which is more important: good resolution or count rate?

- It depends on what you are trying to do.
- Case 1:
  - Measuring only strong photopeaks with negligible background or overlapping peaks
  - **Count rate is most important**. As long as the peaks are resolved and above background, improving resolution beyond this will not matter.
- Case 2:
  - Measuring a single weak peak, where background is important but no overlapping.
  - **Resolution and count rate are equally important**. You can get the same improvement in the measurement if you double the count rate or cut the FWHM in half.
- Case 3:
  - Measuring a weak peak with high background and overlapping peaks
  - **Resolution is most important**. You will gain much more in the measurement result by improving the resolution, even if you decrease our count rate
- For more information, see R. Redus & A. Huber, *Figure of merit for spectrometers for EDXRF*, X-ray Spectrom, Vol 41, Issue 6, pp 401-409, Dec 2012

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Additional Advice

Novice users often underestimate the important of optimizing the excitation source. They assume that, if they are exciting X-rays, the spectrometer is configured properly, and the analysis software has been set right, they will get good results.

One will, indeed, be able to see a signal. But the precision, accuracy, and detection limits will be much worse than they anticipate unless they are careful with the excitation conditions.

Rules of thumb:

- Excitation energy must be 2 keV above line you are measuring to see anything
- Best excitation is usually at a kV 2-3 times the energy of the line
- Best filter will use a K edge about 1.5 time energy of the line

40 kVp with no filter is "jack of all trades, master of none". It will show all lines but will do none well.