Miles, M., *Report on Calorimetric Studies at the NHE Laboratory in Sapporo, Japan.* Infinite Energy, 2000. **5**(30): p. 22.

REPORT ON CALORIMETRIC STUDIES AT THE NHE LABORATORY IN SAPPORO, JAPAN

MELVIN H. MILES

New Hydrogen Energy Laboratory
3-5 Techno-Park 2-Chome Shimonopporo
Atsubetsu-Ku, Sapporo-004, Japan

ABSTRACT

Experiments using China Lake type calorimetric cells produced excess power in three out of three experiments and no excess power in three control studies. A detailed analysis is presented for two experiments using the China Lake cells. Anomalous thermistor signals in Cell A suggest the emission of electromagnetic radiation from the active palladium cathode. Experiments in Fleischmann-Pons type calorimetric cells produced excess power in six out of eight experiments. These studies involved palladium alloy cathodes, co-deposition of palladium and deuterium from the solution, and electromigration using thin palladium wires.

INTRODUCTION

The New Energy Development Organization (NEDO) of Japan made it possible for me to return to cold fusion research and perform calorimetric experiments for a five-month period (October 1997 to March 1998) at the New Hydrogen Energy (NHE) laboratory in Sapporo, Japan. Two types of isoperibolic calorimeters were used in these studies: (1) China Lake type calorimetric cells where the heat transfer is mainly by conduction, and (2) Fleischmann-Pons Dewar type cells where the heat transfer occurs mainly by radiation. The main focus here will be on the China Lake calorimetric cells that are operated at constant current conditions where the complex calorimetric equations can be greatly simplified. The increase in excess power is then readily apparent by the corresponding increase in the cell temperature. A detailed analysis of the results for one experiment using the Fleischmann-Pons calorimetric cells will be presented elsewhere [1]. The NHE laboratory provided excellent facilities for cold fusion research. However, all experimental work at NHE ceased on March 12, 1998 due to the decision to close this laboratory. It was sad to witness the dismantling of this laboratory and the transfer of personnel to other positions as well as the termination of my own experiments.

EXPERIMENTAL

The basic China Lake calorimetric design has been described in previous publications [2-4]. The small electrochemical cell consists of a long, narrow test tube (1.8 cm diameter and 15.0 cm length) that is filled with 18.0 cm^3 of 0.1M LiOD in D_2O (Isotec, Inc. 99.9 at. % D). The electrochemical cell is placed in a calorimetric jacket within a secondary compartment that contains two thermistors positioned on opposite sides of the cell wall and at different heights from the bottom of the cell (1.9 cm and 4.5 cm). Figure 1 shows a schematic of the positioning of the two thermistors relative to the palladium cathode rod (1 mm x 20 mm) and the platinum anode coil.

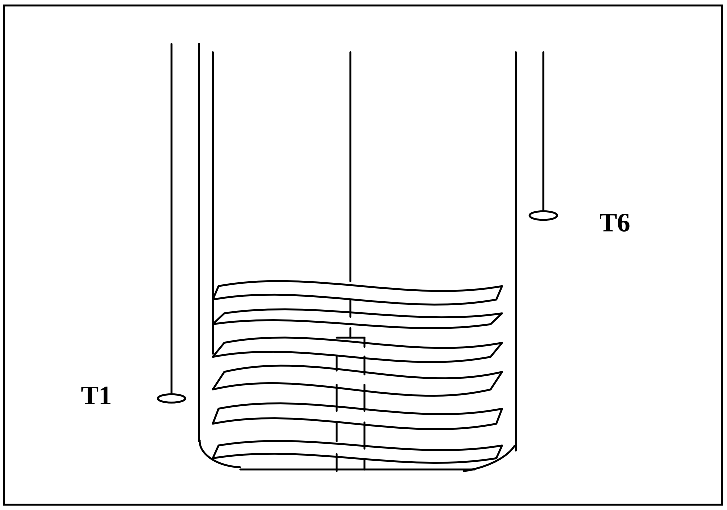

Figure 1. Schematic of positioning of thermistor T_1 and T_6 relative to the palladium cathode rod and platinum anode coil.

It is important to note that the thermistors are placed on the outside wall of the electrochemical cell rather than in the D_2O + LiOD electrolyte. Aluminum foil was wrapped around the outside of the cell including the two thermistors. The secondary compartment was packed with aluminum foil, insulated at the top and then sealed with silicon rubber. Two similar cells were prepared (Cells A and B), placed in a constant temperature water bath, and run in series at constant current. Thermistor channels T_1 and T_6 were used for Cell A and T_3 and T_4 for Cell B. The temperature reading for each thermistor channel was numerically displayed to within 0.01°C and printed as a dot on the chart recording every 18 seconds. There were permanent chart recordings of all six temperatures (four cell temperatures, bath temperature, room temperature) as well as both cell voltages for the entire experiment. In addition, measurements were recorded in a notebook during working hours. The bath temperature was always maintained at 21.51±0.01°C, and the room temperature was generally 1 to 2°C higher than the bath temperature. These experiments were conducted in a thermostatted room at NHE specially designed for calorimetric studies.

SUMMARY OF CALORIMETRIC RESULTS AT NHE

The summary of experiments conducted at NHE using the China Lake calorimetric cells is presented in Table 1.

Table 1. Summary of NHE Experiments Using China Lake Cells

STUDY	CELL A	CELL B
First	Pd Rod Constant Current P_x = 100 mW	Pd Rod Constant Current Control (P_x = 0)
Second	Pt Particles Constant Current Control (P_x = 0)	Pd Particles Constant Current P_x = 90 mW
Third	Pt Particles Pulse Electrolysis Control (P_x = 0)	Pd Particles Pulse Electrolysis P_x= 250 mW

This paper will focus on the first study using palladium rod cathodes (1 mm x 20 mm) from Johnson-Matthey. In previous experiments conducted at China Lake [4], the same palladium cathode used in Cell A (Table 1) produced approximately 200 mW of excess power, but the other palladium cathode used in Cell B produced no measurable excess power effects and serves as a control. Therefore, Table 1 presents excess power (P_x) in three out of three experiments and no excess power in three controls. Details of these experiments are described elsewhere [5].

The experiments conducted at NHE using the Fleischmann-Pons (F-P) Dewar type cells are summarized in Table 2.

Table 2. Summary of NHE Experiments Using Fleischmann - Pons Cells

STUDY	CELL #1	CELL #2	CELL #3
A	Pd-Ce-B Rod P_x =0	Pd-0.5B Rod P_x = 250 mW	Pd-Ce Rod P_x = 200 mW
B	Co-Deposition Cu Rod P_x = 150 mW	Co-Deposition Cu Rod P_x = 400 mW	Co-Deposition Cu Rod P_x = 400 mW
C	Electromigration Thin Pd Wire P_x = 0	Electromigration Thin Pd Wire P_x = 150 mW	Electromigration Thin Pd Wire (?)

Significant excess power was produced using the Pd-B alloy (0.5 wt.% B) prepared by the Naval Research Laboratory. Previous experiments at China Lake using similar Pd-B alloy cathodes produced excess heat in seven out of eight experiments [4]. A much more detailed and accurate analysis of the data from this Pd-B experiment shows an even larger excess power effect as well as positive feedback effects, "heat-after-death", and large excess power effects during cell boil-off studies [1]. It is both surprising and disturbing that the methods developed by NHE for the analysis of the Fleischmann-Pons experiments show no excess heat effects for this same experiment. This indicates serious errors in the NHE alternative method of modifying the Fleischmann-Pons calorimetry [6]. The major problem with the NHE method is that a single calibration involving incorrect procedures was used in determining the heat transfer coefficient for the cell [1]. I developed my own methods of data analysis for the F-P cells while at NHE. As I refined my methods for evaluating the calorimetric measurements, they approached more closely the methods outlined by Fleischmann and Pons in their Icarus Systems handbooks available at NHE [7].

The use of the Pd-Ce alloy in F-P Cell #3 also produced an excess power effect (Table 2), but again the alternative method of analysis by NHE showed no excess power. This same Pd-Ce cathode also produced significant excess power in a previous experiment at China Lake [4]. The Pd-Ce-B alloy in F-P Cell #1 showed no significant excess power effects (Table 2). Three experiments involving the co-deposition of Pd + D onto a copper rod produced large excess power effects. Preliminary results of these experiments have been published [8]. Finally, short experiments involving electromigration produced excess power in one experiment and no excess power in a second experiment (Table 2). Due to an error in assembling Cell #3, the excess power, if any, could not be determined [5]. In summary, excess power was produced in 6 out of 8 valid experiments conducted in F-P cells at the NHE laboratory [5].

The fact that the alternative NHE methods showed no excess heat for F-P cells illustrates the problem in transferring calorimetric methods from one laboratory to another. The second laboratory often fails to follow directions and makes changes that compromise the calorimetry. Similar problems were encountered in the attempt to transfer the China Lake calorimetry to the Naval Research Laboratory (NRL). Various changes made by NRL greatly increased the calorimetric error to ±200 mW, hence excess heat was not observed [4,9]. This failure to reproduce the China Lake experiments at NRL resulted in the termination of Navy funding for cold fusion in 1995. Similarly, the initial failure of NHE to reproduce the Fleischmann-Pons experiments [6], contributed to the termination of the Japan program in 1998.

CALORIMETRIC EQUATIONS

The basic calorimetric equation that describe the heat conduction cells used in this study is

$$P_{EL} + P_x = a + K\Delta T + P_{gas} + P_{calor} \qquad (1)$$

where $P_{EL} = [E(t)\text{-}\gamma E_H]I$ is the power input due to electrolysis and P_x is the excess power [2,4]. The P_{gas} and P_{calor} terms are rather complex and have been described previously [2,4]. In this study, Cells A and B were run under steady state conditions where $(P_{gas}+P_{calor}) << K\Delta T$ and with sufficient insulation above the cells to render the power loss out of the top of cell (term *a*) insignificant. Therefore, our calorimetric equation greatly simplifies to

$$P_{EL} + P_x = K\Delta T \tag{2}$$

The time dependent equation for the cell constant for either Cell A or Cell B at any given cell current (I) was determined experimentally to be expressed by the equation

$$K(t) = K_0 - (I/0.300)\,(1.85\text{x}10^{-4})\,(t\text{-}t_O) \tag{3}$$

where K_O is the cell constant at the time (t_O) expressed in hours when the cell was last refilled with D_2O. The change of the cell constant with time is due to the decreasing volume (V) of the electrolyte in an open cell [2].

The change of the cell temperature with time can now be calculated from Equation 2 where $\Delta T = T_{cell} - T_{bath}$, thus $d\Delta T/dt = dT_{cell}/dt$. Therefore,

$$dT_{cell}/dt = K^{-1}\,(dP_{EL}/dt + dP_x/dt) - K^{-2}(P_{EL} + P_x)dK/dt \tag{4}$$

assuming 100% faradaic efficiency (γ=1.00). If there is no excess power (P_x=0) or if the excess power is constant (dP_x/dt=0) with $P_x << P_{EL}$, Equation 4 becomes

$$dT_{cell}/dt = K^{-1}\,dP_{EL}/dt - K^{-2}\,P_{EL}\,dK/dt \tag{5}$$

Equations 4 and 5 provide the framework for the discussion of palladium rod experiments using the China Lake calorimetry (Table 1).

RESULTS AND DISCUSSION

The constant current electrolysis of the D_2O+0.1M LiOD solution in an open cell slowly increases the conductivity of the solution resulting in a gradual decrease in the cell voltage [2]. Figure 2 presents an example of this behavior for the China Lake Cells A and B using the palladium rod cathodes (first study in Table 1).

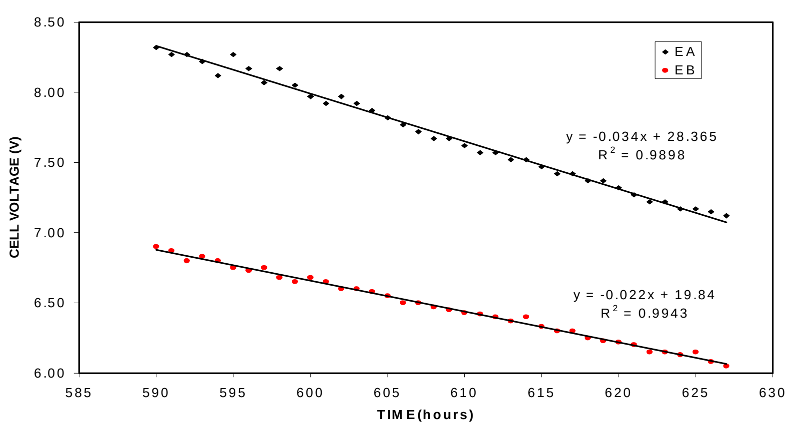

Figure 2. Average cell voltages for Cells A and B showing the linear decrease in voltages.

This nearly linear decrease in the cell voltage with time normally produces a corresponding decrease in the cell temperature with time [2]. The onset of excess power (P_x) can often be recognized simply by the anomalous increase in the cell temperature despite the decreasing applied power. This raises the fundamental question asking how is it possible for the cell temperature to increase when the applied power is decreasing?

Figure 3 presents an example of excess power in Cell A, which covers the same time period as the cell voltage displayed in Figure 2.

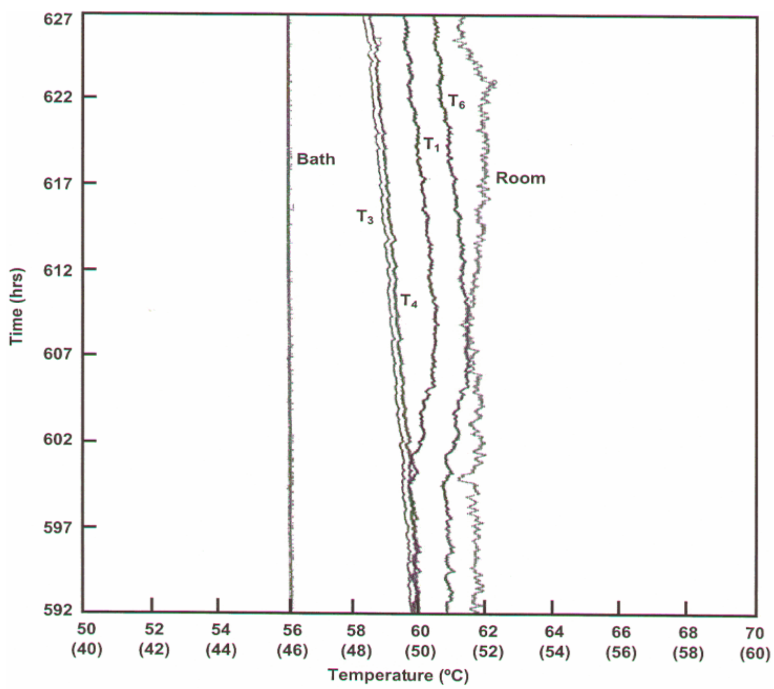

Figure 3. Temperature versus time for Cells A and B. Thermistors T_1 and T_6 show an anomalous temperature increase for Cell A beginning at about 600 hours. Bath and room temperature scales are 20 to 25°C.

Figure 3 is produced directly from the chart recording of the thermistor temperatures, hence time is plotted on the y-axis (ordinate) that feeds out the chart paper at a fixed rate. Two different temperature scales are used in Figure 3 since Cell A was running more than 10°C warmer than Cell B. It is interesting to note that the T_1 thermistor line for Cell A and the T_4 thermistor line for Cell B are merged together until about 600 hours. Both the T_1 and T_6 lines for Cell A then show a substantial increase in temperature that represents a 60 to 70 mW gain in the excess power. The cell voltages shown in Figure 2 show no departures from normal behavior. In fact, the dT/dt slopes calculated from Equation 5 are significantly more negative for Cell A than for Cell B. The increase in the temperature for Cell A shown in Figure 3 is an example of anomalous behavior due to the production of excess power. In contrast, Cell B continuously displays only normal behavior (Figure 3). The room temperature and the bath temperature are also displayed in Figure 3, but their temperature scale ranges from 20°C to 25°C.

Figure 4 displays another anomalous temperature increase in Cell A along with a calibration based on increasing the cell current from 300.22 mA to 320.28 mA for a period of 2.33 hours.

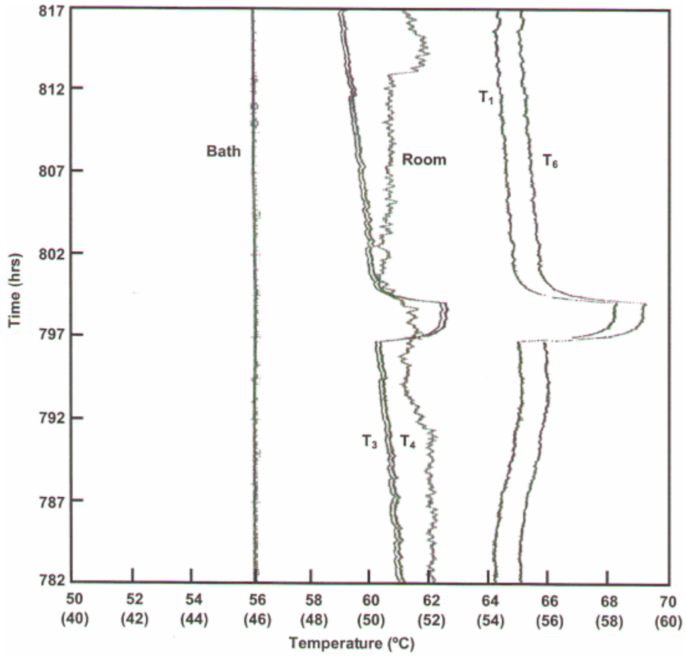

Figure 4. Temperature versus time for Cells A. Thermistors T_1 and T_6 show an anomalous temperature increase for Cell A along with a calibration based on increasing the cell current. Bath and room temperature scales are 20 to 25°C.

Based on notebook data, the power increase of 0.165W produced a temperature increase of 3.29°C in Cell A. Therefore, the anomalous rise of cell temperature of 1.6°C above the expected baseline prior to this calibration indicates an increase in the excess power of about 80 mW. Temperature changes of ±0.1°C are readily detected by these China Lake calorimeters, hence power changes of ±5 mW are measurable. Figure 4 shows that the same baseline is followed before and after the calibration by the thermistors in Cell B. In contrast, Cell A shows different baseline patterns with both positive and negative values of dT/dt. However, both thermistors in Cell A as well as in Cell B always show exactly the same temperature changes. The temperature differences between two thermistors in the same cell is likely due to differences in their positioning relative to the cell wall.

Figure 5 displays the average voltages for Cells A and B during the same time period shown for the cell temperatures in Figure 4.

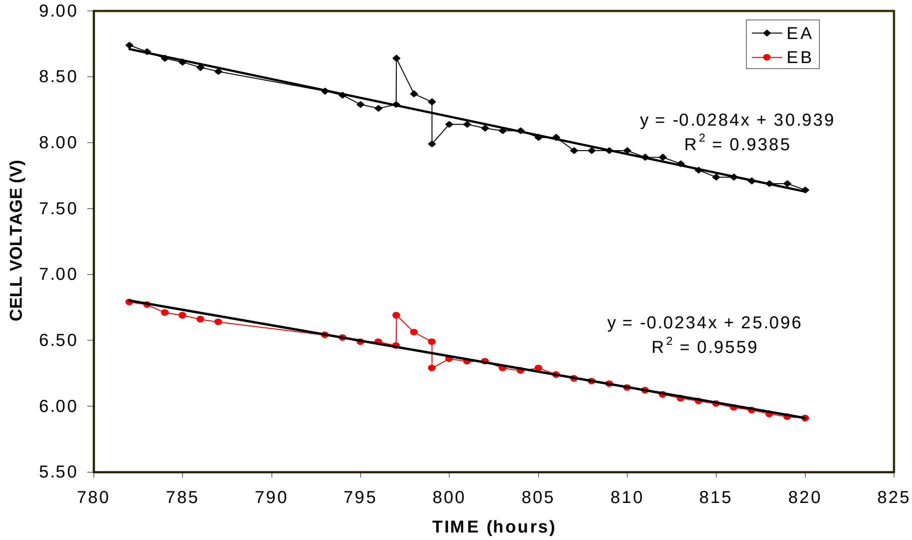

Figure 5. Average cell voltages for Cells A and B showing the linear decrease in voltages along with a calibration based on increasing the cell current.

The effect of increasing the cell current is readily apparent by the change in the cell voltage. However, there is no unusual behavior for the voltage of Cell A that can account for the anomalous increase in the cell temperature. Based on Equation 5, the dT/dt slope should be significantly more negative for Cell A than for Cell B. The episode of positive dT/dt values for Cell A (Fig. 4) requires the assumption of an excess power term as in Equations 2 and 4 along with a significant increase in the excess power ($dP_x/dt>0$).

There were five other examples for excess power in Cell A during this study that gave anomalous increases in the cell temperature despite the decreasing electrolysis power [10]. The calculated values of dT/dt using Equation 5 were always negative for both Cells A and B [10]. Several possible chemical processes including recombination of the electrolysis gases were carefully considered but do not account for the anomalous increases in temperature for Cell A [10].

During the same time period as the excess power production, strange fluctuations were often observed for the temperature readings for Cell A [5]. Stray dots could be seen on the chart recordings for thermistor 1 (T_1), while thermistor 6 (T_6) showed normal readings. An example of these random jumps in the thermistor measurements is shown in Figure 6.

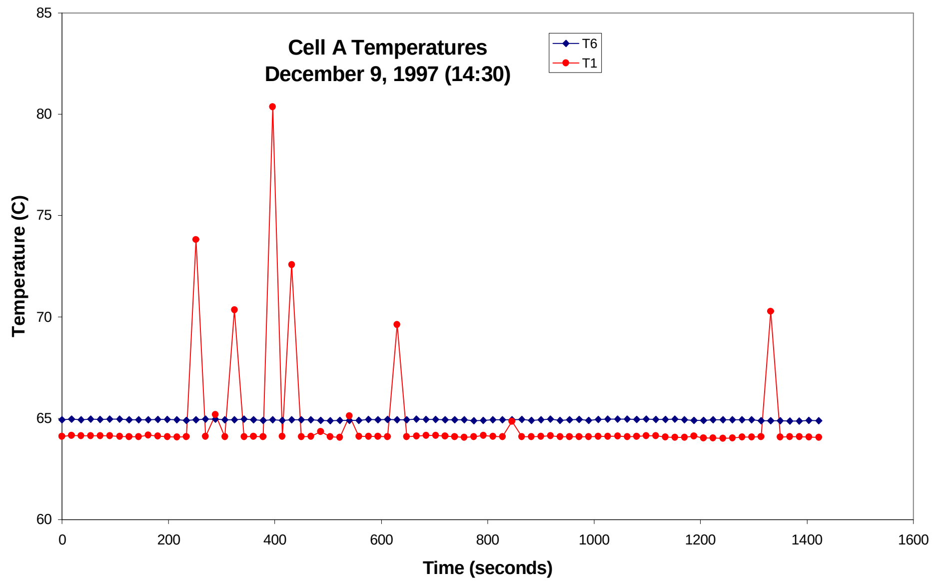

Figure 6. Thermistor readings versus time for Cell A (T_1, T_6).

The thermistor connections were checked and tightened, but these thermistor fluctuations continued over a 23-day period that covered all episodes of excess heat in Cell A. This effect was never observed in any of the following experiments at NHE (Table 1). These thermistors were all identical and consisted of manganese, cobalt, and nickel sintered oxides that were glass coated and covered with a thin Teflon sleeve.

The temperature excursions (Figure 6) quickly returned to normal within the time period of separate measurements (18 seconds). This suggests that the sudden rise and decline in the temperature readings for thermistor T_1 is due to electromagnetic radiation from the palladium cathode that affects the thermistor reading rather than actual increases in the temperature. As shown in Figure 1, thermistor T_1 was located directly in line with the palladium cathode while thermistor T_6 was positioned higher on the outside cell surface. It is readily seen from Figure 1 that any electromagnetic radiation from the cathode could pass directly between the platinum coils to reach thermistor T_1, while thermistor T_6 was completely screened from the cathode by the platinum anode coils. Furthermore, the intensity of such radiation would be much less for thermistor T_6 since its distance from the cathode was approximately four times that of thermistor T_2 (4 cm vs. 1 cm). Later studies at China Lake have shown that gamma radiation sources such as cesium-137 can also produce anomalously high thermistor readings.

ACKNOWLEDGMENT

The author is grateful to the New Energy Development Organization (NEDO) of Japan and the New Hydrogen Energy Laboratory for the opportunity for this research. The author especially thanks Dr. N. Asami for providing for this experience. The author also thanks Masao Sumi and Mari Hosoda for their help and assistance in these experiments along with Linda S. Miles and Kim A. Lincicum for helping with the figures and manuscript preparations.

REFERENCES

1. M. Fleischmann and M. H. Miles (to be submitted to ICCF-8).

2. M.H. Miles, B.F. Bush and D.E. Stilwell, *J. Phys. Chem.* **98** (1994) 1948 .

3. M.H. Miles, K.H. Park and D.E. Stilwell, *J. Electroanal. Chem.* 296 (1990) **241**.

4. M.H. Miles, B.F. Bush and K.B. Johnson, "Anomalous Effects in Deuterated Systems", NAWCWPNS TP 8302, Sept. 1996.

5. M.H. Miles, "Electrochemical Calorimetric Studies of Palladium and Palladium Alloys in Heavy Water", NEDO Final Report, March 31, 1998.

6. T. Saito, M. Sumi, N. Asami and H. Ikegami in "Proceedings of the 5th International Conference on Cold Fusion", Monte-Carlo, Monaco (1995), pp. 105-115.

7. "The Icarus Systems. Isoperibolic Calorimetry: Acquisition, Research, and Utilities System. Icarus 2.00," Technova, Inc., Tokyo, Japan, 1995.

8. S. Szpak, P.A. Mosier-Boss and M.H. Miles, *Fusion Technol.* **36** (1999) 234.

9. D.D. Domingues, P.L. Hagans and M.A. Imam, "A Summary of NRL Research on Anomalous Effects in Deuterated Palladium Electrochemical Systems", NRL/MR/6170-96-803, January 9, 1996.

10. M.H. Miles, *J. Electroanal. Chem.* (Submitted).