Tritium Measurements and Deuterium Loading in D_2O Electrolysis With a Palladium Cathode

R. R. Adzic, D. Gervasio, I. Bae, B. Cahan and E. Yeager
Case Center for Electrochemical Sciences and Chemistry Dept,
Case Western Reserve University
Cleveland, Ohio 44106

ABSTRACT

Measurements have been performed to check on the Fleischmann-Pons (F-P) phenomena. They involved 1) measurements of tritium in the cell solution and the gas above it; and 2) determination of the D/Pd ratio by coulometry. Enhancement of tritium in the D_2O solution was found in these two open glass cells, as well as in another four cells with Ni-anodes. The largest enhancement factor found was ~50. The neutron measurements were inconclusive.

INTRODUCTION

Fleischmann, Pons and Hawkins (1) and Jones et al. (2) have reported evidence for nuclear fusion of deuterium electro-chemically injected into palladium. Excess heat generation, neutron and γ-rays emission and tritium enhancement (1) were found by the first group and neutron emission (2) by the second group. These results have been met with much skepticism, especially after several reports of negative results by other workers (3-5). Subsequently there have been a number of reports of excess heat generation (6-8) and tritium enhancement (9, 10) or both (11).

The present research was motivated by the necessity for experiments under well defined conditions. In order to verify excess heat generation calorimetric measurements were done with a modified (Fleischmann-Pons) type open cell and calorimetric measurements with a closed cell with internal D_2/O_2 recombination. Some excess heat generation was observed. The results, however, require further evaluation and will be reported at a future date. Careful determination of tritium was done in electrolytic solution and also in the water obtained from the recombined gases outside the cell. Surface analysis of Pd cathode was performed and the D/Pd ratio determined. Neutron radiation measurements were attempted, but difficulties in making background corrections interfered with the interpretation.

EXPERIMENTAL

Electrodes

Pd from several sources was used for cathodes. The rods 0.2×10 cm were obtained from Engelhard Corporation. Wire of 0.5 mm diameter and 99.9% purity, was obtained from Johnson Matthey - Aesar. 1 mm wire (grade A) and 4 mm rod of 99.9% purity were obtained from Johnson-Matthey, England. The Engelhard Pd was also used by Westinghouse to prepare 2 mm rods by recasting, drawing or swaging. The pre-treatments used with various samples are indicated in the tables.

Anodes were made of Engelhard's Pt ribbon or wire cages of 99.9% purity Pt or Ni foil (99.9%), obtained from Fischer Scientific Co.

Electrolytes

0.1 M LiOD was used as the electrolyte in all glass cells, while 0.1 M and 1 M LiOD were used in Ni encased cells. LiOD attacks glass, which leads to a deposition of Si and other constituents of glass onto the Pd. The SIMS measurements showed that even in 0.1 M LiOD after several weeks Si could be easily detected on the Pd surface. The electrolytes were prepared from once distilled Norell (99.5%) or Isotech (99.9%) D_2O. 1 M LiOD was prepared from Li20 (99.9%) Cerac, Inc. in a glove bag. Upon diluting this stock solution, 0.1 M LiOD was obtained. Time constants did not permit yet to run blank experiments with LiOH electrolyte solution.

Tritium Measurements

The analyses of the solution for tritium were performed by Westinghouse Electric Company using liquid scintillation counting on a Packard 4030 counter. The liquid scintillation cocktails were prepared with OPTI-FLOUR scintillator. Typically, four consecutive 60 min. counts were recorded and averaged. Another precaution against any possible complications caused by components in the electrolyte solution was a vacuum distillation of the solution before tritium measurements. While this procedure minimized the possibility of interference effects with some constituents of the electrolyte it causes some loss of dissolved T_2 and DT. For this reason some experiments were done without distillation after neutralization of the alkaline electrolyte. The electrolytic solution from the cells with Ni anodes was neutralized, filtered and centrifuged to remove suspended nickel oxide particles. Distillation was also used with a centrifuged sample which showed high counts in order to ensure that solid particles within the electrolytic solution were not responsible for the high count rate.

Neutron Radiation Measurements

The neutron detection was attempted by using two BF_3 counters coupled with a multichannel analyzer. The authors are not satisfied that the background was adequately taken into account with this experimental arrangement and hence the results are considered inconclusive. Solid state track recorders may prove attractive for the measurements in the future.

RESULTS AND DISCUSSION

<u>Tritium</u>

   Fleischmann and Pons (1) reported enhancement of tritium concentration in the electrolyte as one evidence for a cold fusion of deuterium. The enhancement factor (see Eq. 4) is, however, close to the deuterium/tritium separation $S_{D,T} \approx 2$, as recently determined by Corrigan and Schneider (12). Bockris et al. (13) and Wolf et al. (14) reported recently enhancements of 4 to 7 orders of magnitude over the background. In this work we have measured tritium concentrations in open F-P cells, closed cells with the Ni casing and in four open cells designed for studying the tritium enhancement.

   In open cells a change in tritium concentration will occur due to the addition of D_2O containing T and due to removal by electrolysis. The isotopic separation factor of T to D is defined as

$$S = (n_D/n_T)g/(n_D/n_T)_s \tag{1}$$

where n_D and n_T are numbers of deuterium and tritium atoms in the gas (g) and the solution (s) phases.

   It can be shown (14) that the enhancement of tritium in a cell at time (t) obeys the following expression

$$n_T(t)/n_T(o) - S-(S-1)\exp(-t/T) \tag{2}$$

where T is the tritium build-up time constant.

   Thus, at very long times the tritium concentration in the solution due to isotopic separation is S times the tritium concentration in the original solution.

   If the recombination of D_2 and O_2 is carried out in the cell or outside the cell and the D_2O returned to the cell, the change in tritium concentration in the electrolyte could be brought about either by having substantial tritium in the palladium or other cell components or by a nuclear reaction. The palladium was stripped of hydrogen and its isotopes by electrochemical oxidation for 24 h in LiOD solution. This treatment has removed any tritium that might possibly be present in the Pd samples.

   In all our experiments, in which an enhancement of T was observed, a long time electrolysis of the order of several weeks was necessary before observation of the increase in tritium concentration. In two open cells with Pt anodes excess tritium was found. These cells also were the only ones out of five which generated excess heat.

   Table 1 gives the cell parameters for which enhancement of tritium concentration was observed. In four open cells, which were designed to study tritium accumulation and generation, were essentially test tubes 3 cm in diameter with a Teflon cell top. Cathodes were 5 cm long, spot welded to a nickel ribbon as an electrical contact; 0.25 cm thick Ni foil was used as anode. During a long term electrolysis a black residue, most probably nickel oxide, accumulated in the electrolyte in only two cells. The electrolyte was filtered and centrifuged to remove these particles and then neutralized by HCl before determination of T. When large tritium counts the solution was distilled and counted again. In some instances (cells 1, 2) the solution was vacuum distilled and only the

distillate was added to the scintillating liquid to avoid possible complications with various species in the solution.

The results are given in Table 2. All measurements were done in four runs during several to 24 h. The average of 4 counts is given with the standard deviation indicated. The decrease in the count rates for the distilled vs. neutralized samples was negligible. The samples were taken in intervals of 15-20 days. According to Bockris et al. (13) after reaching the maximum level, the tritium content considerably decreases after a few days, eventually down to the separation factor. If this is applicable to our measurements, it may mean that we have not observed the maximum amount of tritium probably due to a low frequency of sampling. The calculation of the tritium concentration was done in the following way:

$$\frac{\text{Net count rate}}{\text{c ml}^{-1}} = \frac{\text{average count rate}}{\text{c ml}^{-1}} - \frac{\text{blank count rate}}{\text{c ml}^{-1}} \qquad (3)$$

$$\text{Enhancement ratio} = \frac{\text{sample net count rate}}{\text{D}_2\text{O net count rate}} \qquad (4)$$

The number of disintegrations per minute per ml was calculated by dividing the net count rate with the efficiency factor, which is 0.224 c/d for this instrument.

$$\text{dpm ml}^{-1} = \frac{\text{net count rate/cpmml}^{-1}}{0.224\,\text{c/d}} \qquad (5)$$

For cells number 1 and 2 for which some excess heat generation was observed, the tritium level amounted to 766.43 and 1730 dpm ml^{-1} respectively. *

Let us compare the excess heat generated in these open cells with the amount of T found. If all of the energy were coming from the tritium generating reaction,

$$D + D \rightarrow T + H + 4.03 \text{ MeV} \qquad (6)$$

this would produce 4.03 MeV = 4.03 • 10^6eV • 1.6022 • 10^{-19}J/eV - 6.46 • 10^{-13} J/reaction. If 1 mW of power is produced in a cold fusion reaction, per 1 cm^3 of Pd the corresponding number of T atoms would be 10^{-3} W/(6.46 × 10^{13} W s/reaction) = 1.55 × 10^9 reactions/s; i.e. 1.55 × 10^9 T atoms would need to be produced. Tritium decays by the reaction

$$T \rightarrow {}^3He + \beta^- + \nu^- \qquad (7)$$

where β^- is a beta particle and ν^- is a antineutrino. Tritium decay is, as with all radioactive nuclei, first order. For N atoms of T, the decay rate is (16)

$$\frac{dN}{dt} = -\lambda N \qquad (8)$$

The decay constant, related to the half life, is (17)

$$\lambda = 1.782 \times 10^{-19} \text{ s}^{-1} \tag{9}$$

For 1 mW one should observe

$$\sim \frac{Dn}{DT} = \left(1.782 \times 10^{-9} \text{ s}^{-1}\right)\left(1.55 \times 10^{9}\right) dt = 2.76 \, \text{dps} = 166 \, \text{dpm of T} \tag{10}$$

Therefore, the observed 766 and 1730 dpm ml^{-1} can account only for ~4-10 mW. Much smaller, but still significant, enhancement of tritium was found in the gas phase for cells 1 and 4 after recombination of O_2 with D_2 and TD. Based on the separation factor one would expect a lower concentration of tritium in the gas phase, if tritium originates from the solution phase. Smaller concentrations of T in the gas phase than in the liquid phase were found by Bockris et al. (13) for long electrolysis times. In certain short intervals, e.g., one day, however, a higher concentration in gas phase was observed. The frequency of our measurements was not sufficient to detect such short-lived changes.

In an experiment of this importance one should address the question of the contamination by T of materials used in the experiments. The question can be ruled out on the grounds that the same materials have been used for the cells that gave zero tritium enhancement and the necessity for the long charging of Pd before observation of T. Electrochemical oxidation of H, D, or T, annealing and catalytic combustion, during the pretreatment make unlikely the possibility that tritium came from the palladium samples contaminated by tritium before the experiments.

Coulometric Determination of D absorbed in Pd Cathodes

Metallic Pd can absorb D to form a solid PdD_x solution. For cathodes equilibrated with air at ambient temperature and pressure, a value of x of about 0.7 to 0.8 is widely accepted. The reactivity of D in Pd may vary considerably with x, particularly if x exceeds 0.8, and therefore a coulometric determination of x was made. The working electrode was a coil of Pd wire (diameter = 0.025 cm, area = 1.86 cm^2, mass = 0.12 g; 99.997%, Puratronic grade, Johnson Matthey) crimped together with the end of a gold wire (d = 0.05 cm; 99.9985%, Puratronic, Johnson Matthey) which served as the lead to the potentiostat. A three compartment electrochemical cell was used. The electrolyte, 0.1 M LiOD was continuously and vigorously degassed with N_2 (99.995%, Ultrapure, Matheson). The counter electrode, a Pd coil (A = 2 cm^2), was in a chamber connected to the working chamber by a glass tube filled with a glass wool plug. The reference electrode was a reversible deuterium electrode (RDE).

The working electrode was rinsed with 0.1 $\underline{M}$ LiOD and D_2O electrolyte solution and cycled between 0.25 and 1.35 V until the steady state voltammogram shown in Fig. 1 (dashed line) was obtained. The cell was then rinsed and refilled with fresh electrolyte solution and sparged with N_2 gas. When the cell was left at open circuit for several days, it came to rest at +0.83 V, and it always returned to +0.83 V at open circuit whether it was offset to positive or negative potentials. When the working electrode was potentiostated at 0.83 V, no current flowed.

Figure 2 shows the chronoamperometry curves for the charging of Pd with D at -0.3 V and discharging of D from Pd at +0.83 V. During the charging at -0.3 V, there was no

noticeable D_2 gas evolution at the working electrode. This suggests that when D is formed on the Pd surface at -0.3 V, initially most of the D is absorbed into the bulk Pd. During charging at -0.3 V, the magnitude of the current was ~10 mA in the first minute, gradually dropped to ~1.5 mA after 10 hours, and finally stabilized at ~0.7 mA after 1 day.

There is an inflection in the chronocoulometry charging curve at ~0.3 V (Fig. 1, bottom curve) after 10 h. It was also noticed that after 10 or more hours that if the N_2 bubbler was lifted over the electrolyte solution, D_2 bubbles were visible and slowly nucleating at the Pd surface. These observations suggest that after 10 h the charging of the Pd with D is nearly completed. The linear diffusion law is stated as, $<x>^2 = 2 D t$, with $<x>$ being the mean distance traveled in time, t, with diffusion coefficient, D. An effective diffusion coefficient of D through Pd for the charging process, $D_{charging}$, can be calculated by using t = 10 hours and $<x>$ = 0.0125 cm (the radius of the electrode), and the value for $D_{charging}$ is ~2×10^{-9} cm^{2}s^{-1}. The transport of D into Pd wire is more complicated than that represented by simple linear diffusion because during D loading the electrode would be modeled better as a cylinder which is slowly expanding.

The endpoint of the charging process was difficult to assign since PdD_x formation and D_2 evolution can occur simultaneously as discussed above. The discharging process at +0.83 V was easily spotted because the current goes to zero when discharge is complete. Although the charging of D into Pd (bottom curves in Figs. 1 and 2) took over 10 h, discharging of D from Pd at +0.83 V (see top curves in Figs. 1 and 2) was completed after ~70 minutes. Using the linear diffusion law, this endpoint suggests an effective diffusion coefficient for discharging D from Pd, $D_{discharging}$, of ~2×10^{-8} cm^{2}s^{-1}. One analogous feature in the discharge chronoamperometry curve, not noticeable in Fig. 1, is a sinusoidal ripple in the curve between 1 1/2 and 8 minutes. This ripple is possibly due to a sudden phase transition in the cathode from the α to β phase as it loses ~40% of the total 106.5 C of charge in this time interval.

Several other charging potentials were tried, but the optimal charging potential was ~ -0.3 V vs. RDE. The rate of charging Pd with D was impractically slow at lower overpotentials (i.e., potentials more positive than -0.25 V). On the other hand, the rate of charging Pd with D was not noticeably increased at greater overpotentials (i.e., more negative than -0.3 V), although the rate of D2 evolution did appear to increase with increasing overpotential. Figure 6 shows the chronocoulometry curve for loading D into Pd at -0.35 V (see bottom curve). Comparing this and the analogous curve in Fig. 7 for loading at -0.3 V, one can see that at any given time there was more total negative charge for the loading of -0.35 V versus at -0.3 V. Furthermore, even though the total charge collected at -0.35 V (-255 C) was greater than the total charge collected at -0.3 V (-142 C), both experiments gave roughly the same total value for discharge at +0.83 V, namely +107 C. Similarly, the total charge collected at -0.4 V was -289 C (and was accompanied by even stronger D_2 bubble evolution), but still discharging the Pd cathode loaded this way gave just +122 C.

The mass of the Pd in the electrode was 0.12 g which corresponds to 1.1×10^{-3} moles of Pd. Assuming that the positive charge collected at +0.83 V is due only to the discharge of D from Pd, the moles of D in Pd can be calculated from Faraday's law, moles(D) = Q(discharge)/nF, in which n is 1 for the discharge of 1 D from Pd and F is Faraday's

constant (9.65×10^4 C/equivalent). The results are summarized in Table 3. All three cases indicate that there was one D absorbed per Pd atom in the electrode over this range of loading potentials with 0.1 $\underline{M}$ LiOD and D_2O as the electrolyte solution.

The loading and discharging processes were essentially reversible when the loading potentials were more positive than -0.4 V. When larger overpotentials, e.g., -0.6 V vs. RDE, were used the value of the subsequent discharge fell to ~+90 C, i.e., the corresponding ratio of D to Pd in the charged electrode was ~0.8. This suggests that charging Pd cathodes at high over-potentials (or alternatively at high constant currents) may result in irreversible damage to the electrode, which may prevent attaining higher ratios of D to Pd.

GENERAL DISCUSSION AND CONCLUSIONS

The results reported here support, to a certain extent, the claims of tritium generated in the electrolysis of D_2O on Pd of deuterium reported by Fleischmann, Pons and Hawkins (1). The following conclusions have been reached:

1. Enhancement of tritium was found in two out of five open cells with Pt-anodes and four out of four cells with Ni-anodes. The largest enhancement factor with respect to D_2O is ~50. The heat level is by three orders of magnitude larger than the heat corresponding to the observed tritium levels.

2. The ratio of deuterium to palladium atoms was found to be 1 for 0.25 mm wire, charged at E = -0.3 - 0.65 V.

3. The neutron radiation measurements were inconclusive due to the uncertainty in determining the background correction.

In general, on the experimental side there are, however, many questions to be answered in order to increase the understanding of this phenomenon. These include the irreproducibility and sporadicity of the phenomenon, necessity for a prolonged electrolysis before the excess heat or tritium production occur, the role of the microstructure and of the trace impurities in palladium, the difference between the amount of excess heat in open and closed cell, the role of surface impurities and the role of lithium, if any.

On the theoretical side the questions appear even more difficult. Table 4 lists possible fusion reaction of deuterium. According to the accepted theories the evidence for fusion of deuterium requires, besides the heat generation, a corresponding amount of neutrons, tritium and ^{3}He. The branching ratio of reaction A and B (Table 3) is approximately one. The cross section for reaction C is on the order of 10^7 lower than for A and B (1). γ-rays should be observed if the reaction C occurs in the electrochemical cell.

No data on electrochemically induced fusion satisfies indisputably any of these requirements. In order to overcome these difficulties several new mechanisms of fusion have been proposed. These include the mechanisms in which the energy from the reaction C dissipates into the lattice as heat rather than γ-photon (17, 18), or in which two deuterons as bosons are squeezed together in a sphere of an octahedral site (19). Dendrites on Pd surface have been suggested as an explanation for tritium generation, due to increased electric fields around the dendrite tips (13). Fracto-fusion was also

mentioned as a possible explanation in analogy with the explanation of neutron generation upon fracturing LiD single crystals (19). There is, obviously, a need for more work in order to estimate the merits of these explanations, as well as reach a complete understanding of the Fleischmann and Pons phenomenon.

ACKNOWLEDGEMENTS

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Table 1: Cell parameters for Tritium measurements

Cell	Electrode size d $\times$ 1 (cm $\times$ cm)	Supplier	Pretreatment	Anode	Current (mA/cm^2)
1	0.4 $\times$ 6	JM[a]	oxid. of H_{abs}	Pt	5-400
2	0.2 $\times$ 10	E[b]	"	Pt	60-120
3	0.1 $\times$ 5	JM	"	Ni	10-120
4	0.2 $\times$ 5	E	"	Ni	10-120
5	0.1 $\times$ 5	JM	Annealing	Ni	10-120
6	0.2 $\times$ 5	E	"	Ni	10-120
7	0.2 $\times$ 10	E	"	Pt	10-120

[a] - JM refers to Johnson Matthey.
[b] - E refers to Engelhard.

Table 2: Tritium Measurements

Cell	Days	ACR[a] (cpm)	NCR[b] (cpm)	$\dfrac{NCR^{sample}}{NCR^{D_2O}}$	DPM/ml[c]
1	42	21.13	5.13	1.6	44
	48	20.72	16.72	1.5	148
	58	35.08	19.08	6.5	170
	68	107.63±1.20	85.99±0.99	24.6±8.3	766
gas phase	68	42.66±2.32	24.06±0.93	8.3±2.3	114
	75	34.05	18.05	6	160
gas phase	75	20.93	4.93	1.6	44
2	25	209.27±0.80	193.80	6.2	1730
	45	120.06±0.49	103.06	4.1	920
3	26	159.14±1.83	143.24±1.88	49.4±13.8	1278
gas phase	26	26.53±0.86	9.63±0.96	3.3±0.9	85
	36	39.14±1.95	22.73±1.97	14.2	202
4	26	91.58±1.58	75.68±1.64	26.1±7.3	675
	36	36.94±0.54	20.53±0.60	12.8	183
5	26	58.11±0.27	42.21±0.51	14.6±4.1	376
	36	40.24±0.34	23.83±0.43	14.8	212
	76[d]	37.03±0.40	19.63	9.67	87
gas phase	76[d]	29.00±0.40	11.60±0.71	5.71±1.94	51
6	26	80.54±0.48	64.64±0.64	22.3±6.2	577
gas phase	26	39.40±0.86	23.50±0.96	8.1±2.3	209
	36	25.35±0.37	8.94±0.46	5.5	79
gas phase	56[d]	33.88±0.55	16.48±0.81	8.12±2.75	73
	76[d]	35.94±0.55	16.48±0.81	9.13±3.09	82
gas phase	76[d]	26.62±0.38	9.22±0.70	4.54±1.56	41
7	16	22.21±0.49	6.31±0.65	2.18±0.65	56
	36	20.93±0.86	4.52±0.63	2.8	40
	76[d]	21.76±0.86	4.36±1.04	2.15±0.88	19

[a] ACR - average (4 replicates) count rate per 0.5 ml.
[b] NCR - net count rate per 0.5 ml.
[c] DPM - disintegrations per minute.
[d] Sample size = 1 ml.
  Typical background count rate = 16 cpm.
  ACR(Norell D_2O) = ~46 cpm; ACR (Isotech D_2O) = ~19cpm.

TABLE 3

Summary of the Calculated Amount of the D

Loaded in Pd[a] at Different Overpotentials

η (volts)	$Q_{discharge}$(C)	moles D
-0.30	106.5	1.1×10^{-3}
-0.35	107.0 [b]	1.1×10^{-3}
-0.40	111.7	1.2×10^{-3}

[a] Mass of Pd electrode was 0.12 g before and after the experiment. This corresponds to 1.1×10^{-3} moles Pd.

[b] In the published version this was 170, which appears to be mistake.

TABLE 4

Nuclear Fusion Reactions

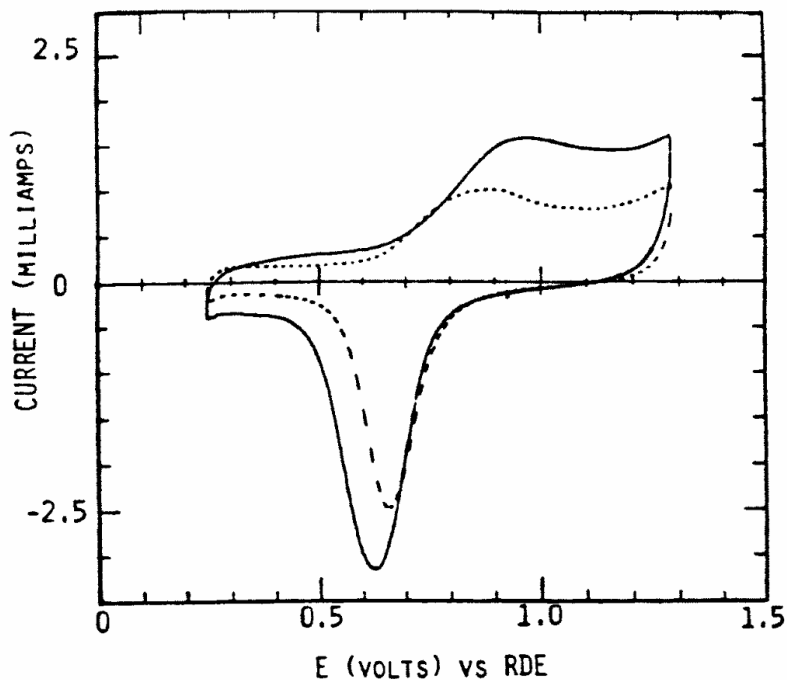| Reaction	Equation
A	$D + D \rightarrow n$ [2.45 MeV] + ^{3}He [0.82 MeV]
B	$D + D \rightarrow H$ [3.02 MeV + T [1.01 MeV]
C	$D + D \rightarrow \gamma$ [23.0 MeV] + ^{4}He [0.08 MeV]
D	$D + {}^6Li \rightarrow n$ [2.96 MeV] + ^{7}Be [0.43 MeV]
E	$D + {}^6Li \rightarrow {}^4He$ [11.2 MeV] + ^{4}He [11.2 MeV]
F	$D + {}^6Li \rightarrow H$ [4.39 MeV] + ^{7}Li [0.63 MeV]
G	$D + {}^7Li \rightarrow n$ [13.36 MeV] + ^{8}Be [1.67 MeV] $\rightarrow$ n [13.36 MeV] + ^{4}He [0.85 MeV] + ^{4}He [0.85 MeV]
H	$D + {}^7Li \rightarrow \gamma$ [16.7 MeV] + ^{9}Be [0.02 MeV]
I	$D + {}^7Li \rightarrow p + {}^8Li$ (endoergic, -1.01 MeV) $\rightarrow \rightarrow p + {}^4He$ [8.05 MeV] + ^{4}He (8.05 MeV]
J	$D + {}^7Li - T + {}^6Li$ (endoergic, -1.81 MeV)

Fig. 1. Cyclic voltammograms of Palladium wire (d = 0.03 cm) in D_2O with 0.1 M LiOD under a N_2 atmosphere. Dashed line is the steady state voltammogram. Solid line is the voltammogram recorded after charging the Pd with D at -0.4V for 24 hours, followed by discharging the D at +0.83 V for 60 minutes. Scan Rate = 100 mV/s. Electrode area = 1.86 cm². Counter electrode: Pd.
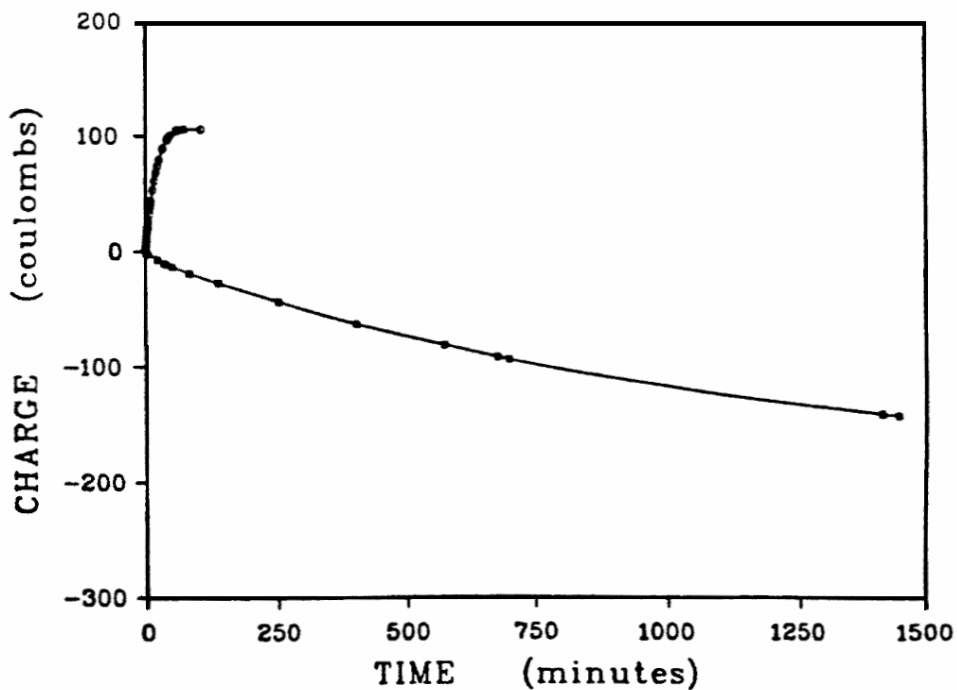

Fig. 2. Chronocoulometry curve for the charging (negative charge) and discharging (positive charge) of D into palladium wire (d = 0.03 cm). Electrode area = 1.86 cm². Electrode mass = 0.12 grams. Electrolyte: 0.1 M LiOD in D_2O. Charging potential = -0.3 V vs RDE. Discharging potential = +0.83 V vs RDE.

moles(Pd) = mass(Pd)/AW(Pd) = 0.12/106.4 = 1.1×10^{-3}.

moles(D) = Q(discharge)/nF = $106.5/9.65 \times 10^4$ = 1.1×10^{-3}.

mole ratio(Pd/D) = 1.

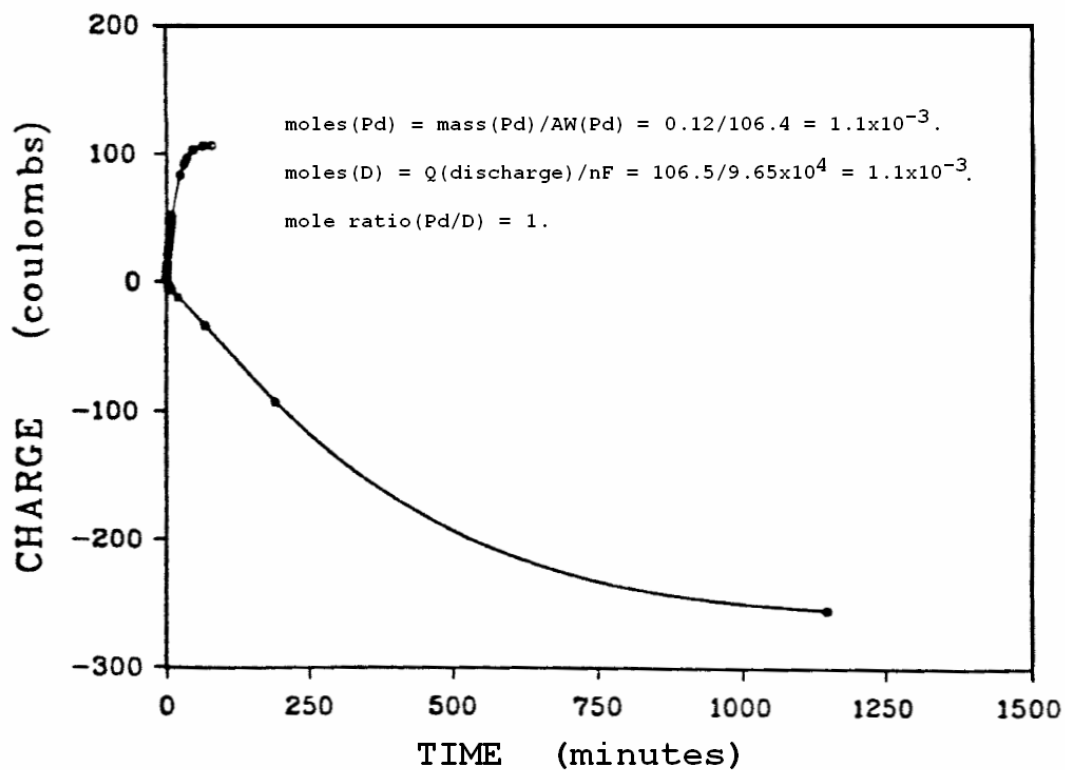**Fig. 3. Chronocoulometry curve for the charging (negative charge) and discharging (positive charge) of D into palladium wire (d=0.03 cm). Electrode area = 1.86 cm^2. Electrode mass = 0.12 grams. Electrolyte: 0.1 $\underline{M}$ LiOD in D$_2$O. Charging potential = -0.35 V vs RDE. Discharging potential = +0.83 V vs RDE.**