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INVESTIGATION OF PHENOMENA RELATED TO D₂O
ELECTROLYSIS AT A PALLADIUM CATHODE

by

R. R. Adzic, D. Gervasio, B. Cahan,
I. Bae and E. Yeager

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Case Center for Electrochemical Sciences and
the Department of Chemistry
Case Western Reserve University
Cleveland, Ohio 44106

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INVESTIGATION OF PHENOMENA OCCURRING DURING D₂O ELECTROLYSIS AT A PALLADIUM CATHODE

R. R. Adzic*, D. Gervasio, I. Bae, B. Cahan and E. Yeager

Case Center for Electrochemical Sciences
and the Department of Chemistry
Case Western Reserve University
Cleveland, Ohio 44106

ABSTRACT

Measurements have been carried out on the phenomena reported by Fleischmann and Pons to occur during the electrolysis of D₂O solutions with a palladium cathode. The measurements include: 1) the determination of tritium concentrations in the cells 2) the determination of the D/Pd ratios by coulometry; 3) surface analysis of the Pd cathode; and 4) neutron radiation measurements. Enhancement of tritium in the D₂O solution was found in two open-type glass cells as well as in four other cells with Ni anodes. The largest enhancement factor found was ~50. The source of this enhancement remains unexplained. Surface analysis revealed a number of impurities on the Pd surface including Si, Pt (with Pt anodes) and Li. The latter was found to penetrate 200 nm into the Pd bulk. The neutron measurements were inconclusive.

INTRODUCTION

Fleischmann, Pons and Hawkins (1) and Jones et al. (2) have reported evidence for the nuclear fusion of deuterium electrochemically injected into palladium. Excess heat generation, neutrons and γ -rays emission and tritium enhancement in the electrolytic solution were found by Fleischmann et al. (1) and neutron emission by Jones et al. (2). 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 (F-P) type open cell and also with a closed cell with internal D₂/O₂ recombination. Some excess heat generation was observed. These results, however, require further evaluation and will be reported at a future date. Careful determination of tritium was done in the electrolytic solution and also in the water obtained from the recombined gases outside the cell. Surface analysis of Pd cathode

*Permanent Address: Institute of Electrochemistry, ICTM, University of Belgrade, Belgrade, Yugoslavia.

was performed and the D/Pd ratio determined. Neutron radiation measurements were attempted, but difficulties in making background corrections interfered with the measurements.

EXPERIMENTAL

Electrodes

Pd from several sources was used for the cathodes. The rods 0.2 x 10 cm were obtained from the 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. Engelhard's Pd was also used by Westinghouse to prepare 2 mm rods by recasting, drawing and swaging. One or more of the following pre-treatments were used with various samples (as indicated in the tables):

1. Annealing in a vacuum with a small amount of Ar for 24 h at -950°C , followed by cooling in D_2 for some samples
2. Recasting, drawing and swaging of some samples
3. Electrochemical oxidation of H_{abs} in 0.1 M LiOD at $E = -0.5 \text{ V}$ vs. S.C.E. for 24 h, followed by immediate transfer into the calorimetry cells for most samples.

The 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 Li_2O (99.9%) Cerac, Inc. in a glove bag. Upon diluting this stock solution, 0.1 M LiOD was obtained. The blank experiments in 0.1 M LiOH in H_2O have not yet been carried out.

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-FLUOR 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 the separation of the water from the cell electrolytic solution by distillation at reduced pressure before the tritium measurements. While this procedure minimized the possibility of interference effects with some constituents of the electrolyte, it causes 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.

Codes
/or

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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 BF₃ 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 such measurements in the future.

RESULTS AND DISCUSSION

Tritium Measurements

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 (11). 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 concentration 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 D₂O containing T and due to its removal by electrolysis. The isotopic separation factor of T to D is defined as

$$S = (n_D/n_T)_g / (n_D/n_T)_s \quad (1)$$

where n_D and n_T are numbers of deuterium and tritium atoms in the gas (g) and solution (s) phases respectively, at steady state. It can be shown (14) that the enhancement of tritium in the electrolytic solution at time (t) obeys the following expression:

$$n_T(t)/n_T(0) = S - (S-1)\exp(-t/T) \quad (2)$$

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 D₂ and O₂ is carried out in the cell or outside the cell and the D₂O returned to the cell, only 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 24h in LiOD solution. This treatment should have removed any tritium present in the Pd samples.

In all of the 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 out of five open-type 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. The 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. The 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 distilled and only the distillate was added to the scintillation 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 distilled vs. neutralized samples was usually 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 experiment, 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:

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

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

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

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

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 power generated in these open cells with the amount of T found. If all of the energy were coming from the tritium generating reaction,

*The tritium concentrations in the samples of electrolyte from the cells in the present work have been checked by S. Landau of Case Western Reserve University and Z. Minevski of Texas A & M University, whose help in this matter is acknowledged.



this would produce $4.03 \text{ MeV} = 4.03 \cdot 10^6 \text{ eV} \cdot 1.6022 \cdot 10^{-19} \text{ J/eV} = 6.46 \cdot 10^{-13} \text{ J/reaction}$. If 1 mW of heat is produced in a cold fusion reaction per 1 cm³ of Pd the corresponding number of T atoms would be $10^{-3} \text{ W} / (6.46 \times 10^{-13} \text{ W s/reaction}) = 1.55 \times 10^9 \text{ reactions/s}$; i.e. 1.55×10^9 T atoms would need to

be produced. Tritium decays by the reaction



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

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

The decay constant, related to the half life is (16)

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

For 1 mW one should observe

$$-\frac{dN}{dt} = (1.782 \times 10^{-19} \text{ s}^{-1}) (1.55 \times 10^9) = 2.76 \text{ dps} = 166 \text{ dpm of T} \quad (9)$$

Therefore, the observed 766 and 1730 dpm ml⁻¹ can account only for ≈ 4 -10 mW, i.e., three orders of magnitude below experimental observations (1). Much smaller, but still significant, enhancement of tritium was found in the gas phase for cells 1 and 4 after recombination of O₂ with D₂ 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.

The impurity layers on the Pd cathodes in the glass and nickel cells can seriously perturb the processes occurring at the cathode surface. Despite this layer, the palladium is still charged with deuterium. Definitive work, however, requires that these impurities are eliminated so that the behavior on clean palladium can be achieved. Then, the impurity effects can be tracked by the addition of each impurity under well defined condition. Such a study is needed but will prove very time consuming and expensive. Furthermore, if tritium is present in some of the materials used in the cell, it is necessary to explain why the large enhancement occurs only after long electrolysis times. One remote possibility, however, is that the Pd contained a relatively stable metal tritide which did not decompose at the temperatures involved in the vacuum pre-treatment of the Pd (e.g., 900°C). If this compound is quite stable and present in a separate phase, then the diffusion of this tritide to the Pd

surface would be extremely slow and probably at a negligible rate even on a time scale of weeks and months. The charging process, however, produces stress cracking and this would then provide a possible mechanism by which the electrolyte solution could reach this tritide and become contaminated with T.

AES and SIMS Analyses of the Composition of Pd Cathodes

Auger electron spectroscopy (AES) and secondary ion mass spectroscopy (SIMS) were the *ex situ* analytical techniques used for examining the surface of the palladium cathodes used to electrolyze D₂O in deuterated aqueous 0.1 M LiOD solutions. The electrodes were removed from the cell, blotted with towel paper, and dried. A dry electrode was introduced into the vacuum chamber ($\sim 10^{-8}$ Torr), where the surface composition was examined with AES and SIMS. The surface was sputtered with an Argon ion beam in order to remove an approximately 90 Å thick layer (sputter rate = -5 Å/s; time interval = 18 s), and the newly exposed surface was then analyzed. A depth profile of the electrode composition was obtained by repeatedly sputtering for a fixed interval of time and then analyzing the newly exposed surface. The depth profiling was continued until the AES and SIMS responses were virtually constant.

The Pd cathodes were examined from the two different types of cells. The major difference in the construction of these two cells, as it pertains to these surface spectroscopy studies, is that the F-P type cell was made of glass, whereas the cell used in the Tronac calorimeter was made of nickel.

Figures 1 and 2 show the Auger spectra for the Pd cathode used in the glass cell for 75 days when the sample was first introduced into the vacuum chamber (Fig. 1) and after an $\sim 3,000$ Å thick layer was sputtered off (Fig. 2). The spectra are markedly different. The spectrum of (Fig. 1) shows peaks for Si, C, O, Mg and Al, but there is no peak for Pd. The spectrum of the surface after sputtering (Fig. 2) showed one major peak characteristics of Pd.

The AES depth profile (Fig. 3) suggests that the composition found for the electrode surface (Fig. 1) prior to sputtering persists for $\sim 1,300$ Å, and then the composition of the electrode gradually changes to the Pd rich bulk surface composition (Fig. 2) between 1,300 to 1,800 Å. No further noticeable changes were observed in the depth profile between 1,800 to 3,000 Å.

A second piece of the same Pd cathode cut off after the electrochemical measurement was examined with SIMS. In principle, the SIMS technique should parallel the AES technique and also should be useful for detecting elements not observable with the AES technique, particularly lighter elements such as Li. There are pronounced peaks for masses 6 and 7 in the SIMS data for the surface (Fig. 4) before extensive sputtering. These peaks, which represent Li⁶ and Li⁷, are larger than the peaks at mass 16, corresponding to O¹⁶, which gave a prominent peak in the Auger spectrum for this surface (Fig. 1). The SIMS peaks for Li persist even after sputtering ~ 9000 Å off the original surface.

Figures 5 and 6 show the Auger spectra for the Pd cathode 1 nm diameter wire used in the Ni cell, when the sample after two weeks of use in the electrochemical cell was first introduced into the vacuum chamber (Fig. 5) and after an $\sim 10,500$ Å thick layer was sputtered off the original surface (Fig. 6). The spectrum of the unsputtered surface shows peaks for C, Ni and O (Fig.

5), but none for Pd. The spectrum of the surface exposed after sputtering shows only one major peak characteristics of Pd (Fig. 6). The AES depth profile (Fig. 7) suggests that the composition found for the virgin surface persists for $\sim 1,000$ Å, and then the composition gradually becomes richer in Pd and poorer in C, Ni and O between 1,100 to 6,300 Å. The Pd rich bulk spectrum (Fig. 6) persisted between 6,300 to 10,500 Å from the original surface.

These *ex situ* studies show impurities masked the Pd from the surface for Pd cathodes used in both the F-P type and Ni cells. Ni was found only in the Pd cathode used in the Ni cell, and probably came from the Ni cell case. Si, Mg and Al were found only in the Pd cathode used in the glass cell, and probably these came from the glass cell case.

Coulometric Determination of D Absorbed in Pd Cathodes

Metallic palladium absorbs D to form a solid PdD_x solution during the electrolysis of D_2O at the cathode. The electrolysis of D_2O in the Pd is sensitive to the conditions at the Pd surface. A coulometric stripping technique was used to analyze the amount of D absorbed in the Pd during the electrolysis. 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. During all electrochemical measurements, the Pd coil of the working electrode was completely submerged in an anaerobic electrolyte solution consisting of 0.1 M LiOD in D_2O . A three compartment electrochemical cell was used. The electrolyte in the working electrode chamber was continuously and vigorously degassed with N_2 (99.995%, Ultrapure, Matheson). The counter electrode, a coil of Pd wire ($A = 2 \text{ cm}^2$), 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) which was submerged into the reference chamber which in turn led to the working electrode chamber via a Luggin capillary. All potentials are given versus RDE unless stated otherwise.

The working electrode was rinsed with 0.1 M LiOD in D_2O and cycled between 0.25 and 1.35 V until the steady state voltammogram shown in Fig. 8 (dashed line) was obtained. The cell was then rinsed and refilled with fresh electrolyte solution and sparged with N_2 gas. The steady state voltammogram was observed after only a few cycles. 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 more positive or negative potentials.

Figure 9 shows the chronoamperometry curves for the charging of Pd with D at -0.3 V (negative current) and discharging of D from Pd at +0.83 V (positive current). During the charging at -0.3 V, O_2 was clearly seen evolving at the counter electrode, but when the N_2 bubbler was lifted out of the electrolyte solution in the working electrode chamber, 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. 10, bottom curve) after 10 h. It was also noticed that after 10 or more hours that if the N_2 bubbler was lifted out of 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. Most of the charge measured before 10 h can probably be associated with the absorption of D by Pd, and the charge measured afterwards is probably associated mostly with D_2 evolution at the Pd surface.

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. 9 and 10) took over 10 h, discharging of D from Pd at +0.83 V (see top curves in Figs. 9 and 10) was completed after ~70 minutes. The relatively quicker discharging versus charging of D through Pd is consistent with a greater D concentration gradient and Pd lattice spacing within the electrode expected during the discharging compared to the charge process.

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 D_2 evolution did appear to increase with increasing overpotential. This is evidenced by two observations: 1) the nucleation of bubbles within minutes of starting the charging of the Pd with D at -0.35 V compared to after ~10 h of starting the charging of the Pd with D at -0.3 V and 2) the difference in the chronocoulometry curves for charging at -0.3 versus -0.35 V. Figure 9 shows the chronocoulometry curve for loading D into Pd at -0.35 V (see bottom curve). Comparing this and the analogous curve in Fig. 10 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, $\text{moles}(D) = Q(\text{discharge})/nF$, 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 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 overpotentials (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 claim 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 .
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. Surface analysis using AES and SIMS revealed a number of impurities on Pd surface after a long time electrolysis including Si, Pt or (Ni) and Li which was found to penetrate into the bulk of Pd.
4. The neutron radiation measurements were inconclusive due to the uncertainty in determining the background correction.

In general, on the experimental side there are 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 of 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 3 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 3He . The branching ratio of reaction A and B (Table 4) is approximately one. The cross section for reaction C is on the order of 10^7 lower than for A and B (16). γ -rays should be observed if the reaction K occurs in the electrochemical cell.

The proposition that electrochemically induced fusion is involved does not satisfy 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 (7, 8), 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 a explanation for tritium generation, due to increased electric fields around the dendrite tips (3). 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 to reach an complete understanding of the Fleischmann and Pons phenomenon.

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Table 1: Cell parameters for Tritium measurements

Cell	Electrode size d x l (cm x cm)	Supplier	Pretreatment	Anode	Current (mA/cm ²)
1	0.4 x 6	JM ^a	oxid. of H _{abs}	Pt	5-400
2	0.2 x 10	E ^b	"	Pt	60-120
3	0.1 x 5	JM	"	Ni	10-120
4	0.2 x 5	E	"	Ni	10-120
5	0.1 x 5	JM	Annealing	Ni	10-120
6	0.2 x 5	E	"	Ni	10-120
7	0.2 x 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)	NCR ^{sample}	DPM/ml ^c
				$\frac{\text{D}_2\text{O}}{\text{NCR}}$	
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 $\pm$ 1.20	85.99 $\pm$ 0.99	24.6 $\pm$ 8.3	766
	gas phase 68	42.66 $\pm$ 2.32	24.06 $\pm$ 0.93	8.3 $\pm$ 2.3	114
	75	34.05	18.05	6	160
	gas phase 75	20.93	4.93	1.6	44
2	25	209.27 $\pm$ 0.80	193.80	6.2	1730
	45	120.06 $\pm$ 0.49	103.06	4.1	920
3	26	159.14 $\pm$ 1.83	143.24 $\pm$ 1.88	49.4 $\pm$ 13.8	1278
	gas phase 26	26.53 $\pm$ 0.86	9.63 $\pm$ 0.96	3.3 $\pm$ 0.9	85
	36	39.14 $\pm$ 1.95	22.73 $\pm$ 1.97	14.2	202
4	26	91.58 $\pm$ 1.58	75.68 $\pm$ 1.64	26.1 $\pm$ 7.3	675
	36	36.94 $\pm$ 0.54	20.53 $\pm$ 0.60	12.8	183
5	26	58.11 $\pm$ 0.27	42.21 $\pm$ 0.51	14.6 $\pm$ 4.1	376
	36	40.24 $\pm$ 0.34	23.83 $\pm$ 0.43	14.8	212
	76 ^d	37.03 $\pm$ 0.40	19.63	9.67	87
	gas phase 76 ^d	29.00 $\pm$ 0.40	11.60 $\pm$ 0.71	5.71 $\pm$ 1.94	51
6	26	80.54 $\pm$ 0.48	64.64 $\pm$ 0.64	22.3 $\pm$ 6.2	577
	gas phase 26	39.40 $\pm$ 0.86	23.50 $\pm$ 0.96	8.1 $\pm$ 2.3	209
	36	25.35 $\pm$ 0.37	8.94 $\pm$ 0.46	5.5	79
	gas phase 56 ^d	33.88 $\pm$ 0.55	16.48 $\pm$ 0.81	8.12 $\pm$ 2.75	73
	76 ^d	35.94 $\pm$ 0.55	16.48 $\pm$ 0.81	9.13 $\pm$ 3.09	82
	gas phase 76 ^d	26.62 $\pm$ 0.38	9.22 $\pm$ 0.70	4.54 $\pm$ 1.56	41
7	16	22.21 $\pm$ 0.49	6.31 $\pm$ 0.65	2.18 $\pm$ 0.65	56
	36	20.93 $\pm$ 0.86	4.52 $\pm$ 0.63	2.8	40
	76 ^d	21.76 $\pm$ 0.86	4.36 $\pm$ 1.04	2.15 $\pm$ 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 D₂O) = -46 cpm; ACR(Isotech D₂O) = -19cpm.

TABLE 3

Summary of the Calculated Amount of the D
Loaded in Pd^a at Different Overpotentials

η (volts)	Qdischarge(C)	moles D
-0.30	106.5	1.1×10^{-3}
-0.35	170.0	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.

TABLE 4

Nuclear Fusion Reactions

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

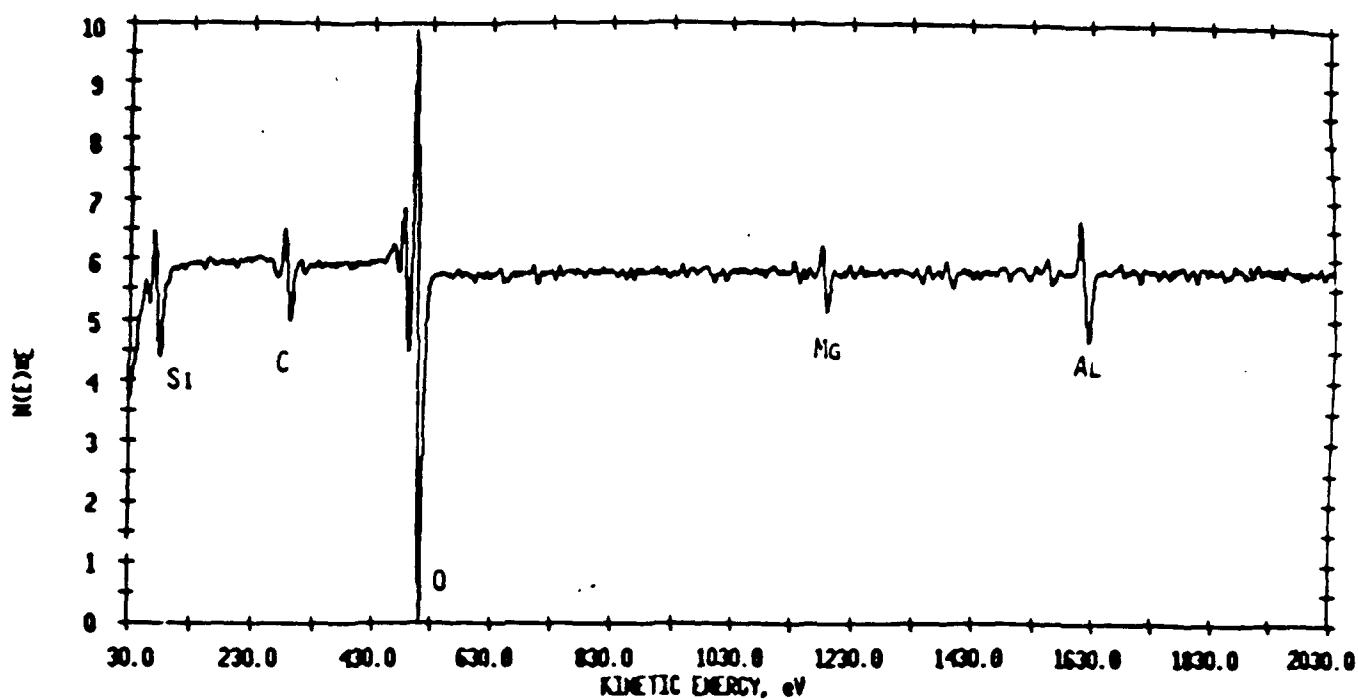


Fig. 1. Auger electron spectrum of Pd cathode (Johnson Matthey, "Fusion rod") used in a glass cell for the electrolysis of D_2O in 0.1 M LiOD. As introduced into the spectrometer with no sputtering of the surface. Incident beam energy = 10 KeV. Sampled area $25 \times 25 \mu m^2$.

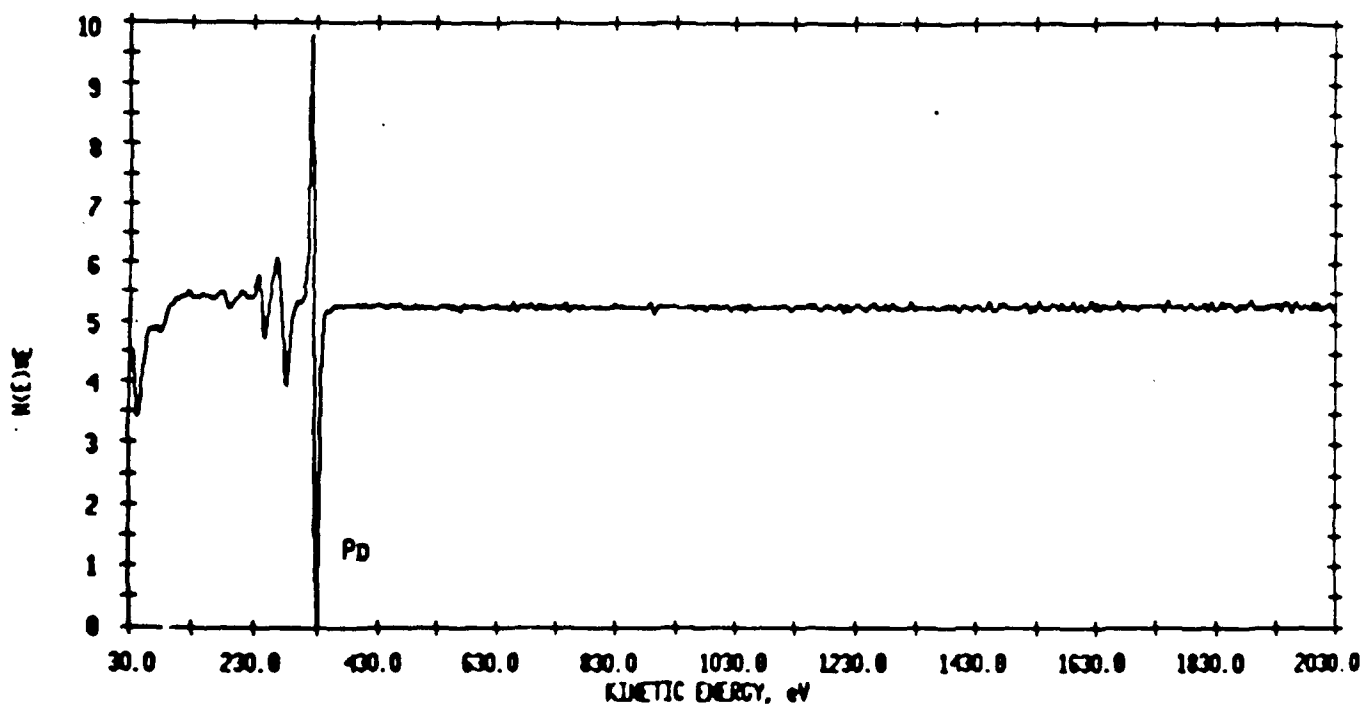


Fig. 2. Auger electron spectrum of Pd cathode (Johnson Matthey, "Fusion rod") used in a glass cell for the electrolysis of D_2O in 0.1 M LiOD. After a $-3000 \text{ \AA}$ layer was sputtered off the original surface. Incident beam energy = 10 KeV. Sampled area $25 \times 25 \mu m^2$.

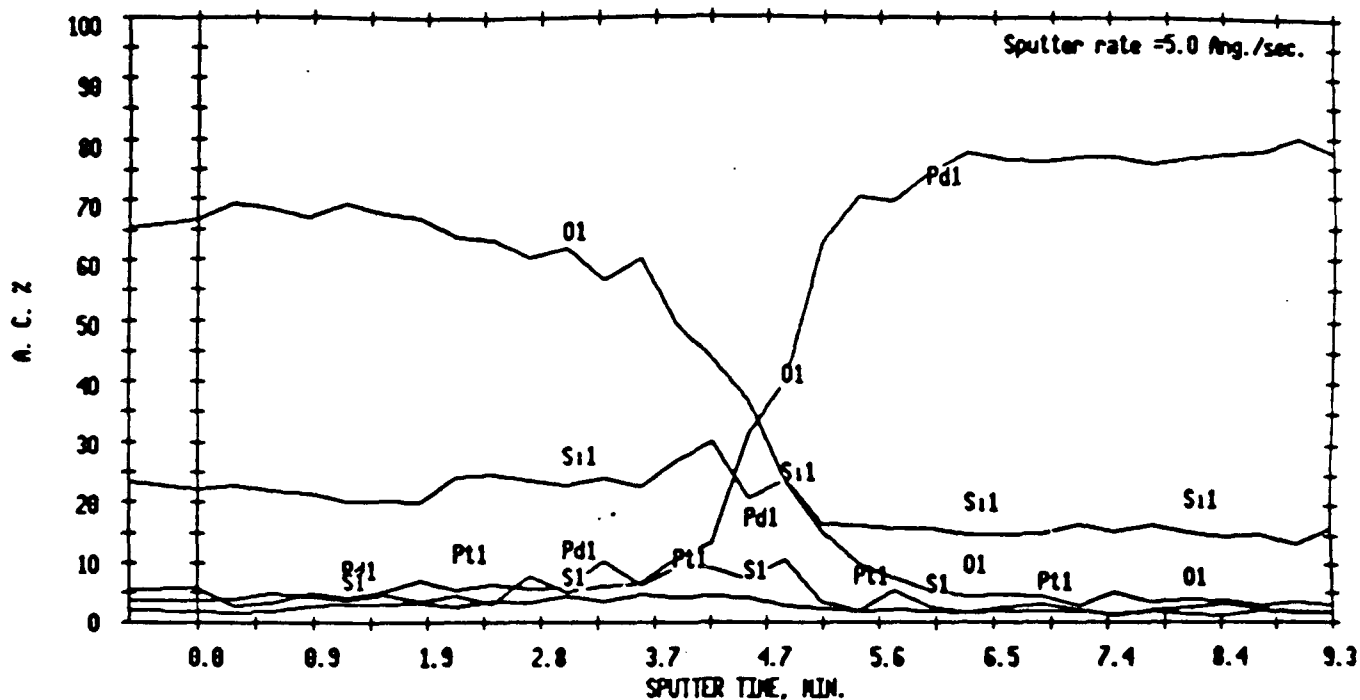


Fig. 3. Auger electron spectrum depth profile of Pd cathode (Johnson Matthey, "Fusion rod") used in a glass cell for the electrolysis of D_2O in 0.1 M LiOD. Sputter rate = -5 Å/sec. Incident beam energy = 10 KeV. Sampled area $25 \times 25 \mu m^2$.

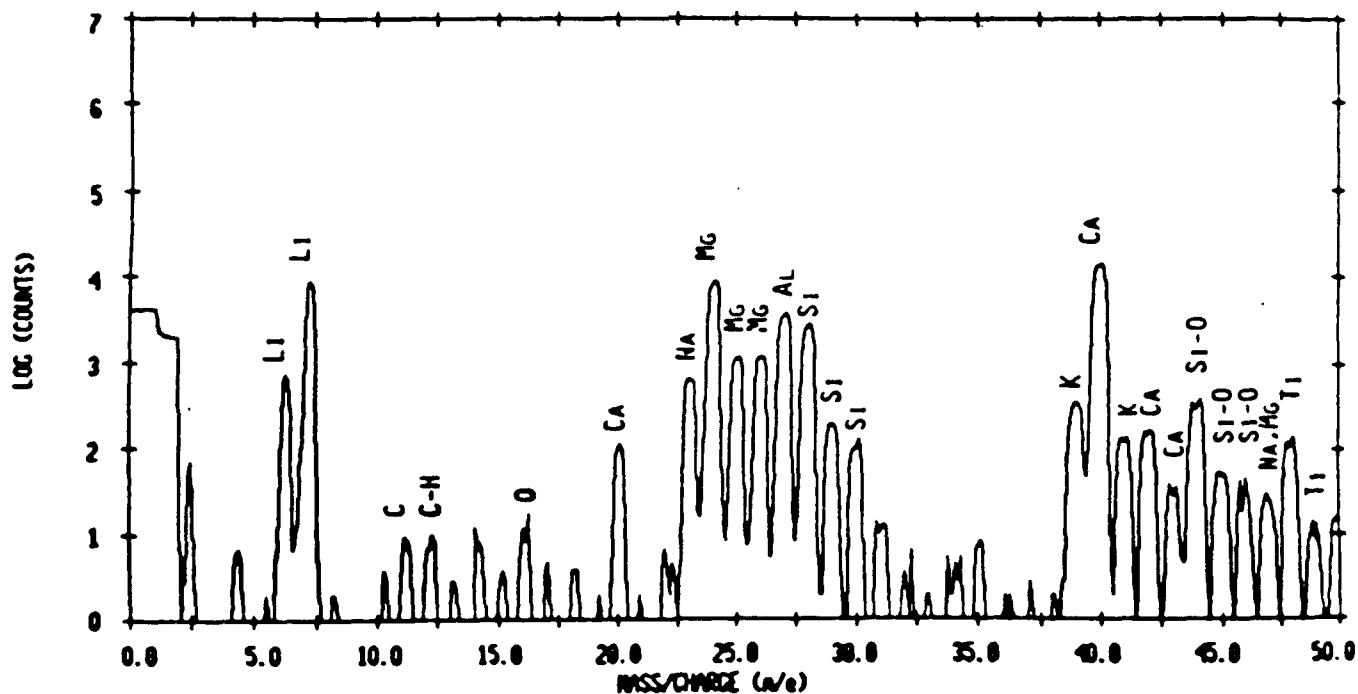


Fig. 4. Positive Secondary ion mass spectrum survey of the Pd cathode (Johnson Matthey, "Fusion rod") used in a glass cell for the electrolysis of D_2O in 0.1 M LiOD. Incident beam energy = -3 KeV. Ion current = -4 μA . Sampled area $1 \times 1 mm^2$.

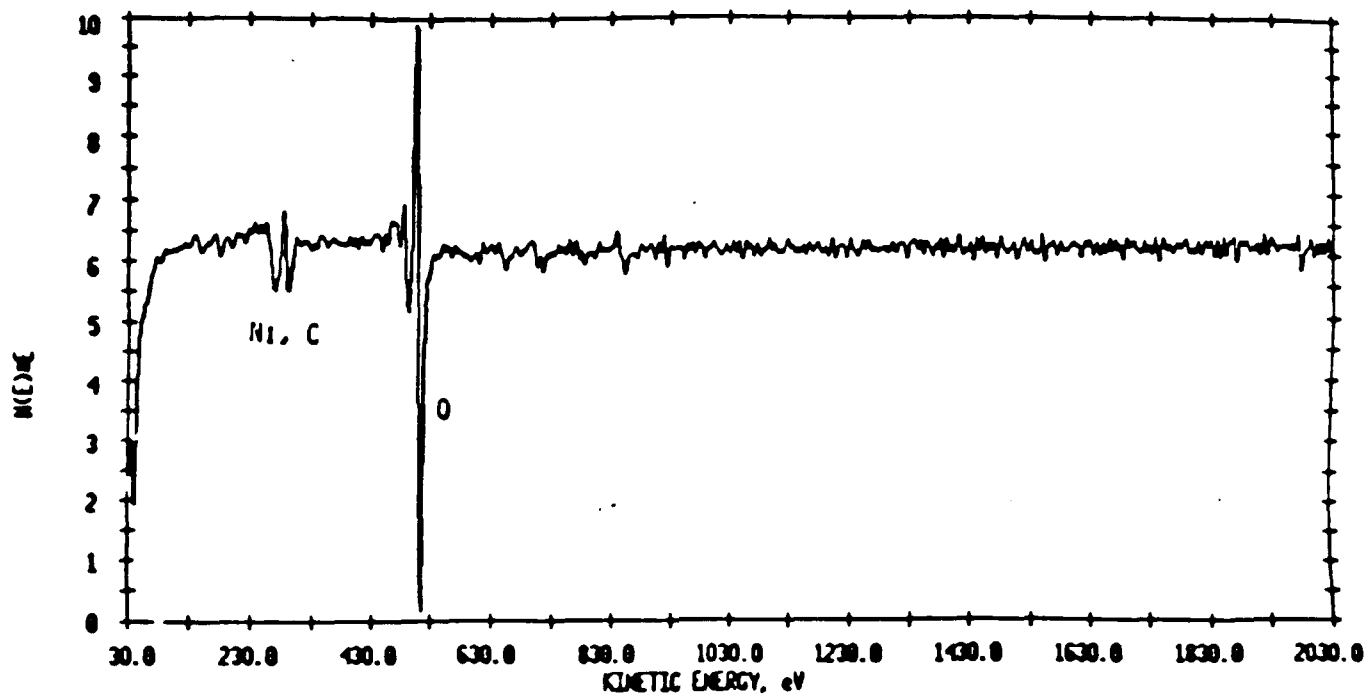


Fig. 5. Auger electron spectrum of Pd cathode (Englehard) used in a Nickel cell for the electrolysis of D_2O in 0.1 M LiOD. As introduced into the spectrometer with no sputtering of the surface. Incident beam energy = 10 KeV. Sampled area $25 \times 25 \mu m^2$.

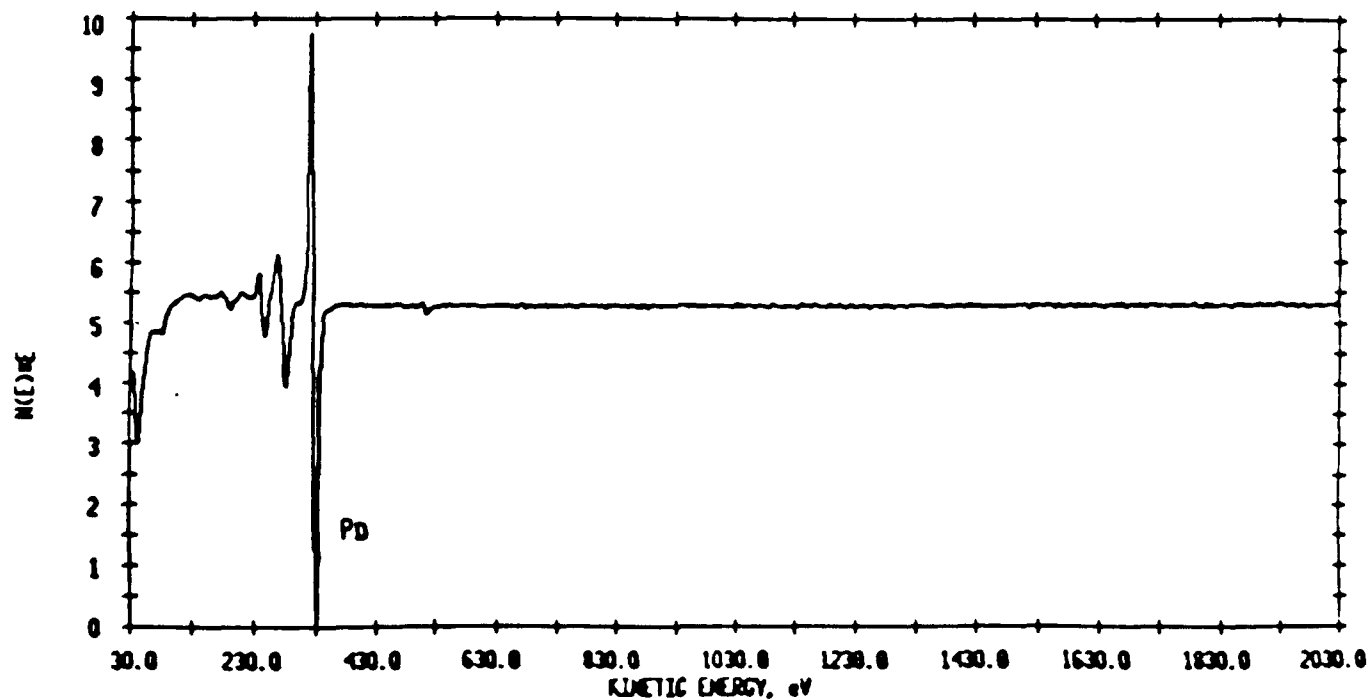


Fig. 6. Auger electron spectrum of Pd cathode (Englehard) used in a Nickel cell for the electrolysis of D_2O in 0.1 M LiOD. After 10,500 Å thick layer was removed from the original surface. Incident beam energy = 10 KeV. Sampled area $25 \times 25 \mu m^2$.

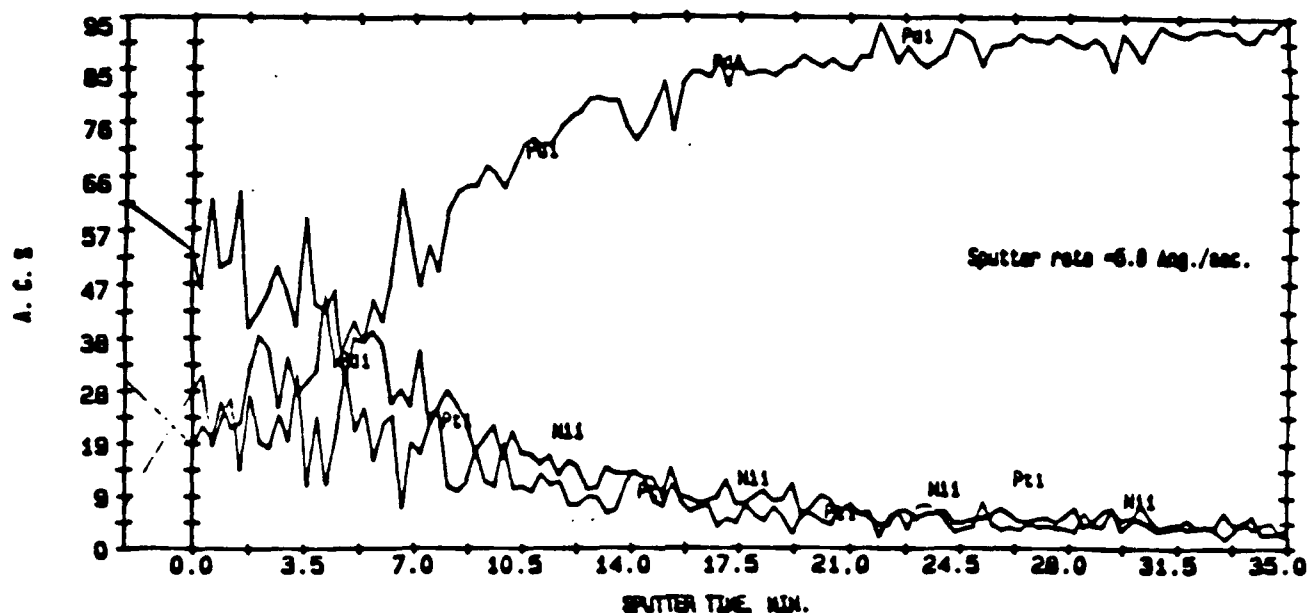


Fig. 7. Auger electron spectroscopy depth profile of Pd cathode (Englehard) used in a Nickel cell for the electrolysis of D_2O in 0.1 M LiOD. Sputter rate = 5 Å/sec. Incident beam energy = 10 KeV. Sampled area $25 \times 25 \mu m^2$.

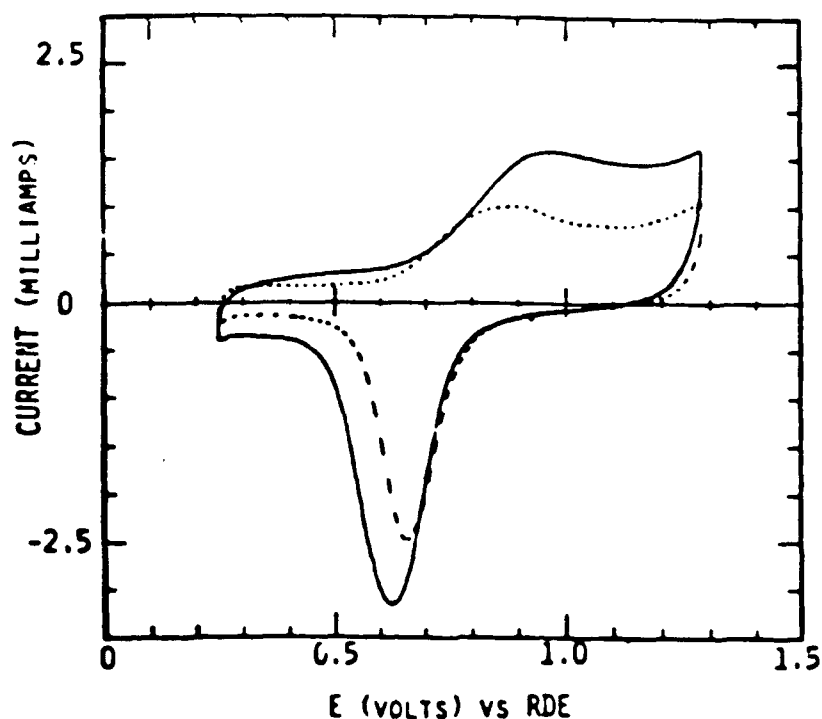


Fig. 8. Cyclic voltammograms of Palladium wire ($d = 0.03$ cm) in D_2O with 0.1 M LiOD under a N_2 atmosphere. Dashed line is the steady state voltammogram. Solid line is the voltammogram recorded after charging the Pd with D at -0.4V for 24 hours, followed by discharging the D at +0.83 V for 60 minutes. Scan Rate = 100 mV/s. Electrode area = 1.86 cm^2 . Counter electrode: Pd.

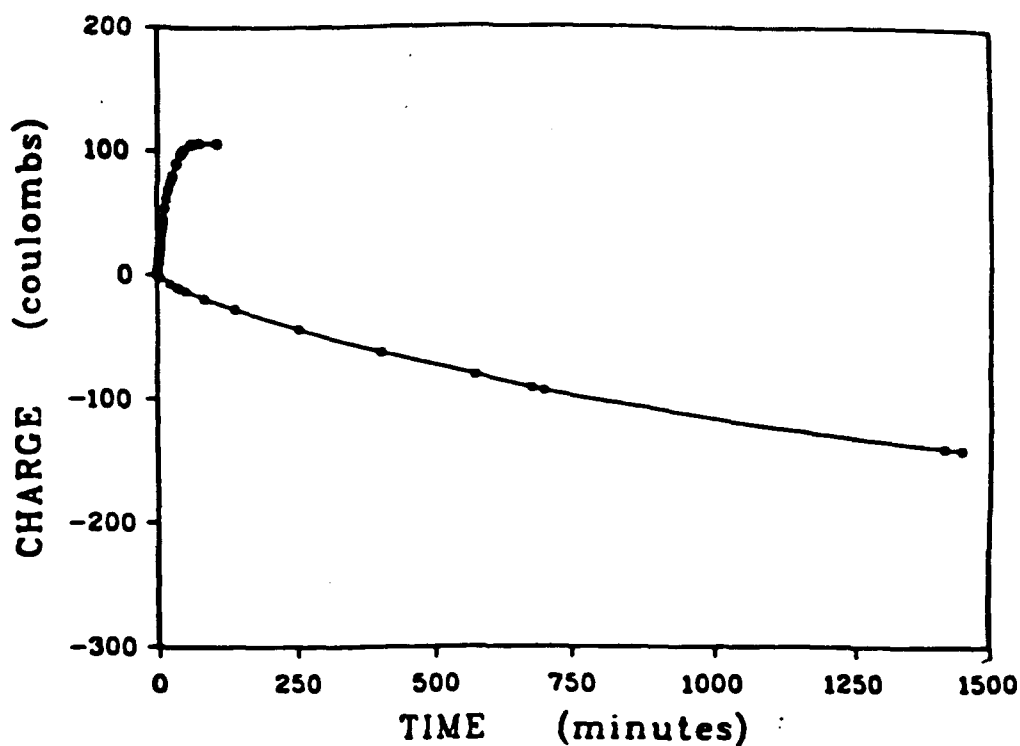


Fig. 9. Chronocoulometry curve for the charging (negative charge) and discharging (positive charge) of D into palladium wire ($d=0.03$ cm). Electrode area = 1.86 cm^2 . Electrode mass = 0.12 grams. Electrolyte: 0.1 M LiOD in D_2O . Charging potential = -0.3 V vs RDE. Discharging potential = $+0.83 \text{ V}$ vs RDE.

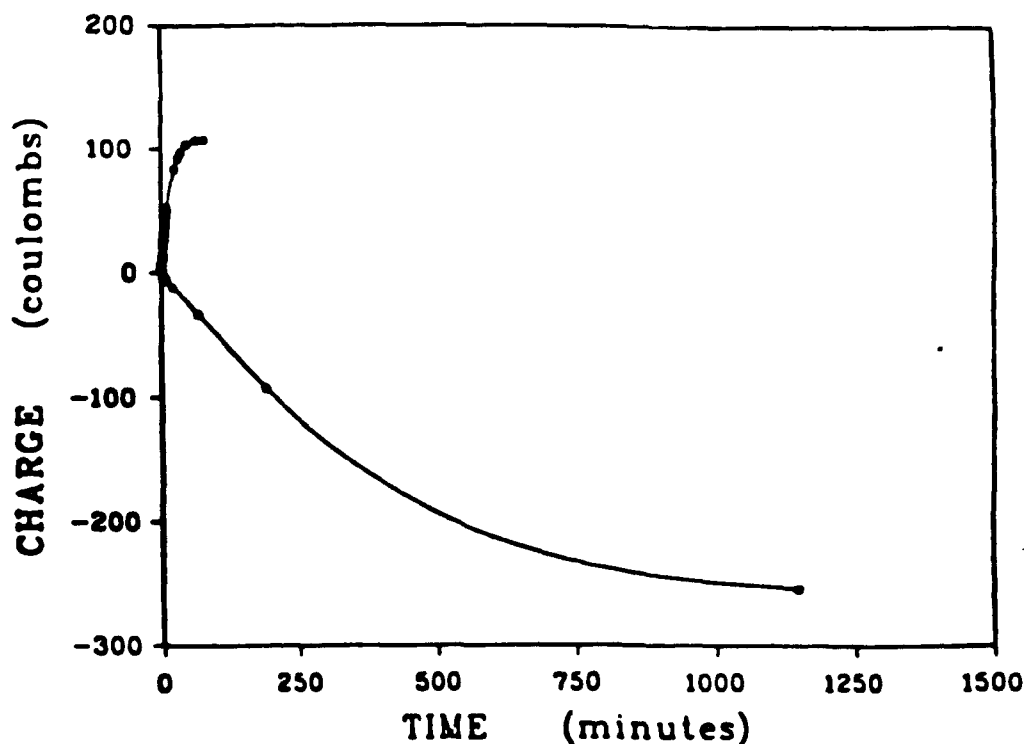


Fig. 10. Chronocoulometry curve for the charging (negative charge) and discharging (positive charge) of D into palladium wire ($d=0.03$ cm). Electrode area = 1.86 cm^2 . Electrode mass = 0.12 grams. Electrolyte: 0.1 M LiOD in D_2O . Charging potential = -0.35 V vs RDE. Discharging potential = $+0.83 \text{ V}$ vs RDE.