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THE CONSTRUCTION OF A MULTI-THRESHOLD FOIL
FOR FAST FLUX MEASUREMENTS

by

Michael Henry Holkenbrink

A

THESIS

submitted to the faculty of the
UNIVERSITY OF MISSOURI AT ROLLA

in partial fulfillment of the requirements for the

Degree of

MASTER OF SCIENCE IN PHYSICS

Rolla, Missouri

1966

Approved by

Dr Edward (Advisor)

Albert E. Bolton

J. H. Miller
Blair

ABSTRACT

The construction of a multi-threshold foil for fast neutron flux measurements is investigated. The factors governing the selection of the materials are the energy response of the given reactor, the half-life of the daughter, the prominent gamma ray emitted during decays, the power level of the reactor, the availability of adequate cross section data, and the alloying properties of the composite foil. A ternary system was chosen as a compromise between the difficulty of fabrication and the amount of information gained from one foil. The particular system chosen was In-P-Fe. The threshold reactions of these elements are $\text{In}^{115}(\text{n},\text{n}') \text{In}^{115\text{m}}$ at 0.40 Mev, $\text{P}^{31}(\text{n},\text{p}) \text{Si}^{31}$ at 2.40 Mev, $\text{Fe}^{54}(\text{n},\text{p}) \text{Mn}^{54}$ at 4.30 Mev, and $\text{Fe}^{56}(\text{n},\text{p}) \text{Mn}^{56}$ at 5.00 Mev. This foil is used to illustrate the feasibility of the construction of a multi-threshold foil by using it to make preliminary measurements of the fast flux.

Acknowledgement

The author wishes to express his appreciation to Dr. D. R. Edwards, Director of the Nuclear Reactor, for his valuable assistance and guidance throughout this very interesting investigation.

He also wishes to extend his gratitude to the staff of the Nuclear Reactor for their assistance and suggestions.

This work has been performed under National Science Foundation Grant GP-3050.

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I. Introduction

A. Problem

Today with the growing need to monitor the neutron environment for health, shielding, and other types of experiments, there is increased research into the different methods of gaining this information. Some of these methods are semiconductor spectrometers, photographic emulsions, threshold foils, and proton recoil methods. For the measurement of the neutron flux within the core of the reactor, however, a method is needed which has a selective energy response, is simple to use, requires very little time for analysis, and is not influenced by the gamma flux. The threshold foil satisfies most of these criteria.

The foil is usually a thin piece of metal or powder encased in plastic or similar material. "A threshold foil is further defined as a material which only has a response to a flux of neutrons having energies above a specific value, or threshold, which is characteristic of the material in question." (1) The reaction threshold is actually not defined as the lowest energy for the occurrence of the specific reaction but the energy for which the reaction is one one-hundredth of its maximum value (2,3).

The value of the cross section as it varies with the energy is of extreme importance. These values are extensively tabulated in the literature even though the values usually differ. It will be seen that most sources of cross section data used for this thesis are fairly recent, to take advantage of the work being done to correct discrepancies in this data.

Upon exposure to a neutron flux, the threshold detector nuclei undergo a reaction, for instance (n,p) , and form a radioactive

daughter or daughters. The daughter then decays, at which time a gamma ray is usually emitted. The number of gamma rays is directly proportional to the number of nuclei which underwent the reaction. This gamma activity is then proportional to the cross section, the neutron flux, and the number of parent atoms. The threshold detector can, therefore, be used to determine the average flux over a particular energy range. This flux can be used to index the normalized fission spectrum, such as that of Watt or Cranberg, so as to indicate the neutron flux at any neutron energy.

This method, however, has many sources for error. The non-reliability of the cross section data is one of these. Another is the error inherent in the assumption that the neutron spectrum is the fission spectrum. Work is presently being done by K. Cage here at the University of Missouri at Rolla on minimizing this source of error. The overall error may be minimized by the use of another threshold foil of a different energy response and averaging the two indexed spectra thus obtained.

The problem then encountered in the use of more energy reactions is that the increased number of foils increases the irradiation time and the counting time. This, however, may be simplified by the use of a single foil which has a number of threshold energy reactions. Thus comes the need for the development of a multi-threshold foil.

This thesis, therefore, is a description of the investigation for the reactions necessary for this type of foil. Also the construction details and difficulties are explored.

B. Method of Attack

Before the construction of a multi-threshold foil could be undertaken, a list of the specifications of the foil material was formulated based on the following criteria:

1. Thresholds. The effective threshold of the materials used would have to be above .1 Mev or sensitive to fast neutrons only. This was necessary because there exist many reactions that occur at thermal energies or energies not associated with fast neutrons and introduce unwanted complications.
2. Cross Section. The available data on the cross section should be fairly recent. The energy dependence of the cross section should indicate a sharp increase at energies just above the threshold energy, preferably similar to a step function.
3. Half-life. The half-life of the daughter nucleus should be of the order of hours to days. This allows the foil to be reused in the immediate future so as to help keep the cost per flux measurement to a minimum. In addition, with a half-life of this order good activation will be attained in reasonable activation times.
4. Gamma Spectrum. The daughter should decay so as to produce a prominent gamma peak which is easy to recognize. The prominent peak will be used not only to gain knowledge of the activation of the material, but also to yield an observation point utilized in the stripping (separation) of the overlapping spectrum of the materials used in the multi-threshold foil.
5. Cost and Purity. The cost of material used, of a purity necessary to yield clear data with little unseparable interference, must be reasonably low.

6. Construction. The materials used in the foil must be of a nature such that they will combine to form an alloy or a compound which may be either encased in some suitable material or formed into a solid foil. A salt might be used where the constituents of the salt have thresholds which are usable.

II Preliminary Investigations

A. Literature Survey

There has been little work done along any direction in the design of a foil with the characteristics described in Chapter I. However, there has been work done by William Gerken (4) using alloys currently available on a commercial basis. There has also been some work done using MgSO_4 as a double threshold detector (5). The work of W. Gerken is the only one of the two papers which has well tabulated results. This work, however, was hampered by a number of factors including impurities, long half-life, and the inability to choose the energy threshold — for instance, the use of inconel with the reactions $\text{Fe}^{54}(\text{n,p})$ Mn^{54} , $\text{Fe}^{56}(\text{n,p})$ Mn^{56} , and $\text{Ni}^{58}(\text{n,p})$ Co^{58} with the respective energy thresholds, $E_{\text{eff}} = 4.3, 7.2, \text{ and } 2.9 \text{ Mev.}$

Pertinent information for reactions with interesting thresholds (1-17) has been tabulated in the literature; this tabulation is in Table I. This data represents the latest references which are in agreement. The inconsistencies in earlier data have been cleared up for the most part. To obtain as consistent a set of data as possible, the data from each source was weighed according to consistency and publication date. The latter is particularly important where major discrepancies exist.

An investigation was then undertaken to determine feasible methods of combining the materials. The books by Hansen (18) and Elliott (19) on binary systems were found to be excellent for metallic alloys. The resultant foil will not necessarily be an alloy but could be a compound; this compound would probably be in the form of a powder. The metallurgical engineering and ceramics engineering departments here at the

University of Missouri at Rolla were consulted on the preparation of a foil from a powder. The work by N. Frigerio (20) was also consulted. In this work some different methods of encasing the powder in some form of teflon on plastic are thoroughly discussed. This concept has the inherent disadvantage that the encasing material may become radioactive causing a high background count and may suffer radiation damage more severely than a sintered foil.

B. Initial Studies

In Table I the effective energy thresholds and the average cross sections are stated. These were examined through actual irradiation to find which elements best meet the necessary requirements set by irradiation time, 1 hour or less, and power level, 10 kw. At least three reactions were desired with these reactions in each of three different energy ranges — 0.1 to 1.0 Mev, 1.0 to 3.0 Mev, and 3.0 to 7.0 Mev. The half-life of the daughter product should not be in excess of about 50 hours so the foil will have a high reusability rate and a high activity after irradiation time.

1. 0.1 to 1.0 Mev

a. Indium. Indium appears to be one of the best materials for use within this energy range. The (n, n') reaction has a high cross section and a 4.4 hour half-life. However, indium does have a resonance reaction that has an extremely high cross section for neutrons in the epithermal range. Fortunately, this latter reaction has a half-life of only 54 minutes and after 9 hours (10 half-lives) a negligible amount remains. One other detrimental factor concerning indium is that it does not alloy readily with many other metals and even when it does the indium percentage is small.

b. Silver. Silver has a low threshold and has the advantage that it is easy to alloy. However, the activation cross section is small as can be seen in Table I. After irradiation no gamma peak identifying a daughter product could be found. Thus silver is not usable under the present conditions.

2. 1.0 to 3.0 Mev

a. Nickel. Nickel is used extensively as a fast flux monitor but due to the long half-life, 71 days, of the daughter Co^{58} it is slow to activate and requires long counting times. It does, however, alloy easily and has a prominent gamma peak. Time must be allowed for the 9 hour half-life Co^{58m} to decay.

b. Phosphorus. The (n, p) reaction of phosphorus producing Si^{31} is promising. Phosphorus alloys easily and the prominent gamma peak is at 1.26 Mev. The magnitude of the cross section is small but preliminary irradiation indicated it to be adequate. Phosphorus requires about the same irradiation time as nickel.

c. Aluminum. The main problem with the aluminum (n, p) reaction is that the half-life is only 9.5 minutes. Irradiation also produces a quantity of Na^{24} by the (n, α) reaction which has a substantial half-life of about 15 hours. The threshold for the Na^{24} reaction is 5.9 Mev so there would be two reactions yielded with one irradiation. However, the short half-life weighs heavily against the use of this (n, p) reaction.

d. Other. There are other materials with an energy response within this energy range. However, not enough cross section data is available, or like the sulphur reaction, $\text{S}^{31}(\text{n}, \text{p}) \text{P}^{31}$, the daughter is a pure β emitter. Another threshold reaction within this range is fission.

For example the U^{238} fission reaction has a threshold of about 1.5 Mev

(4). Fissions reactions have been excluded due to the difficulty in separating their gamma spectrum from that of other possible constituents.

3. 3.0 to 7.0 Mev

a. Iron. Again, as in the case of aluminum two reactions, $Fe^{56}(n,p)Mn^{56}$ and $Fe^{54}(n,p)Mn^{54}$ exist. The higher energy threshold reaction, $Fe^{56}(n,p)Mn^{56}$, is quite productive while the other reaction has a long half-life and as a result is of smaller production. The yield of the long half-life product, Mn^{54} , is not great and the counting time is necessarily long to achieve any reasonable statistics. However, it is simple procedure to take a background count before any irradiation of the material so the foil can be reused. This element is very interesting because of its separable double energy response and its easy alloying characteristics.

b. Magnesium. Magnesium more than any other element appears to meet the criteria listed earlier. The half-life is not excessively long and the cross section appears to be sufficient under irradiation. Unfortunately, it is difficult to alloy.

c. Other. There is not enough data tabulated on any other material to make it of any use as a constituent of this foil.

The activations previously completed indicate that indium is the only available constituent for this foil in the lowest energy range. The range from 1.0 to 3.0 Mev has two good possibilities, nickel and phosphorus, as has the highest energy range iron and magnesium. The following are the possible combinations:

In Ni Fe

In Ni Mg

In P Fe

In P Mg

After examining Hansen (18) and Elliott (19) as to the possible binary systems — InNi, InP, InFe, InMg, PFe, PMg, NiMg, NiFe — it was apparent that the use of indium presents a great alloying problem. Indium will not alloy with any of the possible constituents to any more than a few percent. Another approach, the search for a chemical system proved futile. The search therefore returned to that of an alloy.

On expanding the alloy to a ternary, (21, 22) the possible combinations were reduced to one, InPFe. This being the only one on which a ternary system could be found in the literature. Nothing was found for the other three systems. The most probable system was thus narrowed to InPFe.

<u>Reaction</u>	<u>E_{eff} (Mev)</u>	<u>$\bar{\sigma}_f$ (mb)</u>	<u>Half-life</u>	<u>Reference</u>	<u>Gamma Peak</u>
In ¹¹⁵ (n,n') In ^{115m}	.45	170.0	4.5 hours	12	.35
Ag ¹⁰⁹ (n,p) Pd ¹⁰⁹	0.4	2.0	13.6 hours	12	.087
Ni ⁵⁸ (n,p) Co ⁵⁸	1.2	104.0	72 days	2, 3, 4	.81
P ³¹ (n,p) Si ³¹	2.4	30.6	2.6 hours	4	1.26
Al ²⁷ (n,p) Mg ²⁷	2.7	3.35	9.5 minutes	2, 3, 4	.83
Al ²⁷ (n, α) Na ²⁴	5.9	.57	15 hours	2, 3, 4	2.75 1.38
Fe ⁵⁶ (n,p) Mn ⁵⁶	5.0	.92	2.6 hours	2, 3, 4	.84
Fe ⁵⁴ (n,p) Mn ⁵⁴	4.3	49.0	291 days	4	.84
Mg ²⁴ (n,p) Na ²⁴	6.1	1.25	15 hours	2, 3, 4	2.75 1.38

Table I

Reactions

III Experimental Results

A. Experimental Procedure and Foil Composition

All irradiations were performed in cadmium containers to eliminate thermal neutron activation. The fast neutron activation gamma spectrum of the individual materials and of the ternary were then examined using a multi-channel analyzer.* The spectrum indicated the following peaks with little overshadowing of any peak by that of another material:

In - 0.35 Mev

Fe - 0.84 Mev

P - 1.26 Mev.

This meant, that as an alloy, the three spectrum peaks would be visible. As a further check upon the composite spectrum, a binary compound, a mixture of two chemical compounds, containing iron and phosphorus was irradiated. This irradiated sample when counted clearly indicated the two prominent gamma peaks showing little interference from each other. It was not deemed necessary to irradiate a sample containing indium as considerable time could be allowed before counting. That is, due to the longer half-life of $\text{In}^{115\text{m}}$, a length of time sufficient to allow complete decay of the iron and phosphorus daughters could pass before counting the sample to obtain the indium activation.

The irradiation of the binary containing the iron and phosphorus compounds also gave information concerning the relative size of the two cross sections. This was done because the values listed in Table I for the average cross sections of iron and phosphorus differ considerably.

* The multi-channel analyzer and the reactor facility are described in Appendix I and the calibration of the analyzer is in Appendix II.

However, during irradiation where a number of items--flux spectrum, half-life, etc.--are taken into account, the resultant activities were close to identical. The irradiated binary, made up of equal weights of iron and phosphorus, clearly indicated this equivalence in activities. This showed that the percentages of iron and phosphorus should be about identical in the multi-threshold foil. An indium foil was then irradiated to determine how its activation level compared to that of iron and phosphorus. The counted indium foil had an activity one-hundred fold that of the iron and phosphorus activities. The activation is proportional to the number of atoms present and therefore, the amount of indium present was determined to be about one one-hundredth that of the iron and phosphorus. The multi-threshold foil was then specified to have the following percentage concentration:

49% P

50% Fe

1% In.

The iron has a slightly larger percentage due to the alloying properties of the ternary system.

B. Construction

Because phosphorus has a high affinity for water and oxygen, the Metallurgy Department suggested the following procedure for the alloying process:

1. Sealing the material mixture in a quartz tube.
2. Heating in the oven, taking caution not to break the quartz tube.
3. Removing the resultant material which will probably be in the form of a powder and hot pressing it to sinter it. The sintering process would produce a solid material with the characteristics of a solid metal foil.

This procedure was not followed, however, due to lack of the proper equipment. A search was, therefore, begun for a commercial company with the capabilities to manufacture the desired foil. Cerac (25) was the company which was finally commissioned to do this. Their method of manufacturing differed only in that they proposed to alloy in an oven under an inert atmosphere. They did have adequate sintering facilities to press the foil. Therefore, there was manufactured one pound of the alloy powder and 16 pressed foils. The foils are one inch in diameter and one-eighth of an inch thick. The percentages of materials present are as follows:

1.0% In
43.8% P
55.2% Fe.

C. Result

After delivery, the foils were irradiated to provide a fast flux spectrum. This spectrum is seen in Graph I. The prominent indium, iron, and phosphorus peaks are easily visible. On examining Graph II, however, which is another spectrum of the same foil taken only a hour earlier, the indium peak is very obscure due to overlapping of one of the other spectra. It is, therefore, seen that the time at which the foil is counted is very important. The optimum counting time was found to be at ten hours after irradiation. One of the major reasons for waiting this long is the fact that the indium has its own interfering reaction. As mentioned in Chapter II, this reaction has very little remaining activity after nine hours.

The value obtained for the indium peak activity is not the true activity. The low energy gamma ray emitted by indium is very easily shielded, and this occurs in the foil itself. This self-shielding is discussed in Appendix III.

In Graph III we see the activity of the foil 54 hours after irradiation. This graph shows the activity due to the Mn^{54} . Due to its lower activity, this activity may be observed using a longer counting time than is used to count the foil at the ten hour point. At least 26 hours must be allowed before this activity is counted to allow the Mn^{56} activity to decay beyond the point where it might interfere.

Samples of the materials used to manufacture the foil were also irradiated. These spectra are illustrated in Graphs IV, V and VI.

The magnitude of the spectrum was used in conjunction with the average cross sections listed in Table I to obtain values for the average fast flux. Normally this will be done by separating the individual spectra from the composite using a computer program and then calculating the flux over the energy range of the material using an energy dependent cross section curve and a computer program. These computer programs are currently being developed here by Dr. D. R. Edwards of the reactor facility.

The magnitude of the counts obtained was a minimum of 25,000 over a 30 minute period. This large number of counts should give good statistical results. The values obtained for the fast flux were as follows according to the yielding reaction:

$$In^{115}(n,n') \rightarrow In^{115m} - 4.07 \times 10^9 \text{ n/cm}^2/\text{sec}$$

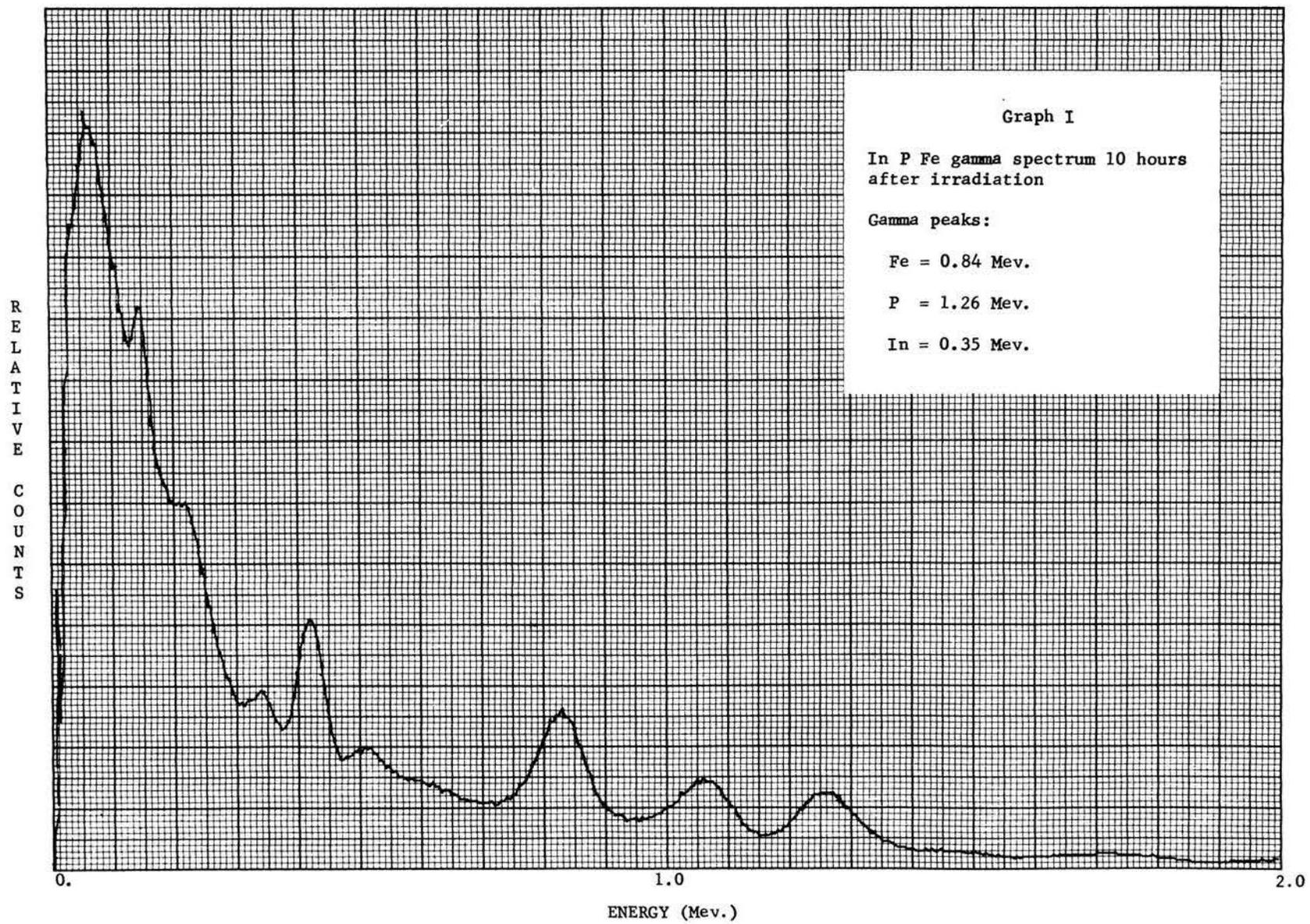
$$Fe^{56}(n,p) \rightarrow Mn^{56} - 6.15 \times 10^9 \text{ n/cm}^2/\text{sec}$$

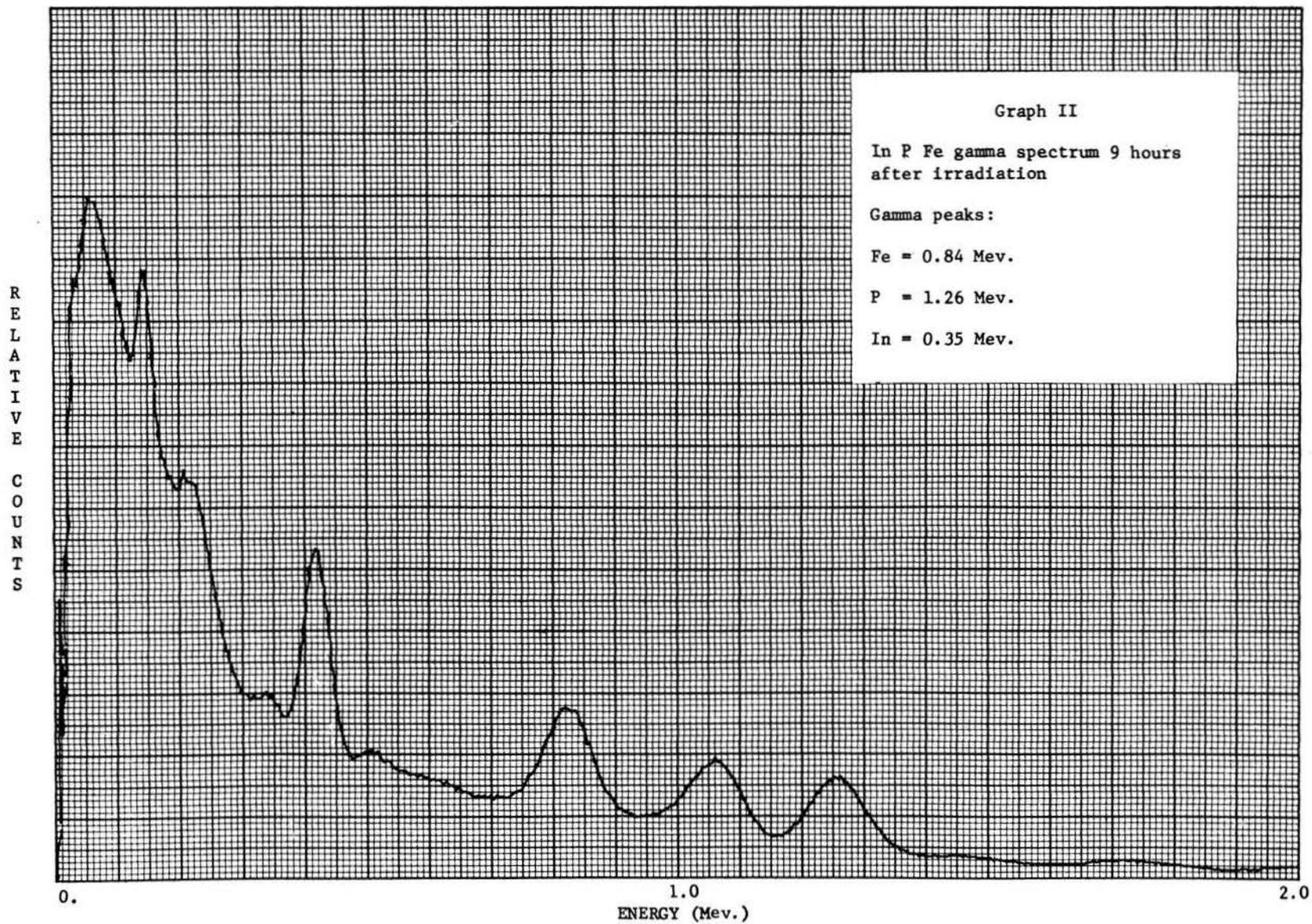
$$P^{31}(n,p) \rightarrow Si^{31} - 2.47 \times 10^9 \text{ n/cm}^2/\text{sec}.$$

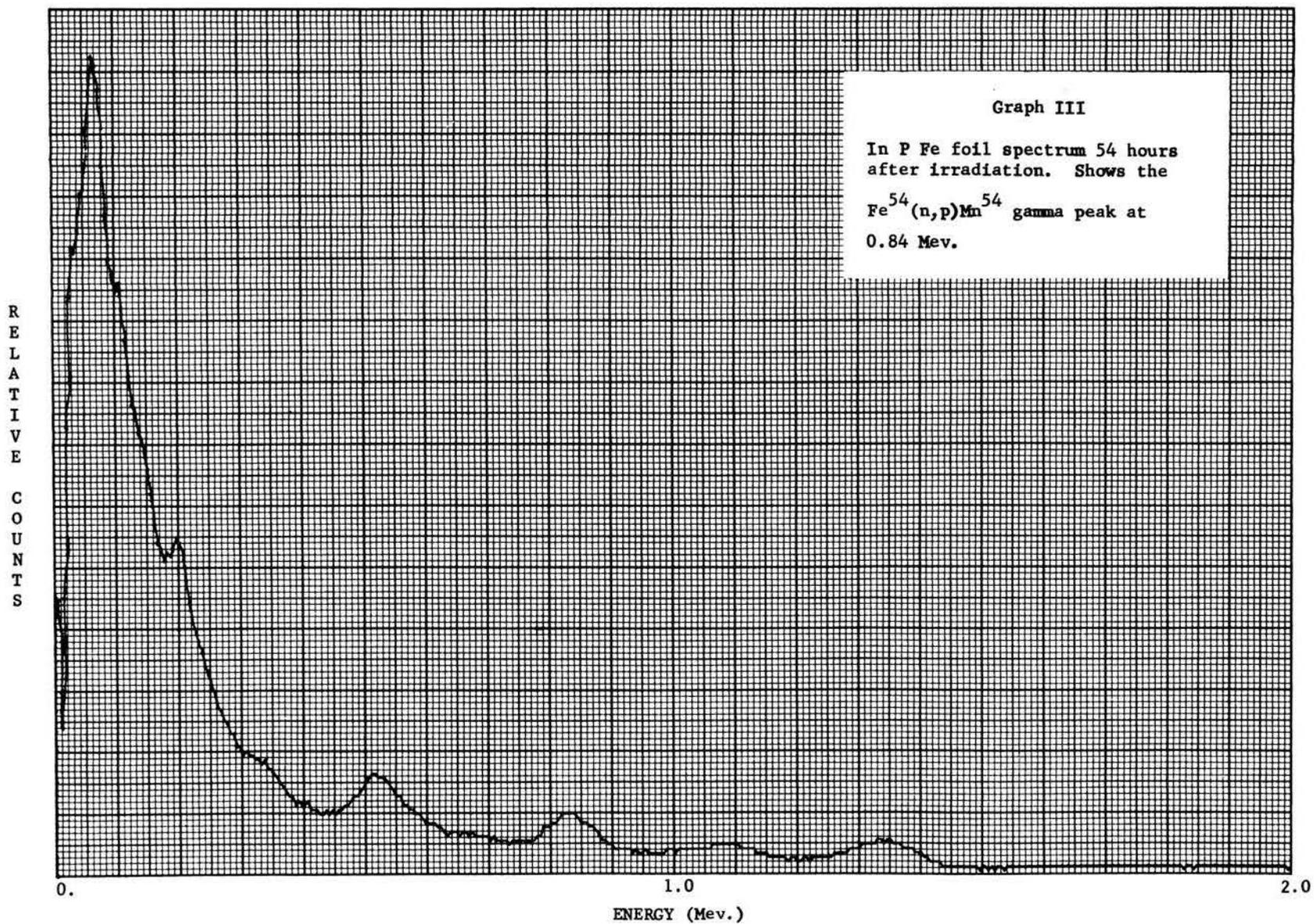
An example of the involved calculations is given in Appendix IV. The result from the Fe^{54} reaction was not computed due to the lack of the spectrum separating program which was needed to separate the Mn^{54} and In^{115m} reactions.

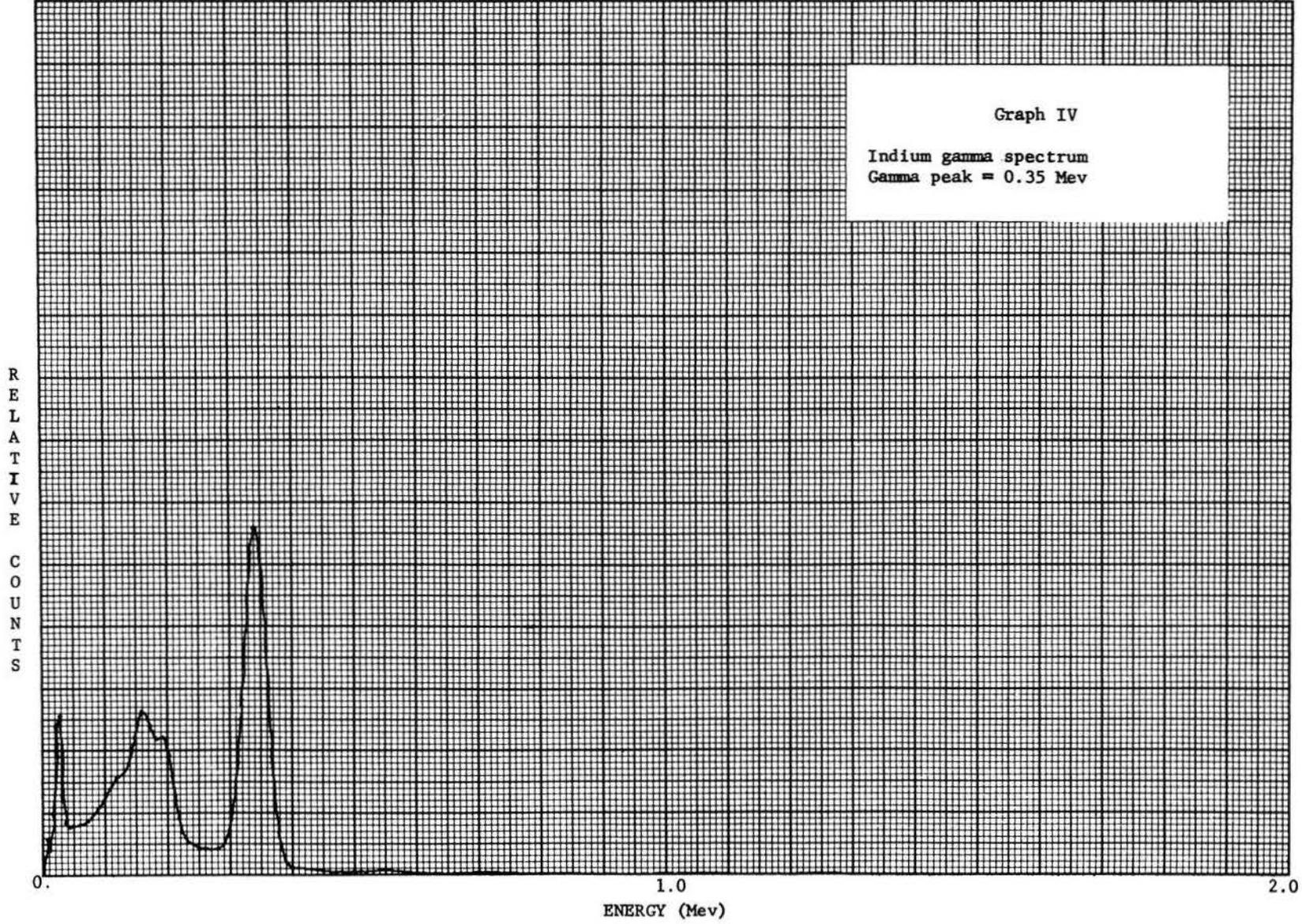
The above calculation has many inherent errors and hence the value found for the fast flux is only approximate. The background activity at a peak from another peak is not accounted for. The calculation is also based upon peak maximum count values rather than the integral number of counts under a peak. The exact composition of the foil is still slightly in doubt as this cannot be checked by activation methods until the spectrum separating program is completed.

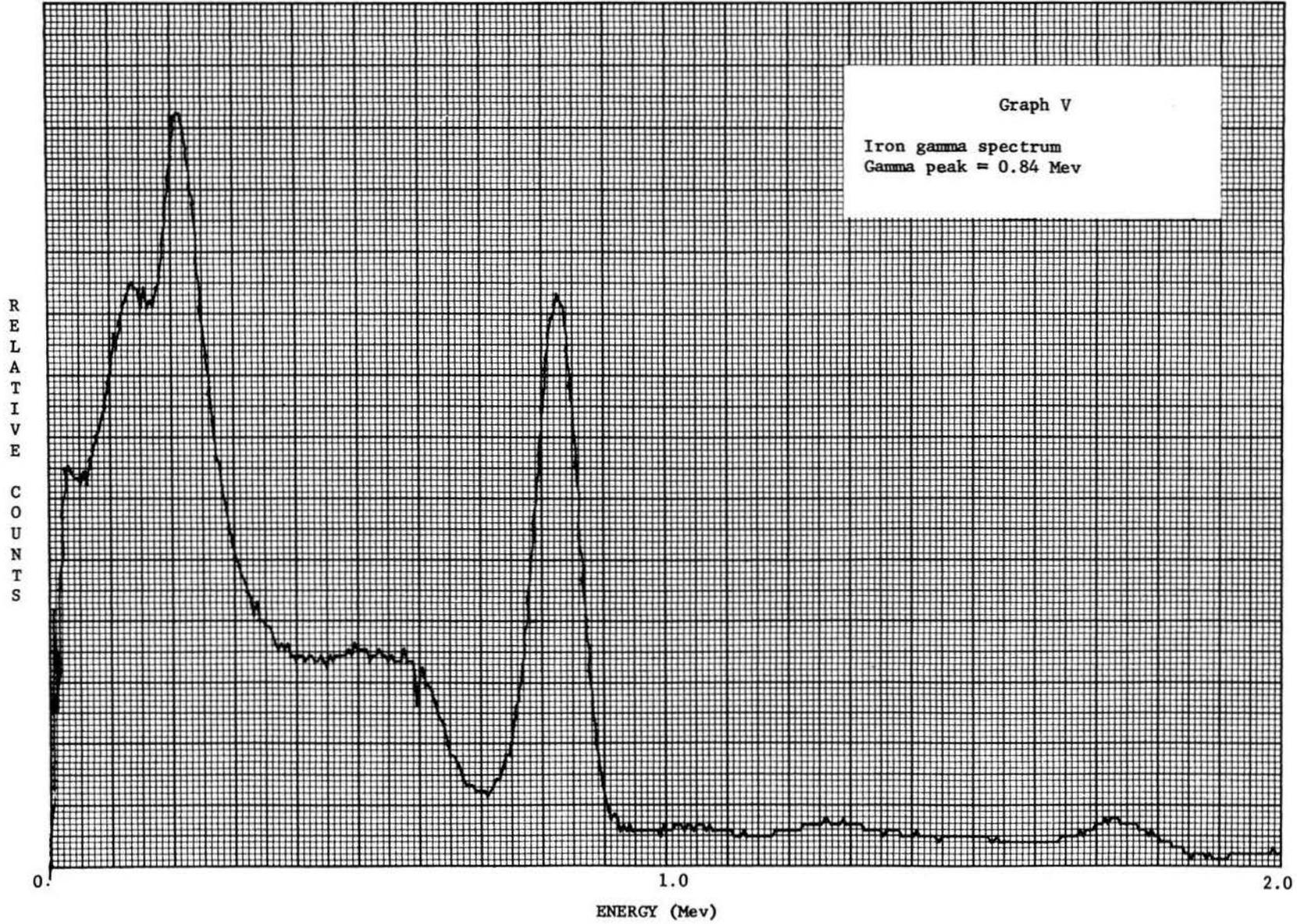
One major factor that was taken into account for the above measurement and those like it is the calibration of the analyzer. This is both for energy per channel and the overall efficiency of the analyzer. This calibration was done and is included in Appendix II.





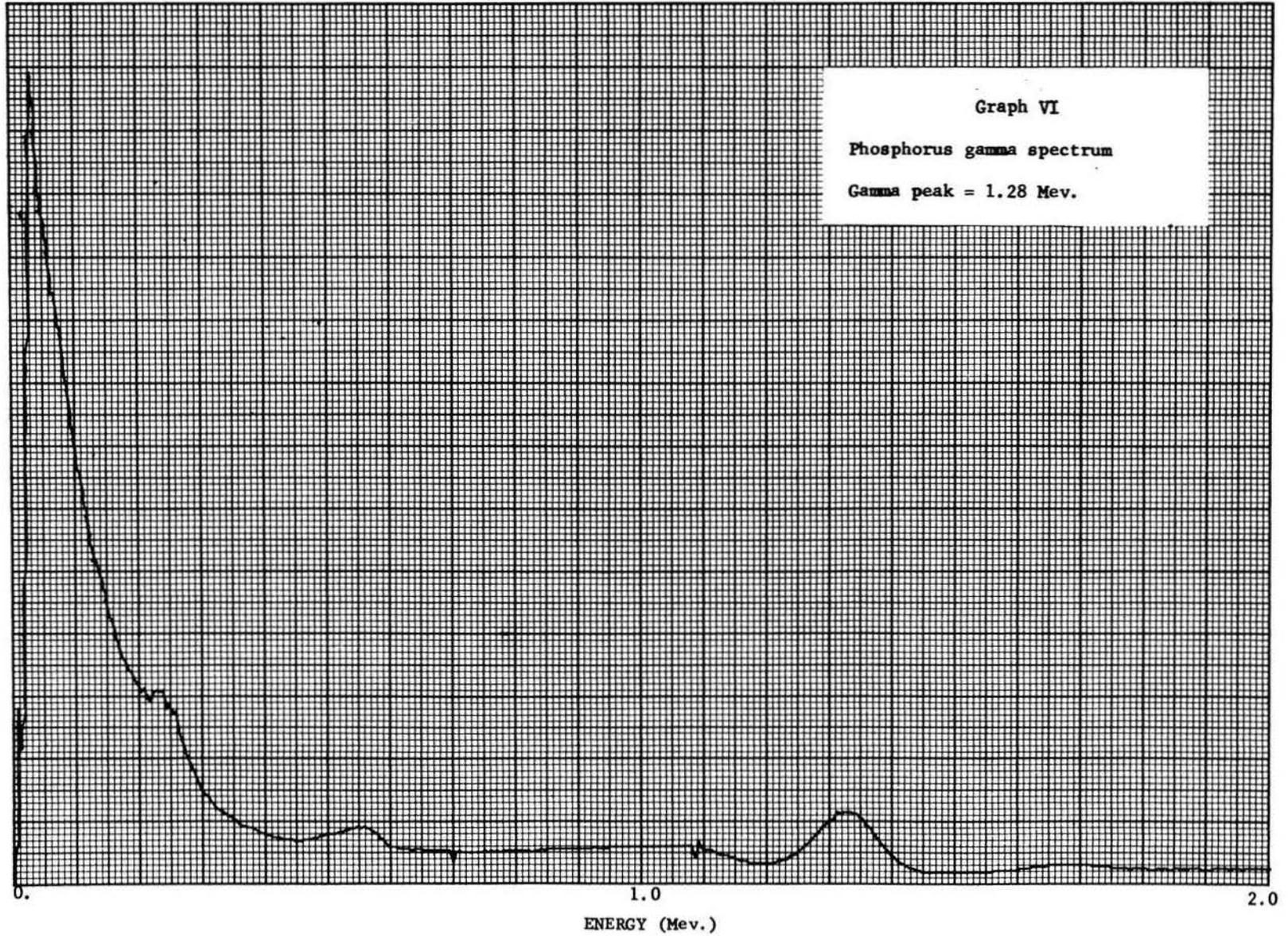






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IV Conclusions and Summary

The construction of a multi-threshold foil was undertaken both as a time saving device and to help minimize errors introduced through erroneous assumptions. A number of different materials were investigated and then four systems proposed. Metallurgical considerations were used to eliminate three of these so that only one remained, InPFe. This system was to provide three threshold reactions -- .35 Mev, $\text{In}^{115}(\text{n},\text{n}')\text{In}^{115\text{m}}$; 2.4 Mev, $\text{P}^{31}(\text{n},\text{p})\text{Si}^{31}$; 5.9 Mev, $\text{Fe}^{56}(\text{n},\text{p})\text{Mn}^{56}$. After the foil was constructed, however, it was found by irradiation that the system provided a fourth reaction, $\text{Fe}^{54}(\text{n},\text{p})\text{Mn}^{54}$. This reaction was not as active as the others but proved to be measurable. It is, therefore, decided that the InPFe system is quite adequate as a threshold detector.

This detector was irradiated for 1 hour at a power level of 10 kw. This proves to be an adequate power level to keep the counting statistics well within a 1% accuracy level. The foil should be usable up to power levels of about 1 megawatt with much shorter irradiation times. It is doubtful that this detector system would have much use below power levels of 10 kw.

V Recommendations

The other three systems of InNiFe, InNiMg, and InPMg should be investigated more thoroughly by a metallurgist. If the alloys can be synthesized, they should be irradiated to examine them as possible multi-threshold detectors. Also, since there is more cross section data available every year, there is nothing which restricts the researcher to the reactions indicated herein. A typical example is the (n,p) reaction of Cu^{65} which is alloyable with indium and nickel.

The possibility of improving the InPFe system also exists. The concentration of indium might be raised slightly with a significant increase in the resolution of the indium peak. Also there is the possibility of adding another detector material to the system to give another reaction.

Appendix I

The reactor used for all irradiations was designed and built by Curtiss-Wright. It is a pool type (modified BSR) reactor. The maximum licensed power is 10 kw. All irradiations were done at the maximum power level for a period of one hour.

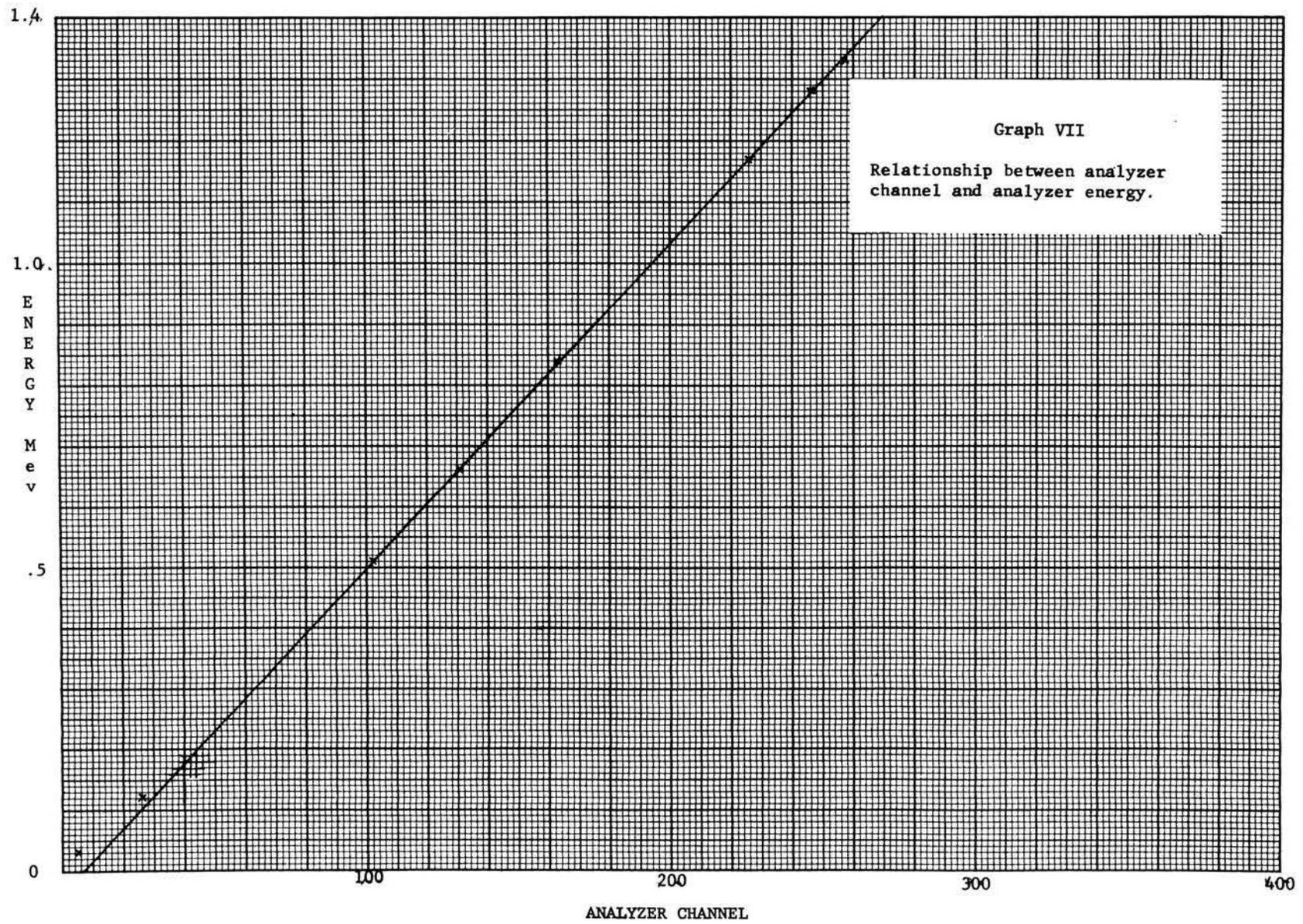
The counting of all samples was done on a 400-channel analyzer. This analyzer was built by RIDL and is their model 34-12B. A one and three quarters inch scintillation crystal and corresponding RIDL preamplifier were also used. The analyzer was adjusted so that the 400 channel memory capacity corresponded to 2.0 Mev.

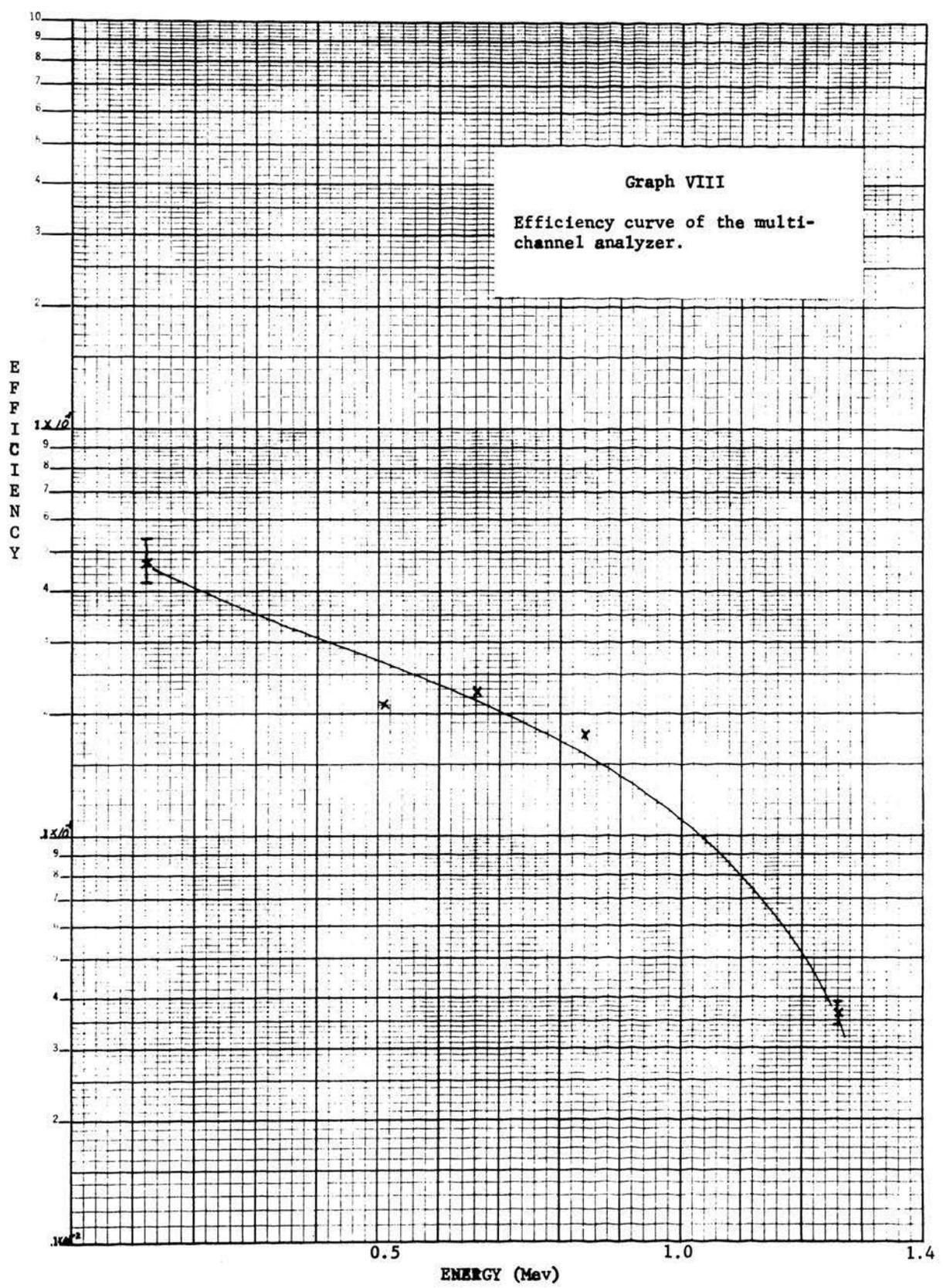
Appendix II

The calibration of the analyzer entailed the determination of two things. The first is the energy to channel correspondence, and the second is the energy dependent counting efficiency.

A. Energy. The energy to channel dependence is the correspondence of each channel to an amount of energy. The calibration was done using 2.0 Mev corresponding to the 400 channels of analyzer memory. A graph of this dependence is shown as Graph VII. This graph shows that the dependence is not a perfect linear relationship. This is due to a shift in the zero energy to zero channel correspondence. This can be corrected but required equipment which we do not have available.

B. Efficiency. The efficiency calibration was effected using four known gamma emitting samples of calibrated strength. The four samples used were Co^{57} , Cs^{137} , Mn^{54} , and Na^{22} . The integral number of counts under a gamma peak less the background radiation was then divided by the known activity of that peak. This gave a value for the efficiency which is plotted in Graph VIII. From Graph VIII the efficiency at any energy value may be taken.





Appendix III

Attenuation of Indium Gamma Ray

Because of the thickness of the foil, some self-shielding of the .35 Mev gamma ray of indium is observed. The gamma rays of iron and phosphorus are energetic enough that little shielding of these is observed and therefore, the self-shielding factor applies only to the indium gamma peak. The shielding is dependent upon the heavier metals and is considered to be dependent upon the percentage of each present. The following is suggested by Goldstien and used by Obenshain (24) to obtain the attenuation coefficient of a homogeneous mixture of different elements:

$$\beta_i = \frac{a_i Z_i / A_i}{\sum_i^n a_i Z_i / A_i}$$

$$\mu_f = \sum_i^n \beta_i \mu_i(E)$$

μ_f = the attenuation coefficient of the mixture

μ_i = the attenuation coefficient of element (i)

a_i = the fraction of element (i) present

Z_i = the atomic number of (i)

A_i = the atomic weight of (i)

The foil is considered to be a homogeneous mixture and with the above equation the attenuation coefficient for the InPFe system was found to be $\mu_f = .265 \text{ cm}^{-1}$.

The attenuation may then be found using the formula (1) for the amount of gamma transmitted, f_γ

$$f_\gamma = \frac{1 - e^{-\mu t}}{\mu t}$$

With this formula the transmittance through the foil was found to be 96%. This value is high despite the thickness of the foil because of the small amount of high atomic number material in the foil.

Appendix IV

The calculations were all done using the following formula:

$$\bar{\phi} = \frac{C \times \lambda \times A}{A_v w \bar{\sigma} E q (1 - e^{-\lambda t_1}) e^{-\lambda t_2} (1 - e^{-\lambda t_3})}$$

$\bar{\phi}$ = average flux

C = number of counts

λ = decay constant

A = atomic weight

A_v = Avagadro's number

w = weight of isotope present

$\bar{\sigma}$ = average cross section

E = analyzer efficiency

q = percent of decays by γ -ray observed

t_1 = irradiation time

t_2 = decay time

t_3 = counting time

The values for the indium average flux are as follows:

C = 52576 cts

λ = $.437 \times 10^{-4} \text{ sec}^{-1}$

A = 115

A_v = 6.02×10^{23}

w = .05 g

$\bar{\sigma}$ = 170 mb

E = .012

q = .48

t_1 = 1 hour

$$t_2 = 10 \text{ hours}$$

$$t_3 = 1/2 \text{ hour}$$

With these values the value of the average flux is $4.07 \times 10^9 \text{ n/cm}^2/\text{sec}$.

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25. Cerac Incorporated, Box 126, Butler, Wisconsin.

Vita

The author was born June 21, 1942 in Bell, California. He received his elementary education in the Los Angeles, California area. His secondary schooling was obtained in Neosho, Missouri.

He entered the Missouri School of Mines and Metallurgy, Rolla, Missouri in September, 1960. He received his B.S. in Physics from the Missouri School of Mines and Metallurgy in June, 1964.

He entered the Graduate School of the University of Missouri at Rolla in September, 1964 as a candidate for the degree of Master of Science in Physics.