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TITLE: TRITIUM GENERATION AND NEUTRON MEASUREMENTS IN Pd-Si UNDER HIGH DEUTERIUM GAS PRESSURE

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# TRITIUM GENERATION AND NEUTRON MEASUREMENTS IN Pd-Si UNDER HIGH DEUTERIUM GAS PRESSURE

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## INTRODUCTION

This paper summarizes some of the methods applicable for low level tritium detection needed in the search for anomalous fusion in metal hydrides. It is also intended to further detail our tritium and neutron results that have been obtained with the Pd-Si-D system, originally presented at earlier workshops [1,2]. A measure of reproducibility that was not evident in our previous work has been achieved partially due to the better detection sensitivity afforded by the use of low tritium deuterium and partially from the fact that the foil-wafer cells can be made with nearly identical electrical characteristics. This reproducibility has allowed us to narrow the optimum conditions for the experiment. While this experiment is rather different from the "standard" electrolytic cell[3,4,5] or the Ti gas hydride experiment[6], similarities exist in that non equilibrium conditions are sought and the tritium generation levels are low and neutron emission is extremely weak. In contrast to many electrochemical cell experiments, the system used in these experiments is completely sealed during operation and uses no electrolyte.

The major improvements to the experiment have been the use of very low tritium deuterium for the hydriding and the replacement of the aluminum neutron counter tubes with ones of stainless steel. These changes have resulted in pronounced improvements to the detection systems since the background tritium level in the gas has been reduced by a factor of 300 and the neutron background has been decreased by a factor of 14.

## MATERIALS

The detailed analysis of our materials has been described previously. The Y12 deuterium gas had tritium levels of 110 to 17 $\mu Ci/m^3$ and was 99.3% pure. Major impurities found in the $D_2$ are $H_2$ (0.6%), $H_2O$, $O_2$, CO, $CO_2$ and $N_2$ ( < 0.1%). The new deuterium from Cryogenic Rare Gases has a tritium level less than 0.15 $\mu Ci/m^3$ and is said to be 99.995% pure in aluminum cylinders. To assess the tritium level in the high purity gas it was necessary to combine the deuterium with

oxygen on a palladium catalyst, collect the water and count the colorless fluid in a Packard scintillation counter as described later.

The palladium powder was obtained from Englehard and formed by precipitation from an aqueous solution of $Pd(NH_3)_4Cl_2$ using reagent quality chemicals. This process results in an powder composed of small (0.3 to 0.5 $\mu$m) spheres that form chains or agglomerates up to 30 $\mu$m in dia. The raw material was said to be virgin sponge obtained from a South African mine. The major impurities in the palladium are oxygen (980 ppm) Chlorine (80 ppm), Nitrogen (65 ppm) and Carbon (47 ppm), all other major impurities are (each) under 35 ppm by weight. A total of 512.7 g of palladium powder has been used in the experiments described in this paper, of that amount, 87.3 g was used in various control experiments to test for tritium contamination. Palladium powder was not reused in experiments once it had been removed from a cell. A total of 43.2 g of palladium foil from Johnson and Matthey was used in the foil cells, 0.44 g of this foil was checked for tritium contamination by dissolution[7]. The 220 micron thick foils were laser cut and then annealed at 850 C for 2 hours at $10^{-6}$ torr. After the dehydride, the foil was reannealed at 850 C and reused. These foils have been hydrided, dehydrided and annealed seven times and show neither a monotonic decrease or increase in tritium production.

Tritium contamination in the palladium was tested by three independent methods, dissolution and scintillation counting, hydriding and dehydriding and suspension in a scintillation gel. By these means we can assign an upper limit on tritium contamination of 0.02 nCi/g (ie. no tritium detectable within experimental error). In addition, since the powder was obtained from a large bottle by pouring, one would expect that if the bottle was contaminated we would find that the tritium production would be dependent on the amount of palladium used in the experiment. In fact, the two cells with the largest palladium loads are among the cells with the smallest excess tritium.

The silicon powder size distribution and morphology has been discussed previously[1], however, we are now using a monosized, sieved, intrinsic silicon with a particle size of 10 to 20 $\mu$m. Added to the silicon powder was 3% (wt) of either PVA or Dow XUS 40303 binder. These binders and ethanol solvents were tested for tritium contamination by dissolving 132 to 460 mg of binder and solvent in water and placing the resultant mixture in a scintillation cocktail. No tritium could be detected over background by this method. Also, no counts over background could be detected when the silicon powder (44 mg) was suspended in a scintillation gel and counted.

In some cells, Sb doped silicon wafers (0.01 ohm-cm in resistivity by 0.5 mm thick disks, 3.07 cm dia.) obtained from Monsanto were used. Between the silicon wafers would be placed the 220 $\mu$m thick palladium foil. Because of surface roughness, the plates would only touch over a small fraction of their surface area.

Four types of cells have been made, those with palladium powder and silicon powder, those with palladium foil and silicon powder, those with palladium foil and silicon wafers and one with palladium foil and silicon powder. A typical cell, made with powders, might contain 12 to 21 grams of palladium in eight layers (one to two grams per layer) and 6 to 8 grams of silicon distributed between seven layers. Silicon layers are typically 0.76 to 2.15 mm thick by 3.17 cm in dia. while the palladium

layers vary from 1.16 to 0.48 mm thick by 3.05 cm in dia. for different type cells. The palladium powder was pressed (11.2 MPa, 2000 psi) into disk form and then oxidized, in air, at 350 C for 2 hours (weight gain of 0.37%). Layers of alternating palladium disks and silicon powder were then pressed into a ceramic form at a pressure of 11.2 MPa resulting a density of 26% and 68% of theoretical density for the palladium and silicon respectively.

## TRITIUM MEASUREMENT TECHNIQUES

Various techniques exist to detect tritium in the environment and in samples with a very high sensitivity. These techniques have been condensed and adapted from reference 8 and are compared and summarized in Figures 1 and 2. Since the morphologies for liquids and solids vary widely, the data in the Figures 1 and two are only approximate.

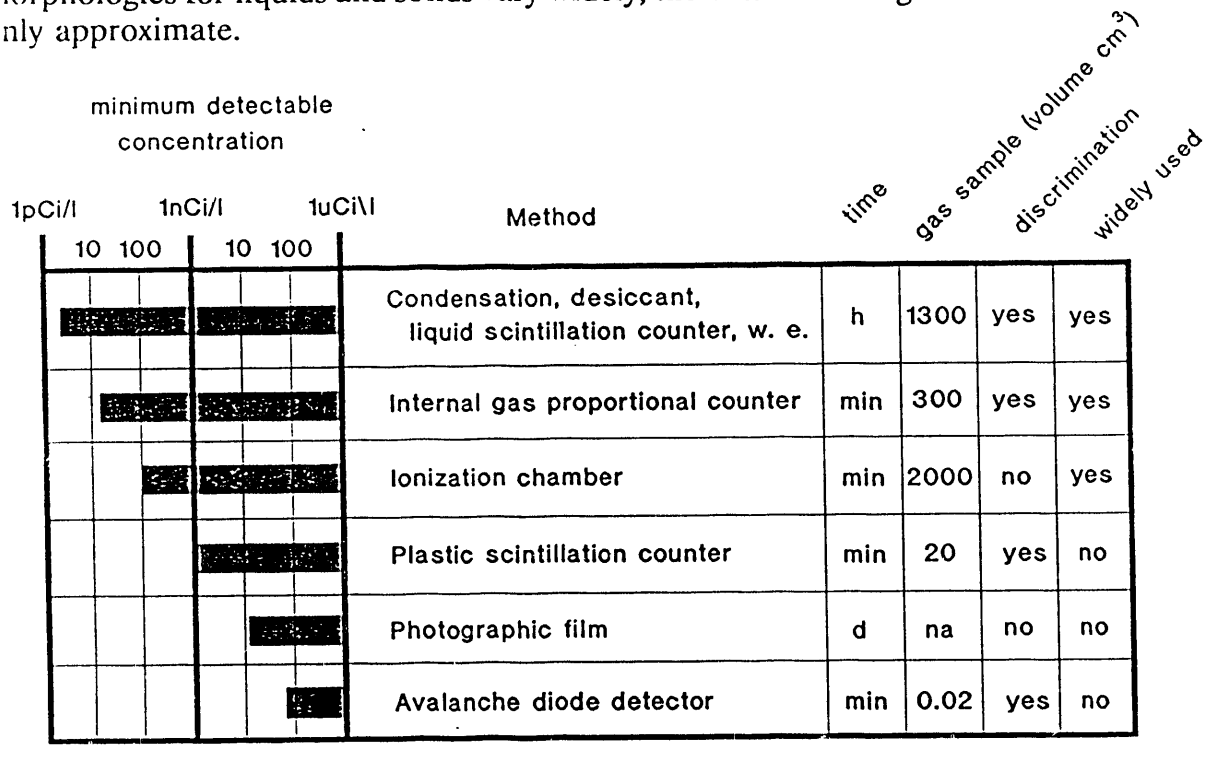


Figure 1. Comparison of tritium detection methods for tritium in gases.

Tritium detection in gases is easily accomplished by the oxidation of the hydrogen and collection of the tritiated water vapor for counting in a scintillation counter. The gas proportional counter is almost as sensitive as the scintillation gauge but involves more effort and chance for contamination than the ionization gauge. The advantage of the ionization gauge over the proportional counter and the scintillation counter is that it is fast, uses no counting gas (which can introduce $^{14}C$ and $^{85}Kr$ isotopes to the chamber), and samples do not need to be handled as in the case of the scintillation counter. Scintillation counting, if done carefully, can take days from the time of sample collection. The other techniques listed in Figure I are too insensitive for consideration.

To detect tritium in liquids one typically uses a scintillation counter (such as the Packard CA 1600)[9] and if additional sensitivity is required for water enrichment by electrolysis at 10 °C will result in enhancements of up to 70 times the original

concentration. The most sensitive method relies on the buildup of $^3He$ over a period of months and the subsequent detection of the He by a mass spectrometer. For rapid sample turnaround this method is obviously impractical. No other methods than the scintillation counter need be considered for liquids.

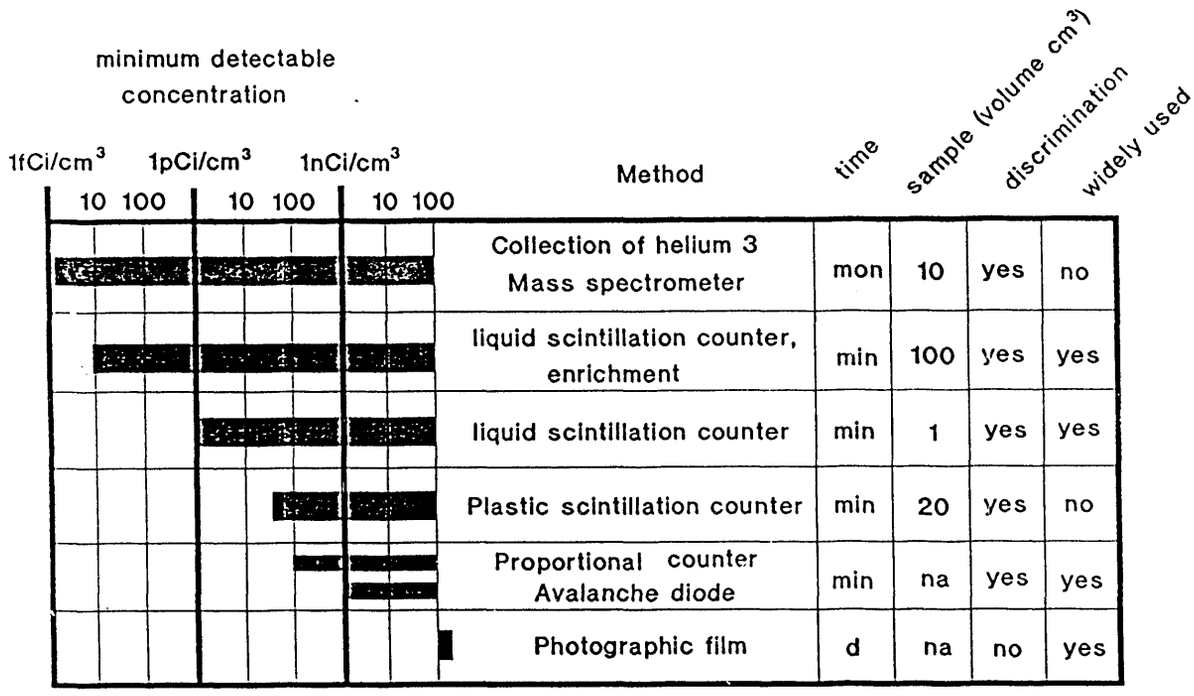


Figure 2. Comparison of tritium detection methods for tritium in liquids.

To detect tritium in solids, a variety of techniques may be applied depending on sensitivity and sample size and sample condition desired at the end of the experiment. If the sample is a powder, suspension of the dilute powder in a scintillation gel will give good results although for very opaque specimens misleading results. For opaque specimens an increase in sensitivity can be achieved by the use of scintillating inserts in the vial which effectively increase the surface area measured. Gas flow proportional counters[10] are the most sensitive method for measurement of small samples if the samples are to be tested non destructively. Semiconductor avalanche detectors[11], photographic film[12], and the measurement of beta stimulated X rays are less sensitive methods.

## APPARATUS

The primary tritium measurement device used in this study was a two liter ionization gauge in a stainless steel recirculating gas loop containing a 310.9 cc calibration volume. Gas ionization gauges are used extensively in the detection of tritium and have been shown to be stable, reliable and sensitive[13]. The instrument rejects pulse type radioactive events which effectively discriminate against radon and cosmic ray ionization. The instrument showed good stability in measuring the tritium background in the deuterium gas in over a year of operation with several different bottles of deuterium (maximum deviation during a year of ± 2.5%). Shown in Figure 3 is the tritium measurement time history for all background tritium measurements made with the Overhoff ionization gauge. The tritium level varies from 4 to 66 $\mu Ci/m^3$ and is relatively constant for a bottle unless the system is

cleaned. Because of tritium adsorbed on the analysis system walls, the minimum background for the Overhoff is effectively 3-4 $\mu Ci/m^3$. This memory effect is commonly found for instruments that have been exposed to tritium containing gases for long periods.

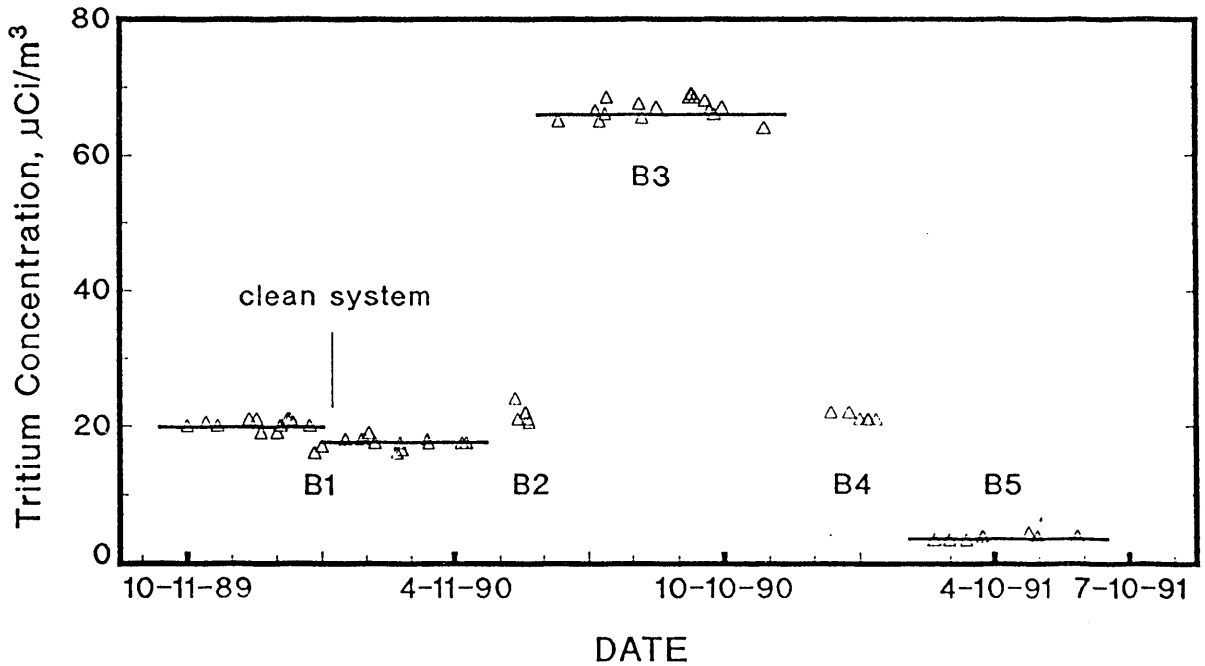


Figure 3. Stability of background tritium measurements for various bottles of deuterium gas.

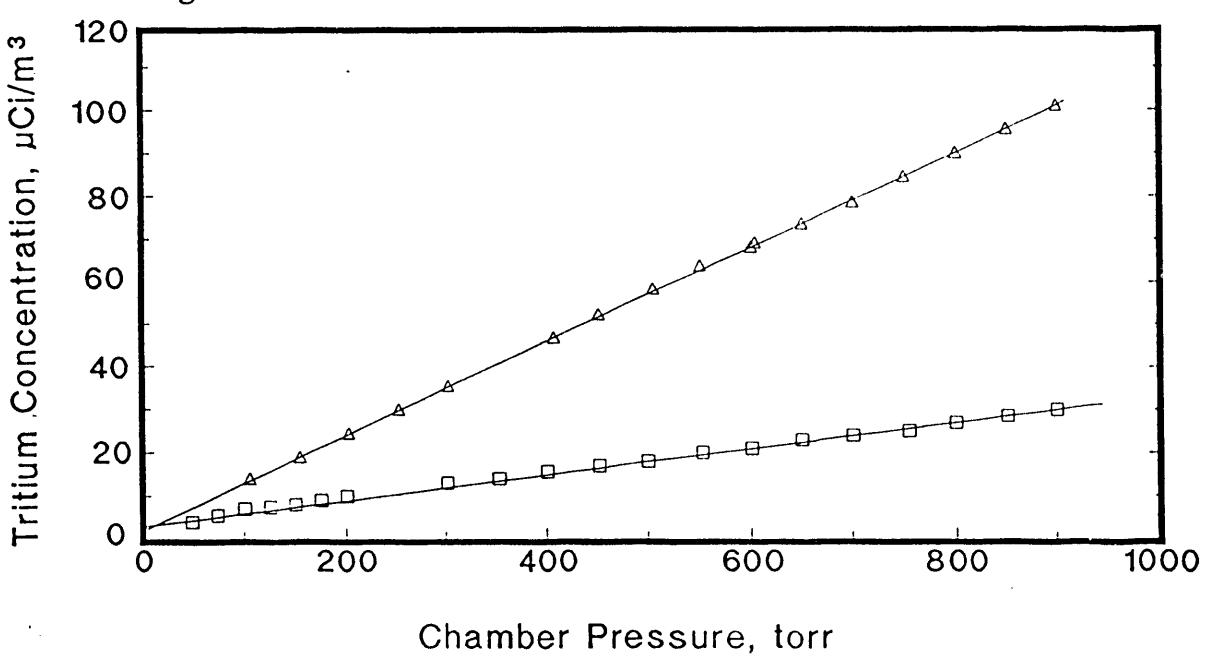


Figure 4. Linearity of Overhoff tritium ionization gauge to trace amounts of tritium in deuterium over a wide pressure range.

Figure 4 shows the linearity of the instrument to two concentrations of tritium in deuterium as a function of chamber pressure. Offsets near zero pressure

correspond to ionization currents caused by small amounts of adsorbed tritium on the chamber walls. Absolute calibration was accomplished by inserting the chamber into a circulating loop containing a standard, calibrated ionization gauge while circulating various concentrations of tritium enriched deuterium. Calibration was performed at twelve points from 498 $\mu Ci/m^3$ to 114 $\mu Ci/m^3$. Sensitivity to air contamination was checked by comparing the response of the meter with deuterium and with deuterium mixed with small amounts of air, no difference was found within experimental error. The response of the gauge to a deuterium water vapor mixture was also measured showing negligible effect at low water concentrations ( <38 torr).

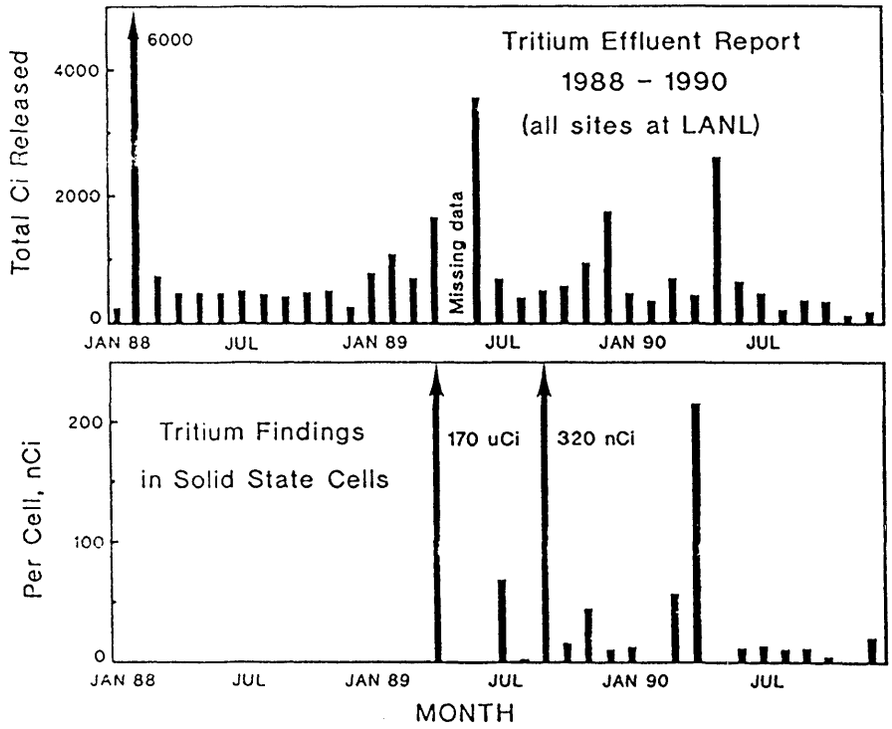


Figure 5. Controlled and accidental tritium releases at Los Alamos and tritium measurements from solid state cells.

As previously discussed, precautions were taken to mitigate the possibility of tritium contamination of our materials. The major precaution was to prepare the samples in a tritium free laboratory, seal the cell and then move it to the filling area which is in a tritium handling area. The cell would be attached to a vacuum system and then opened to a vacuum. Therefore, the inside of the cell was never exposed to the atmosphere in the tritium laboratory. In any case, the atmosphere in the laboratory was not a factor since it was always much less than the background found in the deuterium. In addition, we have compiled the tritium release data for Los Alamos, this data is shown in Figure 5 where it is seen that there is no correlation with the three large releases, which are all accidental and hence unpredictable, with our tritium findings. The fabrication area is also located 3.7 km from the nearest tritium handling facility. All materials handling and assembly work was done with disposable latex gloves and paper bench liners.

The neutron detection equipment is similar to that used by Menlove and has been thoroughly described elsewhere[14,15] The main feature of the counter and

electronics are that they provide data on neutron totals (total counts accumulated in a specified time) and a number designated as reals which are correlated neutron counts in a 128 $\mu$s gate. The reals counts are indicative of a neutron burst. The counters and tube enclosures are environmentally hardened and have shown excellent totals stability in the underground environment in over a year of operation. Two significant modifications to the counter have been made in the past year. First the aluminum counter tubes have been replaced by low background stainless tubes and the counter has been segmented with the addition of a second set of coincidence electronics.

To illustrate the magnitude of underground backgrounds, in Menlove's underground laboratory[15], the average background totals rate and correlated count rate was 870 ±6 c/h (24 hr) and 3.6 c/h (for correlated counts) while at the environmentally controlled underground tunnel (1) (15 m deep), we found 701 ±6 c/h and 0.6 c/h while for the still deeper tunnel (2) (70 m, overburden density ≈1.9 g/cc) the rates were 637 ±6 c/h and 0.15 c/h. When the stainless tubes were installed the background totals dropped to 44 ± 2 c/h and 0.14 c/h for tunnel 2 as shown in Figure 6.

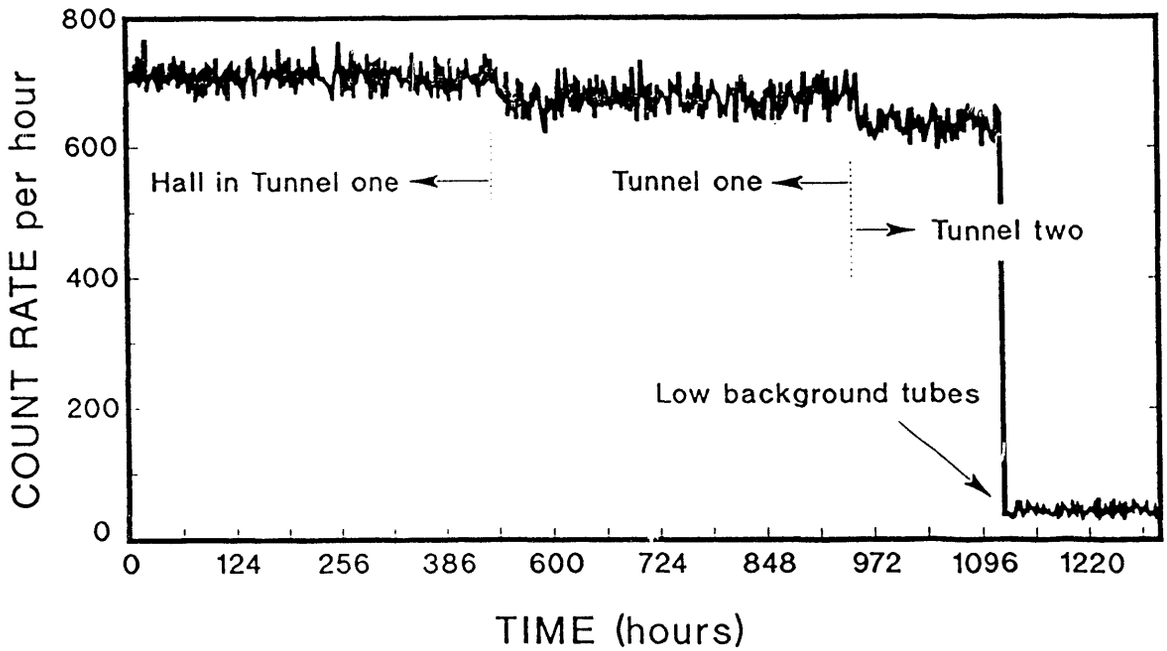


Figure 6. Neutron background summary for two tunnel locations and effect of low background tubes.

The channel counter was calibrated with a $^{252}$Cf source (average energy ≈ 2.3 MeV) both in a cell body and in the open counter. The efficiency was 18.5% for the bare source and 20.0% with the source in the stainless body which has large flanges which reflected the neutrons, subsequently increasing the efficiency.

## PROCEDURE

The procedure for hydriding a cell was to first measure the background tritium concentration in the deuterium fill gas. Then the loop and cell were evacuated and the tritium analysis loop was filled with fresh deuterium gas at a

known pressure less than 1000 torr. The tritium level could therefore be measured in this gas again. The cell was then opened and allowed to absorb the gas. Since the pressure in the analysis system and the volume of the system is known, an accurate measure of the gas absorbed by the cell could be made. Subsequent filling with higher pressures allow a determination of the amount of deuterium gas contained in the cell. A comparison of the predicted amount of gas that should be contained in the cell based on the free volume and the amount of palladium and pct curves agrees within 10 percent. The error is thought to be due to the imprecision of the pct curves at pressures greater than $7 \times 10^5$ Pa (100 psi)[16]. An additional advantage of this filling technique is that after the gas had been let into the cell the remaining gas could be checked for tritium enrichment or deficit. In all cases that were checked, we found that the deuterium remaining in the analysis system had the same tritium concentration the original fill gas in the analysis bottle to within experimental error ± 3%. After the cell was completely hydrided and removed from the loop, another check of the tritium background of the deuterium gas was made.

After the cell had been filled, it was placed in the neutron counter and a voltage of 200 to 2500 V generated by a Velonex model 360 pulse generator was applied to the cell. In typical operation, a unipolar, square pulse with a width of at least 150 $\mu$s at a repetition rate of 100 pulses per second was used at voltages as high as possible before breakdown occurred, typically 1200 to 2500 V. Currents of up to 5 A were used in some experiments. A minimum of 100 hours of pulsing was used, however in some cases the experiment was terminated earlier than 100 hours due to sudden electrical breakdown of the cell.

The gas analysis after the electrical pulsing was the reverse of filling. Background checks were made before and after the dehydride and care was taken to measure the volume of gas evolved from the cell. The cells initially were opened to the evacuated analysis system at room temperature but near the end of the dehydride the cells had to be heated to at least 125 C and then opened to the vacuum of the analysis system to release the remaining deuterium.

The procedure for the tritium detection by oxidation and subsequent scintillation counting was to first oxidize hydrogen from an uncontaminated cylinder and collect two, separate, 2 ml samples of water for use as background samples. Then the deuterium was oxidized from each of the $D_2$ bottles and again 2 ml samples were collected in two separate vials. Hydrogen was again oxidized in two more 2 ml samples to test for tritium holdup and then sample 36 was dehydrided.

One ml of each of the samples was then placed in 19 ml of Ultima Gold scintillation cocktail. These samples as well as standards ( two uncontaminated $H_2O$ samples, two tritium standards and two straight cocktails ) were counted for 100 minutes on three separate days. The data agree to within ± 2 sigma in all cases except the tritium standard which varied by 2.5 sigma. These results are shown in Figure 7 in order of analysis. No tritium was detected above instrument background for the standard water or hydrogen samples combined before or after combining the deuterium gas. We also show dehydride results for cell 36 (filled from the Liquid Carbonics cylinder), but because only 40% of the deuterium was recovered from the cell, we cannot draw a conclusive case for excess tritium in this cell even though the level was elevated over that of the fill gas by four sigma.

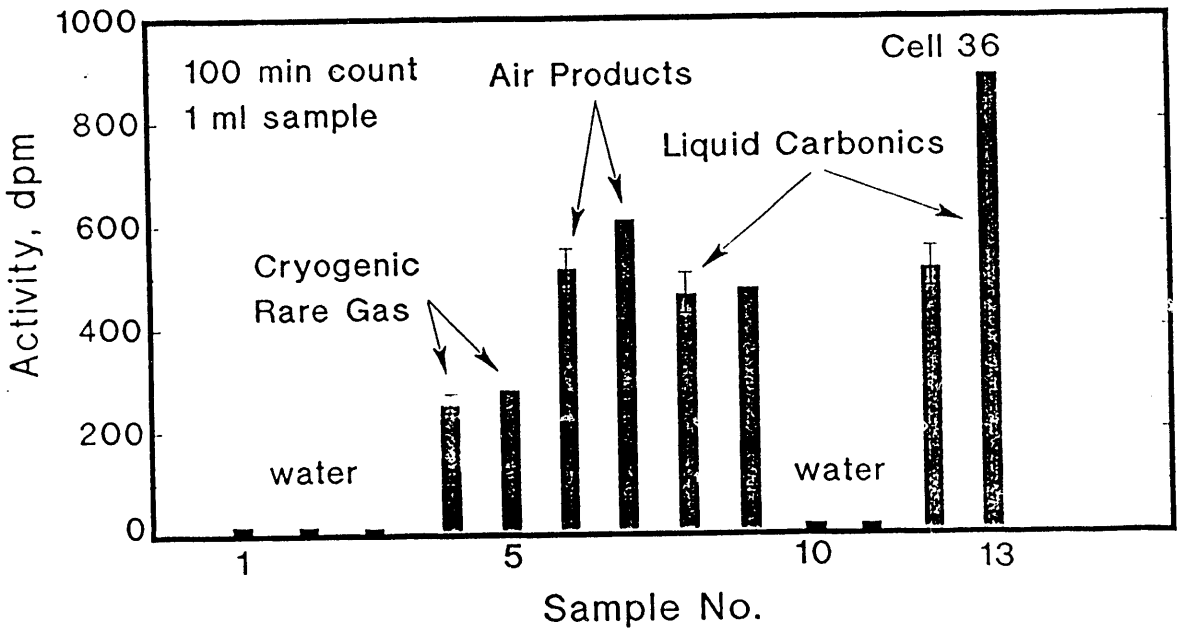


Figure 7. Tritium analysis of several bottles of low tritium deuterium and one solid state cell (36).

## RESULTS

### Tritium Measurements

Shown in Figure 8 is a summary of all the tritium results obtained after the cell designs had become stable and after the background tritium measurement technique had become standard. It is clear that the cells fall into definite categories with some exceptions. We find that the tritium output depends on current and that various types of cells have different efficiencies. A point worth noting is that the current density axis in Figure 8 is really current through the cell divided by the cell area (8 $cm^2$). In all cases, the actual area of contact between the silicon and palladium is a fraction of the 8 $cm^2$. Regardless of this, the contact for a particular type cell should be relatively constant within that cell type.

Our most reproducible cells have been the foil-wafer cells which can have nearly identical electrical characteristics from cell to cell. As can be seen in Figure 8, these cells show the least scatter of any cell type. Unfortunately, these also give the least amount of tritium generation.

Several cells showed current instability which was later determined to be arcing. When those cells were disassembled surface regions of the powder had been melted and pitted over as much as 50% of the surface of the palladium pellets. Arcing did not seem to be beneficial to higher tritium production. Cells that gave the most tritium did not show any obvious degradation due to the current flow.

A significant point to note is that the highest amount of excess tritium found for the foil-wafer cells was obtained with the low tritium deuterium and was obtained after the foils had been dehydrided and annealed 6 times. Incomplete dehydrides and separation effects would have given higher values with the deuterium containing larger amounts of tritium. Any tritium containing impurity would probably have been exhausted or depleted by exchange during the numerous anneals and hydridings.

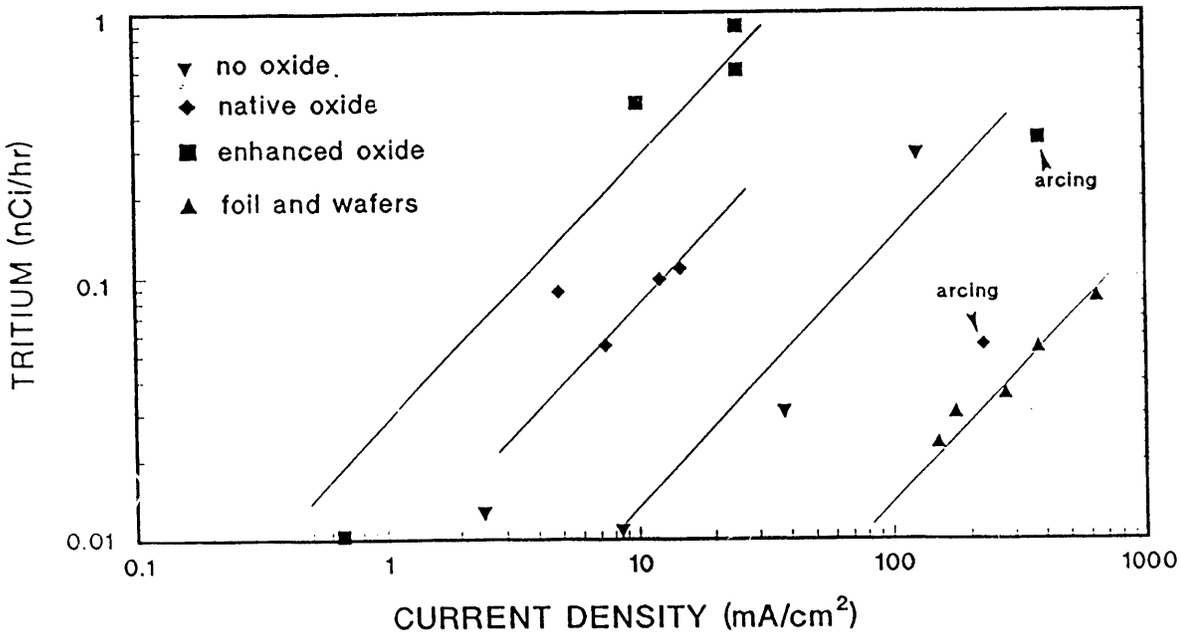


Figure 8. Tritium production of cells correlated with current density and cell type.

Greatest tritium generation rates have been achieved with powder-powder cells with oxidized Pd powder and voltages greater than 800 V at 0.1 A. Originally, it was thought that the binder used in these powder-powder cells had an effect, but it was shown that the only effect of the binder was to increase silicon uniformity and hence raise the breakdown voltage. In addition, the foil-wafer cells had no binder or oxidized palladium and yet produced measureable amounts of excess tritium.

Four hydrogen control cells have been made by either the usual method with layers between silicon or by simply pressing 12 to 30 g of virgin palladium powder into the ceramic form and then hydriding with hydrogen and subsequently dehydriding to test for contamination intrinsic to the palladium. All of these tests give a positive excess tritium result from 6 to 12 nCi total. This can be attributed to the effect of water and hydrogen gas in displacing small amounts of TDO from the ionization chamber and system walls. This TDO finds it's way to the ionization chamber and sticks in the chamber until the system is evacuated. Therefore, this "excess" cannot be readsorbed by the palladium bed as is the case with the tritium in the deuterium gas. These control cells also do not show the same dehydriding signature characteristic for the deuterided material. When palladium is deuterided and then dehydrided after a short period of time as seen for those cells that have shorted out (16, 23, 31), one finds either a very small excess tritium or none at all within experimental error.

Three cells 28, 33 and 35 have been run at low currents or high voltages for greater than 60 hours and then dehydrided. Small amounts of excess tritium or none within experimental error was found. These cells were then rehydrided and operated at higher currents for periods of up ot 300 hours with the result that up to 7 times more excess tritium was found after the longer runs at higher current.

An analysis of the dehydriding behavior of the cells reveals that in all cases the excess tritium is evolved when the palladium is dehydrided. If the tritium was slowly diffusing out of the container walls or ceramic insulator sleeve or other

materials, we would expect to find more tritium than we do in the gas overpressure. If the ionization species was not tritium gas but some other isotope (or even TDO,THO), we would not be able to reabsorb the tritium back onto the palladium bed reversibly as we are able to do with the deuterium containing samples.

While in the majority of cases the amount of excess tritium is small, in cell 20 the excess amount of tritium was 540 times the maximum amount found by the dissolution checks and it was 2.2 times the total amount of tritium contained in all of the deuterium gas used to hydride this cell. Figure 8 indicates that there is a wide range in tritium production rates. It is reasonable to assume that future improvements in the maximum rate will be possible. The greatest rate, achieved reproducibly, equates to a generation of $3.4 \times 10^6$ tritium atoms per second. Obviously if neutrons were generated at parity with tritium one would expect to easily detect neutrons.

## Neutron Detection

Our previous experiments[1,2] have indicated that there is an anomalously low value for neutrons detected to tritium produced ($\leq 4 \times 10^{-9}$). Since we have attained a reproducible but small tritium generation rate, we have been striving to make the neutron sensitivity equivalent to that of the tritium detection apparatus. We anticipate that our improved neutron sensitivity illustrated in Figure 6 will make it possible to detect a neutron signal that is unambiguously above zero.

Shown in Table 1 is a summary of all of the neutron data that have been collected since the counter was moved to the tunnels. Three locations are shown, with slightly different backgrounds. The first entry in Table 1 compares all tritium producing cells (in the group 17 through 24.5) with the current applied to the background which was composed of non tritium producing cells, hydrogen control cells and time when no current was applied to the cells. There is seen to be a slight excess of counts with this comparison. This can be compared to the other foreground background measurements of 24.5-29 and 30-35 where there is practically no difference between the foreground and the background and the tritium production is quite low compared to that of cells 17-24.5.

The column listed as reals/hr and total reals/hr differ in that the reals/hr only counts singles events (two neutrons detected). The total reals/hr includes all singles and higher multiplicity events. It appears that excess neutrons occur primarily as single neutron events (totals) and rarely as bursts (reals/hr). No bursts to rival the hundreds of neutrons detected from the titanium cells have ever been seen from these palladium experiments even though this particular neutron counter has been used for some of the titanium measurements that have detected large bursts.

The set of cells 24.5 through 29 was compared to two different backgrounds, one background was indicative of the neutron background with a deuterium containing cell in the counter but with the current off. The other background was obtained by including data from dummy (palladium, steel) cells. This background varies by more from the previous background than the variation in the foreground to the initial background. However, the total variation over some 2400 hours is, at most, only 0.4% indicating the excellent long term stability of these counters.

| Cell No's | Location | Conditions | Excess Tritium | Hours | Totals/hr | Reals /hr (Singles) | Total Reals/hr |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
| 17-24.5 | Center | Foreground (current applied) | 374 ± 20 | 810.6 | 707.9 | 0.441 | 0.635 |
| | Tunnel 1 | tritium producing cells only | | | | | |
| | | Background (no current) and | | 829.3 | 700.3 | 0.434 | 0.604 |
| | | non tritium producing cells | | | | | |
| | | | | | | | |
| 24.5-29 | Hall | Foreground(current applied) | 39 ± 10 | 837.5 | 713.9 | 0.607 | 0.99 |
| | Tunnel 1 | | | | | | |
| | | Background (no current) | | 958.8 | 712.9 | 0.541 | 0.735 |
| | | Background (no current) and | | 1560 | 710.8 | 0.576 | 0.799 |
| | | dummy cells | | | | | |
| | | | | | | | |
| 30-35 | Center | Foreground (current applied) | 65 ± 16 | 878.8 | 638.1 | 0.084 | 0.109 |
| | Tunnel 2 | | | | | | |
| | | Background and dummy | | 343 | 637.2 | 0.122 | 0.146 |
| | | cells | | | | | |

Table 1. Summary of neutron data for three tunnel locations and a comparison of tritium generation rates with the neutron output.

Care should be taken in the interpretation of this data (especially that of the reals) since the counters could be subject to drift over these long periods and a few very high correlated counts can skew the results significantly. Regardless of these caveats, this data is self consistent and consistent with our other measurements.

If the neutron to tritium partition ratio is $\approx 4 \times 10^{-9}$, this implies that we should be able to see a neutron totals of 5 sigma over background in 24 hours at a tritium production of 0.5 nCi/h with our new neutron background. From Figure 8 it can be seen that a tritium production rate of 0.5 nCi/h has been achieved several times. However, since these tubes were installed only a few months ago, we have been able to run one cell with the new tubes and that cell showed no excess neutron output and gave a tritium output of less than 0.01 nCi/h. Thus, at this point, the results from the new tubes are consistent with the neutron output.

## CONCLUSIONS

A reproducible method of tritium generation has been demonstrated. The tritium output scales with the current applied to various configurations of the cells. The tritium yield is found to depend strongly on the type of palladium metal used (powder or foil) and it may be expected that other parameters that have not been investigated thoroughly will have similar effects. Various tests for tritium contamination confirm that there is little chance of initial tritium contamination in the powder, foil or other materials used in this study. The tritium and neutron results are self consistent, and consistent with other reports. However, more sensitive neutron measurements are required to give a definitive neutron emission result.

## ACKNOWLEDGEMENTS

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## REFERENCES

1. T. N. Claytor, D. G. Tuggle, H. O. Menlove, P. A. Seeger, W. R. Doty and R.K.Rohwer, "Tritium and Neutron Measurements From Deuterated Pd-Si", AIP Conference Proceedings 228, Anomalous Nuclear Effects in Deuterium/Solid Systems, Ed. S. Jones, F. Scaramuzzi and D. Worledge, Provo Ut. 1990.

2. T. N. Claytor, P. A. Seeger, R. K. Rohwer, D. G. Tuggle and W. R. Doty, "Tritium and Neutron Measurements from a Solid-State Cell", LA-UR-89-3946, October 1989, Presented at the NSF-EPRI workshop.

3. S.E. Jones, E.P. Palmer, J.B. Czirr, D.L. Decker, G.L. Jensen, J.M. Thorne, S.F. Taylor and J. Rafelski, "Observation of Cold Nuclear Fusion in Condensed Matter", Nature, 338, 737-740, (1989).

4. M. Fleischmann and S. Pons, "Electrochemically Induced Nuclear Fusion of Deuterium", J. Electroanal. Chem., 261, 301-308, (1989).

5. E. K. Storms, "Review of Experimental Observations About the Cold Fusion Effect", to be published in Fusion Technology 1991.

6. A. De Ninno, A. Frattolillo, G. Lollobattista, L. Martinis, M. Martone, L. Mori, S. Podda, and F. Scaramuzzi, "Emission of Neutrons as a Consequence of Titanium-Deuterium Interaction", Il Nuovo Cimento, 101A, 5, 841, 1989.

7. Dr. Krystyna Cedzynska, private communication, December 1990. see also these proceedings.

8. W. C. Reinig, et. al., "Tritium Measurement Techniques", NCRP Report No. 47, NCRP Publications, Washington DC. 20014. 1976.

9. Packard CA 1600, Packard Instrument Co., 1 State St., Meriden CT 06450.

10. Berthold LB110, EG&G Instruments, Nuclear Products Group, 100 Midland Road Oak Ridge Tn. 37831.

11. SP10R, Radiation Monitoring Devices Inc., 44 Hunt St. Watertown Ma. 02172.

12. LKB Ultrofilm, LKB-Produkter AB, Box 305, S-161 26 Bromma, Sweden.

13. Overhoff & Associates, Inc., "Technical Manual for the Tritium Monitor Betatec", Milford Ohio. also Femtotech Inc., PO. Box 8257, Carlisle Oh. 45005.

14. H. O. Menlove and J.E. Swanson, "A High-Performance Neutron Time-Correlation Counter", Nucl. Technol. 71, 497-505, (1985).

15. H. O. Menlove, M. M. Fowler, E. Garcia, M. C. Miller, M. A. Paciotti, R. R. Ryan and S. E. Jones, "Measurements of Neutron Emission from Ti and Pd in Pressurized $D_2$ Gas and $D_2O$ Electrolysis Cells," Joun. of Fusion Energy, 9 (4) (1990).

16. R. Lasser and K. H. Klatt "Solubility of Hydrogen Isotopes in Palladium", Phys. Rev. B, 28, 748 (1983) and also Dr. Ivar Lindstrom, Private Communication, November 1990.

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