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Research Article

Protocol for a Silicate-based LENR Using Electrodes of Various Metals

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Abstract

This paper reports a protocol that consists of applying concurrent electronic and photonic stimuli in a cell with two or more electrodes at or near the boiling point of the liquid. The liquid in the cell is a solution including a silicate, a lithium salt, and a surfactant. The electrical stimuli are RF signals and, optionally, a direct current. The protocol generates an exothermic reaction characterized by sharp temperature transients. We have successfully used three different silicates and four different metals for electrodes. We believe the exothermic reaction is nuclear in nature. The evidence supporting that statement includes:

- Data logs show brief, intense temperature transients.
- Electron diffraction scattering (EDS) analyses show elements to be present after the reaction that could be transmutation products of several elements in the ingredients of the protocol, specifically including silver, a possible transmutation product of palladium.
- Auger analysis of one experiment also shows evidence of transmutation of the elements in the reaction cell.
- SEM photos show "volcanic sites" and other evidence of metal migration.
- Other SEM photos show large areas where electrodes have spalled during experiments.

Keywords: Gold, Heat transients, LENR, Lithium silicate, Palladium

1. Background

We hypothesized that using a lower surface tension electrolyte in the Fleischman–Pons experiment would better wet the Pd electrode, leading to more efficient reaction and possibly greater excess heat. Anionic silica hydride was chosen because it is a soluble silicate known to have surfactant properties, but not to have a surfactant tail, which we wanted to

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avoid. Anionic silica hydride is commercially marketed for human consumption as an alternative health care product in both a liquid and crystalline form. The liquid is called Super Hydrate and the crystalline is called Mega H^- [1]. It is reported to lower surface tension of water from 72 to 48 dynes/cm [2].

Dr. Michael McKubre told one of the authors in a conversation at SRI International that three things have been demonstrated to stimulate LENRs: electricity, light, and vibration. We chose to apply all three at once, with the vibratory stimulation introduced as a pulse-modulated RF sine wave. That latter technique is used to simulate percussion signals in electronic circuit simulations.

Our experiments required two hours or more gestation time before bursts of heat occurred. We suspected that something must be changing either with the solution or the electrodes in that period that facilitated the observed reaction. To the best of our knowledge, all of the successful LENR experiments conducted either by us or other experimenters have included lithium in the reaction, so lithium appears to be an essential ingredient. Reasoning that both silica and lithium are needed in the reaction and that the reaction does not occur immediately, we suspected that the anionic silica hydride and the lithium in our protocol might form a compound (coordination or stronger) before the reaction occurs. To test that hypothesis, we substituted two other silicates for the anionic silica hydride–lithium metasilicate, Li_2SiO_3, and sodium metasilicate, Na_2SiO_3.

2. Experimental Setup

The reactor used was a stainless steel cylinder with a central well 5.08 cm deep and 5.08 cm in diameter, having a closed bottom and a removable top. A photograph of the vessel is shown in Fig. 1. It was dimensioned to accommodate a glass beaker capped with a quartz top. Electrodes and thermocouples passed through holes in the top.

Four "ultrabright" white LEDs, capable of generating 15,000 mcd each, were spaced equally around the vessel below the surface of the liquid as photonic stimuli. These stimuli were provided through sealed glass ports in the vessel wall.

The electrical stimuli were provided via three electrodes of 0.63 mm diameter: a common cathode, an anode for the RF stimulus, and a second optional anode for the DC stimulus. The electrodes formed a triangle with sides 2.3, 3.7, and 3.7 cm long. The shortest side lay between the RF anode and the common cathode. The electronic stimulus was a signal generated by an arbitrary waveform generator (AWG). The RF sine wave had a frequency of 3.1 MHz and the modulating pulse waveform was 48 MHz with a 50% duty cycle. Those frequencies were chosen for purely intuitive reasons.

All three electrodes were isolated from the steel vessel and sheathed in glass tubing to the surface of the liquid in order to keep them straight and to concentrate the RF stimulus in the liquid. The electrodes passed through the vessel's top via Teflon® seals compressed with Swagelok® fittings. We have used palladium, silver, gold, and platinum electrodes and observed similar temperature transients. All experiments were performed with electrodes of similar metals rather than dissimilar ones.

The temperature of the liquid was monitored via thermocouple wells projecting into the liquid. The wells were made of stainless steel or glass, and the reaction was more robust when glass wells were substituted for the steel ones. The thermocouple wells also passed through the vessel's cap via Teflon® seals compressed with Swagelok® fittings. The electrodes and the thermocouple wells were equally spaced on a bolt circle, so thermocouples would be 2.3 and 3.7 cm away from the cathode.

The headspace above the liquid in the vessel consisted of a static blanket of hydrogen and helium, in approximately equal percentages.

The majority of our experiments used 30 ml. of either H_2O or D_2O in the cell with 1.4 g. of lithium sulfate (Li_2SO_4), 100 mg. of Mega H^-, and 0.8 ml. of Super Hydrate. The solution was buffered with citric acid to keep it in the range of 6.5–8.9 at the beginning of the experiments.

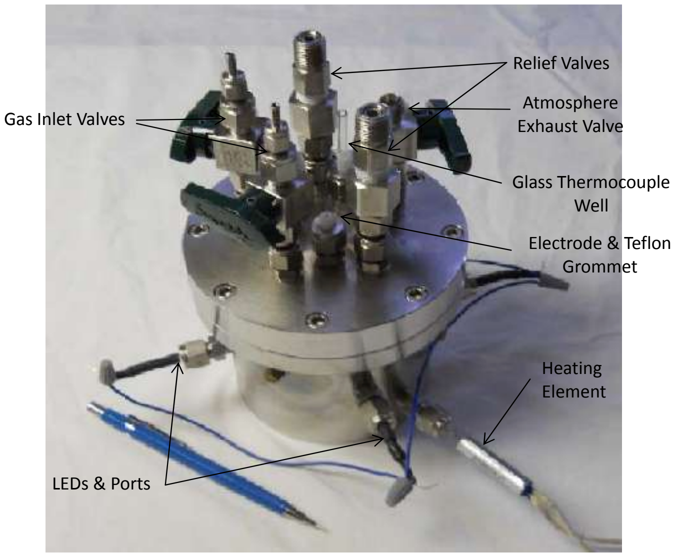

Figure 1. Reaction cell, showing LEDs and ports on the side. Electrodes and thermocouple wells can be seen on the top, along with relief valves and gas valves.

When Li_2SiO_3 and Na_2SiO_3 were used, they were mixed with EDTA to dissolve and/or suspend the silicates in the D_2O. The electrodes used in those experiments were palladium. In the experiments using either lithium metasilicate or calcium metasilicate, the solution was buffered, using sodium bicarbonate, to keep $6.5 < \text{pH} < 8.9$.

The input heater power was determined by measuring the voltage and current applied to a heating coil, located in a cavity in the bottom of the vessel. The temperature of the vessel was initially raised to 101˚C ± 1˚C. After the temperature of the liquid had remained stable within that range for at least two hours, we applied the photonic and RF stimuli. Temperature was controlled by manually adjusting the input power to the heating coil. We did not attempt to deliberately raise the temperature of the solution above the boiling point or to measure pressure.

In latter experiments we applied a signal consisting of a 3.1 MHz fundamental sine wave modulated by a smaller 43.4 MHz sine wave. After reaching equilibrium near the boiling point of the solution, the applied stimuli add less than 100mW of incremental power to initiate the reaction [3,4].

As a safety practice appropriate when working with exothermic reactions in a sealed vessel at or near the boiling point of water, our vessel was equipped with two relief valves set to lift sequentially at different pressures. We strongly recommend that others replicating this protocol do the same thing.

3. Results

Figure 2 shows a datalog from Experiment No. 1, where the horizontal axis is two minutes per division and the vertical axis is either 1˚C or 1 V, as appropriate for the variable measured. Data was logged at 10-second intervals. The experiment was conducted with palladium electrodes in heavy water. The electrolyte was lithium sulfate and the silica reagent was anionic silica hydride. The time-varying electrical stimulus was the sinusoidally modulated pulse stream described above.

- The data was logged with an Agilent 34970A Data Acquisition Unit, which uses a proprietary database system,

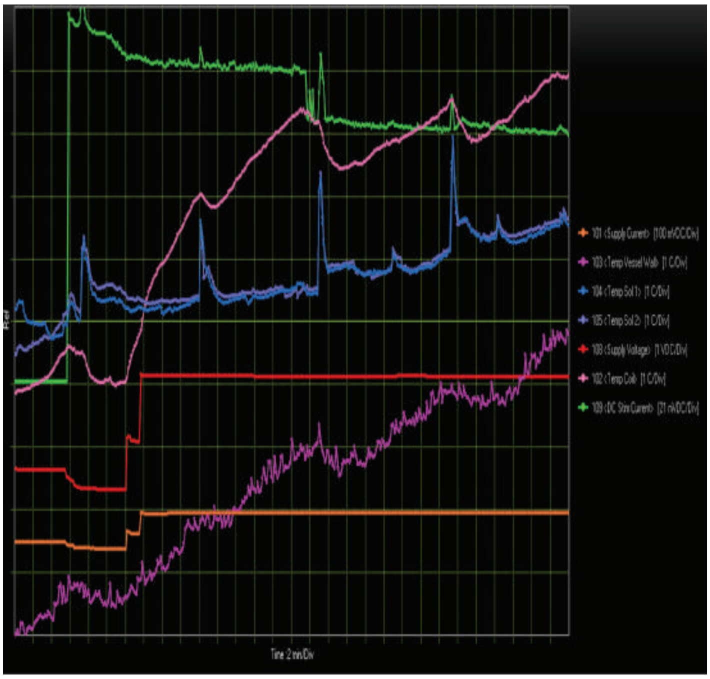

Figure 2. Experiment No. 1 data log showing temperature spikes. Experiment used Pd electrodes, anionic silica hydride, and lithium sulfate as an electrolyte in predominately heavy water.

preventing modification of the raw data. All data logs shown are cropped screen prints from Agilent's Data Logger 3 software.

- The temperatures of the solution are shown with traces 104 and 105, with a centerline of 102˚C. The data log shows at least four distinct bursts of heat that had notable periodicity, occurring at intervals of approximately 13 min. The data was logged at ten-second intervals, so the largest burst rises more than 1.4˚C in 10 s.
- Note that the temperatures of the heating coil (trace 102) and the vessel wall (trace 103) drop after each burst, possibly caused by the vessel leaking some steam or venting through the relief valves. (We consistently noted the loss of approximately one-third of the liquid in the vessel during our experiments when the temperature transients occurred, and that would consistent with steam leaking or venting. Such losses were not observed when the transients were not observed and the reaction presumably did not occur.)
- Note also that the DC stimulus current (trace 109) spikes concurrent with or slightly before the bursts occur. Those current spikes suggest that the resistance between the DC anode and the common cathode decreased at approximately the same time the temperature spikes were occurring.
- After treatment, portions of the wire were coated with a deposit that resembled a field of bubbles when viewed in a SEM. However, they were solid rather than hollow.
- We do not claim that this particular data log is representative of all of our experiments. It shows the pattern of the thermal behavior more distinctly than was typical and also shows the current spikes in the DC stimulus better than our other experiments did.

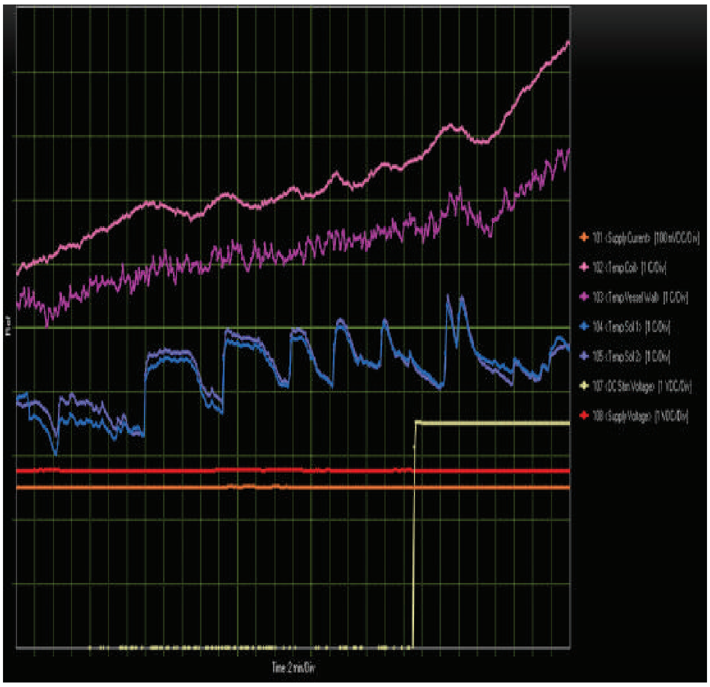

Figure 3. Experiment No. 3 data log showing temperature spikes. Experiment used Pd electrodes, anionic silica hydride, and lithium sulfate as an electrolyte in predominately heavy water.

Glass beads purchased from a craft store were threaded onto the palladium wire for Experiment No. 2, whose results are shown in Fig. 3.

- The electrodes for this experiment were palladium in a solution of heavy water. The electrolyte was lithium sulfate and the silica reagent was anionic silica hydride. The time-varying electrical stimulus was the sinusoidally modulated pulse stream described above.
- The reaction kicked in at a relatively low temperature, just above 100˚C, and showed some sustained bursts lasting as long as 5 min. The temperature of the solution (traces 104 and 105) peaked at 102.4˚C. Note that the temperature spikes last longer than the ones seen in the experiment shown in Fig. 2, presumably meaning more heat was generated with the presence of glass beads.
- There is a strong reaction before the DC stimulus voltage (trace 107) was applied. For that reason, we only used two electrodes, the RF anode and the cathode, in most of our later experiments.
- When viewed in the SEM with the glass beads removed, there were no deposits on this wire and significant portions of the wire had spalled and peeled away. Spalling is caused by stress and could suggest local high temperatures. Figure 4 is a micrograph of a piece of electrode that spalled and became twisted during the experiment.

The data log for Experiment No. 3 using lithium metasilicate instead of anionic silica hydride is shown in Fig. 5. The boiling point of this solution was higher than that in the standard protocol, so the centerline of the two traces (104 and 105) for the thermocouples is 104°C. There were some bursts of heat, but they were not periodic. The data log from one thermocouple showed one of those bursts rose approximately 1.8°C in 20 s.

Figure 4. Spalled portion of Pd cathode after treatment with protocol. This experiment included glass beads on the cathode. Such spalling was only seen when glass beads were used.

The data logs from the experiment with sodium metasilicate showed a temperature transient, albeit a small one.

Figure 6 shows a "volcanic site" observed during SEM analysis of a gold electrode after treatment with the protocol. Interestingly, gold does not form a hydride at or near the boiling point of water, so that means its metal lattice is not being loaded with either hydrogen or deuterium atoms during the protocol. That suggests that the reaction is not driven by the interaction of those atoms while they are immobilized in the metal lattice and allowed to interact in ways that are stochastically impossible as gases, as was postulated by Fleischman and Pons to cause "cold fusion". Something else is going on with this protocol besides that reaction.

We have performed three experiments where the BES analyses of the electrodes show the presence of silver, a possible transmutation product of palladium. We have also observed in SEM examinations that the apparently transmuted material is often associated with different morphologies than the surrounding palladium. Figure 7 shows a SEM photo and its accompanying BES spectra of the treated surface of a palladium cathode; silver is shown to be present on the surface. A similar BES analysis of the interior of the untreated interior of the wire did not show the presence of silver.

BES analysis also detected the presence of nitrogen, fluorine, aluminum, and chlorine during various experiments, which could be transmutation products of elements added to the reactor, assuming these elements undergo Beta decay.

The cathode from one of those three experiments was also analyzed using Auger spectroscopy. That analysis showed the presence of nitrogen, aluminum, chlorine, calcium, and zinc. Each of these also could be a transmutation product of one of the elements added to the reactor, again assuming those elements undergo Beta decay

The results of the BES and Auger analysis are summarized in Table 1. The left-hand column shows the elements added to the reactor. The next column shows the possible transmutation products of those initial elements. Both BES and Auger analysis have detection limits of 1%, and both kinds of analysis show five possible transmutation products to be present. Magnesium is both an ingredient and a possible transmutation product, so its presence is not indicative of a transmutation. The fields that are labeled "NA" indicate that the respective method does not detect the presence of those elements with lower atomic numbers. Na and Cu are shown in red because they were not listed as ingredients in

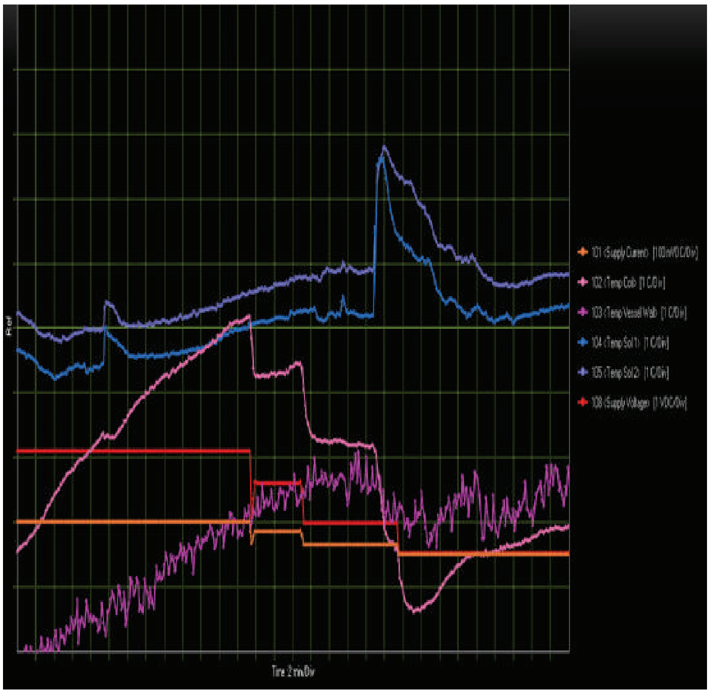

Figure 5. Experiment #3 used Pd electrodes, lithium metasilicate, and lithium sulfate as an electrolyte in predominately heavy water. Glass beads were strung onto the cathode.

the anionic silica hydride; they were presumably contaminants (Table 1).

Table 1. Shows the ingredient elements in the protocol, the possible transmutation products if the transmutation consists of a neutron converting to a proton, and which elements were detected in EDS and Auger analyses

Ingredient	Transmutation	EDS	Auger
^{1}H	^{2}He	NA	NA
^{3}Li	^{4}Be	NA	
^{6}C	^{7}N	Available	Available
^{8}O	^{9}F	Available	
^{11}**Na**	^{12}Mg	Available	Available
^{12}Mg	^{13}Al	Available	Available
^{14}Si	^{15}P		
^{16}S	^{17}Cl	Available	Available
^{19}K	^{20}Ca		Available
^{29}**Cu**	^{30}Zn		Available
^{46}Pd	^{47}Ag	Available	

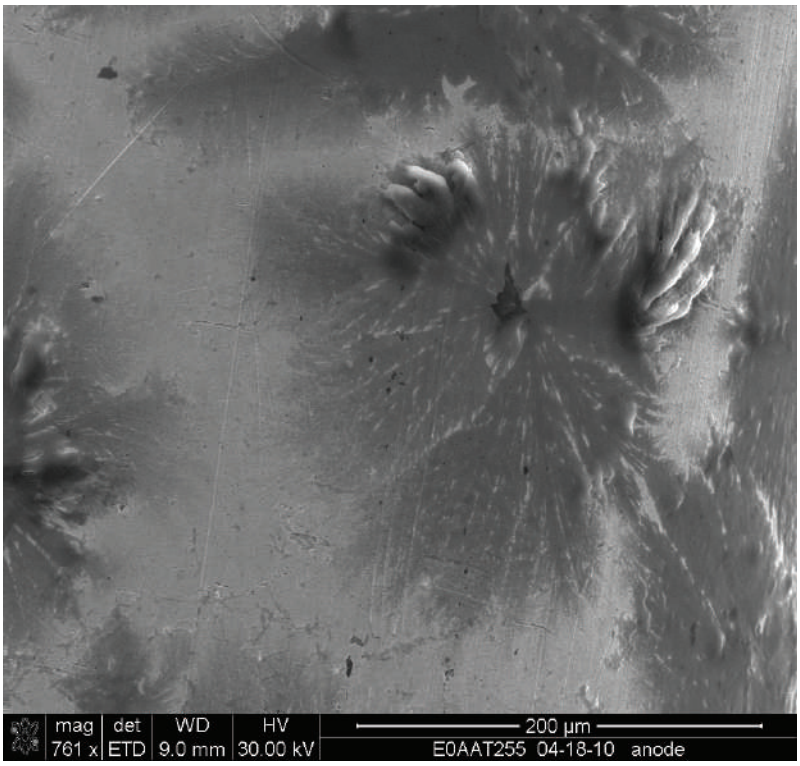

Figure 6. SEM photo showing a "volcanic site" on a gold electrode.

It is important to note that we only saw temperature transients when the solution was within 6°C of the solution's boiling point, and more typically when it was within 2°C of it. We suspect the reaction we are reporting either only takes place at or near the boiling point or only takes place robustly enough to generate the temperature transients

Also, interestingly, experiments conducted with red and blue LEDs instead of white ones did not result in a reaction.

Finally, three things were found to inhibit the reaction: (1) rubber in the reactor, (2) direct contact with Teflon®, and (3) using ultra-pure palladium (99.999%) for electrodes.

4. Conclusion

We conclude that the experimental results above show an exothermic reaction can be generated when a solution including a lithium salt and a silicate is stimulated with modulated electrical and photonic stimuli provided by white LEDs. The reaction presents sharp temperature transients. Examination of the electrodes after the experiments showed evidence of thermal stress in spalling and of material flow in volcanic sites.

Anomalous temperature excursions were observed when using soluble anionic silica hydride, silica (glass or quartz) containers and not when using stainless steel or Teflon®. The reaction was also more robust when glass beads were threaded onto one of the electrodes and when glass thermocouple wells were substituted for steel ones. Hence, the presence of silica in the reactor proved to be a critical to yielding anomalous heat.

The experiments demonstrated similar heat signatures when conducted with lithium metasilicate, anionic silica

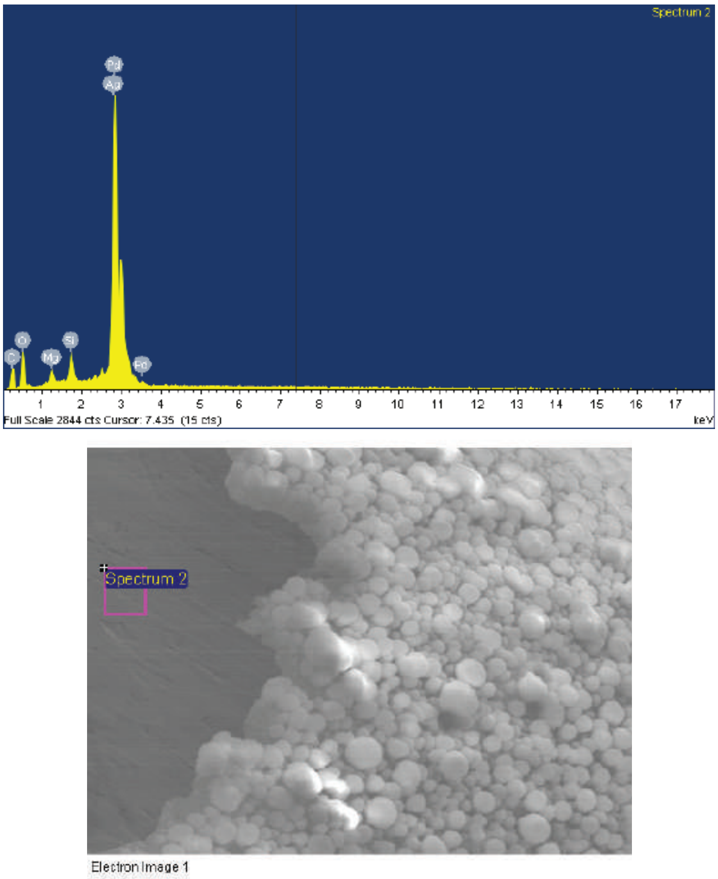

Figure 7. SEM photo of treated surface of a Pd electrode and accompanying EDS analysis showing the presence of Ag, a possible transmutation product of Pd.

hydride, and sodium metasilicate, with the reactions showing a stronger reaction with the silicates in that order. We believe those results support the reasoning that silica is critical to the reaction and that lithium silicate promotes a stronger reaction.

We detected evidence of possible transmutation products of as many as six different elements using two different analytic techniques, EDS and Auger analysis. Taken together, the data suggest that our protocol may have induced nuclear reactions on numerous occasions.

References

[1] Cory J. Stephanson and G. Patrick Flanagan, Synthesis of a novel anionic hydride organosiloxane presenting biochemical properties, *Int. J, Hydrogen Energy* **28** (2003) 1243–1250; Also available online at http://www.ressourcement.info/Synthesis%20of%20a%20novel%20anionic%20hydride%20organosiloxane.pdf

[2] Cory J. Stephanson and G. Patrick Flanagan, Quantitative Analysis of Membrane Diffusion Kinetics and Surface Tension Differentiation by a Colloidal Silicate Mineral, available online at http://www.phisciences.com/20050516CE.pdf

[3] An early version of the protocol using anionic silica hydride is described more fully in United States Patent 7,442,287. It is available online at uspto.gov.
[4] The version of the protocol using lithium metasilicate is described more fully in United States patent application 20110174632. It is available online at us pto.gov.