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XAS EXPERIMENTERS' MANUAL FOR THE 13-ELEMENT GERMANIUM DETECTOR ARRAYS

by

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XAS Experimenters' Manual for the 13-Element Germanium Detector Arrays

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1. Introduction

The detector system is used to measure the intensity of fluorescence photons of a selected energy from samples undergoing XAS analysis. It may appear somewhat daunting to the first-time user, but in practice it is not difficult to use provided that the instructions given in this text are followed carefully. Most of the setting up required will be done by in-house staff, and ***users should not make any adjustments to the system other than those described in section 9.*** Data can be seriously degraded under some circumstances if this instruction is disregarded. Anyone using the system for the first time is strongly advised to read through this text completely before attempting any setting up or operation of it.

The array consists of thirteen separate planar high-purity germanium crystals (referred to as detector elements in this text) in a single cryogenic housing and sharing a common HT bias supply.

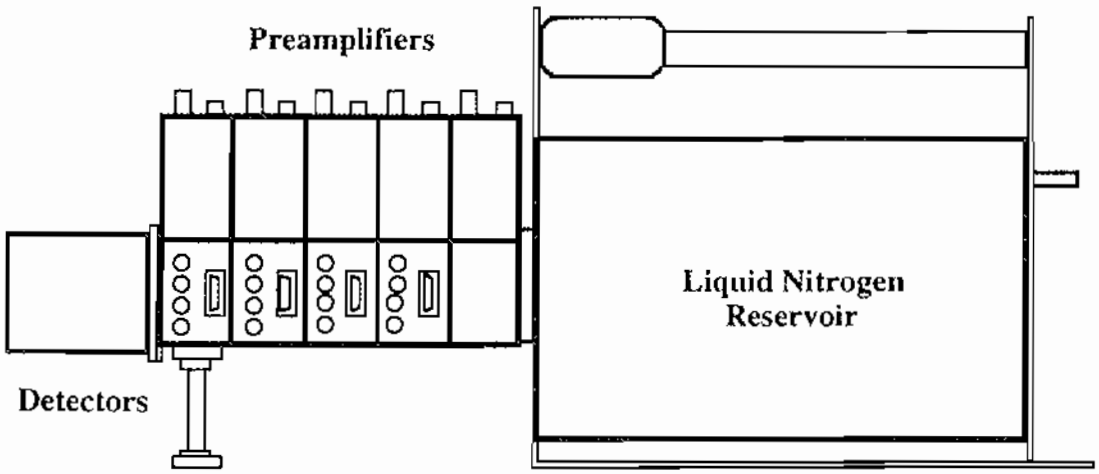


Figure 1. Side view of the detector array.

Each element is in itself a complete and separate detector, and operates independantly of all the others. Thirteen sets of signal-processing electronics are used with the array, one for each element. Each detector element along with its associated electronics and cabling is referred to as one 'channel' of the system. All channels of the system may be used for either data-aquisition or monitoring, or for both simultaneously. Users will be informed of the exact configuration of the system prior to first setting up.

The front face of the detector array is shown in figure 2 below. Both the relative position and channel number of each element are indicated.

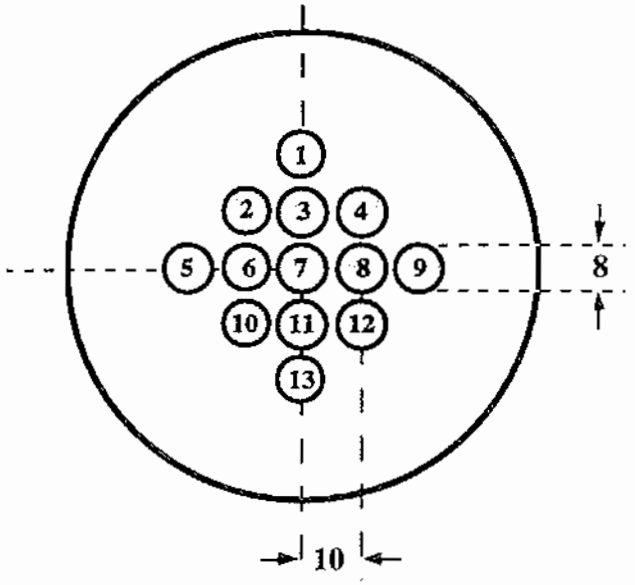


Figure 2. Front face of the array. Dimensions shown in mm.

When aligning with the monochromatic beam and the sample, the array should be treated as if it were a single detector. On stations 8.1 and 9.2 the array is mounted with its central axis horizontal at the level of the beam and at right angles to it in order to minimise Compton scatter. On station 9.3 the array is mounted vertically as part of the permanent REFLEXAFS set-up.

2. The Detector System - Principles of Operation.

This section is intended mainly as a brief introduction for the first-time user. It is not essential for setting up the detector system, and can be omitted if so desired.

Figure 3 shows a schematic of one channel of the detector system. The detector element itself consists of a single cylindrical crystal of high-purity germanium. Application of a high voltage to one face removes charge carriers from the volume of the crystal, producing a deep depletion region with a strong electric field across it. The detector is effectively a diode (of P-I-N structure) under reverse bias, the depletion region forming the sensitive volume of the detector. An X-ray photon of energy up to about 100keV which enters this region will liberate charge carriers (i.e. electron-hole pairs) by photo-electric absorption, the number of carriers produced being proportional to the energy of the absorbed photon. These are swept out of the depletion region by the electric field and collected at the electrodes on the faces of the crystal. The accumulated charge is integrated by a charge-sensitive pre-amplifier to produce an output voltage pulse of amplitude proportional to the photon

energy. This pulse amplitude will be of the order of a few millivolts only, and of very short duration. For any subsequent analysis to be accurate, the pulse must be amplified and lengthened substantially. A "shaping amplifier" both amplifies and integrates the pulse to produce a more manageable signal. The integration process provides low-pass filtering, removing high-frequency noise from the pulse, as well as lengthening it. By amplifying the pulses from the pre-amplifier, any differences in amplitude (caused by differences in photon energy) are likewise amplified, and thereby made easier to detect.

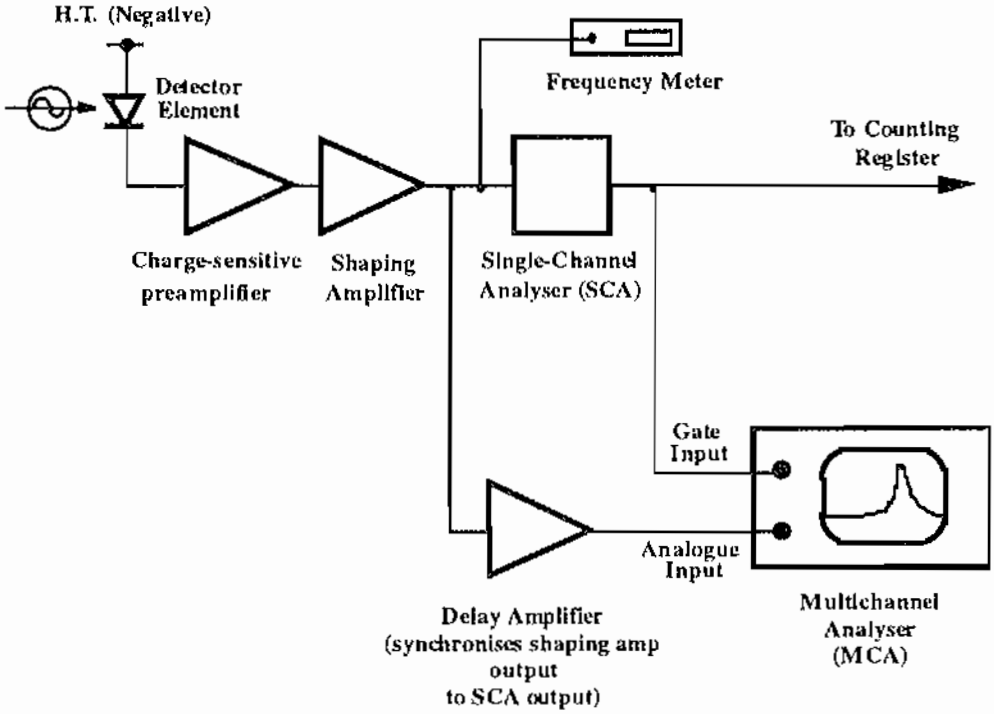


Figure 3. Schematic of one channel of the detector system, with monitoring.

The radiation entering the detector will consist of a mixture of fluorescence X-rays from the sample, plus a much higher flux of scatter from the incident monochromatic beam. The primary objective of a fluorescence XAS experiment is to measure the intensity of one particular type of fluorescence from the sample (Kα for 3d metals or Lα for 4d metals) as a function of incident beam energy. Therefore an important property of the detector system is the ability to ensure (as far as possible) that only pulses due to fluorescence photons are counted. Fluorescence photons are emitted at a single energy. Therefore, their corresponding pulse amplitudes will fall within a narrow range, allowing for some spreading due to statistics, scattering within the sample, etc. If the electronics can discriminate between different pulse amplitudes, all those which do not correspond to the energies of fluorescence photons can be rejected, and the detector system will then be effectively "tuned" to the relevant fluorescence energy. This discrimination is achieved by feeding the output from the shaping amplifier into a "single-channel

analyser" (SCA for short). Here the amplitude of the analogue signal is compared with two preset voltage levels. These correspond to upper and lower limits of the energy range of fluorescence photons, and therefore the energy range to which the counting system must respond. If the pulse amplitude lies between the two levels, the SCA produces an output pulse of fixed voltage and duration, compatible with the digital electronics of the counting register. Users who are familiar with electronics jargon will recognize the SCA as being essentially a window comparator, and the energy range defined by the upper and lower voltage levels is termed the "window" of the SCA.

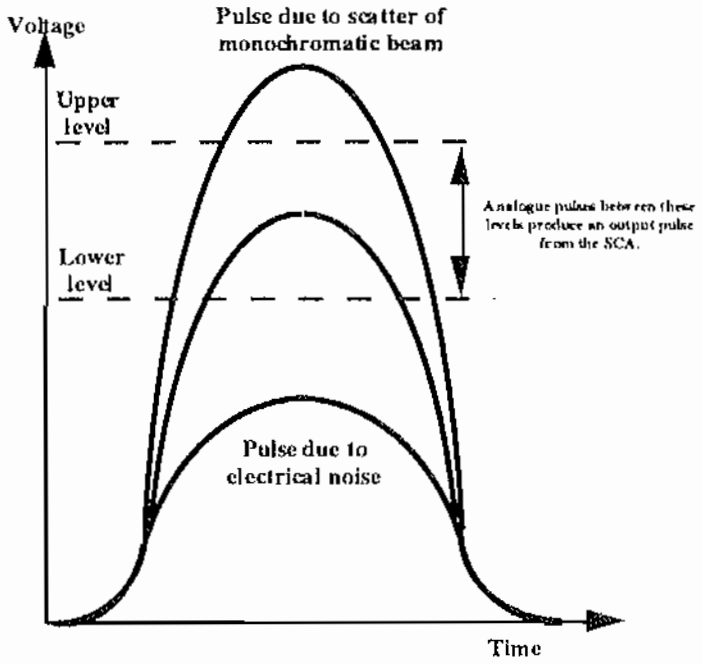


Figure 4. Definition of the SCA window.

From the SCA, the digital pulses are fed to a counting register which interfaces directly to, and is controlled by, the station computer. The XAS software allows the user to set counting-time limits in the scanning conditions, and to select which of the detector system channels are to be included in the data-aquisition.

The range of pulse amplitudes coming out of the shaping amplifier is monitored using a "multichannel analyser" (usually abbreviated to MCA). This can be thought of as a very large array of SCA's, with slightly different window settings on each, plus a memory to record the number of pulses within each window. The display produced is a histogram of the range of pulse amplitudes fed into it. If the output from a shaping amplifier were fed straight into the MCA, the display would correspond to the energy spectrum of X-rays entering the detector.

The MCA can also be set to trigger on an externally-applied digital signal. With this setting the MCA only responds to signals (i.e. amplifier output pulses) which appear at the MCA's analogue input simultaneously with a digital pulse at its 'gate'

input. By using the SCA output pulse to generate this gate pulse, while the output from the corresponding shaping amplifier is fed to the analogue input of the MCA, the energy spectrum shown by the MCA display will be due only to those photons whose energies are within the range defined by the SCA window. Thus the width and position of the SCA window are shown in terms of photon energy. Once the window is correctly matched with the fluorescence photons' energy range, the MCA display gives a visual indication of the fluorescence intensity.

The output pulses from the SCA are too short to trigger the MCA adequately, so are lengthened by a gate-and-delay generator. The finite response time of this unit results in the gate pulse being delayed with respect to its corresponding analogue pulse from the shaping amplifier. Both pulses must arrive at the inputs of the MCA simultaneously, otherwise the analogue amplitude will not be correctly registered. For this reason, the analogue pulse is passed through a delay-amplifier which, as its name implies, delays the analogue pulse sufficiently to compensate for the delay inflicted on the gate pulse. A frequency meter or counter is used to monitor the output count rate from the detector; this is connected to the analogue signal line between the shaping amplifier and the delay amplifier.

3. Detector System Linearity

The integration time-constant of a shaping amplifier imposes an upper limit to the rate at which input pulses can be processed to produce clear and distinct signals at its output. If the input rate exceeds this limit, the pulses effectively become overlapped at the output, a phenomenon known as 'pile-up'. The net result of this is that as the input count rate is increased, a point is reached at which the output rate no longer increases linearly with it. This effect becomes progressively worse as the input rate is further increased, and will eventually reach a point at which an increase in input rate will actually produce a decrease in the output rate.

For practical purposes, the count rate of each detector channel should normally not exceed 40,000 per second, or else linearity will be lost. If, however, the input-to-output rate characteristics of the system are known, and the true input count rates to the shaping amplifiers can be measured, it is possible to operate the system at much higher measured output count rates. Linearity is then recovered through the data-aquisition software. The station master will advise on how to do this, providing that the necessary electronic units are included in the detector system. When setting up the detector system, the count rate per channel should be kept below 40,000 per second or the MCA is likely to show a distorted spectrum.

4. The Detector System - Electronics

The complete signal-processing system is shown schematically in figure 5. The detector preamplifiers are mounted on the array itself (Figure 1). Power for the preamplifiers is taken from a NIM crate power supply through a fan-out module. The HT supply is in the form of a NIM module, and is housed in the same crate as the power fan-out for the preamplifiers. This crate is located inside the station

hutch. Likewise the shaping amplifiers are in NIM format and are located inside the hutch.

A 'signal router' is used to select which channel of the detector system is to be monitored, by switching the MCA and frequency meter between the different pairs of amplifier and SCA signals. This allows rapid checking of each detector output, and removes any necessity for the user to change over any cabling. The 'SCA controller' allows the windows of all the SCA's in the system to be raised or lowered in energy simultaneously.

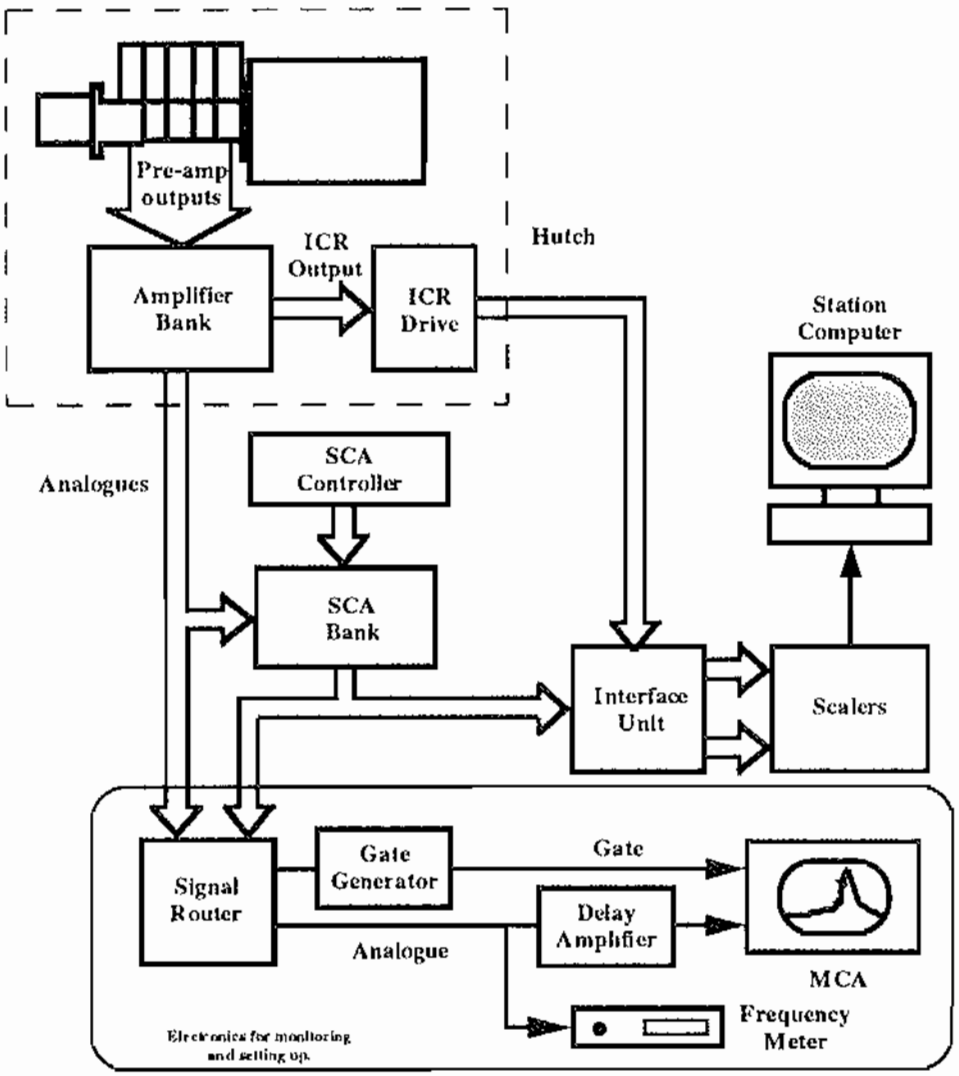


Figure 5. Schematic of the complete detector system.

The 'ICR drive' and 'Interface unit' (if present) are to allow the true input count rates (ICR = input count rate) of the shaping amplifiers to be measured independantly of the output count rates from the SCA's. This in turn allows the

system to be run at count rates that would normally exceed the limit imposed by pile-up (section 3). If the system does not include these units, the SCA outputs are fed straight to the scalers. The ICR drive will be located inside the hutch as close as possible to the shaping amplifiers.

The SCA's are in NIM format while the SCA controller and signal router are separate rack-mounting units. The count registers (and interface unit, if present) are in CAMAC format, and form the link with the station's data-aquisition computer. These will be located in one of the data-aquisition racks, outside the hutch. The MCA and frequency meter are stand-alone instruments, and are placed so as to be within easy reach of the user.

5. Safety Considerations.

1. The front face of the detector array consists of a beryllium foil, which isolates the vacuum inside the detector from the outside air. In order to maximise X-ray transmission this window is very thin, and consequently is extremely fragile. In normal use it is protected by an aluminium mask with a mylar window. **DO NOT REMOVE THIS MASK, AND DO NOT TOUCH THE BERYLLIUM WINDOW.** The station master will advise the appropriate action if the mask interferes with the mechanics of an experiment.

2. The HT bias to the detector array should not be adjusted, and must not be turned off except in an emergency. If the HT is found to be off, then seek assistance. **NEVER SWITCH THE BIAS ON AT FULL VOLTAGE.** The HT supply takes its input power from the DC supply rails of the NIM crate it is housed in. Should this power supply be faulty or be found switched off, do not attempt to bring it on again but wind the HT output control slowly down to zero, switch off the HT and seek assistance.

3. As mentioned earlier, the fan-out module supplying power to the preamplifiers is housed in the same crate as the HT supply. This is because power must be maintained to the pre-amplifiers at all times that the HT is on. **DO NOT SWITCH OFF THE POWER TO ANY NIM CRATE IN THE DETECTOR SYSTEM.** Likewise, the HT supply must not be removed from the crate that it is in, and must not be powered from any other crate supply.

4. The detector array may have its own built-in ion pump. If this is running when you are using the detector it will not affect its performance and should not be switched off, neither should the ion pump HT connector be removed if the pump is connected and running.

5. Some HT supplies are interlocked to the temperature of the detector elements. If this temperature rises above a preset level, due perhaps to loss of the dewar vacuum and subsequent loss of liquid nitrogen, the HT supply will automatically be turned off and an alarm will sound. If this happens, contact the station master as soon as possible, or bleep 359.

6. **DO NOT USE HELIUM GAS NEAR THE FRONT OF THE DETECTOR ARRAY**, as helium will diffuse through the beryllium window on the detector front face and cause a serious degradation of the vacuum inside the detector. If a sample cryostat is being used in conjunction with the detector array and condensation on the cryostat window is likely to be a problem, use a stream of nitrogen or dry air to keep it clear.

6. Setting Up - Outline

The only adjustment that the user is required to perform on the system electronics is to position the energy windows of the SCA's. This can be thought of as tuning the energy response of the system to suit the sample under analysis.

The gains of the shaping amplifiers will already be set up so that each channel produces analogue pulses of (as near as possible) identical size for the same X-ray energy. With the amplifier outputs thus matched, the required SCA window sizes will be similarly matched.

The main part of the setting-up procedure consists of using the signal router to display the signals from the central detector element on the MCA. The SCA controller is subsequently adjusted so that only signals due to sample fluorescence are displayed. The channel is then only sensitive to pulses arising from X-ray photons of the same energy as the fluorescence from the sample.

Users should not make any adjustments to the settings on any of the other units.

Small drifts in amplifier gains are possible, as are differences in resolution between the different elements of the detector itself. It is recommended that each channel's output should be inspected on the MCA before serious data aquisition is attempted. If it seems likely that other adjustments are required, then consult the station master or a member of the Solid-State Detectors Group.

7. The MCA

The MCA has three modes of operation, termed "coincidence" ,"anti-coincidence", and "straight-through". In coincidence mode the MCA is *triggered* by an externally applied digital signal, and only sees those analogue signals which arrive at the same time as the trigger. In anti-coincidence mode, the MCA is continually responsive to analogue signals *except* when inhibited by an external trigger. In straight-through mode all analogue signals are registered. When setting up the detector system, it will be necessary to change the MCA mode. This is done by means of a small toggle switch, located at the side of the MCA close to where the signal cables are connected. The switch positions are labelled to indicate which mode is selected.

7.1 The MCA Display

The appearance of a typical MCA screen layout is shown in figure 6. A lot of information is displayed, most of which the user does not need. Those which are important for setting up the detector system are indicated in the figure; the rest can be ignored. The main part of the display is the histogram of analogue pulse amplitudes from the shaping amplifiers. This is effectively a plot of photon energy (horizontal axis) against intensity (vertical axis) and is best thought of as such. Superimposed on this plot are a vertical "cursor" line and two other vertical lines either side of it. These are termed the cursor limits, and the area of the plot which they define can be displayed in expanded form to show greater detail as and when required. The cursor and its limits can be moved across the display to select any part of it, its position being shown in terms of "channel number" in the MCA memory. This represents a photon energy, and must not be confused with the term "channel" as applied to the detector system itself.The vertical scale of the plot is shown as logarithmic in the figure, but can be changed to a variable linear scale.

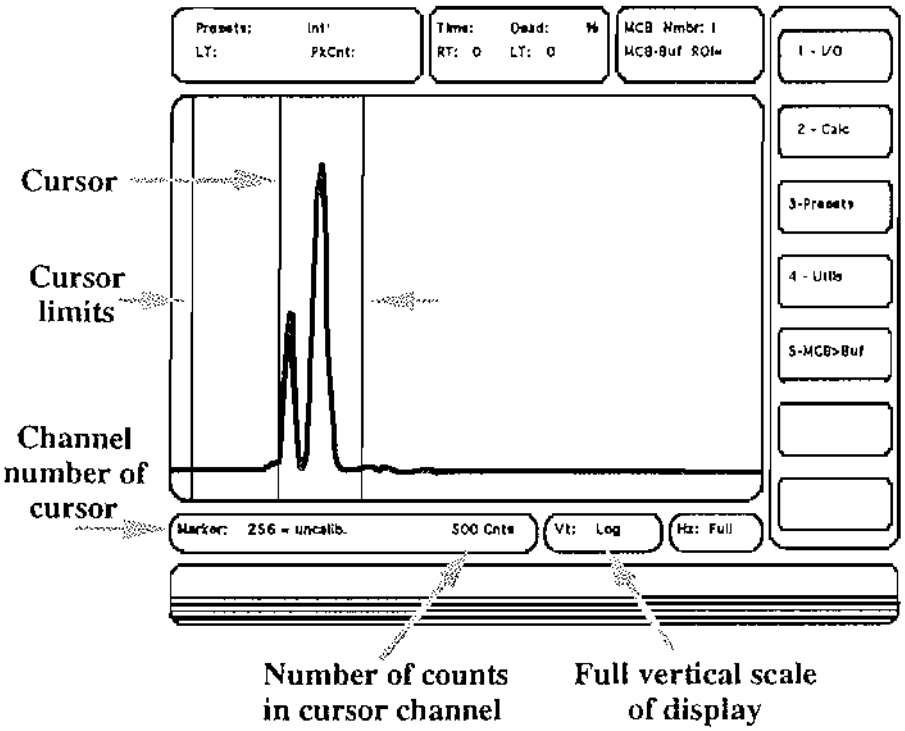


Figure 6. The MCA screen display.

7.2 The Control Keypad

The MCA is controlled by means of the membrane keypad located to the right of the display screen. Figure 7 overleaf shows the layout of the pad, with legends only on those keys which are required by the user. The other keys on the pad should not be used.

Data collection is controlled by the keys under the ADC label. Once the START key is pressed, the MCA begins accumulation, updating the display continuously, and continues until the STOP key is pressed. The CLEAR key erases the accumulated data and clears the display, and can be used to restart data collection,at any time without having to first stop collecting.

The white arrowed keys at the bottom of the pad, along with the blue full/expand key, are used purely to control the display. Again, these can be used at any time to change the display, without having to stop data collection beforehand.

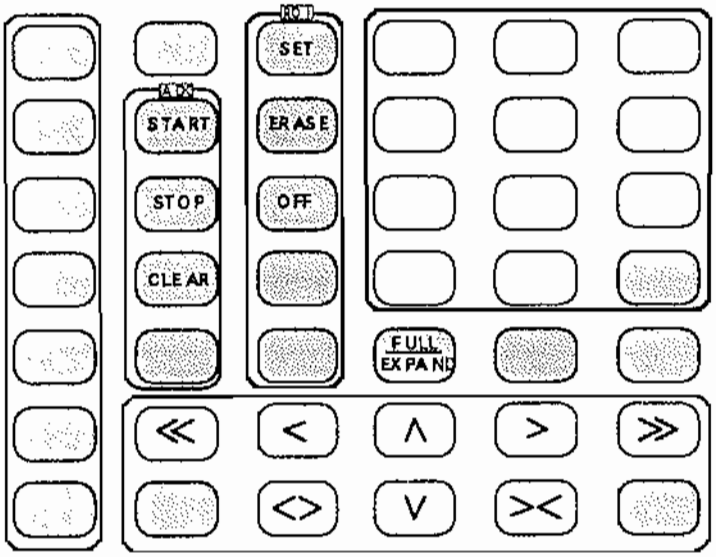


Figure 7. Layout of the MCA control keypad.

The keys labelled with horizontal single arrows, or dual arrows in the same direction (e.g. > or >>) are used to move the cursor and its limits across the display either to left or right (figure 8).

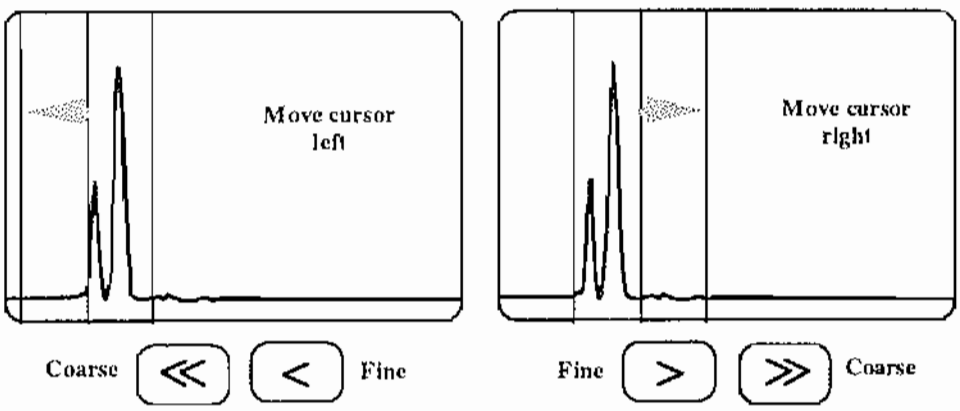


Figure 8. Moving the cursor and its limits across the MCA display.

The width of the region enclosed by the cursor limits is controlled by the keys labelled with dual arrows in opposing directions (figure 9).

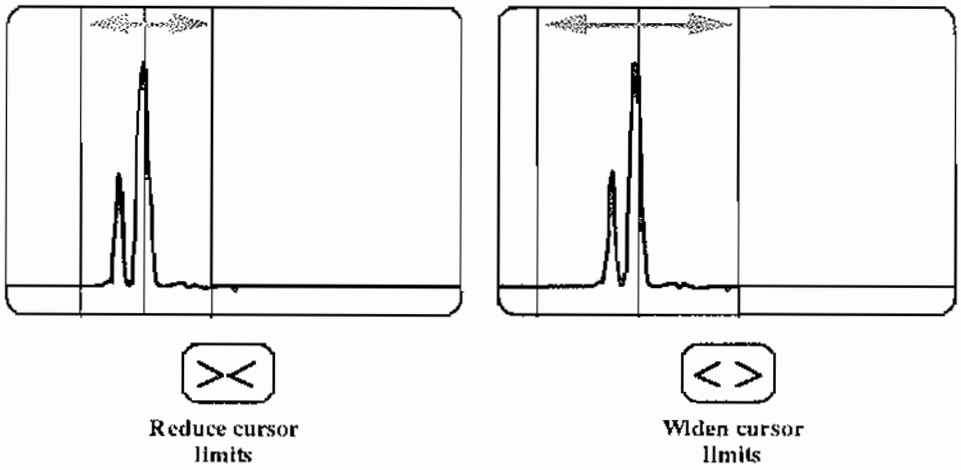


Figure 9. Changing the width of the cursor area.

The FULL/EXPAND key toggles the display between the complete pulse-height distribution and an expanded display of the region defined by the cursors (figure 10).

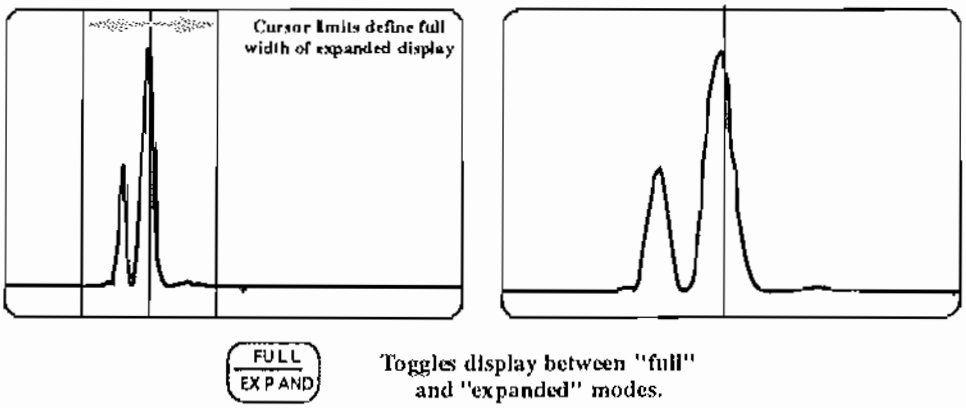
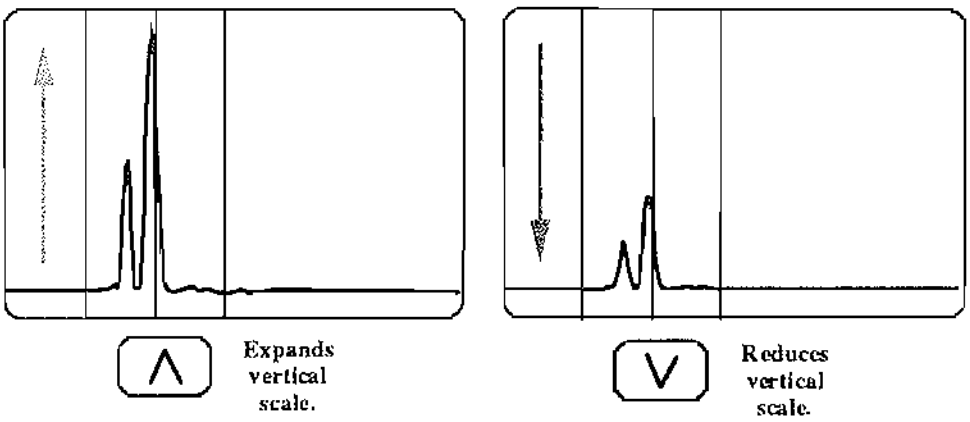


Figure 10. Expanding the display area bounded by the cursor limits.

The vertical-arrow keys control the vertical scaling of the display (figure 11).



Figiure 11. Changing the vertical scale of the display.

The maximum vertical scale possible on the display, obtained by holding down the 'up' arrow key, is logarithmic. Pressing the 'down' arrow key immediately reverts the display to a linear scaling.

The keys under the ROI label (ROI = Region Of Interest) allow one or more segments of the displayed pulse-height distribution to be highlighted for clarity, useful as a way of distinguishing the fluorescence peak from the rest of the distribution, or for identifying the region of the distribution where a fluorescence peak is expected.

To set an ROI on the display, proceed as follows:
1. Position the central cursor line at one end of the region to be highlighted (using the arrow keys) and press the SET key.
2. Move the cursor line to the other end of the region; the ROI will highlight as the cursor is moved. The single-arrow (fine control) keys must be used for setting the ROI. The double-arrow keys will still move the cursor, but the ROI will not set.
3. Once the required segment has been highlighted, press the OFF key. The cursor control keys will then revert to their normal function.

To erase an ROI from the display:
1. Position the cursor at one end of the ROI and press the ERASE key.
2. Move the cursor over the highlighted region (again, use the single-arrow keys for this) until the highlighting has disappeared.
3. Once the ROI is erased, press the OFF key.

8. Using the Signal Router and the SCA-controller

The signal router, also known as the multiplexer, connects the amplifier and SCA outputs from a selected detector channel to the MCA. A numbered LED on the front

panel shows which channel is selected at any one time. Selection is made using the two pushbutton controls on the front panel of the unit. Pressing the "clock" button shifts the selection along by one element or channel, and the "direction" button toggles the direction of the shift.

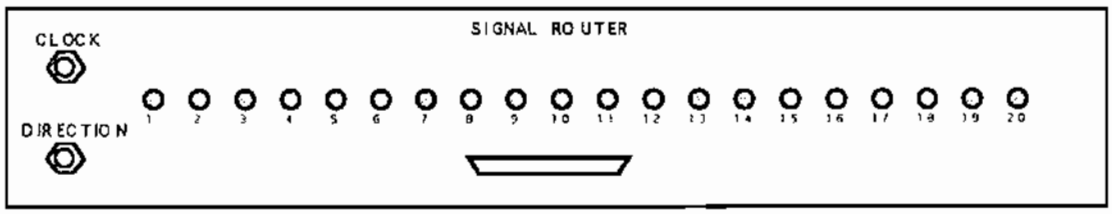


Figure 12. The front panel of the signal router.

The SCA controller allows the window settings of all detector channels to be raised or lowered simultaneously and by the same amount by sending a control voltage out to each SCA to set the lower limit of its energy "window". For each SCA the actual size of the energy window above this lower level is determined by the settings on its own front panel controls. These will have been already set up and should not be adjusted by users. The polarity switch on the front panel of the SCA controller will be set according to the type of SCA in use at the station, positive for Ortec 551 or negative for TCA 452. The mode-select switch should be set to "INT CONTROL".

9. Setting Energy Windows - Procedure

The instructions which follow assume as a starting point that the detector system has previously been operating correctly and that a move to a new fluorescence energy is the only change required. The sample should be in place and a stable monochromatic beam established at an energy somewhere within the new scan range. No changes in cabling are required. The procedure is as follows:

1. Using the signal router controls, select channel 7. This corresponds to the central element of the detector array.

2. Expose the sample to beam, and set the monochromator to an energy just above the sample's absorption edge. Adjust the beam intensity until the frequency meter shows a count rate in the range 10,000 to 30,000 per second. This is necessary to avoid pile-up in the amplifier itself. If the count rate is too high then the beam intensity must be reduced, either by reducing the monochromator entrance slit aperture or by increasing the amount of harmonic rejection used.

3. The MCA should already be switched on; a pulse height distribution may be showing, but not necessarily. If the MCA is off, seek assistance. Set the MCA to either "anticoincidence" or "straight-through" mode (using the switch on the side of the MCA) and wind the SCA controller to zero. Clear the existing display on the MCA, and start the data accumulation (i.e. press CLEAR followed by START).

4. Since the monochromator is positioned above the absorption edge of sample, the fluorescence and scatter counts should appear as distinct peaks in the pulse height distribution. Identify the fluorescence peak required. If in doubt, proceed as follows:

i. Set the monochromator at an energy below the absorption edge, clear the MCA display, and start data accumulation.

ii. Move the monochromator to an energy well above the absorption edge, while accumulating data on the MCA. By pressing the CLEAR button every few seconds while the monochromator is moving, it should be possible to see the flourescence peak appearing as the edge is traversed.

Check that the energy of the required peak is correct. Also check for the presence of any L peaks from other elements in the sample on the tails of the required fluorescence peak.

5. Clear any existing 'regions of interest' on the display, and set an ROI to cover the required fluorescence peak. The new window is now defined on the MCA display, although not yet established in the SCA.

6. Switch the MCA back to "coincidence" mode and wind the SCA-controller level until the fluorescence peak appears in the ROI. Set the controller level at a point where the fluorescence peak is straddled by the SCA window. If the width of the peak appears to be significantly different to that of the SCA window, then consult the station master.

7. Delete the ROI and, using the signal router controls, observe each operational channel on the MCA display to check that they are all counting in the windowed energy range. It is not necessary that each channel should show the fluorescence peak in exactly the same position on the MCA display, but they should not be spread out very much. If the spread appears wide, or if any channel does not show the peak within the SCA window, consult the station master.

8. Run a short trial scan, with short counting times and large step sizes, over the absorption edge and check that the edge contrast is satisfactory. Observe the count rates measured by each detector channel; the LIST ION command is the best way to do this. In particular, check the count rate where fluorescence intensity is greatest, usually just above the absorption edge. This should be done with the monochromator set at the harmonic rejection value intended for use in the proper scan.

For a given count rate, the signal to noise ratio of the data collected will be proportional to the square root of the collection time. Therefore, if the sample under investigation is particularly dilute, it is advisable to use the maximum count rate that the system linearity will permit (Section 3.). Also, the counting time per data point of the scan should be as long as is reasonably possible within experimental constraints.

10. Common Problems

Apart from outright equipment failure, most but not all cases of poor data quality can be attributed either to incorrect SCA window settings or to inadequate positioning of the detector with respect to the sample. No two experimental set-ups are exactly the same, however, and all will have their own subtle sources of potential trouble. In addition, a particular set of symptoms can be due to a number of causes. It is therefore impossible to specify the causes of all problems the user is likely to encounter. The symptoms, and their possible causes, discussed in this section may or may not be applicable to any one set-up. Possible remedies are offered as suggestions only, not as guaranteed solutions. Users should never attempt to fault -find on the detector itself or its electronics, but report any suspected failure of the system to the station master as soon as possible.

10.1 Low Count Rate

A low count rate on some or all all channels could be caused by a misalignment of the detector with the beam-to-sample axis. This should be checked. Also bear in mind the thickness of any attenuators between the sample and detector, such as the sample containment, mylar windows on cryostats etc.

If an unexpected drop in count rate occurs, without a similar loss of monochromatic beam intensity, then something may have fallen between the sample and the detector. If using liquid samples, check for possible leakage from the sample cell. If a sample cryostat is in use, then check that its windows are free of condensation, and that its vacuum has not failed.

Moving the detector close in to the sample will not only decrease the air attenuation of the fluorescence radiation, but will also increase the solid angle subtended by the detector at the sample, and hence the number of photons intercepted.

10.2 Little or No Absorption Edge Contrast

Essentially this is because the ratio of signal to noise in the data collected is low; in other words, there isn't much fluorescence on top of the background count rate. This can be caused by too much background getting into the energy window of one or more channels, effectively swamping the fluorescence. Alternatively, there may simply not be very much fluorescence getting through to the detector. In either case it is worth checking that the energy window is correctly set by repeating step 4 of the setting - up procedure.

Remember that if the window is incorrectly set on the scatter peak it may include the Kβ peak. This will show an absorption edge, but with very poor contrast.

A trial scan using a known sample of high concentration, usually a foil, is a useful check. If the resulting edge contrast is still poor, then the problem is likely to be with the detector or its setting-up. If not, then the sample itself or its containment may be the problem.

10.3 Noisy Data

This often accompanies poor edge contrast, and for the same reason, namely too much background relative to the fluorescence. Increasing the count rate or counting time per point may help reduce this, as indicated at the end of section 9.

If the noise appears in the form of spikes or dips on a spectrum which otherwise looks satisfactory, then some form of high frequency pickup may be present. Any extra electrical equipment brought in as part of the experimental set-up may be a source of noise, especially if it includes a switch-mode power supply. Contact the station master for assistance.

10.4 Distorted Background Shape

This appears to be due to unwanted fluorescence getting into the energy window of some or all of the detector channels. It arises from the scattered radiation falling on any metal of the same type as that of the sample and causing it to fluoresce. Not surprisingly this problem is most noticeable with samples containing iron, as most of the beamline hardware is made from stainless steel. Often the Io ion chamber is the culprit, and a slotted lead mask may be available to fit over the back face of it. Otherwise a few pieces of lead can be positioned to act as baffles and this usually eliminates the problem.