EXPERIMENT 8

High-Resolution X-Ray Spectroscopy

EQUIPMENT NEEDED FROM EG&G ORTEC

Source Kit SK-1X
~1 μCi each of ^{65}Zn, ^{137}Cs, ^{55}Fe, and ^{57}Co (see Table 8.2 for other possibilities)
Bin and Power Supply
SLP-06175 Series Si(Li) X-Ray Detector System; typical specifications: 6 mm diam, 175 eV resolution at 5.9 keV, and 1-mil Be window
572 Amplifier
480 Pulser
459 5-kV Detector Bias Supply
ACE-2K MCA System including suitable IBM PC (other EG&G ORTEC MCAs may be used)
12 each AlFl-2 aluminum and 12 each NiFl-1 nickel absorbers (see Appendix)
ORC-8 Cable Set
Model 311 Chamber (optional)
Oscilloscope

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-resolution x-ray spectroscopy is an equally challenging research field. The Nuclear Spectroscopy Centers suggested in Experiment 7 can be duplicated for the area of x-ray spectroscopy. The state-of-the-art in x-ray spectroscopy with solid-state detector systems is changing almost every day. Both Si(Li) and germanium systems can be used for x-ray spectroscopy although this experiment is written for the use of a Si(Li) system. High-purity germanium (HPGe) detectors are also available for this discipline.

Figure 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: an 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 give 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 $K\alpha$ and $K\beta$ fluorescence x rays for most elements above ^{16}O. The subject of x-ray fluorescence is treated in Experiment 12.

In general, Si(Li) systems provide a little better resolution than can be obtained with HPGe systems. In contrast, the efficiencies of the 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. Figure 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 beryllium window on the efficiency. From the figure it can be seen that the window becomes important for energies below 6 keV, with comparable wave lengths >1 Å. 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.

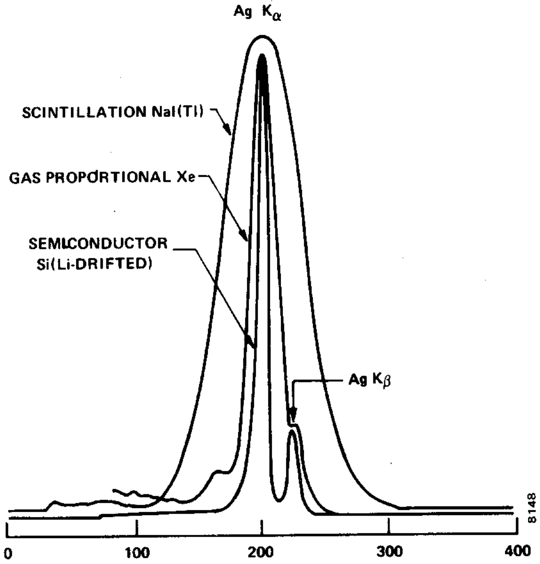

Fig. 8.1. Demonstration of the Resolution Capability of the Three Types of X-Ray Detectors for the Silver K Spectra Obtained from ^{109}Cd Source.
(Courtesy Philip G. Burkhalter and William J. Campbell, U.S. Bureau of Mines, College Park, Maryland.)

EXPERIMENT 8.1

Energy Calibration with a Pulser

Procedure

1. Install the EG&G ORTEC 459, 480, and 572 in the Bin and Power Supply. Interconnect the modules, the preamplifier on the detector, and the MCA as shown in Fig. 8.3. The preamplifier is mounted on the detector, and the signal con-

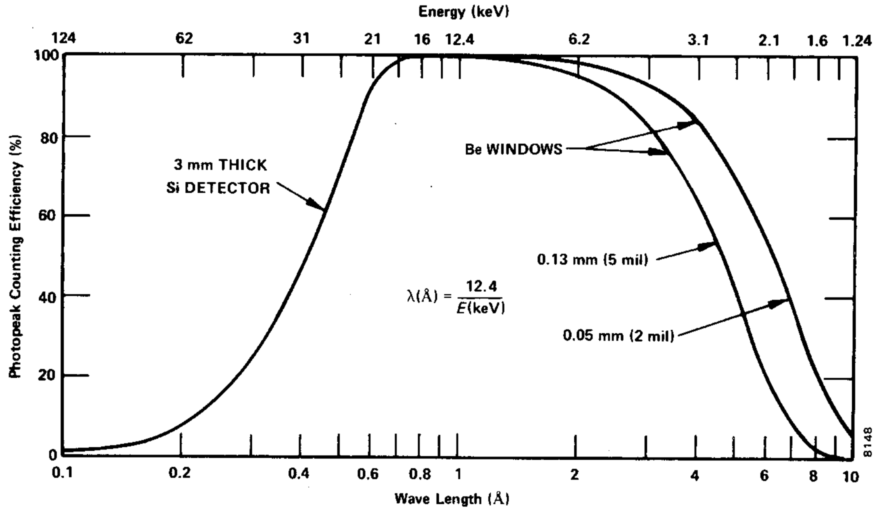

Fig. 8.2. Spectral Response of Si Detector with Be Windows.

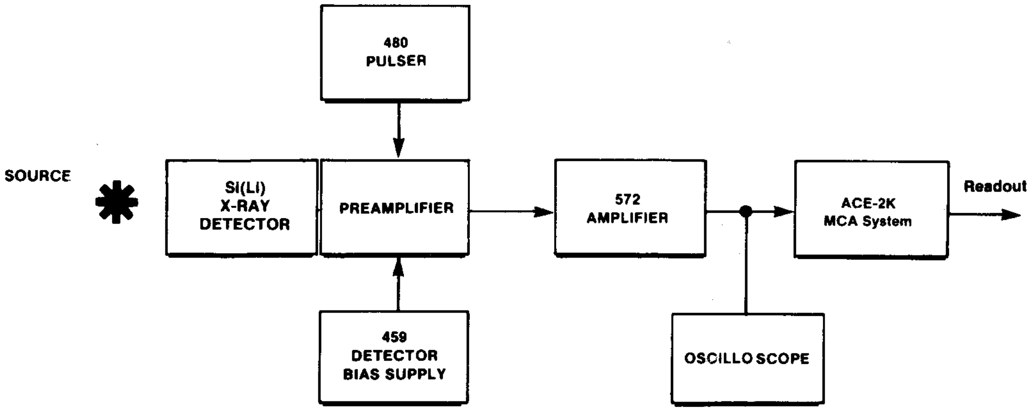

Fig. 8.3. Equipment Interconnections for Experiment 8.1.

nection from the detector to the preamplifier is made internally in the mounting. Check the detector data sheet for the correct polarity and high voltage level, and select the correct polarity with the 459 while its amplitude controls are set at zero. The oscilloscope will be used to check the waveform at the output of the 572 Amplifier.

2. Raise the voltage output of the 459 to the recommended value.

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

4. Adjust the gain of the 572 Amplifier so that the 32.1-keV Kα line for ^{137}Cs falls in the upper portion of the analyzer.

5. Accumulate a spectrum for a time period long enough to have 2000 counts in the peak for the 32.1 Kα 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 dials 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 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. Figure 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 identified easily. These isotope decay schemes are shown in ref. 10.

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 that are used for all of these experiments should be sources that are manufactured specifically for x-ray studies. For x-ray sources, most manufacturers will deposit a spot of the radioactive material onto 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 window (for this experiment, all sources must be placed at exactly the same distance from the detector).

3. Adjust the gain of the 572 Amplifier so that the 32.1-keV K_α line is in the upper portion of the 1024-channel analyzer

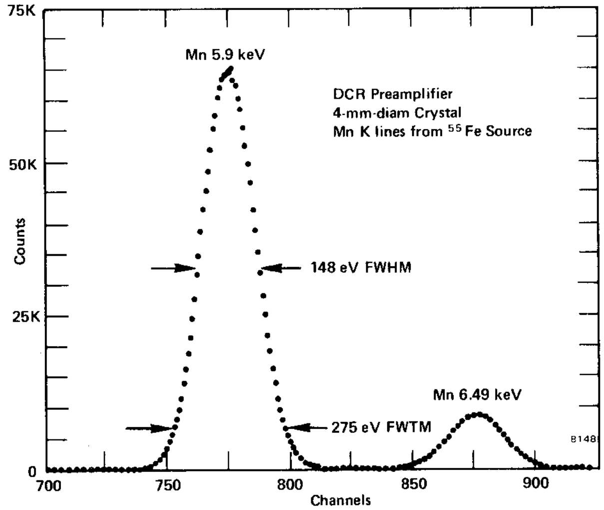

Fig. 8.4. K_α and K_β X-Ray Lines from an ^{55}Fe Source.

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

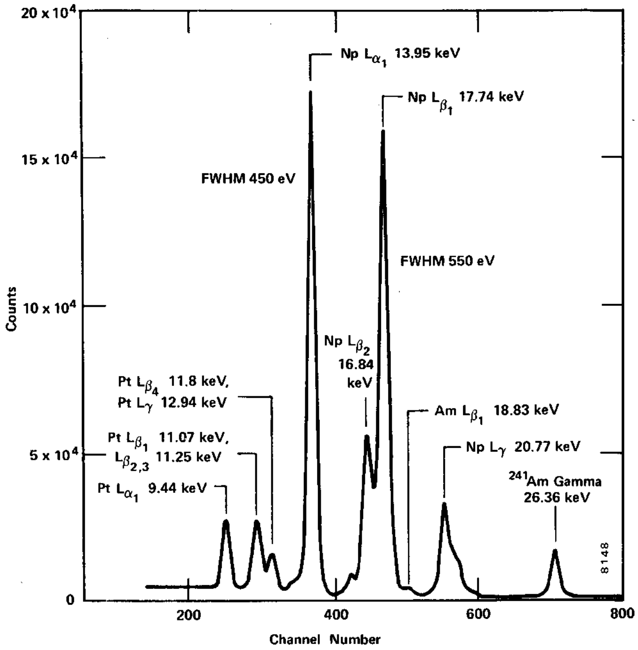

Fig. 8.5. Typical ^{241}Am Spectrum Taken with Si(Li) X-Ray Detector.

range. Accumulate for a time period long enough to obtain ~6000 counts in the Kα peak. Record the live time and readout the MCA. 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. Readout 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, and 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.

c. Determine the theoretical number of x rays for each line by multiplying the activity of the source in gammas/s 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. Figure 8.2 shows the shape of this efficiency curve for various Be window thicknesses.

EXPERIMENT 8.3

Mass Absorption Coefficient for X Rays

In this experiment the attenuation of the ^{55}Zn Kα 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 the detector for insertion of the foils without threatening the thin Be window. The Model 311 Chamber, 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 Kα peak for a long enough analyzer live time to acquire ~6000 counts in the peak. From the analyzer data, determine the number of Zn Kα 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 analyzer and set it for enough preset time to get reasonable statistics in the peak.

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

EXERCISE

Make a plot of counts vs absorber thicknesses. Determine the half-value thicknesses 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 8.3. 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					

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	

References

1. R. E. Wood, P. V. Rao, *et al.*, *Nucl. Instrum. Methods* **94**, 245 (1971).
2. Z. Moroz and M. Moszynski, *Nucl. Instrum Methods* **68**, 261 (1969).
3. G. F. Knoll, *Radiation Detection and Measurement,* John Wiley and Sons, New York (1979).
4. R. J. Gehrke and R. A. Lokken, *Nucl. Instrum. Methods* **97**, 219 (1971).
5. J. C. Russ, Coordinator, *Energy Dispersion X-Ray Analysis, X-Ray and Electron Probe Analvsis.* Available as ASTM Special Technical Publication 485, 1970, 04-485000-39 from American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania.
6. R. D. Giauque and J. M. Jaklevic, "Rapid Quantitative Analysis

by X-Ray Spectrometry," *Adv. in X-Ray Analysis* **15**, 266, Plenum Press, New York (1972).

7. J. M. Jaklevic and F. S. Goulding, "Semiconductor Detector X-Ray Fluorescence Spectrometry Applied to Environmental and Biological Analysis," *IEEE Trans. Nucl. Sci.* **NS-19** (1972).

8. J. L. Campbell and L. A. McNelles, "An Intercomparison of Efficiency Calibration Techniques for Semiconductor X-Ray Detectors," *Nucl. Instrum. Methods* **125**, 205-223 (1975).

9. 14th Scintillation and Semiconductor Counter Symposium, *IEEE Trans. Nucl. Sci.* **NS-22**(1) (1975).

10. C. M. Lederer and V. S. Shirley, Eds., Table of Isotopes, 7th Edition, John Wiley and Sons, Inc., New York (1978).