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A STUDY OF ELECTROLYTIC TRITIUM PRODUCTION

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ABSTRACT

Tritium production is being investigated using cathodes made from palladium and its alloys with various surface treatments. Three anode materials have been studied as well as different impurities in the electrolyte. Tritium has been produced in about 10% of the cells studied but there is, as yet, no pattern of behavior that would make the effect predictable.

INTRODUCTION

Since the first studies by Pons and Fleischmann(1), evidence for excess tritium production in electrolytic cells continues to be reported[2;3;4;5;6;7;8;9]. In a few cases, relatively large amounts of tritium (10^{5-6} counts/min-ml) have been observed in electrolytic cells while smaller but significant quantities (200-800 c/min-ml) have been seen by many others. Electrode materials from many sources have been used as well as cells of various designs including sealed cells with gas recombiners. Tritium also has been made using D_2 gas in a dry electrolytic cell.[10] Although we can now safely assume that the effect is real, no patterns have emerged from this effort to suggest ways to make the phenomenon reproducible. Apparently, a special environment must first be created on or near the electrode surface before the rather novel nuclear reactions can start. Because this special environment is, as yet, seldom achieved, an exhaustive study of the nuclear products using the necessarily expensive equipment is not cost effective and often frustrating. This study was undertaken to learn how the initiating conditions could be produced in order to improve the reproducibility of the nuclear

reactions before attempts are made to study them in detail. Tritium production was used as an indication that a nuclear reaction had occurred.

Over 150 cells were examined involving over 5000 tritium measurements. Only about 10% of the cells produced tritium. Although the success rate is still rather low, a range of conditions can be ruled out as not being critical.

EXPERIMENTAL

The cell design, shown in Fig. 1, was used for most of this work. The cell consists of a 120 ml, wide mouth glass jar with a bakelite lid. Gases generated by electrolysis flow through a stainless hypodermic needle sealed in the lid with epoxy and are routed to an IV drip system where the

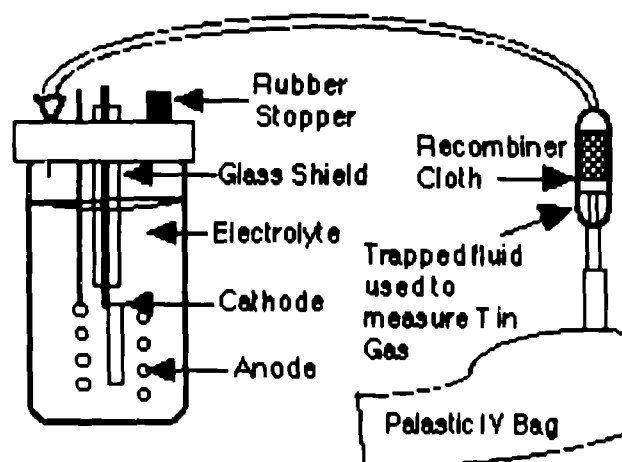


FIGURE 1. Cell with gas collection system

recombination catalyst is located. About 1 ml of recombined liquid collects in this region before the excess enters a plastic IV bag. A rubber stopper seals a small hole in the lid and allows the electrolyte to be

sampled using a 1.00 ± 0.01 ml disposable, automatic pipette. Thus the cell was completely sealed except for a small time during sampling. Tritium content of the cell was determined daily and a 1 ml sample of recombine was taken approximately every other day if the electrolysis rate permitted. These samples as well as tritium-free water were each mixed with 10 ml of Opti-Fluor(11) and counted using a Packard Tri-Carb Liquid Scintillation Spectrometer. Measured quantities of D_2O (12) and electrolyte(13) were returned to the cell to replace that lost by electrolysis and sampling, respectively.

Palladium alloys were made by arc-melting Pd powder(14) in an Ar atmosphere to make the alloys listed in Table I. The X/Pd ratios are indicated for each alloying component. Most of these

TABLE I
Pd Alloys Studied as Cathodes

Pd-Li (0.011, 0.012, 0.013, 0.023, 0.051, 0.22)
Pd-C (0.005, 0.012, 0.026, 0.034)
Pd-Li-C (Li 0.038, C 0.019),
 (Li 0.039, C 0.040)
 (Li 0.012, C 0.012)
Pd-Be (0.01, 0.04)
Pd-S (0.0043)
Pd-B (0.028)

cathodes were studied using Pt gauze anodes although nickel wire was used in a few cases. The cathodes were either coin shaped, made by rolling an arc-melted button or were strips cut from a coin using a low speed diamond saw. Several of the Pd-Li alloys were swaged into 1-2 mm diameter rods. In most cases, the surface was cleaned with concentrated HNO_3 , water and acetone.

Unalloyed Pd was studied sometimes as strips cut from a 0.050" thick sheet and four times as a wire. These cathodes were given various surface treatments as sum-

Washed with	Heated in
HNO_3 ,	Paraffin + H_2S ,
HCl,	Paraffin followed by H_2S , or
H_2O_2 , or	H_2S alone
Acetone	

marized in Table II. Only one treatment was applied to each strip. The paraffin + H_2S treatment was applied by heating the strip in vapor produced by heating paraffin and sulfur together.

Various materials were added to the electrolyte in order to plate a component onto the cathode surface. Some were present as unintentional impurities. The known impurities are listed in Table III. Not listed are the impurities in the D_2O and the components electrolyzed from the anode, both of which are largely unknown at the present time. The neutrons were obtained from a ^{252}Cf source located near the cells.

Several of the electrodes were examined using an electron microscope and an ion probe to determine the surface composition.

RESULTS

The results from the various surface

Added	Unintentional
Thiourea	Bakelite from lid
Wheat flour	Stainless from hypodermic needle
Fe metal	Glass components
Ag metal	
Hg metal	
Li_2S	
As_2O_3	
Ag_2S	
^{238}U metal	
Neutrons	

treatments and alloys will be discussed individually starting with the Pd-Li alloys.

ALLOYS

Pd-Li: The repeated ability of R. Huggins at Stanford University[15] to produce heat using impure Pd containing at least lithium and the observed migration of Li from the electrolyte into the cathode suggests that the presence of Li might be a precondition to a nuclear reaction. Cells were studied using the conditions listed in Table VII located at the end of the paper. No excess tritium was detected in any of the cells. All of the alloys except 0.22 % Li were ductile and easily rolled into a coin shape and swaged into rods.

Two alloys, Li/Pd = 0.011 (3mm Ø) and Li/Pd=0.034 (2mm Ø) were examined as rods for heat production, by T. Guilinger (Sandia National Laboratory) and by S. Gottesfeld (Los Alamos National Laboratory), respectively. No excess heat was observed in either case.

The presence of lithium was found to increase the ease of deuterium uptake and to increase the limiting D/Pd ratio over that observed using cathodes made with ordinary grade, arc-melted powder. However, there is no difference in properties when a comparison is made to ultra-pure, arc-melted powder. Apparently the presence of lithium offsets the effect of impurities that are present in ordinary grade palladium.

Pd-C: Carbon, when present in Pd, resides mainly in the grain boundaries as graphite because of its very low solubility. This impurity would be expected to inhibit the diffusion of D through the grain boundaries thereby reducing the uptake rate, increasing the D concentration gradient near the surface and reducing the limiting, average composition during the early history. When carbon was arc-melted with Pd powder, the resulting

coins showed these effects. None of the cells listed in Table VII produced excess tritium.

Pd-C-Li: When Li was added to an alloy containing carbon, the effects produced by carbon were reduced. Thus, the addition of Li to ordinary grade Pd is thought to improve the D uptake rate because it tends to offset the effect of the usual carbon impurity. These alloys, as listed Table VII, did not produce excess tritium.

Pd-Be: Because beryllium has a relatively weak, neutron rich nucleus, a suggestion was made by Edward Teller that its presence might augment the neutron source provided by the deuterium atom. The two listed compositions were sufficiently ductile to be rolled into a coin shape and cut into strips using a diamond saw. These were used as cathodes in cells described in Table VII. No excess tritium was detected.

Pd-S: Although tritium was produced in some cells when a sulfide treatment was applied to the cathode surface, this Pd-S alloy produced no tritium. However, the sulfur does, in both forms, accelerate recombination of the evolving D₂ with oxygen in the air causing self-heating.

Pd-B: Two cathodes containing boron were studied as listed in Table VII. One which was run with As₂O₃ in the electrolyte produced a small amount of tritium. The other cathode did not produce tritium.

SURFACE TREATMENT

Heated in H₂S: A number of pure palladium samples were heated in H₂S from a gas cylinder. This treatment causes an insulating sulfide layer to form on Pd. Unless the electrode is run as an anode for awhile, the Pd will not take up deuterium.

Samples treated only with H₂S, without paraffin, have not produced excess tritium.

Heated in Paraffin Vapor: Several pure palladium samples, both strips as well as wire, were heated in paraffin or paraffin + H₂S from a cylinder. As can be seen in Table VII, several of these cells produced excess tritium. In this series, the electrolyte dissolved some of the bakelite lid because the lid liner had been removed. Surface examination showed the presence of large amounts of Ca which is a major component of bakelite. This Ca, as well as a lesser amount of Zn, formed a complex surface layer on the electrode. Residual paraffin was also observed as clumps. Electrolytic action was uneven where the paraffin was present.

CONTROL CELLS

The amount of tritium produced in most of the cells described in this study is so small that the possibility of contamination must be considered. This is especially important because the work is being done in an area where tritium might be present in the air. Two methods were used to determine the contamination level produced by the environment and by the cell construction materials. In addition, a cell was contaminated on purpose so that the time behavior of such contamination could be studied.

	<u>Cathode</u>	<u>Anode</u>	<u>Electrolyte</u>	<u># Cells</u>
A.	Pd	Ni	H ₂ O	3
	Pt	Pt	D ₂ O, Aldrich	1
	Ni	Ni	D ₂ O, MSD	1
	Ti	Pt	H ₂ O	1
B.	5 open water jars			
C.	1 cell to which tritium water was added			

Seven inactive cells listed in Table VII were run during this study using materials from the same lot as were used in the cells that produced tritium (active cells). In addition, many cells of conventional, potentially active design did not produce tritium even though they were being studied at the same time, using the same materials and design as the active cells.

Open jars containing 60 ml of initially tritium-free water were situated in the room so that any tritium in the environment would be detected by observing an increase in their tritium content.

Finally, a little tritium water was added to an inactive cell in order to see how this known contamination would behave compared to the time history of active cells. The result from the inactive cells is described first.

(A) Inactive Cells: Three cells (#117, #123 and #132) contained water (LiOH) electrolyte and construction materials from the lots used in several active cells (#73, #98). Tritium measurements for these cells and for tritium-free water were taken over a 35 day period (23 data points in each set). The average excess tritium in the D₂O cells is 4 count/min-ml greater than tritium-free water (instrument background). Although there may be a slight pickup of tritium from the environment, the amount is well within the uncertainty of ±5 count/min-ml (±14 d/min-ml) found in

the tritium measurement for the cells containing D₂O electrolyte.

None of the other control cells show evidence for contamination beyond this level.

(B) Open Jars: One ml samples were taken from each open jar every working day. During times that a small amount of tritiated water vapor was present in the air, the tritium content of

TABLE V
Summary of Count Rate and Standard Deviation for Various Inactive Cells

<u>Data Set</u>	<u>Average</u>	<u>SD</u>	<u># Val-</u>
<u>ues</u>	<u>d/min-ml</u>		
A Closed Cells	138	±14	446
B Open Cell, corrected	120	±14	80
C Open Cell, uncorrected	140	±31	96

the jar increased. When the air became tritium-free, the tritium content of the jars decreased. This reduction occurred because ~4 ml/day evaporated in addition to the loss of 1 ml owing to sampling. This loss was replaced by tritium-free water. The rate of change, corrected for dilution, gives a measure of the tritium content of the air. The quantities present in the room are too small to have biological importance and are too small to affect sealed cells as used in this study. This conclusion will be seen more clearly in the following discussion about individual cells.

(C) **Addition of tritium water:** A small amount of tritium water was added to an inactive cell (#82) so that the count rate was increased to a value similar to that found in some active cells. Corrections were made for enrichment during electrolysis and dilution by replacement fluid. The result is shown in Fig. 2 as the fraction

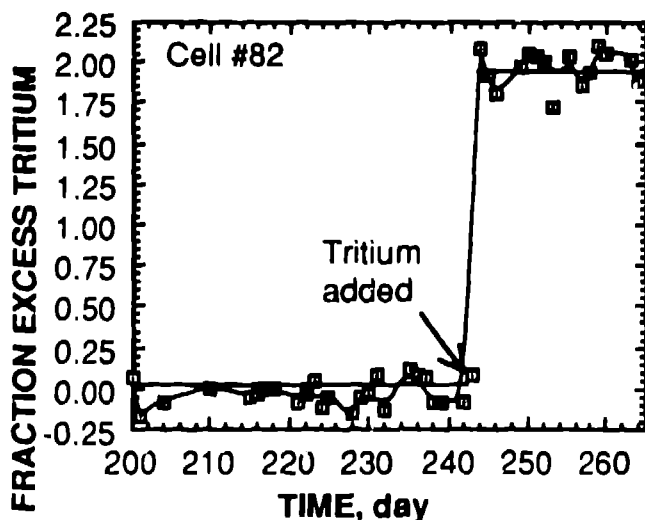


FIGURE 2. Effect of adding tritium water

excess tritium over that at the beginning of electrolysis. No tritium was produced during the 123 days preceding the addition of tritium water. After the addition, the quantity remained constant, consistent with normal enrichment and dilution. This steady value is in sharp contrast to the behavior of tritium loss from active cells as seen below. After this cell was terminated, the electrolyte was replaced by tritium-free water containing 0.2N LiOH and the current was reversed so that the cathode would lose any absorbed tritium to the water. After electrolyzing in this mode for 24 hrs, no increase in tritium content was seen. Therefore, the tritium which had been added to the electrolyte did not react with the Pd cathode to any significant extent.

STATISTICAL ANALYSIS OF DATA

A series of closed cells, where the recombine was allowed to run back into the cell, were studied to determine the total random error in the tritium measurements. These cells were also unsealed such that there was a small hole to allow excess gas to escape through the recombine cloth. No enrichment was observed.

The standard deviation based on 446 measurements using 13 cells over a 40 day period is listed in Table V as (A).

A similar analysis of a sealed cell was made but where the recombine was collected separately. The value is based on 80 data points, after correction for enrichment, and is listed as (B). When an enrichment correction was not made, the standard deviation, designated (C) was larger because of the slow increase in the count rate over the

life of the cell. This study shows that the total random error in the tritium measurement is ± 14 decomposition/min-ml (± 5.3 count/min-ml) with an occasional outlier near ± 25 d/min-ml. Because of such outliers, unusually high or low values are ignored if they are isolated from the trend of the data. In other words, only patterns supported by many points are used as evidence for tritium production. In addition, only data that are greater than 5 sigma from the behavior produced by enrichment and dilution are considered significant.

DISTRIBUTION FACTOR

During electrolysis, the tritium concentration in the gas is generally less than that in the liquid. This difference causes enrichment of tritium in the liquid. In order to calculate the amount of tritium that is lost to the gas phase, thereby allowing tritium enrichment to be calculated, the distribution factor (tritium in the gas/tritium in the liquid) must be determined. This quantity was measured directly for a number of active and inactive cells as listed in Table VI. In addition, this quantity was calculated by an indirect approach using the rate at which tritium was enriched in an inactive cell #70 (slope in Table VI). Values as high as 1.1 have been observed in the past (averaged over the life of the cell) although recent cells above #91 have a mean of 0.55 ± 0.02 based on 13 cells.

BEHAVIOR OF ACTIVE CELLS

A total of 13 cells have shown some evidence for tritium production as of 3/8/90, as listed in Table VII. However, only the more recent observations have been made with sufficient detail to eliminate any doubt about the source of the tritium. The time history of various cells are summarized in Fig. 3. The square points

indicate the time at which the cell was started and the corresponding + indicates when the cell was stopped. A few of these points are connected in the figure to show how the connection should be made. Tritium was produced during the interval between each set of vertical lines. A box is drawn around the cells that were active during this time, and the number of active cells and the total running at the time are indicated. For example, between 100 and 125 days after 6/1/89, seven cells out of a total of 34 produced tritium. These active cells continued to run after day 125 without producing tritium until they were turned off on day 180. Eleven cells were started during the time when tritium was

TABLE VI
Measured Distribution Factor
and Standard Deviation

<u>Cell #</u>	<u>Gas/Liquid</u>	<u>SD</u>	<u>#Values</u>
70 (ratio)	0.91	0.16	15
70 (slope)	0.84		
74	1.12	0.17	13
73*	0.83	0.12	9
82(before)	0.69	0.09	4
82(after)	0.44	0.06	3
85	0.53	0.05	10
86	0.52	0.08	8
87	0.76	0.15	21
91	0.53	0.04	7
98*	0.53	0.04	8
110	0.58	0.02	5
118	0.58	0.05	10
119	0.59	0.09	10
120	0.54	0.06	11
121	0.55	0.04	13
124	0.57	0.07	11
127	0.53	0.08	11
128	0.57	0.05	9
129	0.55	0.08	9
130	0.53	0.06	9
131	0.52	0.06	10

Values before and after addition of tritium to cell #82 are shown,

* Active cells

being produced in these seven cells but none became active. Presence of low level tritium in the room air, as detected by the open water jars, is shown by the boxes at the bottom of the figure. Some tritium could have been in the air before day 125 but no open jars were present to detect its presence. Although some tritium was present in the air when excess tritium appeared in some cells, other cells showed

the measured distribution factor. The small amount of tritium in D_2O that is added to the cell each day to replace fluid loss is also taken into account. The expected inventory of tritium in the cell is calculated and compared to the actual amount. The fraction excess is the difference between these values divided by the initial amount. For example, a calculated excess fraction of 1.0 means that the tritium con-

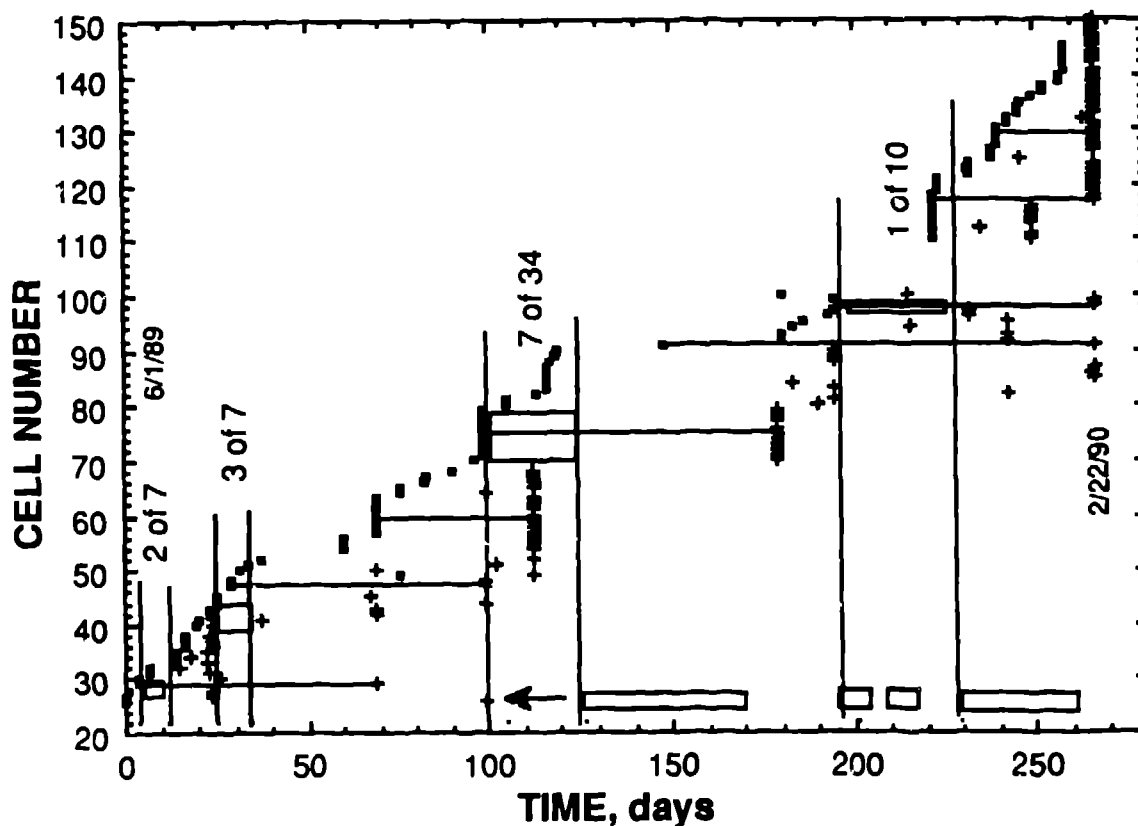


FIGURE 3. Time history of cells

no tritium production. In addition, all of the active cells eventually ceased production even though a small amount of tritium continued to be present in the air.

Two active (#73 and #98) and two inactive (#70 and #99) cells are compared in Fig. 4 where the fraction excess is plotted as a function of time from the start of electrolysis. The fraction excess is calculated as described previously[9]. In brief, the amount of tritium lost to the gas phase is calculated from the electrolysis rate and

tent has doubled over that in the cell when electrolysis started, taking into account tritium from all known sources.

The amount of fluid added to the cells on a daily basis was recorded and compared to the amount calculated using the cell current. Except when a cell occasionally started to leak fluid, the amount added agreed with the amount calculated. Of the two active cells compared in Fig. 4, only cell #98 started to leak after 40 hr. This leak occurred after tritium production

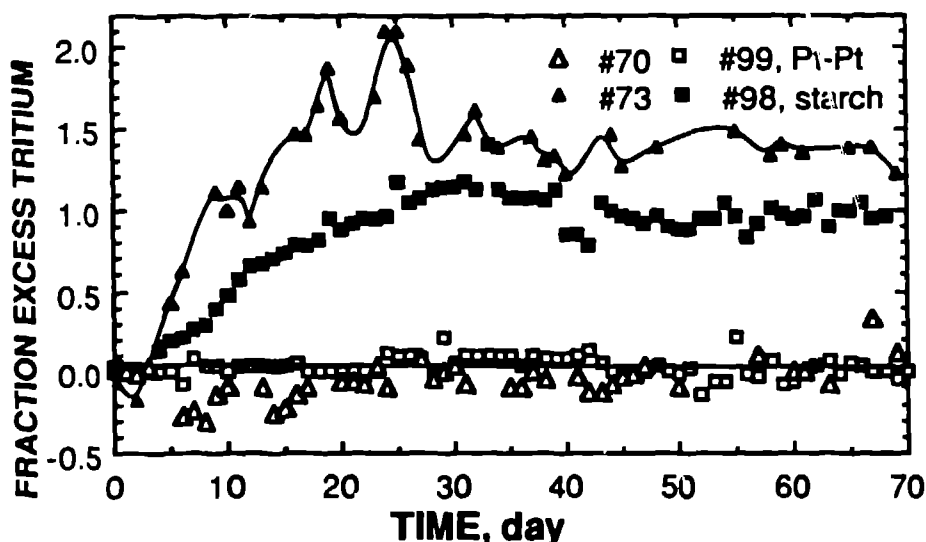


FIGURE 4. Fraction excess tritium calculated for cells #70, #73, #98 and #99.

had stopped.

Cells #70 and #73 were electrolyzed at the same time and at the same current. The cathode in cell #73 had been heated in paraffin vapor before being electrolyzed. Other factors in the cell design are listed in Table VII. This cell produced tritium over a 25 day period with evidence for two bursts. After the last burst, the tritium content decreased to a constant value over a 10 day period. This decrease is thought to be caused by the removal of tritium that is present as DT gas dissolved in the liquid. The tritium that remained was present as DTO. This initial decrease is in contrast to the stable tritium content that results when tritiated water is placed in a cell as seen in Fig. 2.

Cells #98 and #99 were electrolyzed at the same time but not at the same current. The cathode in cell #98 was not pretreated although 3.4 mg of wheat flour was placed in the electrolyte. Such large molecules are thought to inhibit the growth of surface dendrites. Indeed, the tritium production rate was decreased, production lasted for a longer time and there were no bursts. However, attempts to duplicate this effect using other cells

containing various amounts of wheat flour have failed.

CONCLUSION

We have produced tritium in electrolytic cells containing a Pd cathode, a Ni or Pt anode and an electrolyte containing 0.1M or 0.2M LiOD. However, we can not yet suggest a design that will have a high probability of success.

The presence of Li, Be, C or S alloyed with Pd does not increase the probability of tritium production at the concentrations studied in this work. A surface layer of palladium sulfide also does not help. The effect of dissolved boron is uncertain.

A small concentration of ^{238}U , Hg, Fe, thiourea, Li_2S or Ag_2S does not increase the rate of success. The effect of paraffin, wheat flour or As_2O_3 is uncertain although the chance of tritium production seems to be improved by these materials.

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References

- [1] M. Fleischmann and S. Pons, "Electrochemically Induced Nuclear Fusion of Deuterium", *J. Electroanal. Chem.* **261** (1989) 301 and errata **263** (1989) 187.
- [2] N. J. C. Packham, K. L. Wolf, J. C. Wass, R. C. Kalnitha and J. O'M. Bockris, "Production of Tritium from D₂O Electrolysis at a Palladium Cathode", *J. Electroanal. Chem.* **270** (1989) 451.
- [3] C. Sanchez, J. Sevilla, B. Escarpiz, F. J. Fernandez and J. Canizares, *Solid State. Comm.* **71**, #12, (1989) 1039.
- [4] P. K. Iyengar, "Cold Fusion Results in BARC Experiments", *Proc. of Fifth International Conference on Emerging Nuclear Energy Systems, Karlsruhe, Federal Republic of Germany, July 3-6, 1989. See also BARC-1500 Dec. 1989, Government of India, Atomic Energy Commission and this Proceeding.*
- [5] C. D. Scott, J. E. Mrochek, E. Newman, T. C. Scott, G. E. Michaels and M. Petek, "A Preliminary Investigation of Cold Fusion by Electrolysis of Heavy Water", *Oak Ridge National Laboratory, ORNL/TM-11322* (1989).
- [6] C. H. McKubre, S. I. Smedley, F. L. Tanzella and R. D. Weaver, "Calorimetric and Kinetic Observation of D₂-Pressurized LiOD/D₂O/Pd Cells", *Presented at Electrochemical Soc. Meeting, Hollywood, Florida, Oct. 19-20, 1989.*
- [7] R. Alqasbi and H. J. Scaller, "Neutrons and Tritium from Cold Fusion in Pd-D", *Presented at Electrochemical Soc. Meeting, Hollywood, Florida, Oct. 19-20, 1989.*
- [8] R. Adzic, D. Gervasio, I. Bae, B. Cahn and E. Yeager, "Investigation of Phenomena Related to D₂O Electrolysis at a Palladium Cathode", *Presented at Electrochemical Soc. Meeting, Hollywood, Florida, Oct. 19-20, 1989.*
- [9] E. Storms and C. Talcott, "Electrolytic Tritium Production", *accepted by Fusion Technology* (1990).
- [10] T. Clayton and D. Tuggle, *submitted to Fusion Technology* (1990).
- [11] Opti-Fluor was obtained from Packard Instrument Company, 2200 Warrenville Rd, Downers Grove, IL 60515. This fluid shows no chemiluminescence after 20 min when mixed 10 to 1 with 0.2 N LiOD.
- [12] Obtained from MSD Isotopes, Montreal, Canada
- [13] Made by mixing Li metal with the D₂O to give 0.1N or 0.2 N solutions.
- [14] Obtained from Johnson-Matthey Company as 100% Pd, batch #V7114307 and #V8368501.

[15]

A. Belzer, U. Bischler, S. Crouch-Baker, T. M. Gur, M. Schrieber, and R. Huggins "Two Fast Mixed Conductor Systems-Deuterium and Hydrogen in Palladium", *Cold Fusion Workshop, Santa Fe, NM May 22-25 (1989).*

TABLE VII
Description of Cell Treatment

#	Form	wt. (g)	area (cm ²)	Alloy B	Cleaning C	Pre- Treatment D	Electrolyte E	Kind F	Anode G	Container H	lead covering I	Max. D/Pd J	Date Started K	T L
Pd+Li Alloys														
33	coin	4.87	4.7	Li/Pd(1)=0.051	std	none	0.5N,thio	MSD	Pt	plastic(2)	none	0.77	6/15	n
34	coin	4.96	4.2	Li/Pd(1)=0.023	std	none	0.5N,thio	MSD	Pt	plastic(2)	none	0.83	6/15	n
35	coin	4.99	4.7	Li/Pd(1)=0.012	std	none	0.5N,thio	MSD	Pt	plastic(2)	none	0.77	6/15	n
49	wire	0.74	1.5	Li/Pd(2)=0.0343	HNO3	none	0.2N	MSD	Pt, small	plastic(3)	glass		8/15	n
55	coin	3.67	4.2	Li/Pd(4)=0.025	std	none	0.2N	MSD,pe	Pt	plastic(4)	glass	0.73	7/31	n
69	coin*	10.26	7.9	Li/Pd(1)=0.013	std	none	0.2N	MSD	Pt	quartz	glass		9/5	n
88	button	4.05	2.0	Li/Pd(1)=0.22	none	none	0.1N	MSD,Ald.	Pt	glass(5)	glass		9/25	n
111	strip	1.26	2.3	Li/Pd=0.012	HCl	pol.,annealed	0.1N	Aldrich	Ni(2)	glass(5)	glass	0.79	1/9	n
113	strip	0.82	1.7	Li/Pd=0.012	HCl	polished	0.1N	Aldrich	Ni(2)	glass(5)	glass	0.76	1/9	n
Pd + C Alloys														
2	coin	5.00		C/Pd(1)=0.0344	std	none	0.2N	MSD	Pt	plastic(3)	torr seal		7/2	n
52	coin	5.45	5.4	C/Pd(1)=0.026	Na2S	oxidized	0.2N	MSD	Pt	plastic(3)	1.yes, 2.no	0.88	7/8	n
56	coin	3.52	3.7	C/Pd(1)=0.0191	Li2S	oxidized	0.2N	MSD	Pt	plastic(4)	glass	0.66	7/31	n
65	coin	4.10	4.0	C/Pd=0.005	std	none	0.2N	MSD	Pt	plastic(4)	glass	0.77	8/15	n
110	strip	0.92	1.8	C/Pd=0.012	HCl	polished	0.1N	Aldrich	Ni(2)	glass(5)	glass	0.73	1/9	n
116	strip	0.83	1.8	C/Pd=0.012	HCl	pol.,annealed	0.1N	Aldrich	Ni(2)	glass(5)	glass	0.75	1/9	n
Pd + Li + C Alloys														
89	coin	4.90	3.0	Li/Pd(5)=0.038 C/Pd=0.019	H2O2	none	0.1N	MSD,Ald.	Pt	glass(5)	glass	0.70	9/27	n
90	coin	4.65	3.0	Li/Pd(5)=0.039 C/Pd=0.040	none	none	0.002N	MSD,Ald.	Pt	glass(5)	glass	0.53	9/28	n
114	strip	0.97	2.0	Li,C/Pd=0.012	HCl	polished	0.1N	Aldrich	Ni(2)	glass(5)	glass	0.76	1/9	n
115	strip	0.80	1.7	C,Li/Pd=0.012	HCl	pol.,annealed	0.1N	Aldrich	Ni(2)	glass(5)	glass	0.71	1/9	n
Pd-B Alloy														
43	coin	4.69		Pd(1)+B	std	none	0.2N,As2O3	MSD	Pt	plastic(3)	torr seal		6/24	y
118	strip	1.41	2.7	B/Pd=0.028	HCl	none	0.1N	Aldrich	Ni(3)	glass(5)	glass		1/9	n
Pd + S Alloy														
42	coin	4.47		S/Pd(1)=0.0043	std	none	0.2N	MSD,pe	Pt	plastic(3)	torr seal		6/24	n

TABLE VII (Continued)
Description of Cell Treatment

#	Form	wt. (g)	area (cm ²)	Alloy	Cleaning	Pre- Treatment	Electrolyte	Kind	Anode	Container	lead covering	Max. D/Pd	Date Started	T
Pd + Be Alloys														
92	strip	1.63	3.0	Be/Pd(2)=0.01	none	none	0.1N	Aldrich	Ni(1)	glass(5)	glass	0.75	11/28	n
93	strip	1.66	2.9	Be/Pd(2)=0.04	none	none	0.1N	Aldrich	Ni(1)	glass(5)	glass	0.71	11/28	n
PURE Pd														
68	coin	2.09	2.6	ultra pure	std	none	0.2N	MSD	Pt	plastic(4)	glass	0.79	8/30	n
32	coin	5.10		Pd(1)	std	none	0.2N,thio	MSD	Pt	plastic(2)	torr seal		6/8	n
137	coin	6.21		Pd(3), W sw	sanded,HNO3	vac	0.1N	Aldrich	Pt, Ni(4)	plastic(2a)	glass		2/8	
138	coin	7.48		Pd(3), W sw	sanded,HNO3	anneal	0.1N	Aldrich	Pt, Ni(4)	plastic(2a)	glass		2/8	
54	coin	4.08	4.3	Pd(4)	std	none	0.2N	MSD,pe	Pt	plastic(4)	glass	0.77	7/31	n
50	coin	5.26		Pd(2)(am)	std	none	0.2N	MSD	Pt	plastic(3)	torr seal		7/2	n
51	coin	5.89	5.3	Pd(2)(am)	std	anneal	0.2N	MSD	Pt	plastic(3)	torr seal	0.80	7/4	n
58	sheet	1.79	2.5	Pd(2)	HNO3	none	0.2N,thio	MSD	Pt	plastic(4)	glass	0.83	8/9	n
59	sheet	2.44	3.5	Pd(2)	HNO3	none	0.2N,thio	MSD	Ni gauze	plastic(4)	glass	0.71	8/9	n
66	strip	1.71	1.5	Pd(2)	HNO3	none	0.2N	MSD	Ni(1)	plastic(4)	glass		8/22	n
104	strip	0.59		Pd(2)	HNO3	anneal	0.1N	Aldrich	Pt	glass(5a)	none		12/12	n
106	strip	1.11		Pd(2)	HNO3	none	0.1N	Aldrich	Pt	glass(5a)	none		12/12	n
107	strip	1.16		Pd(2)	HNO3	none	0.1N	Aldrich	Pt	glass(5a)	none		12/12	n
108	strip	0.93		Pd(2)	HNO3	none	0.1N	Aldrich	Pt	glass(5a)	none		12/12	n
109	strip	0.77		Pd(2)	HNO3	none	0.1N	Aldrich	Pt	glass(5a)	none		12/12	n
121	strip	0.68	1.5	Pd(2),sw Ni(4)	HCl	none	0.1N	Aldrich	Ni(2)	glass(5)	glass		1/10	n
127	strip	0.82	1.6	Pd(2),sw Ni(4)	HNO3	reverse	0.1N LiOD	Aldrich	Ni(4)	glass(5b)	glass		1/26	n
139	strip	1.06	2.0	Pd(2),sw Ni(4)	none	none	0.1N	Aldrich	Pt, Ni(4)	paraffin	glass		2/12	
140	strip	1.02	2.0	Pd(2),sw Ni(4)	HNO3	none	0.1N	Aldrich	Pt, Ni(4)	paraffin	glass		2/12	
41	wire	1.90	7.6	Pd, Marshall	HNO3	none	0.2N,thio	MSD	Pt	plastic(3)	torr seal	0.86	6/21	y
82	wire	1.66		Pd,Marshall	std	none	0.05N	MSD,Ald.	Ni(1)	glass(5)	glass		9/25	n
70	wire		2.0	Pd, Martin	HNO3	none	0.1N	MSD	Ni(1)	glass(5)	glass		9/5	n
79	wire	0.37	2.0	Pd,Martin	HNO3	none	0.2N,Ag	MSD	Ni(1),Ag	glass(5)	glass	0.94	9/7	y
80	bar	8.45	9.4	Pd investment	none	none	0.2N	MSD	Pt	glass(5)	glass		9/14	n
81	bar	7.44	8.4	Pd investment	none	none	0.2N	MSD	Pt	glass(5)	glass	0.74	9/14	n
Paraffin														
73	strip	0.68	1.0	Pd(2)	C	paraffin	0.2N	MSD	Ni(1)	glass(5)	glass	0.81	9/7	y
74	strip	0.69	1.5	Pd(2)	C	paraffin	0.2N	MSD	Ni(1)+S	glass(5)	glass	0.78	9/7	n
83	strip			Pd(2)	HNO3	paraffin	0.2N	MSD	Ni(1)	glass(5)	glass		9/25	n
84	strip	0.54	1.6	Pd(2)	HNO3	paraffin	0.1N	MSD	Ni(1)	glass(5)	glass	0.76	9/25	n

TABLE VII (Continued)
Description of Cell Treatment

#	Form	wt. (g)	area (cm ²)	Alloy	Cleaning	Pre-Treatment	Electrolyte	Kind	Anode	Container	lead covering	Max. D/Pd	Date Started	T
Paraffin (Continue)														
85	strip	0.64		Pd(2)	HNO ₃	paraffin	0.1N	MSD,Ald.	Ni(1)	glass(5)	glass		9/25	n
86	strip	0.42		Pd(2)	HNO ₃	paraffin	0.1N	MSD,Ald.	Pt	glass(5)	glass		9/25	n
87	strip	0.47		Pd(2)	HNO ₃	paraffin	0.1N	MSD,Ald.	Pt	glass(5)	glass		9/25	n
91	sheet			Pd(2)	HNO ₃	paraffin	0.2N	MSD,Ald.	Ni(1)	glass(5)	glass		10/27	n
100	strip	1.13		Pd(2)	HNO ₃	paraffin	0.1N	Aldrich	Ni(1)	glass(5a)	none		11/28	n
101	strip	0.97		Pd(2)	HNO ₃	paraffin	0.1N	Aldrich	Ni(1)	glass(5a)	none		11/29	n
102	strip	0.69		Pd(2)	HNO ₃	paraffin	0.1N	Aldrich	Ni(1)	glass(5a)	none		11/28	n
105	strip	0.87		Pd(2)	HNO ₃	paraffin	0.1N	Aldrich	Pt	glass(5a)	none		12/12	n
Paraffin+H₂S														
40	coin	6.97		Pd(1)	S	H ₂ S+C	0.2N	MSD	Pt	plastic(3)	no	0.84	6/20	n
44	coin	4.93	4.6	Pd(1)	S	H ₂ S+C	0.2N	MSD	Pt	plastic(3)	torr seal	0.75	6/26	n
45	coin	5.53		Pd(2)	S	H ₂ S+C	0.2N	MSD	Pt	plastic(3)	torr seal	0.67	6/26	n
46	coin	5.15		Pd(1)	S	H ₂ S+C	0.2N	MSD	Pt	plastic(3)	torr seal		6/27	n
71	strip	0.73	2.5	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSD	Pt small	glass(5)	glass	0.55	9/7	y
72	strip	0.73	2.1	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSD	Pt small	glass(5)	glass	0.81	9/7	y
75	strip	0.50	1.4	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSD	Pt small	glass(5)	glass	0.71	9/7	y
77	strip	0.72	1.4	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSU	Pt small	glass(5)	glass	0.69	9/7	y
H₂S														
62	sheet	2.61	3.5	Pd(2)	S	H ₂ S	0.2N	MSD	Pt	plastic(4)	glass	0.56	8/9	n
63	sheet	2.56	3.5	Pd(2)	S	H ₂ S	0.2N	MSD,pe	Pt	plastic(4)	glass	0.67	8/9	n
64	sheet	2.27	3.4	Pd(2)	S	H ₂ S	0.2N	MSD	Pt	plastic(4)	glass	0.69	8/15	n
Wheat Flour														
98	strip	0.79	1.8	Pd(2), sw stainless	HNO ₃	none	0.1N, 3.5mg	Aldrich	Ni(2)	glass(5)	glass	0.93	12/15	y
120	strip	0.86	1.7	Pd(2), sw Ni(4)	HCl	none	0.1N, 15mg	Aldrich	Ni(2)	glass(5)	glass		1/10	n
122	strip	0.68	1.6	Pd(2), sw Ni(4)	HNO ₃	none	0.1N, 5.4 mg	Aldrich	Ni(1)	glass(5)	glass		1/19	n
124	strip	0.96	1.8	Pd(2), sw Ni(4)	HNO ₃	none	0.1N, 4.0 mg	Aldrich	Ni(3)	glass(5)	glass		1/19	n
125	strip	0.75	1.6	Pd(2), sw Ni(4)	HNO ₃	reverse	0.1N, 5.2mg	Aldrich	Ni(3)	glass(5)	glass		1/25	n
126	strip	0.83	1.7	Pd(2), sw Ni(4)	HNO ₃	reverse	0.1N, 4.7mg	Aldrich	Ni(3)	glass(5)	glass		1/25	n
128	strip	0.79	1.6	Pd(2), sw Ni(4)	HNO ₃	reverse	0.1N, 15mg	Aldrich	Ni(4)	glass(5b)	glass		1/26	n
129	strip	0.61	1.4	Pd(2), sw Ni(4)	HNO ₃	reverse	0.1N, 7.2mg	Aldrich	Ni(4)	glass(5b)	glass		1/26	n
130	strip	0.92	1.8	Pd(2), sw Ni(4)	HNO ₃	reverse	0.1N, 4.2mg	Aldrich	Ni(4)	glass(5b)	glass		1/26	n

TABLE VII (Continued)
Description of Cell Treatment

#	Form	wt. (g)	area (cm ²)	Alloy	Cleaning	Pre- Treatment	Electrolyte	Kind	Anode	Container	lead covering	Max. D/Pd	Date Started	T
Thiourea														
26	coin	3.63	3.7	Li/Pd(1)=0.047	std	none	0.2N	MSD	Pt	plastic(2)	torr seal	0.82	6/1	n
28	coin	4.88	3.0	Rh/Pd=0.1	std	none	0.2N	MSD	Pt	plastic(2)	torr seal		6/2	n
29	coin	3.63	3.7	Pd(1)	S	H ₂ S+C	0.2N	MSD	Pt	plastic(2)	torr seal	0.72	6/5	y
31	coin	4.93	4.7	Pd(1)+Rh+Li	std	none	0.2N	MSD	Pt	plastic(2)	torr seal	0.88	6/8	n
32	coin	5.10		Pd(1)	std	none	0.2N	MSD	Pt	plastic(2)	torr seal		6/8	n
33	coin	4.87	4.7	Li/Pd(1)=0.051	std	none	0.5N	MSD	Pt	plastic(2)	no	0.77	6/15	n
34	coin	4.96	4.2	Li/Pd(1)=0.023	std	none	0.5N	MSD	Pt	plastic(2)	no	0.83	6/15	n
35	coin	4.99	4.7	Li/Pd(1)=0.012	std	none	0.5N	MSD	Pt	plastic(2)	no	0.77	6/15	n
36	coin	5.86		Rh/Pd=0.1	std	none	0.5N	MSD	Pt	plastic(2)	no	0.86	6/17	n
37	coin	5.35	4.8	Rh/Pd=0.1	std	none	0.3N	MSD	Pt	plastic(2)	no	0.90	6/17	n
38	coin	5.13	4.7	Rh/Pd=0.1	std	none	0.2N	MSD	Pt	plastic(2)	no	0.85	6/17	n
41	wire	1.90	7.6	Pd, Marshall	HNO ₃	none	0.2N	MSD	Pt	plastic(3)	torr seal	0.86	6/21	y
58	sheet	1.79	2.5	Pd(2)	HNO ₃	none	0.2N	MSD	Pt	plastic(4)	glass	0.83	8/9	n
53	sheet	2.44	3.5	Pd(2)	HNO ₃	none	0.2N	MSD	Ni gauze	plastic(4)	glass	0.71	8/9	n
Uranium														
57	strip	0.46	1.1	Pd(2)	HNO ₃	none	0.2N	MSD	Pt, small	plastic(4)	glass		8/9	n
Iron														
61	sheet	2.08	2.9	Pd(2)	HNO ₃	none	0.2N	MSD	Pt	plastic(4)	glass	0.80	8/9	n
Silver														
79	wire	0.37	2.0	Pd, Martin	HNO ₃	none	0.2N	MSD	Ni(1), Ag	glass(5)	glass	0.94	9/7	y
Mercury														
95	strip	1.19	2.3	Pd(3)	HNO ₃	none	0.1N	Aldrich	Ni(1)	glass(5)	glass	0.88	12/4	n
Li₂S														
60	sheet	1.98	2.8	Pd(2)	HNO ₃	none	Li ₂ S+alcohol	MSD	Ni gauze	plastic(4)	glass	0.71	8/9	n
As₂O₃														
43	coin	4.69		Pd(1)+B	std	none	0.2N, As ₂ O ₃	MSD	Pt	plastic(3)	torr seal		6/24	y
131	strip	1.05	2.0	Pd(2), sw Ni(4)	HNO ₃	reverse	0.127g As ₂ O ₃	Aldrich	Ni(4)	glass(5b)	glass		1/29	n
Ag₂S														
135	strip	0.86		Pd(2), sw Ni(4)	HNO ₃	none	0.1N	Aldrich	Pt, Ni(4)	glass(5)	glass		2/2	n

TABLE VII (Continued)
Description of Cell Treatment

#	Form	wt. (g)	area (cm ²)	Alloy	Cleaning	Pre-Treatment	Electrolyte	Kind	Anode	Container	lead covering	Max. D/Pd	Date Started	T
CONTROL CELLS														
Water electrolyte														
117	strip	0.98		Pd(2)	HCl	none	0.2N LiOH	H ₂ O	Ni(3)	glass(5)	glass		1/9	n
123	strip	0.85	1.7	Pd(2), sw Ni(4)	HNO ₃	none	0.2N LiOH	H ₂ O	Ni(1)	glass(5)	glass		1/18	n
132	strip	0.90	1.8	Pd(2), sw Ni(4)	HNO ₃	reverse	0.1N LiOH	H ₂ O	Ni(4)	glass(5b)	glass		1/29	n
144	strip	0.44	4.2	Ti(1), sw Ni(4)	HNO ₃	none	0.1N LiOH	H ₂ O	Pt, Ni(4)	glass(5)	glass		2/14	n
Pt Cathode														
99	wire			Pt	HNO ₃	none	0.1N	Aldrich	Pt	glass(5)	glass		12/12	n
Ni Cathode														
27	coin	6.97	6.4	Ni	std	none	0.2N	MSD	Pt	plastic(2)	torr seal	0.09	6/1	n
TRITIUM PRODUCING CELLS														
29	coin	3.63	3.7	Pd(1)	S	H ₂ S+C	0.2N, thio	MSD	Pt	plastic(2)	torr seal	0.72	6/5	y
30	coin	5.40		Pd(1)	S	H ₂ S+C	0.2N	MSD	Pt	plastic(2)	torr seal		6/5	y
41	wire	1.90	7.6	Pd, Marsh. 0.032"	HNO ₃	none	0.2N, thio	MSD	Pt	plastic(3)	torr seal	0.86	6/21	y
43	coin	4.69		Pd(1)+B	std	none	0.2N, As ₂ O ₃	MSD	Pt	plastic(3)	torr seal		6/24	y
71	strip	0.73	2.5	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSD	Pt small	glass(5)	glass	0.55	9/7	y
72	strip	0.73	2.1	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSD	Pt small	glass(5)	glass	0.81	9/7	y
73	strip	0.68	1.0	Pd(2)	C	paraffin	0.2N	MSD	Ni(1)	glass(5)	glass	0.91	9/7	y
75	strip	0.50	1.4	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSD	Pt small	glass(5)	glass	0.71	9/7	y
77	strip	0.72	1.4	Pd(2)	C-S	paraffin, H ₂ S	0.2N	MSD	Pt small	glass(5)	glass	0.69	9/7	y
78	strip	0.66	1.4	Pd(2)	none	none	0.2N	MSD	Ni(1)+S	glass(5)	glass	0.79	9/7	y
79	wire	0.37	2.0	Pd, Martin	HNO ₃	none	0.2N, Ag	MSD	Ni(1), Ag	glass(5)	glass	0.94	9/7	y
98	strip	0.79	1.8	Pd(2)	HNO ₃	none	0.1N, 3.5mg WF	Aldrich	Ni(2)	glass(5)	glass	0.93	12/15	y

A coin= arc melted and rolled into coin shape
 sheet= 0.05" thick sheet cut into square shape
 strip= strip cut from sheet or button to give a parallelepiped
 wire= a cylinder having a diameter from 0.03" to 0.04"
 button= used as arc-melted
 am= used after arc-melting
 sw Ni= spot welded to nickel lead
 Marshall or Martin= source of wire

B Alloys made by arc melting Pd with the other component
 Pd(1)= Johnson Matthey powder, Batch V7114307
 Pd(2)= Englehart sheet
 Pd(3)= Johnson Matthey powder, Batch V8368501
 Pd(4)= Englehart Powder, Batch A4934
 Pd(5)= Englehart Powder, Batch F01567
 sw = spot welded using tungsten electrodes, otherwise copper electrodes were used.

TABLE VII (Continued)

- C** Various surfaces applied as described under "D"
 std.= sanded with 200 grit paper and washed with nitric acid
 none=not cleaned
- D** H₂S+C= heated in H₂S mixed with paraffin vapor
 paraffin, H₂S= heated in paraffin vapor first than in H₂S
 H₂S+H₂O= heated in H₂S that was bubbled through H₂O
 H₂S= heated in pure H₂S to form Pd sulfide on surface
 oxidized= heated in air to produce microfractures on surface
 annealed = heated in vacuum
- E** thio= thiourea added to electrolyte
 pe= current was passed through electrolyte using a Pt dummy
 electrode before Pd was used
 All cells after #70 contained stainless hypodermic needles
 Hg= mercury metal in electrolyte
 U=uranium metal in electrolyte
 WF= Whole wheat flour added
- F** MSD = D₂O from MSD Isotopes, 99.9 at. %
 Aldrich = D₂O from Aldrich Chemical Co., 99.9 at. %
- G** Pt=platinum gauze in a large "C" shape around cathode
 Ni wire= nickel wire was made into a spiral (0.3" ID) around the anode
 Ni+S wire= Ni wire heated in H₂S
 Pt small= Pt gauze made into 0.3" in diameter tube that surrounded the cathode
 Ni gauze= gauze obtained from Texas A and M
 Ni(1)=0.04" wire from Driver-Harris Company
 Ni(2)=0.04" welding wire
 Ni(3)=0.06"
 Ni(4)= 0.04" protonic JM batch W12918
- H** Glass= flint glass
 Plastic= polyethylene
 Cell Design #1 = 120ml glass jar
 Cell Design #2 = 120ml glass jar with 40 ml
 plastic insert
 Cell Design #3 = 100ml plastic jar
 Cell Design #4 = 100ml plastic jar with glass tube
 covering cathode lead. Recombined liquid returned
 to cell.
 Cell Design #5 = 120ml glass jar with recombined gas
 collected separately.
 Cell Design #5a = 10ml glass test-tube with
 recombined gas collected separately.
 Cell Design #5b = 120ml glass jar with recombined
 gas collected separately in hot water bath.
 Cell Design #2a = 120ml glass jar with 40 ml plastic
 insert-recombine collected separately
- I** torr seal= Pt wire from Pd electrode was covered with
 Torr Seal to prevent Cu and Pb pickup
 glass=lead covered with glass tube
 no=bare lead
- J** Maximum determined from weight gain
- K** Month and day cell started
- L** y = excess tritium measured
 n = no excess tritium measured
 ? = no tritium measurement made