

## COMMENTS

**Reply to “Examination of Claims of Miles et al. in Pons–Fleischmann-Type Cold Fusion Experiments”**

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**Introduction**

This paper is a response to S. E. Jones and L. D. Hansen,<sup>1</sup> who critically examined our claims of excess heat and helium-4 production during electrolysis of the Pd/D<sub>2</sub>O + LiOD system.<sup>2–5</sup> Many of the allegations regarding our work have been discussed in previous publications.<sup>2–6</sup> We have also critically examined the basic principles and problems in measurements of excess power during Pd/D<sub>2</sub>O + LiOD electrolysis in isoperibolic calorimeters.<sup>7</sup> The claim by S. E. Jones and co-workers<sup>8</sup> that faradaic efficiencies less than 100% can account for reports of excess heat in cold fusion cells is not valid at the high current densities ( $j \geq 100$  mA/cm<sup>2</sup>) required for these experiments.

**Excess Heat Production**

The calorimetric results reported by our laboratory have been used to support both sides of the scientific controversy regarding anomalous effects in deuterated metals. Our first set of experiments conducted over a 5-month period (April–September 1989) produced no significant evidence for any excess enthalpy production.<sup>9</sup> The mean value for the ratio of heat out to joule heat in was  $X = 1.00 \pm 0.04$  in our most accurate calorimetric study.<sup>9</sup> These early experiments at China Lake were listed in the Energy Research Advisory Board report to the U.S. Department of Energy as one of the groups *not* observing excess heat.<sup>10</sup> It is difficult to explain why our early calorimetric studies reporting no excess heat<sup>9,10</sup> are acceptable to critics of this field when later studies, that are actually more accurate, are judged to be in error.

Research groups from MIT, CalTech, and Harwell laboratories also reported no evidence for excess heat,<sup>7,10</sup> thus greatly impacting the general scientific opinion regarding this field in 1989. All three groups discontinued their experiments after only a few months of investigation. We continued to investigate other palladium samples and eventually observed significant evidence for excess enthalpy from the use of Johnson–Matthey palladium rods.<sup>2</sup> In retrospect, it would be impossible for any research group to adequately investigate the multitude of variables involved with this field in only a few months. These variables range from the palladium metallurgy to the D<sub>2</sub>O purity, the type of electrolyte and concentration, the electrochemical cell, the electrode arrangement, the type of calorimeter, proper scaling of the experiments, the handling of materials, the current

densities used, the duration of the experiments, the loading of deuterium into the palladium, the use of additives, and so on.

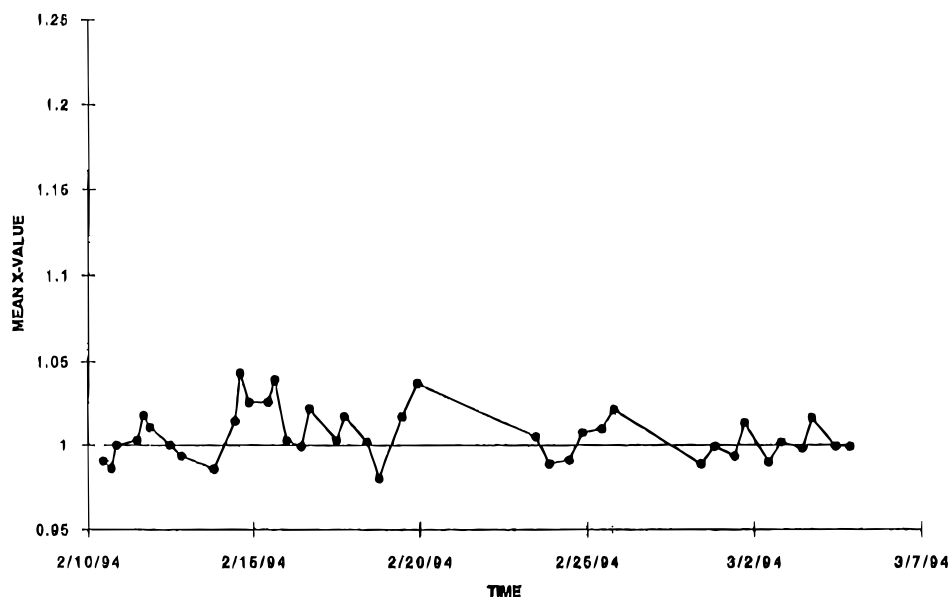
As should be expected, our calorimetry has improved with time. An early version had glass tubes containing the thermistors that protruded considerably above the tops of the cells.<sup>2</sup> Although the effect of these thermistor tubes was not apparent when the room temperature was stable, cooler weather later produced greater fluctuations in room temperature and unstable thermistor readings. This was especially apparent in a water control study (Figure 6 of ref 2). In the following experiments, the thermistor tubes were made flush with the cell top, resulting in much more uniform measurements. Although Jones and Hansen<sup>1</sup> focused considerable attention on Figure 6 of ref 2, they ignored our explanation and correction for this effect (see pp 245–246 of ref 2). A dramatic improvement in the calorimetric stability is seen in the experiment following the H<sub>2</sub>O control study (see Figure 7 of ref 2) where the single tail  $t$  test for excess enthalpy easily exceeds the 99.95% confidence level (see Table 2 of ref 2). Many of these issues were thoroughly discussed in a previous debate with S. E. Jones.<sup>11</sup>

The accuracy of our calorimetry is illustrated in Figure 1 which features one of many experiments that never displayed any evidence for excess power. The ratio,  $X$ , of output power to input power remains close to unity. All measurements of excess power were within  $0 \pm 40$  mW for the entire experiment. Approximately 70% of our experiments never displayed any evidence for excess power and served as controls for our calorimetry. In June of 1995, Roger M. Hart, an expert in the design, construction, and testing of calorimeters, examined our calorimetric design and agreed with our stated error range of  $\pm 20$  mW or  $\pm 1\%$  of the input power, whichever is larger.

A major criticism presented by Jones and Hansen<sup>1,11</sup> of our calorimetry is the variation of the calorimetric cell constants over various experiments. For example,  $K_1$  ranges from 0.135 to 0.141 W/°C over four separate experiments that yield a mean of  $0.138 \pm 0.003$  W/°C (see Table 3 of ref 2). Roger Hart pointed out that this criticism by Jones and Hansen is not valid since all cell components are repositioned in each experiment. The relative positions of the anode and cathode electrodes and of the two thermistors vary somewhat with each new cell assembly; thus, the slight variation in the calorimetric cell constants in different experiments is expected. The many experiments that produced no excess enthalpy, such as shown in Figure 1, indicate that the calorimetric cell constants do not change during an experiment.

Many experiments have proved that the recombination of D<sub>2</sub> and O<sub>2</sub> electrolysis gases does not occur to any significant level for typical cold fusion studies using high current densities and solid, fully submerged palladium cathodes.<sup>4,12</sup> Some scientists, however, prefer to ignore this evidence and continue to claim that the excess heat effect can be explained by faradaic efficiencies less than 100% ( $\gamma < 1$ ).<sup>8</sup> The recombination effects for Ni and Pd cathodes reported by Jones et al.<sup>8</sup> used current densities of only 1–2 mA/cm<sup>2</sup>. Such studies are irrelevant since excess heat effects for the Pd/D<sub>2</sub>O system require a threshold current density of about 100 mA/cm<sup>2</sup> or higher. This requirement of high current densities was reported by M. Fleischman

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**Figure 1.** Ratio,  $X$ , of the calorimetric output power and the electrochemical input power for a palladium sheet cathode. No significant excess power was observed.

et al.<sup>13</sup> in 1990. Lowering the current density in water electrolysis experiments will always decrease the current efficiency due to the slower gas evolution that allows the product at one electrode to more readily invade the vicinity and react at the opposing electrode. Furthermore, the current fraction consumed by the electrode reaction of impurities becomes larger at smaller current densities. Contrary to the comments by Jones and Hansen,<sup>1</sup> we always measured the current efficiency at the time of collection of an electrolysis gas sample for helium analysis. This was done volumetrically by measuring the rate of the displacement of water by the electrolysis gases.<sup>4</sup> For all the runs that appeared in the original table reproduced and criticized in Jones and Hansen<sup>1</sup> as Figure 1, the volume of gases evolved was as expected for no recombination.

Several other measurements and observations provided secondary checks for any recombination of  $D_2$  and  $O_2$  in our experiments. The volume of  $D_2O$  added to replenish the cell was always recorded to provide another test for any significant recombination effects. Furthermore, the rate of the electrolysis gases passing through the oil bubbler could always be directly observed. If recombination of  $D_2$  and  $O_2$  within the electrolysis cell occurs, this would slow or even stop the evolution of gases through the bubbler.

There is only one group of experiments where recombination was detected in our electrolysis experiments over a 6-year period. These studies all involved the codeposition method reported by Szpak et al.<sup>14</sup> where palladium metal is deposited from a  $D_2O$  solution containing 0.05 M  $PdCl_2$  and 0.3 M  $LiCl$  onto a copper cathode in the presence of evolving deuterium gas. This method reportedly produced excess enthalpy, tritium, and emanating radiation.<sup>14,15</sup> The deposition of palladium from  $D_2O$  solutions offers the possibility of generating a high-purity cathode material that is simultaneously loaded with deuterium. In our experiments, however, this palladium deposit was often dendritic in nature. Hence, the palladium became detached from the electrode, floated in solution, and adhered to the cell wall above the  $D_2O$  level. This finely divided palladium acted as an excellent catalyst for recombination in the gas phase; hence, these codeposition experiments sometimes resulted in loud explosions. There was also evidence that the dendritic palladium deposits occasionally contacted the anode, thus allowing some

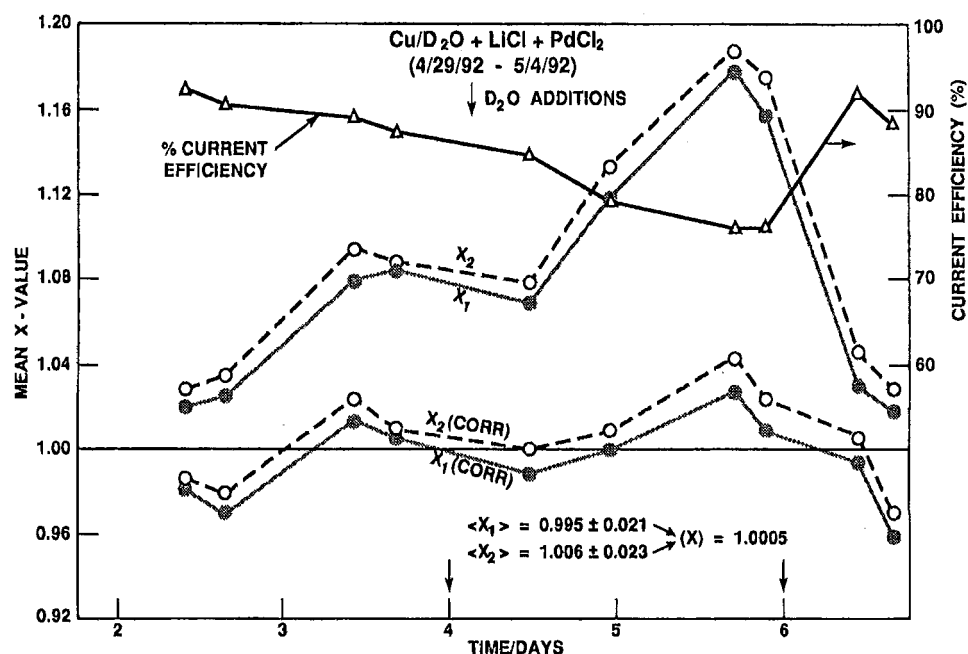
of the current to pass directly through the cell without producing any electrolysis.

The reaction or recombination of the  $D_2$  and  $O_2$  electrolysis gases or any cell shorting could always be readily detected in our codeposition experiments.<sup>16</sup> For example, there were always obvious changes in the rate of gas flow through the oil bubbler. The extent of these effects was determined by measuring the current efficiency ( $\gamma$ ) for the  $D_2O$  electrolysis. This was done by volumetrically measuring the rate of evolution of the  $D_2 + O_2$  electrolysis gases. The resulting  $\gamma$  can be easily applied to the calorimetric equations to correct for any apparent excess enthalpy produced by recombination or other reactions of the electrolysis gases.

A typical codeposition experiment where significant recombination or dendritic shorting occurs is shown in Figure 2. The apparent excess power reaching levels up to 18% could be readily corrected for recombination or other effects by the simultaneous measurement of the rate of evolution of the  $D_2 + O_2$  electrolysis gases. This was used to determine the current efficiency ( $\gamma$ ). The corrected values for  $X$  were then close to unity, and the overall ratio of power out/power in was  $X = 1.0005 \pm 0.022$ ; i.e., no significant excess power was observed after applying the correction for the current efficiency. The results in Figure 2 show that recombination can always be readily detected and easily corrected in our experiments. These results provide further proof that our calorimetric methods are accurate.

### Helium-4 Production in Electrolyte Cells

Perhaps the most important point raised by S. E. Jones and L. D. Hansen<sup>1</sup> was that our helium-4 detection limit was first reported as  $10^{12}$  atoms per 500 mL of effluent gases<sup>3,4</sup> and then later increased to  $10^{13}$   $^4He$  atoms/500 mL.<sup>5,6</sup> Our earlier limit was based on measurements at the University of Texas laboratory where 10 mTorr of air in 500 mL of nitrogen gas yielded the observation of helium-4 at the detection limit of the mass spectrometer (see Table 1 of ref 3). We reported that less sensitive detection limits were expected for  $D_2 + O_2$  electrolysis gas samples versus  $N_2$  gas samples because of their different absorption properties in the cryofilter that was used to separate  $D_2$  and  $^4He$  (see pp 101 and 104 of ref 4). Furthermore, the



**Figure 2.** Palladium–deuterium codeposition experiment where the current efficiency is significantly less than 100%. No significant excess power is observed after correcting for the experimental current efficiency ( $\gamma$ ).

$^4\text{He}$  content in the chemistry laboratory may have been considerably higher than the normal 5.22 ppm by volume assumed in our calculations.<sup>4</sup> Finally, the assumption that the nitrogen gas did not contain any  $^4\text{He}$  atoms may not have been correct, although it was always below the detection limit. Because we did not want to overestimate the amount of helium-4 produced in our experiments, we originally used the conservative estimate of  $10^{12}$   $^4\text{He}$  atoms/500 mL as our detection limit.

Solid evidence that we should have originally reported considerably higher helium-4 production rates was obtained in later studies where the electrolysis gas samples were collected in metal flasks rather than in Pyrex glass flasks and then analyzed by a commercial laboratory.<sup>6</sup> For five control experiments yielding no excess power, the mean background helium concentration in our system was  $4.4 \pm 0.6$  ppb or  $5.1 \pm 0.7 \times 10^{13}$   $^4\text{He}$  atoms/500 mL.<sup>6</sup> These values, therefore, accurately define a minimum helium-4 detection limit for our original studies. To clearly resolve this helium-4 detection limit issue, exactly the same procedures and apparatus were used in these experiments except for the replacement of the glass flasks with the metal flasks. This eliminated the diffusion of atmospheric helium into the sample flasks. These quantitative commercial measurements of background helium-4 concentrations in our calorimetric system dictated an upward revision of our original helium reports. It would be absurd to continue to claim  $10^{12}$   $^4\text{He}$ /500 mL (0.1 ppb) as our detection limit when our background helium concentration is accurately determined to be considerably higher.

In retrospect, the higher helium-4 detection limit resolves the issue of atmospheric helium diffusion into our glass flasks and is consistent with the detection limits reported by a commercial laboratory.<sup>5,6</sup> Furthermore, this higher helium-4 detection limit yields helium production rates of  $10^{11}$ – $10^{12}$   $^4\text{He}$  s<sup>-1</sup> W<sup>-1</sup>, which is the correct magnitude for typical fusion reactions that yield helium as a product.<sup>5,6</sup> The consistent merging of these various results would have been highly improbable if our initial measurements were due to errors or atmospheric contamination. Nevertheless, the revision in our helium-4 detection limit was a major issue raised by S. E. Jones and L. D. Hansen<sup>1</sup> in their

criticism of our work. Our explicit explanations for this change<sup>5,6,11</sup> were completely ignored.

For calculations of our helium-4 production rates, it should be noted that it requires 4410 s to produce 500 mL of electrolysis gases at 528 mA (200 mA/cm<sup>2</sup>) for our normal laboratory conditions ( $T = 296$  K,  $P = 690$  Torr). Therefore, simple calculations show that our results reproduced by Jones and Hansen<sup>1</sup> as Table 2 in Figure 1 yield  $10^{10}$ – $10^{12}$   $^4\text{He}$  s<sup>-1</sup> W<sup>-1</sup>. Later experiments involving much more accurate helium measurements of our electrolysis gases collected in metal flasks yielded approximately  $10^{11}$   $^4\text{He}$  s<sup>-1</sup> W<sup>-1</sup>.<sup>5,6</sup> Therefore, the amount of excess  $^4\text{He}$  found can account for nearly all of the excess power observed in our experiments.

Another criticism by S. E. Jones and L. D. Hansen<sup>1</sup> is our exclusion of run 12/17/90-B in calculations of statistical significance.<sup>4</sup> An unusual voltage increase with time for cell B suggested that the  $\text{D}_2\text{O}$  level was much lower than normal and not completely covering the electrodes in this cell. Eight days later at the end of this experiment, the  $\text{D}_2\text{O}$  level in cell B was 5.1 mL lower than in its companion cell A. These cells initially contained 18 g (17 mL) of 0.2 M LiOD +  $\text{D}_2\text{O}$ .<sup>4</sup> We later demonstrated that the low  $\text{D}_2\text{O}$  level observed in cell B that exposed the electrodes to the gas phase can yield a false excess heat effect. In early reports,<sup>17</sup> we omitted both cell 12/17/90-A and cell 12/17/90-B from statistical treatments of our heat-helium results since the two cells were run in series. In later reports,<sup>4,5</sup> we included cell 12/17/90-A in statistical arguments since this cell actually had an acceptable  $\text{D}_2\text{O}$  level. The question remains: Do you omit both cells run in series if the experiment is flawed in one cell? If the answer is yes, then you have the earlier result,<sup>17</sup> but if the answer is no, then you have the later results.<sup>4,5</sup> Unfortunately, the sample 12/17/90-A was inadvertently left out in our preliminary report of correlated excess power and helium production (see Table 2 of ref 3).

S. E. Jones and L. D. Hansen<sup>1</sup> contend that our observation of helium-4 in four out of ten  $\text{N}_2$ -filled glass flasks must be included for consistent and fair statistical treatment of our data. We totally disagree because air was deliberately introduced into four of these flasks in order to estimate the helium-4 detection

limit, and a fifth flask experienced an obvious air leak probably induced by air freight shipment (see Table 1 of ref 3). The only valid controls were four Pyrex flasks filled with boil-off  $N_2$  at the China Lake laboratory and then analyzed at the University of Texas 9 days later. The diffusion rate of  $3.2 \times 10^{12}$   $^4He$  atoms/day measured for our  $N_2$ -filled Pyrex flasks<sup>5</sup> and 9 days of storage yields a helium-4 concentration of  $2.9 \times 10^{13}$   $^4He$  atoms/500 mL or 3 ppb due to atmospheric diffusion alone. One  $N_2$ -filled Pyrex flask showed the presence of  $^4He$  at the detection limit while no helium could be detected for the other three Pyrex flasks (see Table 1 of ref 3). Results for these four  $N_2$ -filled Pyrex flasks provide additional evidence that our helium-4 detection limit was considerably higher than  $10^{12}$  atoms/500 mL (0.1 ppb) that we claimed initially.<sup>3,17</sup>

Experimental measurements of the diffusion of atmospheric helium into our Pyrex flasks is presented in Figures 1 and 2 of ref 5. Quantitative measurements by the Rockwell International laboratory clearly show that the amount of helium-4 increases linearly with the flask storage time as predicted theoretically.<sup>5</sup>

Plots of our data presented by Jones and Hansen<sup>1</sup> in their Figure 2 also show the helium-4 concentration versus the Pyrex flask storage time. The later graph (1993) gives the correct data points. The lines drawn in these figures were intended to simply illustrate that there is absolutely no correlation between the helium concentration reported and the flask storage time. The correlation coefficients are actually found to have negative values for either graph rather than the expected positive values; hence, bizarre statistics are encountered for arguments that our helium results are due to atmospheric helium diffusing into our Pyrex glass flasks. Note that the effect of atmospheric helium diffusing into our glass flasks should have been measurable even on an order of magnitude scale if our helium-4 detection limit were actually  $10^{12}$  atoms/500 mL as reported initially. For example, the experimental diffusion rate of atmospheric helium into our Pyrex glass flask yields  $2 \times 10^{12}$  atoms/500 mL after 1 day and  $2 \times 10^{13}$  atoms/500 mL after 10 days.<sup>5</sup>

S. E. Jones and L. D. Hansen<sup>1</sup> report that our designation of an experiment as a "control" is done after the experiment is run, not before. Neither Jones nor Hansen has been in our laboratory; hence, they have no basis for such a statement. Permanent laboratory records always defined the amount of excess power prior to any helium measurements. In general, excess power was consistently produced day after day in experiments that yielded excess helium-4 production, while no excess power was ever detected in experiments that served as controls.<sup>16</sup> In retrospect, helium-4 is probably the only nuclear product that could have remained so well hidden from view over the past 8 years of cold fusion experiments.

There is compelling evidence that the anomalous excess heat measured at our laboratory is associated with helium-4 production. For example, 30 out of 33 heat and helium studies yielded either excess helium when excess power was measured or no excess helium when no excess power was present.<sup>16,18</sup> A statistical treatment shows that the probability is approximately one in a million that our complete set of heat and helium results could be this well correlated due to random experimental errors in our calorimetry and helium measurements.<sup>16,18</sup> It is even much more unlikely that random errors could consistently yield helium-4 production rates in the appropriate range of  $10^{11}$ – $10^{12}$  atoms/s per watt of excess power.

## Radiation Measurements

Radiation monitoring was imposed upon our laboratory due to safety concerns but was never intended to be a major focus

of our program. This is one area where the criticism by S. E. Jones et al.<sup>1</sup> may have some validity. Nevertheless, anomalous radiation was detected by X-ray film exposure,<sup>3,4,17</sup> by the use of several different GM detectors,<sup>5,16</sup> and by the use of NaI detectors.<sup>16</sup> There was never any anomalous radiation when the experiments were not running.

Portable equipment for measuring radiation was available within the Navy;<sup>15,16</sup> thus, our laboratory did not accept the offer by S. E. Jones for the use of his portable X-ray spectrometer. Control studies showed that energies less than 50 keV could not escape from our cell and water bath. The revision of our experiments to optimize the X- and  $\gamma$ -radiation spectrometry would have critically compromised our calorimetry. Appropriate experiments were conducted at another Navy laboratory that yielded evidence for the emission of low-intensity X-rays during cathode polarization of the Pd/D system.<sup>15</sup> These experiments required specially designed cells where the palladium electrode is close to the detector window.<sup>15</sup>

## Miscellaneous Issues

S. E. Jones and L. D. Hansen<sup>1</sup> suggest a possible energy storage in the cell where there is a *negative* excess heat early in the experiment. We have never observed any energy storage in cells that produced excess heat. There is no real scope for energy storage in our cells—quite the reverse actually, since if deuterium were to leak out of the palladium, the cell would cool down.

S. E. Jones and L. D. Hansen<sup>1</sup> contend that  $D_2$  and  $O_2$  diffuse rapidly through Teflon; thus, recombination could occur on our anode and cathode lead wires despite our use of thick heat shrinkable Teflon tubing to protect these wires. This effect would certainly be very small and would diminish as the reaction product,  $D_2O$ , accumulated at the surface. Furthermore, cell calibrations performed under similar experimental conditions would zero out any such effects. Finally, volumetric measurements of the evolved gases show that no recombination occurs.

S. E. Jones and L. D. Hansen<sup>1</sup> attempt to explain our lack of helium-4 in  $H_2O$ -control experiments by suggesting that we were simply getting better at keeping out  $^4He$ . They overlook the fact that our very first  $D_2O$  sample (10/17/90-A) produced no significant excess power and no detectable helium.<sup>3–5</sup> Later experiments using metal flasks showed that our techniques yielded very consistent results in keeping atmospheric helium out of our system.<sup>6,16</sup>

Several additional statements by S. E. Jones et al.<sup>8</sup> need to be corrected. The thermoneutral potential,  $E_h$ , is the cell voltage at which the entropic cooling balances the polarization heating. Its numerical value is  $E_h = -\Delta H/zF$ , not  $\Delta H/F$  as in eq 3 of ref 8, with  $z$  indicating the number of charges transferred in one reaction step. The correct equation makes  $E_h$  invariant with the expression of the cell reaction and the direction of the cell current. At the high current densities used in cold fusion experiments, the cell voltage is always considerably larger than  $E_h$ ; hence, concerns by S. E. Jones et al.<sup>8</sup> regarding cells operating close to  $E_h$  do not apply.

More serious errors by S. E. Jones et al.<sup>8</sup> are found in their presentation of the electrochemical aspects of the cell operation. In particular, they stated that the exchange current density depends on the electrode surface area. The exchange current density always has dimensions of A/m<sup>2</sup> or similar units; hence, it cannot depend on the electrode surface area. Furthermore, there is no such thing as an exchange current density for their reaction 4 in ref 8. This cell reaction consists of the oxidation of hydrogen at the anode and the reduction of oxygen at the

cathode; hence, there are two distinctly different exchange current densities associated with the cell reaction.

## Conclusions

Documentation is presented that shows major allegations by S. E. Jones and L. D. Hansen concerning our experiments have already been explained in our previous publications as well as in a 1992 published discussion. The simultaneous measurements of power and the rate of evolution of the electrolysis gases in our experiments prove that faradaic efficiencies less than 100% cannot account for our reports of excess heat. Excess enthalpy for the Pd/D<sub>2</sub>O system generally involves high current densities that exceed 100 mA/cm<sup>2</sup>; therefore, the report by S. E. Jones et al. of low faradaic efficiencies using current densities of only 1–2 mA/cm<sup>2</sup> is not applicable to our cold fusion experiments. Based on experiments at our laboratory, there is compelling evidence that the anomalous excess heat is associated with helium-4 production. For example, 30 out of 33 heat and helium studies yielded either excess helium when excess power was measured or no excess helium when no excess power was present. The probability of obtaining this result by random errors in our heat and helium measurements is about one in a million. Permanent laboratory records always defined the presence or absence of excess power prior to any helium measurement. The measurement of helium in the electrolysis gas samples at three different laboratories places our rate of helium-4 production at 10<sup>11</sup>–10<sup>12</sup> atoms/s per watt of excess power. This is the correct magnitude for typical deuteron fusion reactions that produce helium-4 as a product.

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