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Neutron Emission and the Tritium Content Associated with
Deuterium Loaded Palladium and Titanium Metals*

K.L. Wolf, N.J.C. Packham, D. Lawson, J. Shoemaker,
F. Cheng and J.C. Wass

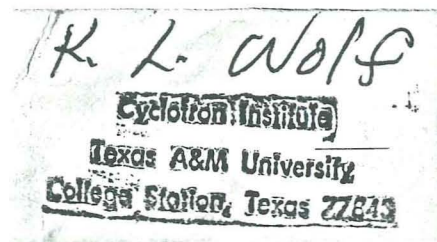
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-R.L. GARWIN-

Cyclotron Institute and the Department of Chemistry
Texas A&M University, College Station, TX 77843

Abstract:

An experimental investigation has been conducted on samples of palladium and titanium metals which have been loaded with deuterium through the electrolysis of D_2O and by absorption of D_2 gas. In approximately 200 experiments on 25 cells, statistically significant evidence for neutron emission was obtained in three separate experiments from one palladium cathode. Observed rates are 3-4 times the background rate and correspond to a source strength of 50 neutrons/min. The pulse height response of the NE213 liquid scintillator-based detectors corresponds to that expected for 2.45 MeV neutrons. Tritium has been identified in nine Pd-Ni electrolytic cells, at levels corresponding 10^{12} to 10^{16} atoms. Activity buildup curves indicate that the apparent production occurs over a time period of several hours.

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Introduction

The present paper reports some positive results for neutron emission and for tritium detection from the Fleischmann-Pons¹ type of electrolytic cells. The neutron emission corresponds to a source strength of up to 50 neutrons/min, similar to that reported by Jones, et al². Most of the results are negative with respect to the detection of neutron emission in the 25 active electrolytic and gas phase cells investigated in the present study. The experimental program involved over 200 experiments with the variation of charging times and electrode potentials. Each cell configuration has a corresponding blank experiment with an identical cell which was unpowered or run with the electrode potentials reversed. Only one cell has shown clear indications of neutron emission and on three separate occasions. The results of tritium assays are more encouraging since **nine cells have shown levels that are factors of 10^2 to 10^6 above background.** Most of the experiments have been conducted on cells with palladium cathodes. The electrodes range from 0.5 mm to 6mm in diameter and were 4 cm in length. The electrolyte generally is 0.1 M LiOD in 99.9 % purity D_2O . Titanium metal rods have been used in 0.5mm and 3mm diameters. All experiments with titanium have proved to be negative, including high pressure D_2 gas cells at 1000psi and at liquid nitrogen temperatures.

The fusion reaction one assumes to be operating is the $d + d \rightarrow {}^4He^* + n$ reaction which produces 2.45 MeV neutrons and 1 MeV tritons with a nearly equal branches, as measured at deuteron bombarding energies where fusion is known to occur. The neutron

experiments are sensitive to neutron energies of approximately 1-50 MeV to avoid an experiment of a highly specific nature, and to aid in measurement of cosmic ray background as described later. The tritium measurements use an integral, sampling technique with samples taken from the cell electrolytes and counted with liquid scintillator-based detectors. The possibility of tritium contamination from outside sources must be considered, as discussed later.

Experimental Method

The neutron experiments were performed with fast time-of-flight counters based on 3" X 5" NE-213 liquid scintillators coupled to 5" diameter RCA 8854 low noise phototubes. The timing feature cannot be used here, but the geometry of the design is well-suited to the low background requirements of the present work, through optimization of the pulse shape discrimination. The counter is located in a low gamma-ray background area but the pulse shape discrimination is needed to differentiate between neutrons and gamma rays, resulting in a background level of 0.5 c/min in the neutron energy range from 1-2.5 MeV. The lower energy level is determined by a constant fraction discriminator set at an electron energy of 0.35 MeV. The neutron response is lower than that of electrons due to saturation properties of the liquid scintillator. The efficiency is approximately 5% including the solid angle for 2.5 MeV neutrons. The contributions due to gamma-ray feedthrough into the neutron region of the pulse shape discrimination spectra are found to be less than 0.1 c/min which was established by testing with gamma-ray sources of various

intensities. Conclusions of Gai, et al ³ about the inadequacy of pulse shape discrimination are found to be in error for the present situation and can be traced to an inappropriate design of the Yale neutron detectors. Sharp corners and long light guides result in light traps which decrease the rejection efficiency of the PSD method. The major contribution to the background level of the present experiment is found to be caused by the neutron component of cosmic ray showers. Most of the electrolytic cells were constructed for compatibility with the neutron measurements and allowed placement of the palladium electrode within 0.5" of the face of the neutron counter for high geometric efficiency and little degradation of the energy spectrum. The counter and cell were surrounded with 12" of parawax and protected on 4 sides with an active cosmic-ray shield of plastic scintillator. Charged particles originating from cosmic rays are rejected, and some neutron rejection is accomplished by the use of a gate veto of 5 microsecond duration to take advantage of the correlation of particles in cosmic ray bursts⁴. The system was protected from noise bursts with an antenna pickup system which was checked with a broad range frequency scan in the location of the electrolytic cell and in the adjacent laboratory where the electronics and the computer system were located. The detector was isolated from the cell with a 0.5 mm steel plate and an air gap, and the detector temperature was monitored. The K500 cyclotron was not in operation during the time of the experiments. Data were recorded with a Lecroy 2280 CAMAC system handled by an 80386-based small computer. The neutron efficiency and response were measured with three techniques, utilizing a Pu-Be source, a ²⁵²Cf source, and

a ^{252}Cf time-of-flight measurement. The latter technique was used to measure the pulse height response for a given energy neutron e.g. for 2 MeV neutrons. A solid state detector provided a 100ps reference which signals the occurrence of a spontaneous fission event for the neutron detector. A 1-meter flight path was sufficient to define the neutron energy to 10% or better. The neutron energy response calculation discussed later was tuned to reproduce the measured spectra, and the calculation was used mainly for the extrapolation to high geometry. This procedure allows a great deal of confidence in interpretation of the energy spectra, i.e. in knowing the response of the detector to 2.45 MeV neutrons.

Experimental Results

The analysis of the neutron data provides the counting rate as a function of time and an energy spectrum for specified time intervals. The system is rather insensitive to "bursts" unless the neutrons are within 200 nsec, corresponding to the gate time of the charge sensitive ADC system. Figure 1 shows the time dependence from the on-line analysis in a broad electron energy interval of 0.35-2.5 MeV. Despite the inclusion of extra background, the signal relative to the background rate is quite prominent, reaching 3.6 c/min compared to the background rate of 0.8 c/min. The data had been grouped into 15-20 min. intervals for preliminary analysis. A cell of identical construction had been used for blank runs prior to installation of the cell labeled JBA5. At an elapsed time of 170 min, the cell was installed and the cell current was increased from a low charging

value to 140 ma. No further changes were made in the cell parameters. The cell was rotated a distance of 5" away from the face of the neutron counter at t=275 min. with a corresponding decrease in count rate, and a corresponding increase when returned back to the usual distance of 0.5" at t=300 min. The rate change is consistent with the efficiency loss due to the loss in solid angle, as expected if the neutrons were emitted from the cell and not from the surrounding material. The rate returned to the background level at t=340 min. with no change in cell parameters, signaling the end of neutron production.

The neutron energy spectrum lends more evidence to the contention that the emission of 2.45 MeV neutrons has been observed here. The pulse height data corresponding to the four points at t=230-310 min. in Fig.1 are integrated and plotted as the upper curve in Fig.2 which shows counts per 0.1 MeV energy interval as a function of energy, in electron energy units. The background curve is the sum of two time intervals each, before and after the neutron production corresponding to t= 140-175 min. and t=350-385 min., respectively. The background energy spectrum is much broader, as expected for secondary reactions in the surrounding material. In fact, the region above 0.8 MeV is used to tag cosmic-ray showers and provide evidence against interpretation of these data as cosmic-ray induced secondary reactions in the detector and in surrounding material. It can be seen in Fig.2 that the net number of counts in the region above 0.8 MeV is nearly zero, and indicates there was no unusual cosmic-ray activity. Of course this argument does not eliminate the possibility that cosmic rays may somehow initiate the $d + d$

reaction in the cell, although there has been no model proposed that fits all of the data in the present paper. Comparisons with neutron response calculations gives good agreement with the present data, and mark the position of the expected broad edge in the spectrum at approximately 0.5-0.6 MeV. The results of a calculation⁵ of the expected response is shown in Fig.3. A more modern calculation⁶ of the predicted response is shown in Fig.4. The latter calculation was performed at ORNL without the benefit of the time-of-flight experimental data described earlier. The experimental data in Fig.5 and Fig.6 are in reasonable agreement with the calculations and suggest strongly that neutrons from the $d + d$ reaction have been observed. The energy spectrum is quite different from that observed from fission spectrum neutrons, neutrons from natural alpha-induced reactions on light elements, and neutrons from cosmic ray shower-induced reactions in the surrounding material.

The response shown in Fig.2 and in Fig.5 is 9-10 standard deviations above the background level when an optimized energy integration interval is used (125 +/-13 counts). Figure 6 shows the energy spectrum for an earlier measurement on the same cell, taken with a different neutron detector (but of the same type) and different shielding arrangements (with 4" of iron surrounding the detector). The statistical significance is somewhat poorer due to a lower rate and a higher background level, caused mainly by cosmic ray reactions in the iron shield. The statistical significance corresponds to 4 standard deviations above the background (60 +/-15 counts). Again, the energy spectrum is indicative of 2.5 MeV neutrons. Of course the

statistics relative to the average background is not the full criterion for determination of the significance of the neutron signal. One must compare the count rate relative to the fluctuations possible in the background, as determined by the cosmic-ray showers. Figure 7 shows a plot of the frequency of occurrence of the background count rate for the on-line data to be compared with values of the count-rate data in Fig.1. The long tail at the highest count rates in Fig.7 is of concern here, but one can see that there is a large margin of safety for the results in Fig.1.

Tritium results constitute the second topic to be covered in this paper. The measurements are quite different and much easier than neutron detection, with the major difficulties caused by the low energy of the beta emission and the possibility of external contamination. The activity levels found here eliminate the need for extensive discussions about backgrounds due to cosmic rays or gamma rays. The standard method of tritium detection at the present time is in situ water soluble liquid scintillation counting. A commercial LKB WALLAC 1219 counter was used for many measurements, and a rather good tritium counter was built at the Cyclotron Institute with a pair of low noise 2"-diameter phototubes situated on opposite sides of a 1cm X 1cm X 3cm rectangular cell, located in a light-tight box. A 50ns coincidence requirement minimized phototube noise contributions. The home-built system allowed careful beta end-point energy determinations to be made, which verified that the activity in question was tritium, and also allowed a careful search of electrolyte solutions for higher energy alpha emitters which

proved to be negative. The activity levels as measured with the two systems at Texas A&M were within 0.5% and rather good agreement has been obtained by at least five outside laboratories. The first outside laboratory to confirm the tritium result was General Motors Research⁷, and researchers at LANL⁸ have performed many variations on the sample preparation, the most definitive being vacuum distillation of samples to leave no doubt as to the purity. With the knowledge that tritium has appeared in the electrolytic cells with no chance that chemiluminescence has caused false signals, one must consider the possibility that the tritium was introduced externally.

Tritium has been observed in nine cells constructed in the Bockris laboratory, and many of the necessary blanks have been run, but not in sufficient numbers. Over 30 cells constructed in the Martin laboratory and those for the Srinivasan heat measurements have shown no nuclear effects, even though the cells have a somewhat similar construction with common sources of many of the components. The major differences in construction of the Bockris cells include the use of nickel mesh for anodes, longer charging times, the use of small glass tubes for cell containment, the particular batch of palladium used, and the laboratory where cell preparation took place. From the data in hand, one must outline some quite special circumstances for explanations in terms of contamination. The background level of the heavy water is not a factor for the data presented in Table II as can be seen in Table I, which shows the results of approximately 10 determinations for each value quoted. Factors of 2-3 are at the limit for tritium buildup from selective

absorption which may explain the cell C-1. The total amount of D_2O added to the cells during the cell lifetime is only a few times the 15 ml cell volume. In the startup of the cold fusion experimental program, many of these cells were not assayed periodically. But those cells labeled C-C and C-G were followed and all solutions were assayed. The tritium appears rather suddenly over a time interval of a few hours as can be seen in Fig.8. The first point in Fig.8 was taken after the cell had charged at low current for several weeks, corresponding to the background level. At that time the current density was increased to 0.5 amps/cm^2 and the cell was assayed with 1 ml samples withdrawn every two hours. More D_2O was added as necessary from an assayed Aldrich sample. New hypodermic syringe packs were used for each transfer. A constant level was achieved after 12 hours which is probably indicative of the end of production. Strictly speaking, the constancy could reflect the equilibrium partial pressure of DT in the gas phase compared to the electrolyte concentration. The gas was free to escape through a tube immersed in mineral oil which prevents H_2O contamination from the atmosphere. This experiment was performed in the Bockris laboratory. In a second timed assay series, the results of cell C-G are shown in Fig.9 as a plot of activity per ml of electrolyte as a function of the date of assay. The cell had been constructed in the Bockris laboratory, charged and run at high current density and then transferred to a plastic cell and transported to the Cyclotron Institute while charging. An assay in the Bockris laboratory proved to be at the background level and a later measurement at the cyclotron was only slightly above

background, which may reflect the use of different tritium counters for the measurements. Assays were performed daily on May 5, 6 and 7 because the cell was situated in the neutron counter arrangement and the current density was increased in an attempt to correlate tritium production and neutron emission. During that period, access to the cell was quite limited since the neutron counter is located in a secure area on the experimental floor of the Cyclotron Institute, and the cell is further protected by 12" of parawax which requires the removal of a shielding block which occurred only daily for assays and refilling of the cell. The rapid rise from the low count rate of May 1 and 5 to the elevated values of May 6 and 7 cannot be ascribed to outside contamination. Again all electrolyte and D₂O samples were assayed before addition to the cell. The decrease in the activity levels at long times is ascribed to an end of production and to losses due to displacement with D₂, since the cell was returned to charging conditions. Later the electrode was transferred to a new cell and electrolyte, and the current was increased to 0.5 amps/cm², but no further production was obtained. No gas phase recombination was performed. At the cyclotron the same syringe is used for a given cell in its lifetime to avoid the possibility that syringe packs are contaminated, since tritium is sometimes used by the manufacturers for checking the quality of needles. Also no tritium tracer work has ever been done in the cyclotron laboratories so the possibility of outside contamination is low.

A final important result comes from the Bockris laboratory in another cell that produced tritium after two months of charging.

Prepared at the time of most of the other cells shown in Table II, cell C-H (not shown in Table II) was charged and run at high current density, but with no indication of tritium above background. At that time a tube filled with platinized alumina beads⁷ was fitted onto the cell for recombination of the deuterium and oxygen gases to water, and collection was made in a separate container. After two weeks of charging an assay of the recombination cell indicated 5×10^7 d/min/ml of tritium, and the electrolyte in the Pd-Ni cell contained 5×10^5 d/min/ml.

Strictly by coincidence, both cell volumes were approximately 15 ml of D₂O. It had been suspected that large losses had occurred into the gas phase in the other cells, since measurements on recombination in the Fleischmann-Pons type cells have shown that less than 1 % of the gases recombine. The number of tritium atoms present here can be calculated to be approximately 10^{16} , and this was not the highest count in the electrolyte, compared to cells C-A and C-B in table II. It should be noted that the current density was not high ($.05 \text{ amps/cm}^2$) during the collection time, although it had been run at 0.5 amps immediately before the installation of the catalyst.

Discussion of Results

The neutron results have shown three indications of 1-2 hour periods of neutron emission. One of these indications is over nine standard deviations in statistical significance and two indications are four standard deviations. All have occurred with the same palladium electrode. Over 20 cells of similar construction have given negative results. The energy spectra are

consistent with those expected for 2.5 MeV neutrons, and the source is from the electrolytic cell. Some mechanism which involves the neutral component of the cosmic-ray spectrum cannot be ruled out, but no plausible mechanism has been suggested. The source rates of 20-50 neutrons/min are comparable to those measured by Jones, et al².

The tritium results show a much higher rate of reproducibility, since cells with eight 1-mm diameter Pd-wires and one 3-mm Pd-rod have shown significant levels. The calculated energy release rate based on the amount of tritium detected is significant, making the results even more important. If the production were through the $d + d$ reaction with a Q-value of 4 MeV, the power level is approximately 10 watts/cc of palladium assuming a 5-10 hour generation period and using the activity levels of cell C-H.

The correlation of neutrons, tritium and heat has proved to be negative. Obvious heat generation (ignition) by a Sirinivassan cell was not accompanied by neutron emission, and these cells show no tritium production. Cell C-D has shown both neutron emission and tritium production, but it is not known if the occurrence was simultaneous. After reprocessing the electrode from cell C-D by melting the Pd, a positive neutron indication was observed but no tritium was produced at detectable levels. The measurement on cell C-G failed to give a neutron signal during the period of a positive tritium indication. If there is no delayed release mechanism for the tritium, one can state that the branching ratio for neutron production is too low by at least a factor of 10^5 compared to the observed tritium level in the

electrolyte, for the d + d fusion reaction. Caution must be used in this conclusion because of the observation of tritium production in cell C-H well after a period of high current density. Neutron generation should occur due to the secondary reaction of $t + d \rightarrow {}^4\text{He} + n$ to produce 14 MeV neutrons even if the d + d reaction has a modified branching ratio or a different mechanism to avoid compound nucleus formation. The neutron detector system is quite efficient for 14 MeV neutrons and the energy spectra would allow easy identification, at a rate of approximately $10^6/\text{min}$. The 3 MeV proton that accompanies the 1 MeV triton from d + d fusion would produce no neutrons since it is below the Coulomb barrier for Pd and is not of sufficient kinetic energy to breakup the deuteron (3.38 MeV). A search has not been made as yet for gamma rays produced by Coulomb excitation. The conclusion can be drawn, however, that if tritium is produced in the cells, an unknown mechanism creates the triton at a low kinetic energy to explain the lack of secondary reactions. Such mechanisms have been postulated, but there have been no detailed calculations performed.

An almost equally unlikely explanation occurs in terms of tritium contamination, but it is the one being pursued with tests and blanks in the cyclotron laboratories. The palladium cathodes originated from the same batch of material, and similarly for the nickel anodes. From the three samples which have been measured for buildup of tritium in the cells, it must be concluded that the tritium was introduced or was contained within a cell component at the time of cell preparation. Similarly, the

observation of approximately 100 times the activity in the gas phase compared to the electrolyte indicates that the tritium was not introduced inadvertently into the electrolyte. The palladium is the most likely material for contamination because of its hydrogen storage capability, but all materials are being tested. Up to 10^{-4} of the hydrogen sites in the Pd must be preloaded with tritium to account for the yields found. This would be a considerable accident and would probably have required a trip to Savannah River for the cell C-H. Two H_2O , LiOH cells prepared from the same batches of the other materials (Pd, Ni, cells, rubber septa, etc.) have failed to produce any tritium. Samples of the Pd and Ni have been sent to LANL for direct analysis for preloaded tritium, but no results have been obtained as yet. Another type of blank was run by extracting the Pd wire from a cell that had charged for 25 days. The cathode was replaced with a 1 mm Pt wire. The cell was run at high current density and assays followed in the usual fashion as shown in Fig.10. No tritium above background was detected with this Pt-Ni cell. Figure 11 shows results for a cell prepared in the Bockris laboratory and run at the cyclotron. The negative result in itself constitutes a type of blank. The program of blanks and testing continues, but at present the strongest argument against tritium contamination is the magnitude of the yields detected at locations where little or no tritium is used, and the unlikeliness of contamination in the refining and manufacture of materials.

References

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TABLE I

TRITIUM BLANK SAMPLES, COUNTER BACKGROUND NOT SUBTRACTED

SAMPLE DESCRIPTION	COUNTS MIN ⁻¹ ML ⁻¹
Millipore H ₂ O	23
Aldrich 99.9% D ₂ O	64
D ₂ O with KHP for pH adjustment	50
D ₂ O + 0.1mM NaCN with KHP	48
0.1 M LiOD	68
0.1 M LiOD with KHP	65
0.1 M LiOD + NaCN with KHP	66
ISOTEC D ₂ O	26

TABLE II

TRITIUM ACTIVITY FROM PALLADIUM - NICKEL CELLS

CELL	ELECTRODE TREATMENT ^a	ELECTROLYTE ^b	ACTIVITY (d min ⁻¹ ml ⁻¹)
C-A ^d	B	1	4.9 X 10 ⁶
C-B	C	2	3.7 X 10 ⁶
C-C	D	1	
	after charging at 0.05 amp/cm ² for 4 weeks		64
	after 2 hours at 0.5amp/cm ²		5290
	after 6 hours at 0.5amp/cm ²		5.0 X 10 ⁵
	after 12 hours at 0.5amp/cm ²		7.6 X 10 ⁵
C-D ^c	B	2	1.2 X 10 ⁵
C-E	A	1	3.8 X 10 ⁴
C-F	B	1	6.3 X 10 ⁴
C-G	A	2	
	after charging at 0.05 amp for 4 weeks; 0.5amp, 12 hours		120
	after additional charging for 1 week,		250
	after 0.1 amp for 24 hours, 0.3 amp for 1 hour		1.5 X 10 ⁴
C-2 (3 mm)	B	1	6.3 X 10 ⁴
C-3 (3 mm)	C	1	0
C-1 (6 mm)	A	1	69

a) electrode treatment: A, no treatment; B, vacuum anneal; C, acid etch; D, electroclean

b) solution type: 1, 0.1M LiOD; 2, 0.1 M LiOD + 0.1mM NaCN

c) cell JBA5 which has shown neutron activity, 50 n/min

d) verified by second ³H counter at TAMU and by 5 other laboratories

All electrodes are 1mm diameter palladium except where noted.

A blank count rate of 65 c/min has been subtracted before calculation of activities.

Figure Captions

Fig.1. The observed count rate in counts per minute for a neutron counter as a function of time, divided into 15-20 minute intervals. The Pd-Ni cell was installed 0.5 " away from the face of the neutron counter at t=170 min marked as "A" and the cell was rotated away from the counter by a distance of 5" at the point marked "B".

Fig.2. The observed energy spectrum associated with the count rate data in Fig.1. The number of counts per 0.1 MeV in electron energy units is plotted as a function of electron energy as established with ^{137}Cs , ^{22}Na and ^{60}Co gamma-ray source Compton edges, and with triggered cosmic ray muons. Above 1 Mev the average of every 5 points is plotted.

Fig.3. The efficiency of the counters used here for 2.45 MeV neutrons as a function of energy in electron energy units, using the monte carlo calculation of ref.4. The crosses include the energy resolution of the detector and should be compared to experimental data in Fig. 5,6.

Fig.4 The shape of the neutron energy spectrum expected for 2.45 MeV neutrons for the geometry of the present experiments. The calculation was performed by J. K. Dickens, ref.6. The abscissa is scaled in channels to correspond approximately to electron energy, 1 MeV = 100 channels.

Fig.5 The background subtracted data from Fig.2 (net) on the same scale as the efficiency calculations in Fig.3 and Fig.4.

Fig.6 The background subtracted data similar to Fig.5 for an earlier run on the same electrolytic cell.

Fig.7 The number of occurrences of a given count rate as a function of that count rate (frequency distribution) for evaluation of the data in Fig.1.

Fig.8 The count rate observed for cell C-C during liquid scintillation counting in a pulse height window appropriate for tritium beta decay, approximately 2-25 keV. Counter background (65 c/min) has not been subtracted. Samples of 1ml volume were withdrawn from the cell and mixed with 15 ml of liquid scintillator. All samples were counted again 24 hours later in a check for chemiluminescence. The zero of time on the abscissa was taken at the time of cell current increase from .05 to 0.5 amps/cm².

Fig.9 The count rate in (d/min/ml) in a tritium window(Fig. 8) for cell C-G in Table II as a function of date of assay. Notice that the abscissa is not linear. The current was increased on May 6 where indicated. Neutron measurements were made simultaneously.

Fig.10 The count rate in a tritium window (Fig.8) as a function

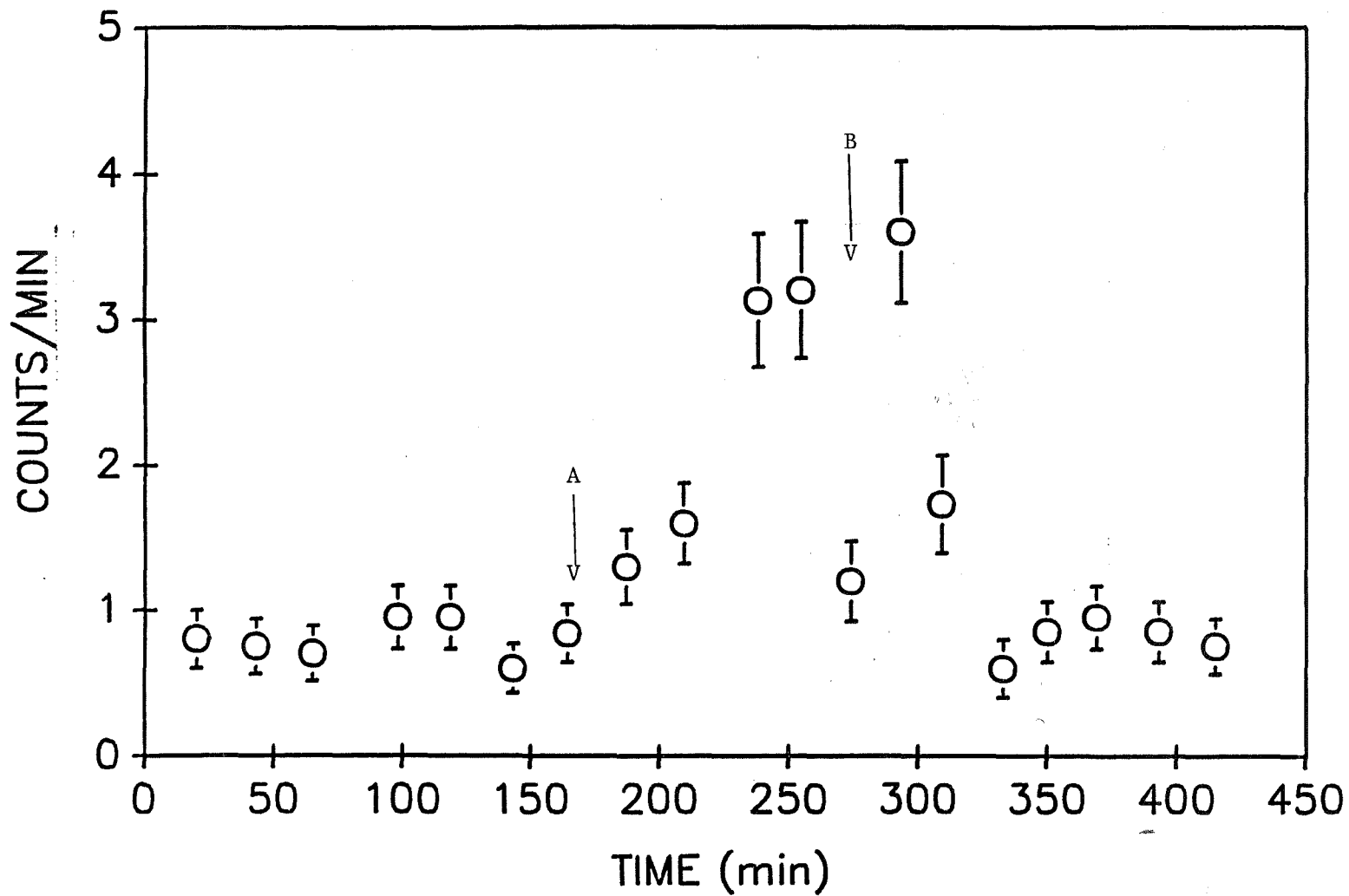
sample number, assayed approximately hourly for cell C-D in which the Pd-electrode was replaced with a 1mm Pt-electrode and the current density was increased to 0.5 amp/cm^2 . ISOTEC D_2O was used (Table I).

Fig. 11 The count rate as a function of sample number, taken approximately hourly for sample JB11. The cell contains a 3mm Pd rod similar to cell C-G in Table II. No counter background has been subtracted. ISOTEC D_2O was used (Table I).

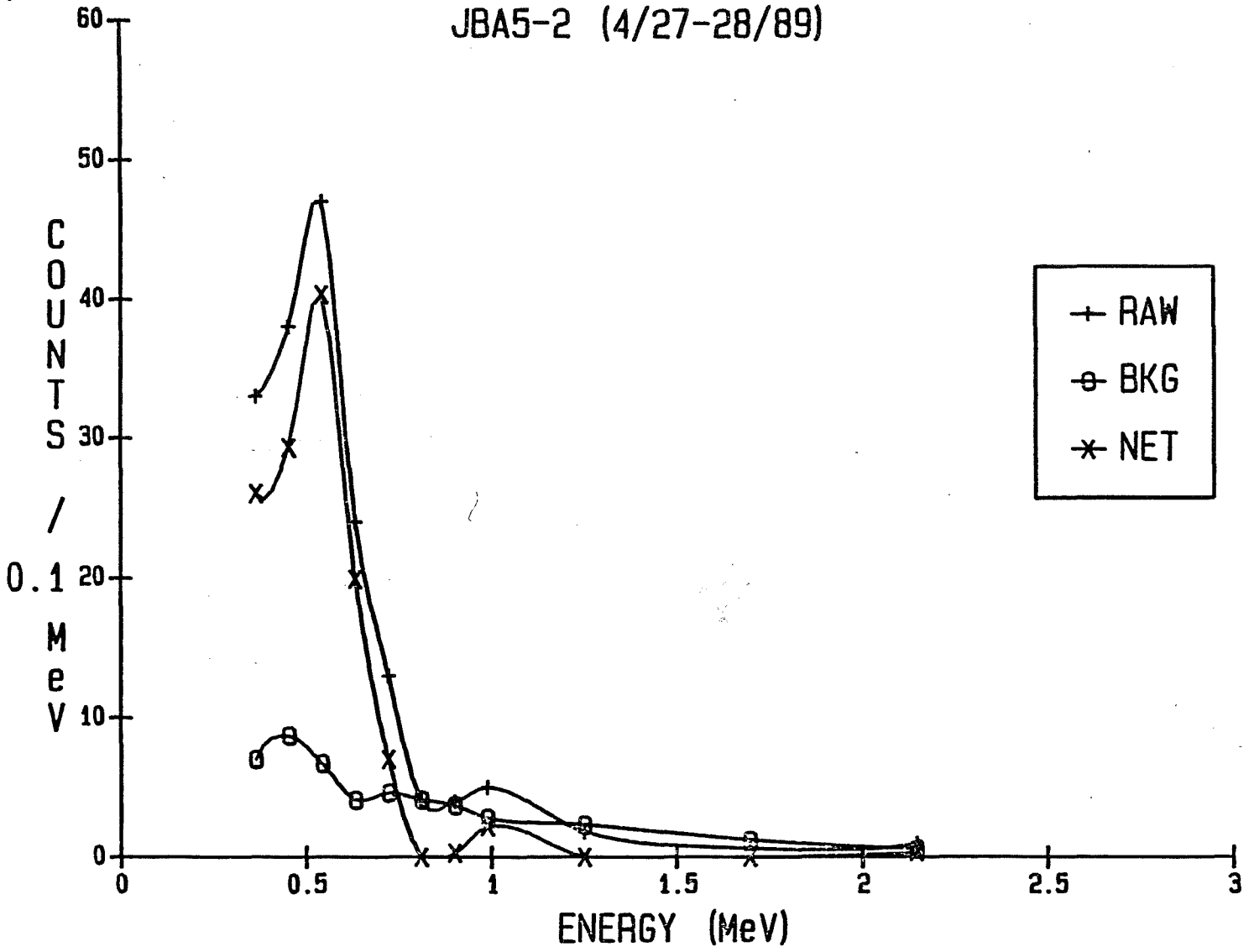
ACKNOWLEDGEMENTS

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JBA5-2 (4/27/89)

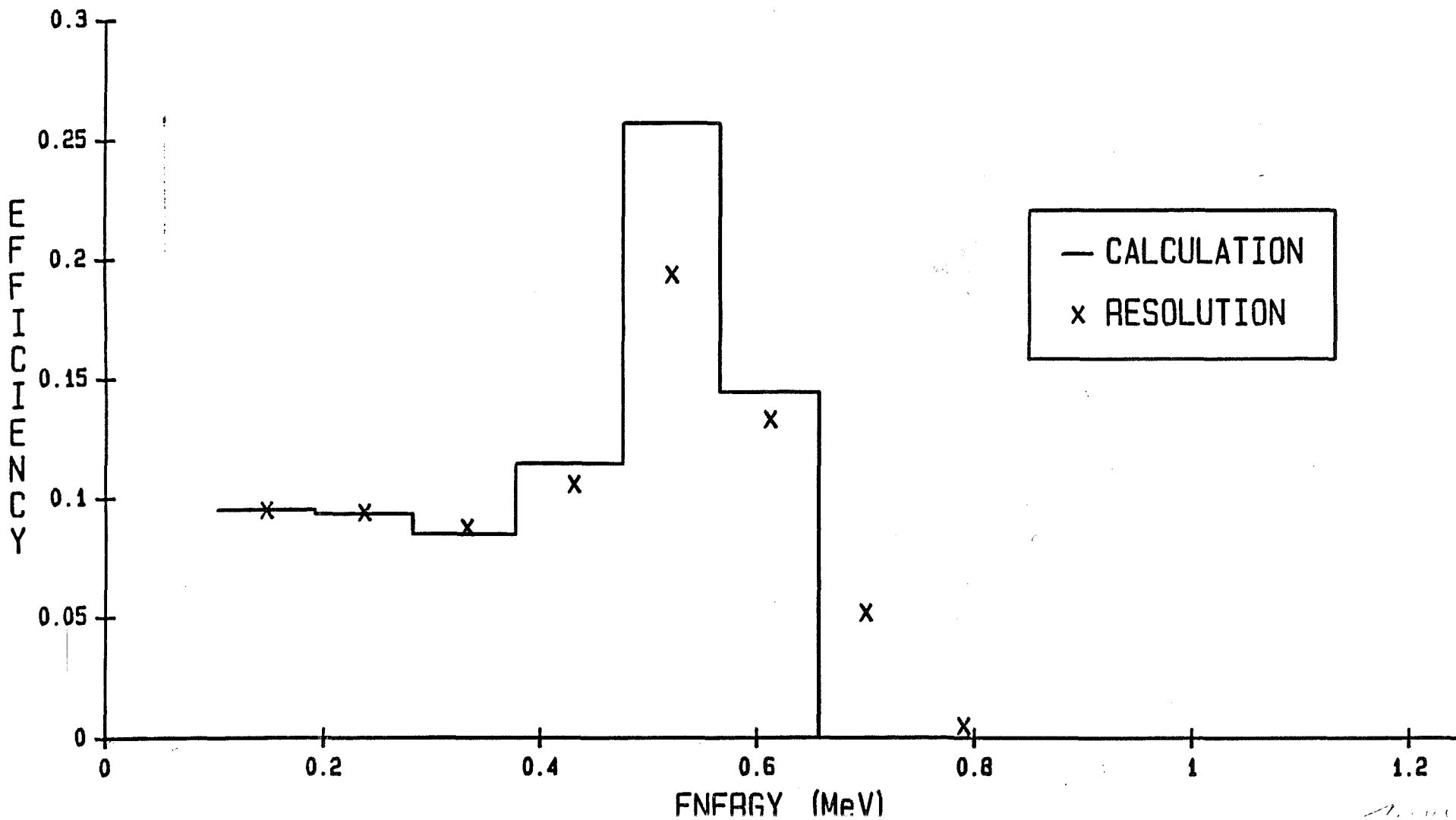


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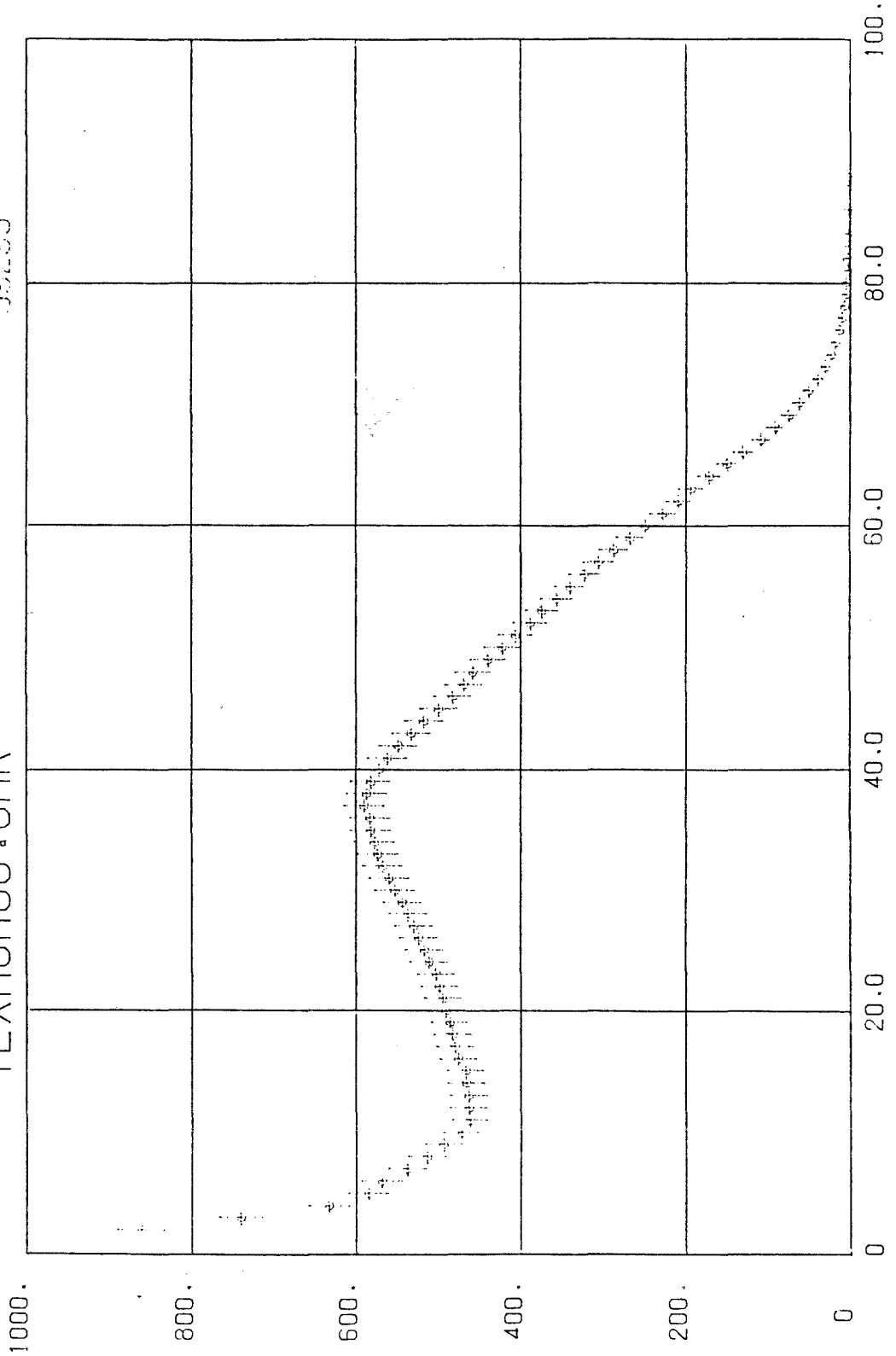
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ENERGY DISTRIBUTION: 2.45 MeV NEUTRONS

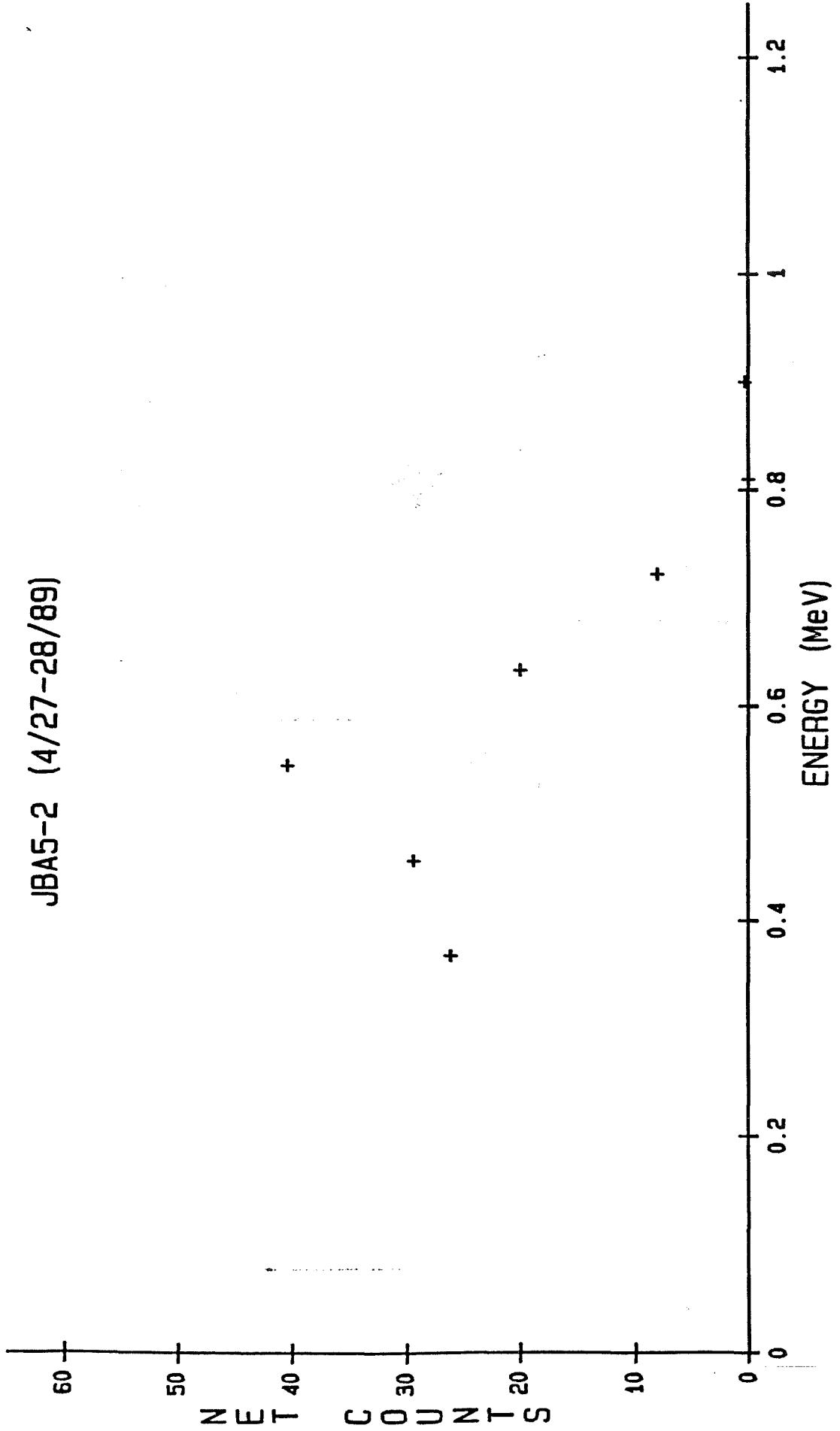


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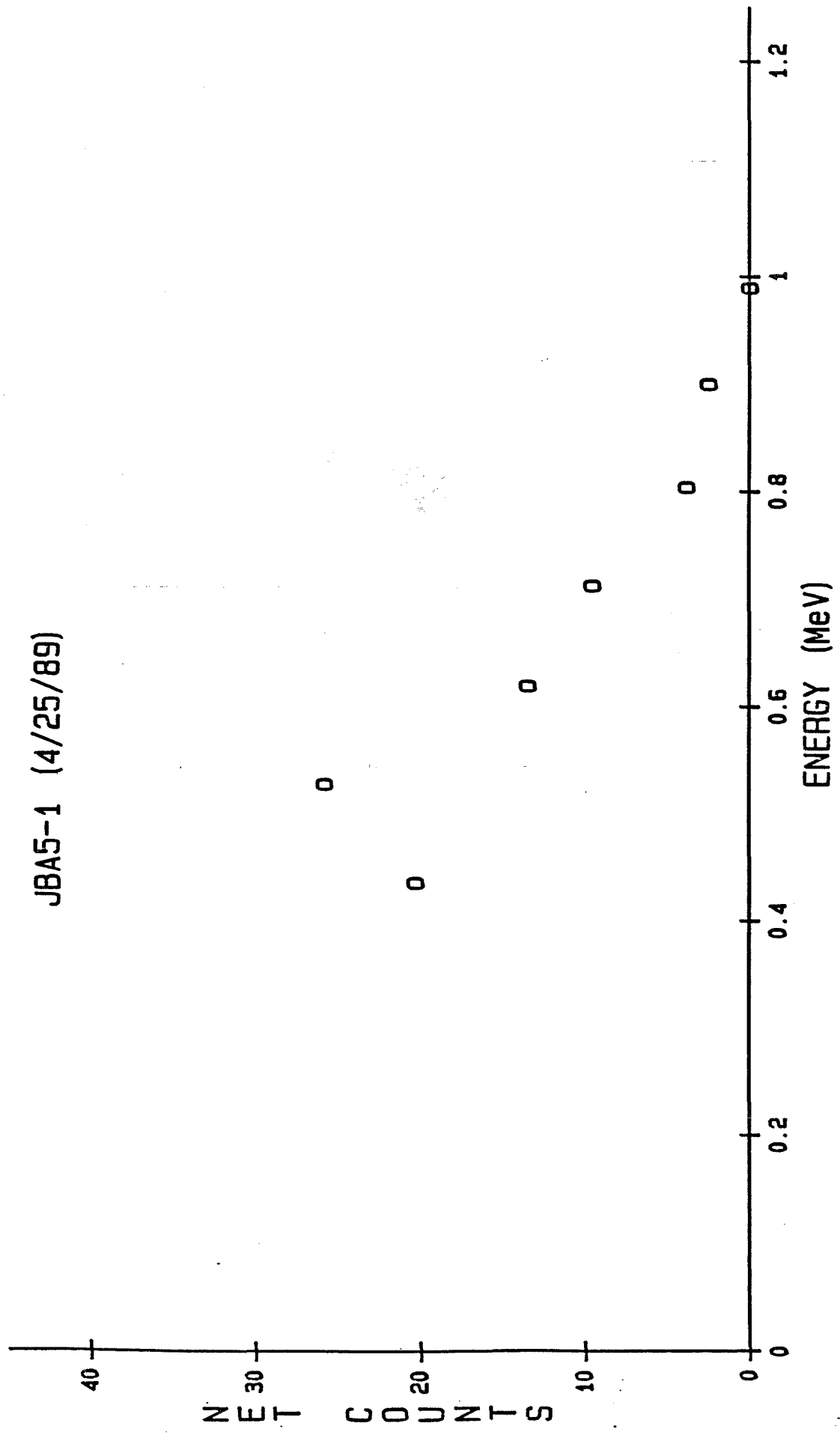


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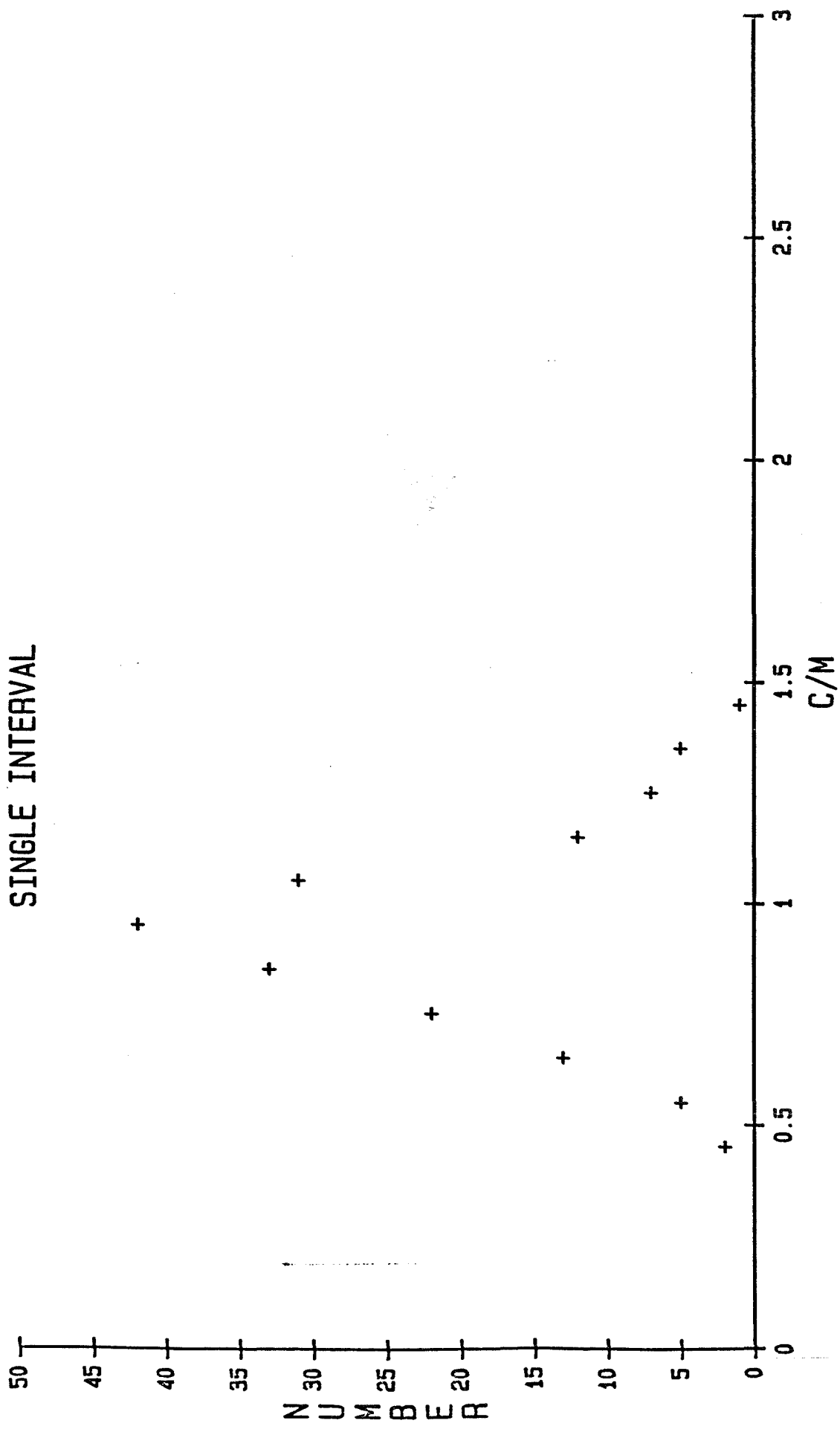
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JBA5-1 (4/25/89)



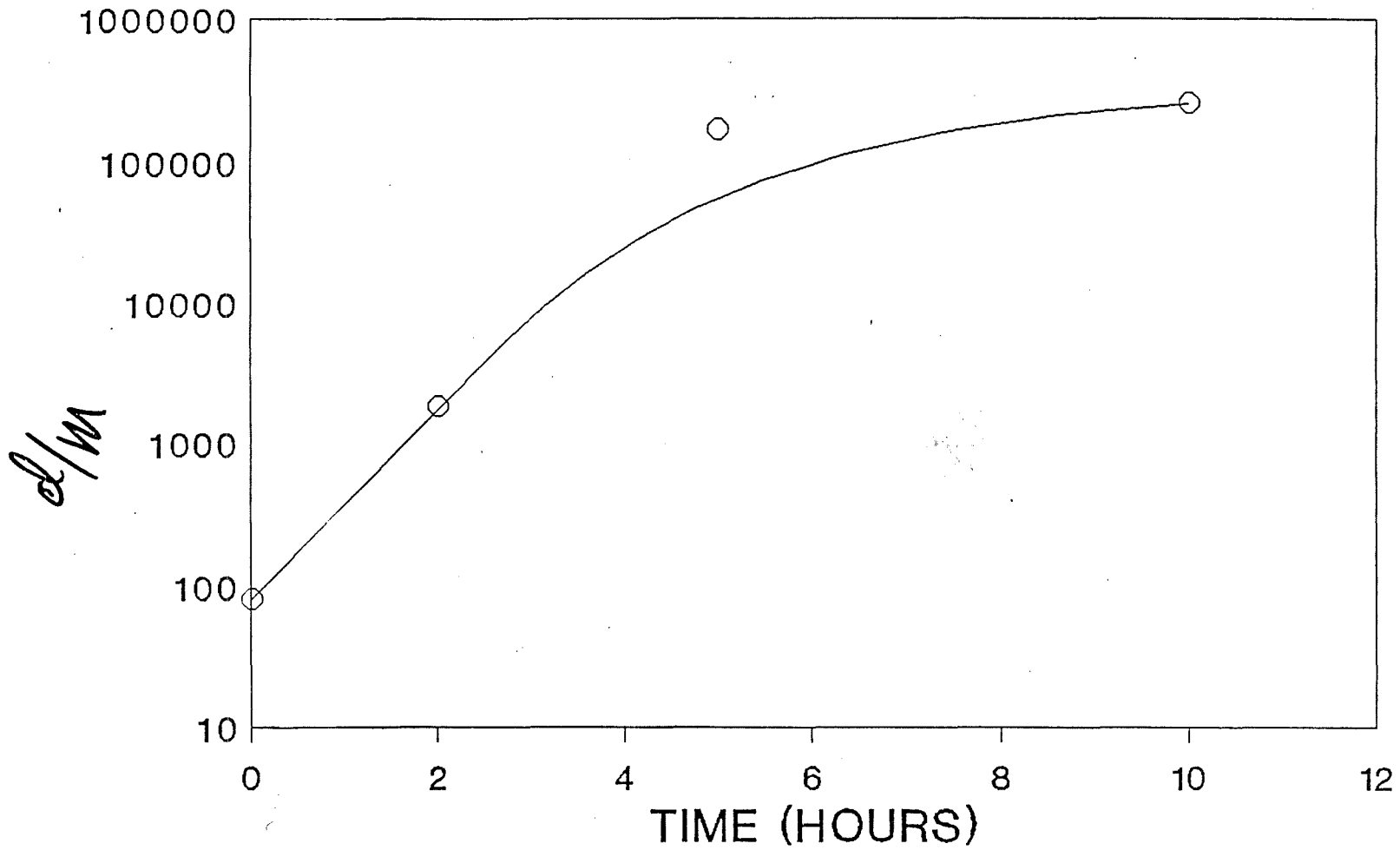
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SINGLE INTERVAL

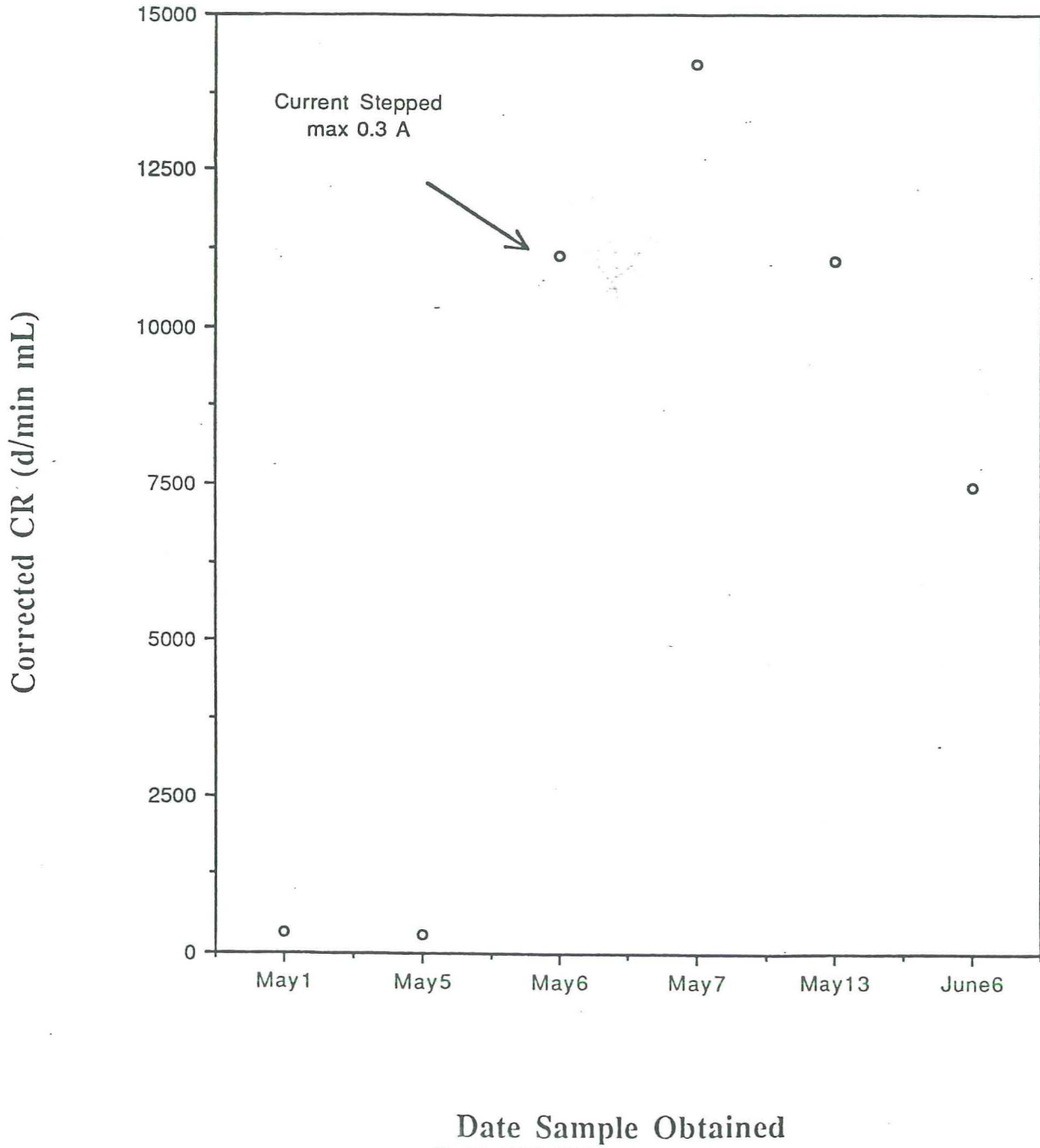


TRITIUM TIME PROFILE FROM CELL C

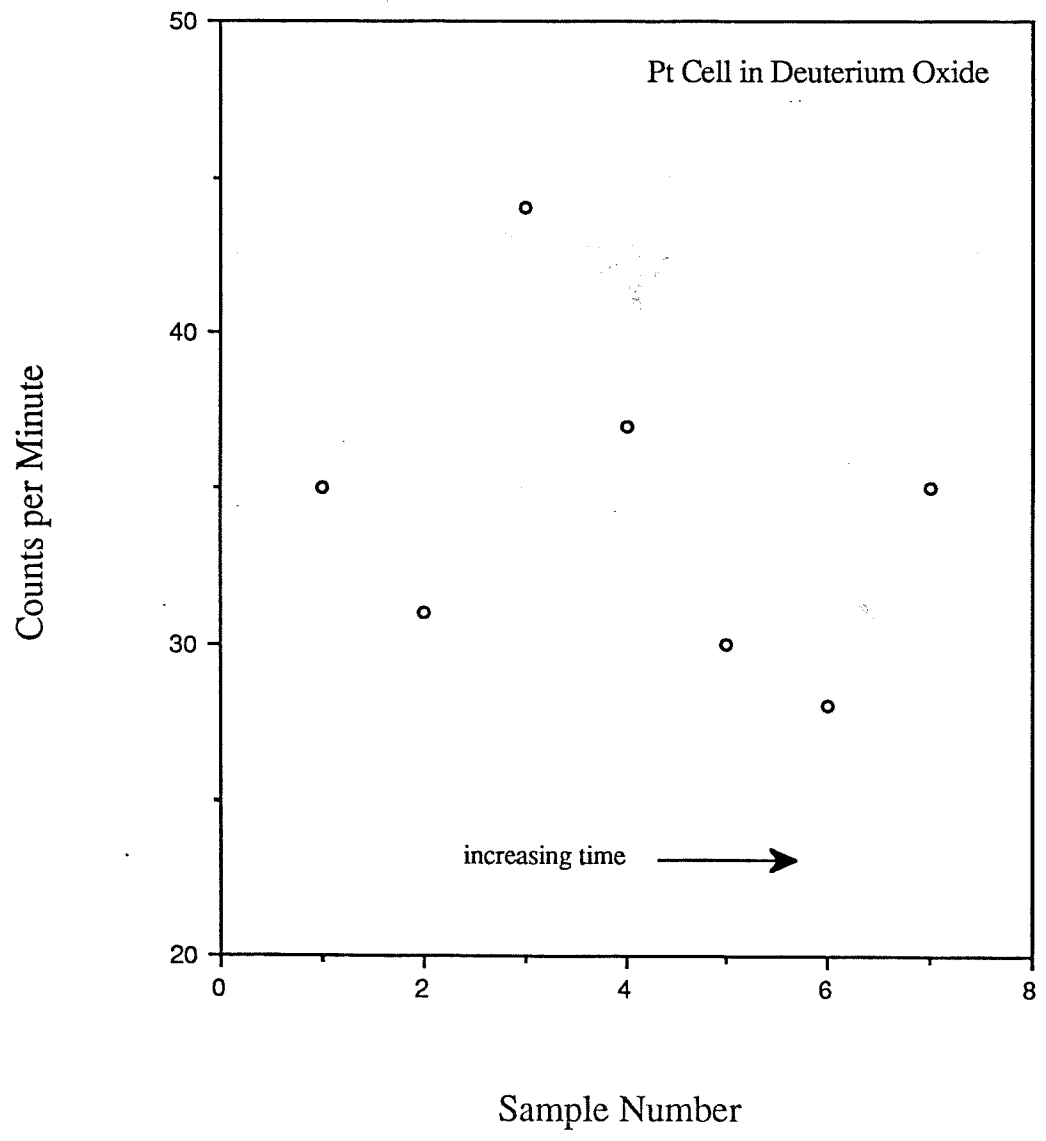
lmm



Plot of Tritium Counts vs. Date Sampled for JBXR1



Tritium Counting - cpm vs. sample number



Tritium Counting- cpm vs. sample number

