

Synthesis and characterization of an Iron pico-hydride. A permanent electric dipole with high enthalpy of formation

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Abstract

Chemical reactions result from the outside shell electrons of the reacting species being shared in various types of combinations. Typical distances involved are tens of nm, resulting in binding energies typically in the order of hundred of kJ/mole(eV/atom) . The synthesis of a novel "deep chemical compound" formed from Iron and Hydrogen has been achieved. The measured enthalpy of formation is some 390 MJ/moleFe and the distance between the Hydrogen proton and the Iron nucleus is some 3 picometers, hence the proposed name: Iron Pico-hydride. This compound is a permanent electric dipole of atomic size. Pico-hydrides could thus play a role in room temperature superconductivity. The synthesis is compatible with the standard model.

Key words: Pico-chemistry. Room Temperature Superconductivity. Clean and abundant energy source.

1.Introduction:

It has been shown [1], that a purely electro-magnetic binding could be formed, under appropriate conditions, between a transition metal M and a hydrogen atom. In this type of binding, the proton of the hydrogen atom is at picometer distance from the transition metal nucleus (at the level of the K shell of M), its electron oscillating around this proton. The resulting specie has been proposed to be called a pico-hydride and will be denoted M-pH. In this study, M is Iron and the corresponding pico-hydride is Fe-pH. The enthalpy of formation of Fe-pH is expected to be in the order of magnitude of the energy of the iron K shell (7.1 keV/atom or 681 MJ/mole). Fe-pH is a "deep chemical compound".

The conditions of the synthesis of Fe-pH (that occurs at high temperature: >1100°C) will be first described.

The apparatus (calorimeter) used to measure the energy released during the synthesis and the experimental data it yields will then be presented.

The analysis by ICP-MS-HR (Thermo ELEMENT 2XR) of the samples (blank and treated) will then be discussed and a first estimation of the enthalpy of formation of Fe-pH will be given. The MS analysis can be interpreted as Fe-pH being a permanent electric dipole of atomic size.

2.Conditions of the synthesis of Fe-pH

For any deep chemical compound to be formed between Fe and hydrogen, two barriers have to be overcome: the Lenhard-Jones barrier and the Coulomb barrier.

Consider a reacting medium where Iron is present in the solid state and Hydrogen together with the vapor of an alkaline metal (Na or Li) in the gas phase. Such a reacting medium is described in **Fig. 1**:

Sodium and Lithium have a low ionization energy (Na 5.16 eV and Li 5.41 eV) and provide at 1100°C the electrons required for the reaction to proceed. Part of the Hydrogen is trapped at the surface of the Nickel in the form of a proton, its electron being shared with the conduction band of the Nickel.

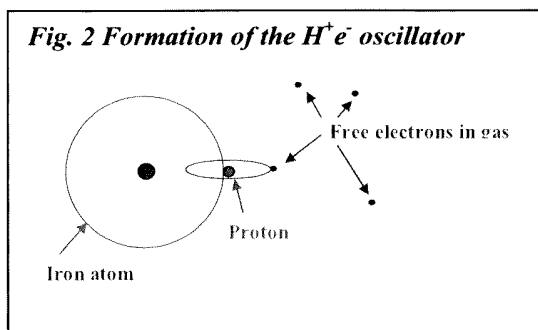
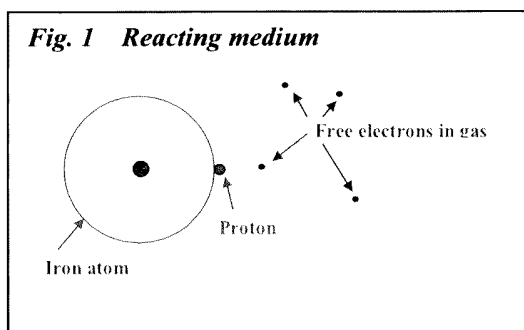


Fig. 2 shows how an electron in the gas phase is attracted by the hydrogen proton trapped at the surface of the iron, resulting in the formation of an H^+e^- oscillator.

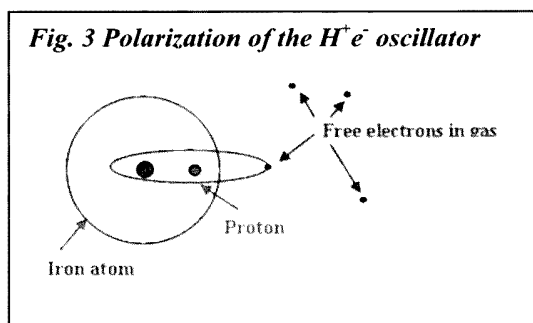


Fig. 3 shows how the dissymmetry of positive charges (Iron 26 and Hydrogen 1) results in the polarization of the H^+e^- oscillator that becomes a polarized $(Fe-H)^+e^-$ dipole. The proton is attracted towards the Iron nucleus, thus overcoming the 2 barriers.

Fig. 4 Final step of the Fe-pH synthesis

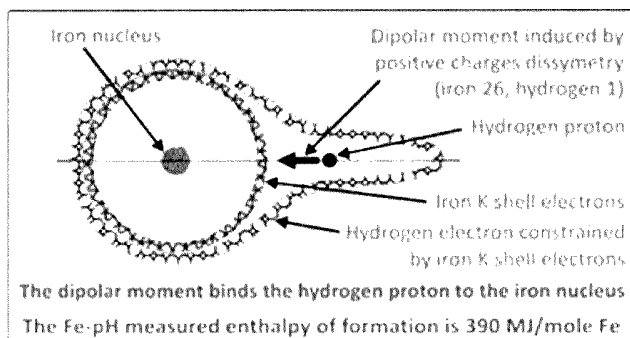


Fig. 4 shows the final state of the Fe-pH synthesis. **The K shell of the iron is a limit for the progression of the proton towards the Iron nucleus: the dipolar moment vanishes when the proton goes towards the iron nucleus and the proton is attracted back by the K electrons of the iron.** A crude quantum mechanical treatment of the synthesis is presented in [1].

The synthesis of Fe-pH has been carried out under following conditions:

-Temperature from 1075 to 1150°C

-Gas phase composition: Total pressure 7 bar. Hydrogen partial pressure 3 bar and Sodium partial pressure 4 bar in the case of Sodium. Hydrogen partial pressure 6.6 bar and Lithium partial pressure 0.4 bar in the case of Lithium. Sodium is introduced (at room temperature) in the form of metallic Sodium. Lithium is introduced (also at room temperature) in the form of LiAlH₄ powder. The detailed experimental procedure is given below.

3.Measurement of the heat released by the synthesis of Fe-pH:

Measuring the heat released by a reaction at high temperature (1100 °C) is a difficult task. It is even more difficult when this reaction proceeds on long periods of time (days). A simple, robust and reliable calorimetric system meeting these requirements has been developed. This calorimetric system is a low cost, high temperature DSC type device: a high precision is not necessary for this first exploratory study.

3.1 Principle of the heat measurement:

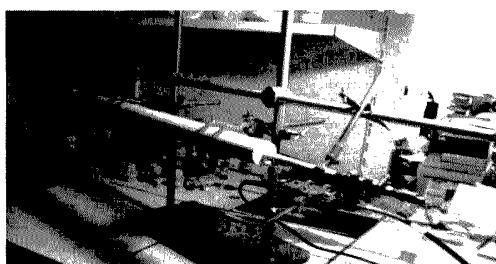
The heat measurement is based on the heat exchange between 2 coaxial cylinders made from alumina 99.9 %. The inner cylinder is 8x12 mm diameter and 85 cm long. The outer one is 38x45 mm diameter and 50cm long. The relative position of these 2 cylinders is kept fixed (compressed vermiculite disks and High Alumina Cement). The assembly is inserted in the tube (50 mm inside diameter) of a horizontal heater able to operate up to 1200°C with the heating zone 45 cm long and the total length 75 cm (ThermConcept ROS 50/450/12). The inner tube is connected (O rings and clamp collars) to a manifold allowing the inner tube to be submitted either to a primary vacuum (10⁻² mb) or to a regulated hydrogen pressure. The connection is outside the heater and can withstand 10 bar H₂.

Inside the inner tube are inserted two small cells (diameter 6x7.5 mm and length 8 cm). These cells are maintained in a symmetrical position on both sides of the longitudinal symmetry axis of the heater (8 cm long alumina tube, diameter 6 mm between the 2 cells and 4x2 mm alumina tubes between each cell and the corresponding extremities of the inner tube.

One of the cells contains the powder to be tested and the other one a reference powder. Both cells are made from the same material. Two materials were tested: AISI 304 L steel and Alumina 99.9 %.

The differential thermal signal between the two cells is measured by 4 thermocouples (50 cm long): 2 thermocouples are inserted in a 2x4 mm alumina tube fixed (alumina cement) against the outer surface of the 8x12 mm inside tube and 2 in a similar 2x4 alumina tube fixed against the inner surface of the 38x45 mm outer cylinder. The tips of the thermocouples are positioned so as to face the middle of each cell. *Fig.5* below is an overall view of the calorimeter during construction (centering the 2 tubes)

Fig.5 Overall view of the calorimeter



The heater containing the calorimeter is placed in a ventilated cabinet (air extraction by a fan in the cabinet's roof) that contains the required manifold (Pressure regulated H2 source/Vacuum). The laboratory is air conditioned but the temperature is not regulated.

3.2 Temperatures measurement:

The temperatures T_c (°C) of the cold junctions of the thermocouples are measured (PT 100). From these measurements and from the measurements of the potential differences V of the thermocouples, the 4 temperatures T (°C) of interest can be computed. Use has been made of well known norms, namely CEI 584.1 (1995) for N thermocouples. Following correlations, derived from CEI 584.1, were used. C_{cj} is the cold junction voltage correction. (1) and (2) apply for N thermocouples:

$$C_{cj} = 1.828 \times 10^{-5} T_c^2 + 2.587 \times 10^{-2} T_c - 4.588 \times 10^{-4} \text{ mV} \quad (1)$$

$$T = 2.4 \times 10^{-3} (V + C_{cj})^3 - 0.2063 (V + C_{cj})^2 + 31.195 (V + C_{cj}) + 23.846 \text{ °C} \quad (2)$$

Expressed in voltage, the precision on temperature differences up to 10°C in the range 700 to 1150°C is a few μV .

N thermocouples were used, because K thermocouples have a too important uncontrolled drift with time. The treatment of the data eliminates experiments with unacceptable differential drift of the thermocouples. N thermocouples can be safely used in the temperature range of interest (700 to 1100°C).

3.3 Power calculation:

W_A is the power flowing from the active cell that contains the active powder to be tested. W_R is the power flowing from the reference cell that contains an inert reference powder.

The total power W_A exchanged between the outer surface S_1 of the 8x12 mm tube, outer radius R_1 (that contains the active cell) and the inner surface S_2 of the 38x45 mm tube, inner radius R_2 is:

$$W_A = \frac{2\pi}{Ln(R_2/R_1)} \lambda L (\Theta_1^A - \Theta_2^A) + LS_1 T_r \sigma ((\Theta_1^A)^4 - (\Theta_2^A)^4) \quad (3)$$

Θ_1^A and Θ_2^A are the surface temperatures (K) of the 8x12 mm and of the 38x45 tubes respectively.

The upper-script A denotes the active cell. Similar equation holds for the reference cell, A being replaced by R . L is the length of the powder in the cell (edge effects are neglected). The variation of the air thermal conductivity λ with temperature Θ (K) has been approximated by a second degree polynomial:

$$\lambda = 0.024083 + 7.78 \times 10^{-5} (\Theta - 273) - 3.68 \times 10^{-8} (\Theta - 273)^2 \quad (\text{W/m.K}) \quad (4)$$

σ is the Boltzmann constant and T_r is the transmission coefficient, relating the radiation transfer to the geometry and material of the tubes:

$$T_r = \frac{1}{\frac{1}{\varepsilon_1} + \frac{1}{\varepsilon_2} \frac{S_1}{S_2} - \frac{S_1}{S_2}} \quad (5)$$

The emission coefficients have been taken equal to 0.9 (99.9 % alumina at 1100°C)

Powers are counted positive when flowing from the inner tube to the outer one. When no reaction occurs, these powers are negative (the heat flows from the heater to the outer tube, then to the inner tube and finally to the surrounding air).

3.4 Data monitoring and processing:

Data are monitored using an AOIP LS 20 monitoring system. The precision on the voltage measurements is 1 μ V. Acquisition time has been varied from 5 seconds to 2 minutes.

Data are processed using a spreadsheet.

4.Heat measurement experimental results:

4.1 Summary of the experiments run

5 experiments were run, that are summarized in following *Table 1*

<i>Table 1 Summary of experiments run</i>				
Experiment number	Active cell	Reference cell	Electron source	Cells material
1	Na + Fe + SiC	Na + SiC	Sodium	304 L steel
2	LiAlH ₄ + Fe + SiC	LiAlH ₄ + SiC	Lithium	304 L steel
3	Na + SiC	Na+ SiC	Sodium	Alumina
4	Na + SiC	Na + SiC	Sodium	304 L steel
5	Na + Fe +SiC	Na+ SiC	Sodium	Alumina

4.2 Experiment 1: no thermocouples drift

4.2.1 Active and reference cells loads:

0.259 g of Sodium (Alfa Aesar L 13285 Lot 10185964) is compressed in the active cell at one of its extremities that had first been closed by a 0.5 cm long ceramic felt stopper. A physical mixture of 1.087 g of Iron (Alfa Aesar 000170 LOT A29123 <10 μ m) and 1.087 g of Silicon Carbide (Alfa Aesar 43 332 Lot Q10B002 <44 μ m) is then introduced in the tube that is finally closed by a ceramic fiber stopper. In a similar way, 0.260g of Sodium and 1.704 g of Silicon Carbide are introduced in the reference cell. Both cells are then introduced in the inner tube of the calorimeter, the ceramic felt stoppers facing each of the extremities of this tube.

4.2.2 Experimental procedure:

The calorimeter is then introduced into the heater (both extremities of the heater tube being then closed by ceramic fiber stoppers), connected to the manifold and the experiment is left overnight under vacuum (10^{-2} mb) at room temperature. Hydrogen is then introduced in the reactor at a pressure of 7 bar (gauge). The regulating point of the heater is then set at 1075°C and the parameters are registered every 5 seconds. W_A and W_R are then computed as a function of time using a spread sheet (Note that the small manifold background leakage {0.03 cm³/s: one electromagnetic safety valve and 14 conical steel fittings} maintains the total reactor pressure at 7 bar during Sodium vaporization).

4.2.3 Start of the thermal effect:

The thermal effect of interest starts when the heater temperature reaches 1050°C. **Fig.6** below shows the evolution of W_A and W_R with time.

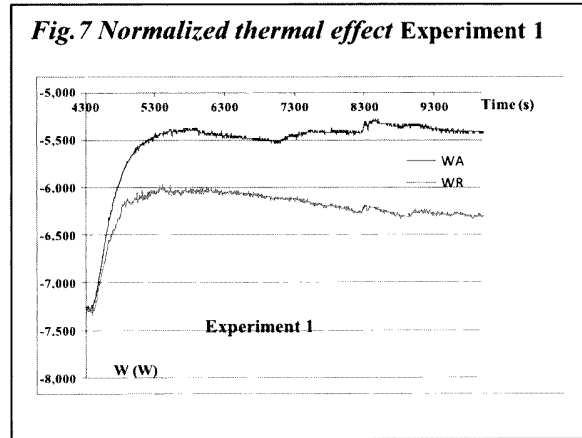
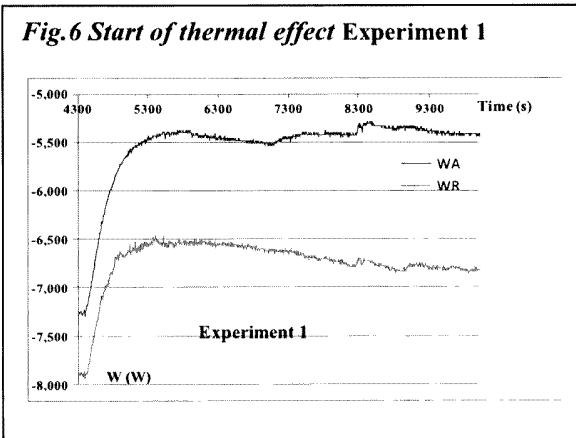
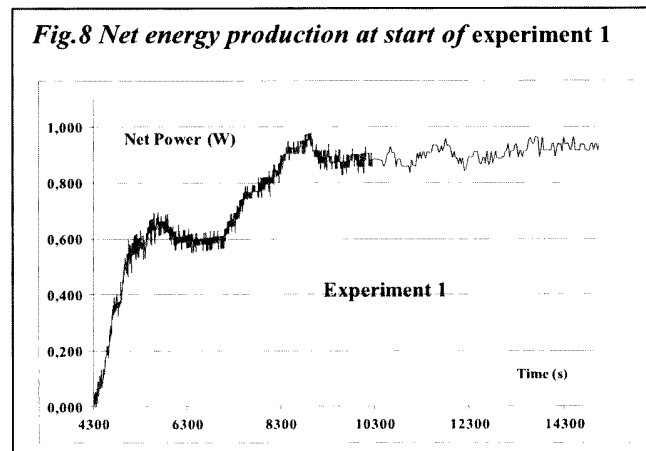


Fig 6 shows that the increase of W_A is higher than that of W_R and that this increase starts for different values of W_R . This is attributed to the inherent dissymmetry between the 2 cells and is taken into account by normalizing the cells (using a normalization coefficient D). For $D=0.924$, the net energy production $W = W_A - D W_R$ is 0 at time 4300s (**Fig. 7**) and then increases with time as can be seen on **Fig.8**



Note on **Fig.8** that the acquisition time has been changed from 5 seconds to 2 minutes at time 10,300 seconds.

The synthesis of Fe-pH last on long periods of time (this experiment was shut down after 9 days and the formation of Fe-pH was far from being completed).

The question thus arises of the stability of D with time.

4.2.4 Trace of the experiment, stability of D with time:

The stability of D is checked using following procedure. W_A and W_R are sampled once per day (at 5 to 6 am) and the net power W is calculated as $W = W_A - D W_R$ with $D = 0.924$. W_A is then plotted against W_R , introducing in the correlation $W_A(0) = \text{mean value } (W_A - D W_R) = 0.598 \text{ W}$

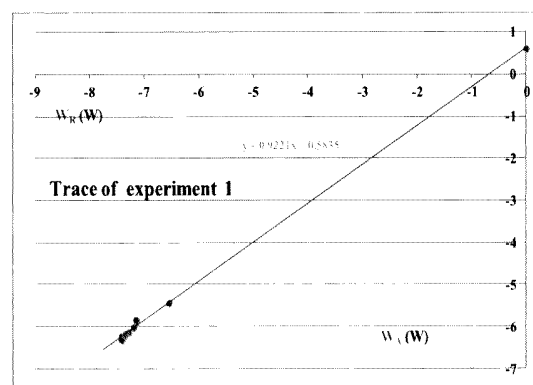
Table 2 below shows that the linear correlation $W_A = f(W_R)$ returns for D the value 0.922 (slope of the straight line) and for the intersection with the W_A axis, the value 0.584 W

It can be seen on **Fig.9**, that the trace of the experiment shows a very small scatter, meaning that the calorimeter drifts coherently as a whole, D being constant all over the experiment (the correlation coefficient of the linear regression yielding D is 0.9994)

Table 2 Correlation $W_A=f(W_R)$ Experiment 1

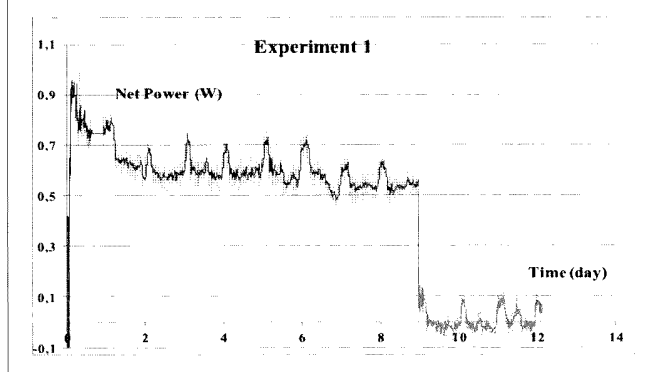
		D	Calculation	Correlation
		Mean power	0.598	0.922
Date	W_R	W_A	$W_A - D * W_R$	
	0.000	0.598		
09/07/2015	-6.542	-5.446	0.599	
09/07/2015	-7.151	-5.846	0.761	
10/07/2015	-7.195	-6.023	0.625	
11/07/2015	-7.287	-6.146	0.587	
12/07/2015	-7.344	-6.188	0.597	
13/07/2015	-7.360	-6.220	0.580	
14/07/2015	-7.408	-6.242	0.603	
15/07/2015	-7.386	-6.257	0.568	
16/07/2015	-7.410	-6.314	0.532	
17/07/2015	-7.413	-6.325	0.524	

Fig. 9 Trace of experiment 1



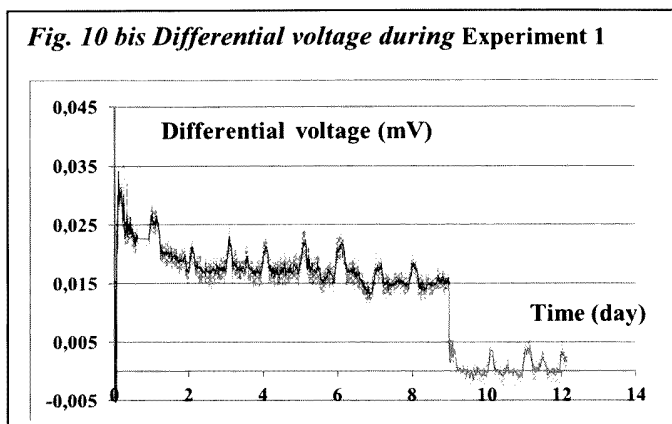
After 9 days, the reaction was stopped by keeping the heater set point at the same value, the inner tube being put under vacuum (10^{-2} mb). This situation was kept for 4 days to get the base line. Note that under vacuum, the normalization coefficient changes (0.848, assuming that the Fe-pH synthesis-and the associated heat release-no longer proceeds in the absence of Hydrogen). After 4 days, the experiment is cooled down to ambient under vacuum. The overall experiment is summarized in **Fig.10**.

Fig. 10 Net power during Experiment 1



The peaks observed are due to the daily variations of the laboratory temperature (air conditioning but no temperature regulation). After cooling down, the inner 8x12 mm tube is filled with water that recovers most of the sodium in the form of NaOH (or Li in the form of LiOH). The cells are then recovered and the active sample is prepared for analysis (see below).

Fig. 10 bis Differential voltage during Experiment 1



For comparison purpose, the differential voltage of the thermocouples used to measure Θ_1 and Θ_2 for the active and reference cells (Equation (3) p.4) have been used to calculate the DSC signal of the system (mV). The shape of the signal is comparable to the shape of the response in W . The dissymmetry coefficients are similar (0,81 and 0,76 for 7 bar H_2 and vacuum).

The calibration constant of the DSC system is thus 0.034 mV per W .

The total energy released by the reaction is calculated as $H = \int_0^T W(t) dt$, T being the total duration of the synthesis.

4.3 Experiment 2: thermocouples drift

4.3.1 Active and reference cells loads:

A physical mixture of 0.031 g of $LiAlH_4$ (Alfa Aesar A 18116 Lot 21555946), with 2 g of Iron and 2 g of SiC (same origins as experiment 1) is introduced in the active cell after one of its extremities was closed by a 0.5 cm long ceramic felt stopper. The other extremity is closed by a ceramic fiber stopper.

A physical mixture of 0.028 g of $LiAlH_4$ with 3.304 g of SiC is introduced, according to above procedure in the reference cell.

4.3.2 Experimental procedure:

The procedure is the same as for **experiment 1** except that the regulating point of the heater is set at 1150°C instead of 1075°C.

4.3.3 Trace of the experiment, non stability of D with time:

At start of the experiment, the normalization coefficient is $D = 0.732$. But in sharp contrast with **experiment 1**, **Fig.11** bellow shows that the trace of the experiment is erratic, D continuously increasing with time, resulting in a non valid calculation of the net power W with time (**Fig.12**), when keeping $D = 0.732$ for the whole experiment. After some 15 days, one of the thermocouples failed.

Fig.11 Trace of experiment 2

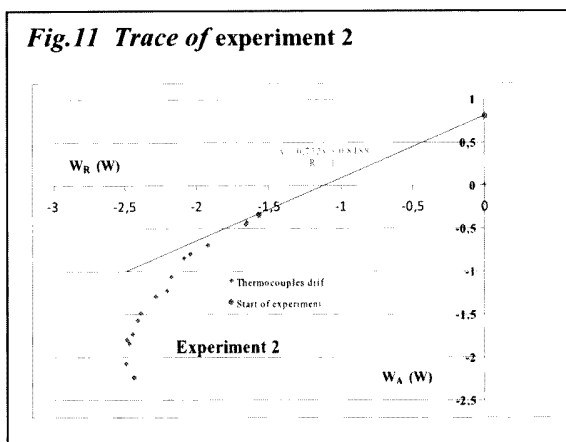
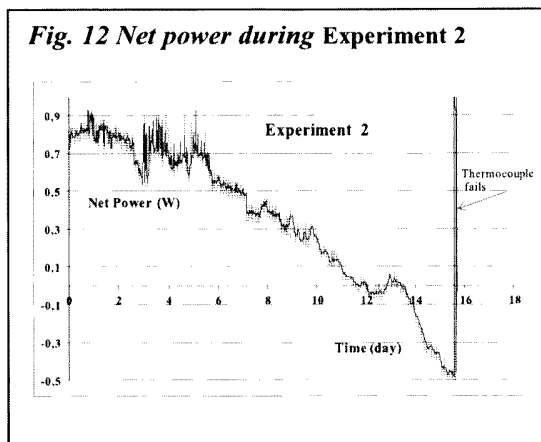


Fig. 12 Net power during Experiment 2



Only for comparison purpose the net power has been recalculated, taking into account the variation of D with time. A mean net power of 0.510 W during 15 days was found. Upon dismantling of the experiment, it was observed a deep scaling of the tip of the envelope of the thermocouple measuring the inner 38x45 cylinder temperature of the active cell. It can however safely be concluded that the synthesis lasted at least 12 days.

4.4 Blank experiments 3 and 4:

It was observed that the 304 L steel of the cells reacted in the conditions of the experiment, resulting in small bright golden yellow scales on the metal. In order to quantify this effect, 2 blank experiments were run: **Experiment 3** was run with Alumina cells filled with Sodium and SiC. The trace of the experiment returns $D = 0.434$ (correlation factor 0.9871) and net power mean value W (intersection with W_A axis) 0.002 W. **Experiment 4** was run with 304 L steel with same cells load. The trace of the experiment returns $D = 1.335$ (correlation factor 0.9863) and net power mean value W (intersection with W_A axis) = - 0.084 W, indicating a small dissymmetry between the two 304 L steel cells reactivity.

4.5 Experiment 5:

Experiment 1 was duplicated with Alumina cells, the loads in the cells being the same as **Experiment 1**. **Experiment 5** yielded a mean net power of 0.450 W over 9 days, the difference with **experiment 1** being explained by the reactivity of the 304 L steel used in **experiment 1**. From this result it can be estimated that some 0,330 g of the steel of the cell reacted thus increasing the power measured in **Experiment 1**.

4.6 Heat measurement discussion:

4.6.1 Experiment 1 (Sodium + Iron) resulted in a mean net power W of 0.590 W and a total energy production $H = 23.45$ MJ/mole Fe (14.83 MJ/ moles Fe+Na) during 9.1 days. Assuming an enthalpy of Fe-pH formation of 7.1 keV/atom Fe (or 681 MJ/Mole Fe), the conversion would have been 34,400 ppm (3,780 ppm/day).

4.6.2 Experiment 5 (sodium+Iron alumina cells) resulted in a mean net power of 0.450 W and a total energy production $H = 18.03$ MJ/MoleFe (11.40 MJ/ moles Fe+Na) during 9.1 days. Assuming an enthalpy of Fe-pH formation of 7.1 keV/atom Fe (or 681 MJ/Mole Fe), the conversion would have been 26,470

ppm (2,910 ppm/day). The difference with *experiment 1* can be explained by some 0.330 g of the 304L steel of the cell having reacted in *experiment 1*.

4.6.3 Experiment 2 (Lithium + Iron), corrected for comparison purpose, resulted in a mean net power W of 0.510 W and a total energy production $H = 18.46$ MJ/mole Fe (18.04 MJ/moles Fe+Li) during 15 days. The conversion would have been 27,080 ppm (1,805 ppm/day).

The Sodium or Lithium are recovered as explained above. It is observed that the parts of the alumina tubes in contact with the gas phase at high temperature show a very superficial grayish/blackish color on some places. This can be attributed to the formation of a superficial thin layer of sub-stoichiometric alumina oxide.

The difference in conversion per day between Na and Li as catalysts, is in line with the predictions of the Saha equation (Sodium at 1075°C and 4 bar partial pressure generates more electrons than Lithium at 1150 ° C and 0.6 bar partial pressure).

5. Analysis by ICP MS HR of the samples from experiment 1:

5.1 Preparation of the samples. Analytical tools:

The reaction product was first extracted from the active cell (304 L steel) with much care to avoid pollution from the 304 L steel from the cell, crashed in an agate mortar and separated in 3 fractions: **1** < 30µm (64.3 w%), **2** >30µm and <80µm (31.7 w%) and **3** >80µm (7.3 w%). The first 2 fractions looked homogeneous (binocular magnifier). The 3rd one was very heterogeneous. The total sample weight recovered represents 85% of the initial Fe+SiC load.

In order to get a representative liquid sample of the reaction product, the mineralization (12N HNO₃ MS grade) was done in the following way:

- fraction **3** was completely dissolved in 50 ml and diluted 200 times resulting in a concentration of 7.2 mg/l

- 14.265 mg of fraction **2** were dissolved in 50 ml and diluted 5 times, resulting in a concentration of 57.06 mg/l.

- 11.574 mg of fraction **1** were dissolved in 50 ml and diluted 2 times, resulting in a concentration of 115.75 mg/l

Mixing the 3 solutions results in a **Composite** mineralized sample representative of the total reaction product sample. Note that most of the Silicon Carbide present in the 3 fractions is not dissolved (ICP-AES and MS spectra).

The blank Iron was a sample of the virgin Alfa-Aesar Iron and will be referred to as **Iron**.

The analytical tools used were ICP AES (Thermo iCAP 6500) and ICP MS HR (Thermo ELEMENT 2XR). A resolving power round 10,000 was used (thus resolving Ar-O interferences) and the scan was from mass 23 to mass 68.

The mechanical preparation of the treated sample and the interpretation of the MS Spectra were done in S*PIC*E. The preparation of the **Composite** and the **Composite** and **Iron** spectra were done in "Institut des Sciences Analytiques" UMR 5280 Equipe inorganique Lyon.

5.2 Interpretation of the Composite and Iron spectra:

5.2.1 Isotopic composition:

Table 3 below gives the Iron isotopic compositions found for the **Iron** and **Composite** samples respectively (and the natural Iron composition)

Table 3 Isotopic composition of Iron and Composite

		⁵⁴ Fe	⁵⁶ Fe	⁵⁷ Fe	⁵⁸ Fe	Total counts
Natural	Mass	53.938812	55.934839	56.935396	57.933277	
Iron	%	5.9	91.72	2.1	0.28	
Iron	Mass (meas.)	53.9456	55.9400	56.9403	57.9386	
	%	5.98	91.51	1.94	0.56	12,275,755
Composite	Mass (meas.)	53.9456	55.9400	56.9403	57.9386	
	%	5.06	92.24	2.32	0.38	11,398,997

The **Iron** composition is closed to that of Natural Iron. By contrast **Composite** shows, when compared to **Iron**, a sizeable decrease (-15.4%) at mass 54, a stability (+0.8%) at mass 56 and a sizeable increase (+19.6%) at mass 57. For further analysis, **Composite** has been normalized to **Iron** (1.0769). In the following, **N.Composite** refers to normalized **Composite**.

5.2.2 Interpretation at mass 55:

Table 4 gives the required data for mass 55 interpretation and for the determination of ⁵⁵X, a sizeable signal at mass 55 in **N.Composite** that is not in **Iron**.

Table 4 Interpretation at mass 55

		²³ Na	⁵² Cr	⁵⁵ Mn from cell	⁵⁵ X
Natural	Mass	22.989767	51.940509	54.938047	
	%	100.0	83.79	100.0	
Iron	Mass (meas.)	22.9929	51.9672		
	Total counts	28,740	46,377		
N.Composite	Mass (meas.)	22.9922	51.9672		
	Total counts	109,080	59,089	1,686	42,205

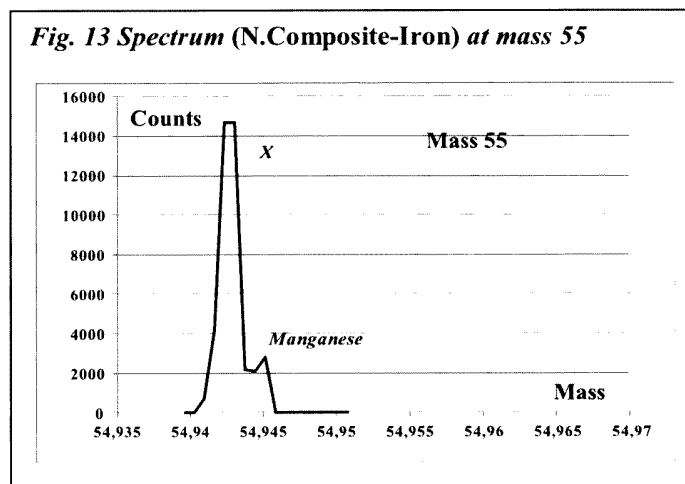
A very small pollution of **N.Composite** by the 304 L steel of the cell has occurred. The increase observed at mass ^{52}Cr between **N.Composite** and **Iron** is $59,089 - 46,377 = 12,712$ counts. The maximum pollution of **N.Composite** by Mn is thus: $(12,712/0.8379) \times (2/18) = 1,686$ counts (304 L steel contains less than 2% Manganese and has a chromium content between 18 and 20 %)

The pollution of **N.Composite** by Manganese (1,686 counts) cannot explain the total counts observed at mass 55 (42,205).

Counts at mass 23 show that Sodium was present in **Iron** (# 2300 ppm). This figure increases up to 8800 ppm in **N.Composite**, confirming that most of the sodium does not react and is recovered when washing the calorimeter with water (a small part may have been left deposited on the iron surface).

5.2.3 What is ^{55}X :

Fig. 13 below is the spectrum (**N.Composite-Iron**) at mass 55



The size of the small peak at the right side of the ^{55}X peak is in line with the counts that can be expected from the pollution of the sample by Mn (1,686 counts).

This peak is attributed to manganese, thus allowing a normalization of the masses.

The Manganese peak is at measured mass 54.9452. The mass of ^{55}Mn is 54.938047. The mass shift is thus: $+ 0.0072$. The measured mass of ^{55}X being 54.9424 its normalized mass is 54.9352. The expected mass for $^{54}\text{Fe-pH}$ is 54.9474. Thus, as such, ^{55}X does not match $^{54}\text{Fe-pH}$.

Among the radioactive species at mass 55 (^{55}Cr , ^{55}Co , ^{55}Ni and ^{55}Fe), ^{55}Fe has the mass closest to that of ^{55}X (54.9383 compared to 54.9352). But the measured radioactivity of the solid sample extracted from the cell and from which **Composite** has been obtained is within the background: 8cps (measured by a SAPHYMO CoMo 170 contaminamètre α , β particles and X, γ photons). By contrast a 157 Bq ^{55}Fe sealed

source returns 57 cps. This source –CEA DAMRI LMRI- was 1,320 Bq on 2007/03/06 and the measurements were on 2015/08/20.

The half life $T_{1/2}$ of ^{55}Fe is 2.737 year or 86,314,000 s. The disintegration constant is thus $\lambda = \ln 2 / T_{1/2} = 0.693 / 86,314,000 = 8.03 \times 10^{-9} \text{ s}^{-1}$. The time elapsed between 2007/03/06 and 2015/08/20 is round $t = 2.648 \times 10^8 \text{ s}$. The activity at the time t of the measurement is thus $A(t) = 1320 \exp(-\lambda t) = 157 \text{ Bq}$ (Note that, for solid angle reasons, the detector measures less than half of the total emission)

Should ^{55}X be ^{55}Fe , the total number of ^{55}Fe atoms contained in the solid sample extracted from the cell would be: $N = 6 \times 10^{23} \times 34,400 \times 10^{-6} \times 0.059 \times 1.087 / 53.939 = 2.45 \times 10^{19}$ (taking for the conversion the value obtained in 4.6.1, for the weight of Iron the value of 4.2.1 and known values for iron mass and isotopic composition).

The activity would then be $dN/dt = \lambda N = 2.45 \times 10^{19} \times 8.03 \times 10^{-9} = 1.97 \times 10^{11} \text{ Bq}$. Such a level of activity could not have been missed.

The object $^{54}\text{Fe-pH}$ under consideration is far from being a usual atom. Compared to its counterpart (^{55}Co), its positive charge is complex: a positive nucleus with charge 26 and some 10 fm diameter, and a positive charge 1 at a few picometers, bound to the Iron nucleus by an oscillating electron. It is possible that this "atom" with a "composite nucleus", is a permanent electrical dipole resulting from the modification of all its electron shells by this "composite nucleus". The Fermi surface of a $^{54}\text{Fe-pH}$ crystal is likely to be drastically different from that of a crystal of Cobalt (the void cat-ion model certainly does not apply and this might have an important incidence on its physical properties, including electrical conductivity).

It is then conceivable that the single charged $^{54}\text{Fe-pH}^+$ permanent dipole could behave during its time of flight through the electric and magnetic fields of the MS machine as if its apparent transient effective electrical charge is slightly higher than e . The mass spectrometer sorts atoms according to their m/e ratios. If the apparent transient effective charge of $^{54}\text{Fe-pH}^+$ is e increased by 223 ppm, then the actual mass of ^{55}X is 54.9474, which is the mass of $^{54}\text{Fe-pH}$.

5.2.4 Conversion of ^{54}Fe to $^{54}\text{Fe-pH}$

Using **Table 3** and **4**, this conversion is: $39,866 \times 10^6 / (39,866 + 621,711) = 60,260 \text{ ppm}$

5.2.5 Interpretation at Mass 57:

The interference with ^{57}Fe prevents a detailed analysis. The only thing that can be said is that there are more counts in **N.Composite** than in **Iron** and that this excess of counts tends to concentrate on the left of the ^{57}Fe peak of **Iron**.

6. Enthalpy of the Fe-pH synthesis :

From the thermal measurements (4.6.1), the conversion is 34,400 ppm, when taking for the enthalpy of formation $H = 681 \text{ MJ/moleFe}$ (7.1 keV/atom Fe). Corrected for the pollution by the 304 L stainless steel of the cell, the conversion is 26,700 ppm.

From the MS analysis the conversion is 60,260 ppm for ^{54}Fe (5.2.4). The enthalpy of formation is thus 390 MJ/mole Fe (4.05 keV/atom Fe) for $^{54}\text{Fe-pH}$, assuming the same reaction rate for all iron isotopes.

The conversion from thermal results can be reconciled with the conversion from MS analysis in the case of $^{54}\text{Fe-pH}$: referring to [1], the effective charge of the hypole proton would be 19.5 instead of 26, the dimension of the radius of this proton orbit being in the order of 3 picometer.

Assuming that the reaction rate is the same for all Iron isotopes, the enthalpy of formation of Fe-pH is some 390 MJ/Mole Fe.

Note that no high energy X-ray photons emission was observed during the synthesis (experiments are monitored with a GM counter placed inside the cabinet).

7. Conclusion:

Iron is very abundant, ranking 4th in the earth crust. Sodium (that acts as catalyst in the Fe-pH synthesis), ranks 6th. Should Fe-pH synthesis be harnessed as an energy source, the energy this synthesis releases (390 MJ/mole Fe) is considerably higher than the energy released by the synthesis of CO₂ from Carbon (0.39 MJ/mole C). Fe-pH synthesis could then be an abundant and clean energy source producing neither CO₂ nor nuclear waste. Finally the Iron pico-hydride is a permanent electric dipole of atomic size. The mechanical properties and the electrical conductivity (high temperature super conductor) of an Iron Pico hydride are potentially interesting.

Ref

[1] J.J Dufour, X.JC. Dufour and J.D Vinko "*Pico-Chemistry: the possibility of new phases in some Hydrogen/Metals Systems*" IJMP-B Vol 27 N° 15 (2013) 1362038.