

# Anomalous Heat Produced by Electrolysis of Palladium using a Heavy-Water Electrolyte

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## ABSTRACT

Significant heat was generated for about 740 min when a sample of palladium foil was electrolyzed as the cathode in  $D_2O+LiOD$ . A very stable Seebeck calorimeter is described and used to make the measurements. The source of this anomalous energy is unknown. However, the observed energy and production of unexpected elements based on EDX examination are similar to the behaviors claimed by many people who study what is called low energy nuclear reactions.

## I. INTRODUCTION

A very large and anomalous amount of heat equal to 106 kJ was released from a small electrolytic cell containing 30 ml of 0.55 molar LiOD, 0.3 g of palladium as the cathode, and about 5 g of platinum as the anode. This observation is so unusual that the source of this energy needs to be explored. Observations similar to this one have been published by many laboratories and by the author, which suggests the existence of a universal and important phenomenon. This paper provides one more example of this anomalous effect.

Similar anomalous heat production was first reported in 1989 when Profs. Fleischman and Pons [1, 2]<sup>1</sup> electrolyzed a platinum anode, a palladium cathode, using a LiOD +  $D_2O$  electrolyte. This heat was proposed to result from D-D fusion, so called cold fusion. Since then, many similar observations have been reported [3, 4]. In 1993, Bush et al. [5-8] showed that helium was associated with this anomalous heat. Since then, many other studies [9-17] have arrive at the same conclusion. When the amount of energy generated is compared to the amount of  $He^4$  collected, the observed energy per event is consistent with the energy known to result from d-d fusion. However, the neutron radiation and tritium production normally associated with this reaction are not found in sufficient amounts to be consistent with experience using plasma or high energy methods. This failure has resulted in much skepticism about the reality of the claims. [18] Nevertheless, studies now underway in laboratories in eight countries<sup>2</sup> report observing similar behavior.

Because the effect is produced infrequently, heat measurement must be made using a calorimeter that is very stable, accurate, and free of prosaic possibilities to avoid challenges such as published by Miskelly et al. [19] and Shanahan [20-22]. Therefore, the design and characteristics of the Seebeck calorimeter used here are fully described.

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<sup>1</sup> See [www.LENR-CANR.org](http://www.LENR-CANR.org) for full text copies of most citations.

<sup>2</sup> US, Japan, China, Italy, Russia, Ukraine, France, and Israel

## II. EXPERIMENTAL

A Seebeck calorimeter completely surrounds the source of heat by a wall that generates a voltage when a temperature difference occurs across its thickness. The inner surface is heated by the source of heat and the outer surface is held at constant temperature. As a result, the generated voltage is proportional to heat flux through the wall. If designed properly, the voltage responds equally to heat flow no matter where heat passes through the wall or where it originates within the enclosure. However, use of a fan to minimize temperature gradients makes the device more accurate. Although the relationship between heat flux and Seebeck voltage should be linear, experience has shown that use of a quadratic equation improves accuracy. When these precautions are taken, the device is simple in concept, easy to operate, and very accurate over a long period of time.

This type of calorimeter measures power based on generated heat, which is converted to energy by summing measured power over time. Consequently, uncertainty in calculated energy can grow even if the error in measured power is small.

### II. 1 Calorimeter Design

The calorimeter used in this study is constructed by gluing together 16 thermoelectric panels<sup>3</sup>, as shown in Fig. 1. All of the panels are connected electrically in series. Each

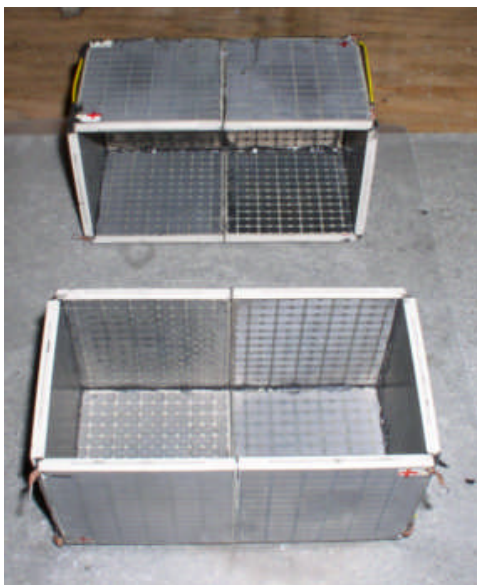


FIGURE 1. Picture of the thermoelectric panels after having been glued together to form two boxes.

box assembly is coated with water-proof paint on the outside and placed in a water-tight plastic structure that allows constant-temperature water ( $\pm 0.01^\circ\text{C}$ ) to flow over its outer

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surface. Wires that make electrical connection to the interior of the boxes are routed through channels that run the full length of the box to insure maximum thermal contact with the cooling water and thermal isolation from changes in room temperature. Two channel designs can be seen in Fig. 2 and 3, where the assembled boxes of two calorimeters are shown. Only the calorimeter shown in Fig. 2 was used when excess energy was measured. A thermistor is located in front of the fan to measure interior temperature. All voltage measurements are made at the thermal boundary.

A Pyrex glass cell, shown in Fig. 4, contains the anode and reference electrode, both of which are platinum mesh. A thermistor is located in the electrolyte to allow electrolyte temperature to be determined and a resistor is present to generate a known amount of heating power for calibration, both of which are glass covered. Using a Teflon clamp, the cathode is connected to a platinum wire that passes out of the cell through a gas-tight seal, which allows the cathode to be easily removed. A recombining catalyst consisting of platinum applied to carbon cloth is located in the gas space. In addition to the cathode, the cell contains only platinum, Teflon, Pyrex glass and chemicals used in the electrolyte. Gas is communicated to an oil reservoir located outside of the calorimeter through a small tube. Displaced oil is weighted on a balance and recorded. Lack of unusual weight change is used to insure that the recombiner is working and that the cell is sealed. The D/Pd ratio of the cathode also can be determined using the orphaned oxygen method.

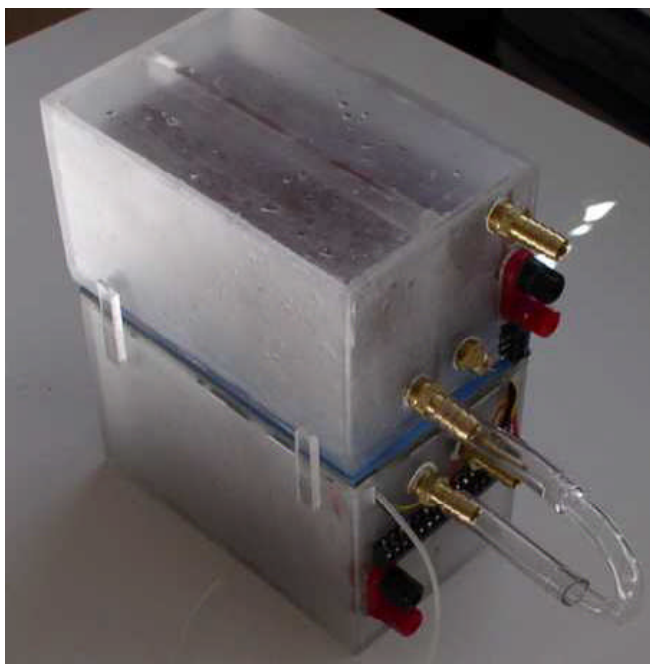


FIGURE 2. Picture of assembled Calorimeter #1 after the thermoelectric boxes are surrounded by a water-tight structure. The wires pass through plastic channels on their way out of the calorimeter.

The electrolyte used in Calorimeter #1 is 30 ml double distilled  $D_2O$  (99.6% D) + 0.148 g LiD, which was prepurified by electrolyzing a dummy cathode before making the studies described here.

## II. 2. Electrical Connections and Data Acquisition

The electrical connections are shown in Fig. 5. Measurement of voltage applied to the cell and to the fan are made at the thermal boundary. Current is measured as a voltage across resistors in series with the cell and fan. Current and voltage are calibrated using a precision digital voltmeter. A switch is used to change from Joule heating to electrolysis, which allows both measurements to use the same data acquisition (DA) channel and series resistor, thereby avoiding a potential error. When the open circuit voltage is measured (OCV), voltage produced by the power supply is turned off for 0.004 sec, the cathode is isolated from the power supply using a relay, and the anode DA channel is switched to the reference electrode. Data used to calculate excess power are taken every 10 minutes after averaging 20,000 measurements of cell temperature, Seebeck box interior temperature and room temperature. In addition, voltage and current applied to the cell and fan, and Seebeck voltage are measured and recorded about every 10 minutes.

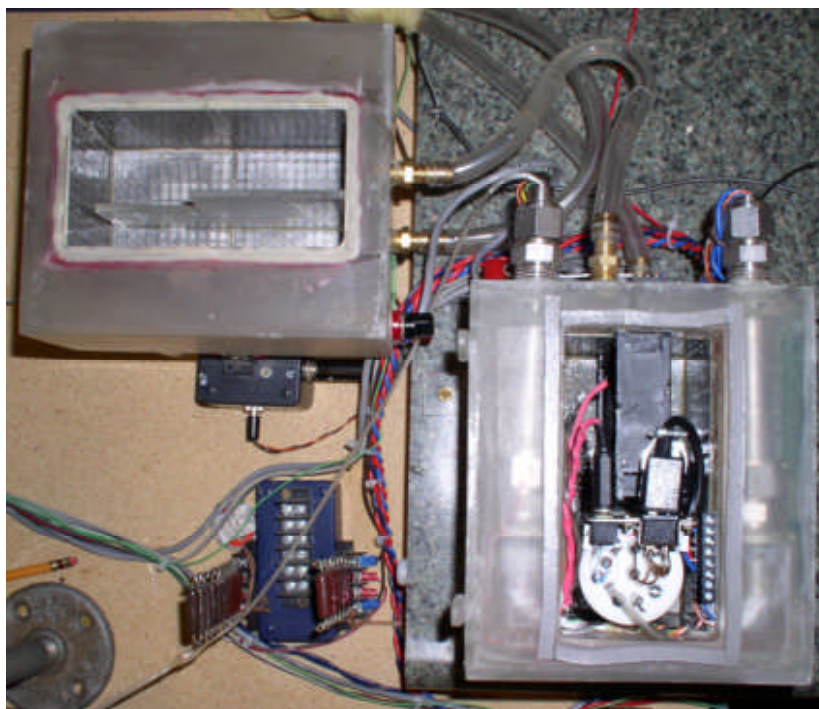


FIGURE 3. Interior of Calorimeter #2 showing location of cell and fan. The wires pass through stainless steel tubes on their way from the interior to the exterior of the thermal envelope.

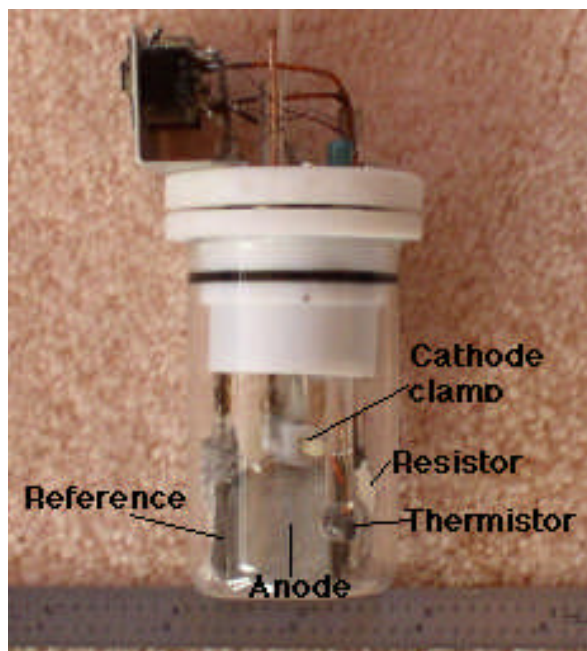


FIGURE 4. Picture of the cell. The recombining is located within the white Teflon structure at the top where it is protected from reaction with spray from the electrolyte.

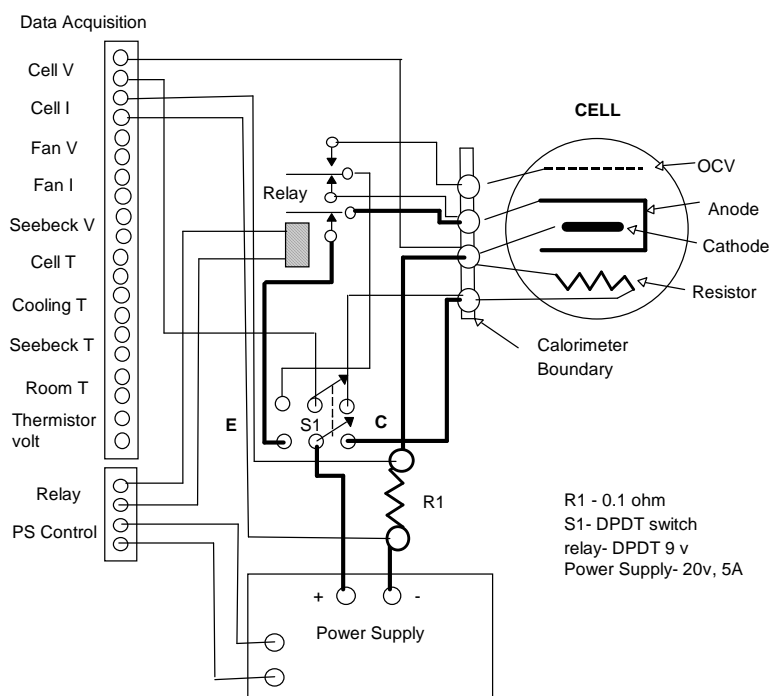


FIGURE 5. Circuit diagram of electrical connection to the cell and power supply. Only those circuits involved in applied power and OCV measurements are shown.

The amount of heating power ( $W$ ) being generated within the calorimeter, expressed in watts, is equal to  $W = A + B \cdot V + C \cdot V^2$ , where  $V$  is the Seebeck voltage and  $A$ ,  $B$ , and  $C$  are constants obtained from a calibration. Excess power is equal to  $W - (V_c \cdot A_c + V_f \cdot A_f)$ , where  $V_c$  and  $V_f$  are the voltages being applied to the cell and fan, respectively, and  $A_c$  and  $A_f$  are the currents passing through these items. The amount of heating power ( $W$ ) being generated within the calorimeter, expressed in watts, is equal to  $W = A + B \cdot V + C \cdot V^2$ , where  $V$  is the Seebeck voltage and  $A$ ,  $B$ , and  $C$  are constants obtained from a calibration. Excess power is equal to  $W - (V_c \cdot A_c + V_f \cdot A_f)$ , where  $V_c$  and  $V_f$  are the voltages being applied to the cell and fan, respectively, and  $A_c$  and  $A_f$  are the currents passing through these items.

### II. 3 Calorimeter Calibration and Test

The calorimeter is calibrated by applying electrical power to the internal resistor. This is done automatically in steps, first going up in power and then going down. In addition, electrolytic current can be applied to a dead cathode to determine whether the location of heat production has any effect on the calibration constants, because when Joule heating is

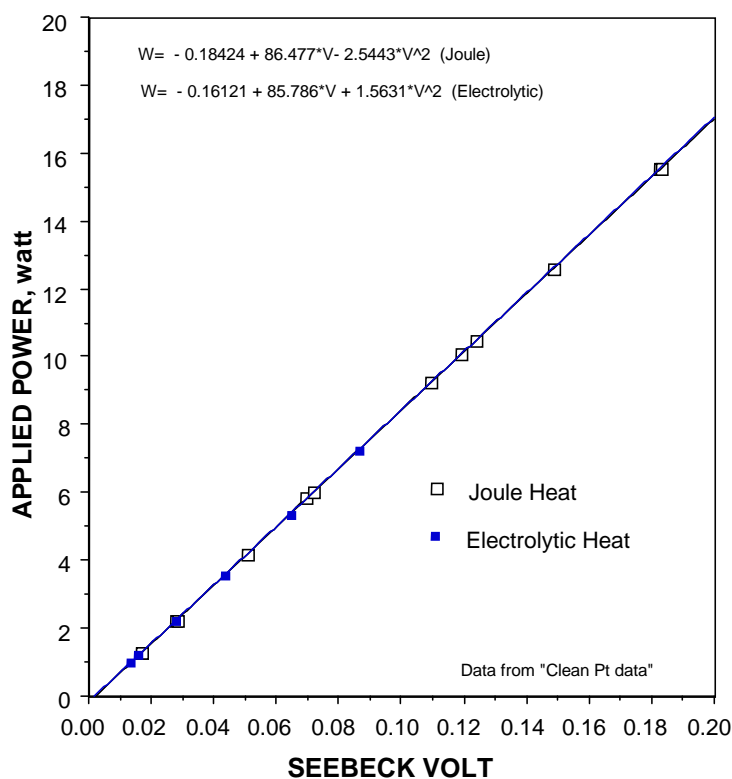


FIGURE 6. Typical calibration results by applying Joule or electrolytic power.

used, heat is produced only at the resistor without bubble formation. On the other hand, applied power that produces electrolytic action generates heat at the electrodes and at the recombiner - two entirely different locations. The difference between these two methods is  $2 \pm 10$  mW within the applied power range of this study. Therefore, the calorimeter design is insensitive to where heat is being generated within the cell and gives the same

result for power being generated by Joule or electrolytic heating. Scatter of points from the least squares line gives a value for the random variation of individual measurements. Typically, this value is  $\pm 15$  mW for Calorimeter #1. In addition to this uncertainty, drift in the base line over 24 hours of  $\pm 25$  mW can occur, caused mainly by changes in room temperature. The long term drift of about  $\pm 50$  mW over several months is reduced by repeated calibrations. Calibration equations are listed in Table 1. All but the last two values use a reference temperature of 20° C for which an average is given.

TABLE I  
Coefficients in  
Calibration Equation  
for Calorimeter #1

DATE	A	B	C	error, mW
2/14/05	-0.020	83.02	0.12833	7
2/17/05	0.006	82.85	2.012	17
2/21/05	0.001	83.04	0.366	17
2/26/05	0.001	83.35	-1.220	11
3/10/05	-0.051	83.18	0.325	24
3/15/05	-0.022	83.86	-1.927	10
3/21/05	0.002	83.30	-0.764	16
3/24/05	-0.027	83.76	-1.737	28
3/29/05	0.000	83.40	-1.556	19
4/29/05	-0.013	83.82	-1.998	16
5/7/05	-0.052	83.40	-1.516	24
5/19/05	-0.076	83.25	-1.258	10
5/22/05	-0.116	83.33	-0.841	5
5/26/05	-0.093	82.94	1.053	17
5/29/05	-0.065	83.04	0.587	16
6/1/05	-0.078	83.46	-2.371	13
7/2/05	-0.203	83.49	-1.940	15
7/26/05	-0.184	83.59	-1.099	5
8/16/05	-0.101	82.38	3.609	34
8/23/05	-0.169	83.65	-2.819	8
9/16/05	-0.086	81.21	1.564	10
12/23/05	-0.188	86.54*	-2.598	14
1/29/06	-0.184	86.48*	-2.544	13
average=	-0.064	83.21	-0.5429	15

\* Reference at 5° C.

The average internal temperature, measured using a thermistor located in front of the fan, can also be used to calculate power production, with the calorimeter acting as a second

wall isoperibolic type calorimeter. Because this average temperature is measured using a different DA channel from that used to measure the Seebeck voltage, this method provides a check on generated power based on Seebeck voltage. A typical calibration using this method is shown in Fig. 7.

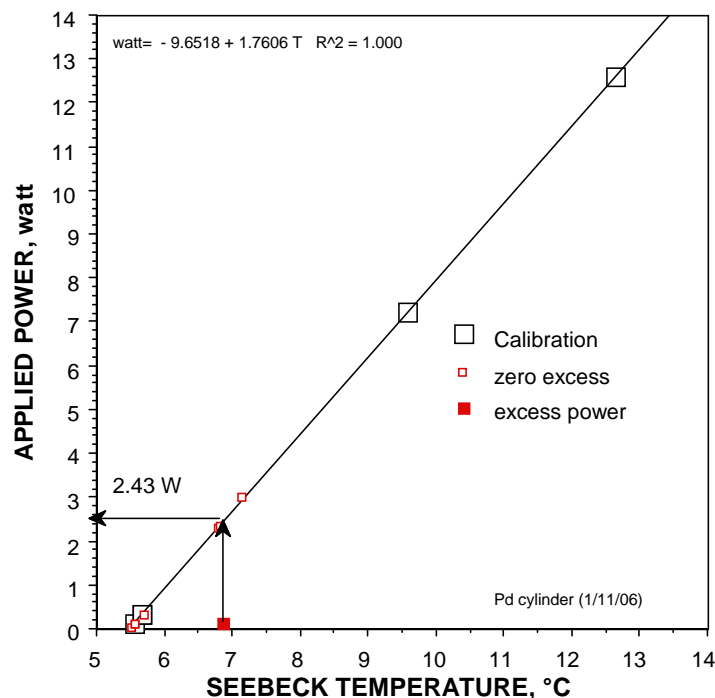


FIGURE 7. Calibration of power production based on internal temperature.

Unfortunately, temperature within the electrolyte can not be used to obtain accurate values for power production, because variable temperature gradients are present. Nevertheless, this temperature is sensitive to changes in power production within the cell, thereby providing another indication of when power production changes.

Once calibrated, the calorimeter can be tested by measuring a known chemical reaction. When current is applied to a Pd cathode,  $D_2O$  is decomposed into  $D_2$  at the cathode and  $O_2$  at the anode, with the  $D_2$  and Pd reacting to produce  $\beta$ -PdD<sub>1-y</sub>. The reaction is endothermic because more energy is used to decompose  $D_2O$  than is produced when PdD<sub>1-y</sub> forms. As the cathode takes up deuterium, the Pd first converts to alpha-PdD followed by a layer of  $\beta$ -PdD<sub>1-y</sub> of increasing thickness with a range of composition. Eventually, after most of the sample is converted to the final composition, reaction stops and conditions return to zero power (energy). At this time, the composition is in steady-state with a stable gradient and a constant rate of diffusion between the surface and random cracks through which dissolved deuterium is lost as  $D_2$  gas. Upon reaching steady-state, reaction energy can be calculated from the reaction  $(x/2)D_2O + Pd = PdD_x + (x/4)O_2$ , where x is equal to the average D/Pd ratio.



The energy used during the loading reaction can be calculated by integrating the data shown in Fig. 8, between zero power and the curve drawn through the data points over the duration of the loading process. This curve gives a value of -106 kJ/mol Pd. Termination of the reaction is assumed to occur at an average D/Pd of 0.8, the measured value for this sample, and at 460 min. The reaction equation becomes:

$0.4\text{D}_2\text{O} + \text{Pd} = \text{PdD}_{0.8} + 0.2\text{O}_2$ , where  $\Delta H_f(\text{D}_2\text{O}) = 294.6$  kJ/mol. Pd and  $\text{O}_2$  are assumed to be at standard-state, at which their enthalpy values are zero. The enthalpy of formation of  $\text{PdD}_{0.8}$  is  $\Delta H_f(\text{PdD}_{0.8}) = -106 + 0.4 * 294.6 = 11.8$  kJ/mol. This value can be compared to that calculated from a published equation[23]:

$$\Delta H_f(\text{PdD}_x) = 44.99 - 41.89 * x = 11.5 \text{ kJ/mol for } x = 0.8.$$

Because the power measurements have some random variation and because the sample does not have a uniform composition, the potential error in this measurement is about  $\pm 2$  kJ/mole. Nevertheless, agreement between these two measurements is well within the error of both results, thereby demonstrating the potential accuracy of this calorimeter. The calorimeter is able to easily detect this endothermic reaction when cathodes as small as 0.1 g are used. The weight of palladium used in this measurement is 2 g.

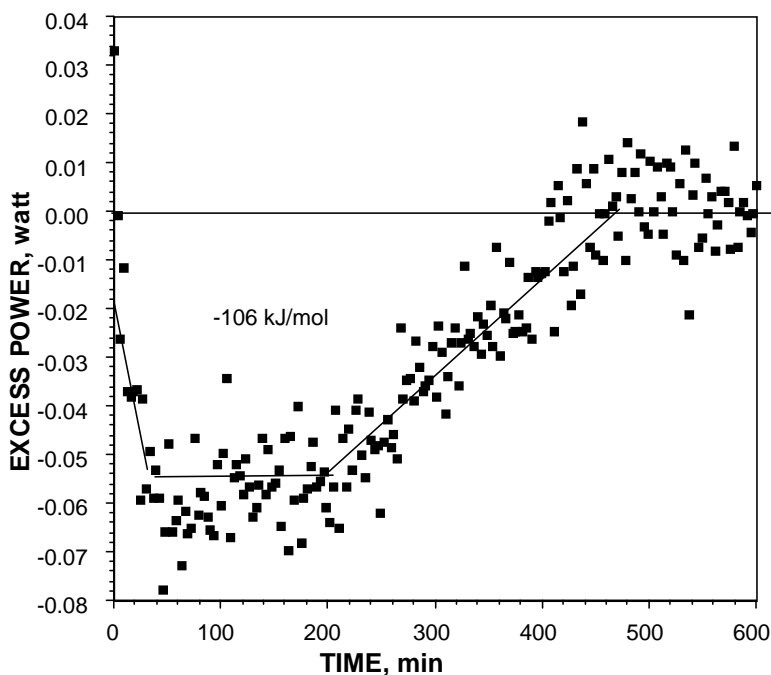


FIGURE 8. Endothermic reaction that occurs as beta-PdD is formed by electrolysis.

#### II. 4. Construction of the Palladium Cathode

A sheet of commercially supplied Pd foil (0.076 mm thick, 0.3 g) was rolled into a cylinder having a diameter of 3.8 mm and a length of 20 mm with about 1.0 mm overlap. No pretreatment was used other than to clean the assembly with acetone before it was

placed in the calorimeter. A picture of the assembly is shown in Fig. 9. Glass rings are used to prevent the foil from unrolling as it is loaded. Electrolysis takes place on the outer surface, with some deuterium diffusing through the sheet to exit as gas at the interior surface. A value for the final D/Pd ratio was not obtained.



FIGURE 9. Picture of cylinder made using palladium foil.

## II. 5. Measurement of Energy Production

Electrolysis was started at 0.049 A and continued for 129 min without excess power being produced. At the end of this time, a small air leak was discovered in the cell at the O-ring seal. Because the cell was initially filled with air and because any generated  $D_2$  and  $O_2$  was removed by reforming  $D_2O$ , this additional air would have caused little change in gas composition within the cell. Current was turned off, the cell was removed from the calorimeter, and repair was quickly accomplished by adding a little silicon vacuum grease to the O-ring. Many similar procedures in the past never resulted in excess energy production. When current at 0.049 A (0.140 W) was again applied, the sample was found to produce 2.6 W of excess power, which slowly decreased as shown in Fig. 10. No change was seen in the amount of applied power or in cell resistance, which indicates that no change in cell characteristics had resulted from the repair. Nevertheless, cell temperature increased from  $5.80^\circ$  to  $7.10^\circ$  C, which is consistent with extra power being generated in the cell. After 706 min, applied current was increased to 0.098 A (0.334 W). Shortly after 33 min, excess power suddenly stopped and the calorimeter returned to a null condition at a rate consistent with the time constant of the calorimeter, as shown in Fig. 11. Interrupting applied power once again and application of current between 0.02 A and 1 A did not result in excess power production being restored.

The OCV between the anode and cathode continued to increase from 1.02 V to 1.10 V even after excess power production stopped, showing that no sudden change had occurred in the activity of deuterium at the cathode surface, which might explain the abrupt termination of energy production. However, the slow increase indicates that the activity, hence the D/Pd ratio at the surface, was gradually increasing.

In order to verify that this extra power was not caused by an error in Seebeck voltage, the Seebeck calorimeter was used as a second wall isoperibolic calorimeter, as previously described. Figure 7 shows the relationship between interior temperature of the Seebeck and power being generated within the cell. The line is based on values taken when the Pd cylinder was not making excess energy and on a previous sample that was also dead. A value is plotted as an example and compared to the calibration line to give 2.29 W (2.43

W- 0.14 W). Similar calculations using all points are compared in Figs. 10 and 11. The excess/applied power ratio is about 16 for the data in Fig. 10.

A second sample was made using the same material, the same construction, and subjected to the same procedure in the same calorimeter as the first sample. No excess power was produced when current from 0.05 A to 2.5 A was applied. A third sample was made the same way and electrolyzed in a  $D_2O+D_2SO_4$  electrolyte using Calorimeter #2. No excess power was produced when current was set at 0.018 A, 0.048 A, 0.100A and 0.509 A.

## II. 6. Observation of Surface and Surface Analysis

The surface was examined using a scanning electron microscope. Figure 12 shows the surface of the foil before being rolled into a cylinder and electrolyzed. The surface of Cylinder #1 is shown in Fig. 13 after electrolysis. Electron dispersive X-ray (EDX) examination of the surfaces revealed the absence of significant Cu and Fe before the study but the presence of significant Cu, Fe and Si after the study, as listed in Table 2.

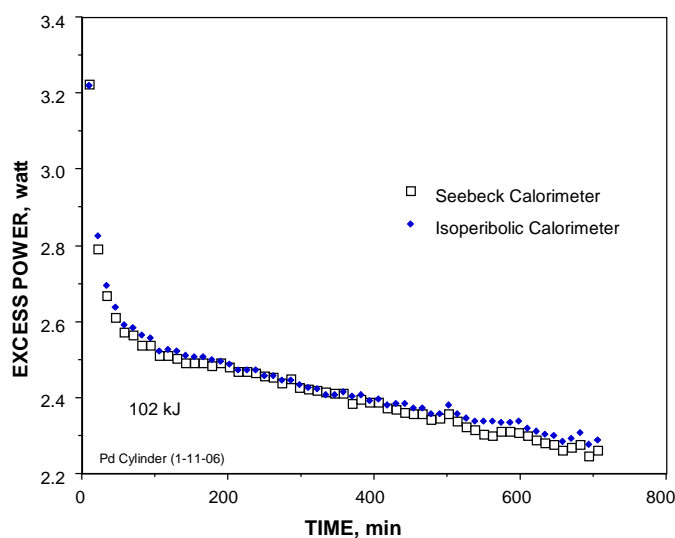


FIGURE 10. Excess power production while 0.05 A is applied, starting on 1/11/06 at 10:30 PM MDT. The values before 60 min are not correct because the calorimeter was gradually approaching steady-state after generated power had increased.

The values were taken at random locations on the samples and with a sufficient number of points to reduce the random variation of each element to less than  $\pm 0.01$  Atomic % for each data set. According to the review by Miley and Shrestha [24], Cu and Fe are frequently detected in anomalous amounts in other studies made under similar conditions.

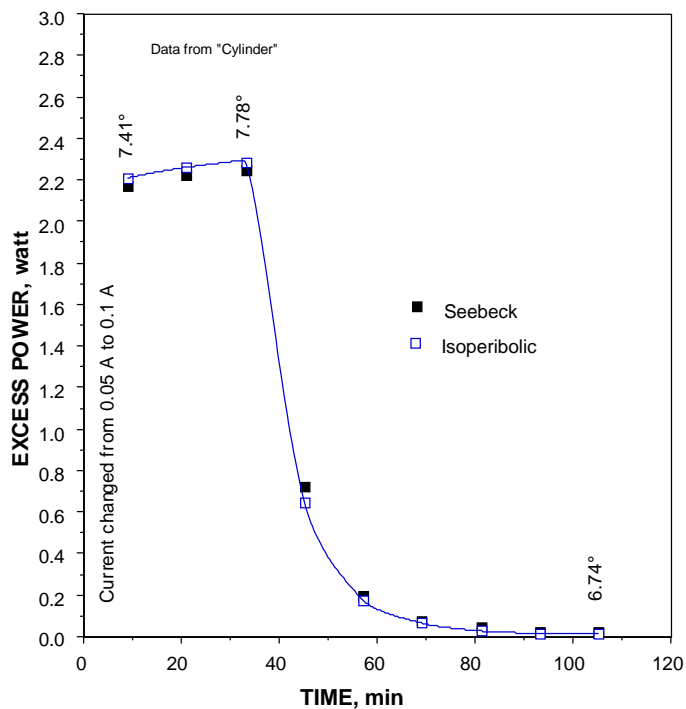


FIGURE 11. Excess power production while 0.1 A is applied. Temperature within the cell is shown. The drop in apparent excess power is consistent with the time constant of the calorimeter after power production had suddenly stopped.

TABLE 2

Atomic Percent of Indicated Elements on the Surface  
Foil before  
electrolysis

	Pt	Cu	Fe	Si
	0	0	0.01	0.06
	0.01	0.02	0.01	0.2
	0	0	0.01	0.14
	0.17	0.01	0.02	0.01
	0.06	0.07	0.1	0.35
	0.06	0.05	0.12	0.47
	0	0	0.01	0.11
	0.01	0.01	0.01	0.27
	0	0.02	0.07	0.79
	0.02	0	0.01	0.11
<b>AVERAGE</b>	<b>0.03</b>	<b>0.02</b>	<b>0.04</b>	<b>0.25</b>
<b>STDEV</b>	<b>0.05</b>	<b>0.02</b>	<b>0.04</b>	<b>0.24</b>

	Cylinder #1 After electrolysis			
	0.44	0.34	0.9	0.79
	0.04	0.15	0.02	0.02
	0.07	0.15	0.03	0.08
	0.03	0.21	0.15	0.92
	0.41	0.21	0.24	
	0.24	0.21	0.1	0.51
	0.03	0.02	0.02	0.48
	0.03	0.01	0.01	0.12
	0.16	0.12	0.09	0.71
	0.17	0.17	0.11	0.62
	0.02	0.02	0.01	0.16
<b>AVERAGE</b>	<b>0.12</b>	<b>0.13</b>	<b>0.08</b>	<b>0.40</b>
<b>STDEV</b>	<b>0.15</b>	<b>0.10</b>	<b>0.26</b>	<b>0.33</b>

### III. DISCUSSION

A calorimeter having a demonstrated stability of less than  $\pm 25$  mW recorded an anomalous generation of power of about 2.5 W, a value 100 times the expected uncertainty. Confidence in this measurement is especially high for the following reasons. Good agreement was found between two independent methods for calculating energy production, the cell temperature behavior was consistent with extra power being generated within the cell, no change in cell characteristics occurred, and the calibrations before and after the study show excellent agreement. In other words, all behavior was consistent and as expected based on two years of experience using this calorimeter. Consequently, the observed energy of 106 kJ does not appear to be caused by error in the power measurement.

What is the source of this energy? The measured amount is too large to be produced by normal chemical reactions, given that less than 30 ml of  $D_2O$  and 0.3 g of palladium are present in the cell. All other materials within the cell are chemically inert. A total of only 1.08 kJ was applied after the cylinder was put in the cell, but before energy generation started. If energy storage during electrolysis is the source, it is an important observation worthy of further study. However, previous evaluation of various chemical process concluded that they could not be the source of similar reported energy[25]. The amount of gas in the cell is about 30 ml, which if it were a stoichiometric mixture of  $D_2$  and  $O_2$ , would generate about 0.4 kJ upon reacting to form  $D_2O$ . However, this reaction was ongoing because of the internal catalyst, and would not have suddenly changed without showing a change in oil weight, which was not observed. Release of deuterium from palladium is endothermic, thereby eliminating this possibility. Because the cell is very small and contains very few chemical components, a chemical source does not appear plausible.

The original foil was dead soft and had insignificant amounts of Pt, Fe, and Cu on its surface, but contained a small amount of Si. The surface was not smooth but had a matted appearance, with a subtle regular structure that runs vertically through the picture in Fig. 12. Cylinder #1 gained additional amounts of each of these elements during the study, which were very nonuniform in their distribution. The additional platinum is expected to have transferred from the anode and additional silicon could have come from the Pyrex glass. The source of Fe and Cu is unknown although these elements have been reported previously after similar treatments of palladium[24]. Loading caused parallel ridges to form that appear to be a magnification of the subtle structure seen in the original foil. No cracks or other gross changes were visible at a resolution of 1  $\mu\text{m}$ , thereby eliminating stress relief as an energy source.

If this observation were an isolated event, it would be interesting but not particularly important. In fact, many similar observations have been made in laboratories all over the world as have been reviewed by Storms[3, 4, 26-29]. In addition, the author has published claims for similar heat production in the past using a variety of calorimeter types and cathode material. [30-32] Therefore, this claim is not an isolated event but is part of a general phenomenon that is not yet understood but is clearly too well documented to ignore.

Everyone who has studied this effect has found it to be difficult to reproduce, although certain batches of palladium and certain methods for applying a surface deposit are more reproducible than others. The effect obviously can not occur in "normal" materials but apparently requires a very unusual and difficult to create condition to be created within in the surface region of the cathode. This unusual condition is only loosely related to the gross conditions that people can control and measure. For example, the effect is known to be more frequently observed if the D/Pd ratio is above 0.85. However, the reported D/Pd ratio is always the average value, not the actual D/Pd ratio that exists in the surface region. The D/Pd ratio in the surface region can be as high as 2.0, a value that is only loosely related to the average. Therefore, conditions in the active region can be much different from what is reported or required. This is only one of many examples of the disconnect between gross conditions and the conditions that actually exist in the region where the anomalous effect is proposed to originate. The difficulty in replicating the effect will remain until someone actually studies the region in which energy is actually produced and succeeds in controlling the required conditions in that region.

#### **IV. CONCLUSION**

Significant anomalous energy was produced when 0.3 gm of palladium foil was electrolyzed in only 30 ml of 0.55 molar LiOD. In addition, anomalous amounts of Cu and Fe were observed on the surface. This amount of energy, especially when measured using a very accurate calorimeter, requires attention and an explanation. Although all aspects of this observation could not be explored in this study, many similar published observations provide some guidance [27, 33] about the process that might be the source of this energy.

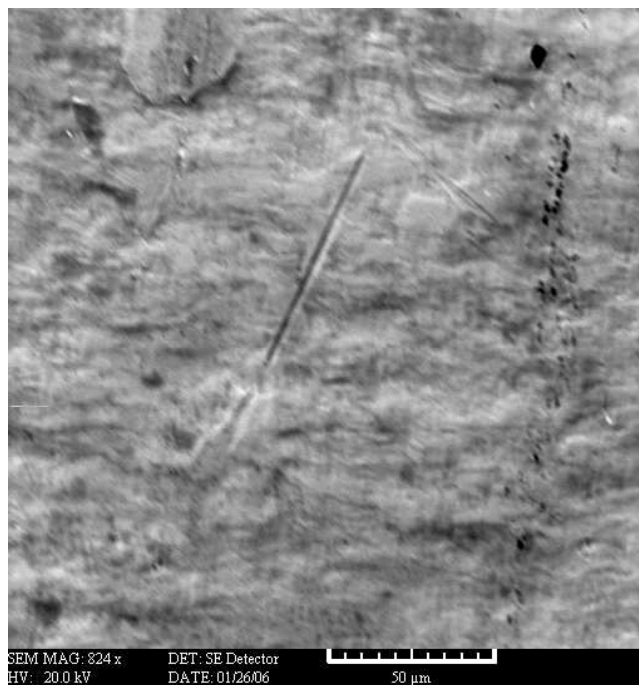


FIGURE 12. SEM view of foil before study.

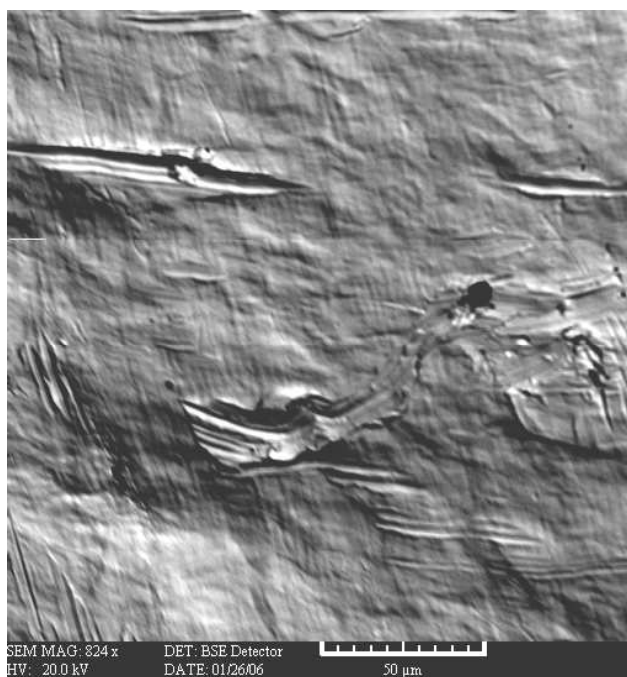


FIGURE 13. SEM view of cylinder #1 after study. The scars were produced when the glass rings were added and removed.

If the heat observed here is related to that reported by previous studies during which various nuclear products have been produced, this is the first example of such anomalous

energy resulting when a foil of Pd was electrolyzed from only one side using a very small current density. This configuration was thought not able to produce such anomalous energy because the required D/Pd ratio could not be achieved. Consequently, this is an unusual observation even in the context of the LENR claims. In this context, it is the largest reported ratio of energy out/energy in for electrolytic energy production.

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