

# Investigating the Emission of Neutrons During the Fracturing of D<sub>2</sub>O Ice and During the Fracturing of Ti Shavings Immersed in D<sub>2</sub>O



## FINAL REPORT

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## Abstract

We developed an experimental apparatus to repeat the deuterated ice ( $D_2O$ ) fracturing experiments reported by the Soviet group of Deryagin<sup>1</sup>. In particular, we designed and characterized both a low-level neutron detection system, and in parallel, a high velocity projectile technique to fracture ice. The primary objective of this experiment was to reproduce if possible the claimed rate of neutron production during the fracturing process. A key factor was distinguishing any such signal from the low-level cosmic ray background of neutrons. We performed several sets of preliminary neutron-production measurements that involved fracturing  $D_2O$ -ice with a projectile fired from a high velocity air gun. These experiments were then analyzed in detail, statistically, using Poisson simulation methods. The results were found to be inconclusive, because neither the neutron detection efficiency nor the rate of firing shots were high enough. We then developed a second-generation neutron detection system that had a tenfold increase in detection efficiency. In addition to the ice fracturing experiments, we performed an experiment in which titanium shavings immersed in  $D_2O$  were fractured in a ball mill. This experiment produced no evidence for neutron emission. Also, we performed some exploratory experiments using microwave-plasma-assisted chemical vapor deposition to produce carbonized titanium that was loaded with deuterium. In this process, a high density of cracking occurred, that could have produced fractoemission of neutrons. We observed no evidence for neutron emission, although electronic noise problems greatly reduced the experimental sensitivity.

## I. Introduction

During the last several years, a group of researchers in the Soviet Union have reported in several publications that the cracking of solids produced neutrons which were measurable above the natural background<sup>1-3</sup>. In their interpretation, these neutrons were the product of nuclear reactions that involved the fusion of deuterium nuclei which had been accelerated to 10 or more keV of energy in the interior of cracks whose walls were of opposite electrical charge. Because these results were unprecedented in the nuclear physics literature and because no established theory predicted them, the validity of the results were, a priori, open to scrutiny. Moreover, the statistical significance of the net neutron counting rates reported by the Soviet group was not adequately addressed in the papers regardless of the origin of the detected events. Despite the obscurity of these early experiments and the concomitant questionability of the reported results and conclusions, it appeared that the reported "fractoemission" process could have been in fact real and not an experimental artifact. The technical benefit that could arise if mechanical cracking were to induce low-energy nuclear reactions could be potentially very significant.

Because these fractoemission processes would open a new area of research in condensed-matter physics, we began an experimental program to address the question of whether the production of neutrons in these cracking experiments in fact arises from some type of nuclear interaction. Alternatively, we sought hard evidence to determine whether the reported results were either an experimental artifact or were produced by a real process that proceeds with an insignificant rate. Similarly, some recent theoretical work suggests that ions formed in cracks may be accelerated, because large electrostatic potential differences are present during mechanical cracking of solids<sup>4</sup>.

Given this information, we designed experiments that are very similar to the design of the Soviet experiments, namely, the cracking of D<sub>2</sub>O ice with an air gun and the fracturing of the chips in a ball mill. However, because the Soviet experimental designs were limited in several aspects and because we did not have enough detailed information to completely duplicate their design, we designed our experiments to incorporate the essence of the Soviet experiment but we introduced what we believe are some significant improvements. In particular, the Soviet experiment employed BF<sub>3</sub>-filled proportional counters to detect neutrons which were positioned in an oil bath that acted as a neutron moderator. From the published schematic diagram,<sup>2</sup> it was clear that the geometric arrangements of the neutron detectors did not provide optimal detection efficiency. Furthermore, the published pulse-height distributions, which were the primary experimental data, indicated that the experiments could have been significantly affected by spurious counts and low-energy events. During the course of the project, we contacted the Soviet scientists to query them about several

apparent inconsistencies in their published papers. They did reply to our questions; and, indeed they admitted that their papers contain numerous errors. Hence, the value of the Soviet papers is at this point problematical. Nonetheless, we performed investigation independently of the vagaries associated with the Soviet works.

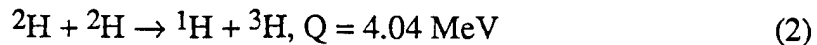
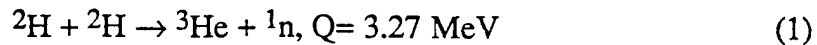
In low-level neutron detection experiments, the two primary sources of background are (1) neutrons that either originate from the cosmos or are produced by other cosmic ray interactions and (2) low-energy signals that arise from gamma radiation interactions and spurious signals that arise from electronic noise. The former produce electronic signals in the detector that are indistinguishable from the signals that the neutrons of interest would produce. The latter produce signals that differ in character and that can be eliminated to a large extent by using the appropriate electronic discrimination circuitry, which in this case consists of pulse-shape analysis. Another means of distinguishing noise-produced spurious counts is to use the characteristic shape, i.e., the signature, of the pulse-height distribution that is produced by neutron interactions in the detector. This characteristic shape is used to discriminate against signals that are either too small or too large to have originated from a neutron interaction in the detector. Since spurious signals that are caused, for example, from a cable jiggling, can be large enough in amplitude to appear as a neutron-produced event in the pulse-height distribution, using the pulse-height signature is a necessary but not a sufficient condition to assure that the events represented by the pulse-height distribution were produced by neutron interactions in the detector. Thus, the best neutron detection design for low-level measurements should employ a detector type that gives a well-defined pulse-height distribution when the neutrons of interest interact in the detector. In addition, pulse-shape analysis should be used to reduce the number of spurious events, and shielding should be arranged to reduce the cosmic-ray neutron background as much as possible. With this information in mind, it was clear that the Soviet experiment was lacking some of these features. In this context, since we were not interested in producing results that would have been subject to the same vagaries that have plagued the Soviet work, we approached the neutron detection part of the experiment as is described in the next section<sup>5</sup>.

Finally, we mention that while our experiments were underway, investigators at Sandia National Laboratories performed a much more sensitive experiment<sup>6</sup> on lithium deuteride, using a very large, efficient neutron detection system<sup>6</sup>. They found no evidence of neutron emission that resulted from the fracture of lithium deuteride.

## II. Experimental Details

### A. Neutron Detection System

The nuclear process that deuterons can undergo are given by:



Equation 1 represents the reaction of interest. From the kinematics, the neutron can carry 2.46 MeV of the total kinetic energy, 3.27 MeV.

The three general considerations associated with designing a neutron detection system are (1) the overall neutron detection efficiency, (2) the reliability associated with separating the signals that originate from neutron interactions in the detectors from noise, and (3) the extent to which the cosmic ray background can be reduced. These three factors are interrelated. More importantly, the available resources greatly constrain the extent to which the available technology can be employed to achieve these design considerations. During the initial phase of this project, the primary objective was to repeat the Soviet experiment, which involved fracturing thin D<sub>2</sub>O ice layers with a high velocity projectile from an air gun. To accomplish this task, we used BF<sub>3</sub>-filled detectors that were similar to those used by the Soviets. Unlike the Soviets, we constructed a polyethylene half-annulus to improve the counting geometry (a full annulus was not practical because of space restrictions around the existing air gun). Figure 1 shows the detector system, schematically. To improve the quality of the pulse-height signature, we selected a BF<sub>3</sub> gas-fill pressure of 20 mmHg, which was probably lower than the pressure used in the Soviets' detectors. To compensate for the loss of neutron detection efficiency that was associated with less boron being in the detectors, we used eleven tubes instead of seven tubes, which is the number reported by the Soviets. To reduce the background, the half-annulus was surrounded by a layer of polyethylene, and this layer was separated by a cadmium sheet. Figure 2 shows a pulse-height distribution that resulted from an Am-Be neutron source being incident on the system. This distribution is a characteristic signature of the neutron interactions in this type of detector.

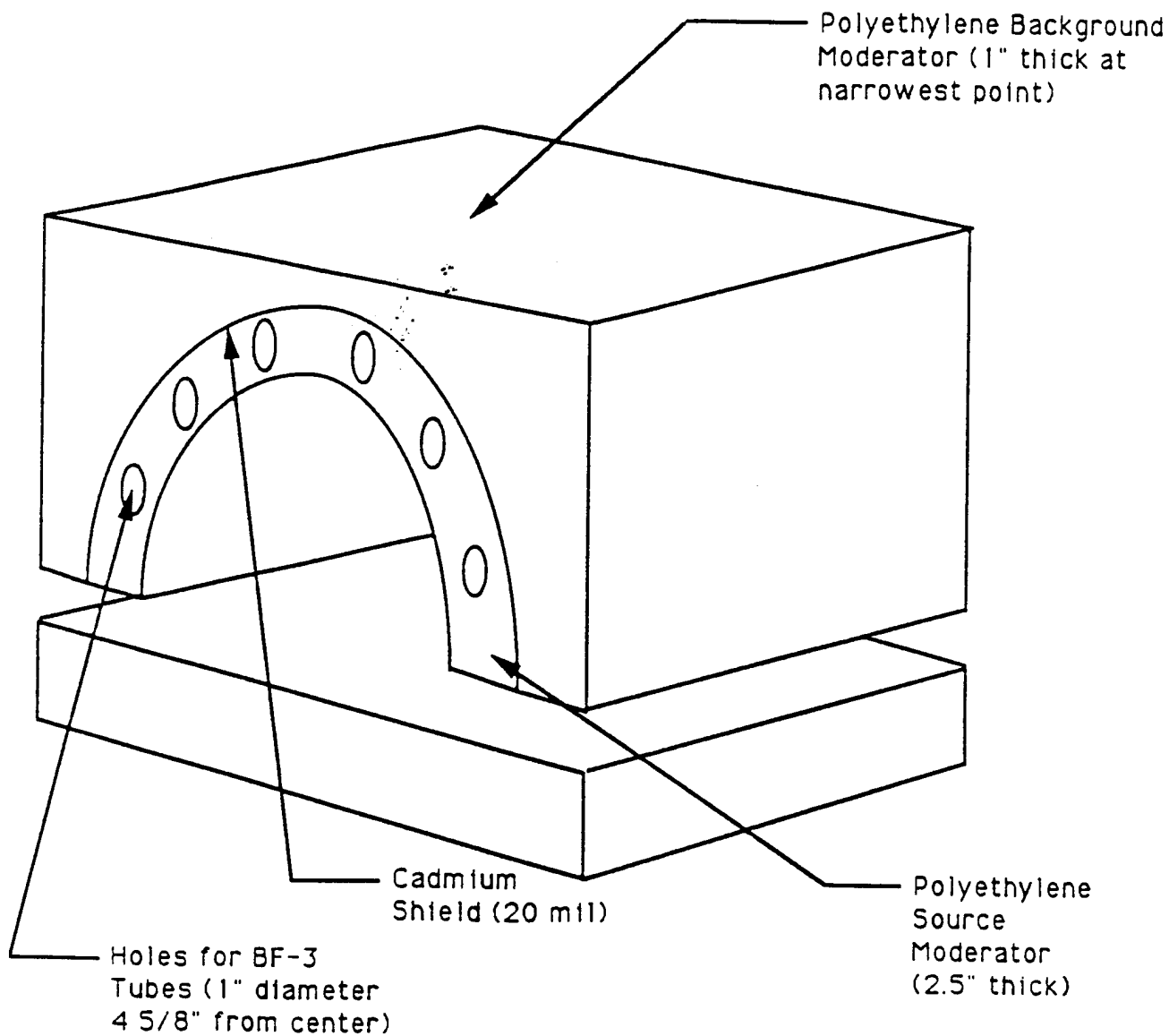


Figure 1. Low-Level Neutron Detector Moderator and Background Shield. Two half annuli were constructed: one having holes for six tubes and the other having holes for eleven tubes.

AUG 03 1989 11:15:19 AM MODE: PHA ADD A DEAD TIME: 00  
 GROUP: F US: 512 CTS GAIN: 2048 CHLS OFFSET: 0000 CHLS ID: GATED E 3SEC

FUNCTION KEY

F1	F2
OPTNS	IDENT
F3	F4
STORE	LOAD
F5	F6
170	USER
F7	F8
CTRD	CHLNR
F9	F10
DOS	MAIN



CHANNEL: 1703 COUNTS: 00000014 ROI #1: OFF ROI #2: OFF  
 PK #: 00 CTRD: 0.00000 CHL FWHM: 0.00000 CHL GROSS: 000000000 NET: 000000000  
 TIME LIVE PRESET: 000000 ELAPSED: 000084 REMAINING: ∞ SECONDS

Figure 2. Energy-Gated Pulse-Height Distribution from Eleven Low-Pressure BF<sub>3</sub>-Filled Proportional Counters Connected in Parallel to a Single Preamplifier.

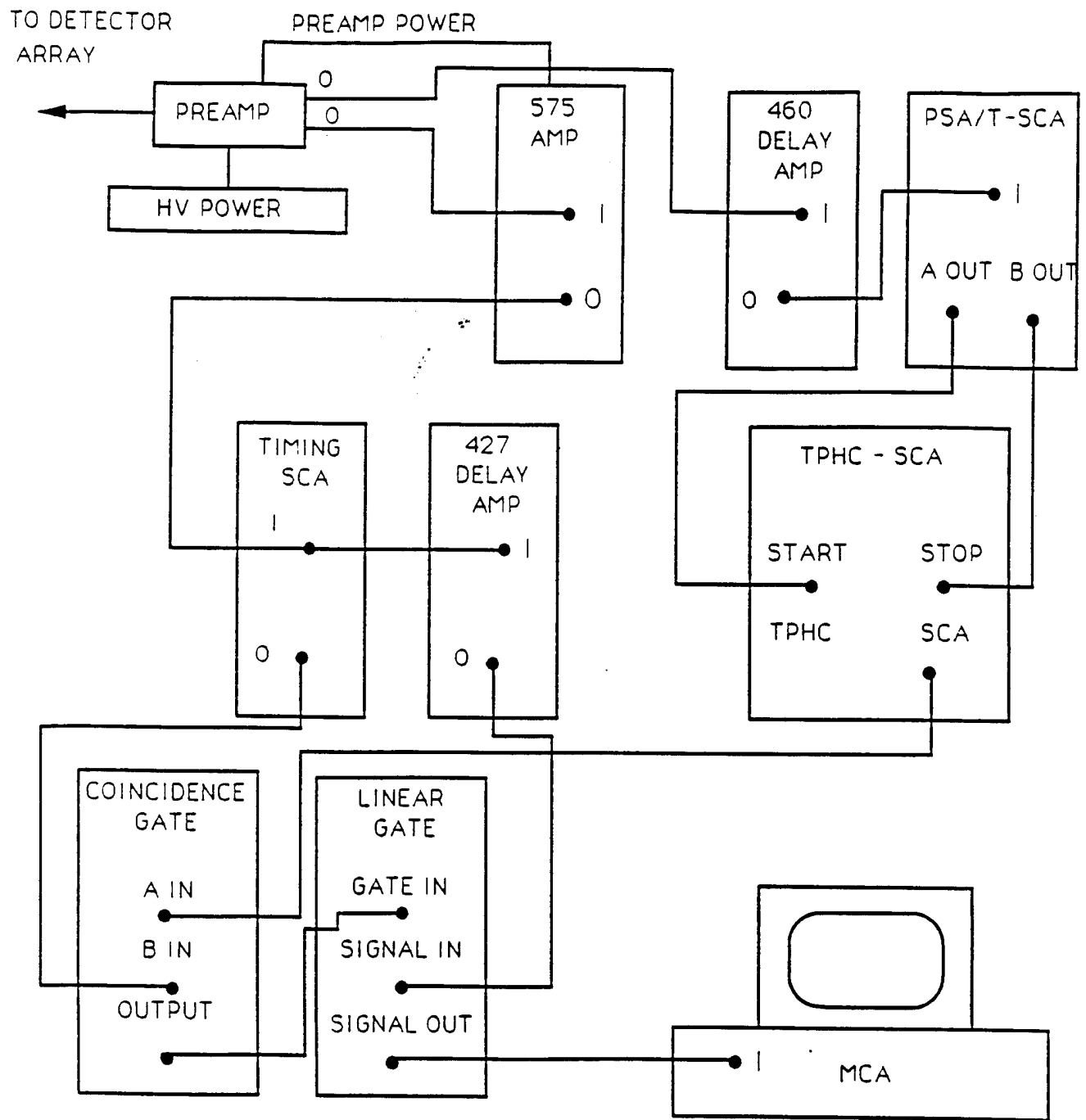
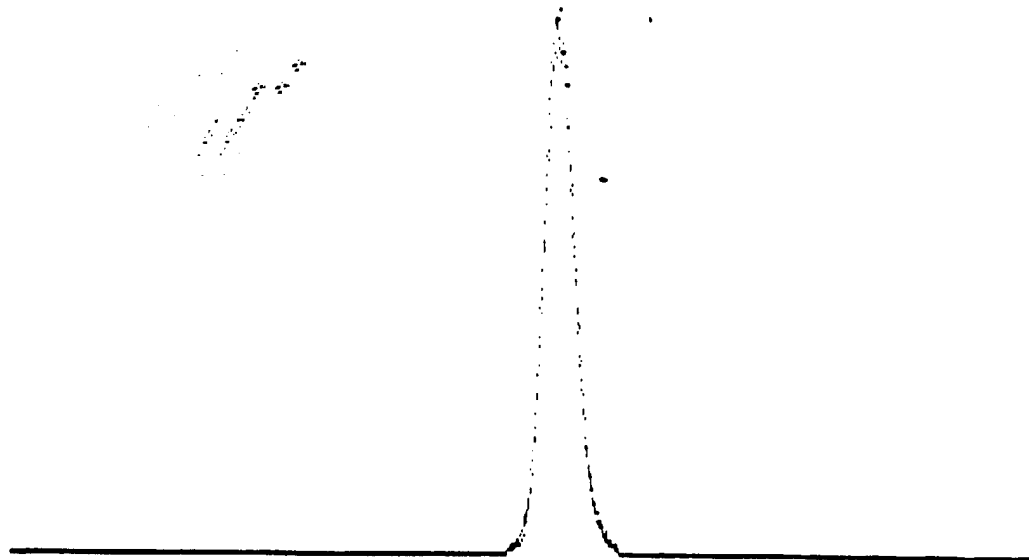


Figure 3. Schematic of Two-Fold Logic Pulse-Analysis System.

AUG 03 1989 10:24:48 AM MODES: **DATA** **ADT** A DEAD TIME 00  
 GROUP: F US: 2K CT5 GAIN: 3048 CHLS OFFSET: 0000 CHLS ID: T FHC T SPEC

FUNCTION KEY

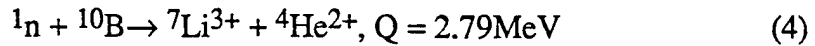
F1 OPTNS	F2 IDENT
F3 STORE	F4 LOAD
F5 <b>MODES</b>	F6 USER
F7 <b>CHRS</b>	F8 <b>FWHM</b>
F9 DOS	F10 MAIN



CHANNEL: 1885 COUNTS: 00000000 ROI #1: OFF ROI #2: OFF  
 PK #: 00 CTRD: 0.00000 CHL FWHM: 0.00000 CHL GROSS: 000000000 NET: 000000000  
 TIME LIVE PRESET: 000000 ELAPSED: 000060 REMAINING: 00 SECONDS

Figure 4. Time Distribution from the Pulse Shape Analyzer and Time-to-Amplitude Converter.

Now, it is important to note that the BF<sub>3</sub> detector response is insensitive to the incident neutron kinetic energy. That is, the measured pulse-height distribution does not give information about the incident neutron energy. The reaction on which the detector is based is:



Similarly, the cross section for this reaction is 3838 b for thermal (0.025eV) neutrons and is much less for higher-energy neutrons. For this reason, it is desirable but not always achievable to completely moderate the neutrons en route to the detector. In this particular detector system, the moderator, polyethylene, also acts as a weak neutron absorber. So there is a tradeoff between moderation and absorption. Preliminary experiments indicated that the optimal position for the detector tubes is near the inner edge of the annulus<sup>5</sup>.

In addition, for more energetic neutrons, the detector response is not very sensitive to the additional kinetic energy carried by the neutrons, because the Q-value is rather large. One or two collisions of a several-MeV neutron with protons in the moderator are sufficient to reduce the kinetic energy to well below the Q-value. The advantage of this property is that the detector response is insensitive to differences between the energies of the neutrons emitted by the calibration source and by the process in question, i.e., the process indicated by Equation 1. Also, since the low-pressure BF<sub>3</sub>-filled detectors are operated at relative low biases, e.g., 1200 V, they are less subject to spurious signals than higher-pressure tubes, which must be operated at significantly higher biases. Thus, the low-pressure BF<sub>3</sub>-filled detectors provided the basis for a reliable neutron detection system.

However, as the efficiency measurements indicate below, the addition of more BF<sub>3</sub> tubes to the detector semi-annulus did not fully compensate for the lower efficiency caused by using counters that had low gas-fill pressures. One solution to this problem was to use <sup>3</sup>He-filled detectors, which produced a distinct neutron pulse-height distribution. These detectors have large fill pressures that give a much higher neutron detection efficiency than the BF<sub>3</sub>-filled detectors can give. The <sup>3</sup>He-filled counters, however, are more expensive than BF<sub>3</sub>-filled tubes and they are more finicky to operate. For these reasons, we initially chose the BF<sub>3</sub>-filled detectors for the D<sub>2</sub>O fracturing experiment. The subsequent efficiency measurements made on the BF<sub>3</sub> system (see Section III-A) indicated that the sensitivity was not really adequate. Thus, we purchased six tubes filled to a pressure of about 4 atm of <sup>3</sup>He. Figure 2 shows a typical spectrum.

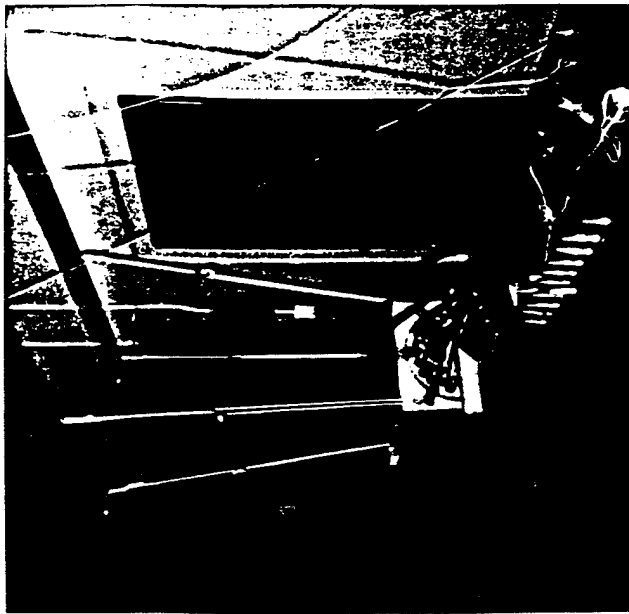
To further enhance the reliability associated with separating neutron-produced signals from spurious signals, we designed and set up a two-fold logic pulse-analysis system. This system, which Figure 3 shows schematically, requires that a pulse produced by the preamplifier pass both

energy and rise-time criteria to be counted as representing a neutron-produced interaction in the detector. Figure 4 shows a time distribution that resulted from pulse shape analysis. The events represented by the peak had associated pulse rise times that corresponded to neutron-produced events in the detector. Similarly Figure 2 shows a gated energy distribution. The gate requirement is that both the pulse height and the pulse rise time be consistent with those that a neutron interaction would produce in one of the tubes.

### B. D<sub>2</sub>O Ice Fracturing Apparatus

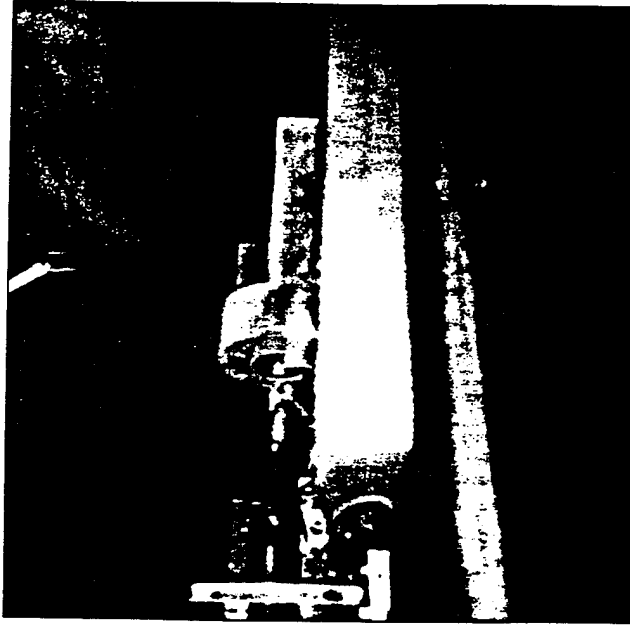
Fracture of solid tap water and solid deionized deuterated water occurred by impact of a WC-Co hemi-spherical tipped projectile onto a filled conical depression in a lead container. The depression in the lead container was filled with either liquid, and the container was then frozen in the freezer of a conventional refrigerator. The samples were left in the freezer for a minimum of three hours and a maximum of 72 hours, but typically for the shorter time. The samples obtained in this fashion were not bubble free. They ranged from largely clear to opaque. The cone was covered with a (~ 0.5 mm ) thin brass plate. Prior to firing a shot, the lead container was placed into an aluminum holder which was pre-cooled, then it was placed in a steel tray and covered with liquid nitrogen before firing. During this step, the majority of the samples exhibited severe cracking sometimes accompanied by a loud report. The aluminum holder was aligned by means of screws through the bottom of the steel tray and table upon which the gun was placed. The WC-Co composite impactor was embedded in a nylon container (total length of 60 mm) and projected by means of a gas gun (generally used in Hopkinson bar experiments). The velocities of the projectiles were between 100 and 200 m/s. The mass of the projectile was several grams. The gun barrel was 30 mm in (outside) diameter (20 mm inside diameter) and propelled using nitrogen gas. The pressure was released using a double (copper) diaphragm system.

The experiments consisted of projecting the impactor at high velocity past photodiode detectors located near the end of the gun barrel. These photo-detectors were intended to be used to measure the velocity of the projectile and to trigger the signal for the neutron counting system. But because they were unreliable, they usually were not used. The neutron detection system was suspended from supporting steel frame structure by means of ropes. This is shown in Figures 5 and 6. For most of the experiments, the timing "gate" for counting neutrons was opened for approximately 1.5 seconds. One set of experiments was run with the gate opened for approximately 5-6 seconds and another was run with the gate opened for approximately 100 seconds. Usually, background radiation was measured for 300 seconds between the impact



*Oct 10, 1989*

Figure 5. Neutron Detector in Place.



*Oct 10, 1989*

Figure 6. Projectile Impinging on D<sub>2</sub>O Ice Target.

experiments. Most of the experiments were run at 10 -15 minute intervals unless some technical problem arose.

After impact, the projectile recoiled and landed in front of the target. The brass cover usually remained intact, but had a hole the size of the projectile. The ice was totally crushed and shattered with some of the ice remaining at the bottom of the conical depression for at least several minutes after each firing.

### C. Titanium Fracturing Apparatus

The second of these experiments involved using a vibratory ball mill, model, make, specifications to fracture titanium shavings immersed in D<sub>2</sub>O. Specifically, this experiment was run by simply putting liquid D<sub>2</sub>O and titanium shavings along with several stainless steel bells of various sizes into a stainless steel vial. The vial was then fastened in the clamp of a Spex ball mill.

The neutron detector (the version containing six <sup>3</sup>He-filled tubes) was positioned over the ball mill with the same steel frame and rope arrangement used in the D<sub>2</sub>O ice fracturing experiment. The detector was placed as close to the vibrating vial as possible, and extra polyethylene was stacked around the mill to reflect source neutrons into the detector. The absolute neutron detection efficiency of the detector in this position measured with an Am-Be source placed in the stainless steel vial was 2%. Initially several runs were performed in which the ball mill was left on for long periods of time. For reasons mentioned below, these runs were unsatisfactory, a second set of neutron measurements was made with this system by turning both the neutron detector and ball mill on for periods of several minutes.

### III. Results

#### A. Neutron Detection Efficiency Measurements

Most of the detector system characterization experiments were done with a six-tube half-annulus. To understand how various geometrical configurations would affect the measured counting rates, we performed several simple experiments using an Am-Be source and a Pu-Be source. Figure 7 shows the source-to-detector configuration for these experiments. Table 1 gives the efficiencies for several different moderator configurations in which the source was located in the normal position (as indicated in Figure 6) and the detector system was positioned horizontally on a table top. These measurements indicate that reflectors can enhance the efficiency significantly. The Cd foil, which is used to absorb cosmic-neutrons thermalized in the shield, also reduces the detection efficiency. This effect indicates that more and better shielding were needed. In particular, several additional layers of polyethylene in which a boron-containing material was added between the layers to provide better shielding without diminishing the detection efficiency as much. Table 2 gives the detection efficiencies for a single detector tube in each hole in the polyethylene moderator. These efficiencies confirm that the moderator is uniform and that edge-loss effects are small. Table 3 gives the detection efficiencies for several source-to-detector distances, in which the detector system was positioned vertically on the floor. Notice that the efficiency at zero distance, 0.50%, is less than the efficiency that Table 1 gives for the normal configuration, 0.68%. This difference arose because the table top acted as a better neutron reflector than the concrete floor. As Tables 3 and 6 indicate, the product of distance squared times efficiency is relatively constant at sufficiently large distances. This result is expected, and it indicates that the detector system function is consistent with its physical construction, i.e., no unusual geometrical effects were observed. These same series of measurements were repeated for the six tube  $^3\text{He}$ -filled detector system, and the results are shown in Tables 4 through 6. Figure 8 shows a pulse-height distribution for  $^3\text{He}$ -filled detector system.

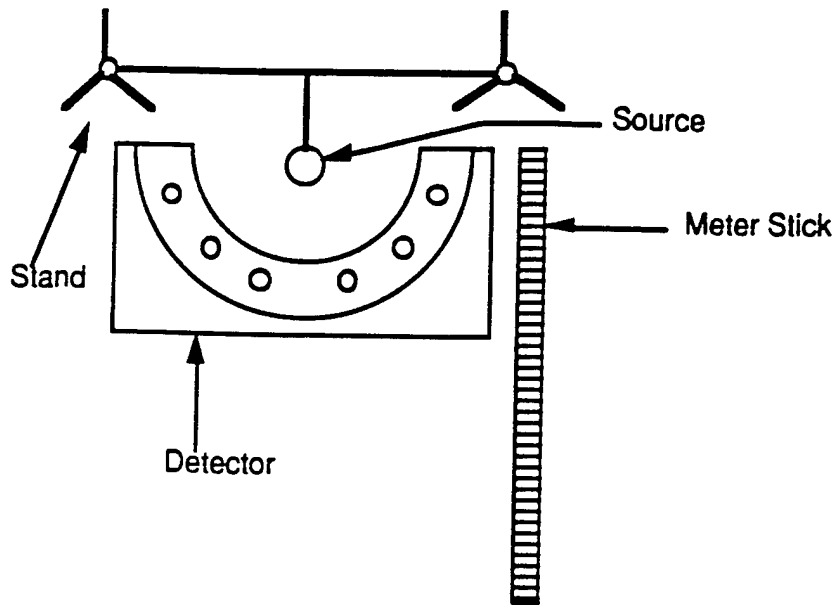


Figure 7. Source to Detector Configuration for Efficiency Measurements.

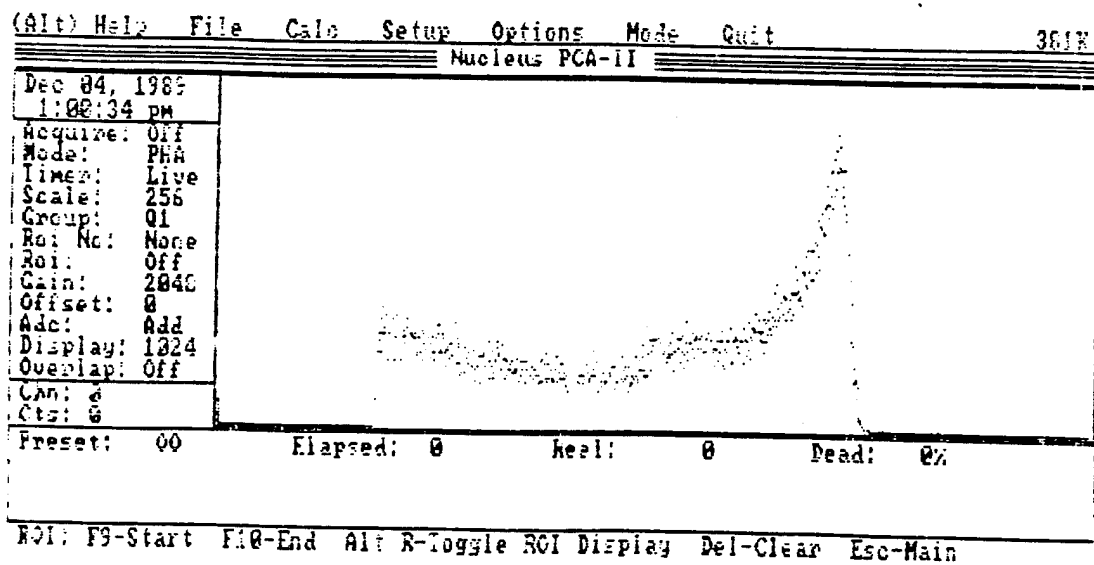


Figure 8. Pulse-Height Distribution for the Neutron Detector System Containing Six  $^3\text{He}$ -Filled Proportional Counters.

Table 1. Six-Tube BF<sub>3</sub> Efficiencies for Several Moderator Configurations

Condition	Counting Efficiency	$\epsilon^{a,b}(\%)$
Normal		0.68 <sup>c</sup>
Polyethylene Reflector in front of detector		0.97
Outer Polyethylene Shield Removed		0.58
Cd foil removed but polyethylene shield in place		0.83
Additional polyethylene shielding a in place (w/Cd)		0.85

<sup>a</sup> Source was Am-Be; neutron emission rate was 58,000 neutrons per second. Source position was the same for each condition.

<sup>b</sup> Statistical uncertainty is less than one percent.

<sup>c</sup> The eleven tube normal efficiency,  $\epsilon = 0.93\%$ .

Table 2. Efficiencies for a Single BF<sub>3</sub> Tube in Each Hole

Hole	$\epsilon (\%)$
1	0.11
2	0.13
3	0.12
4	0.12
5	0.12
6	0.09

$$\epsilon = \sum_i \epsilon_i = 0.69$$

Table 3. Dependence of the Detection Efficiency on Distance for the Six Tube BF<sub>3</sub> System Measured with Pu-Be Source (strength = 1.75 x 10<sup>6</sup> n/sec)

r(cm)	ε (%)	εr <sup>2</sup>
0	0.50	0
10	0.24	24
25	0.11	69
50	0.05	125
75	0.02	113
100	0.01	100

Table 4. Six-Tube <sup>3</sup>He Efficiencies for Several Moderator Configurations

Configuration	ε(%)
normal*	3.84
polyethylene reflector in front of detector	6.28
Cd foil removal but polyethylene shield in place	4.78
Cd foil removed and polyethylene reflector in front of detector	7.37

\*The detector had to be placed vertically on the floor rather than horizontally on a table top because the Au-Be source was strong enough to cause large dead times and pulse pile-up in the horizontal position.

Table 5. Efficiencies for a Single <sup>3</sup>He Tube in Each Hole

Hole	e(%)
1	0.68
2	0.78
3	0.70
4	0.81
5	0.74
6	0.62

\*<sup>3</sup>He-filled detector was placed horizontally on a table top for these measurements.

Table 6. Dependence of the Detection Efficiency on Distance for the Six-Tube  $^3\text{He}$  System Measure with Pu-Be Source

$r(\text{cm})$	$\epsilon(\%)$	$\epsilon r^2$
0	2.05	0
25	0.52	325
50	0.22	350
75	0.12	675
100	0.07	700
150	0.04	900
200	0.02	800

Comparing the numbers from Tables 1 and 4, one can see that the  $^3\text{He}$  system is about 5.6 times more efficient than the 6-tube  $\text{BF}_3$  system and 4 times more efficient than the 11-tube  $\text{BF}_3$  system.

### B. Neutron Background

Neutron background measurements were made over periods ranging from several hours to several days. They were made in the Co-60 Bay of the Penn State Breazeale Reactor (PSBR) Facility, in Room 9A of Hammond Building (where the air gun is located), and in the basement of the Co-60 Bay. Typically, the background rate in the PSBR Facility was found to range from 30 to 40 counts per minute, and in Room 9A it was found to range from 2 to 3 counts per minute ( $2.34 \pm 0.180$ ). The rate in Room 9A is about a factor of ten lower, because the room is located in the basement where a considerable thickness of concrete is overhead. In the basement of the Co-60 Facility where the Ti fracture experiment was set up, the background measured with the six-tube  $^3\text{He}$  detector was  $12.6 \pm 0.1$  counts/min. The neutron detection system can be turned on for arbitrarily short periods of time to reduce the background rate. For example, if the nominal background rate is 5 counts/min and the system is turned on for 5 seconds, the average number of background counts during a single 5 second shot is  $5/\text{min} \times 1\text{min}/60 \text{ sec} \times 5 \text{ sec} = 0.42$ . So, an initial simple estimate of a small-but-measurable neutron detection rate is twice the background rate, which is about one neutron per shot.

### C. Ice Cracking Experiments

Table 7 summarizes the cracking experiments that were performed. Table 7 summarizes the data from the  $\text{D}_2\text{O}$  ice fracturing experiments. To understand these data, we must recognize that the time intervals during which the neutron detection system was turned on was very important. It is desirable to make this interval as small as possible but not so small that emitted

neutrons would be missed by the detection system. Typically we found that  $\Delta\tau \approx 1$  sec was a reasonable time interval. In principle, however, intervals on the order of 10-100 msec would be optimal because the intervals of this length would allow detection of any emitted neutrons while the system was turned on yet this short length of the time interval would minimize the number of background neutrons detected. To use a very short interval such as  $\Delta\tau \approx 100$  msec requires that a precision device be used to synchronize the projectile gun and the counter. Because such a device was not available within the constraints of the present study we had to use longer intervals, typically about 1 sec in length. Intervals longer than several seconds allowed too many background neutrons to be counted. Thus, only runs in which  $\Delta\tau$  was short provided an opportunity to observe neutron emission above the background. If we exclude the one case in which three neutrons were detected in one eight-second interval (Run 4), no positive evidence remains for the production of neutrons during ice fracturing.

Table 7. Summary of Experimental Runs for the Fracture of D<sub>2</sub>O Ice.

Run	$\Delta t$ (sec)	# Shots	Target	# Counts Observed	$R_t$ (counts/sec)	background <sup>+</sup> $R_b$ (counts/sec)
4	1.1a	20	D <sub>2</sub> O	5 <sup>a</sup>	$\sim 0.17 \pm 0.08$	$0.049 \pm 0.004$
5	1.1	22	H <sub>2</sub> O	0	0	$0.042 \pm 0.003$
3	5.8	17	D <sub>2</sub> O	1	$0.0095 \pm 0.0095$	$0.0395 \pm 0.0027$
1	5	7	D <sub>2</sub> O	0	0	-
2	100	13	D <sub>2</sub> O	56	$0.043 \pm 0.020$	$0.041 \pm 0.013$

<sup>a</sup>Three counts were observed in one 8 second interval. The other intervals were 1.1 sec. long; total time for this run was  $\sim 29.5$  sec.

+measured over longer time intervals in between fracture shots.

#### D. Titanium Fracture Experiments

Table 8 summarizes the results of the first set of ball-mill experiments. For the first two runs, the ball mill shut off after an unknown amount of time (because its motor overheated), but the detector system continued to collect data. Thus, if there were neutrons given off during these runs, they would have been obscured by the background. The titanium chips were turned into a powder by about the third or fourth run. A second set of runs were performed using fresh Ti and D<sub>2</sub>O, and the counting times and the associated shaking times were limited to several minutes or

Table 8. Summary of the First Set of Experimental Runs for Fracture of Ti Chips Immersed in D<sub>2</sub>O

Run	Time of Run (sec)	Counts	Rate (sec <sup>-1</sup> )	Background
1	64507	12432	0.193 ± 0.002	0.2
2	17388	3493	0.201 ± 0.003	0.2
3	3752	793	0.211 ± 0.008	0.2
4	4200	807	0.192 ± 0.007	0.2
5	4200	1022	0.243 ± 0.008	0.2
6	2110	419	0.198 ± 0.010	0.2

Table 9. Summary of the Second Set of Experimental Runs for Fracture of Ti Chips Immersed in D<sub>2</sub>O.

Runs	Time of Run (sec)	Counts	Rate (sec <sup>-1</sup> )	Comments
1	300	326	1.09 ± 0.06	mill off
2	300	312	1.04 ± 0.06	mill on
3	300	325	1.08 ± 0.06	mill off
4	300	290	0.97 ± 0.06	mill on
5	30	27826	929 ± 6	position 1, Am Be source, Eff.=1.6%
6	30	29711	890 ± 5	position 2, Am Be source, Eff.=1.5%
7	30	30946	1032 ± 6	position 3, Am Be source, Eff.=1.8%
8	30	31354	1044 ± 6	position 3, Am Be source, Eff.=1.8%
9	60	62544	1043 ± 4	position 3, Am Be source, Eff.=1.8%
10	60	62661	1044 ± 4	position 3, Am Be source, Eff.=1.8%
11	60	71154	1186 ± 4	position 3, w/poly Am Be source, Eff.=2.0%
12	60	70538	1176 ± 4	position 3, w/poly Am Be source, Eff.=2.0%
13	72100	15173	0.210 ± 0.002	Background
14	25362	50118	0.195 ± 0.001	Background
15	15605	2418	0.187 ± 0.003	Background
16	64507	12432	0.193 ± 0.002	Ti + D <sub>2</sub> O, mill on
17	17388	3443	0.201 ± 0.003	Ti + D <sub>2</sub> O, mill on
18	3752	793	0.211 ± 0.008	Ti + D <sub>2</sub> O, mill on
19	4200	807	0.192 ± 0.007	Ti + D <sub>2</sub> O, mill on
20	4200	1022	0.243 ± 0.008	Ti + D <sub>2</sub> O, mill on
21	2110	419	0.198 ± 0.010	Ti + D <sub>2</sub> O, mill on

less to prevent pulverization of the Ti chips. Table 9 summarizes the results of these experiments. Runs 1-4 were used to determine the effects of microphonics. Because the running of the unit did not cause the counting rate to increase, it was clear that microphonics were not a problem. Runs 5-

12 were used to measure the neutron detection efficiency. The results were reasonable for  $^3\text{He}$ -filled detectors. As expected, the results indicated that the detection efficiency was sensitive to the source position and the presence of a moderator, i.e., polyethylene plate. Runs 13-15 were used to measure the neutron background, and they were made over a period of several days. The observed fluctuation are reasonably small and consistent with our previous experience. Runs 16-21 were the actual "shaking" experiments. None of these runs yielded neutron detection rates that were significantly greater than the average background rate. Thus, this series of experimental runs yielded no positive results.

#### E. Microwave Plasma Enhanced Cracking of Deuterium-Loaded Titanium

An experiment that used a microwave-plasma-assisted chemical vapor deposition system was performed. Because Ti can be loaded with large amounts of hydrogen or deuterium, deuterium-loaded Ti that was carbonized was a good choice for a cracking experiment. In particular, titanium can incorporate as much as 50 atomic percent of H and the  $\text{TiH}_2$  phase does exist. Dissolving D in Ti and carbonizing the Ti was performed in a microwave plasma. This process produced a high density of cracks in the material. This particular process also has the advantages: (1) a stable D plasma was easily attained, (2) Ti was heated by the plasma and the temperature was controlled by the  $\text{D}_2$  pressure and the magnetron power, and (3) CVD reactions of Ti with  $\text{CH}_4$  are controlled by the  $\text{CH}_4/\text{D}_2$  ration, temperature, and time.

With this information in mind, several experiments were performed. The neutron detections system, which had  $^3\text{He}$ -filled proportional counters, was mounted within several centimeters of the microwave cavity. Table 10 summarizes the experiments performed. In addition to the experimental runs in which various plasma conditions were present, several measurements of the background were made. The primary question was to discuss whether the various power supplies and other electrical equipment were giving rise to spurious signals. The levels of electronic noise were sufficiently high in this particular laboratory that the delay-line amplifier, which was part of the pulse shape analysis part of the detection system, could not reject electronic noise and would not operate properly. Thus, we only used energy discrimination as the only means of rejecting spurious counts produced by noise. The series of background runs indicated a large number of events in the low-energy region of the neutron energy distribution. These events appeared with different frequency depending on the other experimental conditions, i.e., whether other equipment such as power supplies were turned on. As a result of this condition and not being able to totally reject noise-produced events, the background counts did not give consistent results. Similarly the runs in which the plasma was being produced and subsequent

Table 10. Summary of the Experimental Runs for the Plasma Enhanced Cracking.

Run	T °C	Gas	Flow (SCCM)	Counting	Time (sec)	Counting Rate (counts/sec)	Remarks
<u>July 2, 1990</u>							
1. Annealing	950	D2	5	1917±44	3600	0.532	
2. Annealing	1185	D2	5	1351±37	3600	0.375	
3. Background	25	D2	0	1378±37	3600	0.382	
4. CVD	1035	CH4/D2	5/95	1788±42	3600	0.496	
5. Background	25	CH4/D2	0/0	1423±38	3600	0.395	
6. CVD	1190	CH4/D2	5/95	1307±36	3600	0.363	
7. CVD	1050	CH4/D2	5/95	1427±38	3600	0.396	like run 4
8. Annealing	1050-1240	D2	pressure 80-425 Torr	1010±32	2527	0.399	
9. Background	25	D2	0	1329±36	3600	0.369	
<u>July 5, 1990</u>							
10. Background	?	Vacuum power	0	3817±62	3095	1.233	sample in vacuum power corresponding run 6
11. Background	25	Vacuum no power	0	577±24	871	0.662	
12. Background		Vacuum power no sample	0	4207±65	3600	1.168	power corresponding run 6

cracking was taking place did not show any evidence for neutron production. However, examination of the Ti strip after the experiments were completed indicated that the Ti strip was brittle and easily disintegrated. These observations indicated that the cracking process in the plasma was indeed successful. Therefore, this series of experiments was inconclusive.

#### F. Statistical Model of Background Detection

To analyze the case of separating purely background neutron counts from neutron counts produced by both the background and the process in question, we used the Poisson distribution:

$$P(x) = \frac{\lambda^x e^{-\lambda}}{x!}, \quad x = 0, 1, 2, \dots$$

in which  $x$  refers to a specific number of counts observed and  $\lambda$  is the average number of counts. When  $\lambda$  is small compared to unity, the Poisson distribution is a good approximation to the exact model of nuclear processes, namely, the binominal distribution. In the case of interest, the average number of counts  $\lambda$  is indeed small. To represent the average number of background counts, we take  $\lambda = R_b \Delta t$ , in which  $R_b$  is the background rate in counts/sec and  $\Delta t$  is the time interval in sec that the detection system is turned on.

As Table 7 indicates, the neutron detection rate for the ice-cracking experiments was very low. Typically in a series of twenty shots, perhaps only one or two neutrons were detected. That is, during most shots, no neutrons were detected. To test whether this observation was reasonable, we considered the background rate  $R_b = 0.04 \text{ sec}^{-1}$  and a time interval of  $\Delta t = 1 \text{ sec}$ . These conditions give  $\lambda_b = 0.04$  neutrons. We sampled the Poisson distribution by drawing random numbers that reflected the shape of a Poisson distribution in which  $\lambda_b = 0.04$  neutrons. Each sampling or computer trial yielded a small number, either zero, or one, or two, or three. A tally of twenty trials was a simulation of one of our experiments that consisted of twenty shots. As shown, we found that strings of many zeros were not uncommon. This simulation confirmed the observed experimental effect, namely that the background produced very few non-zero counts per shot (i.e.  $x = 1, 2..$ ).

To develop a more sophisticated model, which was also a means of testing the Soviet claim, we sampled two Poisson distributions, one that represented the effects of the background only and one that represented the effects of a "real" process on which the background effect was superposed. The former was characterized by  $\lambda_6 = 0.27$  neutrons or true (background plus true neutrons from fracture process) process and the latter was characterized by  $\lambda_t = 0.54$  (These

values approximately represented the values of the background and total rates that the Soviets claimed.) After a sample of integers,  $X_i$  ( $i = 1$  to  $s$ ), was sampled from the distribution representing the real process, the average was calculated:

$$\bar{N}_t = \frac{1}{s} \sum_{c=1}^s X_i$$

The sample size  $s$  was chosen to correspond to the number of experimental shots. Similarly after an array of  $s$  numbers,  $Y_i$ , was sampled from the distribution representing the background, the average

$$\bar{N}_b = \frac{1}{s} \sum_{c=1}^s Y_i$$

was calculated. Thus  $\bar{N}_t$  and  $\bar{N}_b$  simulate an experiment in which  $2s$  fracture events (shots) were performed impairs, one with the D2O sample exhibiting fractoemission of neutrons the other a background with no sample. The difference  $\Delta_N$  of these two average values is the random variable that we tabulated:

$$\Delta_N = \bar{N}_t - \bar{N}_b$$

in which the subscript  $N$  refers to the net number of counts above background after completing  $s$  samplings (shots). By choosing  $s = 75$ , we simulated the Russian experiment. By repeating this experiment many times and tabulating the relative frequency of  $\Delta_N$ , we obtained, as expected, a Gaussian distribution for the random variable  $\Delta_N$ . We also repeated this computer simulation a second time, only this time we took both samplings from the background distribution (characterized by  $\lambda_b$ ). From this second simulation we tabulated

$$\Delta_B = \bar{N}_b - \bar{N}'_b$$

The prime indicates that the two sets of neutron counts used to obtain any value of  $\Delta_b$  were from independent sampling sequences. Again, we tabulated the relative frequency of  $\Delta_b$ .

Now if the sample size were very large, (egn  $\gg 75$ ),  $\Delta_N = \lambda_t - \lambda_b = .27$ . Since the sample size was relatively small, i. e., 75,  $\Delta_N$  and  $\Delta_B$  fluctuate. Repeating the computer simulation of the Russian experiment many times is a way of determining accurately the extent of these fluctuations. In other words, we are simulating the probability distribution of the neutron counts which would be obtained from a single experimental series of 75 shots assuming the neutron generation rates given in the Russian paper are correct. Figure 9 shows the results of the simulated computer experiments. We see that the centroid of the curve corresponding to  $\Delta_N$  is at 0.225.

This is the value expected for subtracting two random variables that are sampled from two distributions that differ in their corresponding mean values by 0.27. The shaded area of the former curve represents the probability that the background is mistaken for real effects. The shaded area of the latter curve represents the probability that the real effect appears as background. Thus we see that when  $\lambda_T \cong \lambda_B$ , it is easy to confuse the issue.

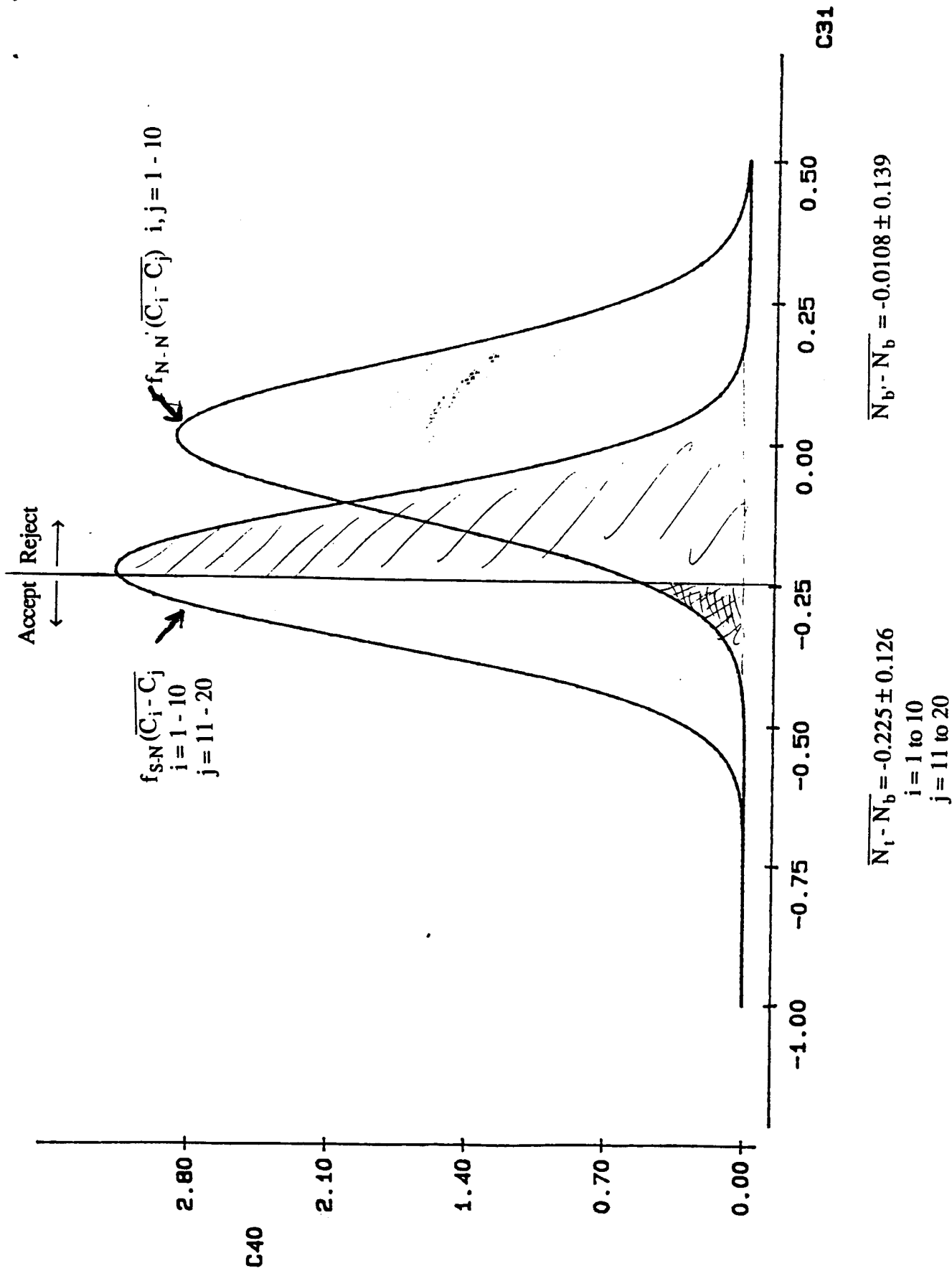


Figure 9 Test on Null hypothesis.  $H_0$ : Neutrons with Solid D<sub>2</sub>O in Projectile Beam are Real, i.e., from Mean<sub>1</sub> = 0.54 vs. alternative  $H_1$  Neutrons with Solid D<sub>2</sub>O in Projectile Beam are from background with mean<sub>b</sub> = 0.27

#### IV. Conclusions and Future Work

We developed and characterized a sophisticated neutron detection system that is based on both BF<sub>3</sub>-filled and <sup>3</sup>He-filled proportional counters. The initial measurements on D<sub>2</sub>O ice showed no neutron production above the average neutron background rate. The results of a statistical analysis, that used the Poisson model, are consistent with the measured neutron rates. These results suggest that the Soviet's published results<sup>1,2</sup> may be experimental artifacts.

Clearly, the absence of a statistically significant neutron production rate in the fracturing experiment is an inconclusive result. The two major limitations, which have prevented us from obtaining conclusive information, are (1) the inherently low efficiency of the neutron detection system and (2) the inability to fire a large number of shots with the available apparatus. Since we cannot change the latter limitation without obtaining more funds, we chose to address the former. To significantly increase the detection efficiency, we incorporated six <sup>3</sup>He-filled proportional counters into the detection system in place of the BF<sub>3</sub>-filled detectors. They increased the efficiency of the entire system by approximately a factor of ten. But, we did not have an opportunity to repeat the D<sub>2</sub>O ice fracturing experiment using the more-efficient <sup>3</sup>He-filled detectors because we did not have further access to projectile firing apparatus. However, using the detectors, we did perform an experiment in which titanium chips were fractured while they were immersed in D<sub>2</sub>O. This experiment showed no neutron emission above the background rate.

Also, the plasma enhanced-cracking experiments showed no conclusive results. Since these particular experiments involved sources of noise that produced spurious counts, very little reliable information could be obtained. Similarly, to perform a more sophisticated experiment of this type would require an extensive (and expensive) effort to reduce the effects of electronic noise.

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