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ELECTROCHEMICAL COLD FUSION TRIALS AT IPP GARCHING

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Abstract: Following the report of Fleischmann and Pons [1], we (The Bavarian Bubble Bottle Team) have attempted to reproduce their claims of cold nuclear fusion, and failed. We note that our measurements would not be able to detect neutrons at the level of Jones *et al.* [2]. Three electrolytic cell experiments were conducted using palladium cathodes and Platinum anodes, in a 0.1 Molar solution of LiD in heavy water, without any signs of neutrons, tritium or gammas above backgrounds, and within $\pm$ 0.3 Watt accuracy calorimetry, no excess heating. Excess heating at the levels of F&P would have been easily detected, if present. Intrinsic tritium, differing from each D_2O bottle tested, was however observed. The longest duration experiment ran for 21 days, and was an attempt to duplicate the large "melting incident" of F&P. This was terminated on April 28, 1989, by throwing the vacuum-cast 22 gram, deuterium-loaded palladium cathode directly into liquid nitrogen, immediately next to a bare BF_3 counter (backed by 25 cm of moderator), in order to attempt one of the Italian ENEA neutron production variants. No neutrons above backgrounds were seen, while counting for one hour, and also none while the piece warmed to room temperature over the next hour. Post mortem analysis of the darkened, hardened Pd piece showed large crystal grains (up to 2mm$\times$2mm), and continuing evolution of gas bubbles at the grain boundaries even four days after the experiment was ended. Eight-weeks after loading, the catalytically active palladium piece continued to create heavy water (with exposure to oxygen in the air).

Contents

1 Introduction

During the long holiday weekend of Easter, 1989, we first began to hear radio and TV reports of "fusion in a test tube", originating from the University of Utah. Most of us rejected the brief news reports as quackery and fluff. A televised video of a laboratory scene with a bubbling cell, without any heavy shielding, caused us immediately to ask "If this could be real, why aren't the experimenters dead from exposure to radiation?", which would accompany nuclear fusion in quantities to account for the claimed (a few Watts) energy releases.

Then, with rumors beginning to fly across computer networks and faxes, we received the reports of "vaporized electrodes", and that one of the principals (MF) was a respected Fellow of the Royal Society, and the other (SP) was head of the U of Utah chemistry department. Still with considerable doubt and a lot of skepticism, mixed with some wonderment if it just might really be possible... a few of us begin to look around in the IPP for the necessary chemicals, detectors, and a safe place to operate. We conducted a basic library search on properties of palladium and metal hydrides, and began to interact with metal hydride experts at the Technical University...who indicated that Fleischmann should be taken at his word. Furthermore, we were certain that a pair of chemists would have done careful calorimetry, as this is a basic tool of the physical chemist's profession. We felt that calibration of the nuclear measurements might be another question, but this didn't concern us greatly at the time. *Any* chemical process driving a reaction creating nuclear products is in of itself, interesting.

Our only initial "engineering" information was the March 23, 1989 "Financial Times" article [3], which had a crude diagram, and a March 25, 1989 article in "Die Welt" newspaper showing a picture of Fleischmann holding one of his cells in his hand. This information was dispersed at the highly publicized initial press conference announcement. Conspicuously missing from these accounts were the electrolytic "salt" and concentrations used. We gathered more articles, and discussed possible lines of action among our self-assembling team on Tuesday March 28 (Monday was an Easter holiday). We turned on our first experimental cell on Thursday afternoon March 30, 1989. Minor delays for machining appropiate glass cell fittings, and setting up the necessary radiation detectors, chart recorders, etc., in a fume hood of the electrochemical plating laboratory of the institute, progressed in parallel with information gathering.

It was obvious from the beginning that the F&P experiment was either going to be one of the biggest flops of the year, or else one of the most important discoveries in this century. Since our interest in achieving fusion energy in a controlled fashion for the production of energy is not necessarily tied to any one technique, (ie, the tokamak), we felt it important to verify these astonishing claims, if possible. If something unexpected might be occurring in a lattice of palladium fully loaded with deuterium, that just maybe no one else had noticed before, we wanted to *know*.

Discussions with Los Alamos, Princeton, and Harwell Laboratories, told us that other people were also taking the claims seriously. As it has been stated in the press, "the race

was on". Computer bulletin boards were critical for early accounts of seminars, or phone discussions from the Utah principals involved. Through these means, we first found out to use lithium as the electrolytic solute, and that it might be important to minimize exchange of H_2O from the air with D_2O in the cell. Over the weekend of April Fool's, we continued to sort and assimilate the wild speculation floating around on the computer networks.

2 Properties of the Pd/D/Li System

For the purposes of this report, we describe here some of the relevant information on the extremely complicated Pd/D system, which is complicated even further by the addition of lithium, which is also soluble in palladium. We make no attempts at completeness, for entire books are written on differing aspects of the subject[4,5]. Since we are a team of physicists and chemists, right away we found it convenient to make a small table of conversion factors, which is reproduced here as Table 1, so that we could all speak the same language. Tritium data comes from Sauter[6].

Table 1: Useful conversions & constants for Chemists and Physicists

1 cal	=	4.184 Joules
1 kcal/mol	=	0.04335 eV/molecule
1 eV/molecule	=	23.069 kcal/mol
1 Faraday of charge	=	96487 Amp-sec
1 Mol T_2	=	2.15×10^{15}Bq
1 dpm/ml	=	1 decay/minute/milliliter
1 dpm/ml	=	1.66×10^{4} Bq/m^3
Tritium in rain(1973)	=	1.1 dpm/ml

Palladium, a silvery colored noble metal, has a density of 12.0 gm/cm^3, an atomic weight of 106.4, a melting point of 1554°C, is known for its catalytic ability, and capability of absorbing large amounts of hydrogen (900-2800x its own volume in H_2). The hydrogen-poor state is referred to as the α phase, and the hydrogen-rich state, at about a 0.5 D/Pd ratio, is the β phase. Overloading to ratios between 0.8-1.3 D/Pd, where the deuterium is thought to continue accumulating in crystal voids, is also reported to be possible with high electrolytic current densities ($\sim$ 1 A/cm^2). Palladium metal consists of a face-centered cubic lattice, with four atoms in the elementary cell. The "empty" α-phase crystal constant is $a = 3.88$Å, with the nearest neighbor distance of $a/\sqrt{2}$. The crystal lattice expands approximately 5% as hydrogen is absorbed. There is considerable uncertainty in the literature, as to the exact nature of the bonding sites of hydrogen in palladium.

It has also been reported for more than 125 years, that a palladium piece, loaded with hydrogen, when removed from solution and placed in the air, may in a short time *spontaneously heat to a red, or orange-hot glow* and can set filterpaper *aflame* or even "explode". This effect depends strongly on the previous surface conditions of the palladium, and its prior history of "loading/unloading" cycling. This effect is a result of catalytic recombination of the hydrogen with oxygen, and nearly the entire load of hydrogen can be released in the runaway heating reaction, over a brief timespan[4]. As early as 1823, a man named Döbereiner invented a cigarette lighter (the so-called "Döbereiners Feuerzeug") using a similar effect of hydrogen on platinum.

The actual loading of D_2 into Pd is exothermic, and endothermic when the deuterium unloads (if no further catalized reaction with oxygen occurs). Numbers for H_2 gas loading of the α-phase are 6-8 kcal/mole H_2, followed in the β-phase by 9.7 kcal/mole H_2, with an integrated value of 8.7 kcal/mole at 30°C, have been reported[7]. The heat of absorption for deuterium is reported by Lewis as being about 15% smaller than for hydrogen. Furthermore, the absolute values of the heat of absorption climb with increasing hydrogen loading into the β-phase. However, additional reactions (such as atomic recombination of D^o to D_2, or formation of water) at the surface of the Pd may dominate the net kinetics. Some of these reactions for hydrogen are shown in Table 2 below.

Table 2: Some relevant chemical reactions

$H_2O_{(l)} \rightarrow H_{2(g)} + 1/2\ O_{2(g)}$	+68.4 kcal/mole(H_2)
$D_2O_{(l)} \rightarrow D_{2(g)} + 1/2\ O_{2(g)}$	+70.2 kcal/mole(D_2)
$H_2O_{(l)} \rightarrow H_2O_{(g)}$	+10.76 kcal/mole
$D_2O_{(l)} \rightarrow D_2O_{(g)}$	+11.15 kcal/mole
$H_{2(g)} + 2\ Pd \rightarrow 2\ PdH$	-8 kcal/mole(H_2)
$H_2 \rightarrow 2\ H$	+104 kcal/mole(H_2)
$2\ H + 2\ Pd \rightarrow 2\ PdH$	-112 kcal/mole(H_2)

Hence in any case, one *should* see a thermodynamic difference between a heavy water and light water test cell, without necessarily invoking any nuclear processes. Consequently, the publicized persistence of R. A. Huggins[8] (Standford) of reporting heat differences between his heavy and light water cells, is at first glance plausible, depending on the nonequilibrium conditions in his cells.

2.1 Estimate of loading times

To give the reader an idea of the absolute minimum timescales for loading a piece of palladium electrolytically, let us consider a hypothetical 1 cm^3 piece, containing 6.8×10^{22} atoms of palladium. If a current of 2.5 Amps was 100% efficient at loading a D^+ ion into the lattice, allowing no extraneous bubbles, essentially instantaneous diffusion rates, and

no other recombination effects, while neglecting the lithium, then the loading time for a 1:1 fraction of Pd:D would be approximately 1.2 hours. This only serves as an absolute lower limit.

In reality, the diffusion velocity of deuterium through palladium depends on the surface conditions of the palladium, and its prior history. At current densities of order 10 mA/cm^2 or higher, deuterium can not diffuse into the metal fast enough, and the excess appears in the form of bubbles on the cathode. Hence for a cubical configuration, identifying the useful current density as only 10 mA/cm^2, one might expect a loading time of approximately 50 hours.

It is believed that the loading time scales with the thickness of the piece, squared[4]. The e-folding diffusive time constant is of the form $\tau = r^2/\pi^2\Delta$, where r is the radius of the Pd rod, and a diffusion rate (pessimistically) $\Delta \sim 10^{-7}$ cm^2/s. Approximating the cubical case above, then one obtains by this estimate a time of 70 hours. More detailed measurements of the α and β-phase diffusion rate constants at room temperature, show the β-phase to have about a $10 - 30\times$ higher diffusion rate at room temperature[5].

As a rod of palladium is loaded with deuterium, the β/α transition zone slowly sweeps from the edge into the center of the rod[4]. Existence of neighboring metal crystals in different phases has complicated crystallographic determination of the loaded-lattice constants. Under electrolytic-loading at 1.6 mA/cm^2, it has been observed that the phase boundary advanced upwards out of the surface of the solution, in a piece of Pd only partly submerged, at a velocity of 0.2 mm/hour[9]. This implies loading times for a 5 mm radius piece would be greater than 25 hours.

Summarizing this section, the deuterium loading time for a 5 mm radius Pd piece is estimated to be of the order of 30-90 hours at a current density of 10 mA/cm^2 or higher, if the surface isn't chemically poisoned.

2.2 Comments on heat balance

Observations of "excess heating", or changes in the heat balance of the system, after hours of stable operation, must be extremely carefully considered. In particular, changes *are* expected after the palladium saturates with deuterium, because of differences between when deuterium is lost in the bubble form at the surface of the palladium, as opposed to when the same deuterium is loading into the palladium. In addition, whether or not the dissolved deuterium is effectively in atomic or molecular form as it is bound in the lattice, or even later, simply in interstitial cracks, all affect the net heat balance. This doesn't even include possible Li-D, or Pd/Li reactions...and is only discussed above for the part of the palladium immersed in water! If for some reason a portion of the palladium becomes catalytically activated above the liquid surface during the course of the experiment, then considerable heat is released from the catalyzed recombination of the electrolytically disassociated heavy water, instead of being counted as a loss to the system. This effect is typically of order 10-50% of the total work delivered to the cell (depending on the cell voltage and current density), and can explain the so-called

#	Nuclear reaction	Q-value (MeV)	specific reaction rate ($s^{-1}W^{-1}$)	Ref.
1	p+d $\rightarrow\gamma+^3$He	5.49	1.13×10^{12}	[12]
2	d+d $\rightarrow$p+t	4.03	1.54×10^{12}	[13]
3	d+d $\rightarrow$n+^{3}He	3.27	1.90×10^{12}	[13]
4	d+d $\rightarrow\gamma+^4$He	23.85	2.61×10^{11}	[14]
5	p+t $\rightarrow\gamma+^4$He	19.81	3.14×10^{11}	[12]
6	d+t $\rightarrow$n+^{4}He	17.59	3.53×10^{11}	[13]
7	d+t $\rightarrow\gamma+^5$He	16.63	3.74×10^{11}	[12]
8	p+^{6}Li $\rightarrow^3$He+^{4}He	4.02	1.55×10^{12}	
9	d+^{6}Li $\rightarrow^4$He+^{4}He	22.4	2.77×10^{11}	
10	p+^{7}Li $\rightarrow^4$He+^{4}He	17.5	3.55×10^{11}	

Table 3: A (short) list of nuclear reactions discussed for cold fusion.

"excess" heat output of the F&P cell in every published case[10].

2.3 Comments on nuclear processes

Here we quickly review some information about known nuclear reactions which might possibly be involved in the so-called cold fusion experiments. First news reports talked about powers of the order of "Watts" from nuclear reactions. With a conversion factor of 1.609×10^{-13} Joules per MeV we get the specific reaction rates (reactions per second per Watt) listed in Table 3. The energies released with these reactions have been calculated from the atomic mass excesses as listed in [11].

The reactions (2) and (3) are those used in existing hot fusion plasma experiments and their cross-sections are roughly equal. The thermal reaction rate in a deuterium molecule for these reactions is in the order of 10^{-64} reactions/second/particle pair[15]. Although for reaction (4) the cross-section is a factor of about 10^{-7} lower[16], it received a lot of attention since it liberates large amounts of energy, with no direct neutrons. However there has been no convincing idea of how to overcome its small cross-section or how to deal with the absence of the normal, powerful (23.8 MeV) gamma-ray, although speculation did center on "coherent processes" (P. Hagelstein, MIT).

Almost all the nuclear reactions in this table create a helium isotope, and therefore the detection of He in the Pd samples would be a significant indication of fusion, and conversely, absence of helium in a "working cell" would weigh heavily against fusion as the source of any "excess heat". Widely publicized reports concerning the presence of high levels of helium (Wallings and Simons, Utah) were withdrawn, and a cell from Texas A&M which *is* reported as having excess heat, has much too low a level of helium to associate the excess heat with nuclear fusion[17]. For those reactions which don't

create directly gammas, the generation of fast charged particles would create secondary gammas due to bremsstrahlung or from Coulomb excitations of palladium nuclei[18]. The idea of a nuclear-equivalent Mössbauer effect, where up to 24 MeV of reaction energy could be absorbed by the metal crystal lattice through the generation of thousands of phonons, without accompanying gammas, is highly exotic in our opinion. In summary, we know of no nuclear fusion process, which would not be accompanied by the generation of detectable amounts of nuclear radiation of some sort!

3 Experimental Setups

Following information gleaned from news reports, newspaper photos, computer network mail, we began experiments in a chemical fume hood, on the second floor of the chemistry lab of the IPP. Concern about possible chemical explosions from recombination of the deuterium and oxygen electrolysis products required a location with good ventilation. A location with good radiation shielding from a health/safety viewpoint was only a secondary consideration, since we didn't have any good reason to believe in hazardous radiation production, but we were certain about the chemical hazards. From the viewpoint of shielding against background radiation in order to see lower signal levels, we were confident that we could easily see levels as large as P & F were initially reporting, without special precautions. (Had we started our experiments later, we would have, in retrospect, gone for better background shielding.)

The form, and amount of palladium immediately available for experiments was initially a limiting factor. We had several grades of heavy water, and a variety of deuterated salts and acids available. Platinum mesh anodes, and sheets of platinum-irridium were also available. We had both metallic lithium (in kerosene) and pure LiD powder. For the second and third experiments, 99.75% purity D_2O was used, originating from a total of three 100 ml, and one 250 ml Merck flasks, all more than 12 years old. The third experiment used a low purity D_2O water bath for a time, in order to minimize possible H/D exchange to the high purity central cell over extended periods of operation. Towards the end of the third experiment, this was replaced with a normal H_2O water bath, and insulation of the water bath was modified to reduce evaporative losses and isolate heat from the magnetic stirrer motor.

Thermo-resistor elements and multi-pen strip chart recorders were borrowed from ASDEX, as well as direct readout thermocouple elements. Since the experiment would run for long periods, without constant human monitoring, the "paper tape" readout was essential. Standard precision ($\pm 0.1^{\circ}C$) thermometers were also placed for "human readout" as needed, in both the center cell, water bath, and as an air temperature indicator. We looked for a constant temperature water bath regulator with known cooling/heating inputs, but did not find one to suit our needs, and hence ended up using a water bath cooled by a constant flow of room air. The cell current, voltage, air temperature, and water bath temperature were continuously monitored on a 4-pen strip chart recorder.

The temperature scale was 4°C/cm, with a reading accuracy of 0.5 mm, corresponding to 0.2°C, comparable to the additional manually read thermometers.

NaI detectors, normally used for measuring radiation produced by runaway electrons in ASDEX, were borrowed from Dr. Fussmann. A 2" diameter, 10" long BF_3 detector, encased in a polyethlene moderator, was our main neutron diagnostic. We borrowed an automatic, absolutely calibrated radiation monitoring system normally used for safety measurements in the ASDEX building. A pulse height analyzer was used for both gamma spectra from the NaI detector (later also a Ge(Li) crystal), and to observe the proper pulse energy from the large BF_3 counter.

3.1 Exp. #1, Thin-wall Pd tube

Our first attempt principally served as a "shake-down" experiment for later efforts. This experiment was performed with materials at hand, and stock chemistry glassware. A 3.85 gm thin-walled palladium tube (1.5 mm dia, 0.2 mm wall, 19.4 cm length, outgassed in a vacuum oven at 300°C overnight) was coiled in a vertical spiral, with both ends of the tube extending above the surface of the electrolyte. The underwater portion had a length of 15 cm, for a useful loaded-volume of 0.25 cm^3. A 26 gm cylindrical platinum mesh anode surrounded the cathode, and the cell was a 250 ml Pyrex beaker, surrounded by a stirred 800 ml water bath.

This run began on Thursday afternoon, March 30, before we had gotten any information about the desired electrolyte makeup or concentration. We only had vague rumors about lithium, and we fortuitously chose to use 100 mg of LiD per 100 ml of D_2O, which turned out to be almost exactly what F&P used in their experiments (0.1 Molar)! Low-grade heavy water (95% isotopic purity) was used (only in experiment #1) as the solvent to makeup the electrolyte. Later, we continued to use LiD instead of metallic lithium, because the heat of reaction upon solvation is lower for LiD, and, in addition, we didn't have to worry about removing kerosene or oil from the available metallic lithium. In particular, we didn't want to contaminate our electrolyte with any hydrogenated compounds.

3.1.1 Evolution of bubbles

With this first attempt, we watched (as the cell was initially turned on) the delay between bubbles first appearing at the anode, and then 1-2 minutes later (at 130 mA and 3 volts) bubbles appearing on the surface of the palladium cathode... indicative of the initial absorption of deuterium into the palladium lattice. The chosen operating point was 1 Ampere, and a voltage of 5.4 Volts, which with the 2.5 cm^2 surface area (one-sided), corresponded to a 400 mA/cm^2 loading current. We noticed a large, continuing flux of gas (presumably deuterium) escaping up the center of the cathode, and realized that since the inner wall of the cathode was *not* exposed to electrolysis pressure, that it was unlikely we could "overload" the Pd with deuterium. Hence the run was stopped after

an overnight vigil, and a duration of 16 hours. (A 10 minute interruption in the current after 7 hours was made to watch the bubble release from the cathode, and observe the voltage decay). The mass increase of the cathode was 40 mg, or if all deuterium, this corresponds to a ratio of 0.7:1 D:Pd atoms. We later made estimates of the loading timescale to be longer than 45 minutes.

3.1.2 Temperature(s)

During this first experiment only, one of the two (strip-chart monitored) thermo-resistors was encased in a thin glass capillary tube, and actually immersed directly in the center cell. Water in the capillary conducted heat to the metal probe jacket. A meter-readout thermocouple element was similarly encased but measured the center cell temperature in the solution, near its surface. (The reason for the glass jackets, was to avoid unwanted electrolysis action on our sensing elements.) First crude open-cell heat balance

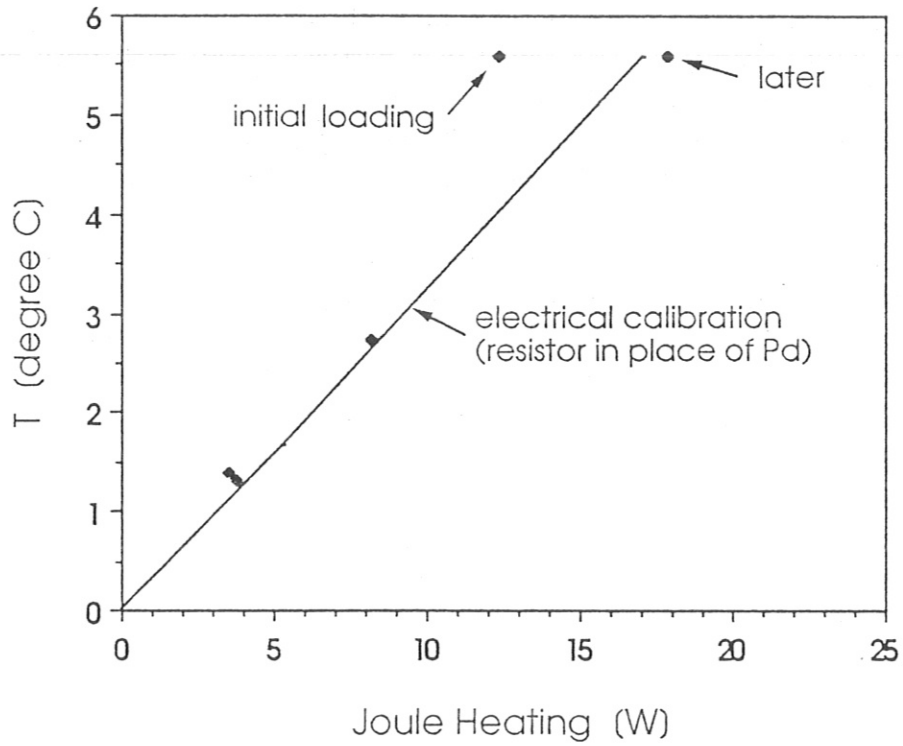


Figure 1: *Experimental temperature measurements in the # 1 cell. Temperature rise of TC at bottom of center cell relative to the water bath. Time constant is only 3 minutes. The cell was hotter during the first 10-15 minutes during the loading phase, than hours later in equilibrium.*

measurements are shown in Figure 1. As a zeroth order approximation, a 5.5 Ω resistor was used for comparative ohmic heat inputs, in the position of the Pd (but without bubbles). With the actual Pd/Pt cell, at its nominal operating point of 5.4 Watts, one could measure a 2°C temperature differential between the top and bottom of the cell, along

a vertical chord just *outside* the Pt anode mesh. Near the Pd itself, the gradient was even larger. We calculate the Joule heating from the electrolysis current, by subtracting 1.54 volts from the applied voltage (as F&P have done). Initial nonequilibrium effects of the exothermic loading was apparently observed, but clearly better measurements (in the following experiments) were needed to draw more significant conclusions.

Even though we noted "strong" bubbling, it was quickly clear from multipoint measurements with the three thermometers directly in the center cell, that a "single point" temperature reading in the center cell would not be representative of its energy content, due to substantial thermal gradients. We did not know at that time, that this was precisely what F&P had done.

3.2 Exp. #2, Large surface area Pd sheet

The second cell was put into operation on April 4, 1989, after our first receipt of the F&P paper on Monday evening April 3.

This cell was more sophisticated, and some parts actually took a day in the shop to prepare! A 1/2-scale sketch of the cell is shown in Figure 2.

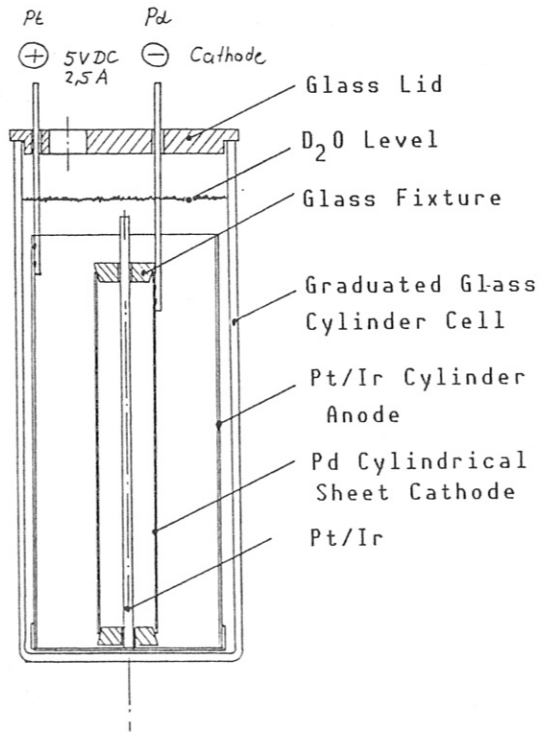


Figure 2: *Schematic of the second experiment, showing concentric Pt/Ir anode, and inner palladium cylindrical sheet, with central Pt/Ir wire, to insure a well-defined field pattern on the 0.2 mm thick palladium sheet. The hole in the glass lid was plugged with a conventional glass-encased mercury thermometer.*

We still hadn't located any solid rods of palladium, so we employed a cylindrical thin sheet of palladium as the cathode. A two part anode, with a central Pt wire, as well as a

concentric surrounding Pt/Ir sheet, allowed both "sides" of the palladium to be subject to electrolysis. The electric field configuration was well-defined, though undoubtedly giving a differential current flux on the two surfaces of the palladium sheet. The sheet was 0.2 mm thick, 9.4 cm tall, and had a cylindrical diameter of 1.5 cm, for a mass of 11 grams. Pretreatment of the rolled sheet was limited to scouring the surface with a wire mesh, followed by vacuum-baking overnight at 300°C. The total (two-sided) surface area of 88 cm^2, was designed to be completely submerged in solution and a thin-walled Pd (1.5 mm dia) tube formed the spot-welded electrical connection to the cathode. Losses out the top of the cell were restricted to small holes in the close-fitting glass lid.

Initially to get an idea of the chemical purity of the 99.75% D_2O, we turned up the cell voltage (before adding the LiD salt) to a value of 48 volts, obtaining only 60 mA of current. Then after the salt was added, bringing the concentration up to 0.11 Molar, we raised the current up to a value of 2.5 amperes, at a voltage of 5.5 volts.

Based on our #1 experiment, we placed the chart-recorded thermo-resistors in the stirred water bath, and in the air. Air flow was maintained constant in the fume hood, by always keeping the fume hood door in a fixed position. With an air temperature of 24-25° C, the stirred water bath was in equilibrium at 34° C, and a thermometer in the center cell read 36.5° C near the bottom of the center cell. At a current of 2.5 Amps and a voltage of 5.0 volts, the Pd had an average current density of 25 mA/cm^2. This was maintained, with minor interruptions (to watch the bubbles unload at one point, and at another, to repair a bad tack weld contact mechanically) for 45 hours. After the first four hours of operation, thermal conditions in the cell were essentially stationary for the remaining 41 hours of the run. Upon completion of the run, a 2 ohm resistor was put in place of the palladium, to crudely simulate the ohmic heating (although admittedly without the effects of bubbles), directly in the center cell, while monitoring power, air and bath temperatures with the strip chart recorder.

3.3 Exp. #3, Cast Pd stone

After our initial searches for palladium yielded only thin-wall palladium tubes from hydrogen purification systems, and 0.2 mm thick sheets of palladium from purification fingers, we realized we needed to buy, or make our own pieces. Since the time for purchasing palladium was long (about a month), we took pieces of palladium we had, and looked for a suitable furnace, preferably in a vacuum. The electroplating group at IPP also has a vacuum apparatus with an e-beam for melting metals. We thought this would be satisfactory, and we could also pump away desorbed gases.

Using this facility we created a 22 gram vacuum-cast (1.8 cm^3) "stone", to reproduce the so-called "ignition?" experiment of F&P, as shown in Figure 3. Due to the available water-cooled copper crucible, the shape was irregular, much like a pebble that you would choose to skip across the water at a stream. A large quantity of gases were evolved during the 10-minute melting process. No machining was done on the piece, which was allowed to cool gradually to room temperatures in vacuum. The resulting shiny silvery piece had

a surface area of 10 cm^2, with the long dimension of 2.8 cm, thickness of 5-6 mm, and a width of 1-1.4 cm. A thin-hollow palladium tube (approximately 2 gms worth) was also attached with the e-beam, to serve as the electrical lead, and mechanical support to the stone. During later operation, the stone was fully immersed, with its top approximately 1.5 cm underwater.

The reactor then consisted of a machined 200 ml pyrex beaker, with a close fitting glass lid, having four small holes, one for a thermometer, one each for the palladium and platinum electrical connection. A cylindrical platinum mesh anode surrounded the palladium cathode. Gas and a small amount of spray could escape through two of the holes (as evidenced by a gradual white powder residue build-up, known to be Li_2CO_3, from carbon dioxide in the air interacting with the strongly basic LiOD solution). A photo depicts the preassembly in Fig. 3, and a ruler is shown for scale.

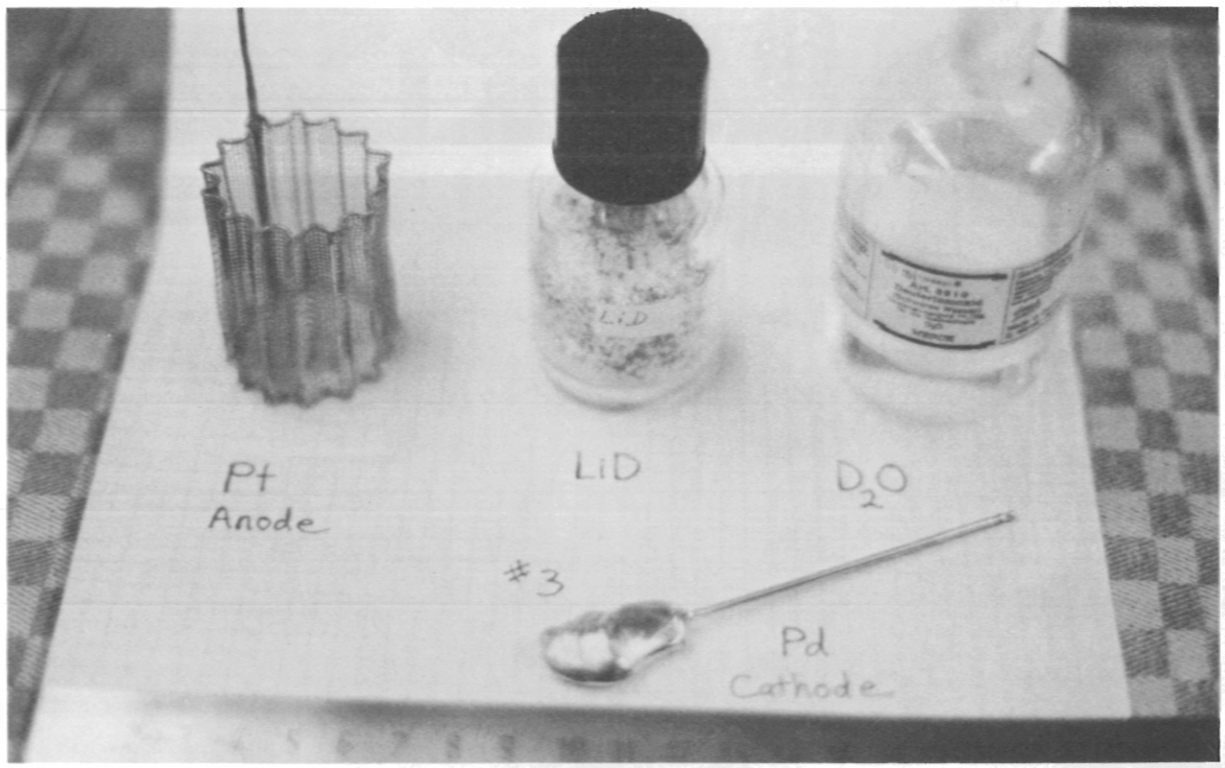


Figure 3: *Picture of the third, and longest running experimental cell components, including from left to right, the Platinum mesh anode, the bottle of LiD powder used, the Merck 99.75% heavy water, and the e-beam melted 22 gram palladium "stone".*

The assembly was then supported in a larger 1-liter beaker, which was filled at first with 750 ml of low grade D_2O, and then later in the run with the same amount of normal water. A thermo-resistor monitored the bath temperature in the middle of the bath, and a digital readout thermometer monitored the surface temperature of the water bath.

A photo of our final working setup in the lab is shown in Figure 4a, and a closeup of the #3 cell in operation in Fig. 4b. In the overview picture, the pulse height analyzer

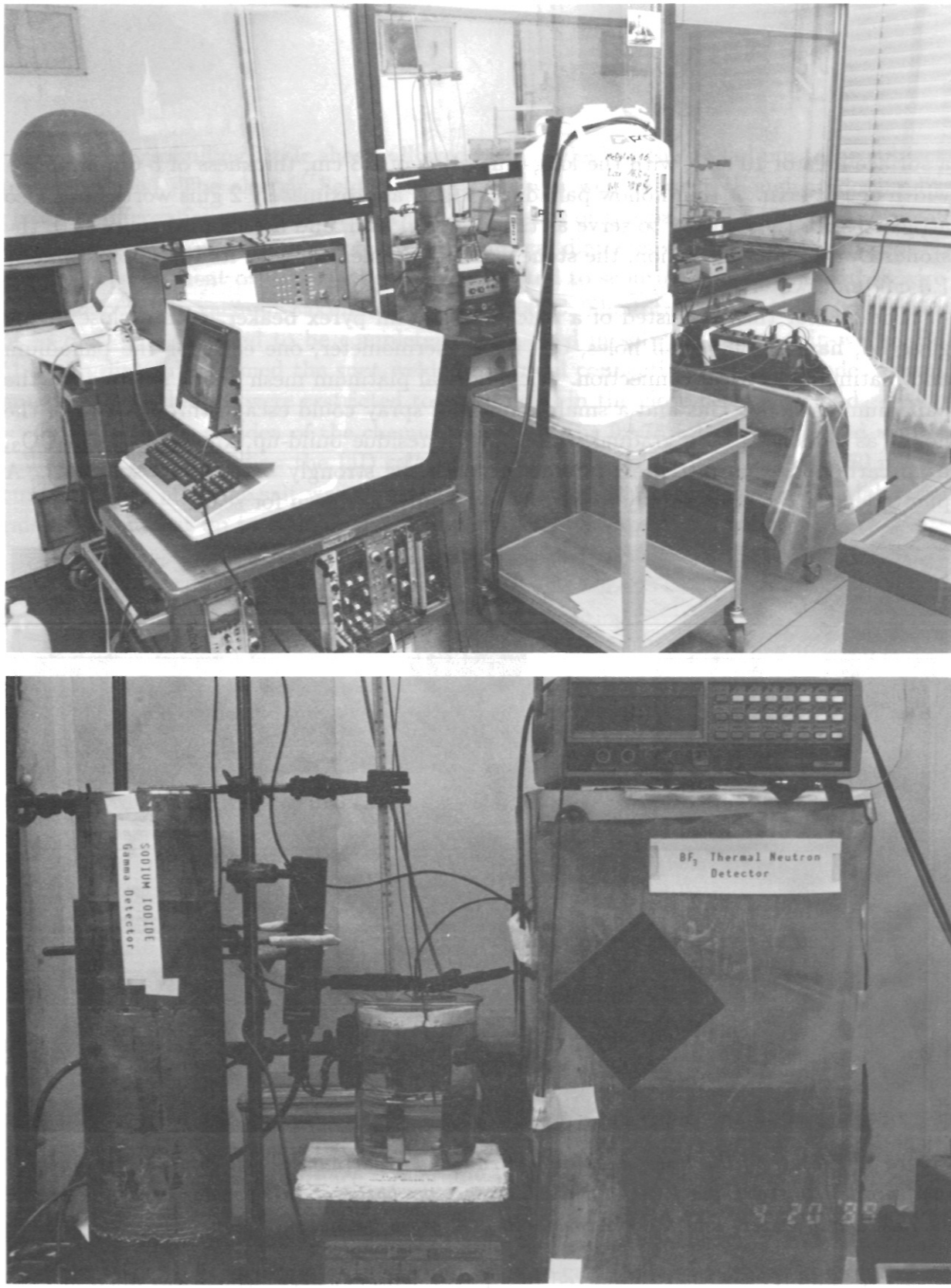


Figure 4: *(a) General lab overview picture. (b) Closeup of the #3 experimental configuration on April 20, 1989.*

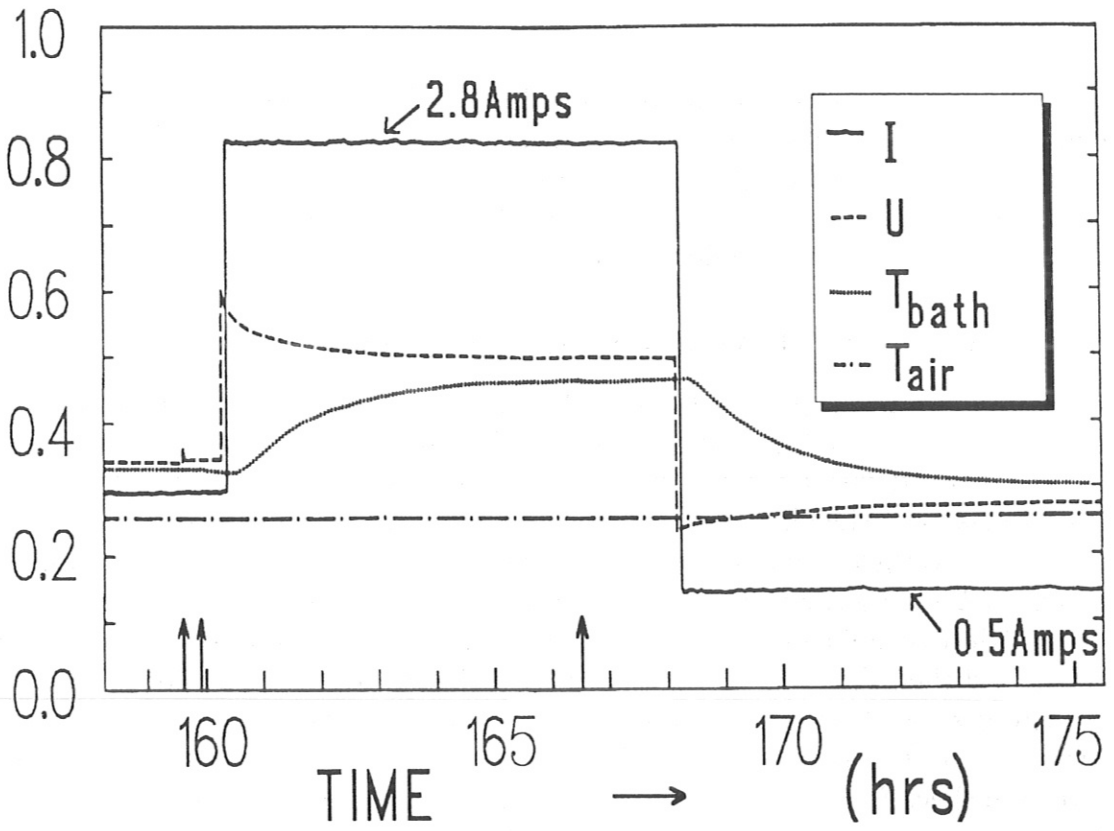


Figure 5: *A sample of the multi-pen chart recorder traces during forced transients, 160 hours into the #3 experiment. Cell current, voltage, and waterbath and air temperatures from the strip chart record are shown for a 17 hour period. Full scale readings correspond to 3.4 Amps, 18.8 Volts, and 100 °C for the respective traces.*

and chart recorder are visible in the foreground, while the cell itself is almost hidden by the Princton GammaTech Ge(Li) high resolution gamma detector. The Bonner sphere of the absolutely calibrated Berthold monitoring system, and controller, sits in the left fumehood. In the closeup photo, one sees the lead-shielded NaI detector to the left, the water bath of the cell in the middle, and the rectangular shielding of the BF_3 neutron detector to the right. Insulation has been added between the stirrer motor and the waterbath, and also some floating insulation on the waterbath itself, to further reduce long term evaporation. Thermometers and thermal-resistor probes are also visible.

Typical loading currents were 2-2.5 Amperes, with a current density then in the neighborhood of 200-250 mA/cm^2. The surface of the stone was noticeably darkened after a day of operation. The current was maintained for constant for approximately 145 hours, before being changed as an adjustable parameter over the subsequent 360 hours of operation. Rapid current increases from 1 A to 3 Amp were peformed, and also rapid decreases down to 0.5 A. After waiting for several 100 minute e-folding times of the water bath, new readings would be taken. An example of such changes are shown in Figure 5, where the response of the voltage and bath temperature are recorded on the

strip chart, in addition to the controlled current and monitored air temperature traces. Perturbations due to topping off the cell with room temperature D_2O, and of refilling the water bath to its marked height, are also visible and denoted with arrows. Replenishment of the center cell D_2O averaged 16 ml/day. No unexplained excursions were apparent when we tried to force nonequilibrium conditions.

3.3.1 Termination: "Fusione superfredda" attempted

We stopped the #3 experiment after 21 days of operation, by shutting off the cell, disconnecting leads, and quickly (in 10 seconds) dropping the loaded Pd piece directly into a Dewar filled with a few hundred ml of liquid nitrogen. The Dewar was surrounded by a plexiglas blast shield, and placed immediately next to the bare BF_3 detector, which had 20" of polyethylene moderator directly behind it. In this way, we should have been sensitive to both thermal, and fast neutrons, if any came from stress-induced fusion as the Pd lattice unloaded[19,20]. No neutrons above backgrounds were seen for 40 minutes, and then the piece was removed from the liquid nitrogen allowed to warm up. No neutrons above background were seen in this phase either.

After removal from the liquid nitrogen, we rested the Pd stone on a piece of wood (in order to check for possible catalytic scalding, due to recombination[10]). The surface of the darkened piece, which quickly became frost covered (from humidity in the air) warmed to 0°C after 15 minutes. A thermocouple in close contact with the main Pd stone, saw no thermal excursions, during the next hour, and so we let it sit overnight on a piece of tissue paper. No evidence of catalytic recombination of deuterium at the surface of our piece of Pd was seen. It is interesting to note here, that Lewis[5] indicates on pg. 68 of his book, that "specimens with (originally) smooth bright surfaces generally have relatively poor catalytic activites"....but "the quantity of hydrogen which is finally absorbed in the steady-state may in fact substantially exceed the quantity of hydrogen which can be absorbed by electrodes with surfaces of a high activity..."

3.3.2 Mass increase

The Pd stone was weighed before and after the experiment. It clearly had devolved a significant amount of deuterium (as evidenced by vigorous bubbling after cell shut-off) during the one hour it was in liquid nitrogen, and the further night it sat exposed to the air, while warming up. Nevertheless, the mass uptake (24.0290 - 23.7759 gms) of 0.31 gms corresponds to a ratio of 0.70:1 D:Pd, if any possible lithium is neglected. This is close to values of 0.67 reported in the literature.

Weighing the Pd after the experiment is in general problematic. The piece must be dry, and yet at the same time, you don't want to let the deuterium unload significantly, so the measurement needs to be made quickly. From videos of the bubbles unloading from the Pd, we crudely estimate a loss of 0.3-1 liter of D_2 from the Pd, before it was weighed the next morning. Titrating the electrolyte puts a limit of the Lithium that was unaccounted for at 5-10% of the original amount added, or equivalently, less than 15 mg.

Subtracting the mass of Pd in the thin connecting lead, we are confident that the Pd stone itself was loaded in excess of 0.8 D:Pd, and most likely in the range of 0.9 to 1.2 D:Pd.

3.3.3 Post-mortem analysis

After a period of four days, we cut the stone into two pieces, without much difficulty, using a hacksaw. The stone was hardened, and embrittled, relative to an unloaded piece of palladium. We etched the smaller piece with a weak solution of *aqua regia* acid in order to better see the crystal structure. After rinsing in distilled water, we placed it underwater to view the continuously outgassing bubbles. Figure 6 shows Polaroid images of the cut, and etched surface, at 40× magnification. Bubbles are easily visible,

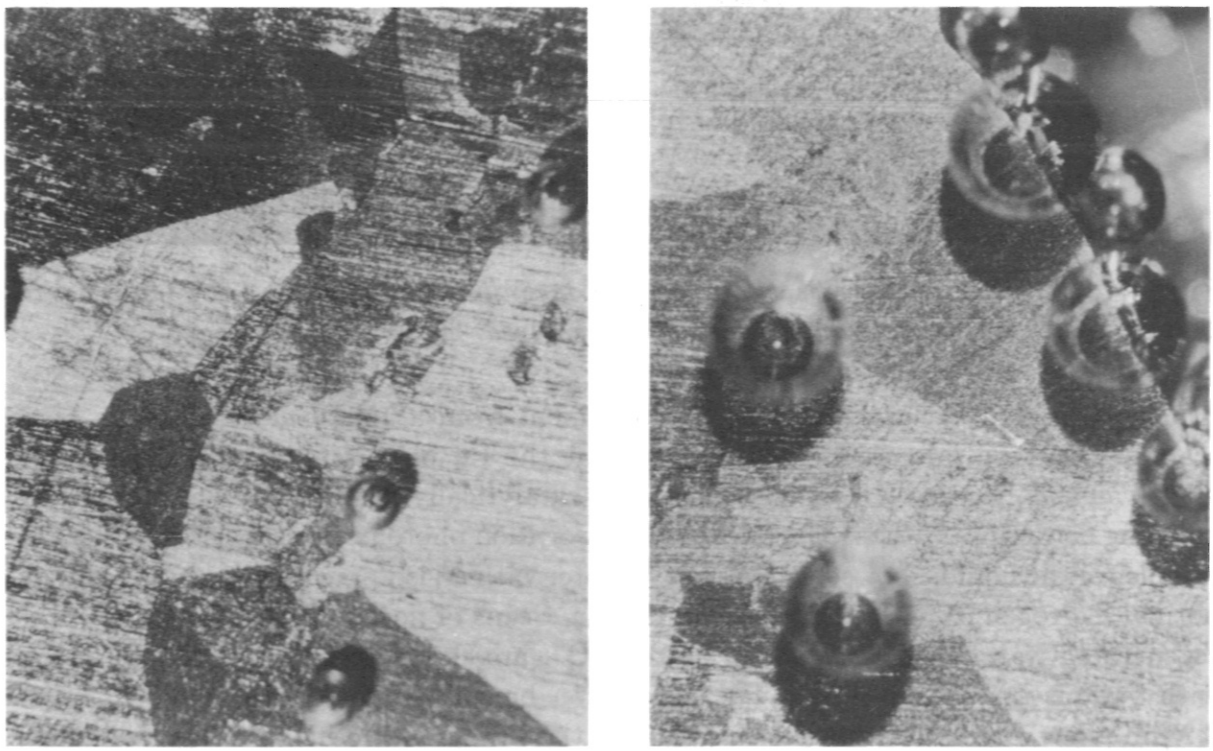


Figure 6: *Microphotograph of a slice of the Pd stone, showing the large crystal grain structure, and escaping gas bubbles, four days after termination of the experiment.*

and originate at the edges of the stone, and also along crystal grain boundaries. We were surprised at the large size (2mm×2mm) of the grains. We also could see evidence of small voids in the stone, evidently a relic of the original casting.

We noticed six weeks later, that significant fractures in the stone had developed, as the stress of the escaping deuterium fissured the piece predominantly at crystal boundaries. At this time, electron microscope pictures were taken, showing the fractures occurring along crystal boundaries, as can be seen in Figure 7. Interestingly, three days after

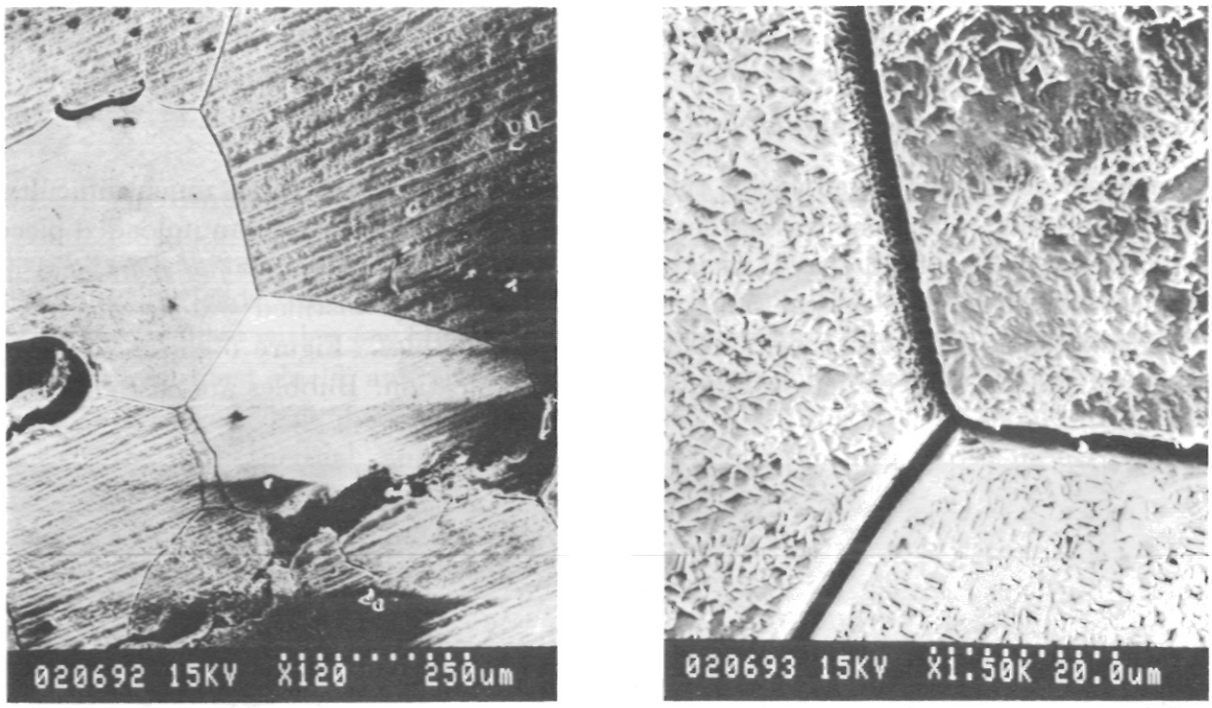


Figure 7: *Electron microscope photos at two magnifications, showing the large fissures that developed in the Pd stone, as deuterium escaped, weeks after loading.*

exposure to high vacuum, and the bombardment from a 15keV electron beam of the scanning electron microscope, we noticed that the Pd piece (stored in a capped glass vial) was visibly wet. It had finally become catalytically active, and was recombining escaping deuterium with oxygen in the air! Absorption photometer scans of 100 microliter samples of the liquid, showed it to be heavy water with an isotopic purity of 85%, with the normal hydrogen coming from water in the air. Scans of normal water, the catalytically regenerated water, and that of the "pure D_2O", are shown in Figure 8 for comparison with various known dilutions. The absorption bands of water between 3000 Å and 3.2 μm allow a quantitative determination of the isotopic ratio between light and heavy water. However, due to the 8-week (post experiment) intermediate exposure to air, immersion in normal water for previous bubble photographs, etc., this represents only a lower bound on the deuterium fraction in the freshly unloading Pd piece. Heavy water continues to be generated, ten weeks after the run was ended. Just as a check, similar scans of stored samples of the #3 electrolyte after the run, showed that its isotopic purity remains greater than 99% D_2O.

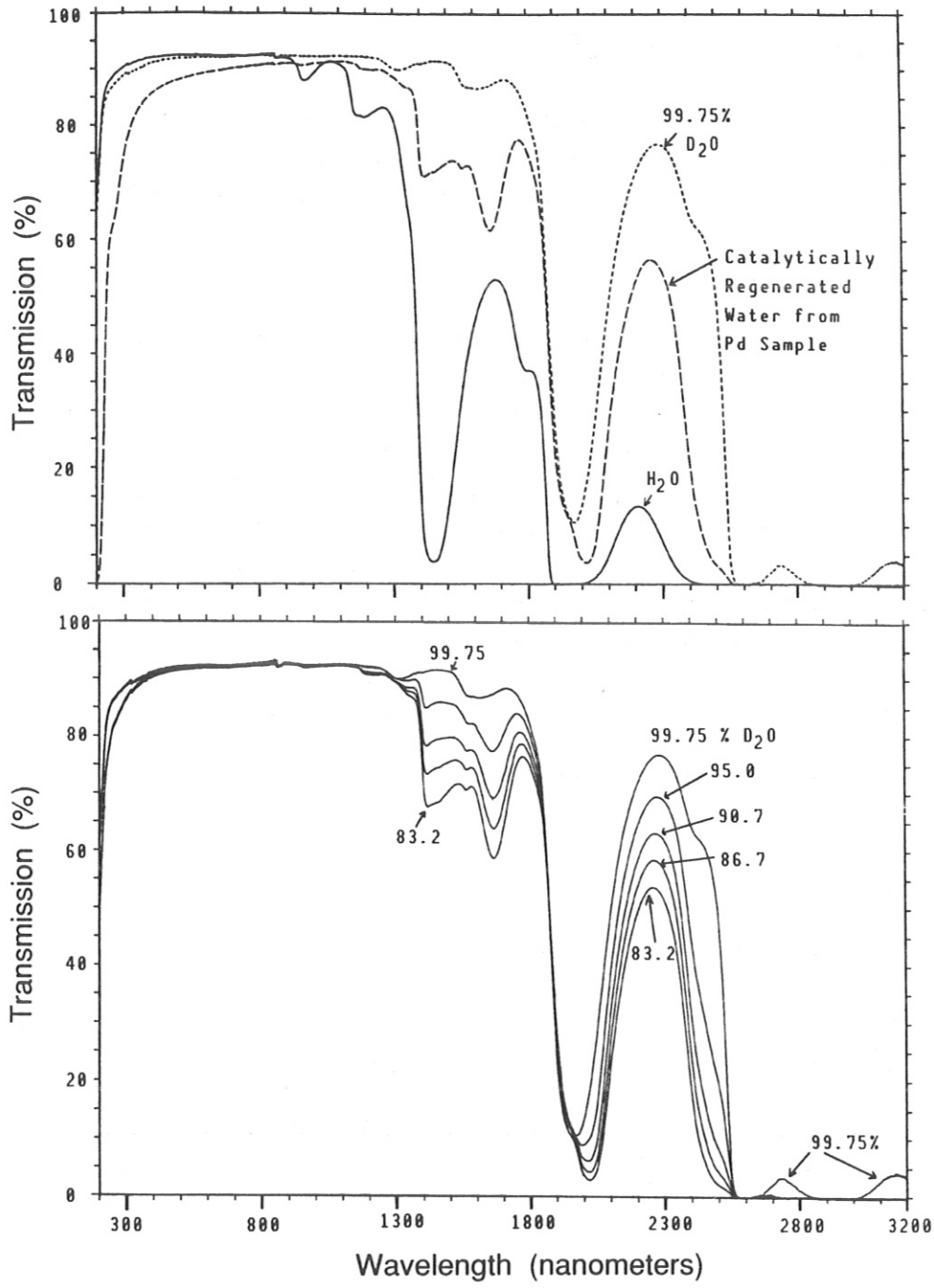


Figure 8: *(a) Absorption spectroscopy between 300 nm and 3200 nm, comparing the catalytically regenerated water from the surface of the Pd, with normal water, and 99.75% purity heavy water. (b) A series of dilutions of the high purity D_2O with known amounts of light water, enabling a quantitative determination of 85% deuterium concentration in the catalytically regenerated 100 μliter sample from the Pd.*

4 Neutron Diagnostics

We decided initially to look for thermalized neutrons, and not to attempt neutron spectroscopy until we first saw some neutrons. Therefore we used a sensitive BF_3 detector and an absolutely calibrated radiation monitor system (Berthold LB1026).

4.1 BF_3-detector

The BF_3 detector was a 2" diameter proportional counter with an active length of 31.1 cm, filled with 0.933 bar of BF_3 (Reuter-Stokes, Mod. Nr. RS-P1-1613-203, Ser. Nr. W-4452) enriched in ^{10}B. It was operated at a bias voltage of +2100 V with a Canberra 2006E preamplifier and a Canberra 2015A Amplifier/Timing Single Channel Analyser. The counting pulse from the Amp/TSCA was fed into an Canberra 1772 Counter/Timer (and onto a strip chart recorder), and the amplifier output was checked on a Pulse Height Analyzer Nuclear Data ND 66 with a Nuclear Data ADC ND750. The counter as well as the spectrum analyser were used to measure the neutrons, in short time intervals (about 10 min.) and in longer integration periods (up to 26 hours) respectively.

Figure 9 shows the BF_3 detector pulse height distribution, with markers indicating the region of interest corresponding to expected neutron pulse heights.

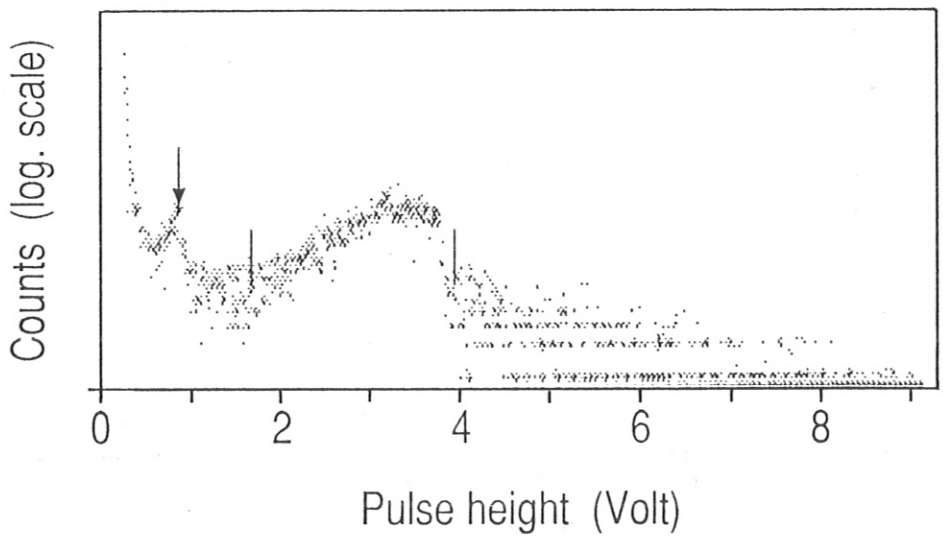


Figure 9: *Pulse Height Distribution of the BF_3 proportional counter (2" at 0.933 bar). Background measurement for 25 hours. The region of interest used as neutron signal is indicated with the markers.*

This, however, is not a spectrum of the neutrons, but one of the charged products which are created in the BF_3 gas with the reaction $n + {}^{10}B \rightarrow {}^{7}Li + \alpha + 2.792$ MeV [21], and gives no information about the actual neutron energies. The low energy peak (marked by the arrow) is due to pulses counted in the dead volume at the ends of the

detector [22], and its position is independent of bias voltage (unlike the main spectrum). The spectrum doesn't show the expected structure with two distinct peaks, but that might be due to wall effects and unoptimized electronics.

The detector was in a polyethylene moderator block 25 cm × 25 cm wide and 50 cm high. Not knowing the structure of the neutron background this moderator block was surrounded by 0.5 mm thick cadmium sheets to "block" thermal neutrons from background. This cadmium shielding, however, didn't change the background rate significantly. Before its installation we measured 3.81 ± 0.07 counts per minute (cpm) in the spectrum, and with the Cd shield 3.75 ± 0.13 cpm. A day before this installation the background rate was 3.33 ± 0.06 cpm, which simply indicates the variability of the cosmic ray background in time.

The lower and upper thresholds on the TSCA were set correspondingly to count roughly the same part of the spectrum.

After the first experiment we calibrated the BF_3 detector with a ^{238}PuB neutron source, absolutely calibrated in August 1977 as emitting 6.81×10^6 neutrons per second, therefore emitting 6.21×10^6 neutrons per second in April 89.

The source was positioned above the electrolytic cell, just in the same distance from the BF_3 tube as the Pd cathode (20 cm), but without any water inbetween. Actually there is about 3 cm of water between the palladium and the moderator which would moderate neutrons before entering the Cd sheet therefore giving smaller count rates. Thus our calibration factor (neutrons/count) will be a lower limit to the true value. With the strong source at this position the neutron counter showed 14992 ± 27 counts per second. From a similiar detector used for neutron flux measurements on ASDEX we know that the dead time is about 6 μs. This results in a dead time corrected countrate of 16.48 ± 0.27 kHz and a calibration factor of 377 ± 6 neutrons per count.

The pulse height analyzer had more problems with the high count rates for its particular calibration. For a real time of 60 seconds, the ADC had a live time of only 39 seconds and the spectrum showed strong pile-up. When we also used the pile-up peak we got 7.4×10^5 counts, corresponding to 327 neutrons per count. Since it is not clear how to take the pile-up into account correctly we attached a large uncertainty of at least 20 % to this calibration factor, and used only the separate counter as a calibrated diagnostic.

4.2 Neutron results

Figure 10 shows the neutron measurement (from the counter) during experiment #3. The counter data were not recorded automatically, but only manually, and therefore are not continuously available.

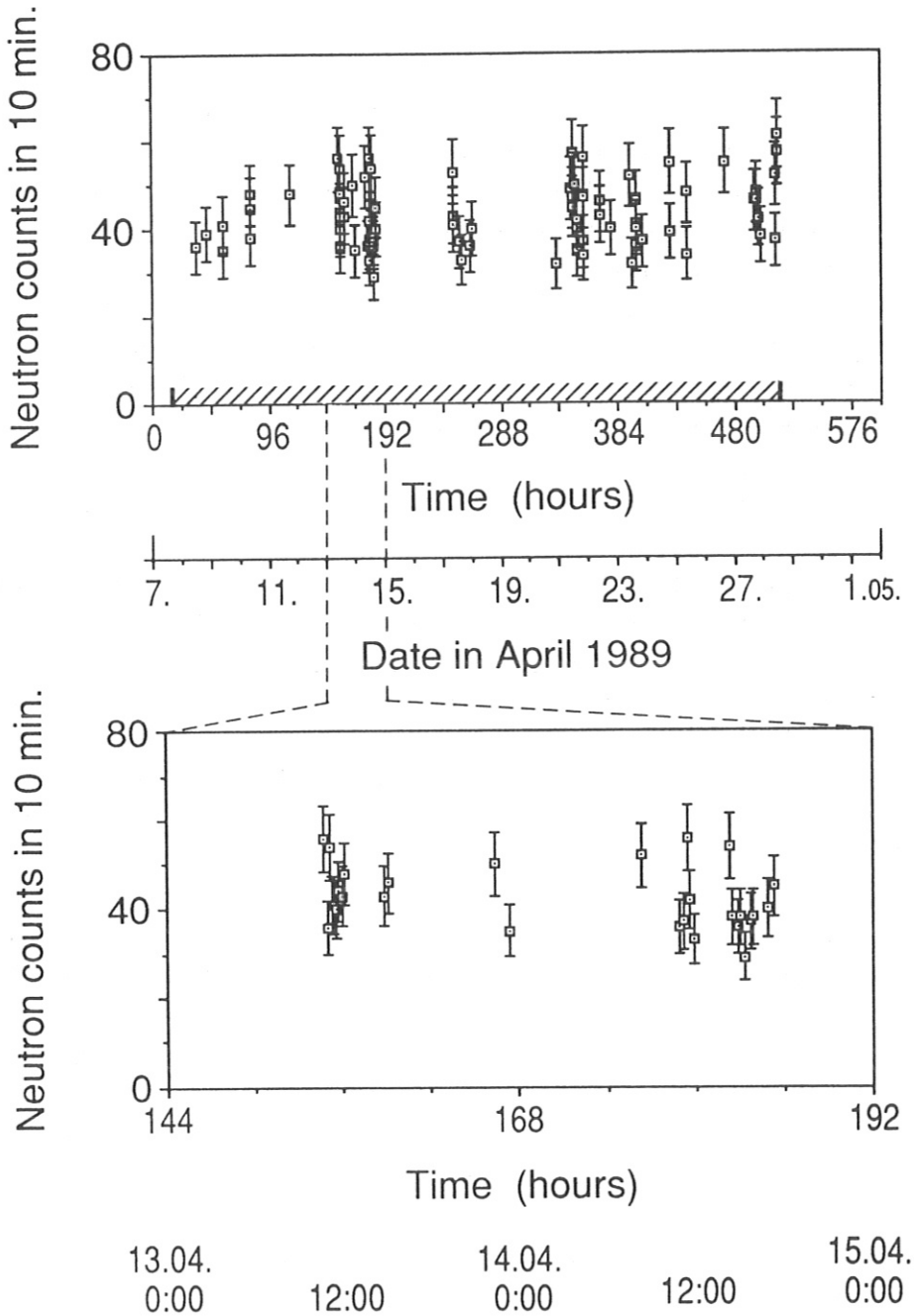


Figure 10: *Neutron background rate during experiment #3. The time scale starts at April 7 00:00 with 0 hours. The data are taken in 10 minute intervals. The average count rate of 4.2 ± 0.7 counts per minute corresponds to a neutron source rate (if from the test cell) of 26 ± 4 neutrons per second. The lower part of the curve shows a 2 day period with a larger time scale to demonstrate the background fluctuations. Cross hatch denotes duration of #3 cell operation.*

The experiment #3 was started April 7 at 17:12 which is 17.2 hours on the time-axis and it was stopped April 28 at 15:50 (519 hours). Large fluctuations are seen in the neutron background counting statistics, but there is no systematic change in the count rate observable over the course of the experiment.

Figure 11 shows neutron levels from evaluation of the BF_3 pulse height spectra. Although we do not have an absolute calibration for this figure, it is quite obvious that

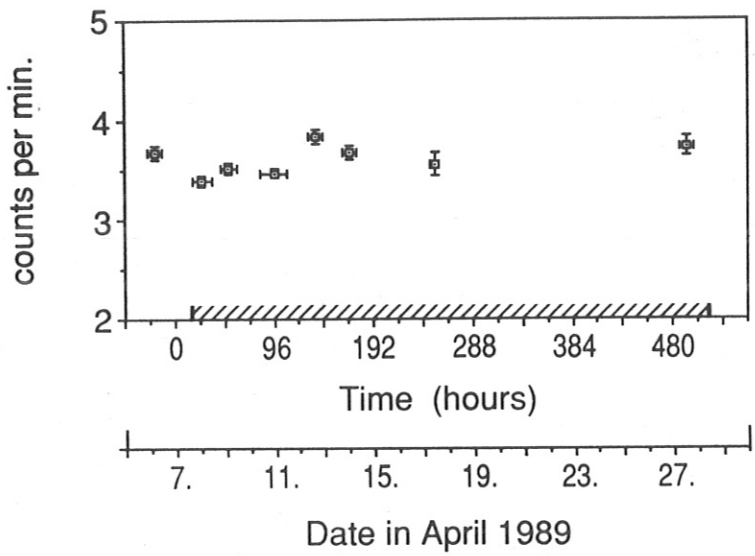


Figure 11: *Neutron background evaluated from the BF_3-spectra. The sampling durations vary between 4 and 26 hours, indicated by the error bars.*

due to the long sampling times of the spectra the background fluctuations are reduced. The average rate during the experiment #3 is 3.6 $\pm$ 0.15 cpm which is a standard deviation of only 4.2 % instead of 16.6 % for the 10 minute counter measurements. The spectrum taken before experiment #3 has a mean count rate of 3.75 cpm thereby showing that the neutron rate during the experiment did not only show no change over time but also its absolute value was not larger than before switching on the electrolytic cell.

In summary, if we use our calibration factor for the BF_3 neutron counter (as if these neutrons would come from the test cell) its count rate corresponds to a neutron source rate of 26 $\pm$ 2.3 neutrons per second, for a 30-minute counting interval. A neutron signal at a level of 3 σ above background, would correspond to 7 neutrons per second. We therefore can conclude that the neutron rate from our electrolytic cell was smaller than 0.3 neutrons per second per gram of palladium, or equivalently, $< 1 \times 10^{-22}$ (ddn) fusions/dd pair/sec.

5 Gamma-Measurements

In the course of these experiments we used an NaI crystal detector as well as a Ge(Li) detector borrowed from the accelerator lab of the TU Munich. Pair-production photons were also monitored, in case any high energy (24 MeV) photons might be produced in the cell...although none were seen. The principal intention was to check the γ-line at 2.224 MeV from neutron capture in Hydrogen [n(p,d)γ], which is the way F&P claimed to prove the existence of fusion neutrons [1]. Additionally we used the radiation monitoring system to monitor the total γ-dose in the lab.

5.1 NaI-scintillator

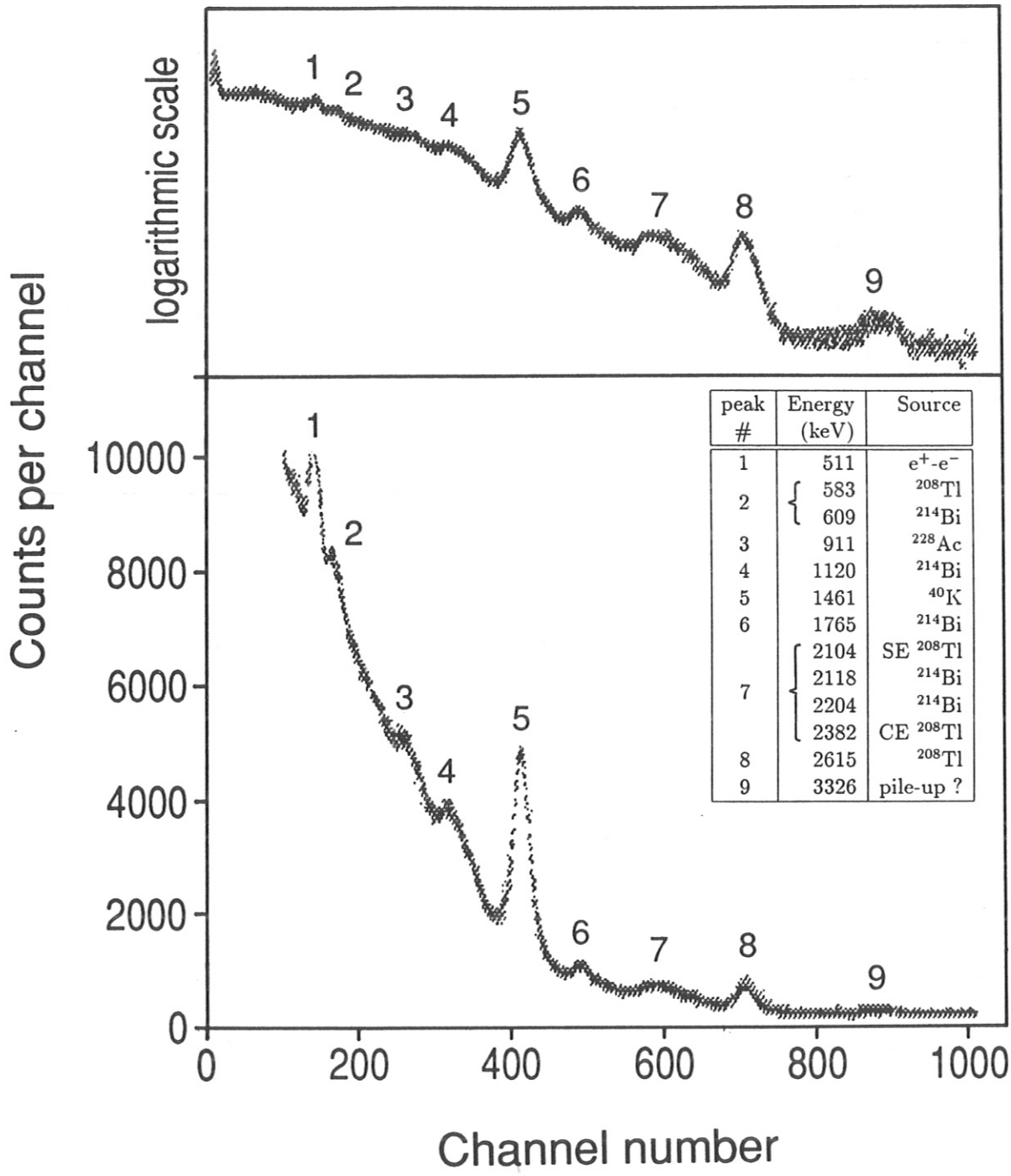
We started with a 2" $\times$ 2" NaI(Tl) scintillator to measure the background spectrum which looked exactly like a typical concrete dominated background spectrum, as published (for example) by Knoll [21] (page 795). The spectrum shown in Fig. 12 clearly shows the dominant contributions from ^{40}K at 1461 keV (line 5), ^{208}Tl at 2615 keV (line 8), and different Bismuth lines (mainly from ^{214}Bi, line 6&7) in combination with the Compton edge from the ^{208}Tl line (line 7). ^{208}Tl and ^{212}Bi are parts of the thorium decay chain, ^{214}Bi is from the uranium decay chain, while ^{40}K is a natural radioactive isotope (natural abundance in potassium is 0.012 %, $\tau_{1/2} = 1.26 \times 10^9$ years). The feature labeled "9" is an artifact of the NaI and its electronics, as it was not seen by the better Ge(Li) detector, and is most likely due to summation peaks from coincident, lower energy counts.

To check eventual γ-emission from neutron capture at 2224 keV, a ratemeter output (30 second time constant) of the NaI output in a window from 1.75–2.5 MeV was hooked to a strip chart recorder. It showed, however, only background variations and was discontinued later. We then used 10 minute counting intervalls (read manually) or collected spectra for long times (up to 72 hours).

We also looked to energies up to 14 MeV, to be certain we might not be missing something above 2.8 MeV, but we saw nothing of interest. A lead shield was later added to the NaI detector, to enhance e^+-e^- pair production, should any 24 MeV photons just happen to be generated by our experiment. No signals above the backgrounds were seen here either.

5.2 Ge(Li)-spectrometer

After we recognized that ^{214}Bi emits γ-rays at 2118.5, 2204.1, 2293.4, and 2448 keV (and at other energies) [23], we decided to look for a detector with much higher energy resolution to isolate the neutron capture line (2224 keV) from the interferring backgrounds. Mr. Thies and his collegues at the accelerator laboratory (of the Technical University of Munich) kindly provided a lithium-drifted germanium detector. This was a 61 mm diameter, 61 mm long germanium crystal from Princeton Gamma-Tech (Mod. Nr. LGTC 34, Ser. Nr. LD-421), operated at a bias voltage of +4000 V with a Canberra 2021 spec-



peak #	Energy (keV)	Source
1	511	e^+-e^-
2	583	^{208}Tl
	609	^{214}Bi
3	911	^{228}Ac
4	1120	^{214}Bi
5	1461	^{40}K
6	1765	^{214}Bi
7	2104	SE ^{208}Tl
	2118	^{214}Bi
	2204	^{214}Bi
	2382	CE ^{208}Tl
8	2615	^{208}Tl
9	3326	pile-up ?

Figure 12: *γ-background spectrum measured with a 2" × 2" NaI-scintillator for 96.4 hrs after #3 (May 4–8). Identification table is made by using a high resolution Ge(Li) detector.*

troscopy amplifier. The energy calibration was performed with uranium salts available in the chemistry lab.

In Fig. 13a the measurements of the γ-background with the NaI- and the Ge(Li)-detector are compared. The line at 2104 keV is the single escape peak of the ^{208}Tl line at 2615 keV. This shows dramatically that the spectrum measured with the NaI detector is dominated by unresolved background lines, easily resolved with the higher resolution (2 keV instead of 90 keV at 1.46 MeV) Ge(Li) detector.

Fig. 13b shows spectra from a ^{226}RaBe neutron source. Since ^{226}Ra is a member of the uranium decay chain, this source also contains ^{214}Bi. The single escape peak of the ^{208}Tl line is not visible since this spectrum has been collected for only 30 minutes. The lower curve shows the pure γ-spectrum of the source, while for the upper one a 9 cm thick water tank had been put between source and detector to generate the 2224 keV line from neutron capture in hydrogen. This figure shows clearly that this weak line is too close to the Bi-lines to ever hope to separate it with an NaI-detector.

If one wanted to measure the neutron capture line on top of the broad Bi-spectrum, one would have to reduce this background (namely the concrete surrounding the experiment), but even then the sensitivity (for detecting neutrons) would be poor in comparision to standard neutron diagnostics.

6 Radiation Safety Monitoring System

In addition to the above described detectors, we used an absolutely calibrated radiation monitoring system from Berthold (LB1026-2) which measures separately the neutron and the γ-dose. It prints out every 24 hours, if ever a pre-set threshhold was exceeded (this was useful if anything happened while we weren't in the laboratory!).

Its neutron detector is a ^{6}Li-I detector in a Bonner sphere, and was located in an adjacent fume hood, about 1.5 m from our electrolysis cell. Gammas were measured by a wire proportional counter, placed directly next to the electrolysis cell, about 15 cm away from the Pd piece.

Figure 14 shows the neutron dose (upper curve) as well as the γ-dose (lower curve) in μSv per day. Both radiation doses show no significant change over the period the experiment #3 was operated (April 7 to April 28), exept on April 26 and 27. On those two days we used a 2 mCi ^{226}RaBe source in the lab to calibrate our other detectors. The γ-dose for these days was 99 and 161 μSv respectively.

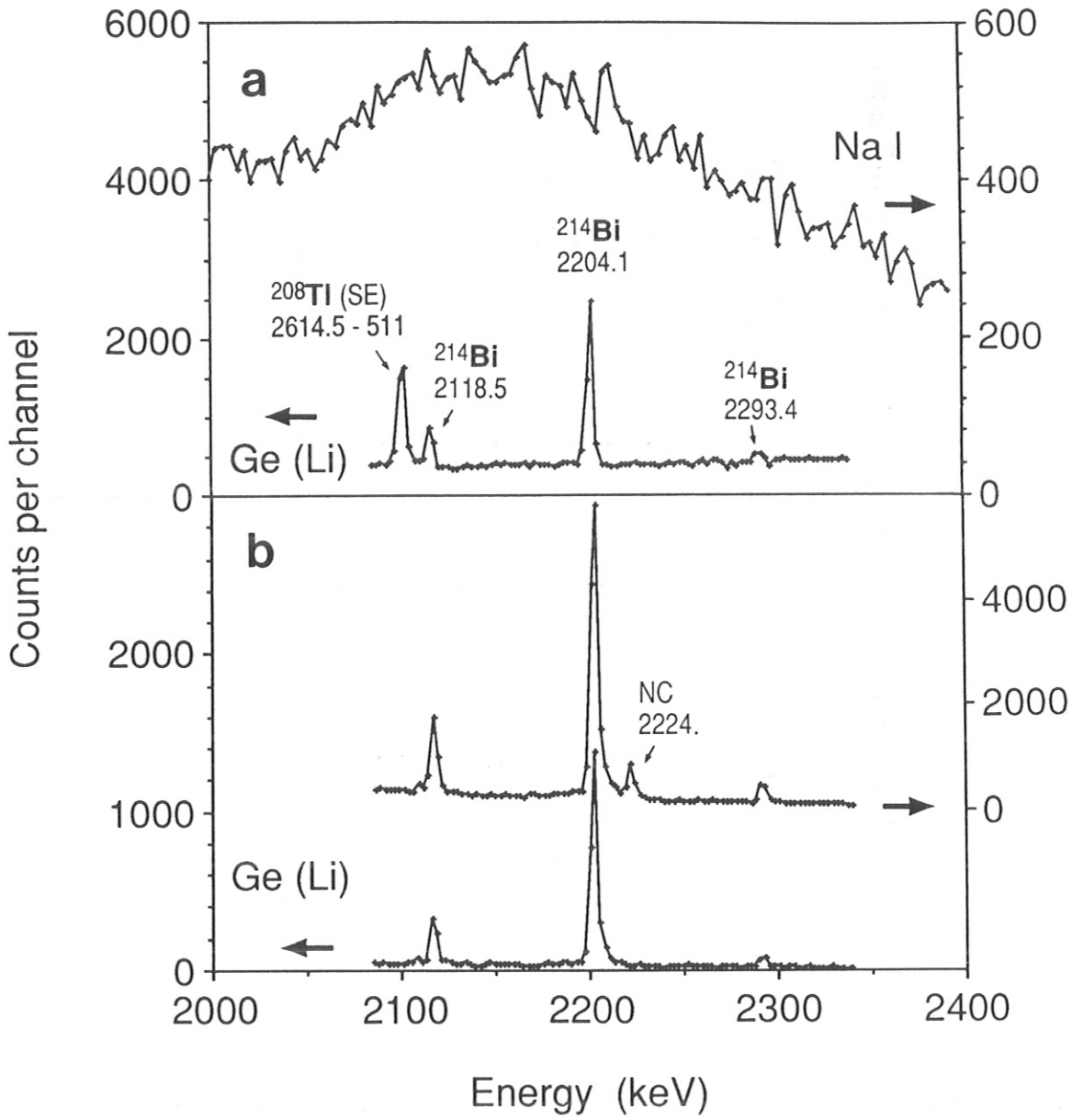


Figure 13: a) *Comparison of the γ-background spectrum from 2.0 – 2.4 MeV measured with a NaI-scintillator (upper curve) and with a Ge(Li)-detector (lower curve).*
b) *γ spectra of a 2 mCi ^{226}RaBe neutron source, collected for 30 minutes. The lower curve shows the pure γ-spectrum of the source, while for the upper one a 9 cm thick water tank had been put between source and detector to generate the 2224 keV line from neutron capture in Hydrogen.*

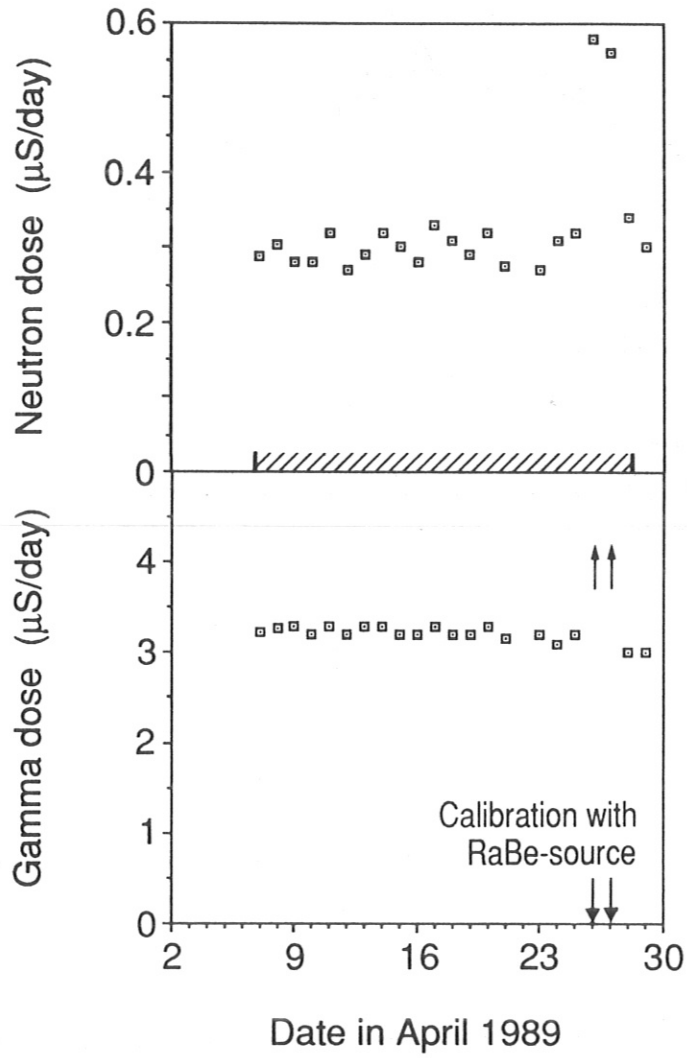


Figure 14: *Absolutely measured radiation doses in the chemistry lab. The electrolysis experiment #3 was operated from April 7 to April 28 and there is no significant change during this time. There was, however, an increased radiation level on April 26 and 27 when we used a 2 mCi $^{226}RaBe$ source to calibrate our other detectors.*

7 Calorimetry Measurements

Calorimetry measurements form the heart of Fleischmann and Pon's experimental claims. Without the measurements of "excess heat" which are claimed to be nuclear, and not chemical in origin, the entire world would never have paid attention to their press conference[3]. Truly careful microcalorimetry was beyond the level of sophistication of our initial experiments. Nevertheless, we recognized we could do an initial job at calorimetry with a large piece of palladium, detecting heat at levels above 100 mW, to easily see amounts claimed from the larger Pd pieces reported at Utah. In particular, we wanted to verify any correlated neutron activity (should we actually see any neutrons). We also knew that a careful accounting of our input powers would be necessary to determine if in fact we actually had "excess heat", and to differentiate against chemical sources, if the need arose.

However, our calorimetry effort from the start was a second order effort, and we believe only marginally better than that of F&P. In particular, we had continuous temperature readings and good mixing at the location (in the water bath) where our temperature measurements were made. But like F&P, our cell was not closed or divided, nor was the energy content and identity of the escaping gases monitored, either which is required for accurate accounting of the energy balance.

7.1 Current-voltage behaviour

Current levels were largely determined by deciding what was an acceptable stable operating temperature...we did not want to evaporate large amounts of electrolyte, in our rather "open" first system. We used a current stabilized DC power supply in each experiment. Whenever the current was increased, the voltage would, after first increasing, then fall to a new (higher) level, over a period of time. This effect is at least partly due to increased mobility of the charge carriers in the solution as the temperature rises. Conversely, simple addition of cold water to the water bath was observed to transiently cause an increase in the cell voltage, for the same current. In a similar fashion, refuelling the center cell with 5 to 10 ml of D_2O at a time, would temporarily raise the cell voltage.

Determining the input power into the cell, requires first monitoring the I-V characteristics of the cell. The nonlinear, equilibrium, I-V curve for our large Pd stone (#3) experiment, is shown in Figure 15. We plot the measured differential voltage between the palladium and platinum leads, and the total current. Note that the cell itself *is* in fact a small battery, with a cell voltage (measured at 1 mA current) of 2.0 volts. We will later take the assumed Joule heating to be $I*(U-1.54\mathrm{V})$, as also *assumed* by F&P, where the 1.54 volts represents work performed by electrolytic dissociation of the water, as well as other losses due to surface bubble conditions, and contact potentials. In truth, the assumed constancy of the 1.54 volts *should* be directly measured[24], but we *did not* do it in our experiments. This could be a source of error, especially when making comparisons to different equilibrium currents (and hence rates of bubbling).

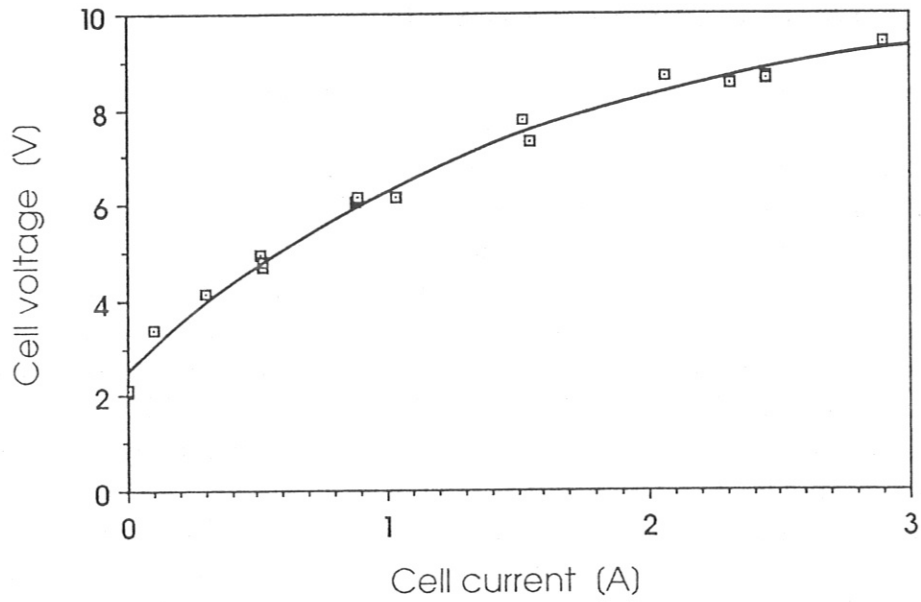


Figure 15: *Equilibrium cell impedance is a function of the electrolyte temperature, bubbling rates, and the applied power. U-I curve for the #3 cell shows the characteristic decrease in resistivity for higher cell currents (temperatures), as the ion mobility increases.*

7.2 Temperature differentials

The principal results of our calorimetry efforts, other than generating rolls of straight lines on the multipen strip chart recorder, are shown in two parts. Data from before (Figure 16a) and after modifications to the waterbath, (Fig. 16b) taken during the long duration #3 experiment, are plotted against the calculated Joule input power to the cell. In both cases, we plot the temperature rise of the approximately 750 ml stirred water bath, over and above the ambient air temperature of constant flow cooling air being sucked into the fume hood, versus the assumed Joule heating.

The first plot in Figure 16a shows a substantial offset temperature for low currents. This effect turned out to be due to a second source of heat to the cell, coming from the motor of the magnetic stirrer used to stir the water bath! It represents the effects of 1.6 W of heating power. The palladium in this experiment has a volume of 1.8 cm^3, so the reader gets a good comparison to the much larger expected magnitude of the reported 20 W/cm^3 excess heating of F&P, should it be present! When the stirrer was turned off, this temperature differential went to zero, as suspected.

Consequently, we improved the water bath geometry (with the cell continuing to run!) at 315 hours into the experiment, by adding insulation and improving the waterbath circulation. A schematic of the cell, and issues affecting heat flow is shown in Figure 17. We found by accident, (as one should in retrospect, expect), that simply leaving the hood in an "up" position, decreased the cooling by an equivalent of 1.7 watts. Consequently

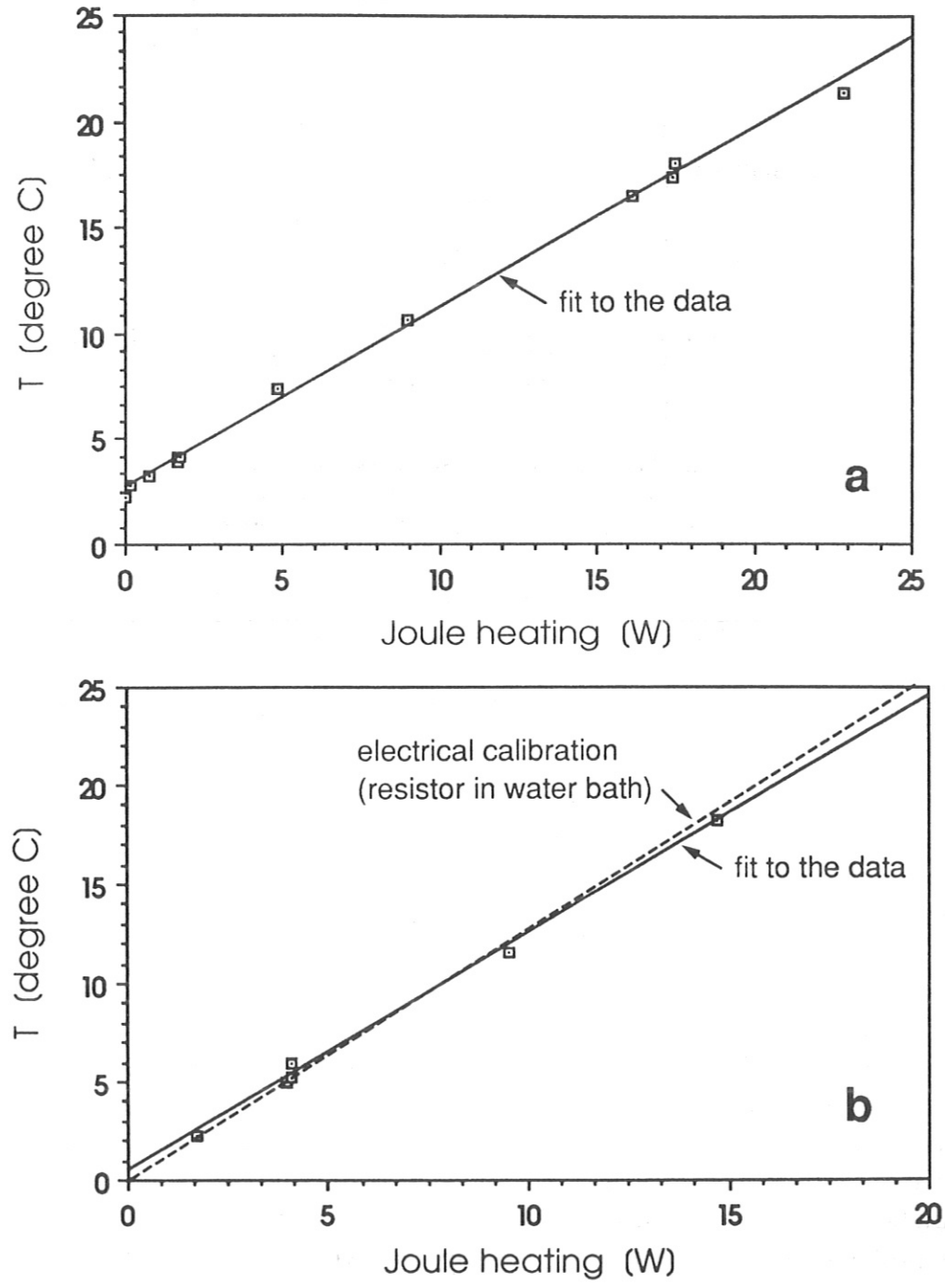


Figure 16: *(a) Temperature differential of water bath relative to cooling air, as a function of cell Joule heating, obtained by scanning the cell current, but always waiting at least three hours for new equibrium in the water bath.(b)Temperature response in #3 palladium stone experiment, after bath modifications. A strip heater resistor in the waterbath allows direct electrical equivalent comparisons, while the cell is in operation.*

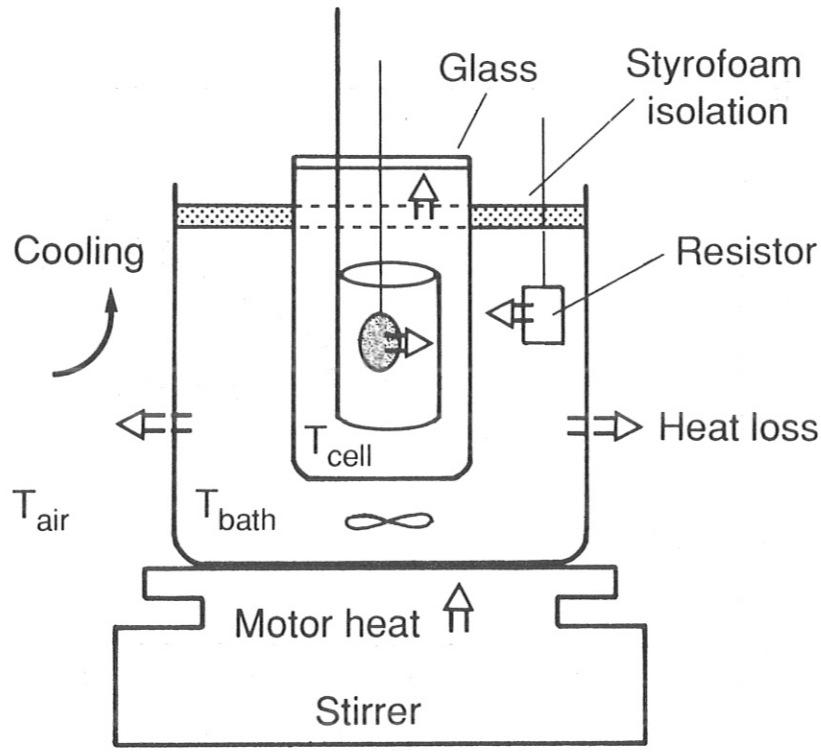


Figure 17: *Schematic of final configuration for heat flow in the #3 experiment.*

we were careful to make measurements in our so-called "hood down" position, which also gave us a more well-defined air flow. Effects from the stirrer motor were reduced to 300 mW power, absolute. The new temperature vs. power line is shown in Figure 16b, including also a strictly Ohmic heating calibration set taken by running a small current through a Kapton encapsulated 40 Ohm resistor placed in the well-stirred water bath. These calibration points were taken simultaneously with the cell operating, at three different equilibrium waterbath temperatures, between 29 and 49 °C. We do not fully understand why the electrical calibration has a larger slope than the curve produced by running the cell at different currents (temperatures). We may be seeing the effects of enhanced evaporative losses from the cell, as less of the cell heat is efficiently transferred to the water bath, and a larger fraction is lost in the saturated vapors escaping from the cell at higher temperatures and/or bubbling rates. This effect was in fact mentioned as a possible problem by A. E. Pontau from Sandia National Labs, at the Santa Fe Workshop, May 24, 1989.

In summary, our calorimetry, which in and of itself is filled with difficulties, nevertheless would have allowed us to easily observe effects an order of magnitude smaller than reported by F&P. At the 5% level, we saw no excess heating. Furthermore, we looked for transient effects resulting from rapid decreases in cell current, and after waiting for our waterbath equilibrium (In the #3 setup, time constant $\sim$ 100 minutes), could not see any unusual effects. In our thin sheet experiment (#2), thermal conditions were in equilibrium after four hours, and remained identical for the remaining forty odd hours of the experiment. Fast transients due to loading (or unloading) were most easily seen in the

first thin-walled tube experiment (#1), due to the short three-minute cell thermal time constant in the center cell itself, but it was precisely these types of measurements which made us appreciate the difficulties with large thermal gradients in the "gas sparged" center cell.

8 Tritium Measurements

As one part of their "proof" of nuclear reactions occurring in their electrochemical experiment, Fleischmann and Pons offered the measurement of tritium in the electrolyte as a sure sign of the fusion of deuterium. Unfortunately their paper[1] *does not* describe the levels of tritium found in the D_2O used before the experiment even began!

What we found, as have others [10], is that the tritium in the D_2O differs from bottle to bottle by as much as two orders of magnitude, as supplied by Merck, irregardless of the effects of further concentration/separation by several weeks of electrolysis. The tritium level (100 decays/minute/milliliter) found by F&P easily lies in the middle of the range we detected in our 99.75% D_2O, Merck Article # 2919 UVASOL flasks.

We used a model 2260XL Tri-Carb Liquid Scintilation Analyzer by Canberra-Packard to measure 6 ml samples of our stock D_2O flasks, samples with LiD (pH $\sim$ 12, unneutralized before making up the cocktail gel) in both D_2O and H_2O, and the actual electrolyte after the first, and during and after the third runs. An efficiency factor of 30% was measured from a tritium standard, and used to convert counts/minute (cpm) into decays/minute (dpm). The measurements were kindly performed on the new PS/2 controlled analyzer, in the Nuclear Chemistry group of the Reactor at the Technical University. Spectra from the instrument indicated that chemical fluorescence was not a significant problem, and had no bearing on the measurements. The first measurements were performed 19 days into the third (and final) palladium experiment. We had also saved samples from earlier runs, and also the stock D_2O bottles. Unfortunately, prior to this, we had not located a suitable tritium measurement instrument that was accessible for our purposes.

We learned from these measurements, that premixing of *all* D_2O to be used in a run, would be absolutely essential for an accurate determination of any alterations in the prexisting tritium levels. However, we did not do that, and also initially didn't have a feeling for how much (expensive) heavy water we would be "using" up over the course of the experiment. Consequently, as we used solutions made up from three 100 ml Merck flasks (one of which was 100% used up before a tritium measurement was made), and one 250 ml Merck flask, we are unable to precisely calculate the various dilutions which occurred.

The results of the tritium measurements are shown in Table 4.

Table 4: Tritium levels in IPP experiments

Sample #	(dpm/ml)	Description
1	6.6	H_2O+LiD
2	9.9	99.75% D_2O 250ml Merck flask
3	210	99.75% D_2O 100ml Merck flask
4	750	D_2O+LiD After #1 Exp. (14 hours)
5	150	D_2O+LiD During #3 Exp. (19 days)

They indicate, that levels ranging from 10 dpm/ml to 750 dpm/ml, were found in the bottles of high purity heavy water from Merck, even though they were bought at the same time (about 15 years ago), and have the same label numbers on the bottles! We also worried about tritium being added through the lithium deuteride that we used as the "salt", but this was measured by testing a blank mix of LiD in H_2O, at 7 dpm/ml, and therefore was not a problem. Another concern was that one of our hot neutron calibrations could have actually bred some tritium in the lithium. A quick worst case estimate from the 12 Curie Plutonium source used in this example, showed this would have only a trival effect from the less than hour long exposure. Due to heavy water replenishment of the Pd Stone (#3) experiment mostly from the 4^{th} and largest bottle of D_2O, which also had a low tritium level, the final solution in the third experiment had a lower concentration of tritium when it ended, than when it began.

We conclude that all of the tritium levels which we saw in our experiments, could be accounted for, simply, from the tritium initially present in our heavy water. While changes in the concentration undoubtedly occur due to distillation and separation by the Pd metal as it loads, we don't expect more than factors of 3-9x here[25]. Furthermore, our results span those of Fleischmann and Pons (from a factor of 10 lower, to a factor of 10 higher), and make us concerned whether F&P also did not premix their heavy water (since the variability of tritium in heavy water is so high), as well as what the absolute levels of the tritium in their heavy water actually were before they began their published experiments. As an erratum[1], F&P have finally stated that their tritium level went from 41 dpm/ml to 141 dpm/ml, in their electrolyte. This is well within the range of expected isotopic separation effects!

Some critics of F& P have indicated that chemical fluorescence (due to poor neutralization of the strong base, and subsequent chemical reactions in the "cocktail gel") accounts for spurious tritium measurements. We do not support this point of view in our equipment, and indeed, did not even need to neutralize the sample electrolyte at all prior to mixing the "cocktail". Fluorescence was accounted for in our measurements, and made an effect only at the 5% level.

9 Conclusions

9.1 Our experiments

We have performed integrated experiments in three electrolytic cells; two test experiments and one "final" long run-time (for 3 weeks) setup. During this experiment the cathode was a 22 gram vacuum-cast "stone" of palladium. After the run the Pd was loaded with D_2 in excess of 0.8 D:Pd, probably even in the range of 0.9 to 1.2, as determined by mass uptake. Absorption spectroscopy of catalytically regenerated water from the outgassing Pd sample weeks after the electrolytic loading, confirmed the presence of large amounts of deuterium. Forced, rapid changes in cell current, after long periods of equilibrium, yielded nothing unusual.

During the runs we didn't observe any neutron emission above the background, which for our setup means an upper limit on the neutron emission from the electrolysis cell of 1×10^{-22} fusion reactions/dd pair/second. The gammas from neutron capture were also monitored, first with a NaI-scintillator, later with a Ge(Li)-detector which is able to separate the neutron capture gammas from the natural background, but we did not see a signal attributable to the cell there either.

Our calorimetry was simple, but trustworthy. We should have been able to see excess heat production an order of magnitude smaller than reported by F&P, if present, but *did not.*

Our measurements of the tritium content in heavy water demonstrated that there are large variations between different source bottles, even those bought at the same time and from the same vendor. The tritium content measured in our experiments can easily be explained in this fashion, or with well-known isotopic separation effects.

We did not attempt to measure helium isotopes in our palladium samples, although we discussed the need for extremely high resolution mass spectrometry to resolve among the various mass combinations of H, D, and T, against possible He isotopes.

By virtue of our quick start in this affair, we recognized at an early date (in comparison to others who came later with sometimes more precise measurements), that extensive and expensive efforts would be fruitless. It is our opinion, based on our own experiments, other papers cited here, and visits to other laboratories involved, that no fusion processes are required to explain the results of Fleischmann and Pons.

9.2 Probable errors in the F&P experiment

After 4 weeks of experiments, the three described above, and additional mock-up normal water cells, as well as a heavy water cell used for fast loading/unloading experiments, we have concluded that the work of Fleischmann and Pons was unfortunately hastely written and not up to normal scientific standards. The lack of controls was particularly conspicuous.

We have identified weak points in our own experiments, and based on what we know

of the F&P experiment, likely errors in the F&P experiment. We have also pointed out differences, and open questions.

- The thinking and design of the F&P calorimetry is not carefully laid out in their publication[1]. Supposedly it was to follow in their larger, more detailed article (later withdrawn from Nature[26]). Our own calorimetry was easily capable of seeing the large (~30%) effects claimed by F&P. We did not see any such effects in our experiments. Issues of evaporative losses (dependent on the operating temperature), unknown products being lost in the open system (through the exhaust gases), variable effects of bubbling at different current levels, uncontrolled catalytic recombination, all remain for researchers with finer calorimetric equipment than ours.

- Arguments by Pons at the time of the Los Angeles ACS meeting, and his showing of a video that a tracer dye was well-mixed in their tube after 20 seconds, by action of the bubbling, *in no way* counters the fact that stable convective cells would allow the existence of significant thermal gradients in the tube, while still having eventual mechanical mixing, so long as the heat source is generated at one place in the tube (as was the case).

- Our doubts about the F&P gamma and neutron measurements, and their internal inconsistencies, have now been dealt with by a number of papers, and in particular, fully confirmed by the work of Petrasso, et al[27]. In a so-called "rebuttal" of the Petrasso work, by Fleischmann, Pons, Hawkins, and R. J. Hoffman[28], in *Nature*, F&P have only shown[29] that they never did a simple energy calibration of their gamma spectrum, nor looked up such a spectrum in numerous books on the topic. They have demonstrated a complete lack of credibility by shifting the energy of their so-called signal line by +300 keV (without explanation), and at the same time showing background lines where none are known to exist!!

- While we do not doubt that F&P indeed measured tritium at the 100 dpm/ml level, it is not clear that this tritium was actually *produced* in their experiments. Instead, we believe it to have been originally present in their feed D_2O, and weakly enhanced by well-known isotopic separation processes.

- The Pd/Pt/D/Li system can make a good "battery". Any discussion of *bursts* of excess energy from these cells, had better have extremely careful accounting of all energy balances, in order to discriminate against chemical origins.

  Based on the suprisingly careless work that has been so-far presented from this University of Utah group, we halted our own efforts in this endevour (in spite of having further cells "ready to go"). We have not seen any "believable proof" from F&P that the excess heating is nuclear in origin. This ideally means time-dependent data showing the relationship between the supposed "excess heating" and any nuclear products.

We thank everyone who helped without asking for an account number, and all the people who hoped it could be true. In particular: the initial gamma calibrations by Dr. Guenter Janeschitz using the ASDEX spectroscopy sources; the late night checks by Herr Spitzer; the cell manufacturing and lead shield machining in the ASDEX shop; the friendly loan of an expensive high efficiency Ge(Li) detector by Mr. Thies in the Technical University of Munich (TUM for short) accelerator group (thanks to Frau Dr. Schneider's connections); the tritium measurements by Frau Dr. Kim in the reactor group; the loan of calorimetry aids and advice by electrochemist Prof. F. Mayinger at the TUM; the raster electron microscope scans and x-ray impurity analysis of a piece of our #2 cell palladium by the Applied Electrochemistry Group of Prof. Dr. D. W. Wabner (also at the TUM); the scanning electron microscope photos of the Pd stone by the Max Planck Institute of Quantum Optics (Garching); and finally help from many interested on-lookers and kibitzers.

A Faxes, rumors, and papers: When, What and How we knew

In the fast-breaking world of press reports, computer networks, and publication by FAX, staying on top of the scanty developments following the initial F&P news conference, was a crucial part of our initial efforts. This effect was magnified by the paucity of details coming from the two Utah principals.

March 24: Some of us see an American video clip on Bavarian (Bayerische Rundfunk) TV news.

March 25: Short news clip on Armed Forces Radio.

March 26: Telephone calls from/to US. One of us (GAW) received a phone call from the USA, from his brother stating "Ok, I have my palladium, platinum, and heavy water....so what do I do next to make fusion?"

March 27: Computer network mail messages sent to associates in US, asking if they know anything about "this cold fusion stuff". Still an Easter Holiday in Germany.

March 28: First newspaper articles with some details. Our first library search turns up a wonderful handbook[4], 430 pages thick, entitled "Palladium", published by Verlag Chemie, edited by the Deutsche Chemische Gesellschaft in 1942, which as we continue to discover, is full of information useful in the coming weeks. We also obtain articles on various calorimeter configurations typically used by physical chemists.

March 29: We didn't fully realize why the cell was tightly closed, and had arguments about whether the cell might be pressurized (in order to aid the $\alpha - \beta$ crystal phase transition without boiling away the heavy water at temperatures of 150°C). We then realized that the electrochemical pressure itself easily drives this phase transition in the palladium cathode, and also that a pressurized glass vessel was unlikely for safety reasons.

March 30: Our first cell is switched on. We make a guess to use LiD as our salt. Low grade (95%) heavy water is used in our first trial.

March 31: Background measurements, following the shutdown of our #1 experimental cell. Fleischmann gives seminar at CERN.

An improved cell is "in the works". During the weekend of April 1-2, 1989, we continued to make background measurements, and collect summaries of news accounts, computer bulletin board rumors & facts, phone discussions with scientists in the US and England, generally trying to glean new information about the F&P experiments. Fleischmann had been at Harwell and at CERN, so a few more details were apparent.

April 2: Still no Fax copy of the rumored F&P paper, or the "other Utah" experiment of Jones... whom we knew to have been involved in previous muonic fusion measurements at Los Alamos, although some pretty good summaries of the contents of the papers were on the computer network.

April 3: We have a copy of the Jones preprint, but have no desire to use the so-called "Jones witches brew" of salts in the cell. Phone discussions with D. Robinson at Harwell.

April 4: Our #2 cell goes into operation, after our first receipt of the F&P paper on Monday evening April 3. Our copy came from Princeton Plasma Physics Lab, via an exchange scientist's suitcase (hence it suffered one less FAXing than many others here in Europe)! After reading it, we could confirm that the computer network news of the previous three days, which contained summaries of the paper, were reasonably accurate.

April 7: Our #3 cell goes into operation.

April 11: Newspaper accounts of Texas A&M claiming excess heat production. First rumors of "non-reproducibility" begin to surface from Harwell's discussions with Fleischmann.

April 14: We hear that it is essential to have a cast Pd piece, not drawn or extruded. By luck, this is exactly what we are running. Georgia Tech experimenters withdraw their erroneous neutron claims.

April 17: Pons press conference, claiming He^4 detection at U Utah... We obtain the Paneth & Peters 1926 paper. Our amazement that someone at Los Alamos had dug up an old German paper, was caused by a request from the editor of Nuclear Fusion to get a good copy of it out of our library. This short-lived report was on the transformation of hydrogen to helium in 1926, *with palladium*. It still causes us to wonder (in truth) whether if F&P ever knew about it, in relation to their own studies...

April 18: We obtain the retraction of the 1926 paper, and information from Sweden concering Prof. Tandberg, of the ElectroLux Laboratory, and his "fusion" experiments in 1928-1930 with electrolytic-loading of palladium in heavy water.

April 19: Pons gives seminar at Los Alamos. Los Alamos wants to have a collaboration to verify the "active" Utah cells, either by hauling cells to LANL, or sending people with measuring equipment to U of Utah.

April 28: We receive the palladium that we ordered at the beginning of our experiments. On the same day, we shut off our long-running #3 experiment.

May 3: APS Cold Fusion Seminar, Baltimore....physicists attack cold fusion claims.

May 5: Raster scanning electron microscope elemental analysis of a piece of Pd from our #2 cell, by the Applied Electrochemistry Group (Dr. Wabner), shows over-

whelmingly the presence of palladium, followed by trace calcium, and minute amounts of copper, and iron. Tiny particles consisting of potassium, chlorine, and of platinum could also be found. The platinum-black evidently came from the anode in the undivided cell. Lithium could not be seen by this technique, as its x-ray energy is too low. However, one could see the lithium by putting the Pd piece in a Bunsen burner flame, and noting its characteristic color.

May 23-25: Workshop on Cold Fusion Phenomena, Santa Fe, New Mexico, sponsored by Los Alamos National Laboratory. We submit a long abstract[30], but are personally unable to attend. Computer summaries become available on the network, and we later obtain video tapes of the entire conference. F&P do not even submit a paper, and the previously touted LANL/Utah collaboration is still frustrated by lawyers. Over 120 papers are presented, the vast majority having "null" results.

May 25: Initial reports at Santa Fe Cold Fusion Workshop, from Texas A& M [31] of Tritium levels in their experiments 10^4 times higher than we have seen in ours, require close scrutiny into their stock D_2O feed supply, or any other sources of Tritium contamination. Also, as they know, should they ever repeat these measurements, they should try it on samples taken before the beginning of the experiment. Texas A& M shows interesting microcalorimetry, claiming to see "excess heat" in the realm of F&P.

We have visited several laboratories, on two continents, and had phone conversations twice directly with M. Fleischmann. Members of our team have had friendly visits with the "cold fusion experiments" at Harwell, Frascati, Karlsruhe, Texas A&M, BYU, and Los Alamos, but were denied access to Pon's lab in Utah. We were also at two meetings of Euratom, the first in Brussels, and the second in Harwell, on the topic of European cold fusion efforts.

B

Sante Fe Workshop on Cold Fusion Phenomena, May 23-25, 1989

Electrochemical "Cold Nuclear Fusion" Attempts at IPP

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Abstract

Following the report of Pons and Fleischmann, we (The Bavarian Bubble Bottle Team) have attempted to reproduce their claims of cold nuclear fusion, and failed. We note that our measurements would not be able to detect neutrons at the level of Jones *et al.* Three experiments were conducted without any signs of neutrons, tritium or gammas above backgrounds, and within ±5% accuracy calorimetry, no excess heating. Intrinsic tritium, differing from each D_2O bottle tested, was however observed.

The third, and most significant attempt used a 22 gram, 10 cm^2, cast (vacuum melted by an e-beam) palladium piece, which was electrolytically loaded with deuterium (99.75% purity D_2O, 0.11 M LiD, Pt mesh anode) at current densities of 200-250 mA/cm^2 for a period of 21 days. Current, voltage, water bath (well-stirred) and air temperature were monitored continuously with a strip chart recorder. The fully covered 170 ml central cell did not partition the electrolysis products, all calorimetry was done in steady state, (bath time constant 100 minutes), and air flow across the 1 liter water bath was kept constant. No isolation Dewar was used. Measurements of the actual temperature(s) directly in the center cell showed strong thermal gradients ($3-4°C$), so calorimetric measurements are only relevant for the well-stirred water bath/air temperatures (typically 45 and 26°C, respectively). An absolute resolution of better than 200 mW, out of 20 Watts typical input power, was obtained. The equilibrium temperature rise of the water bath was strictly linear with input power, calculated from $P_{in} = I * (V - 1.54v)$. A Kapton encapsulated 40 Ohm strip resistor was used in the water bath for reference ohmic input measurements. Fast reductions in current density, after waiting for a new thermal equilibrium, yielded nothing unusual. A BF_3 thermal neutron counter, (calibrated efficiency of 1 count/370 neutrons from the same location as the cell, backgrounds of 0.05 counts/sec, with 12 cm polyethylene moderator and a Cd shield), as well as a moderated Li^6I neutron detector, a large high-resolution, high efficiency (170 cm^3) GeLi gamma detector, a 2" NaI detector, and a proportional counter were used to look for radiation. Gamma backgrounds from K^{40}, Bi^{214} and Tl^{208} (Thorium decay in concrete), were easily seen in pulse height spectra. Backgrounds at our second floor location were 120 mrem/year gammas, and 10 mrem/year thermal neutrons, as measured by a Berthold LB1026 Radiation Monitoring system. No special shielding precautions against cosmic rays were used. A 4 Megawatt swimming pool nuclear reactor, 600 meters away from our building, was one of our additional background considerations! Tritium was measured in the electrolyte, D_2O samples, and H_2O (both with and without the LiD solute). A model 2260XL Tri-Carb Liquid Scintillation Analyzer by Canberra-Packard was used, with the old ($\geq$ 15 years) Merck heavy water yielding 210 dpm/ml from one vial, and 9.9 dpm/ml from another used for refilling. By contrast the LiD dissolved in pure H_2O (unneutralized) gave 6.6 dpm/ml. The cell was replenished with D_2O at a rate averaging 16 ml/day. The electrolyte after 19 days of operation measured 150 dpm/ml. In comparison, the D_2O from another bottle (with same Merck #, and purchased at the same time!) used in our first 14 hour experiment had 750 dpm/ml. Pulse height analysis suggests that true Tritium decay signals are present, and chemical fluorescence in the "cocktail" was not important (although measurable). Tritium can reasonably be explained from that originally present in the various D_2O flasks.

The experiment was terminated on April 28, by throwing the loaded palladium sample directly into liquid Nitrogen, immediately next to the bare BF_3 counter (backed by 25 cm of moderator), in order to attempt one of the Italian ENEA neutron production variants. No neutrons (sensitivity of 5 n/sec equivalent source strength) above backgrounds were seen, while counting for one hour, and also none while the piece warmed to room temperature over the next hour. Post mortem analysis of the darkened, hardened Pd piece showed large crystal grains (up to 2mm×2mm), and continuing evolution of gas bubbles at the grain boundaries even four days after the experiment was ended.

* Los Alamos National Laboratory, presently at ASDEX, supported by USDOE

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