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INSTRUCTIONS TO EXAFS EXPERIMENTERS FOR USING THE 13-ELEMENT GERMANIUM DETECTOR ARRAY

by

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Instructions to EXAFS Experimenters for Using the 13-Element Germanium Detector Array

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1. Introduction

It is strongly recommended that experimenters using this detector array for the first time should read through sections 1 to 4 of this document before attempting to make use of the system. In particular, users should take note of the precautions necessary to avoid damage to the detector system. These are listed in section 3 under the heading 'Warnings'. If in doubt about anything concerning the detector system, then a member of the solid-state detectors group or the appropriate station master should be consulted. The complete detector system as described in this document is subject to continuing development, and new operating instructions will be produced as necessary.

The detector array consists of thirteen separate planar high-purity germanium elements in a single cryogenic housing and sharing a common HT bias supply. Twelve of the elements are used for collecting spectra and one for monitoring. Thirteen sets of signal-processing electronics are used with the array, and each detector element along with its associated electronics is referred to as a 'channel' in this text.

The detector system may appear somewhat daunting to the first-time user, but in practice it is quite simple 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 5. Data can be seriously degraded under certain circumstances if this instruction is disregarded. On the station, the electronics modules and their interconnecting cables have been labelled with appropriate channel numbers to aid identification, and care must be taken not to mix up any of these when using the system. Figure 1 shows the face of the detector array as viewed directly from the front, and the channel number of each element is indicated. Throughout this text, all references to channel numbers are always taken from this numbering convention. At the time of writing, the usual practice is to use channels one to twelve for data-aquisition and channel thirteen for monitoring. This may not always be the case, and it is possible to use one channel for both purposes if necessary. Users will be informed of the exact configuration of the system as appropriate.

2. The Counting System

Figure 2 shows a schematic of the whole detector system, and should be referred to when reading through this section. A check-list of control settings for the various electronic units involved is given at the end of this document.

The counting chain for each of the data-aquisition channels consists of the following: A pre-amplifier (mounted on the detector itself) and pulse-shaping amplifier (Ortec 570 ,571 or 572) produce an output pulse whose voltage is proportional to the energy of the detected X-ray photon. A single-channel analyser, hereafter abbreviated to SCA, senses the size (in terms of voltage) of the analogue pulse from the amplifier. If the pulse size lies between two preset limits the SCA produces a short output pulse of fixed voltage. Two such outputs are available from the SCA, one of positive polarity and one negative. Pulses from the negative polarity output are fed to one input of a 12-channel count register (LeCroy 2551), also termed a scaler, to be read by the station computer. The voltage limits in the SCA effectively define an energy bandpass or 'window' to which that channel of the counting system will respond.

In the monitor channel, the analogue pulses from the shaping amplifier are fed into a multi-channel analyser, or MCA, which displays a pulse-height distribution of the analogue signals. The MCA in use at the present time is a PC-based system, with a separate analogue-to-digital converter, or ADC (Canberra 8077) as its 'front-end'. The output pulses from the SCA are used as 'gate' pulses to trigger the ADC, which thus accepts only those analogue signals which fall within the energy window of the SCA. The pulses from the SCA are too short to trigger the ADC adequately, so are lengthened by the (Ortec 416A) gate-and-delay generator. This has two inputs available, one for positive polarity and one for negative. It is important that whichever input is used, it is connected to the SCA output of the same polarity. If one or more elements of the detector itself are not operative, then any one of the other channels may be used for both monitoring and counting. This is achieved by simply using the positive output from its SCA to generate the MCA gate pulses, whilst using the negative output for counting as usual. The very process by which the gate pulse is produced results in it being delayed behind the analogue pulse, which must be similarly delayed if it is to be synchronous with the gate pulse at the inputs to the ADC, otherwise the ADC will not trigger on the correct analogue pulse. At present this delay in the analogue line is achieved by means of a long length of cable; it is intended to replace this in the near future with a delay amplifier. Likewise it is intended to replace the PC-based MCA with a stand-alone MCA unit which should be both quicker and easier to operate than the present arrangement. A frequency meter or counter is used to monitor the output count rate from the detector; this may be connected at the output of the shaping amplifier (as shown in the figure) or at the input of either the SCA or the ADC (analogue input). Also, an oscilloscope may be used to monitor the size and shaping of the output pulses from the amplifier. Again, this may be connected at the either amplifier output, the SCA input, or the ADC analogue input.

3. WARNINGS

1. The beryllium window on the front face of the detector array 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. 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. ABOVE ALL, NEVER SWITCH THE BIAS ON AT FULL VOLTAGE. The Ortec 459 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. The power to the detector pre-amplifiers is taken directly from a NIM crate power supply through a fan-out module 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 is kept under vacuum by 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. 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.

4. Setting Up - Outline

The only adjustments that the user is required to perform are those of setting the limits of the energy window of each SCA. This consists of using the MCA to examine the analogue signals from each detector channel in turn, adjusting the SCA window 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 K-alpha fluorescence from the sample.

Users should not make any adjustments to amplifier gain settings. These will have already been set by the in-house staff such that each channel should produce analogue pulses of (as near as possible) identical size for the same X-ray energy. With the amplifier outputs thus matched, the required SCA settings should be similarly matched, although small drifts between amplifier gains are possible, as are differences in resolution between the different elements of the detector itself. It should not be taken for granted that once the SCA settings for one channel have been determined then the settings for all the other channels will be identical. Each channel must be checked separately, and the matching of the amplifier outputs is intended only to simplify this. The shaping times of the amplifiers will have been set to suit the energy of the fluorescence X-rays concerned. The present practice is to use 3 microseconds of shaping for fluorescence X-rays of energies below 10keV, and 6 microseconds for energies of 10keV and above.

Each SCA can be operated in one of three modes, selected by a switch on the front panel of the module. Only two of these are relevant for this application, namely the 'normal' and 'window' modes, labelled as "NOR" and "WIN" respectively on the module. Users should be aware of the distinction between the two, as the mode determines the manner in which the level controls operate. In the 'normal' mode the lower and upper level controls operate independantly over a range of 0 to 10 Volts, and separately define the lower and upper limits of the energy bandpass. In the 'window' mode, the lower level control defines the lower energy limit as before, while the upper level control defines the size of the energy bandpass (i.e. the size of the window) over a range of 0 to 1 Volt measured from the value set by the lower level control (which operates from 0 to 10 Volts as in 'normal' mode). In other words, the upper level now sets the size of the window and the lower level sets its position (in terms of energy). Either operating mode is valid, and the choice is left to the user. It must be remembered, though, that the upper levels take on different values if the mode is changed. It is not valid to set the levels in 'normal' mode and then operate the SCA in 'window' mode, or vice versa.

5. 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. IMPORTANT: The count rate displayed on the frequency meter should not be allowed to exceed 13,000 per second for a 3 microsecond shaping time on the amplifier, or 8,000 per second for 6 microseconds of shaping. This is necessary to avoid too much 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.

The only changes in cabling required will be at the SCA and the gate-and-delay generator. As mentioned in section 2, each SCA has two outputs, one of positive polarity and one negative. Users should be aware that the inputs of the 12-channel scaler will only respond to pulses of negative polarity; once the SCA has been set up, ensure that the correct (i.e. negative) output is connected to the scaler. The procedure is as follows:

1. Identify the channel currently configured as the monitor, i.e. the channel connected to the frequency meter and ADC. Trace the cables back to the SCA.

2. The SCA has three adjustment controls on its front panel, namely upper and lower levels, and delay. The delay control must not be moved. The mode-select switch may be found in either the NORmal or WINdow position. Make a note of the upper and lower levels, as it may be necessary to return to these values for checking. If the SCA is found to be in NORmal mode, and if the new fluorescence energy is higher than the old one, then leave the lower level control where it is and increase the upper level to its maximum setting. If the move is to a lower energy, leave the upper level where it is and decrease the lower level to its minimum setting. If the SCA is found in WINdow mode, first switch to NORmal mode and set the upper level about 2 Volts above the lower level. Then proceed as before. The purpose of this step is to open the energy window so as to easily encompass the new fluorescence energy.

3. The IBM PC should already be running the MCA program, called MCA.EXE, and will probably be showing a pulse height distribution. Ensure that the vertical scaling is set to 'AUTOSCALE' and 'EXPANSION ON' is active (these will be found under the 'DISPLAY' header). Clear the display either by using the menu or by typing
<CTRL><E> (both keys together, no carriage return).
If the program is not running, seek assistance.

4. The monochromator should now be positioned above the absorption edge of sample, such that the fluorescence and scatter counts appear as distinct peaks in the pulse height distribution. The mouse of the PC can be used to move the expansion box of the display to the new area. Identify the fluorescence peak required. If in doubt, adjust the SCA levels to widen the energy window further. Also, if necessary, move the monochromator over a short range, say 30 millidegrees or so, while aquiring data on the MCA; the scatter peak will move but the fluorescence peaks will not. Check that the energy is correct for the fluorescence and check for the presence of any L peaks from other elements in the sample on the tails of the K-alpha peak of interest.

5. Using the MCA menu clear the existing 'regions of interest' on the display (this is abbreviated to ROI in the menu and may be found under the 'DISPLAY' header). With the mouse move the MCA cursor to a point on the pulse height distribution corresponding to the lowest energy of the new window required, and 'click' the mouse at that point. Hold down the Shift key on the PC keyboard and slowly move the cursor to the point corresponding to the highest energy of the new window. The energy range thus selected will change colour on the display; release the Shift key when satisfied. If a retry is required, clear the ROI and start again. The new window is now defined on the MCA display, although not yet established in the SCA.

6. If the monitor channel is also being used for counting, the ADC trigger will will be taken from the 'POS' output of the SCA, with the 'NEG' output connected into the LeCroy 2551 scaler. For windowing purposes, disconnect the trigger lead from the 'POS' input of the gate-and-delay module and reconnect it to the 'NEG' input. Likewise, change over the connection at the SCA from the 'POS' output to the 'NEG' output. The cable which was already in the 'NEG' output (which is normally the output lead to the scaler) should be left to one side for the present. If the monitor channel is not included in the counting system, the ADC trigger lead will already be connected between the 'NEG' output of its SCA and the 'NEG' input of the gate-and-delay module. Clear the MCA display with <CTRL><E> and check that the fluorescence peak is still within the ROI. If not, clear the ROI and redefine it accordingly.

7. The energy window of the SCA must now be closed up so as to encompass only the fluorescence peak. The SCA may be left in NORmal mode for this, or switched to WINdow mode, either way the procedure is the same. First increase the LOWER level control in stages, clearing the MCA display after each adjustment, until no counts are registered below the lowest energy of the ROI on the display. When this is so, lock the level control to prevent any accidental readjustment. Now decrease the UPPER level control, again clearing the MCA display after each adjustment, until no counts occur above the highest energy of the ROI, and lock the control as before. Once the SCA levels have been set, the operating mode of the SCA must not be changed, otherwise the values at which the levels are set will change. The monitor channel is now set up. Disconnect the delay cable from the SCA input. Also remove the trigger lead from the 'NEG' output of the SCA and, if this channel is used for counting, reconnect the output lead to the scaler.

8. Select the next channel for setting up. Connect the delay cable to the T-piece on the input of the newly selected SCA in place of the 50-Ohm terminator. Disconnect the output lead from the 'NEG' output of the the SCA and plug in the trigger lead.

9. Switch the SCA to NORmal mode if it is not in this mode already, and open up the energy window as described in step 2. Clear the MCA display and observe where the fluorescence peak now occurs. It should lie more or less within the ROI as defined in step 5. If necessary change the ROI to encompass the fluorescence peak. Either remain in NORmal mode or switch to WINdow mode, and close up the energy window around the fluorescence peak as described in step 7. Once the window is satisfactory, lock the level controls as before. Disconnect the delay cable and trigger lead, and reconnect the 50-Ohm terminator and output lead.

10. Repeat steps 8 and 9 for each remaining SCA in turn until all channels are set up. Reconnect the delay cable to the SCA of the monitor channel, and reconnect its output and trigger leads in their original configuration.

After setting up, run a short trial scan and check the height of the absorption edge above the pre-edge background. If this is not satisfactory, then connect up each SCA in turn to the setting-up configuration, but without switching the SCA mode from WINdow trim the upper and lower level controls to reduce the scatter counts, particularly on the high-energy side of the fluorescence peak. Remember: Although the amplifier gains have been set to match the responses of the detector channels as closely as possible, some drift or variation may be expected with time. Hence it is not safe to assume that a peak appearing in the same place on the MCA display as that from another detector channel is the same peak. The full setting-up procedure should therefore be carried out for each detector channel. If this is not done there is a risk of setting the SCA window on the scatter peak. This may include the K-beta fluorescence peak so that a FLEXAFS spectrum can still be obtained, albeit with a very poor signal-to-noise ratio. This may in turn lead to time being wasted in checking sample alignment etc. when the real problem lies elsewhere.

Control Settings for 13-Element Detector Electronics

NOTE: EXCEPT IN A DIRE EMERGENCY, THE NIM CRATE POWER SUPPLIES MUST NOT BE SWITCHED OFF AT ANY TIME.

Power distribution to the detector pre-amplifiers is provided by a NIM crate power supply through an EC 586 fan-out module. Do not disconnect any power lead from this. Also, it is most important that the HT power supply and pre-amplifier power fanout should be housed in the same crate; do not remove the HT power supply from its crate or power it from any other supply.

Ortec 459 HT Power Supply :

-450 Volts from 0 - 500V output on rear panel of module.
THIS SHOULD NOT BE ADJUSTED.

Ortec 571 (or 572) Shaping Amplifier :

Gain : This will already be set, and SHOULD NOT BE ADJUSTED.
Shaping Time : 3 microseconds (X-ray energies below 10keV)
6 microseconds (X-ray energies of 10keV and above).
BLR switch : To centre position (PZ adj.). NB the pole-zero will already be set, and SHOULD NOT BE ADJUSTED.
Delay switch : To 'out'.
Pos/Neg switch : To 'pos'.

Input : From pre-amplifier (Canberra 2008) mounted on the detector itself.
Output : Unipolar ('Uni' socket) to Ortec 551 SCA input.

Ortec 551 Single-Channel Analyser (SCA) :

Upper level : Empirically determined by energy range of experiment.
Lower level : As for upper level.
Delay : minimum.
Delay range switch : 0.1 - 1.1 microseconds.
Int/Nor/Win switch : To "Nor" or "Win".

Input : Positive polarity pulses from Ortec 571/572 amplifier. Terminated with 50 Ohms for channels used in counting. Branching to ADC (via delay line) for monitor, or for other channels when setting up.
Output : Negative polarity, fast NIM logic, to LeCroy 2551 scaler for counting channels. Also to Ortec 416A gate-and-delay generator for monitor channel if this is not included among the counting channels.
Positive polarity to gate-and-delay generator for monitor channel if this is also used for counting.

Ortec 416A Gate-and-Delay Generator :

Delay : Minimum.
Delay range switch : 1.1 microseconds.
Amplitude : Just past mid-way position.
Width : Just past mid-way position.

Input : Fast NIM pulses from Ortec 551 SCA, using input of the same polarity as the SCA output.
Output : Positive polarity rectangular pulses (from positive delayed-output socket) to gate input of ADC.

Canberra 8077 Analogue-to-Digital Convertor (ADC) :

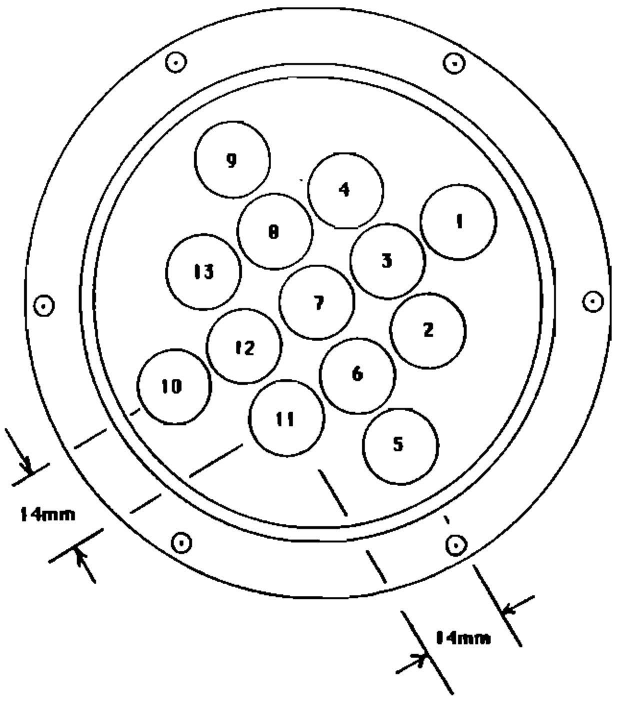
Range : 4k.
Gain : 8k.
Offsets : none (all six switches in downward position).
LLD : 0.3
Peak detect/Delayed switch : To 'delayed'.
PHA/SVA switch : To 'SVA'.
Coinc/Anti switch : To 'Coinc'.

ADC Input : Positive polarity analogue pulses from Ortec 571/572 amplifier via 200ns delay line, terminated with 50 Ohms.
Gate Input : Positive polarity rectangular pulses from Ortec 416A gate-and-delay generator.
Output : To IBM PC through ribbon cable from rear panel.

Oscilloscope Settings (if used to display analogue pulses):

Timebase : 5 microseconds/division.
Deflection : 1 Volt/division.
Triggering : Positive-going.

N.B. The analogue signal cable from the amplifier must have a 50-Ohm termination if the oscilloscope does not have a 50-Ohm input.



Detector Area

Total Area Subtended = **36.5%**

Figure 1. Front face of 13-element SSD.

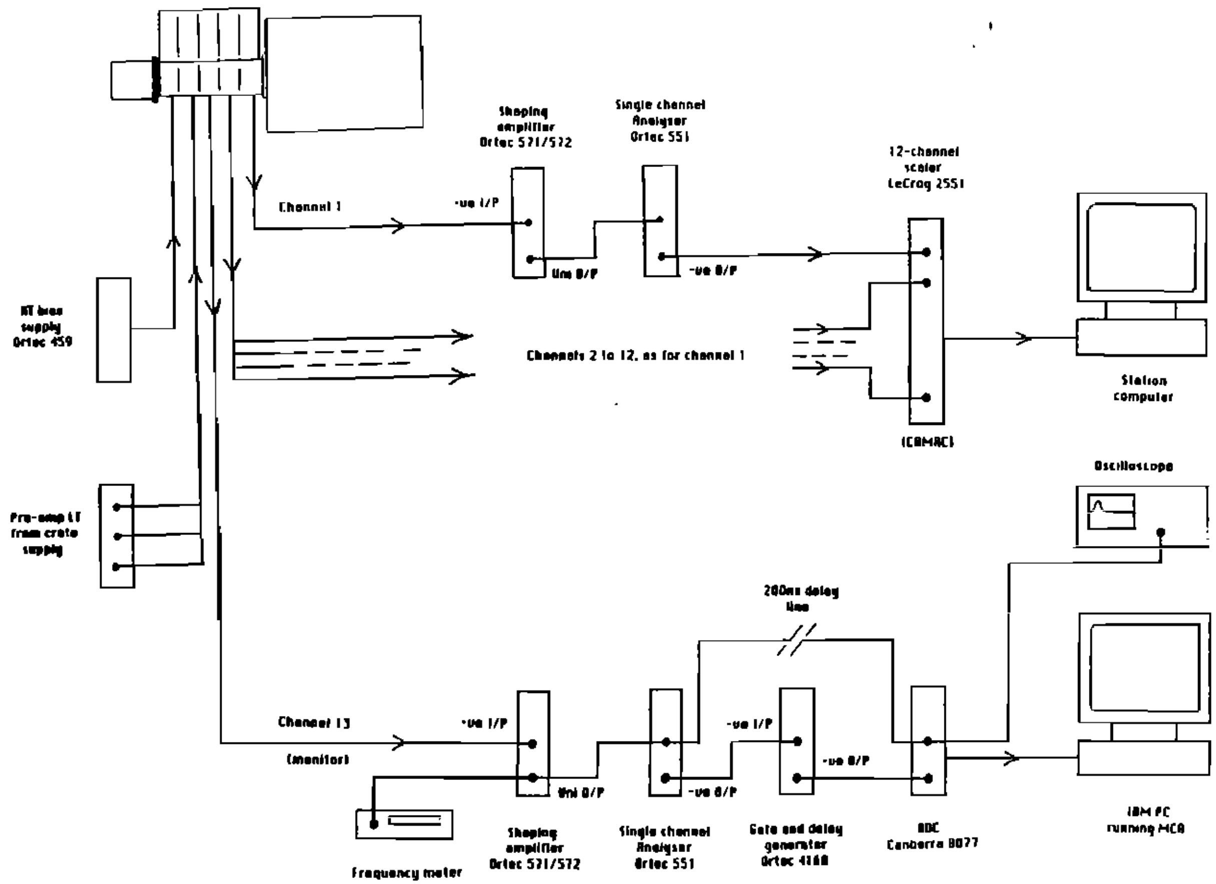


Figure 2. Interconnections for 13-element SSD.