Title

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ABSTRACT

Lithium-drifted germanium gamma-ray spectrometers have been used for the nondestructive analysis of a sulfide ore by neutron activation. The concentrations of ten elements have been determined in the range 1.8×10^5 p.p.m. (iron) to 2.0 p.p.m. (gold). The analytical sensitivity obtained with available Ge(Li) detectors was compared with sodium iodide scintillators for the estimation of manganese in the ore. The various detector properties which effect sensitivities are discussed.

INTRODUCTION

The application of high-resolution, lithium-drifted germanium gamma-ray detectors to nondestructive neutron activation analysis has been reported by Prussin, Harris, and Hollander (13) for the study of trace impurities in aluminum and by Girardi, Guzzi, and Pauly (2) for the determination of minor amounts of hafnium in zirconium oxide. These studies illustrated the benefits to be gained from high resolution gamma-ray spectra in the special case of noninterfering bulk material. In many practical analyses, however, minor and trace constituent concentrations are desired for samples in which the activation of major constituents may contribute a high gamma-ray background. In such cases the sensitivity of analysis for a given component may be severely limited by the complexity of the gamma-ray spectra as well as by the masking effect of the radiations from the activated bulk material. The masking effect is of particular concern in the application of available lithium-drifted germanium detectors since the ratio of Compton to photopeak efficiencies for these detectors is considerably larger than the corresponding ratio for the commonly used 3 × 3 inch (diameter) sodium iodide detectors.

In this paper we report the results of the application of lithium-drifted germanium detectors to the nondestructive analysis of minor and trace constituents in a sample of a sulfide ore in which the major constituents iron, zinc, and copper have substantial neutron activation cross sections and yield (n,γ) products with reasonably long half-lives. Results are given for analysis of a number of elements in the ore in the concentration range 1.7×10^5 p.p.m. (iron) to 2 p.p.m. (gold). Sensitivities for analysis of manganese in the ore were estimated from gamma-ray spectra obtained with two Ge(Li) detectors of

different active volumes and with a very good 3 × 3 inch (diameter) sodium iodide scintillator. This comparison suggests that sensitivities obtained with gamma-ray spectra from available germanium detectors are comparable to sensitivities obtained using spectra from sodium iodide detectors.

EXPERIMENTAL

Spectrometers. The construction and general characteristics of the lithium-drifted germanium detectors and detector assemblies are described in References (1, 3, 4, 6, and 11). The two Ge(Li) detectors used in this study had dimensions of about 2 sq. cm. × 7 mm. (active thickness) and 6 sq. cm. × 10 mm. (active thickness). The resolutions of these detectors for the 122-k.e.v. gamma ray of Co^{57} were approximately 2 k.e.v. (FWHM) and 4 k.e.v. (FWHM), respectively. The associated electronics consisted of a low-noise, low-capacity preamplifier and a linear-amplifier, biased amplifier system designed by Goulding and Landis (5) and constructed at this laboratory. The 3 × 3 inch (diameter) sodium iodide (thallium) detector system was a Harshaw integrally-mounted unit which exhibited a resolution (FWHM) at 662 k.e.v. of slightly less than 46 k.e.v. (7%). Pulses from the various detector systems were routed to a Victoreen, SCIPP 1600-channel pulse-height analyzer for sorting.

Sample Preparation. The bulk sulfide ore was ground to a fine powder and mixed to insure homogeniety of sampling in the range 50-100 mg. Weighed samples were then sealed in polyethylene film or quartz tubing for irradiation. Quantitative analyses were performed by comparison of photopeak intensities of activities induced in the ore to those induced in appropriate standards. To minimize

neutron flux variations between samples and standards, the latter were prepared by evaporating aliquots of stock solutions onto thin polyethylene films and heat sealing a polyethylene cover film over the deposit. The volume occupied by the standards was thereby minimized, permitting the positioning of standards close to the samples within the irradiation capsules.

Irradiations. Neutron irradiations were carried out at the Livermore LPTR nuclear reactor. The thermal neutron flux at the irradiation position was approximately 5×10^{12} n./sq. cm.-sec., and the neutron spectrum exhibited a cadmium ratio of approximately 11. Irradiation periods ranged from 2 hours to 72 hours, and samples were available for counting within 2-4 hours after the end of irradiation.

RESULTS AND DISCUSSION

Analytical. The qualitative identification of the radioisotopes induced in irradiated ore samples was made through determination of photopeak energies and half-lives and by comparison of experimental gamma-ray relative intensities to literature values for isotopes yielding more than one photon in their decay.

A sample of sulfide ore of approximately 150 mg. was irradiated for 2 hours in the LPTR facility to provide gamma-ray spectra of the shorter-lived activities induced in the sample. The spectra were obtained with the smaller Ge(Li) detector [2 sq. cm. × 7 mm. (active thickness)] using an energy interval of approximately 1 k.e.v. per channel. Data were accumulated for 60 minutes (live time) at two-hour intervals over a period of twelve hours. The first spectrum, taken 4.5 hours after the end of the irradiation, is shown in Figure 1 (Spectrum 2). For comparison we have included a spectrum of the ore obtained

with the sodium iodide scintillator (Spectrum 1). Photopeak energies and half-lives were estimated from the Ge(Li) spectra, and isotope assignments were made by comparing these data to literature values (7, 8, 12). Assignments were confirmed, where possible, with relative intensity measurements. These data indicated the presence of In^{116m}, Mn^{56}, Zn^{69m}, Sb^{122}, and Na^{24} in the activated sample. In Table I are listed the experimental measurements together with the literature values of each quantity. It is interesting to note that much of the relative intensity data obtained from NaI(Tl) gamma-ray spectra may be subject to greater experimental uncertainties than those obtained through the germanium spectra. The Ge(Li) intensity data are estimated to have uncertainties of 5-10% for the more intense lines in the spectra. This was checked by using the relative intensities of photopeaks in a spectrum from an irradiated indium standard. These values are shown in Table II in parentheses beside the literature values.

Enhancement of radioisotopes of longer half-lives was obtained through a 49-hour irradiation of 50 mg. of the sulfide ore. After a decay period of 24 hours, spectra were obtained over a two-day period at intervals of four hours. A typical spectrum representing data accumulation for 120 minutes (live time) is shown as Spectrum 3 of Figure 1. These spectra indicated the presence of Au^{198}, Ga^{72}, Fe^{59}, and Zn^{65} in the activated ore in addition to those isotopes listed above. Pertinent data for assignments to these isotopes have been included in Table I.

Quantitative estimation of the elements in the ore giving rise to these activities was obtained through subsequent irradiations of similar samples. These data are given in Table II.

The simplicity of both qualitative and quantitative analysis afforded by the use of Ge(Li) detectors is well demonstrated by considering the neighboring 559-k.e.v. gamma-ray line of As^{76} and the 563-k.e.v. line of Sb^{122}. It is obvious from Figure 1 that accurate resolution of these photopeaks in the sodium iodide spectrum would be difficult, if not impossible, because of the large 511-k.e.v. annihilation photopeak from the β^+ decay of Cu^{64}. The difficulty is intensified by the necessity of interpolating the complex background beneath these photopeaks over an energy range of approximately 200 k.e.v. (40 channels). Figure 2 shows this energy region of a Ge(Li) spectrum obtained with the smaller detector. The spectrum from which Figure 2 was prepared represents a 500-minute count of 19 mg. of the ore, taken 36 hours after a 72-hour irradiation. Peak-shape resolution of the two lines was accomplished for each spectrum, in a decay sequence as shown in Figure 3, by using the single 438-k.e.v. gamma-ray line of Zn^{69m} in the same spectrum as the standard peak-shape (line widths increase by less than 0.5 k.e.v. over this energy range). The analytical data shown in Table II for arsenic and antimony were obtained by this method.

Sensitivity. The value of the germanium detector for nondestructive activation analysis rests in large part upon the analytical sensitivity obtained with its use. In order to provide a basis for judging relative merit, we have made measurements on the same samples of the ore with Ge(Li) detector systems and with the commonly used 3 × 3 inch (diameter) sodium iodide scintillator. The major characteristics of the systems which must be considered in such a comparison are the overall efficiencies, relative photopeak to Compton efficiencies, and the resolutions of the detectors. It is obvious that the vastly improved

resolution of the germanium systems will improve sensitivities in complex spectra when the major limitation of importance is the ability to resolve cleanly two or more gamma-ray lines which lie close in energy. Clean resolution of such lines will also improve the accuracy of analysis in cases where the spectrum exhibits gamma-rays of similar energies and half-lives. In order to assess the effects of resolution and efficiencies upon sensitivity in the general case, it is necessary to examine the error in the estimation of a photopeak which is superimposed upon a large background from higher-energy gamma rays in the spectrum. For the present purposes we consider only the statistical counting error in such a determination (9).

The standard deviation in the measurement of the number of counts, N_p, in a photopeak may be approximated by

$$\sigma_p = \sqrt{\sigma_t^2 + \sigma_b^2} = \sqrt{N_p + 2\,N_b} \qquad (1)$$

where the subscripts t and b refer to total and background counts in the region of the photopeak, and $N_p = N_t - N_b$. If it is required that the photopeak intensity be known within some specific error limit, then

$$\sigma_p = K\,N_p \qquad (2)$$

The number of counts in the photopeak necessary to give rise to this limit of error for a given background, N_b, is

$$N_p = \frac{1}{2K^2}\left[1 + \sqrt{1+8K^2 N_b}\right] \qquad (3)$$

It is seen from Equation (1) that N_b will introduce a major contribution to the error in N_p when the intensity of Compton distributions from higher-energy lines is comparable to the photopeak intensity. The error in the estimation of a photopeak will be reduced by factors which tend to increase the ratio of N_p to N_b and which tend to increase the total number of counts accumulated.

There are several response characteristics of Ge(Li) and NaI(Tl) detectors that will affect the errors discussed above. The higher resolution of the Ge(Li) detectors tends to increase the ratio of N_p to N_b since the background under a photopeak is summed over a smaller energy interval than required in sodium iodide spectra. However, the higher ratio of photopeak to Compton efficiencies characteristic of NaI(Tl) scintillators will tend to produce a more intense photopeak and thereby increase the ratio of N_p to N_b relative to germanium. The ratios of the photopeak efficiencies and linewidths of NaI(Tl) relative to Ge(Li) both increase with increasing gamma-ray energies, and these effects will tend to compensate for one another. As a result, the major difference in detector characteristics of importance is in total detection efficiencies. The high efficiency of a 3 × 3 inch (diameter) sodium iodide scintillator will result in greater total counts than that obtained with the small germanium detectors for given experimental conditions.

The above considerations are illustrated by the estimation of the sensitivity for the determination of manganese in the sulfide ore using the two Ge(Li) detectors and the 3 × 3 inch (diameter) sodium iodide detector. Gamma-ray spectra obtained with each detector in the region of the 845-k.e.v. photopeak of Mn^{56} are shown in Figure 3. The photopeak in the NaI(Tl) spectrum (Spectrum 1) covers an energy range of about 175 k.e.v. and appears to be

single, while the spectrum from the larger Ge(Li) detector (Spectrum 2) shows complete resolution of the 819-k.e.v. line from In^{116m}. The spectrum obtained with the smaller Ge(Li) detector (Spectrum 3) shows, in addition, the complete resolution of the 835-k.e.v. line of Ga^{72}.

The decay of 2.6-hour Mn^{56} was followed by using each of the three detectors and the resulting decay curves are shown in Figure 4. The data from which this figure was prepared are given in Table 3. Because of the difficulty in peak-shape analysis, the complex photopeaks in the sodium iodide spectra were treated as single photopeaks. The initial portion of the NaI(Tl) decay curve is then in error to the extent of contribution from the 54-minute In^{116m}. The error caused by the inclusion of the 845-k.e.v. photopeak from Ga^{72} is negligible. The error bars in Figure 4 represent $\pm 1\sigma$ limits as determined from Equation (1).

The quantity $(100\ \sigma_p)/(N_p)$ as a function of time after irradiation has been determined for the three Mn^{56} decay curves and is shown in Figure 5. These curves clearly show the effects of the level of Compton background upon the error in photopeak determinations. In the curves for the two Ge(Li) spectra, the decrease in error at early times is due to the rapid decay of the In^{116m}, which increases the ratio of N_p to N_b. The apparent linearity in the sodium iodide data during this time interval is due to the inclusion of the In^{116m} photopeak. In all three sets of data the error increases roughly exponentially for times long compared to the half-life of In^{116m}. In this region a reasonable comparison of the sensitivity for estimation of manganese may be attained by comparing the relative photopeak intensities for a given statistical error limit. In the present case, we have determined the fraction of manganese remaining at the times corresponding to the error limit $\sigma_p = 0.1\ N_p$. The

corresponding limits of manganese concentration were determined from these data to be 21 p.p.m. (small Ge(Li)), 4.4 p.p.m. (large Ge(Li)), and 0.73 p.p.m. (NaI(Tl)).

It should be noted that the statistical treatment presented here has neglected several sources of error which are of particular importance for the sodium iodide data. The unresolved 835-k.e.v. line of Ga^{72} will contribute significantly to the apparent Mn^{56} photopeak near the sensitivity limit and decrease this limit from the statistical estimate. Although the interpolation of a background under the narrow photopeaks in germanium spectra can generally be performed with certainty, the interpolation over the large energy interval required in the sodium iodide spectra can contribute greatly to the error in estimation of the photopeak intensity.

The sensitivity limits set for the less efficient Ge(Li) detectors are strongly dependent upon the counting interval and sample size in the experiments reported here. With reasonable increases in these variables, the total counts accumulated with the germanium detectors may be increased and sensitivities comparable to sodium iodide can be obtained.

CONCLUSIONS

The results of this study show the applicability of available lithium-drifted germanium gamma-ray spectrometers to nondestructive neutron activation analysis of samples in which the major constituents as well as the minor and trace constituents are activated. The simplicity of the spectra obtained with these semiconductor detectors allows rapid and accurate estimation of constituents whose concentrations may vary over a very wide range. Similar treatment of data obtained using sodium iodide scintillators would be very

difficult and, in many instances, totally impractical. In the present work, for example, the simplest photopeak in the sodium iodide spectra (845-k.e.v.) was seen to be composed of three gamma-ray lines superimposed on a complex background.

The analytical sensitivities obtained with the various detectors for the particular case of estimation of manganese in the ore indicate that the larger Ge(Li) detector is capable of yielding sensitivities comparable to the sodium iodide detector. This comparison shows the sodium iodide system at its best, in that the gamma-ray line studied was fairly well resolved from other lines in the spectrum. It is clear that the relative sensitivities obtained with the germanium detector would be much better for many of the gamma-ray lines which are not so well resolved in the NaI(Tl) spectra. Also it should be noted that the conditions of sample size, counting interval, and counting geometry could be optimized for use with Ge(Li) spectrometers. The data in Table III show that the sample activity used in the sensitivity comparison is near the practical limit for the sodium iodide detector, while a much more active sample could be used with the less efficient germanium systems.

As a final note we wish to point out that the small volumes of available Ge(Li) detectors have been set by the techniques used in fabrication and not by the intrinsic properties of the system. Recently Malm, Tavendale, and Fowler (10) have reported the construction of a Ge(Li) detector of about 16 cc. (active volume) by a new drifting technique. The efficiency reported for this detector was 2.5% at 1300 k.e.v., comparable to a 1 × 1 inch (diameter) NaI(Tl) scintillator. The detector resolution was 3.3 k.e.v. (FWHM) and 4.8 k.e.v. (FWHM) at 122 and 1333 k.e.v., respectively. The new drifting technique should make available Ge(Li) detectors of excellent resolution with several times the volume of this detector.

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Table I. Isotope assignments for Activities Induced in the Sulfide Ore

	Experimental			Literature[a]		
Isotope	$T_{1/2}$	E_γ(k.e.v.)	Rel. Inten.	$T_{1/2}$	E_γ(k.e.v.)	Rel. Inten.
In^{116m}	54m	137	14.2	54m	135	12 (14.8)
		416	100[b]		406	100 (100)
		819	39.1		820	- (43.2)
		1100	233		1085	216 (221)
		1300	320		1270	300 (318)
Mn^{56}	2.7 h	845		2.6 h	845	
Cu^{64}	12.5 h	1345		12.8 h	1340	
		511(annihilation)				
Zn^{69m}	14 h	438		13.8 h	439	
Zn^{65}	-	1119		245 d	1119	
Ga^{72}	14.4 h	835		14.3 h	835	
Na^{24}	15.5 h	1369		15 h	1368	
As^{76}	26 h	559	100[b]	26.3 h	559	100
		657	14.6		657	14.6
		1216	-		1216	9.7
Sb^{122}	~60 h	563		62.7 h	566	
		686			686	
Au^{198}	~60 h	411		64.8 h	412	
Fe^{59}	-	1095		45 d	1100	
		1292			1290	

[a]Nuclear Data Sheets (Reference 12).

[b]Relative intensities normalized to this value.

Table II. Results of Quantitative Analysis of Sulfide Ore

Element	μg. Present	Sample weight mg.	Irradiation time hours	E_γ k.e.v.	p.p.m.[a]
Mn	6.0	62.9	2	845(Mn^{56})	95 ± 7
In	7.6	62.9	2	1300(In^{116m})	120 ± 5
Ga	3.8	62.0	2	835(Ga^{72})	61 ± 16
Zn	3020	24.5	24	438(Zn^{69m})	$(1.20 \pm 0.01) \times 10^5$
As	60	19.0	72	559(As^{76})	$(3.16 \pm 0.04) \times 10^3$
Sb	8.2	19.0	72	563(Sb^{122})	430 ± 6
Cu	1240	19.0	72	511(Cu^{64})[b]	$(6.50 \pm 0.01) \times 10^4$
Na	16	19.0	72	1369(Na^{24})	840 ± 25
Fe	3370	19.0	72	1095(Fe^{59})	$(1.78 \pm 0.01) \times 10^5$
Au	0.038	19.0	72	411(Au^{198})	2.0 ± 0.2

[a]Errors shown represent statistical counting error only.

[b]Copper concentration corrected for contribution to 511-k.e.v. annihilation photopeak from decay of Zn^{69} and Na^{24}.

Table III. Data for Comparison of Sensitivities between Ge(Li) Detectors and 3 × 3 Inch (Diameter) NaI(Tl) Scintillator.

Sample weight: 62.9 mg.
Irradiation time: 2 hr.
Counting interval: 60 min.
Flux: 5×10^{12} n./sq. cm.-sec.
Detector: small Ge(Li)

Time	N_p ($\times 10^{-3}$)	N_b ($\times 10^{-3}$)	$(100\ \sigma)/(N_p)$
4.0	2.98	10.0	5.07
6.3	1.55	3.76	6.14
8.5	0.923	2.24	7.96
10.7	0.592	1.89	11.20
13.0	0.240	1.13	20.80

Sample weight: 62.0 mg.
Irradiation time: 2 hr.
Counting interval: 60 min.
Flux: 5×10^{12} n./sq. cm.-sec.
Detector: large Ge(Li)

Time	N_p ($\times 10^{-3}$)	N_b ($\times 10^{-3}$)	$(100\ \sigma)/(N_p)$
2.0	38.85	209.3	1.74
5.0	16.34	45.0	2.00
7.4	9.27	25.0	2.63
9.7	4.63	17.0	4.24
12.1	2.30	14.0	7.58
14.3	1.26	11.0	12.15

Sample weight: 62.9 mg.
Irradiation time: 2 hr.
Counting interval: 60 min.
Flux: 5×10^{12} n./sq. cm.-sec.
Detector: 3 × 3 inch NaI(Tl)

Time	N_p ($\times 10^{-3}$)	N_b ($\times 10^{-3}$)	$(100\ \sigma)/(N_p)$
5.2	209.4	310.3	0.43
7.4	102.3	182.0	0.67
9.6	59.3	125.0	0.94
11.9	32.9	94.3	1.43
14.1	18.5	82.4	2.32

FIGURE CAPTIONS

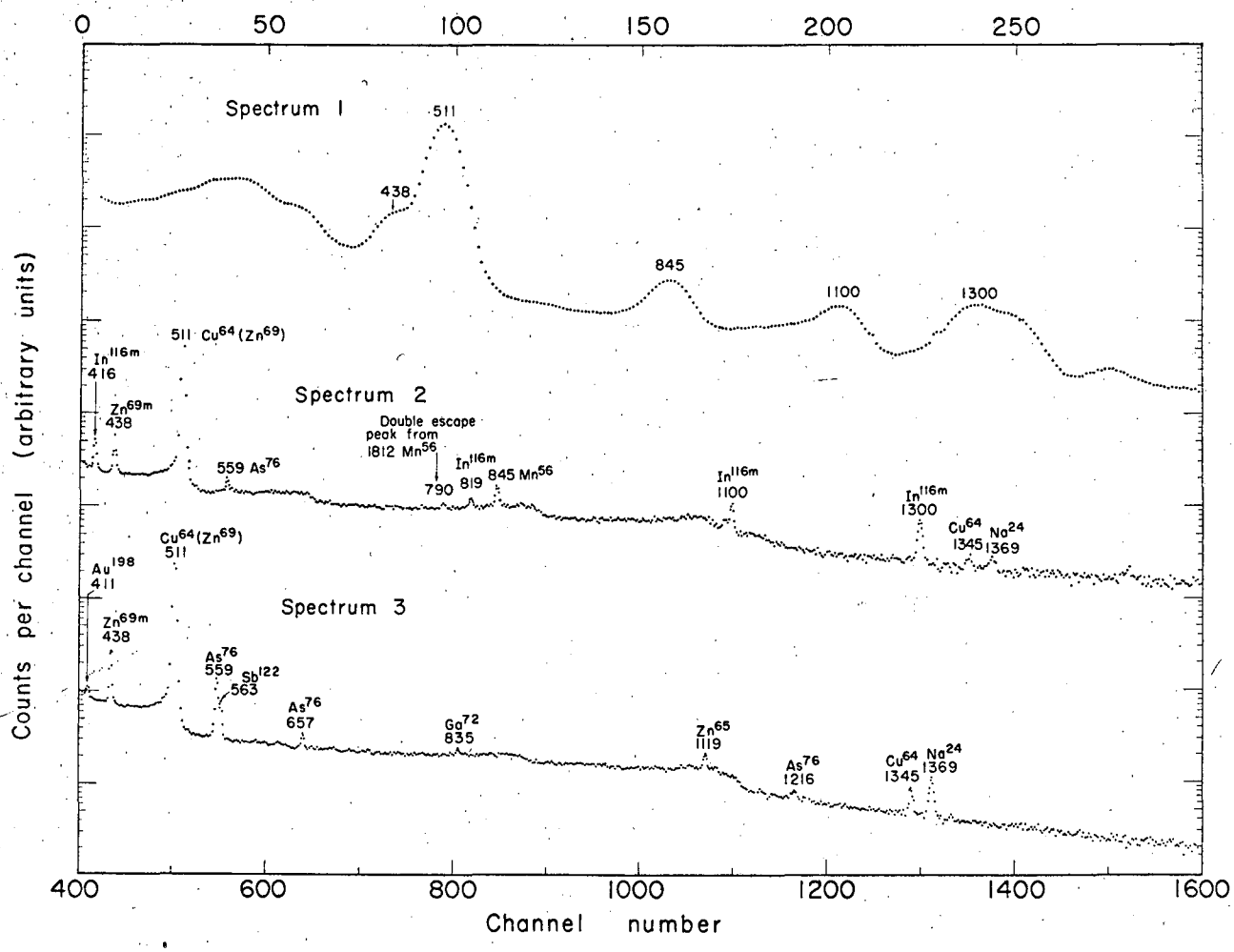
Fig. 1. Gamma-ray spectra of activated sulfide ore. Spectrum 1 was obtained with the 3 × 3 inch (diameter) sodium iodide scintillator 5.2 hours after a 2-hr. irradiation of 62.9 mg. of sulfide ore. Spectrum 2 was obtained with the smaller Ge(Li) detector 4 hours after a 2-hr. irradiation of 62.9 mg. of the ore. Spectrum 3 was obtained with the smaller Ge(Li) detector 24 hours after a 49-hr. irradiation of 50 mg. of the ore.

Fig. 2. Peak-shape fit for the 559-k.e.v. gamma-ray line of As^{76} and 563-k.e.v. gamma-ray line of Sb^{122} in the activated sulfide ore. The spectra were obtained with the smaller Ge(Li) detector. The energy interval is approximately 1 k.e.v. per channel. The upper spectra represent the total counts in the regions of the photopeaks and the lower spectra represent the counts in the photopeak (N_t-N_b).

Fig. 3. Energy region of the 845-k.e.v. photopeak of Mn^{56}. Spectrum 1 was obtained with the sodium iodide scintillator. Spectra 2 and 3 were obtained with the larger and smaller Ge(Li) detectors, respectively.

Fig. 4. Decay curves for 2.6-hr. Mn^{56} in the activated sulfide ore. Curve 1 represents data obtained with the sodium iodide scintillator. Curves 2 and 3 represent data obtained with the larger and smaller Ge(Li) detectors, respectively.

Fig. 5. Percent standard deviation in photopeak intensity vs time after irradiation for the 845-k.e.v. gamma-ray of Mn^{56}. Curves 1 and 2 represent $(100\ \sigma_p)/(N_p)$ for the smaller and larger Ge(Li) detectors, respectively. Curve 3 is for the NaI(Tl) data.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50
100
150
200
250
Spectrum 1
511
438
845
1100
1300
Spectrum 2
In116m 416
Zn69m 438
511 Cu64(Zn69)
559 As76
Double escape peak from 1812 Mn56
790
In116m 819
845 Mn56
In116m 1100
In116m 1300
Cu64 1345
Na24 1369
Spectrum 3
Au198 411
Zn69m 438
Cu64(Zn69) 511
As76 559
Sb122 563
As76 657
Ga72 835
Zn65 1119
As76 1216
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Counts per channel (arbitrary units)
400
600
800
1000
1200
1400
1600
Channel number

Fig. 1

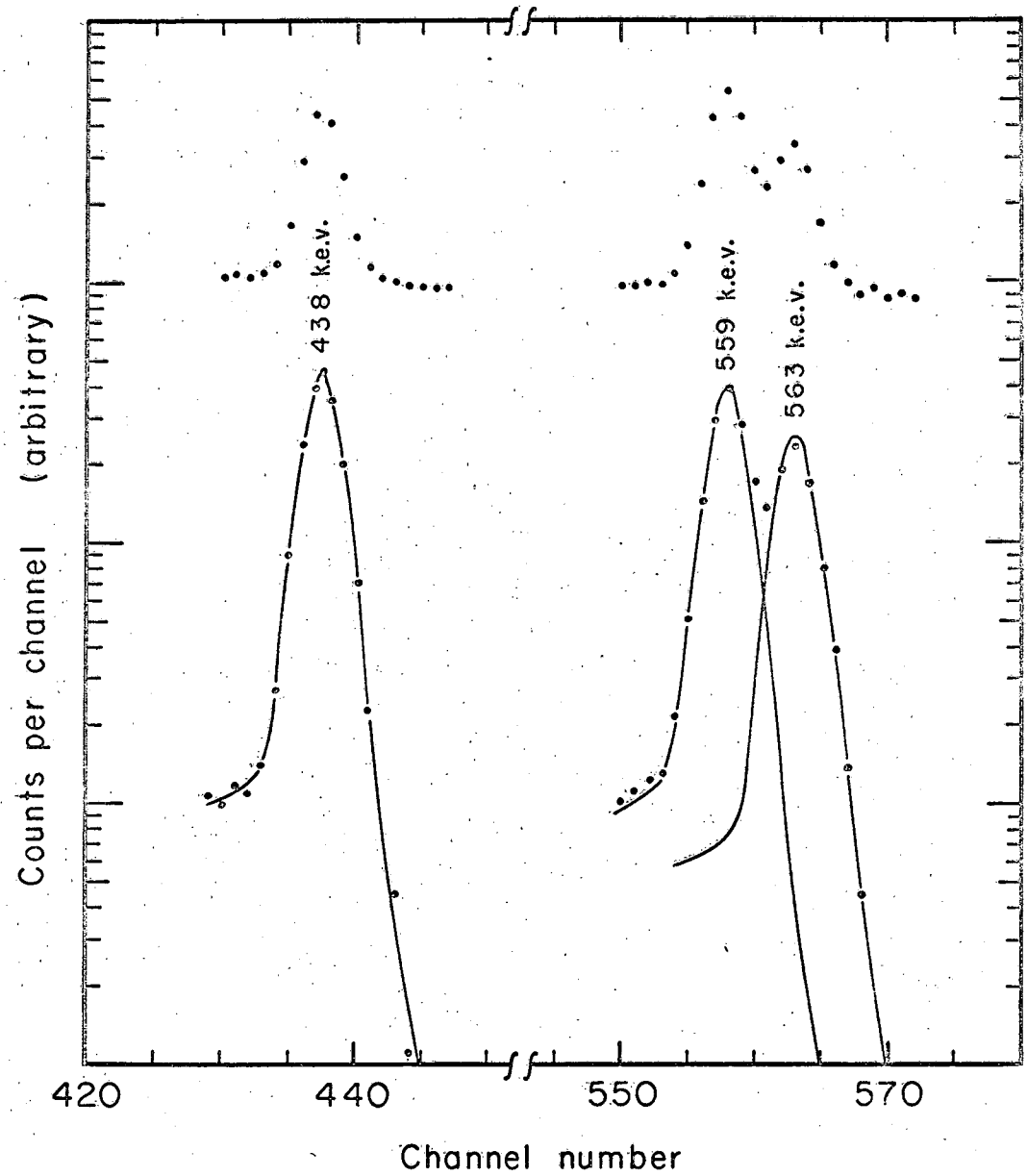

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Fig. 2

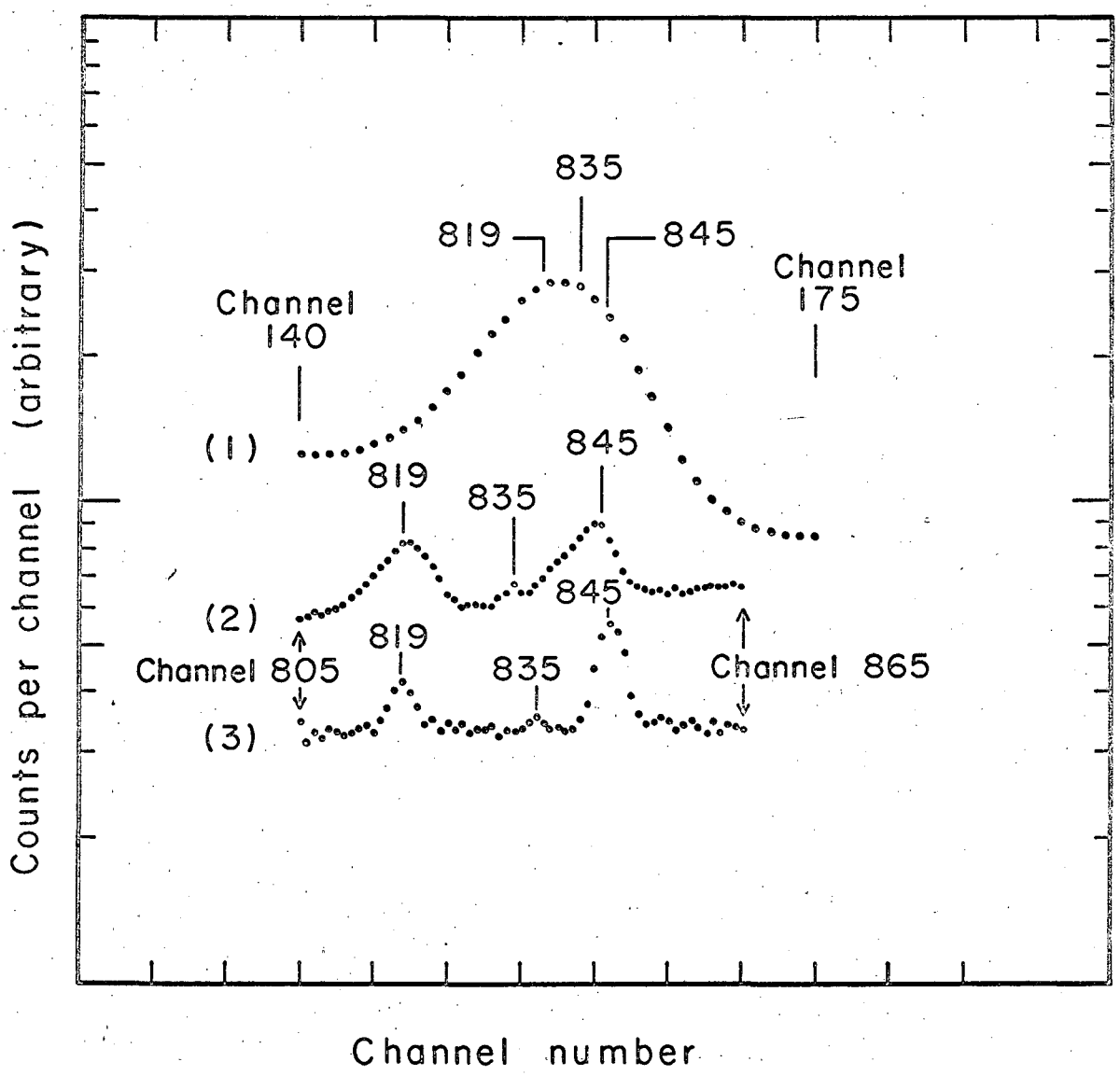

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Fig. 3

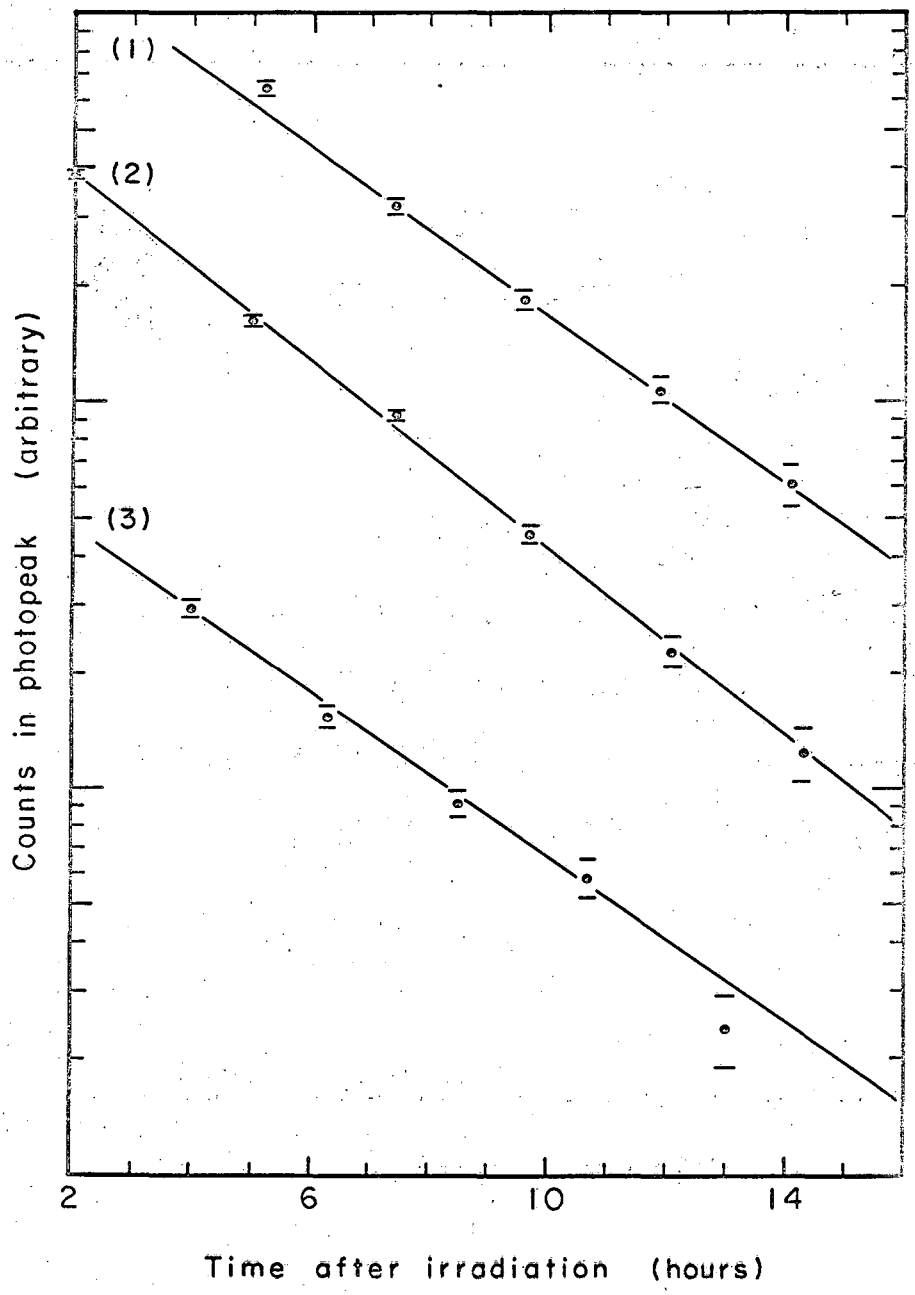

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Fig. 4

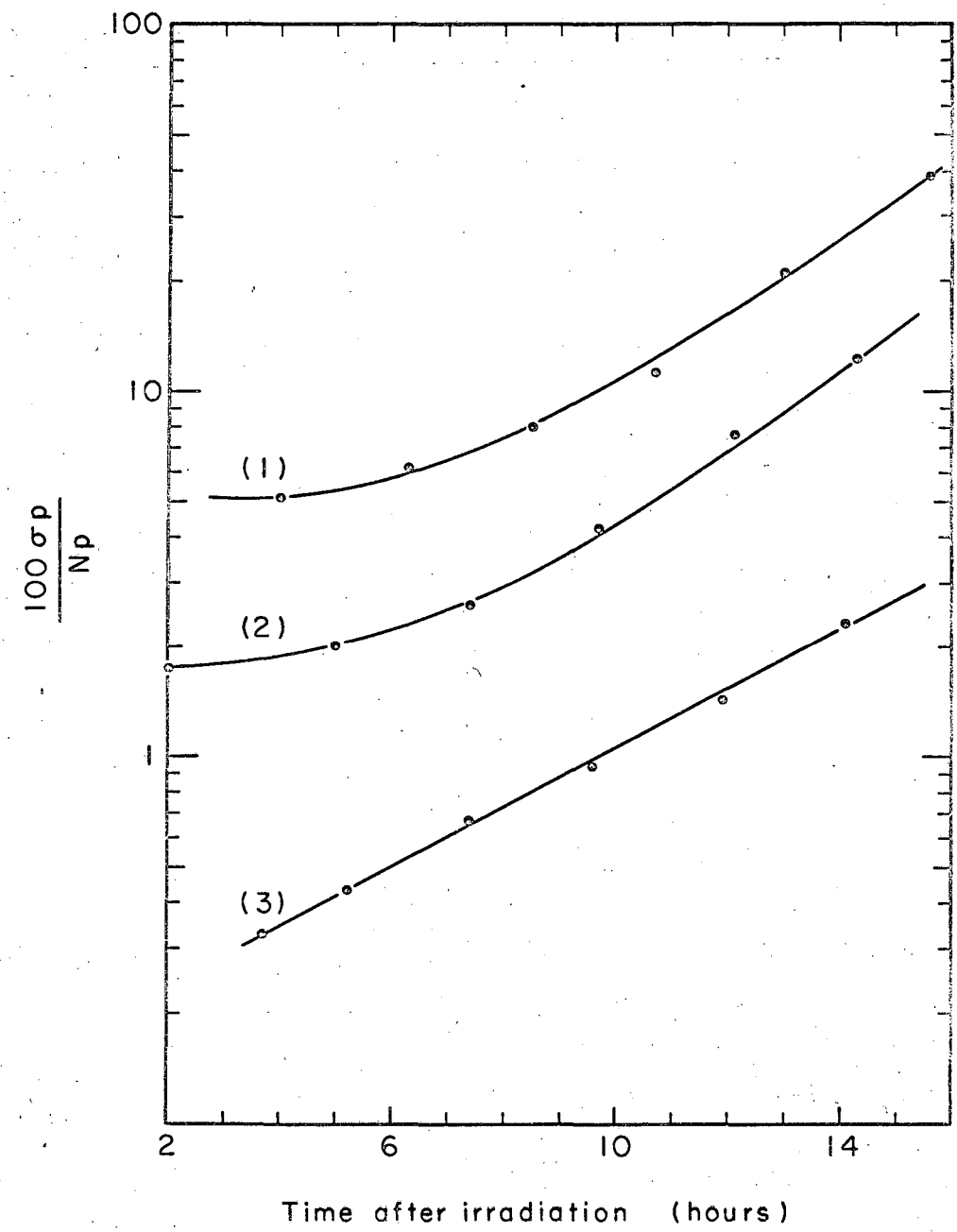

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Fig. 5