ORTEC®

AN34 Experiment 8

High-Resolution X-Ray Spectroscopy

Equipment Need from ORTEC

- 4001A/4002D NIM Bin and Power Supply
- SLP-06175-P/CFG-PV4/DWR30 Si(Li) X-Ray Detector System. Includes Vertical Dipstick Cryostat, 30-liter LN_2 Dewar, Preamplifier, HV Filter, and 12-ft. Cable Pack. Typical specifications: 6 mm diameter; 175 eV resolution at 5.9 keV, and 1-mil thick Beryllium entrance window.
- 659 0–5 kV Detector Bias Supply
- TRUMP-PCI-2K PC Plug-In MCA
- 672 Spectroscopy Amplifier
- 480 Pulser
- C-24-12 Cable (2 ea.)
- C-29 BNC Tee Connector

Other Equipment Needed

- PC operating Windows 98/2000/XP
- Oscilloscope
- ~1 µCi each of ^{65}Zn, ^{137}Cs, ^{55}Fe, and ^{57}Co (see Table 8.2 for other possibilities)
- Sealed X-Ray Sources (Disk Type) 1–5 µCi, ^{57}Co, ^{55}Fe, ^{65}Zn (substitute alternate sources with similar energies)
- 12 ea. Aluminum Foil, 2 x 2 in. x 5 mg/cm^2
- 12 ea. Nickel Foil, 2 x 2 in. x 5 mg/cm^2
- Optional: chamber for x-ray fluorescence measurements with a Si(Li) detector; 8-in. diameter x 3-in deep. Designed to fit onto an upright Si(Li) detector. Four each selectable sample positions under vacuum.

Purpose

X-Ray energies in a range below 35 keV will be measured with a Si(Li) X-Ray Detector System, and x-ray spectra will be obtained for different samples.

Introduction

In Experiment 7, it was indicated that high-resolution gamma spectroscopy is a rewarding research area. High-resoution x-ray spectroscopy is an equally challenging research field. The nuclear spectroscopy centers suggested in Experiment 7 can be duplicated for x-ray spectroscopy. While both Si(Li) and High Purity Germanium (HPGe) systems can be used for x-ray spectroscopy, this experiment is written for the use of a Si(Li) system. HPGe detectors are also available for this discipline.

Fig. 8.1 shows the spectral response of the 22.162 keV K_α line from silver (Ag) as it is measured with three different types of detectors: a NaI(Tl) detector, a proportional counter, and a Si(Li) x-ray detector system. The amplifier gains were carefully matched so that the width of each peak would be a true indication of the relative resolution for that type of detector. Note that the high resolution of the Si(Li) detector not only defines the $K_{\alpha 1}$ peak to advantage, but also provides a definite valley below the adjacent $K_{\beta 1}$ peak. Silicon systems have been developed that will produce a resolution of 148 eV and better on the 5.9 keV line from ^{55}Fe. With the resolution capabilities of these systems it is possible to study the K_α and K_β fluorescence x-rays

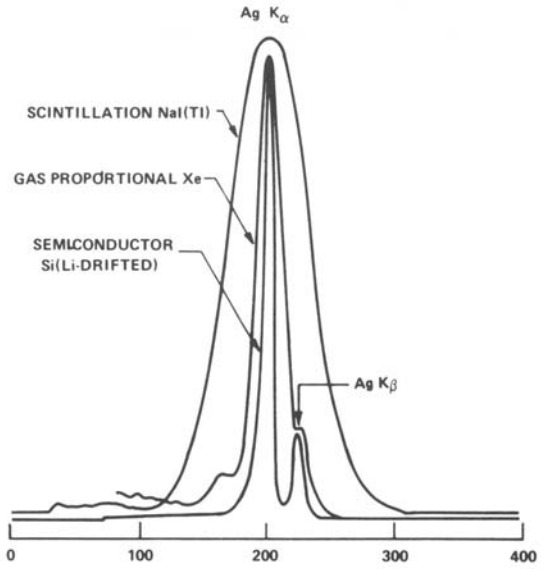

Fig. 8.1. Demonstration of the Resolution Capability of the Three Types of X-Ray Detectors for the Silver K Spectra Obtained from ^{109}Cd Source.
(Courtesy Philip G. Burkhalter and William J. Campbell, U.S. Bureau of Mines, College Park, MD.

for most elements above ^{16}O. The subject of x-ray fluorescence is treated in Experiment 12.

In general, Si(Li) systems provide somewhat better resolution than can be obtained with HPGe systems; however, the efficiencies of HPGe systems are better at higher x-ray energies (>30 keV).

Most Si(Li) x-ray detector systems are equipped with a beryllium window over the detector element. The x-ray proportional counters used in Experiment 11 also have beryllium windows. Fig. 8.2 shows typical photopeak counting efficiency as a function of the x-ray wave length (reciprocal of energy) and the effects of a 2-mil and a 5-mil thick beryllium window on the efficiency. From the figure it can be seen that the window

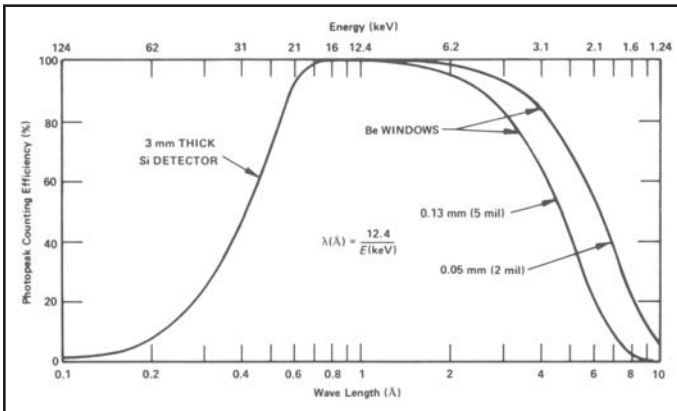

Fig. 8.2. Spectral Response of Si Detector with Be Windows.

becomes important for energies below 6 keV, with comparable wave lengths >1Å. The curve drops off at higher energies (>15 keV) because the 3-mm thick Si(Li) detector starts to become more transparent to the x-rays.

EXPERIMENT 8.1
Energy Calibration with a Pulser

Procedure

1. Install the ORTEC 659, 480, and 672 in the 4001A/4002D NIM Bin and Power Supply and interconnect the modules, the detector preamplifier, and the TRUMP MCA as shown in Fig. 8.3 (the preamplifier is mounted on the detector). Check the detector data sheet for the correct HV bias polarity and level, and select the correct polarity on the 659 while its bias amplitude controls are set at zero. The oscilloscope will be used to check the waveform at the output of the 672 Amplifier.

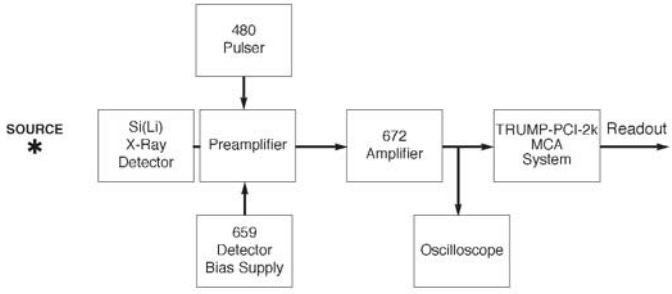

Fig. 8.3. Equipment Interconnections for Experiment 8.1.

2. Raise the bias voltage output of the 659 to the recommended value.

3. Place a ^{137}Cs source ~2 cm from the detector window.

4. Adjust the gain of the 672 Amplifier so that the 32.1 keV K_α line for ^{137}Cs falls in the upper portion of the analyzer.

5. Accumulate a spectrum with at least 2000 counts in the peak of the 32.1 keV K_α line.

6. Determine the channel number at the centroid location of the 32.1 keV peak and call this channel C_0.

7. Turn on the 480 Pulser and set the Pulse-Height dial at 321/1000. Adjust the Attenuation switches and the Calibrate control to place the pulser peak in the same channel (C_0) as the ^{137}Cs x-ray peak. The pulser is now calibrated so that full scale (1000/1000) is equal to 100 keV. Lock the dial on the pulser.

8. Clear the analyzer and accumulate pulser peaks for the values in Table 8.1. Store at least 1000 counts in each peak channel. Record the corresponding channel numbers in Table 8.1.

Table 8.1

Pulser (Pulse Height)	Equivalent Energy (keV)	MCA Channel Number
300/1000	30	
250/1000	25	
200/1000	20	
150/1000	15	
100/1000	10	
50/1000	5	

EXERCISE

From the data in Table 8.1, plot an energy vs. channel number calibration curve. Determine the keV/channel and the resolution of both the pulser and the 32.1 keV line from the ^{137}Cs source. Compare these resolutions with those which the instructor has recorded for the detector being used.

9. Obtain an unknown x-ray source from the laboratory instructor and accumulate a spectrum for a period of time long enough to determine the channel numbers for each pronounced peak in the spectrum.

For example, Fig. 8.4 shows the K_α and K_β peaks for an ^{55}Fe x-ray source. This source, which is listed in Table 8.2 decays by electron capture. The daughter nucleus for the decay is ^{55}Mn, and this accounts for the Mn K_α at 5.9 keV and the Mn K_β at 6.49 keV. If this had been one of the unknowns, there would be no question as to the daughter nucleus. Fig. 8.5 shows an ^{241}Am spectrum with the Np L x-rays and the single 26.36 keV gamma line. As in the case for ^{55}Fe, the parent nucleus ^{241}Am could be easily identified. These isotope decay schemes are shown in ref. 10.

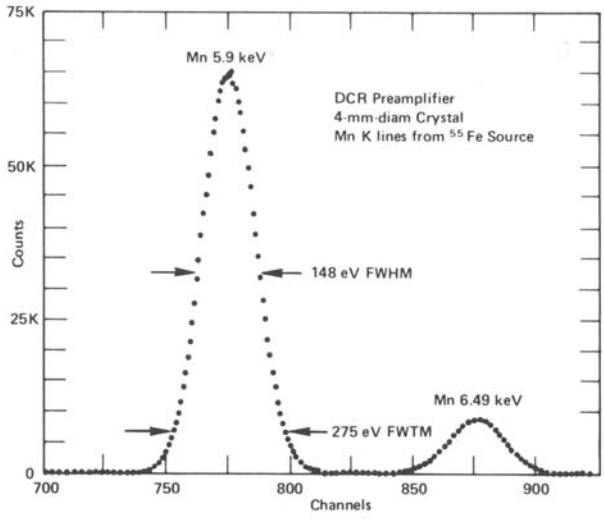

Fig. 8.4. K_α and K_β X-Ray Lines from an ^{55}Fe Source.

Table 8.2. Recommended Calibration Sources for Si(Li) Detectors.
(Taken from ref. 7) (Calibration sources for x-ray studies should be deposited on 0.25-mil. Mylar or be electrodeposited.)

Nuclide	Energy of X-Rays and Low-Energy Gamma (keV)	Energy of High-Energy Gamma (keV)	Intensity Ratio *X/γ*
^{54}Mn	5.414 (K_α) 5.946 (K_β)	834.8	0.2514 (±0.5%) $K_\alpha + K_\beta$
^{57}Co	6.40 (K_α) 7.06 (K_β) 14.41 (γ)	122.1	0.5727 (±2.0%) 0.7861 (±2.9%) 0.112 (±1.8%)
^{65}Zn	8.04 (K_α) 8.9 (K_β)	1115.5	0.6596 (±0.8%) 0.0911 (±2.0%)
^{85}Sr	13.38 (K_α) 15.0 (K_β)	514.0	0.5020 (±0.65%) 0.0880 (±1.4%)
^{88}Y	14.12 (K_α) 15.85 (K_β)	898.0	0.5491 (±1.2%) 0.0989 (±1.9%)
^{109}Cd	22.10 (K_α) 25.0 (K_β)	88.0	22.02 (±4.9%) 4.68 (±5.0%)
^{113}Sn	24.14 (K_α) 27.4 (K_β)	391.7	1.219 (±3.5%) 0.267 (±3.6%)
^{137}Cs	32.1 (K_α) 36.6 (K_β)	661.6	0.0666 (±3.0%) 0.0159 (±3.1%)
^{139}Ce	33.29 (K_α) 38.0 (K_β)	165.9	0.808 (±11%) 0.195 (±11%)
^{198}Au	70.15 (K_α) 80.7 (K_β)	411.8	0.0229 (±2.3%) 0.00635 (±2.4%)
^{203}Hg	72.11 (K_α) 83.0 (K_β)	279.2	0.1247 (±2.1%) 0.0348 (±2.3%)

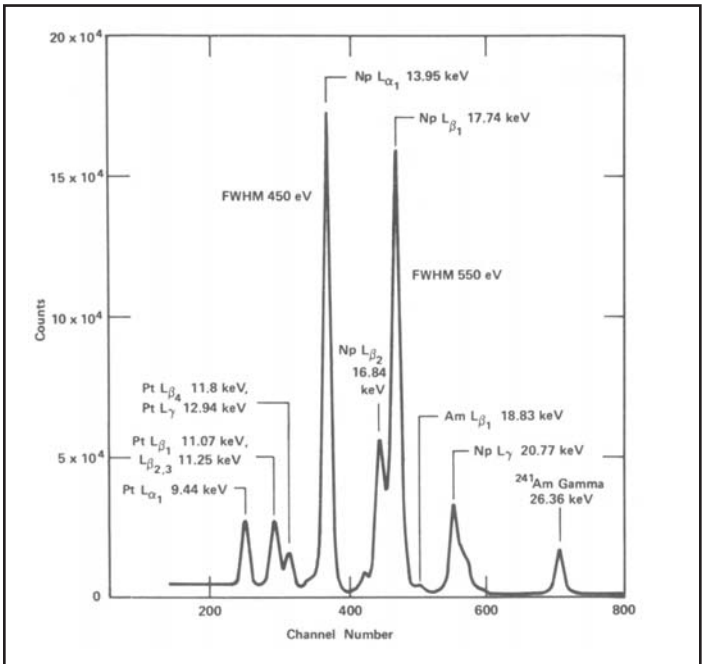

Fig. 8.5. Typical ^{241}Am Spectrum Taken with Si(Li) X-Ray Detector.

EXPERIMENT 8.2
Efficiency Measurements and Energy Calibration with Standard X-Ray Sources

This experiment will be similar to Experiment 8.1 except that standard x-ray sources of known activity will be used to measure the efficiency of the detector while the energy calibration curve is being determined. The x-ray sources used for all of these experiments should be sources that are manufactured specifically for x-ray studies. For x-ray sources, most manufacturers deposit a spot of the radioactive material onto a 2.5 x 10^{-4} in. thick (0.25-mil) Mylar foil. The back side of the source is then covered with another piece of Mylar of approximately the same thickness. The spot size is usually maintained at ~1 mm. The sources used for this experiment are standard sources whose activities are known to ±5%. Thin electrodeposited sources can also be used for x-ray studies.

Procedure

1. Set up the electronics as shown in Fig. 8.3 and adjust the various parameters exactly as described in Experiment 8.1.

2. Place the ^{137}Cs standard activity source at a distance of exactly 2 cm from the detector entrance window (for this experiment, all sources must be placed at exactly the same distance from the detector).

3. Set the horizontal display on the MCA to 1024 channels full scale, and adjust the gain of the 672 Amplifier so that the 32.1 keV K_α line is in the upper portion of the 1024 range. Accumulate ~6000 counts in the K_α peak, read out the MCA, and record the live time. Since the efficiency of the detector is being determined, the live time must be recorded for each part of this experiment.

4. Replace the ^{137}Cs standard with the ^{109}Cd standard and accumulate for good statistics. Read out the MCA and record the live time.

5. Repeat for as many standard x-ray sources as you wish to use for the calibration and efficiency measurements (see Table 8.2 for suggested sources).

EXERCISES

a. From the MCA readouts and the recorded live times, determine the centroids and the measured number of x-rays/second for each of the lines used in Table 8.2. Record the information in Table 8.3.

b. Plot a curve of energy vs. channel number for the data that have been recorded in Table 8.3. Determine the slope of the calibration curve in eV/channel. For each peak, multiply the slope of the calibration curve by the FWHM (in channels) of each peak; from this, determine the resolution of each line. Record these values in Table 8.3 and plot a curve of resolution vs. energy for the data in Table 8.3. Why does the resolution appear to get better at lower x-ray energies? Recall from Experiment 3 that for gamma-ray measurements with NaI(Tl) detectors, the resolution got considerably worse at the lower gamma energies.

Table 8.3. X-Ray Efficiency and Calibration Data						
Isotope	**X-Ray Energy (keV)**	**Channel Number**	**X-Rays/s Measured**	**X-Rays/s Theory**	***E* (Efficiency)**	**Resolution of Peak**
^{54}Mn	5.414 K_α					
	5.946 K_β					
^{57}Co	6.40 K_α					
	7.06 K_β					
	14.41 γ					
^{65}Zn	8.04 K_α					
	8.90 K_β					
^{85}Sr	13.38 K_α					
	15.00 K_β					
^{88}Y	14.12 K_α					
	15.85 K_β					
^{109}Cd	22.10 K_α					
	25.00 K_β					
^{113}Sn	24.14 K_α					
	27.40 K_β					
^{137}Cs	32.1 K_α					
	36.6 K_β					
^{139}Ce	33.29 K_α					
	38.0 K_β					
^{198}Au	70.15 K_α					
	80.7 K_β					
^{203}Hg	72.11 K_α					
	83.0 K_β					

c. Determine the theoretical number of x-rays for each line by multiplying the activity of the source in gammas/second by the x-ray/gamma intensity ratio shown in Table 8.2. Remember the intensity of the gamma source should be determined at the calibration gamma energy shown in Table 8.2. From these data, determine the efficiency of the detector for each line and record in Table 8.3. The efficiency is defined as the ratio of the measured x-ray intensity to that which would theoretically be possible for the source. Plot the efficiency vs. energy curve for the detector. Fig. 8.2 shows the shape of this efficiency curve for the various Be window thicknesses.

EXPERIMENT 8.3
Mass Absorption Coefficient for X-Rays

In this experiment, the attenuation of the ^{65}Zn K_α x-rays at 8.04 keV will be measured as they pass through aluminum and nickel foils.

Procedure

1. Set up the electronics as shown in Fig. 8.3. Adjust the ROI (Region of Interest) of the MCA to select the channels that make up the peak for the 8.04 keV line from the ^{65}Zn source. For this measurement, the source should be placed ~1 cm from the face of the detector.

Be sure there is plenty of space between the source and detector for insertion of the foils without threatening the thin Be window. The chamber for x-ray fluorescence measurements, which is optional for this experiment, can conveniently be used

for these measurements. With this chamber, four foils can be loaded at one time and then sequenced as desired.

2. Accumulate the Zn K_α peak for a long enough MCA live time to acquire ~6000 counts in the peak. From the analyzer data, determine the number of Zn K_α x-rays detected per second. From this point on, record only the integrated total number of counts accumulated in the peak.

3. Insert the first foil thickness from Table 8.4. Clear the MCA and set it for enough preset time to obtain reasonable statistics in the peak.

4. Count the preset time interval. Record the integrated count total in Table 8.4; then repeat for each of the other thicknesses listed in Table 8.4.

EXERCISE

Make a plot of counts vs. absorber thickness and the mass attenuation coefficients for aluminum and nickel. Refer to Experiment 11 for examples of these plots as they were obtained when using a proportional counter rather than a Si(Li) detector. How do your data compare to Figs. 11.5 and 11.6 in Experiment 11?

Table 8.4

Aluminum Thickness (mg/cm^2)	Counts	Nickel Thickness (mg/cm^2)	Counts
5		5	
10		10	
15		15	
20		20	
25		25	
30		30	
35		35	
40		40	
45		45	
50		50	
55		55	
60		60	

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