Experiments in Prompt γ-Ray Spectroscopy II

THERMAL NEUTRON RADIATIVE CAPTURE

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In Part I of this series[2], an experiment was described in which an isotopic neutron source can be used to demonstrate and measure the Doppler broadening of the $^{9}Be(\alpha,n\gamma)^{12}C$ reaction 4.43 MeV γ-ray. Continuing in this same vein, the neutron source can be used for further experiments in prompt γ-ray spectroscopy by utilizing the thermal neutron radiative capture process. As before, these experiments are also meant to introduce both the concept and the methodology of making on-line measurements to observe nuclear reactions while in progress. The measurement of prompt γ-rays from neutron capture is of continuing research interest for nuclear level structure studies (1). Although this in itself could be developed into experiments of considerable interest, the capabilities for elemental analysis is perhaps more apropos for a radiochemistry laboratory.

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[2]See J. Chem. Ed.

The isotopic (α,n) neutron sources have been used for many years in the radiochemistry laboratory for a variety of activation experiments [cf. Vorres(2)]. As a result, thermal neutron activation analysis has become the classic experiment to demonstrate radioanalytical methods. Several excellent experiments have been described (3-6). In this and a subsequent article, two non-activation radioanalytical methods using neutron sources will be described. The two methods are based on thermal neutron capture γ-ray and fast neutron inelastic scattering prompt γ-ray measurements. In the past decade considerable effort has been made to develop in situ and remote nuclear logging methods including the two afore mentioned methods. These efforts have been due mainly to interest in lunar and planetary exploration (7-16), marine and geologic studies (17-24), oil and mineral prospecting (23-37), and continuous analyzes of industrial process streams (38-39)[3]. A listing of the nuclear logging methods and their current status has recently been given by Sowerby (32). Techniques based on neutron irradiation followed by γ-ray spectroscopy appear to be the most general and useful for remote analysis (11).

Experiments in thermal neutron radiative capture and inelastic neutron scattering will therefore not only increase the student's breadth of radioanalytical methods beyond conventional activation analysis, but also introduce him to nuclear methods used for in situ and remote analyses.

Thermal Neutron Radiative Capture

When thermal neutrons are captured by a nucleus, the compound nucleus is left in a highly excited state, usually 6-10 MeV above the ground state. The lifetime of this excited state is typically a femto-

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[3] A more complete bibliography can be obtained from the many useful references given in two I.A.E.A. symposia (40, 41) and the theses of ~~[illegible]~~ A.A. ~~[illegible]~~ El Kady (1969) and P.F. Wiggins (1970) from the University of Maryland ~~[illegible]~~

second (10^{-15} sec) or shorter. The decay of the excited state is usually accompanied by a large cascade of γ-rays which returns the nucleus to the ground state. Frequently, the number of transitions is well over 100. As an example of a typical case, the de-excitation of the 7.916 MeV level in ^{64}Cu following thermal neutron capture in ^{63}Cu is shown in Figure 1. This level scheme shows only those transitions for which the intensity $100 I_\gamma > 1$, where I_γ is the fractional number of γ-rays per neutron capture. In addition to those shown in Figure 1, there are 52 other known transitions in ^{64}Cu [42]. These level schemes are well documented and have been compiled [42,43] for most stable nuclei with a reasonable natural isotopic abundance.

Because of the extremely short lifetimes of the excited states, measurements of these capture γ-rays must be made during the period of irradiation. Spectra from these <u>on-line</u> measurements will contain not only the radiative capture γ-rays, but also any decay γ-rays emitted by activation products and any prompt γ-rays from other neutron interactions (e.g. inelastic neutron scattering). Normally high resolution Ge(Li) detectors are required since the poor resolution of NaI(Tl) scintillators are prohibitive at the high energy and large spectral density. At these high energies (3-8 MeV) pair production is the major γ-ray interaction and the escape peaks are seldom resolved using NaI(Tl) crystals. Unfortunately, even large volume Ge(Li) crystals have very low detection efficiencies at these high energies. Therefore, either a high neutron flux is necessary or the <u>on-line</u> measurement time must be very long. The latter condition is the requisite with laboratory neutron sources.

Elemental Analysis by Neutron Capture γ-Rays

The analytical capability of thermal neutron capture γ-rays has been established (cf. Ref. 10, 12-14, 21, 24-30). As in conventional activation analysis, the γ-ray count rate R must be directly proportional to the elemental mass m

$$R = km \quad . \tag{1}$$

The count rate for a capture γ-ray of energy E_γ is given by

$$R = m \frac{N_o}{W} \epsilon_\gamma f I_\gamma \sigma_{th} \phi_{th} \quad , \tag{2}$$

where ϵ_γ is the overall detection efficiency for the γ-ray; f is the fractional isotopic abundance; m is the mass of the element; N_o is Avogadro number; W is the element atomic weight; I_γ is the fractional γ-ray intensity per capture; σ_{th} is the isotopic thermal neutron capture cross section; and ϕ_{th} is the thermal neutron flux. This neglects any attenuation or self-absorption of the γ-ray. Duffey, Senftle, et. al. (45-47) have compiled a table of sensitivity factors for the principal capture γ-rays of most elements. The sensitivity factors are essentially [4]

$$S = \frac{N_o}{W} f I_\gamma \sigma_{th} \quad . \tag{3}$$

These factors are useful in anticipating the most intense lines in a spectrum and in identifying unknown lines. As cautioned by the authors (45-47), spectral contrast of a particular line, i.e. the line intensity compared to intensities of adjacent lines, can be as important as the sensitivity factor in observing a line. This spectral contrast

[4] Their (45-47) sensitivity factors are listed as $S = I_\gamma \sigma / w$ where σ is the thermal neutron absorption cross section for the element. The value of σ is used instead of the product $f \sigma_{th}$ above.

with adjacent energy peaks can have the effect of either diminishing or enhancing the response expected from the sensitivity factor alone. This effect has been studied in some detail by Duffey, et. al. (24). In general, the elements which give the best spectral response to this enhancement are those which have only a few high energy transitions with large I_γ values and at least a moderate sized neutron absorption cross section. Elements for which these conditions are met include Mn, Co, Cu, Ni, Cl, Ti, Cr, Au, K, V, Ga, W, Se and Hg (24).

The analytical proportionality constant of equation 1, thus depends on only two experimental conditions,

$$k = S \epsilon_\gamma \phi_{th} \quad , \qquad (4)$$

the thermal neutron flux at the sample position, ϕ_{th}, and the total detection efficiency for the γ-ray, ϵ_γ.

Thermal neutron radiative capture analysis has a number of distinct advantages over conventional activation analysis. Unlike activation analysis, the method does not depend on producing a radioactive product with a suitable halflife. As an example, consider the activation of mercury. Data for thermal neutron capture in the Hg isotopes is presented in Table 1. Thermal neutron activation analysis is limited to the $^{196}Hg(n,\gamma)$ and the $^{202}Hg(n,\gamma)$ reactions which are hindered by a low abundance (0.146%) for the former and an unfavorable cross section (4.8 b) for the latter case. Inspection of Table 1 indicates that the $^{199}Hg(n,\gamma)$ reaction is the most favorable case, but it cannot be used for activation analysis since its product is stable. The radiative capture method does not depend on the induced activity and this reaction could be used. The use of this reaction with $f\sigma_{th} = 342$ makes the analysis nearly two orders of magnitude more favorable than the best case ($f\sigma_{th} = 4.38$) for the activation method.

A second advantage of the radiative capture method is that a constant γ-ray count rate is present until the source of neutrons is removed. Therefore, for a given neutron flux and detection efficiency, the total number of events detected can be increased by counting for longer periods of time. This is not possible with activation analysis since the radioactive sample continuously decays. The analysis is limited to the induced saturation activity. The radiative capture method also has the advantage that the high energy capture γ-rays will penetrate even bulk samples with little attenuation.

The disadvantages of the method are the higher background and lower detection efficiency for high energy capture γ-rays. The higher background is due to making the measurement during the irradiation. The spectrometer not only observes and records the capture γ-rays for the sample, but also any other capture, prompt or decay γ-rays produced by the neutrons in the surrounding environment. When using neutrons from an isotopic (α,n) source, the background will also contain the intense 4439 keV radiation from the $^{9}Be(\alpha,n\gamma)^{12}C$ reaction and the decay γ-rays from the source α-emitter (e.g. ^{239}Pu) and its daughters. Although less intense, fission γ-rays from these sources are also present in the background. A ^{252}Cf spontaneous fission neutron source has been used for radiative capture analysis and has a less troublesome background (12-14).

^{252}Cf current trend

Determination of Copper

The determination of the amount of copper in a U. S. 25-cent piece (quarter) provides an interesting example of the method. A U.S. Currency 25-cent piece (circa 1965) consists of a sandwich of a Cu core and a Cu-Ni alloy cladded to the core. Since the cladding material contains a fixed percentage of Cu, the weight percentage of Cu is nearly constant with wear and is 82.3% (~~49~~).

In conventional thermal neutron activation analysis, quantitative measurement of the ^{64}Cu radioactivity is troublesome. The difficulty arises since the 12.8 hr ^{64}Cu decays predominantly (38% β^-, 43% EC, 19% β^+) to the ground state of its daughters. In order to validly assay the beta radiation,

the absorption in the unknown sample must be known. This is nearly impossible, especially for thick samples of unknown constituency. This may be circumvented by using identical radiators with both reference and unknown samples and detect and measure the positron decay annihilation radiation. Extreme care must be exercised to insure that all of the positrons annihilate in the sample or radiator.

These problems are completely obviated by using a thermal neutron radiative capture method. Determination of Cu by this method is a favorable case for two reasons. First, the $^{63}Cu(n,\gamma)$ reaction cross section is of moderate size (4.5 b), and secondly two of the highest energy ^{64}Cu capture γ-rays have a reasonable intensity (see Figure 1). These transitions are:

E_γ = 7.916 MeV I_γ = ~~27.3~~ .273

E_γ = 7.637 MeV I_γ = ~~14.8~~ .148

Interference should be minimal since the intensity of ^{66}Cu capture γ-rays in this energy region is fairly low as well as for the γ-rays from nickel in the Cu-Ni alloy cladding.

Thus, the relative intensity of these γ-rays from the Cu in the 25-cent piece normalized to that in a reference Cu sample should provide a determination of Cu in the 25-cent piece.

Experimental

A detailed procedure is dependent on the equipment available, but the methodology is relatively the same. A description of the experimental apparatus and procedure used by our laboratory follows.

The experimental arrangement is shown in Figure 2. The neutron source which was described in Part I of this series was enclosed in several inches of paraffin to moderate the neutrons to thermal energies.[4] The thermal flux at the sample was $(4.1 \pm 0.8) \times 10^4$ n/cm^2-sec. measured by absolute counting of the neutron induced 54.0 min activity in indium foils using a (lower case) cadmium ratio method (52,53). The Ge(Li) spectrometer used to obtain the capture γ-ray spectra was identical to that described in Part I. Because of the relatively high flux of thermal neutrons at the detector position in the present configuration (Figure 2), the Ge(Li) detector was shielded with a 3.5 mm cadmium jacket.[5] A biased amplifier was used in the spectroscopy system to select regions of interest in the spectrum. Without the biased amplifier, the multichannel analyzer (MCA) would have to record and store the entire energy range from 0-8 MeV. By selecting a bias level, a smaller region can be obtained, e.g. 4-8 MeV. Thus, one can obtain a reasonable energy dispersion (keV/channel) without a large memory capacity MCA. A pulse generator is used to find the approximate energy region of interest and to make an approximate energy calibration. A description of this calibration procedure was previously described in Part I. A more accurate calibration could be obtained later by using the energies of known lines in the high energy spectrum.

"Unknown" 25-cent pieces of known weight were then simultaneously irradiated and counted for approximately 6-12 hours followed by 6-12 hours of irradiating and counting reference copper samples. The size of the

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[5] This was done to avoid neutron damage to the Ge crystal over prolonged periods of time.

[4] (50) (51)

samples used as copper standards was nearly identical to that of the 25-cent pieces. These samples consisted of a stack of three 2.5 cm diameter, approximately 1.8 g copper plachets.

The intensity of the ^{64}Cu γ-ray peaks in the spectra from the reference copper samples were then compared to those from the 25-cent piece spectrum to determine the amount of Cu in the 25-cent piece.

RESULTS and DISCUSSION

RESULTS and DISCUSSION

The high energy portion of the thermal neutron capture spectrum of the 25-cent piece is shown in the upper half of Figure 3. Directly beneath it is a background spectrum obtained in the same counting geometry, but without the 25-cent piece in the sample position. The full-energy (A and B), single-escape (A' and B') and double-escape (A'' and B'') peaks for both the 7.916 and 7.637 MeV transitions in ^{64}Cu are readily apparent in the 25-cent piece spectrum. As shown, however, the peaks (B, B' and B'') due to the 7.637 MeV transition are partially masked by the peaks from the $^{56}Fe(n,\gamma)$ reaction 7.646 MeV (C, C' and C'') and 7.632 MeV (D, D' and D'') doublet. These doublets, which are also present in the background, primarily arise from the iron in the stainless steel cryostat

of the Ge(Li) detector. The other structure in the background spectrum is due to neutron interactions with the detector and surrounding environment. Studies of neutron interaction with Ge(Li) detectors have been made by Rodda, et. al. (54) and Chasman, et. al. (55). Especially in lower energy regions, where the neutron reactions with the Ge crystal of the detector are evidenced[6], these studies are quite useful in identifying some of the background lines. In addition to the peaks due to the two major transitions in ^{69}Cu (A and B), several other peaks ascribible to low intensity transitions in nickel and copper are also present in the 25-cent piece spectrum. These lines

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[6] A more thorough discussion of these interactions with the Ge(Li) detector is made in Part III of this series.

are due to the 7.307 MeV transition in ^{69}Cu (H), the 7.814 MeV transition in ^{61}Ni (F), and the 8.525 MeV (E) and 8.996 MeV (G) transitions in ^{59}Ni. The single escape and double escape peaks for these transitions are labelled in Figure 3 by superscript primes and double primes, respectively.

The double escape peak from the 7.916 MeV transition in ^{69}Cu (A") is most favorable for the copper analysis since it is free of interfering lines and since the intensity of double escape peaks in this energy range is greater than that for the full energy or single escape peaks. Five copper standards ranging in mass from 1.8 to 9.1 g were used to establish the linearity of the 7.916 MeV transition double escape peak count rate as a function of the copper mass. The data from

these measurements are provided in Table II, and the resultant calibration line is shown in Figure 4. From the data in Table II, the weighted grand average for the average count rate per unit mass is 142.3 ± 3.1 cph/g. With a knowledge of the appropriate experimental quantities in equation (2), it should be possible to calculate ~~[illegible]~~ this value of R/m. The sensitivity factor[7], given by equation (3), for the 7.916 MeV γ-ray from the $^{63}Cu(n,\gamma)$ reaction is $S = 8.05 \times 10^{-3}$ cm²/g. Since the thermal neutron flux at the source position was 4.1×10^{4} neutrons/cm²-sec and the detection efficiency[8] for the double escape peak of the 7.916 MeV γ-ray is of the order of $1-2 \times 10^{-4}$, the expected count rate per unit mass is $R/m = S\epsilon\phi = 120-240$ cph/g. The approximate range of this result is

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3 footnotes prior page

7 The value of the sensitivity factor was obtained using $N_0/W = 0.958 \times 10^{-26}$ atoms/g, $f = 0.6909$, $I_\gamma = 0.273$, and $\sigma_{th} = 4.5$ b.

8 The value for the detection efficiency was estimated from curves for various sized Ge(Li) configurations given by Cline and Heath (56), Cappellani and Restelli (57), Arnell et. al. (58), Ewan et. al. (59) and Tavendale (60). It was obtained using either their values for intrinsic efficiencies corrected for the subtended solid angle or relative efficiencies normalized to our detector at low energies. It may be of value to note at this time that most efficiency calibration measurements at high energies (>3 MeV) are made using thermal neutron capture γ-rays. This is usually done by establishing the relative efficiency curves over the entire energy range using well known relative intensities of capture γ-rays from an appropriate element (that having many intense γ-rays over a wide energy range, e.g. ^{14}N). Absolute efficiencies are then obtained by normalizing these curves to lower energy data obtained with calibrated radioactive sources.

in good agreement with the experimentally observed value.

Table I

Thermal Neutron Capture in the Mercury Isotopes*

Target Isotope	Fractional Isotopic Abundance f	Thermal Neutron Capture Cross Section (barns) σ_{th}	Halflife of Radioactive Product	f σ_{th}
^{196}Hg	0.00146	~~2930.~~ 3000.	~~65~~ 69.1 hr	~~4.28~~ 4.38
	0.00146	~~125.~~ 120.	~~24~~ 23.8 hr	~~0.182~~ 0.175
^{198}Hg	0.1002	0.018	~~44~~ 43 min	0.0018
	0.1002	negligible	stable	-
^{199}Hg	0.1684	~~2500.~~ 2030.	stable	~~421.~~ 342.
^{200}Hg	0.2313	< 60	stable	< 13.9
^{201}Hg	0.1322	< 60	stable	< 7.95
^{202}Hg	0.2980	~~4.5~~ 4.8	~~47~~ 46.6 day	~~1.34~~ 1.43
^{204}Hg	0.0685	~~0.43~~ 0.4	~~5.2~~ 5.5 min	~~0.0294~~ 0.0266

*Data obtained from Chart of the Nuclides (48).

Table II

Calibration Data for Determination of Cu by Measurement of the 7.916 MeV Double Escape Peak from $^{63}Cu(n,\gamma)$ Reaction

Mass Copper (g)	(7.916-1.022) MeV Count Rate† (cph)	cph/g
1.8261	276 ± 22	151.1 ± 12.1
3.6378	544 ± 29	149.5 ± 8.0
5.4700	812 ± 35	148.5 ± 6.4
7.3343	998 ± 49	136.1 ± 6.7
9.1421	1255 ± 47	137.3 ± 5.2

weighted average: 142.3 ± 3.1

†Average count rates obtained after 13.889 hours.

Table III

Determination of Cu in Three U.S. 25-Cent Pieces

	No. 1	No. 2	No. 3
Copper Standard			
weight	5.4234 g	5.3985 g	5.4079 g
count time	11.300 hr	6.129 hr	6.569 hr
peak area	811 ± 43 cph	768 ± 51 cph	744 ± 46 cph
cph/g	149.5 ± 7.9	142.2 ± 9.4	137.5 ± 8.5
25-Cent Piece			
weight	5.5528 g	5.5863 g	5.5492 g
count time	15.340 hr	6.729 hr	8.433 hr
peak area	703 ± 23 cph	678 ± 43 cph	666 ± 41 cph
Results for Cu in 25-Cent Piece			
weight Cu	4.70 ± 0.29 g	4.76 ± 0.43 g	4.84 ± 0.42
% Cu	84.7 ± 5.3	85.3 ± 7.8	87.2 ± 7.6
Weighted Average		85.5 ± 0.5%	
"True"		82.3	

Figure Captions

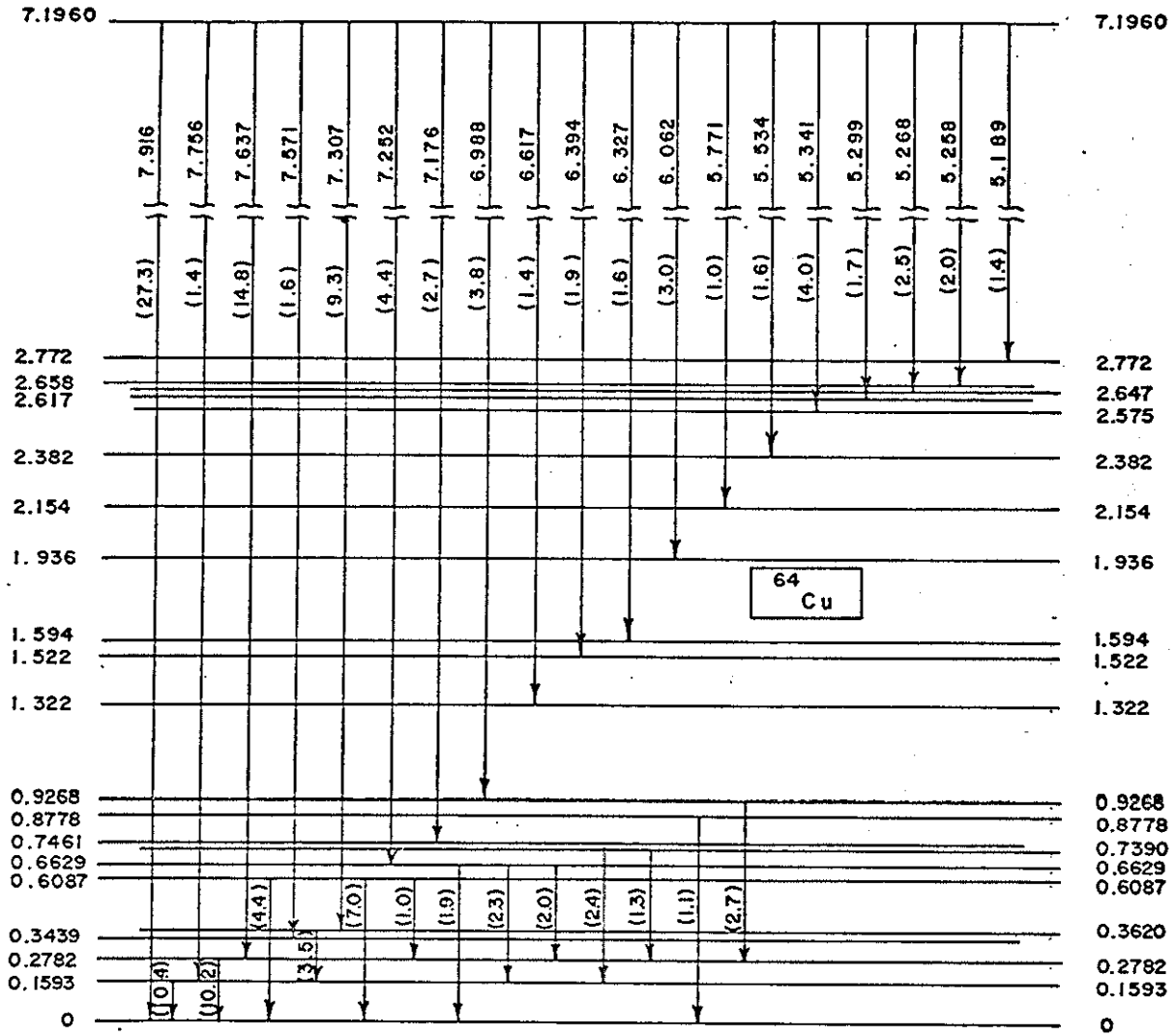
Figure 1. De-excitation of 7.916 MeV level in ^{64}Cu following thermal neutron capture in ^{63}Cu. Only transitions with intensities, given in parantheses, $100 I_\gamma > 1.0$ (see text) are shown. Adopted from Groshev and Bartholomew, et. al. (42).

Figure 2. Experimental arrangement for thermal neutron capture γ-ray measurements.

Figure 3. Typical thermal neutron capture γ-ray spectra. Upper: 25-cent piece showing 7.637 and 7.916 MeV transitions in ^{64}Cu; Lower: background showing interference from $^{56}Fe(n,\gamma)$ reaction 7.6 MeV doublet.

Figure 4. Calibration line for determination of Cu by measurement of the 7.916 MeV double escape peak [7.916-1.022 MeV]. The error bars on the data points representing the statistical uncertainty (one standard deviation) are in the range 4-8%. The slope of the line (weighted least squares fit) is 139.7±0.6 cpk/g.

Figure 3. Typical thermal neutron capture γ-ray spectra of 25-cent piece (upper) and background (lower). The labelled peaks are due to the following transitions: A, 7.916 MeV in ^{64}Cu; B, 7.637 MeV in ^{64}Cu; C, 7.646 MeV in ^{57}Fe; D, 7.636 MeV in ^{57}Fe; E, 8.525 MeV in ^{59}Ni; F, 7.814 MeV in ^{61}Ni; G, 8.996 MeV in ^{59}Ni; H, 7.307 MeV in ^{64}Cu. ~~Those~~ The ~~marked~~ peaks labelled with superscript primes and double primes correspond, respectively, to the single-escape and double-escape peaks for the transitions. See text for a discussion.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7.756
7.637
7.571
7.307
7.252
7.176
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6.617
6.394
6.327
6.062
5.771
5.534
5.341
5.299
5.268
5.258
5.189
(27.3)
(1.4)
(14.8)
(1.6)
(9.3)
(4.4)
(2.7)
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(2.0)
(1.4)
2.772
2.658
2.617
2.382
2.154
1.936
1.594
1.522
1.322
0.9268
0.8778
0.7461
0.6629
0.6087
0.3439
0.2782
0.1593
0
64
Cu
(4.4)
(7.0)
(1.0)
(1.9)
(2.3)
(2.0)
(2.4)
(1.3)
(1.1)
(2.7)
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(10.4)
(10.2)
7.1960
2.772
2.647
2.575
2.382
2.154
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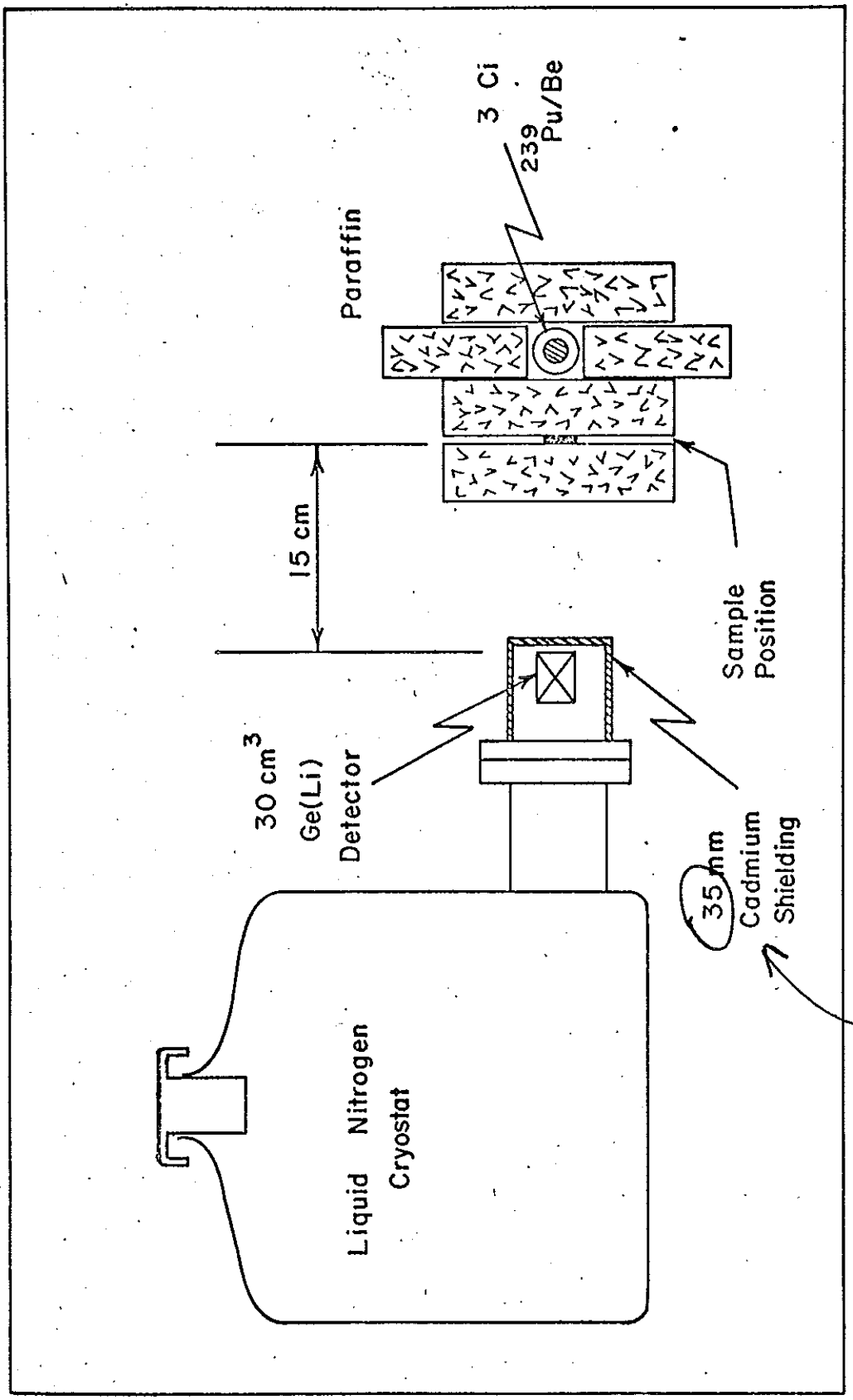
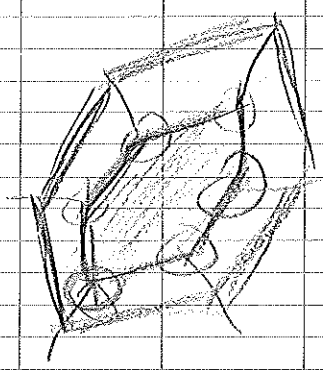

3 Ci
239 Pu/Be
Paraffin
15 cm
Sample Position
30 cm³ Ge(Li) Detector
35 mm Cadmium Shielding
3.5mm
Liquid Nitrogen Cryostat

where is 7117.02 ± 0.19 keV
^{16}O line 2

D''	7636 − 1022	6614	
B''	7637 − 1022	6615	
C''	7696 − 1022	6629	
F''	7814 − 1022	6792	
H'	7307 − 511	6796	
A''	7916 − 1022	6894	6894 + 124 = 7018
D'	7636 − 511	7125	
B'	7637 − 511	7126	
C'	7696 − 511	7135	
$F' + H$	7819 − 511	7303	
A'	7916 − 511	7405	
E''	8525 − 1022	~~8014~~ 7503	
D	7636	7636	
B	7637	7637	
C	7696		
F	7814		
A	7916		
G''	8996 − 1022	8985	
E'	8525 − 511	8014	



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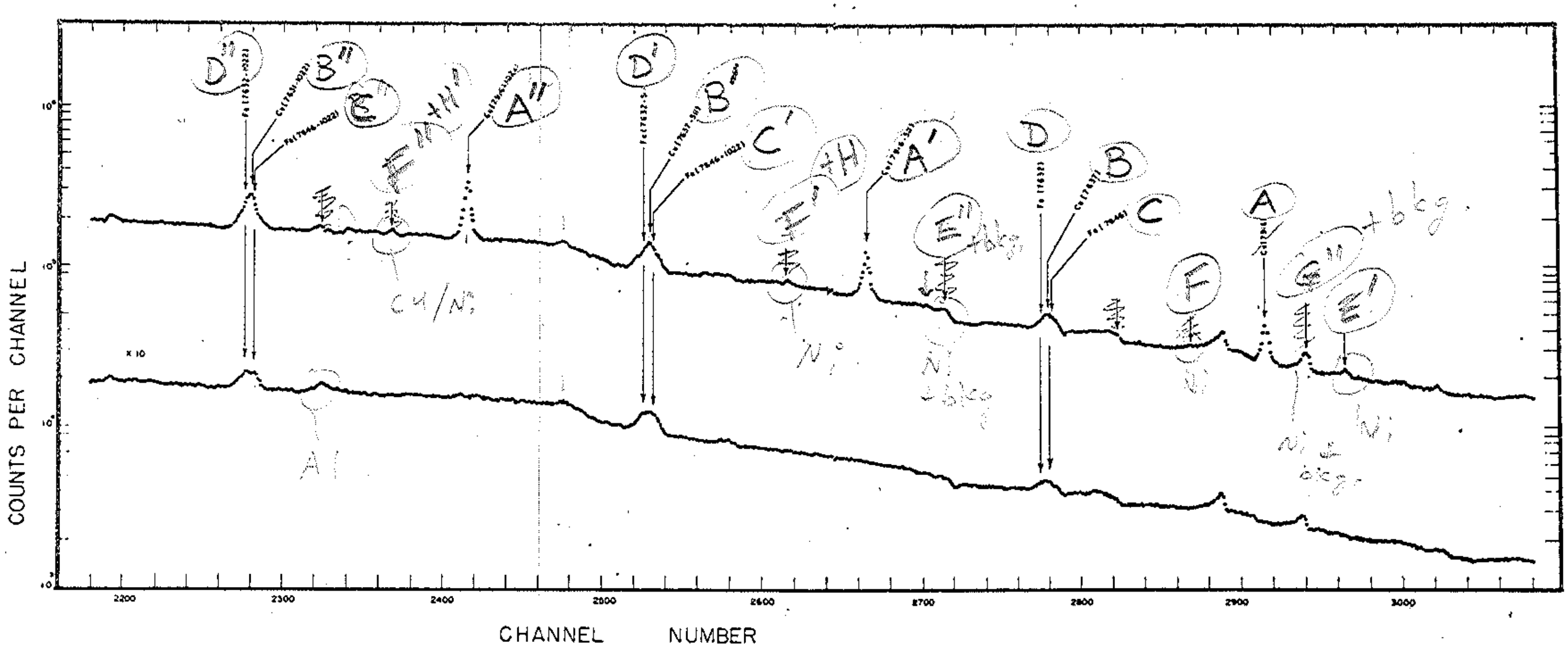
COUNTS PER CHANNEL
CHANNEL NUMBER
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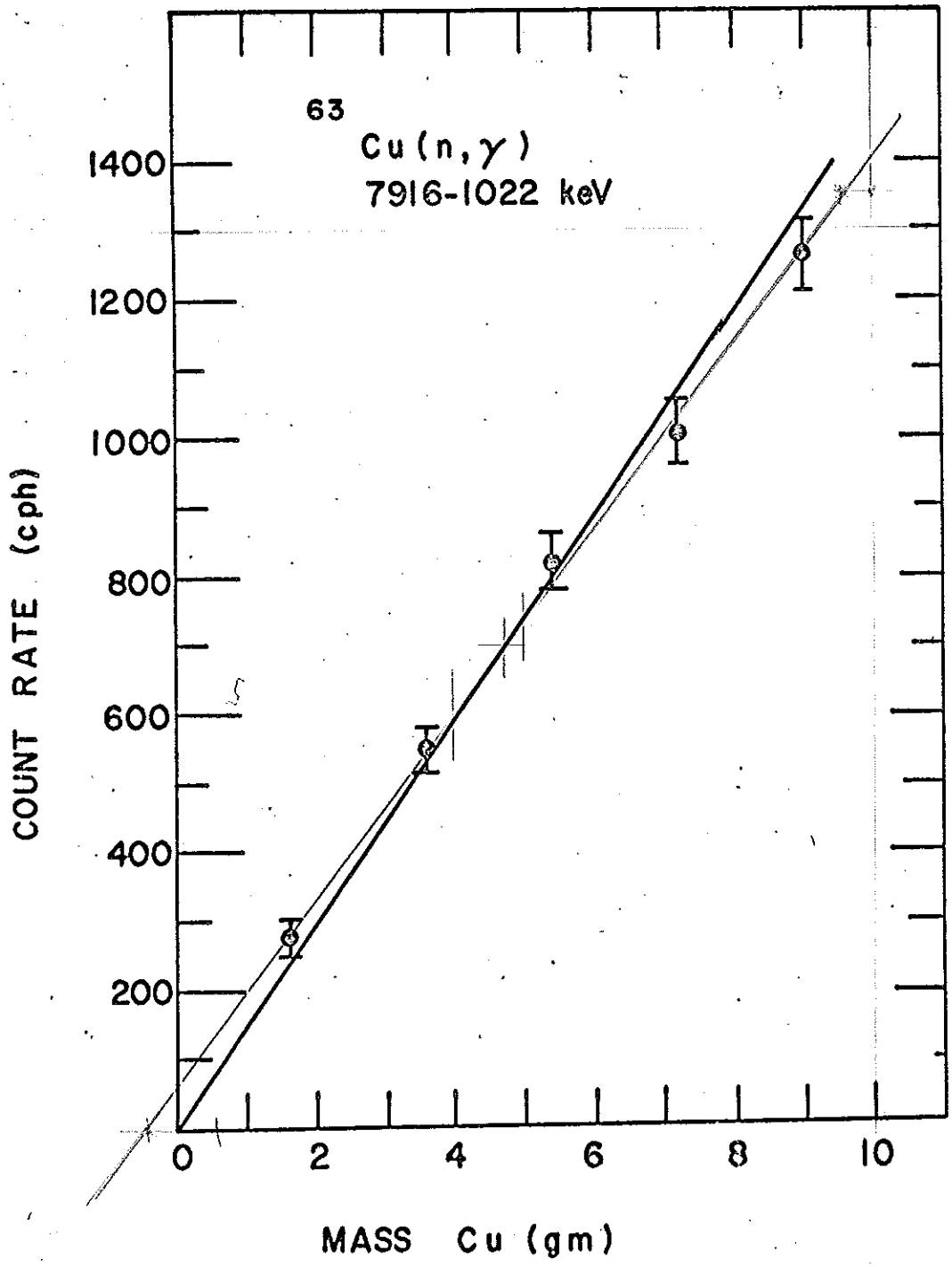
2400
2500
2600
2700
2800
2900
3000
x 10
D''
B''
E''
F''+H''
A''
D'
B'
C'
F'+H
A'
E'' +bkg
D
B
C
F
A
G'' +bkg.
E'
CU/Ni
Ni
Al
Ni & bkg



weighted least squares fit: slope = 139.7 ± 5.9 cph/g
intercept = −40.8 ± 27.7 g

weighted ave. of 5 pts = 142.3 ± 3.1 cph/g

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