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ELECTROCHEMICAL CALORIMETRIC STUDIES OF THE COLD FUSION EFFECT

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ABSTRACT

Several types of calorimetric cell designs were used in attempts to measure excess enthalpy during the electrolysis of LiOD/D₂O using palladium cathodes. Control experiments were run by using light water in place of D₂O or by using platinum cathodes in place of palladium. Initial experiments using thin palladium cathodes of an unknown purity gave no significant differences between the Pd/D₂O cells and the controls. For example, the ratio of heat out to Joule heat in was 1.00 ± 0.04 for one study and 1.065 ± 0.04 for another study in LiOD/D₂O compared to 1.075 ± 0.07 in LiOH/H₂O. The use of a much thicker palladium rod (99.96%, d = 0.635 cm) from Johnson Matthey, however, resulted in calorimetric evidence for excess enthalpy in five out of six cells. The excess rate of heating averaged 0.39 W/cm^3 over a 9-day period in one experiment. The total excess enthalpy observed was 110,000 J. This excess enthalpy is difficult to explain by chemical reactions. Similar experiments conducted in H₂O did not produce significant amounts of excess enthalpy. Possible experimental errors in these calorimetric studies are being investigated.

INTRODUCTION

The electrochemically induced nuclear fusion of deuterium using a palladium electrode reported by Pons, Fleischmann, and Hawkins [1] has sparked a flurry of experimental measurements and considerable controversy [2-6]. The conditions under which this fusion may or may not occur will eventually be determined by many experiments at various laboratories. Enthalpy excesses that can exceed 10 W/cm^3 of the palladium electrode have been claimed [1]. Similar experiments by Jones and coworkers [7] also report evidence for cold nuclear fusion; however, the fusion rates reported are far too small to be detected by calorimetry. The experiments described below are an attempt to detect any excess heat output by calorimetric studies during the electrolysis of deuterium oxide containing LiOD at palladium cathodes. Control measurements were run using light water with palladium cathodes or heavy water with platinum cathodes. Radiation levels were also monitored by various methods.

EXPERIMENTAL

The calorimetric cell design used in most experiments is shown in Figure 1. The electrolysis cell in this configuration can be visualized as a resistive heater with the temperature being measured in the secondary compartment (gap) surrounding the electrolysis cell. The electrolysis cell initially contained 18-20 g of 0.1 m LiOD/D₂O (99.9%, Cambridge Isotope Laboratories)

while the gap contained 68-70 g of distilled water. The alkaline solutions were prepared from lithium metal (ROC/RIC, 99.95%). This cell design minimized the decrease in the calorimetric cell constant with the decrease in the electrolyte solution volume which occurs during electrolysis. Both the electrolysis cell and plastic bottle (polyethylene) were stoppered and wrapped with parafilm to reduce evaporation and contamination. The evaporative losses from both the inner and outer glass vessels were 1% by weight per day. Make up water and heavy water were periodically added to the two compartments. After correcting for evaporation, the measured loss of D₂O due to electrolysis was always within $\pm 1\%$ of the calculated value. The palladium rod cathode (Johnson Matthey, 99.96%, $d = 0.635$ cm, $A = 2.64$ cm²) was spot-welded to a nickel lead. Both the anode and cathode leads were covered with heat shrinkable Teflon tubing to prevent exposure of the bare metal to the gases in the headspace. Two thermister thermometers (Cole-Parmer, Model 8502-16) calibrated within $\pm 0.01^\circ\text{C}$ were inserted into small glass tubes placed in the gap H₂O and positioned 4.6 cm (T₁, T₄) and 1.9 cm (T₂, T₅) from the bottom of the cell. Two identical calorimetric cells containing two thermistors each as shown in Figure 1 were always run simultaneously in a constant temperature bath ($T_{\text{bath}} = 27.50^\circ\text{C}$). Identical coils of Pt - 20% Rh (5.35 g, $d = 0.1$ cm) served as the counter electrodes.

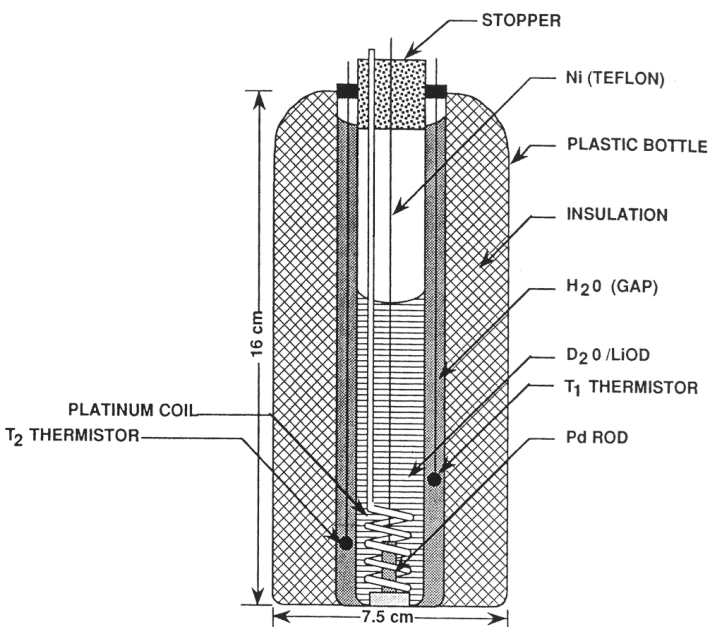


Figure 1. Calorimetric Cell Design.

Earlier experiments used a palladium wire cathode ($d = 0.14$ cm, $A = 2.64$ cm², Wesgo) of an unknown purity spot-welded to a Pt - 20% Rh lead that was covered with heat-shrinkable Teflon tubing. Platinum wire cathodes ($d = 0.12$ cm, $A = 2.64$ cm²) were used in several control experiments. Precision thermometers graduated in units of 0.1°C were used in these earlier studies. Several other calorimetric cell designs were also used that involved measuring the temperature directly in the electrolysis cell and using a correction factor to compensate for the decrease in the calorimetric cell constant with solution volume.

The constant current source for electrolysis was a Princeton Applied Research (PAR) potentiostat/galvanostat (Model 373) set at 264 mA (100 mA/cm²). Calorimetric cell constants were usually determined during the first day of electrolysis when no excess enthalpy is expected. Experiments using palladium cathodes in H₂O or platinum cathodes in D₂O gave nearly the same

cell constants. In earlier experiments, calorimetric cell constants were determined by Joule heat calibrations with a 29 ohm resistor.

RESULTS

Calorimetric studies using the thin palladium and platinum wire cathodes in 0.1 m LiOD/D₂O are presented in Figure 2. The equation

$$X = \frac{\text{Heat out}}{\text{Joule heat in}} = \frac{K \cdot \Delta T}{(E - E_H^o) \cdot I} \quad (1)$$

was used for these experiments where K is the calorimetric cell constant, E is the cell voltage, E_H^o is the thermal neutral potential for the cell reaction, and ΔT is the temperature difference measured within the gap and the outer surface of the polyethylene bottle (Fig. 1). The daily mean values from a series of measurements are shown in Figure 2. The overall mean value of $\bar{X} = 0.97 \pm 0.06$ for the platinum cathode indicated that the calorimetric design was satisfactory. Furthermore, the overall mean value of $\bar{X} = 1.00 \pm 0.04$ for palladium showed that there was no measurable excess enthalpy generation due to the cold fusion effect in this experiment.

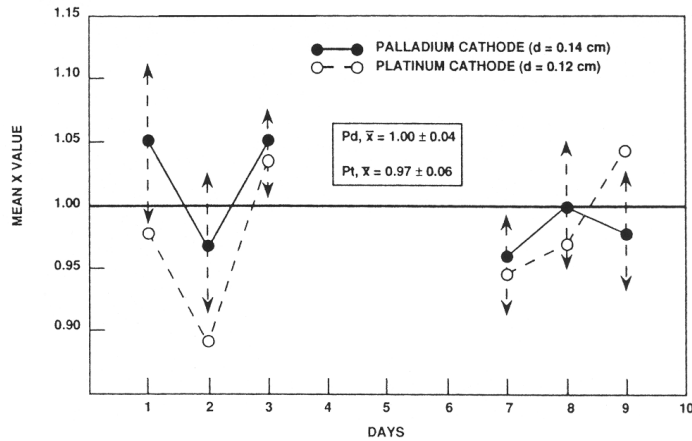


Figure 2. Comparison of Palladium and Platinum Wire Cathodes in LiOD/D₂O. The standard deviation for each day is indicated for the palladium cathode. Both cells were charged at 20 mA/cm² between days 3 and 7. Current density was 100 mA/cm² for all other days except day 3 (200 mA/cm²).

Earlier experiments in a thermos-type cell with a precision thermometer placed directly in the electrolyte solution yielded mean values of $\bar{X} = 1.065 \pm 0.04$ for the palladium wire cathode in LiOD/D₂O compared to 1.075 ± 0.07 in LiOH/H₂O. In these studies, the equation used to correct the K values due to the volume change was

$$K_t = (0.964 V_t/V_0 + 0.036)K_0 \quad (2)$$

where K_0 and V_0 represent the initial cell constant and volume, respectively. The initial cell constants determined by Joule heat calibrations were 0.0435 ± 0.0015 W/°C for the D₂O cell and 0.0458 ± 0.0016 W/°C for the H₂O cell. These experiments support the conclusion that no excess enthalpy was generated using the palladium wire cathode. However, most of these experiments were of relatively short duration (1-3 days). Details of these earlier studies that failed to show any excess enthalpy are reported elsewhere [8].

The use of the much thicker palladium rod from Johnson Matthey resulted in calorimetric evidence for excess enthalpy as shown in Figure 3. For the two cells run simultaneously, one cell gave a heat ratio of $\bar{X} = 1.31 \pm 0.05$ for the 11th day while the other cell gave a ratio of only $\bar{X} = 1.05 \pm 0.04$ for measurements that day. The major difference between these two cells was the tighter wrapping of the counter electrode coils about the palladium in the cell that gave the greater X values. The discontinuation of the electrolysis overnight after the 18th day showed that the X values returned to near unity. Results from the thermistors positioned higher in the gap were similar (Figure 4), hence any thermal inversion is unlikely. The daily average for the room temperature ranged from 21.8 to 23.8°C in this study with a mean of $23.0 \pm 0.6^\circ\text{C}$.

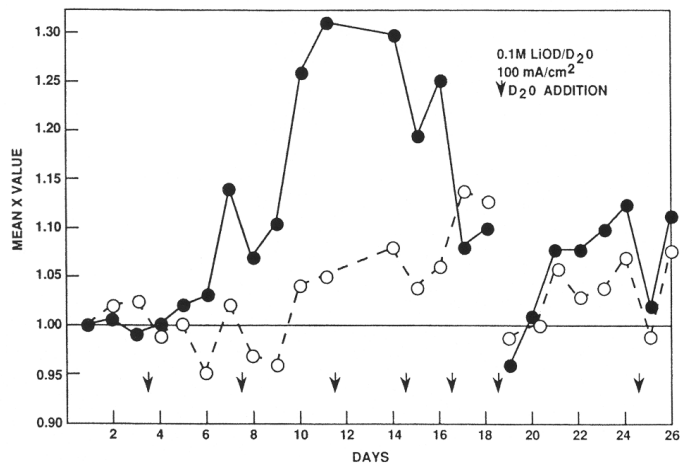


Figure 3. First Cold Fusion Study Using Palladium Rod (Johnson Matthey, $d = 0.635$ cm). Cell A (broken line, T_2) and cell B (solid line, T_5).

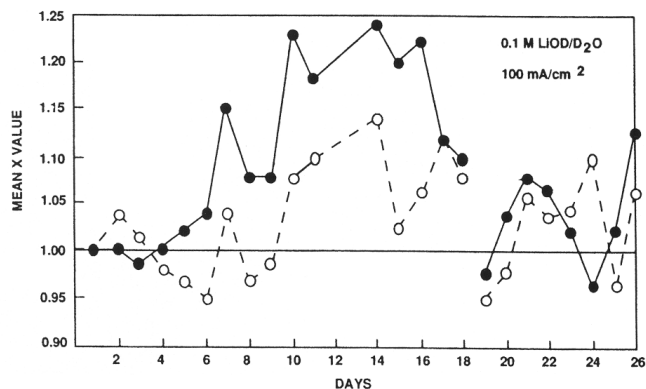


Figure 4. First Cold Fusion Study Using Palladium Rod (Johnson Matthey, $d = 0.635$ cm). Cell A (broken line, T_1) and cell B (solid line, T_4).

A continuation of this cold fusion study following a 10-day shutdown is shown in Figure 5. The excess enthalpy peaks are not as large and appear to be much more periodic with the D_2O additions than before. The additions of small amounts of ZnO to one cell and sulfur to the other cell had no measurable effects. The room temperature control was not as good in this study since the time frame (October, November) involved the onset of cooler weather. The daily average for the room temperature ranged from 19.7 to 23.4°C with a mean of $22.0 \pm 1.0^\circ\text{C}$.

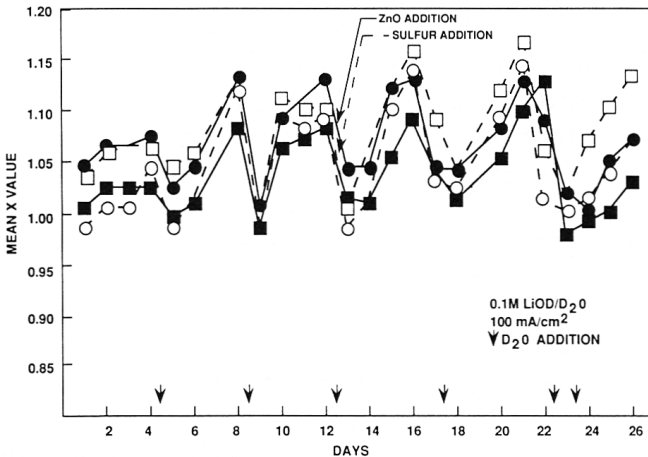


Figure 5. Second Cold Fusion Study Using Palladium Rod (Johnson Matthey, $d = 0.635$ cm). Cell A (broken lines), cell B (solid lines), T_1 (open circle), T_2 (open square), T_4 (solid circle), and T_5 (solid square).

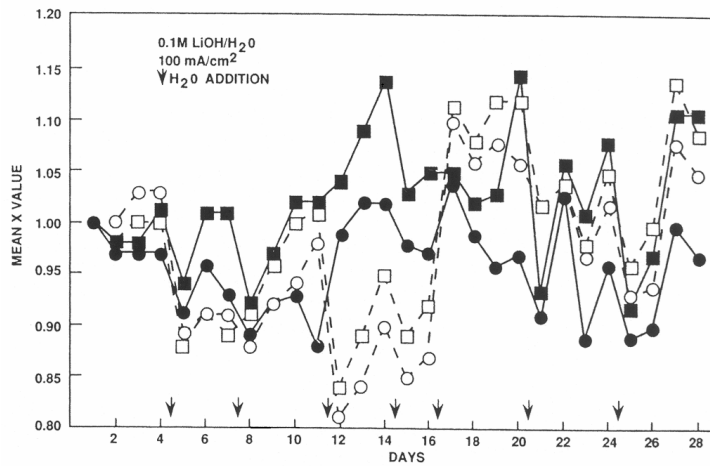


Figure 6. Studies in Water Using Palladium Rod (Johnson Matthey, $d = 0.635$ cm). Symbols for cell A, cell B, T_1 , T_2 , T_4 , and T_5 same as in Figure 5.

Similar studies using the same palladium rod electrodes in cells containing H_2O rather than D_2O are shown in Figure 6. The striking difference from the previous experiments in D_2O are the excursions of the daily mean X values into regions of less than unity. Some rather large differences between the two thermistors in cell B (solid lines) suggest that thermal inversions were occasionally occurring. At times, a temperature instability was noted that was likely due to a mixing of the air in the glass thermistor tube. A portion of the thermistor tube extended above the calorimetric cell and was subjected to cooling by the room air. In later experiments, the thermistor tubes were made flush with the cell top resulting in more uniform temperatures and X values within the same cell. All H_2O measurements were made within an average daily room temperature range of 19.9 - $22.8^\circ C$ with a mean of $21.5 \pm 0.7^\circ C$. Only a weak relationship between the daily X values and the room temperature could be established in this study with a slope less than 0.02 per degree Centigrade and a correlation coefficient less than 0.3 .

A third cold fusion study using the same palladium rod cathodes in fresh $LiOD/D_2O$ solutions is shown in Figure 7. Excellent agreement between the two thermistors in each cell was realized

in this study where the thermistor tubes were flush with the cell top. This experiment shows a nearly consistent excess enthalpy production with only a few days yielding near unity for the heat ratio. However the daily mean X values are noticeably less than those for the first study with a freshly prepared palladium cathode (Figures 3 and 4). The daily average room temperature ranged from 22.4 to 24.5°C in this study with a mean of $23.4 \pm 0.5^\circ\text{C}$.

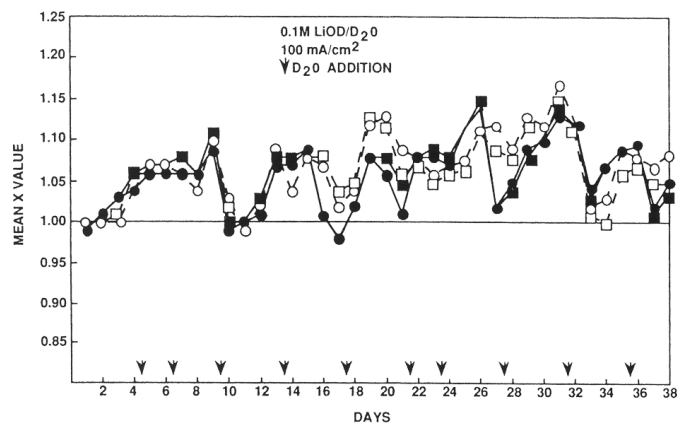


Figure 7. Third Cold Fusion Study Using Palladium Rod (Johnson Matthey, $d = 0.635$ cm). Symbols for cell A, cell B, T₁, T₂, T₄, and T₅ same as in Figure 5.

Tritium measurements on the LiOD/D₂O solutions following the first two experiment using the palladium rods were negative. The two LiOD/D₂O solutions gave 22.99 ± 2.63 cpm and 22.18 ± 1.06 cpm versus 19.34 ± 0.57 cpm for a LiOD/D₂O sample that was never subjected to electrolysis. Results from a second laboratory gave similar conclusions. Theoretical calculations based on the total observed excess enthalpy (563 kJ) yield 4.4×10^7 T atoms mL⁻¹ when the 10^{-9} disparity factor between excess heat and tritium production is used [1]. Assuming no loss of tritium to the gas phase and a 100% counting efficiency, this amount of tritium would only yield 4.7 cpm, hence, the tritium produced would have been difficult to detect from the background.

DISCUSSION

Interpolations of the results reported by Pons, Fleischmann, and Hawkins [1] yield an expected heat ratio of $X = 1.95$ for our palladium rod at 100 mA/cm^2 . Such a high X value would unquestionably be well outside the range of any calorimetric errors. Statistical tests must be applied to our smaller X values to determine any significant difference from unity. Nevertheless, the comment [5] that it is most unlikely that the excess thermal energy due to a fusion process would be of the same order of magnitude as the electrical energy input is perplexing. An order of magnitude larger fusion effect involving D₂O electrolysis with a palladium cathode, however, likely would have been discovered years ago.

The overall mean heat ratios obtained from the various experiments using the palladium rod cathodes as well as the 99% confidence intervals are presented in Table I. The first five days of data were omitted to allow the palladium to become charged with deuterium (n is the number of days of data considered). The excess enthalpy for cell B (\bar{X}_4, \bar{X}_5) was significant at the 99% confidence level in all three D₂O experiments. For cell A (\bar{X}_1, \bar{X}_2), the excess enthalpy was significant at the 99% level in all except the first D₂O experiment that had the widely spaced counter electrode coils. The experiment in H₂O yields mean heat ratios close to unity for both

cells A and B. The overall mean heat ratios and confidence intervals using all data yields similar conclusions. The main difference is that the mean X values are generally somewhat smaller for the Pd/D₂O experiments when the first five days are included.

Table I. Mean Heat Ratios and Confidence Intervals for Data After 5 Days of Charging, $\mu - X = \pm 2.58 \sigma/\sqrt{n}$ (99%).

Experiment	\bar{X}_1	\bar{X}_2	\bar{X}_4	\bar{X}_5	
Pd ROD/D ₂ O (I)	1.04 ±0.05	1.04 ±0.05	1.145 ^a ±0.06	1.17a ±0.08	(n=11)
Pd ROD/D ₂ O (II)	1.06 ^a ±0.03	1.10 ^a ±0.03	1.08 ^a ±0.03	1.05 ^a ±0.03	(n=19)
Pd ROD/H ₂ O	0.96 ±0.05	0.995 ±0.05	0.96 ±0.03	1.03 ±0.03	(n=23)
Pd ROD/D ₂ O (III)	1.08 ^a ±0.02	1.07 ^a ±0.02	1.06 ^a ±0.02	1.07 ^a ±0.02	(n=33)

^aExcess enthalpy (99% confidence level).

The t-test can also be used to determine if there is a significant difference from unity for the various mean heat ratios in Table I. Results for the t-test at the 99.5% confidence level are given in Table II. The same Pd/D₂O cells as before show a significant positive difference from unity at the 99.5% confidence level. Both cell A and cell B in the Pd/H₂O experiment fails to show any significant positive differences from unity. The value for t₄ (-4.21), however, is a significant negative difference at the 99.5% confidence level. Although the third cold fusion experiment (Figure 7) did not yield the highest heat ratios, the length of this study (n = 33 days) along with its small mean standard deviation (s = ±0.02) yielded exceptionally large t values.

Table II. The t Test for Excess Enthalpy for Data After 5 Days of Charging, $t = (X - \mu) \sqrt{n/s}$.

Experiment	t ₁	t ₂	t ₄	t ₅	t (99.5%)
Pd ROD/D ₂ O (I) (n=11)	2.30	2.10	6.64 ^a	5.56 ^a	3.58
PdROD/D ₂ O (II) (n=19)	5.27 ^a	8.27 ^a	7.84 ^a	4.93 ^a	3.25
PdROD/H ₂ O (n=23)	-2.23	-0.28	-4.21	2.25	3.15
PdROD/D ₂ O (III) (n=33)	10.94 ^a	10.25 ^a	8.55 ^a	9.01 ^a	3.08

^aExcess enthalpy (99.5% confidence level).

Table III shows that the differences in the calorimetric cell constants measured in various experiments were not a significant factor. The cell constants for cell A (K₁, K₂) tended to be slightly larger than for cell B (K₄, K₅). The cell constants determined in H₂O were very close to those determined in D₂O.

Table III. Calorimetric Cell Constants Determined in Various Experiments Using $K_{\text{cell}} = (E - E_{\text{H}}^{\circ})I \div \Delta T$
Where $E_{\text{H}}^{\circ} = 1.53\text{V}$ for D_2O and $E_{\text{H}}^{\circ} = 1.48\text{V}$ for H_2O .

Experiment	K_1 (W/°C)	K_2 (W/°C)	K_4 (W/°C)	K_5 (W/°C)
Pd ROD/ D_2O (I)	0.141	0.145	0.133	0.132
Pd ROD/ H_2O	0.135	0.138	0.137	0.134
Pd ROD/ D_2O (III)	0.139	0.143	0.133	0.134
Mean	0.138 ± 0.003	0.142 ± 0.004	0.134 ± 0.002	0.133 ± 0.001

Possible calorimetric error sources for our cell design (Figure 1) include room temperature fluctuations, the exposure of the glass thermistor tubes to the room air, the level of H_2O in the gap, the level of the electrolyte in the electrolysis cell, thermal inversions in the gap or thermistor tubes, hydrogen/ oxygen recombination within the cell, and deviations from Newton's Law of cooling. Many of these error sources are small and they all tend to cancel since cell constant determinations were made under nearly the same conditions. Furthermore, these error sources should affect the Pd/ H_2O experiments in the same manner, yet mean heat ratios close to unity were obtained in the H_2O studies. However, the level of the gap H_2O must be carefully controlled in our cell design. Experiments where measured amounts of the gap H_2O up to 10 mL were withdrawn showed nearly a 2% increase in X per mL of H_2O withdrawn. Although the loss due to evaporation was small (and could be entirely eliminated), the effect of the gradual loss of gap H_2O often became experimentally measurable after several days and likely contributed to the periodic trends of Figure 5. The effect of the electrolyte level was much less important for our cell design, hence no correction for this was needed. There was never any evidence for deuterium/oxygen recombination within the cell.

It is interesting to consider the excess enthalpy produced by the various Pd rod/ D_2O cells. For example, using the data from either Figure 3 or 4, an average of at least 18% excess enthalpy is observed for a 9-day period (days 9-18). This corresponds to an average excess power of 0.14 W (0.39 W/cm^3) and a total excess enthalpy of 110 kJ. It can be shown that the complete combustion of the palladium electrode in the form of Pd_2H to yield PdO and H_2O would give an excess enthalpy of only 6.2 kJ. The recombination of D_2 and O_2 to yield 110 kJ of enthalpy would require the formation of 0.37 moles (7.4 g) of D_2O within the cell, yet only normal amounts of make-up D_2O were required during this 9-day period. Hence, it is difficult to explain the 110 kJ of excess enthalpy by chemical reactions. Similar calculations based on data in Table I yields total excess enthalpies of 213 kJ for cell B in Exp. I, 217 kJ for cell A in Exp. II, and 301 kJ for cell A in Exp. III. No excess enthalpies were measured in the H_2O experiment.

Deviations from Newton's Law of cooling have been recently discussed as a possible calorimetric error source [9]. Although this concern applies mainly to Dewar calorimeters where most heat transfer occurs by radiation, it is interesting to see how such errors would affect experimental X values in any type of calorimeter. If heat transfer from the calorimetric cell occurs only by radiation (QR) and conduction (Qc), then

$$Q_R + Q_C = K_R (T_i^4 - T_0^4) + K_C (T_i - T_0) \quad (3)$$

The substitution $T_i = T_0 + \Delta T$ yields

$$Q_R + Q_C = [K_R^0 (1 + \Delta) + K_C] \Delta T \quad (4)$$

where $K_R^0 = 4K_R T_0^3$ and $\Delta = 3 \Delta T / 2 T_0 + (\Delta T)^2 / T_0^2 + (\Delta T)^3 / 4 T_0^3$. Thus the ratio of heat in to heat out becomes

$$X = \frac{[K_R^0 (1 + \Delta) + K_C] \Delta T}{(E - E_H^0) \bullet I} \quad (5)$$

rather than Eq. 1. If there is Joule heating only, then $X = 1.00$, $\Delta T = \Delta T_J$, and $\Delta = \Delta_J$. If there is a fusion contribution to the heat, then $X > 1.00$, $\Delta T = \Delta T_J + \Delta T_F$, and $\Delta = \Delta_F$. Thus

$$X = \left(1 + \frac{\Delta T_F}{\Delta T_J} \right) \bullet \left[\frac{(1 + \Delta_F) + K_C / K_R^0}{(1 + \Delta_J) + K_C / K_R^0} \right] \quad (6)$$

If the heat transfer occurs mainly by conduction as in our calorimeter, then $K_C \gg K_R^0$, hence

$$X = 1 + \frac{\Delta T_F}{\Delta T_J} = X_N \quad (7)$$

which corresponds to Newton's Law of cooling. If the heat transfer occurs mainly by radiation, then $K_C \ll K_R^0$, hence

$$X = \left(1 + \frac{\Delta T_F}{\Delta T_J} \right) \left(\frac{1 + \Delta_F}{1 + \Delta_J} \right) \quad (8)$$

Since $\Delta_F > \Delta_J$, larger X values would be obtained using Eq. 8 rather than Newton's cooling law (Eq. 7). For example, assuming $\Delta T_J = 10$ K, $\Delta T_F = 2$ K, and $T_0 = 300$ K yields $X_N = 1.20$ from Newton's law and $X = 1.21$ from Eq. 8. Hence calibrating a Dewar type cell under conditions of Joule heating only and measuring a fusion heat contribution would yield somewhat higher X values if the more exact expression (Eq. 8) is used rather than Newton's cooling law.

The exposure to neutrons and other harmful radiation can be a matter of concern in cold fusion experiments. Theoretical calculations of dose rates were performed for our cell design (Figure 1) using the Group 16 model for neutron energies. Assuming a source of 10^4 neutrons per second from the palladium, calculated dose rates were 0.28 mrad/hr at the outer surface of the bottle and 0.012 mrad/hr at the top of the bottle (1 rad = 0.01 J/kg). These calculations suggest safe dose rates for people near the cold fusion experiment. Radiation exceeding weekly safe limits could only be reached by holding the cold fusion bottle against the body for an entire 40-hour-work week. These theoretical calculations also showed that the flux of thermal neutrons would not be sufficient to activate gold or copper metal placed at the surface of the bottle. Gold and copper metal samples were always kept near the cold fusion experiments, but radiation measurements gave essentially only background levels. Neutron monitoring for safety concerns carried out with a Ludlum Model 15 counter never gave any response significantly above the background levels.

The upper limit for isotopic changes in metals due to an electrochemical fusion process that produces neutrons can be illustrated by a simple calculation. Assuming an excess power of 1 W/cm^3 resulting from the fusion reaction



yields 1.91×10^{12} neutrons/cm³ s. If all of these neutrons were absorbed by the palladium electrode, then the time required to convert 1% of the palladium to other isotopes is given by

$$t = \frac{(6.79 \times 10^{22} \text{ Pd atoms/cm}^3)(0.01)}{1.91 \times 10^{12} \text{ neutrons/cm}^3 \text{ s}} = 3.55 \times 10^8 \text{ s} \quad (10)$$

which translates into 11 years. Considering the relatively short duration of most electrochemical fusion experiments and the low thermal neutron capture cross-section for the various palladium isotopes, any change in the isotopic composition of the palladium electrode would be difficult to detect. The rate of ³He production would be equally small and difficult to detect, especially considering its possible escape through cracks and fissures.

CONCLUSIONS

Heat ratios greater than unity can be measured in calorimetric experiments for Pd rod/D₂O cells that are significant at the 99.5% confidence level. However, these heat ratios are frustratingly small in many experiments. Nevertheless, the total excess enthalpies are difficult to explain by chemical reactions.

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