DIGITAL SPECTROMETER FOR AUTOMATING XAS DATA COLLECTION

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Introduction

Energy dispersive x-ray fluorescence is a well developed experimental technique for studying both the atomic composition of unknowns (fluorescence analysis) and the atomic structure of matter at the atomic level (EXAFS). Table 1 lists various common applications. In order to assist researchers interested in extending their capabilities with multi-element detectors, X-ray Instrumentation Associates recently introduced the DXP-4C (Figure 1) which contains 4 complete channels - amplifier through MCA - of electronics in a single width CAMAC module. As shown in Table 2, using these new electronics can improve throughput considerably, compared to either an conventional analog SCA system or a multiplexed MCA system, while also allowing complete computer control of all parameters. This new module allows even detector arrays with large numbers of elements to be instrumented very compactly, as shown for a 19 element array in Figure 2.

Table 1: Applications for Energy Dispersive X-ray Fluorescence
Atomic Composition - X-ray Fluorescence Analysis
Semiconductors: contamination control
Petrochemicals: feedstock monitoring
Coatings: composition monitoring
Geology: ore & mineral analysis
Forensics: unknown determinations
Environmental: cleanup & toxics monitoring
Atomic Level Structure - X-ray Absorption Spectroscopy
Biology: metalloprotein structures
Chemistry: structure of catalysts
Surface Science: local binding structures
Geology: structures at water/mineral interfaces
Environmental: toxic/mineral bound state structures

Application

The DXP-4C contains four complete spectrometers on a card. Input from the preamplifiers enters through the front panel and complete spectra, one per detector, are output through the CAMAC interface. While it might initially seem that the fact that the DXP-4C is a CAMAC module might hinder its use with several other common instrumentation systems, such as VME, this is not generally a significant problem. This is because, for best results, the DXP-4C's should be installed in the hutch close to the detector, in a CAMAC mini-crate, in order to minimize cable lengths from preamplifiers to spectrometers and thereby minimize noise pickup. In this mode only digital spectra are shipped out of the hutch into the noisy synchrotron laboratory environment. CAMAC crate controllers are available to work with many standard interfaces, including VME and SCSI, allowing the detector plus its CAMAC electronics to operate as a stand-alone instrument. Because all DXP-4C controls are digital, no hutch access is required during normal operation. Further, by supplying the detectors' preamplifiers from a CAMAC module, as shown in Figure 2, potential ground loops are eliminated, further reducing noise.

Functional Design of the DXP-4C

Figure 3 sketches the functional design of a single DXP-4C channel. The preamplifier input is conditioned using analog electronics and then digitized using a 10 bit 20 MSA ADC. The primary spectroscopy functions are implemented in a "FiPPI" (for Filter, Peak Detector, and Pileup Inspector) using digital logic, typically in a field programmable gate array (FPGA), using a fast-channel/slow-channel architecture. Its functions include pulse detection, pileup inspection, pulse filtering, peak capture, and input count rate counting. Each time the FiPPI fast channel detects a pulse which does not suffer pileup in the slow channel filter, it captures its slow filter peak value plus some related signal processing values. These captured values are passed to a digital signal processor (DSP) which corrects them to achieve good energy resolution and then bins them to generate spectra. The DSP also handles the interface to the outside control computer, accepting operating parameters and sending back spectra. The DSP has adequate internal memory to generate 1000 channel spectra. Additional memory can be added for up to 16K channels.

FiPPI Structure

Figure 4 shows the FiPPI's internal structure in more detail. The slow channel consists of a decimator, a slow trapezoidal filter, and an output buffer. Standard filter peaking times range from 0.5 to 20 µs with flattops of 0 to 0.5 µs. The fast channel has a fast triangular filter (typically set at 300 ns peaking time) and detects pulses using threshold crossing. The times between successive pulses are counted, using the system clock, to determine if they are sufficiently isolated to not pile up in the slow channel filter. Good pulse amplitudes are captured by the output buffer at a sampling interval following their detection and these values sent to the DSP for further processing. The FiPPI processes new data values continuously, while the DSP only works with the captured values. This division of labor allows the DXP-4C to achieve output throughputs of 300,000 cps in a high density package at a low cost.

Note that all the FiPPI control parameters shown entering the FiPPI diagram in Figure 4 may be adjusted by the experimenter. In contrast to analog spectrometers, where typically only the slow filter parameters are adjustable, the DXP-4C allows all parameters to be optimized, including pileup inspection parameters. This allows efficient operation with x-rays over a very broad range of energies. Pulse pair resolution depends on settings, but is typically 200 ns at 5.9 keV.

DXP-4C: Spectrum and Energy Resolution

Figures 5 through 8 show the DXP-4C's performance characteristics. Figure 5 shows a complete total external reflection (TXRF) spectrum taken, using a Kevex detector, from a Si wafer contaminated with metals at the level of ppm of a monolayer. Even the Si and Cl peaks are well resolved. This is one example of a situation where full spectrum collection is essential to obtaining experimental success. Figure 6 shows energy resolution vs peaking time for 5.9 keV x-rays from Fe-55. These data were collected using a prototype DXP-4C with an Ortec 1 cm diameter HPGe detector at low count rates. More recent values typically fall 10 to 20 eV lower. In comparing Figure 6 to similar curves from other systems, it is important to recall the approximate factor of 2 between triangular pulse peaking times and semi-Gaussian pulse shaping times. I.e. a 1 µs shaping time produces about the same energy resolution and throughput as does a 2 µs peaking time. These results are comparable to those from good laboratory analog spectrometers. Also shown are values of the preamplifier's electronic noise, which are captured as part of the DXP-4C's baseline restoration operations and which act as a diagnostic for monitoring correct spectrometer operation without requiring a pulser input.

DXP-4C: Pileup Inspection and Throughput

Figure 7 shows the DXP-4C's superior pileup rejection capability, which results from the ability to adjust the pileup inspection parameters. These data were collected from a Cu target illuminated by synchrotron radiation, which is a pulsed source. As a result, many of the doubles counts are real, being pileups in the source itself. The background region between the singles and doubles is reduced by almost 4 orders of magnitude compared to the maximum rate. This is about 100 times better than can be obtained from the typical commercial analog amplifier under the same input count rate conditions.

Figure 8 compares throughput between the DXP-4C and a good analog amplifier/SCA combination, versus input count rate (ICR). The DXP's peaking time is 2 µs, twice that of the analog system's 1 µs, giving it about twice the effective count rate capability. The DXP also gives a solid ICR measurement which can be used to generate highly accurate deadtime corrections.

Figure 9 shows two reasons for the enhanced throughput. The first is that digital pulses (Fig. 9b) terminate more cleanly than analog pulses (Fig. 9a), allowing closer approach without pileup. The second is that SCAs require an inspection time, which adds to deadtime, to determine whether a given pulse falls between the LL and UL settings or is too large.

Automating Data Collection

As noted, all the DXP-4C control parameters are set through the CAMAC interface, allowing all data taking operations to be completely automated. Such operations will fall into three categories: a) system calibration and tuning, b) data taking, *per se*; and, c) data integrity assurance.

System Calibration and tuning

Because the DXP-4C delivers full spectra, the major difficulty associated with tuning a multi-element detector system - setting the SCA windows - is eliminated. The data of interest are stripped from the collected spectra by the user's software. Once operating parameters have been established for a particular XAS edge, returning to operation there is as simple as downloading a stored parameter file. Even determining operating parameters for a new situation can be automated by constructing a program to measure energy resolution and throughput versus filter parameters and then selecting the desired combination from the output. The DXP-4C, using its adjustable gain stages and DACs, is also capable of being programmed to measure and calibrate its internal gain as well as to adjusting itself to cause x-rays of a specified energy to fall into specified MCA bins.

Because all these operations are carried out by computer, they are no more difficult to execute for 13 or 19 detectors than for a single detector.

Data Taking

Data taking is straightforward. The module is commanded to accept counts for a fixed time interval or number of counts. It then returns the following: a spectrum, the total number of input x-rays detected, and its own measure of its actual livetime (from a FiPPI counter, see Fig. 4). This latter measures the actual time the DSP is available to collect data, excluding time spent during preamplifier resets or otherwise when data to the ADC are invalid. Combined with the input x-ray count, this produces an accurate ICR for use in correcting the number of output counts when operating at rates where deadtime is significant.

Data Integrity Assurance

Because the DXP-4C supplies a complete spectrum for each detector at each XAS energy, it offers significant advantages in assuring data integrity. Automated routines can be set up, for example, to inspect the data for the development of unexpected emission lines (such as might arise from hitting the sample holder with the input beam). Similarly, the output of each detector can be compared both to its own recent outputs and to the outputs of the other detectors in the array. This would allow detection both of malfunctioning elements (or DXPs) and other sources of data degradation, such as Bragg scattering into a detector. Further, as noted above, the DXP routinely reports its own electronic resolution, which can similarly be used to monitor data quality and spot aberrations.

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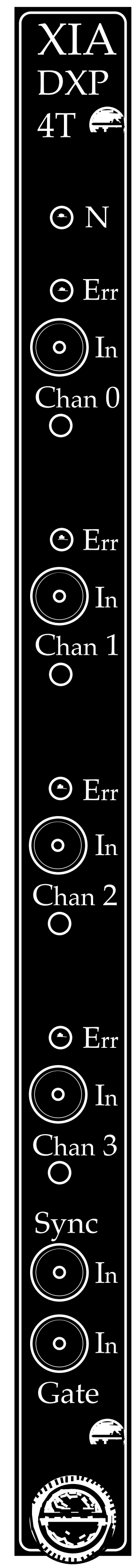

Figure 1: The XIA 4-channel DXP-4T X-ray Spectrometer

Table 2: Three approaches to instrumenting a 13 element X-ray detector system. The XAS SCA system has 13 spectroscopy amps and 26 SCA channels. The XAS MCA system uses commercial, computer controlled amplifiers multiplexed to 4 computer controlled MCAs.

Feature	DXP 4C	XAS SCA	XAS MCA
Cost	$28,900	$69,800	$67,200
Max Rate OCR (kcps) (for 0.5 µs Shaping)	1,600	662 (wide open SCAs)	400
Shaping Type (Values in µs)	Trapezoidal (0.5 to 20 in 60 steps)	Semi-Gaussian (0.5, 1, 2, 4, 8, 12)	Triangular or Semi-Gaussian (0.5, 1, 2, 4, 6, 12)
Resolution (1) FWHM (for X µs Shaping)	155 eV (4 µs Shaping) 250 eV (0.5 µs Shaping)	250 eV (0.5 µs Shaping)	200 eV (4 µs Shaping)
Data Captured	13 full spectra, (1 per channel)	13 SCA & 13 ICR values (1 each/ channel)	4 full spectra (grouped channels)
Pileup Inspection (resolving time)	Yes (<200 ns)	No	Yes (<500 ns)
Computer Interface			
Coarse Gain	Yes	No	Yes
Fine Gain	Yes	No	Yes
Shaping Time	Yes (Peaking & Gap)	No	No
Peak Dxetection Threshold	Yes	No (Nor Manual)	No (Nor Manual)
Pileup Rejection Criteria	Yes	No (Nor Manual)	No (Nor Manual)
Preamp Matching	Yes (Polarity, Offset)	No	Yes (Polarity Only)
Output Data Collected	Yes – Full Spectra and ICR	Yes – Counts and ICR Only	Yes – Full Spectra
Physical Volume	4 CAMAC Cards	1 Full Rack	1 Full Rack

(1) Resolutions reported from different detectors/preamplifier systems may not be strictly comparable since detector/preamp noise can be a major noise performance factor.

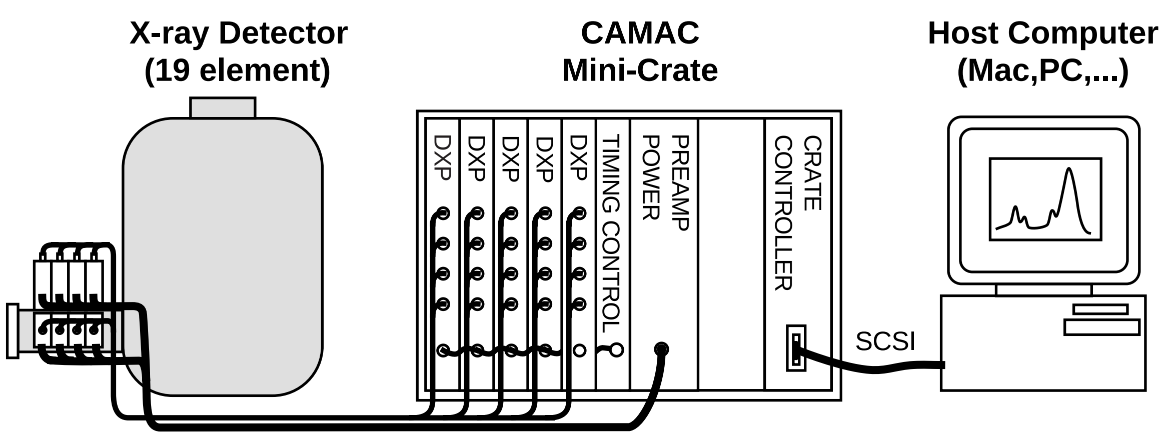

Figure 2: Multi-element X-ray Detector instrumented using DXP-4C spectrometer electronics.

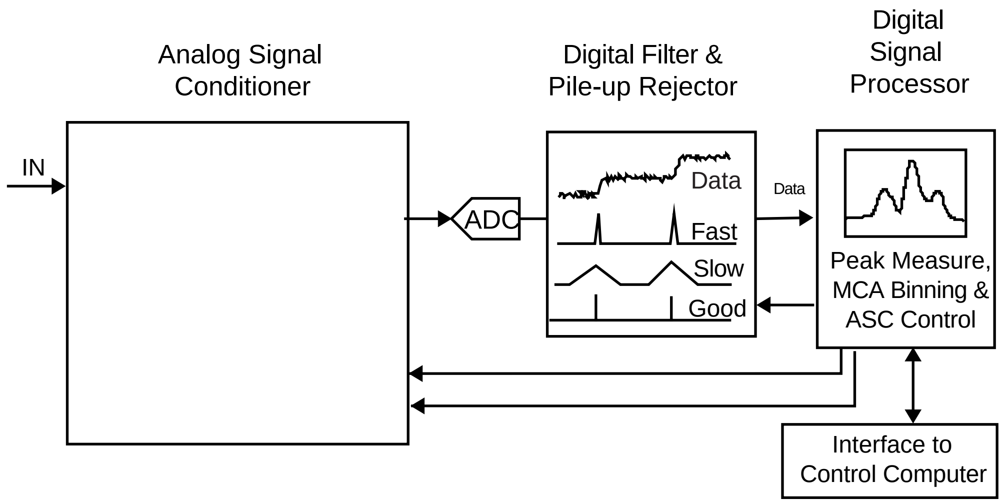

Figure 3: Block diagram of DXP-4C showing major functions

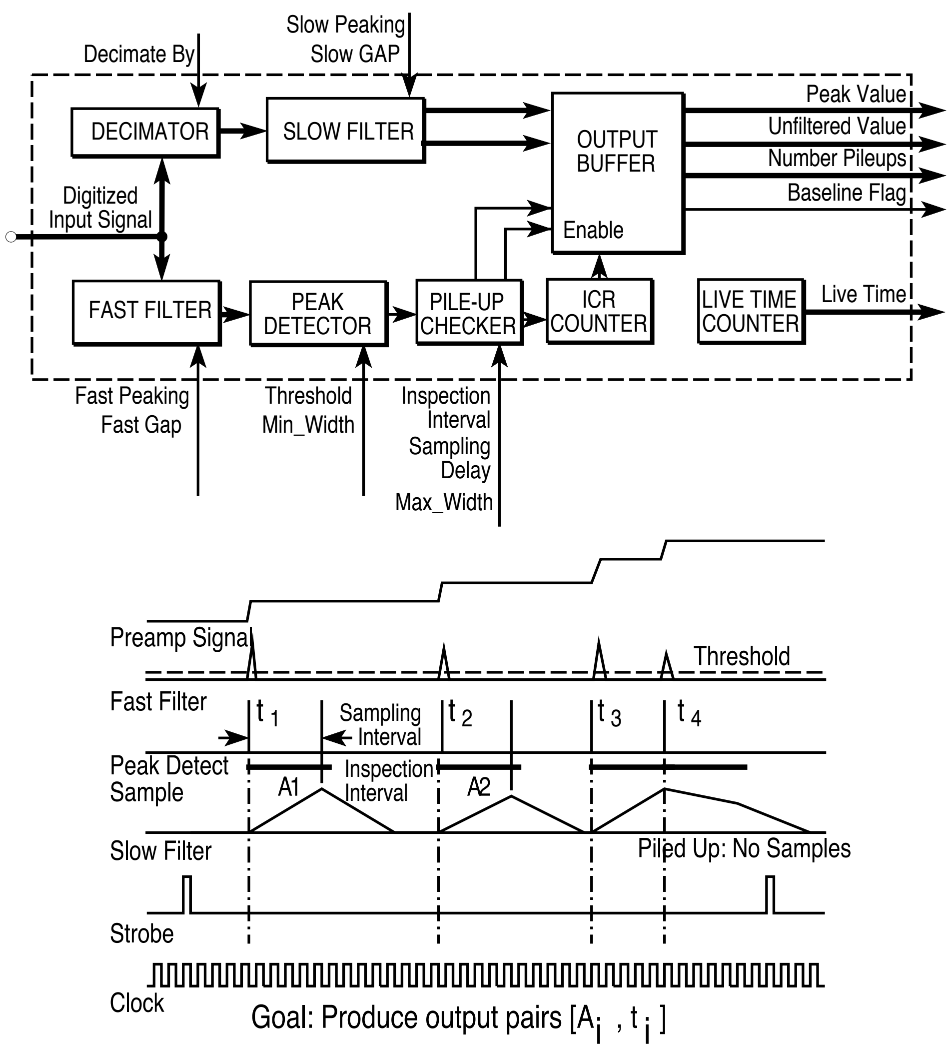

Figure 4: Structure of the DXP_4C's digital filter and pileup inspection block (FiPPI)

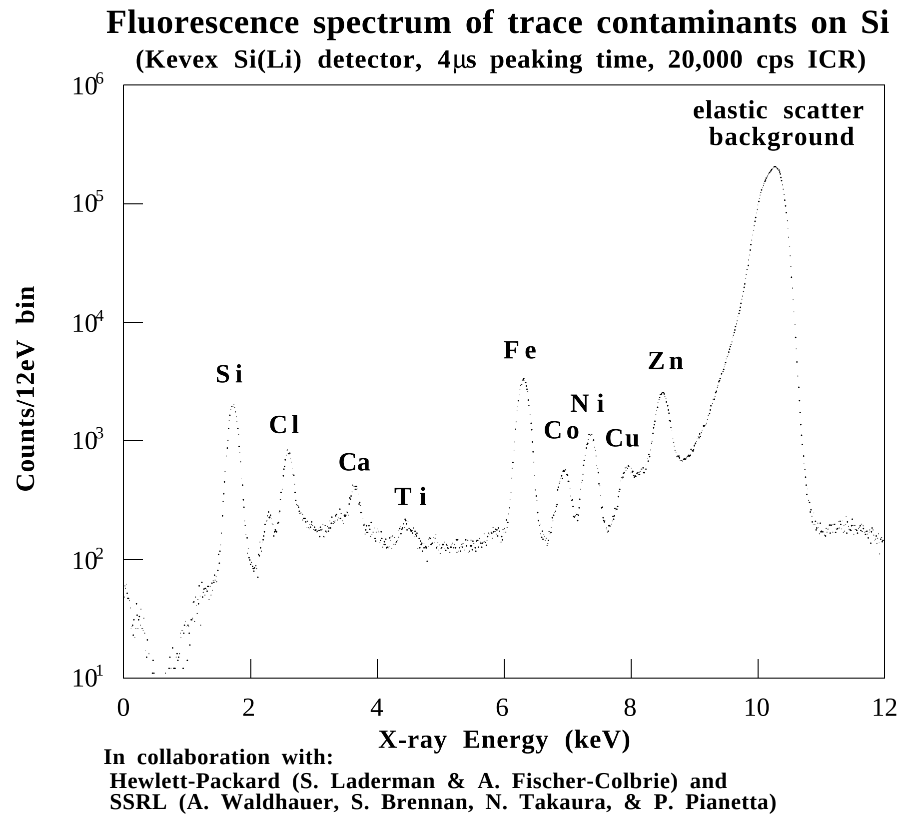

Figure 5: A TXRF spectrum taken using the DXP-4C

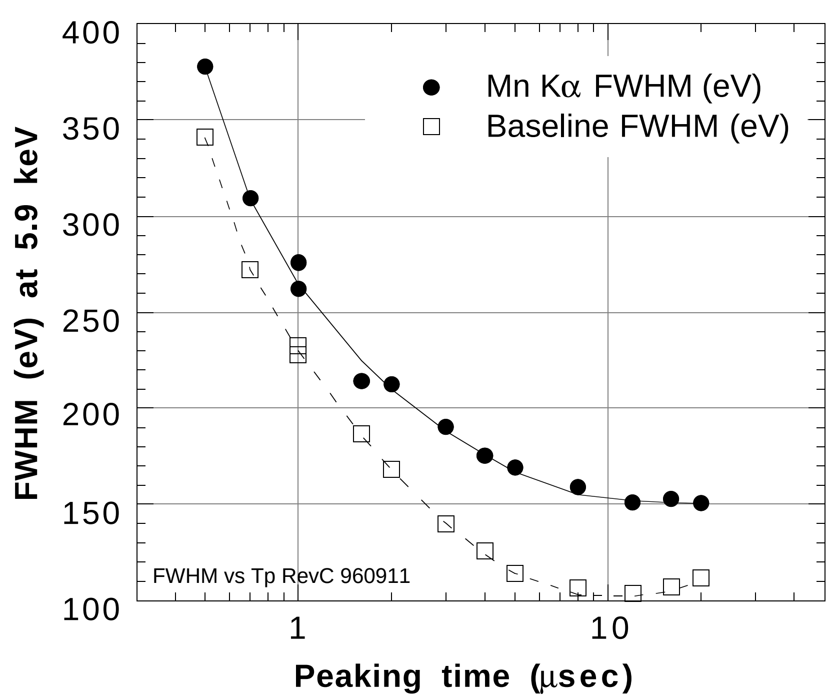

Figure 6: Energy resolution and electronic noise of DXP-4C versus peaking time

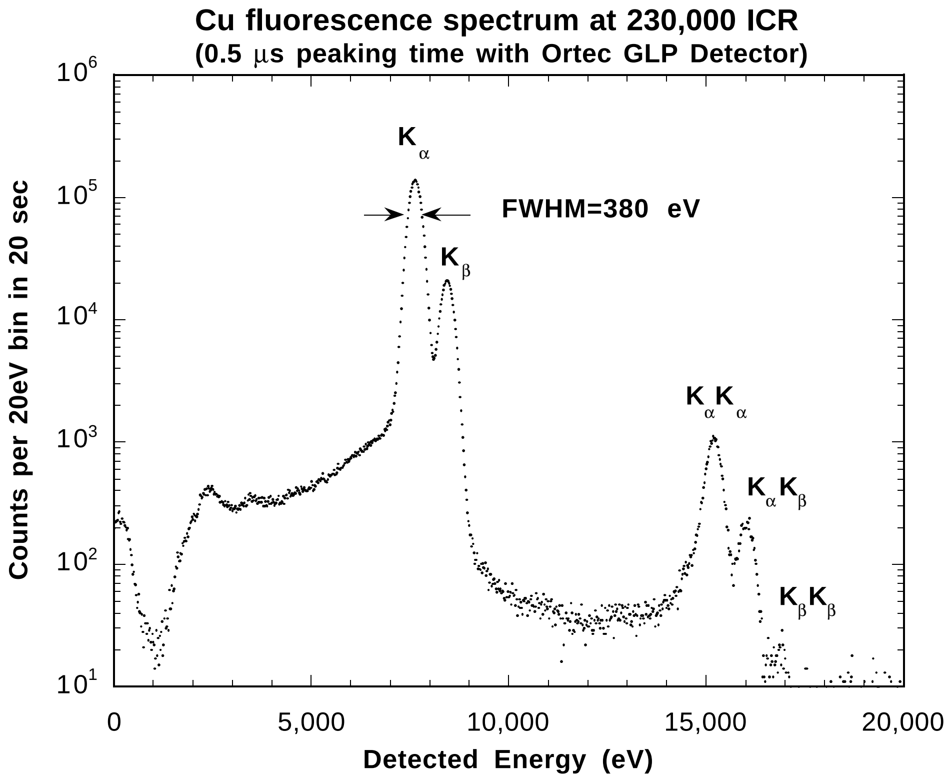

Figure 7: Pileup rejection demonstration using Cu radiation

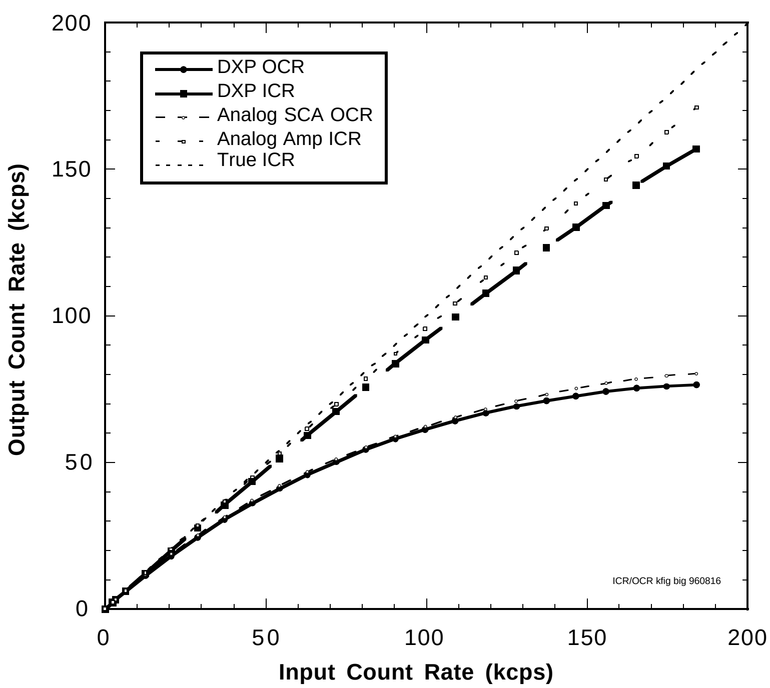

Figure 8: Throughput comparison between DXP-4C and common commercial analog amplifier plus SCA system

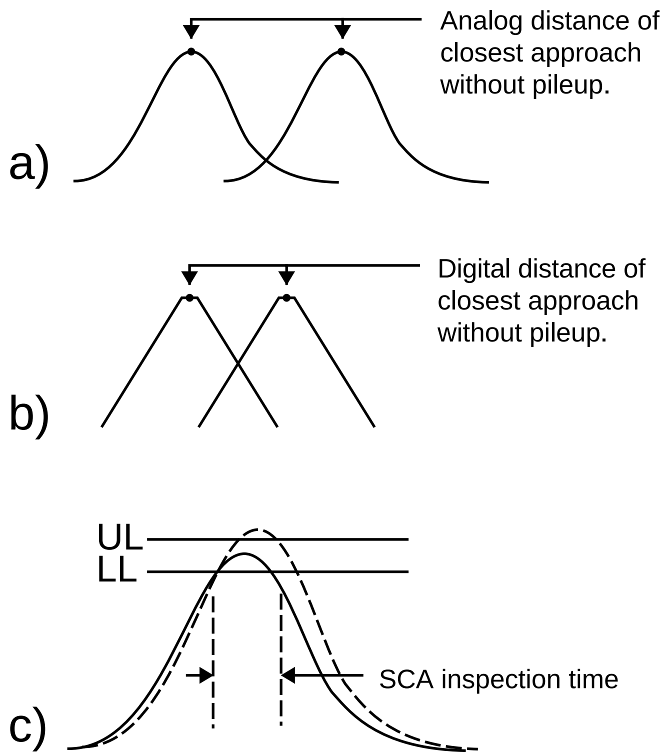

Figure 9: Sources for enhanced DXP-4C throughput: a, b) effect of pulse tails on pileup; c) SCA inspection time.