McKubre, M.C.H. The Importance of Replication. in ICCF-14 International Conference on Condensed Matter Nuclear Science. 2008. Washington, DC.

# The Importance of Replication

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# Abstract

Much discussion in the Condensed Matter Nuclear Science or "Cold Fusion" fields centers on the subject of replication. It is a topic that comes up in essentially every conversation about the Fleischmann Pons Effect (FPE). Assembled here is a set of essentially personal views on this subject of replication.

# Why is replication important?

We might begin with the dictionary definition of replication as the action of copying or reproducing something [1]. More specifically in science we mean the repetition of a scientific experiment or trial to obtain a consistent result. Note that replication is defined in terms of reproduction and that the key test in science is consistency, not identicality.

Reproducibility is the "Touchstone" of Scientific Method. Scientific method does not work if you cannot perform trials with different input parameters, learning something certain from the observed change in output. Critics argue that for something to be "not reproducible" means that it is "not science", implying that it is "not real". This last inference is a false argument. Reality and reproducibility are different concepts.

If the effect one is attempting to study is rarely seen, then it is hard to study. It would be a great advantage for experimenters to have a more reproducible effect. But its lack does not make the effect unreal. Without <u>quantitative</u> reproducibility it is harder (but <u>not</u> impossible) to perform parametric studies which are the basis of the empirical method that we use to gauge the effect of input variables that should – or should not – influence the effect under study.

# What does it mean to "replicate"?

Replicability conveys the ability to demonstrate the effect that you are observing and studying <u>on demand</u>. We can extend the definition and difficulty of replication to include the ability to transport and transplant a successful experiment from one laboratory to another. This is something that the group at SRI has been very much involved with. In the extreme case we would like the ability to interchange experiments between laboratories on the basis of a written instruction set alone. This is more or less the basis of US Patent Law that applies the test of a fictional "person having normal skill in the art" following written instructions (the Patent). Although we would consider ourselves to meet the standard of "normal skill" our experience in replication at SRI, in essentially every instance, written instructions alone have been insufficient to allow us to reproduce the experiments of others. It is not clear that this inability is limited to the FPE; other classes of replications have not been attempted.

Another extreme and desirable condition particularly on the pathway to application is the ability to reproduce (in every case) the magnitude and timing of the effect. If one has an effect completely under control then the input variables lead immutably to the output variables. In this extreme case it is not necessary to perform the experiment, the result is predetermined. To have complete empirical understanding requires a comprehensive, quantitative, fundamental understanding of the effect under study. This can be based on physical theory, although it should be noted that many physical laws that represent apparently entirely reproducible and predictable phenomena are not so based.

#### Have we succeeded?

More particularly, to what extent have we succeeded? The answer depends on what one is looking at; what effect, what evidence? We have succeeded in a number of regards and I will go through some of the examples and review some evidence. It is appropriate to note that a number of effects claimed in the CMNS field have not been replicated with sufficient rigor or diversity to have achieved widespread support of success.

We have certainly successfully transported and replicated the Fleischmann Pons Effect, by which is meant the production of nuclear level heat from the electrochemical stimulation of the heavy water – palladium system. This effect has been observed by hundreds of people in dozens of laboratories around the world, and published in hundreds of papers as recently reviewed [2, 3]. In fact the very breadth of diversity in experiment and calorimeter choice has contributed to the illusion of irreproducibility (although, as will be argued later, the primary cause of apparent irreproducibility is the lack of measurement and control of variables critical to the effect).

The observation of <sup>4</sup>He also appears to meet the criterion of transportability. Miles and Bush were the first to demonstrate semi-quantitative correlation between the rates of creation of <sup>4</sup>He and excess heat [4]. That effect has now been observed in a number of laboratories around the world [5-7] including SRI [2, 8] with considerable assistance from Dr. Benjamin Bush. The Case experiment was successfully replicated at SRI [2, 8]. However, we were not able to replicate this experiment simply based on written instructions alone, and required the intervention of Dr. Leslie Case. The Arata experiment in its original form involving electrolysis of a double structured cathode [7] was successfully replicated at SRI [2, 8] after soliciting help from Professors Arata and Zhang. Again, however, we were also not successful in replicating this experiment simply based on published materials.

Experiments involving laser triggering of excess heat by laser triggering of deuterium loaded palladium cathodes with specially modified surfaces were initially discussed by Letts and Cravens [9]. This class of experiments has been successfully transported from laboratory to laboratory although again requiring hands-on tuition and involving long periods of unexplained failure [10-12].

At SRI we have been unsuccessful in a number of attempts at experiment replication. We were not able at any time to reproduce the claims of heat from nickel – light water electrolysis experiments. We were able uncover one source of systematic error in the experimental procedures involving large area nickel – carbonate electrolyte experiments that blunted our interest. This inability should not be taken to mean that the claims are wrong or an effect not real, particularly in light of previous failures to replicate before personal, hands-on guidance was sought. We were not able to replicate the Patterson-CETI experiments [13]. Despite the very able hands on support of Dr. Dennis Cravens we were never able to observe an excess heat effect for this experiment in our mass flow calorimeters, although it is now understood that an important experimental element may have been lacking. A similar situation exists in respect of the Stringham [14] ultrasonically induced Pd-D<sub>2</sub>O excess heat effect using SRI mass-flow calorimeters, although this condition of uncertainty was exacerbated by the complexities of input energy measurement and coupling between the ultrasonic power source and the transducer and experiment.

These last two cases (Patterson and Stringham) highlight a feature and two rules to be observed in any attempt to replicate calorimetrically an already difficult experiment and study a fragile effect.

- i. The calorimeter is part of the experiment. This is true whether the "effect" is a systematic error or an unexplained heat source. Changing the calorimeter may change the triggering or amplitude of the phenomenon under test. One simple example of this phenomenon is the extent to which the unaccounted excess heat changes the temperature of the experiment; other feedback systems are possible.
- ii. The experiment cannot be compromised and become subordinate to the calorimetric needs. If the experiment needs to be changed <u>in any significant way</u> to accommodate the requirements of the calorimetric method that is preferred (by some criterion or that happens to be on hand), then the test of replication is compromised. This condition can be relaxed only after all variables critical to the effect are known and measured.

To the extent that it occurs, the lack of reproducibility is reflected in the magnitude, timing, gain, and termination of the effect. We perform intentionally identical experiments repeatedly and obtain clear evidence of the effect, but with variable (and inconstant) magnitude, variable initiation time, or the power and energy ratios output:input are not in every case the same. Even the termination of the experiment is not fully under experimental control. One may turn the stimulus off, discontinuing laser, ultrasonic, electrochemical current or other stimulus, and still continue to observe the effect, sometimes requiring minutes, hours or even days to decline to the thermal baseline. The effect may persist after discontinuation of all externally generated stimuli, the so-called "heat-after-death" or more logically "heat-after-life" effect. Thus we cannot even stop our experiments always on demand and we do not yet have quantitative reproducibility in any case of which I am aware.

Every one of the CMNS subtopics still needs further research. To get from where we are to where we need to go requires a substantial level of physical materials support. Much as it was in the early days of semiconductors, today it is our materials that are letting us down. In current FPE studies it is the metallurgy of palladium that is the principle barrier to complete quantitative replicability. On the other hand we do have a limited empirical model. We are able to explain experiment failures and the failure to produce excess heat in terms of our inability to meet certain input conditions: loading, deuterium flux, input stimulus. This ability to explain failures, and thus learn from them, is very valuable.

# Why are there failures?

At this point a reasonable question might be: if you know what you need to do, why can't you always do it? Why is there any degree of irreproducibility? What are you waiting for? The

answer is straightforward: the material conditions of our experiments are not completely under our control. Solids are more complex than liquids; liquids more complex than gases or plasmas. The solid:liquid interface may be the most complex structure of all material science. This is a structure that we are only just beginning to understand in any level of detail and are still struggling to control.

Electrochemistry takes place at an electrified interface between two "difficult" materials, neither of which are fully under our control. In the early days of studying the FPE at SRI experiments were designed to probe the parameters of reproducibility. Sets of 12 cells were prepared, intentionally identically, and operated simultaneously to monitor the time evolution of electrochemical and physico-chemical parameters believed to be or potentially pertinent to the FPE.

A single length of palladium wire was used from a known source and sectioned into 13 identical lengths (12 active electrodes plus a reserved blank). These wire sections (typically 3 or 5 cm in length and 1 or 3 mm in diameter) were machined to remove surface damage and inclusions, spot welded with 5 contacts (one cathode current and 4 wires for axial resistance measurement), annealed, surface etched (to remove surface contaminants) and mounted in 12 identical cells of the type shown in Figure 1. These processes all were performed in the same batch and by the same person. The twelve cells were filled with the electrolyte from a single source and then operated electrically in series, in a 3 x 4 matrix in the same constant temperature chamber.

The variables measured continuously were current (one measurement), cell voltage, pseudoreference cathode potential, temperature and electrical resistance (D/Pd loading) all being monitored in a multiplexed manner with the same instruments. Intermittent measurements were made of the cathode interfacial impedance. With 12 intentionally identical experiments, every one behaved differently. Not only in terms of their heat production, significant and marked differences were observed in: the current-voltage-time profile for both the cell voltage and reference potential; the ability and willingness of each electrode to absorb deuterium measured by the resistance ratio-time curve; the maximum loading achievable; the interfacial kinetic and mass transport processes reflected in the interfacial impedance. Every one of these parameters was importantly different for each of the 12 electrodes.

This set of experiments was repeated several times in an attempt to understand the origins of the irreproducibility, and therefore control it. Trace impurity differences were observed to be contributory and these divided into two sets: deleterious impurities (poisons) that we learned to avoid; impurities that were beneficial to high loading in controlled amounts. This was a highly useful (and somewhat surprising) exercise. Although we were able to make progress and reduce the dispersion, we were not able to control the irreproducibility simply by electrochemical (and trace chemical) means. We continued to operate sets of cells in what we called "farms", selecting the most promising for promotion into calorimeters of the sort shown in Figure 2.



Figure 1. Electrochemical cell for loading and calorimetric studies.



Figure 2. Labyrinth Mass Flow Calorimeter incorporating Degree-of-Loading Cells

Four important understandings developed from these intensive studies at SRI of deuterium loading and calorimetry:

- i. Irreproducibility in FPE experiments can be fully or at least sufficiently explained in terms of the electrochemistry of loading D into Pd.
- ii. In the absence of a measure or knowledge of the D/Pd loading the experimenter has no basis to judge whether an experiment could or should have produced excess heat.
- iii. After basic precautions are taken the irreproducibility of loading and interfacial kinetics is not largely or even primarily controlled by the electrolyte or the electrochemistry, it is controlled by the bulk palladium metallurgy.
- iv. An empirical and near quantitative understanding of the measured magnitude of excess heat effects, and more particularly of the failure to achieve the FPE, can be obtained from measurements made of the controlling variables, and the failure to achieve critical threshold values.

If the metallurgy varies so greatly between adjacent sections of the same wire, annealed and surface treated in the same way and at the same time, how great might the difference (irreproducibility) be between metal samples from different sources? Until recently we have not had sufficient resource of time, money or talent to begin a serious campaign to understand the issues of materials irreproducibility as these pertain to the defect and impurity structure of polycrystalline palladium. Many people recognized this as one of or the most important problems to be faced right from the beginning in 1989 or before, Fleischmann, Bockris, and Huggins amongst others.

Several people have taken up the challenge of palladium metallurgy with limited resources, amongst others Imam at NRL, Letts in his own lab and Violante at ENEA. These efforts have resulted in significant progress and even patents. Most recently the efforts of Vittorio Violante's group to control the metallurgy and surface morphology of palladium foils has contributed significantly to the formal laboratory-to-laboratory replication of Energetics FPE results at ENEA and SRI [15]. Recent experiments at ENEA and NRL [16] have demonstrated that identical annealing and rolling treatments of different starting batches of palladium results in markedly different foil characteristics. To make rapid progress into the light of reproducibility, significant resource must be directed as was done previously for electrochemistry, to identify those characteristics of bulk palladium that are crucial and those that are detrimental.

There has been some discussion of "hidden" variables; a controlling or contributing parameter that so far remains unidentified. In a difficult experiment or situation it can be comforting but also debilitating to attribute failure to a hidden agent. Without further empirical knowledge or theory to guide us, the only rational position is the middle ground. We must to proceed along the path of increasing control of the electrochemical and metallurgical variables that we understand and can measure, in the attempt to exert full control over the FPE. Along this path we need to remain alert to the possibility of a critical undiscovered parameter.

Much of this uncertainty would be resolved and a great deal of tedious and repetitious matrix study avoided with a theory to guide us on the path and point towards the answer. While the FPE is the first and most concrete demonstration of a condensed matter nuclear effect it is highly unlikely that this is the only or best manifestation of a new physical effect. Either by means of theory or ingenious engineering it is likely to be far more practical to <u>avoid</u> the challenges and

limitations present in the electrochemical loading of bulk palladium rather than climb the mountain of ever increasing materials control of difficult systems.

# What have we accomplished so far?

This is not intended as a review but simply is a set of examples taken from the works at SRI and our immediate collaborators. By 1992 we had demonstrated a nuclear scale heat output, and had determined that the effect was real if you controlled the loading variable. There was also an initiation effect that we (and others) had discovered at that point. Figures 3 and 4 show two of the controlling variables, the effect of current density and D/Pd loading on the excess heat effect [2, 3].

By 1995 we had uncovered the importance of flux, the movement of deuterium through the interface and that this was not an equilibrium effect, and we had formed these various terms into an empirical heat equation [2, 17]. Between 1996 and 1998 we measured associated nuclear products, specifically <sup>4</sup>He [2] following Miles in 1992 [4]. By 2000 we had measured ash uncorrelated with heat (<sup>3</sup>He). Figures 5 and 6 show our results replicating the Arata double-structured (DS) cathode electrolysis experiment [8]: Figure 5 plots the excess power measured in heavy and light water electrolyte, and the percentage excess in heavy water; Figure 6 shows the profile of <sup>3</sup>He measured through the wall of the DS cathode produced by the disintegration of tritium diffusing from the inner void where it was created, to the outer electrolytic surface.

In 2003 we published [10] a replication of the Letts-Cravens laser triggering result [9] and a significant new result came in 2007 working in collaboration with ENEA and Energetics. As discussed above the knowledge gained at ENEA helped achieve an improved control (if not mastery) of the palladium metallurgy. The knowledge developed at Energetics using the Dardik SuperWave concept enabled us to achieve much greater reproducibility in the control of loading and interfacial deuterium flux. With this improved understanding we gained a higher degree of command over two parameters crucial to the production of excess heat.

As a consequence we were able to achieve high levels of reproducibility in excess heat production. Table 1 shows the complete set of experiments performed at SRI [15, 18] following Energetics protocols and SuperWave current excitation profiles [19], in all except one case using Pd foil cathodes fabricated at ENEA [16]. The rows labeled "E" were performed at SRI with Energetics electronics and data acquisition; those labeled "S" were performed at SRI with SRI electronics and data acquisition. Of the 15 experiments performed in the latter mode, 11 (73%) demonstrated power excess at or above the 5% level that was determined to be the calorimeter  $3\sigma$  accuracy.



Figure 3. The effect of Electrochemical current density on excess heat production in FPE electrolysis experiments.



Figure 4. The effect of deuterium loading on excess heat production in FPE electrolysis experiments.



Figure 5. The effect of input power (and time) on excess heat production in Arata DS cathode experiments.



Figure 6. Logarithmic diffusion profile of <sup>3</sup>He following on excess heat production in Arata DS cathode experiments.

Table 1. Energetics Replication Results.

	Cell -	Cathode	Min.	Max.	Excess	Power	Energy
	Calorimeter			D/Pd	$\%$ of $P_{In}$	(mW)	(kJ)
1	9-7 E	Lot A	1.77	0.895	<5%		
2	11-8 E	L5(2)	1.67	0.915	60%	340	514
3	12-9 E	Lot A	1.84	0.877	<5%		
4	15-7 E	L5(1)	1.77	0.895	<5%		
5	16-8 E	L5(4)	1.86	0.871	<5%		
6	17-9 E	L1(1)	1.55	0.939	20%	460	<b>407</b>
7	21-7 E	# 830	1.92	0.836	<5%		
8	22-8 E	L5(3)	1.8	0.888	30%	200	188
9	35-7 <mark>S</mark>	L17(1)	1.32	0.985	12%	1800	553
10	35-8 <mark>S</mark>	L17(2)	0.95	1.059	13%	2066	313
11	35-9 <mark>S</mark>	L17	1.39	0.971	1%		
12	43-7 <mark>S</mark>	L14-2	1.73	0.903	<b>80%</b>	1250	245
13	43-8 <mark>S</mark>	ETI	1.63	0.923	5%	525	65
14	43-9 <mark>S</mark>	L14-3	1.61	0.927	1%		
15	51-7 <mark>S</mark>	L25B-1	1.55	0.939	12%	266	176
16	51-8 <mark>S</mark>	L25A-2	1.52	0.945	5%	133	14
17	51-9 <mark>S</mark>	L19	1.54	0.941	43%	79	28
18	56-7 <mark>S</mark>	L24F	1.55	0.939	15%	2095	536
19	56-8 <mark>S</mark>	L24D	1.84	0.877	4%		
20	56-9 <mark>S</mark>	L25B-2	1.56	0.937	3%		
21	57-8 <mark>S</mark>	Pd-C	N.A.	N.A.	300%	93	115
22	58-9 <mark>S</mark>	L25A	1.69	0.911	200%	540	<b>485</b>
23	61-7 <mark>S</mark>	L25B-1	1.63	0.923	50%	105	146
	-		1 0	CDID			

E = Energetics and S = SRI Data Acquisition.

In addition to the high levels of loading and reproducibility of excess heat production this set of experiments had several other desirable characteristics. Experiment failures (null results) were understandable in terms of our inability to meet and exceed known threshold criteria: loading, maximum loading, loading duration and interfacial deuterium flux.

One striking feature is the power of the SuperWave in moving deuterium through the interface. Figure 7 shows the measured resistance ratio and deuterium loading calculated for three points in the 20 minute SuperWave current exercise cycle: the minimum, mean and maximum values. From these values we can calculate the net interfacial flux of deuterium absorption and desorption. For an electrode with an active surface these values were measured to be as high as  $20 \text{ mA cm}^{-2}$ . This value is higher by far than any previously measured at SRI using dc or any other waveforms except under strong transient conditions.



Figure 7. Resistance ratio and deuterium loading during SuperWave electrolysis of an  $80 \times 7 \text{ mm } 50 \text{ mm}$  thick ENEA Pd foil in 0.1 M LiOD.

The existence of data sets demonstrating high power gain and, even more particularly, high energy gain offers several specific benefits to FPE researchers:

- i. High power gain and large values of excess power compared to calorimeter accuracy makes it easy to rule out systematic mismeasurement of input power or temperature as being the source of the FPE, even in discussions with individuals unfamiliar with calorimetric principles and practice.
- ii. High energy gains and large values of integrated excess energy (often expressed in terms of eV per Pd atom) makes it easy to rule out energy storage or anomalous unseen chemical effect as being the source of the FPE, even in discussions with scientists and engineers not having specialized knowledge of materials science and chemistry.
- iii. One spectacular experiment performed at Energetics designated as experiment 64 exhibited energy gains exceeding 25 and accumulated several keV/Pd atom of energy at temperatures at or above the boiling point of water [19]. Results such as these suggest the potential for practical application and the possibility that FPE studies may move from experiment and research into engineering and development.

# Conclusions

At least in this field the "reproducibility" standard is generally far harder to meet than most experimenters or the US Patent and Trademarks Office anticipate. The reasons for this are several:

- 1. Particularly in the early stages of invention the experimenter himself or herself may not be aware of all critical details of his or her experiment, and succeeds because of a knack or skill or habit that is not recognized and not easily transported or identified except by direct, careful observation.
- 2. All communication is imperfect. The advantage of written communication is that it creates a record capable of being improved. However writing suffers two great weaknesses. It is slow and painful and therefore shortcuts are taken and tedious and seemingly superfluous detail truncated or eliminated. It is therefore incomplete. Because of its permanent nature and the possibility (usually viewed as certainty) that posterity will be watching, scientists tend to make written reports appear far more rigorous than their actual procedures. The written record may therefore be over-complete and over-complex while possibly being a false or misleading report of what in fact was done.
- 3. In my experience scientists cannot resist the compulsion to improve. When first they hear of an experiment they begin immediately to think of a new and better way to get a bigger and more interesting effect. Don't! If the effect is artifactual it is important to reproduce the artifact that may precisely lie in the means of temperature measurement or the experiment geometry, *etc.* If the effect is real it may depend critically a peculiarity of the pump or power source, or on an interaction between variables that is unseen, unanticipated or misunderstood.

From nearly twenty years of work at SRI two important conclusions have emerged. Scientists can sometimes be wrong, and often oversimplify, but they are not incompetent or dishonest. Even after significant effort (and none of the experiments we worked on in this field required less than 3 months sustained effort to form even a provisional opinion), don't assume you were lied to or that "the other guy" is "an incompetent boob". If it doesn't work it is your fault.

Reproduce exactly first. Work with the originator directly, in person, understand their procedures at every step until the original effect is recreated. In 1996 Lonchampt *et al* [20] set themselves the task of reproducing the original FPE work, in their words "*simply to reproduce the exact experiments of Fleischmann and Pons - to ascertain the various phenomena involved in order to master the experiments*". The phrase underlined is critical. Only from the point of mastery can systematic effects be studied, whether these are errors, artifacts or new physical processes.

As far as I am aware Lonchampt and his team were and remain the only group ever to attempt an exact engineering replication of the original Fleischmann Pons experiment. It helps considerably that they were engineers, not scientists. Lonchampt *et al* may also have been the only group ever to publish a paper with the goal of FPE replication in its title [20]. They also were successful and closely specified the conditions under which replication was possible:

- *i.* The Fleischmann Pons calorimeter with precautionary measures taken is simple and precise.
- *ii.* Their calorimeter is very accurate and well adapted to study cold fusion.

- *iii.* The maximum error might be in the higher temperature range, and under any condition should not exceed 1% of the input power.
- iv. The effect measured [now called the FPE] is:
  - below 70°C, between 0 and 5%
  - between 70°C and 99°C, about 10%
  - at boiling, up to 150%

At this point I hope I am permitted to conclude with a personal remark or two. If the claim is made that replication is crucial to the development of our field to determine the parameters for advancement, to prove reality to critics, or to uncover systematic error, then it is astonishing that attempts to replicate the FPE have been so few, and methodologically so limited. I do understand the reasons, and am as guilty as any FPE researcher. But it must be completely understood that this lack of attention to detail and the failure to assimilate what Lonchampt taught us, is precisely the reason that the question of replicability remains on the table, and is the motivation for this paper.

One important reason for this lack of focused attention on the question of reproducibility is that this is not the most important question now, and was not in 1989 although it may become so in the near future. The question of reproducibility, while undeniably important, is almost trivial compared with the question of whether or not there is evidence of a new physical effect. Confining attention to the FPE, the pressing and fundamental question is: is there evidence for heat production consistent with nuclear <u>but not</u> chemical effects in the deuterium-palladium system? Clearly the answer is yes. This fact has been established at a level far above working hypothesis as a "working reality" to a by a large numbers of experimenters in many and diverse experiments.

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