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**SOME EXPERIMENTS ON COLD FUSION BY DEUTERIUM/HYDROGEN  
GAS INFUSION IN TITANIUM METAL/ALLOY**

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**ABSTRACT**

New results on cold fusion are reported where three different experimental situations have been tried: a) deuterium gas loaded titanium ; b) deuterium gas loaded  $Ti_{0.8}Zr_{0.2}CrMn$  alloy and c) titanium and the  $Ti_{0.8}Zr_{0.2}CrMn$  alloy loaded with a mixture of deuterium and hydrogen gases. With these experiments, new thermodynamical non equilibrium conditions were achieved and the possibility of cold fusion between protons and deuterons was also tested. Three independent neutron detectors and one  $NaI(Tl)$  were utilized. Despite some large values reported in the literature for the fusion rate, an upper limit of only  $8 \times 10^{-24}$  fusions/s per deuterium pair or per deuterium-hydrogen pair was determined within the attained accuracy.

ALGUNS EXPERIMENTOS SOBRE FUSÃO A FRIO ATRAVÉS DE REAÇÕES DIRETAS DE  
DEUTÉRIO/HIDROGÊNIO GASOSOS COM METAIS/LIGAS DE TITÂNIO

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RESUMO

Neste trabalho são mostrados novos resultados de experimentos sobre fusão nuclear a frio, nas quais foram tentadas três situações experimentais distintas: a) absorção de gás deutério em titânio; b) absorção de gás deutério na liga  $Ti_{0,8}Zr_{0,2}CrMn$  e c) absorção de mistura de gases de deutério e hidrogênio em titânio e na liga  $Ti_{0,8}Zr_{0,2}CrMn$ . Com esses experimentos, foram atingidas novas condições de não equilíbrio termodinâmico e foi testada também a possibilidade de fusão nuclear a frio entre prótons e deuterons. Foram utilizados três detetores de nêutrons independentes e um detetor cintilador NaI(Tl). Ao contrário de alguns valores grandes para a taxa de fusões apresentados na literatura, foi determinado um limite superior de apenas  $8 \times 10^{-24}$  fusões/s por par de núcleos de deutério ou por par de núcleos de deutério/hidrogênio, de acordo com a precisão obtida nos presentes experimentos.

## INTRODUCTION

The experimental evidence on the feasibility of the cold nuclear fusion of two deuteron nuclei, announced by Fleischmann and Pons<sup>(1)</sup> and by Jones et al<sup>(2)</sup>, stimulated a great number of researchers around the world to study this new and exciting phenomenon. A lot of experimental and theoretical work has been done since then.

Some carefully carried out experiments have been published in which the authors claim to have observed the neutrons and gamma radiations produced by nuclear fusion of deuterium atoms introduced into palladium or titanium metals by electrochemical method<sup>(2,4)</sup> or by pressurized gas loading<sup>(3,4)</sup>. On the other hand there are other similar publications with negative results for both types of experiments<sup>(5,6,7,8,9,10)</sup>. The works due to Fleischmann and Pons<sup>(1)</sup>, Iyengar<sup>(4)</sup> and another one from the Frascati group<sup>(11)</sup>, report on the observation of a highly significant emission of neutrons from deuterated metals. The work from Iyengar<sup>(4)</sup> also reports on the formation of large quantities of tritium. All these results were attributed to the occurrence of cold nuclear fusion.

From a theoretical point of view, a cold fusion of two deuteron nuclei is only possible by a tunneling process through the mutual Coulomb barrier. The earlier estimation of the fusion rate by molecular deuterium resulted in a value of  $\approx 10^{-74}$  fusions/s per deuteron pair<sup>(12)</sup>. After the Fleischmann and Pons's experiment more recent calculations showed that this value may be 10 orders of magnitude higher<sup>(13)</sup>.

In order to achieve a fusion rate of about  $10^{-25}$  fusions/s per deuteron pair, (the fusion rate observed in the reported experiments), the deuteron pair distance must be reduced to about  $0.1\text{\AA}$ <sup>(14)</sup>, a very improbable situation to occur in the usual Pd-d system, according to the electronic densities calculations of Sun and Tomanek<sup>(15)</sup>. On the other hand, other authors have different points of view: according to Lee and Kalotas<sup>(16)</sup> the fusion rate may be enhanced if the screening of the electrons between two deuteron atoms inside a metal lattice is sufficiently strong; Hargitai<sup>(17)</sup> pointed out that the Fleischmann and Pons's results may be understood if it is considered that the Pd-d system forms a semiconductor with high dielectric constant, which is not an unusual situation. A re-

port due to Tajima et al<sup>(18)</sup> shows that a pair of deuterons exhibit attractive interactions inside a metal lattice for relative distances in the range of 0.1A to 0.7A. Furthermore, several authors<sup>(13,14,19)</sup> have pointed out that the  $p+d \rightarrow {}^3\text{He} + \text{gamma} (5.5 \text{ MeV})$  reaction is favoured compared to the reaction  $d+d \rightarrow {}^3\text{He} + \text{neutron} (2.2\text{MeV})$  by various orders of magnitude, due to the smaller reduced mass of the proton-deuteron system.

After all these negative and positive results on cold fusion it is tempting to formulate the questions: is there a realistic way to obtain reproducible cold fusion reaction and then to search for more efficient systems for practical applications? Have the positive results reported so far occurred due to some triggering processes, e.g., by mesons<sup>(17,20)</sup>? It is reasonable to think that as these and other questions are still unanswered, the cold fusion subject remains an open field of research.

In this article we report on the experimental results of neutron and gamma ray detection from deuterated and hydrogenated metals such as titanium and the  $\text{Ti}_{0.8}\text{Zr}_{0.2}\text{CrMn}$  alloy. The process used was the direct chemical reaction of the metals with deuterium and hydrogen gases. More pronounced thermodynamical non-equilibrium conditions have been also tried.

#### EXPERIMENTAL FACILITIES DESCRIPTION

A sketch of the experimental set-up is presented in figure 1. A tank with 62 cm external diameter, 95 cm height and with a central hollow cylinder of 10 cm diameter, was filled with deionized water. Three He-3 detectors (6 atm), 60 cm long and 1 inch diameter were vertically placed inside the tank 3 cm from the hollow cylinder and protected by PVC tubes. These counters have a high efficiency for thermal neutrons detection and a negligible sensitivity to gamma rays.

Such an arrangement provides a neutron detector with a nearly constant efficiency over a wide range of neutron energies, and due to that it is usually called "long counter". The efficiency of the counter is also nearly constant over a large axial extension when the neutron source is displaced around the central position. Furthermore the efficiency of the long counter is two or three orders of magnitude higher than for an iso-

lated detector placed near a 2-3 MeV neutron source such as that expected in the present experiment. These features arise because the neutrons are slowed down to the sub-eV range before they are absorbed in the detectors.

A borated paraffin shield, 20 cm thick, was also placed around the long counter in order to prevent neutrons from the environment to be detected.

The three detectors inside the water bath, as shown in the figure 1, were operated with three independent electronic lines. With this setting, any variation of neutron emission from the source should be necessarily observed simultaneously by the three detectors and this virtually eliminates the problem of spurious counting due to other causes than the effect to be observed, which is quite possible in long term experiments with this type of detector.

Another neutron detector, similar to those utilized for the long counter was placed about 10 meters from the experimental apparatus to monitor the ambient neutron background.

For a second set of experiments, a 3" x 3" NaI(Tl) scintillation detector was also installed with its own 10 cm thick Pb shielding outside the long counter and looking at the deuterated metal sample placed inside the long counter (see figure 1). The counting system for this detector was set-up to register the 2.2 and 5.5 MeV gamma rays range expected from the following nuclear reactions:



A multichannel analyser was connected to the system to distinguish whichever reaction was eventually taking place. The first of these reactions should be due to the interaction of the neutrons emitted from the deuterated metal with the water bath of the long counter. The detection of neutrons by this way is analogous to the method utilized by Fleischmann and Pons and is sensibly affected by the natural gamma ray background due to the presence of Bi-214 (2.117 MeV and 2.204 MeV) and Tl-208 (2.615 MeV) nuclides usually found in buildings. The second reaction



is expected to occur with more probability in systems where hydrogen is mixed with deuterium<sup>(13)</sup>. In this case there is no gamma ray background with energy around 5.5 MeV, allowing thus for a more sensitive detection of any proton-deuteron cold fusion event.

The efficiency of the long counter was determined after neutron counting from a calibrated californium-252 source (yielding about 200 neutrons/s) placed inside the long counter. The mean neutron energy from this source is 2.2 MeV and thus approximately equal to the energy of the neutrons expected to be emitted from the deuterated metal sample (2.45 MeV)<sup>(22)</sup>. The efficiency obtained was 3.9% and the efficiency of the gamma ray system was estimated to be around 0.3%.

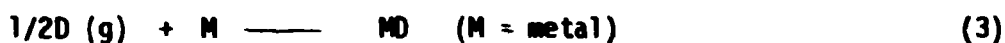
The apparatus where the metal samples were handled with deuterium gas was an extension of a Sievert's type apparatus usually utilized for studies with metal-hydrogen systems. It consists of several stainless steel valves and tubes by which it is possible to extract the gases by a vacuum system or to introduce gases up to 100 bar inside the sample holder. It is also possible to estimate the amount of gas absorbed by the metal sample by means of calibrated volumes. A digital pressure gauge permitted to monitor the gas pressure to which the metal samples were exposed and a copper-constantan thermocouple, placed in contact with the stainless steel sample holder, made it possible to read the sample temperature at least at an approximate level.

On running the experiments, the sample vessel was always kept inside the long counter and inside a thermally insulated glass bottle which was frequently filled with liquid nitrogen or other hotter liquid, by means of a plastic tube extending to the outside of the long counter. As the glass bottle could present some boron contamination, a check for neutron attenuation was performed and no appreciable effect was observed. Once the bottle was filled with liquid nitrogen and the thermal equilibrium was attained, it took about six hours to evaporate completely.

## COLD FUSION EXPERIMENTS AND RESULTS

The experiments consisted of monitoring the neutron counting while the metal sample was exposed to deuterium gas, or monitoring the gamma

counting while the metal sample was exposed to a mixture of deuterium and hydrogen gases. In both cases, the gas absorption and desorption cycles were performed in a similar way as that described by the Frascatti group<sup>(3)</sup> or by Iyengar<sup>(4)</sup>. In these circumstances the chemical reactions:



which takes place in these experiments, is not in thermodynamical equilibrium and this is a necessary condition to stimulate the occurrence of cold fusion in deuterated metals, as pointed out by Fleischmann and Pons<sup>(1)</sup> and by Jones and coworkers<sup>(2)</sup>.

Seven independent runs of such experiments were carried out in this work for a total of about 500 hours, during the period from May to August of 1989, and may be divided in three basic groups: four experiments with deuterated titanium where the amount of metal, the origin of the starting material and the degassing procedure before the first gas absorption cycle were varied; one experiment with a deuterated  $Ti_{0.8}Zr_{0.2}CrMn$  alloy and two experiments where hydrogen and deuterium gases have been absorbed by the titanium and by the cited metallic alloy. These experiments are described in more details below.

In a first experiment, 10 g of commercial grade titanium chips, 0.2 mm thickness, obtained by machining a rod, were initially rinsed with acetone, dried and degassed at 673 K in high vacuum ( $10^{-6}$  torr) for about 10 hours. After that, 99.8% purity deuterium gas was introduced into the system, at ambient temperature and slowly raising the pressure up to 5 bar. Simultaneously the radiation counting systems were started, giving the initial time of the experiment. In this and in all the subsequent experiments the counting time was established to be 1000 s after which the counts were recorded and the counting reinitiated automatically.

After about 15 minutes, it was observed that apparently no gas absorption reaction was taking place, as neither the pressure lowered nor the temperature increased. The gas pressure was then increased in steps of about 5 bar until a very slow chemical reaction at 25 bar was observed indicated by the system pressure decrease. At this point the temperature was lowered to 77 K by immersing the sample holder in liquid nitro-

gen and the deuterium gas pressure was increased to 40 bar. As the system was left closed, the pressure decreased continuously at a rate of about 5 bar/h while the liquid nitrogen was evaporating. The liquid nitrogen level in the bottle and the deuterium gas pressure were restored to the initial conditions at every period of about 4 to 6 hours. In the last cycle, after about 50 hours, the gas pressure rose to about 100 bar when the deuterated titanium attained the environment temperature, indicating the saturation of the titanium sample with deuterium. For the last period of the experiment the deuterated titanium was exposed to vacuum (0.001 torr) and thermal cycling was performed several times between 77 K and 293 K. No gas desorption was detected during this period. After the experiment, it was assured by X-ray analysis that all the titanium sample was converted to  $TiD_2$  over the first part of the experiment.

The experimental data analysis showed that no neutron counts above background (131 counts/1000 s) was observed within 99.5% statistical confidence level. The upper limit for neutron emission thus yielded:

$$\theta_n \leq 0.11 \text{ n/sg (Ti)}$$

Following this, the upper limit for d-d reaction rate per deuteron pair resulted in:

$$\lambda_f \leq 8 \times 10^{-24} / \text{s}$$

It is known that the uptake of deuterium gas by metals follows basically two steps: the catalytic effect of molecular dissociation on the surface of the metal and the in bulk diffusion of deuterons<sup>(23)</sup>. The slowest of the two steps determines the chemical reaction rate.

For titanium, the reaction rate for deuterium absorption can be greatly increased if an efficient surface cleaning is made before the reaction is carried out and also if deuterium gas and titanium metal with high purities<sup>(24)</sup> are used. This indicates that the chemical reaction rate is likely to be limited by the first step outlined above, i.e., by the surface catalytic effect.

All the following experiments have been planned in such a way to get

a metal-deuterium reaction as much limited as possible by the diffusion process instead of the surface catalysis effect, in order to try in bulk non-thermal equilibrium conditions.

In this way, a second run of experiment with titanium sample was carried out, after making a careful initial degassing for 25 hours at 673 K and  $10^{-6}$  torr. As soon the first deuterium gas was introduced at 1 bar and at ambient temperature the chemical reaction was prompt: as the system remained closed, the pressure quickly dropped and the temperature of the sample holder increased, indicating that heat was being generated by the chemical react. After immersing the sample holder in liquid nitrogen and after the thermal equilibrium was attained, the experiment was carried out in a similar way as that of the first run.

The neutron counting profile for this experiment is presented in figure 2 whereas in figure 3 the counts due to the ambient neutron are shown. The neutron background inside the long counter has been measured before and after the experiment and it presents similar features as viewed in figure 2. The arrows in this figure indicates the filling with liquid nitrogen and deuterium gas. The fluctuations observed by the monitor in figure 3 may be explained by the fact that the IEA-R1 research reactor and a linear accelerator were in operation during that part of the experiment. However the absence of these fluctuations in figure 2 indicates the effectiveness of the long counter shielding. As in the case of the run n° 1, no net counting of neutrons was observed.

For runs n° 3 and n° 4 the samples were 30 g of titanium sponge, with 99.7% purity, produced by "Riedel-de-Haen AG Seelze-Hannover", from West Germany. The deuterium gas absorption by these samples was unsuccessful probably due to the presence of a surface oxide layer.

Run n° 5 was carried out in a different way. The metal sample was 20 g of powdered  $Ti_{0.8}Zr_{0.2}CrMn$  alloy which was developed for hydrogen storage purposes<sup>(25)</sup>. This alloy is unstable with respect to deuterium or hydrogen gas (the equilibrium pressure at ambient temperature is about 20 bar), it is not sensible to gas impurities and the absorption/desorption cycling processes are very easy to be performed<sup>(26)</sup>.

Initially an amount of deuterium gas was introduced into the system,

sufficient to fill 80% of the saturation level of the alloy. In this case, at 77 K, all the deuterium gas is in the absorbed state whereas at about 400 K, practically all the gas is liberated by the alloy. On running the experiment a thermal cycling between 77 K and ambient temperature (or eventually up to 400 K) was performed, allowing the deuterium gas to be cyclically absorbed and desorbed.

Several cycles were performed in which the reaction rate of the deuterium gas with the alloy was varied: a) firstly cooling the metal alloy and then exposing it to deuterium gas with different pressure steps; b) cooling (or even "quenching") the metal alloy at the same time it was exposed to deuterium gas; c) performing a slow (6 hour) or fast (5 min) transition from 77 K to 293 K (or 400 K) on the gas desorption cycle.

In the last cycles more deuterium gas was introduced into the closed system until the alloy was saturated with about 20 bar deuterium gas pressure upon it at 77 K. It is believed that with these cycles different in bulk situations of non-thermal equilibrium, were achieved as pointed out previously.

The neutron counting profile for this run was similar to that seen in figure 2 and again no net counting was observed.

For the experiment number 6, 73 g of titanium chips, carefully degassed, were initially put to react with deuterium gas, up to 20% of its total capacity. After that, the experiment was conducted in a similar way as the run n° 2 but hydrogen gas was also introduced into the system. The idea was to observe the 5.5 MeV gamma rays emitted from the  $p + d$  fusion which is theoretically<sup>(13,14,19)</sup> more favourable than the  $d + d$  reaction.

The neutron counting profile for the first part of this experiment, i.e., during the absorption of deuterium gas, was very similar to that presented in figure 2. However, in the second part of the experiment, i.e., during the sample filling with hydrogen gas, a nearby cyclotron machine was in operation over two periods of measurements, and the neutron, gamma and background counting for this particular situation are shown respectively in figures 4, 5 and 6.

From these last results it can be observed that the response of all

detectors were in accordance, showing that if any cold fusion event had taken place with neutron or gamma emission, even for short period of time, the system should have detected it. It is also important to note that even with this high neutron background, the large values for the fusion rates reported by some authors, would be easily identified. As can also be seen in these figures, again no net counting of neutrons or gamma rays was observed.

In the last run, 75 g of the  $Ti_{0.8}Zr_{0.2}CrMn$  alloy were forced to absorb and desorb a mixture of hydrogen and deuterium gases by making a thermal cycling in a similar way as was carried out in the experiment number 5, while at the same time the neutron and gamma ray counting was monitored. In this way, new thermodynamical non equilibrium situations have also been performed in order to search for p + d fusion reaction. The results for gamma and neutron counting were similar to the previous experiment, and also no net counting was observed.

#### DISCUSSION AND CONCLUSION

In these experiments, three distinct situations were tried in order to observe the cold fusion results reported on by some authors: a) deuterium gas loaded titanium chips; b) deuterium gas loaded  $Ti_{0.8}Zr_{0.2}CrMn$  alloy; c) titanium and the alloy loaded with a mixture of deuterium and hydrogen gases.

The first type of experiments is analogous to the experiments carried out by the Frascati group<sup>(3)</sup> and by Iyengar<sup>(4)</sup>. It is believed that in this case the metallic surface effects play an important role since it is known that the titanium surface cleanliness strongly influences the reaction rate of hydrogen gas with this metal<sup>(24)</sup>.

In contrast, the second type of experiments in which the metallic alloy  $Ti_{0.8}Zr_{0.2}CrMn$  was deuterated, the effects in the bulk of the material are more important than in the first experiments, since there are less severe restrictions on surfaces conditions<sup>(25)</sup>. Due to that, different thermodynamical non-equilibrium conditions were attained when compared with the first type of experiments.

In the third type of experiment an attempt was made to observe the nuclear fusion between deuteron plus proton and two different thermodynamical situations were created.

The upper limit for the rate of fusion events determined from these experiments was  $8 \times 10^{-24}$  fusions/s per deuteron pair and a similar value was obtained for deuteron-hydrogen pair. This value is compared with the experimentally observed fusion rates from other authors in table I.

TABLE I - Comparison between various cold fusion rates

Author (reference)	Value (fusions/s per dd pair)
I) SUCCESSFUL EXPERIMENTS	
Fleischmann and Pons(1)	$9.4 \times 10^{-19}$
De Ninno et al.(11)-Frascati	$3.2 \times 10^{-22}$
Jone et al. (2)	$1 \times 10^{-23}$
Bertin et al.(3)-Gran Sasso	$6.4 \times 10^{-24}$
Iyengar(4)	$3.8 \times 10^{-21}$
II) UNSUCCESSFUL EXPERIMENTS	
Broer et al.(5)	$< 2.2 \times 10^{-24}$
Lewis et al.(6)	$< 1.5 \times 10^{-24}$
Gai et al.(7)	$< 2.0 \times 10^{-25}$
Brudanin et al.(9)	$< 7.0 \times 10^{-28}$
Badurek et al.(10)	$\approx 3.6 \times 10^{-23}$
Present Work	$< 8.0 \times 10^{-24}$

It is seen that except for the value quoted by Bertin et al.<sup>(3)</sup>, in the Gran Sasso experiment, our limit of detection was sufficiently small to observe easily the successful reported fusion rates<sup>(1,11,2,4)</sup>, even with the presence of a higher background during the cyclotron operation

in a part of the experiment. In this context we can conclude that our experiments do not confirm the high cold fusion rates as claimed by some authors<sup>(1,11,2,4)</sup> and thus we are more in accordance with the reported negative results on cold fusion as can be seen in Table I.

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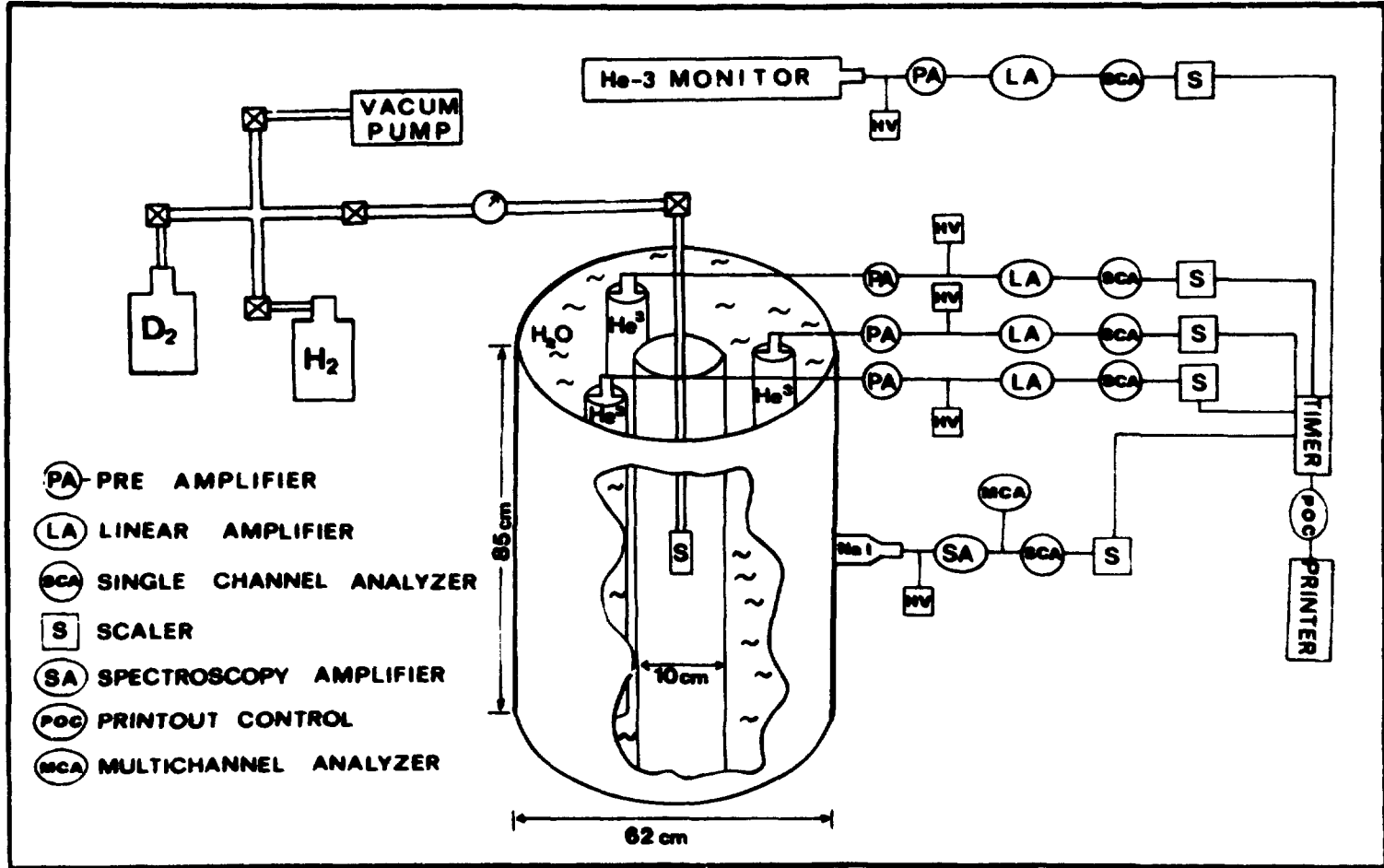


FIG 1

FIGURE 1 - Sketch of the cold fusion experimental set-up.

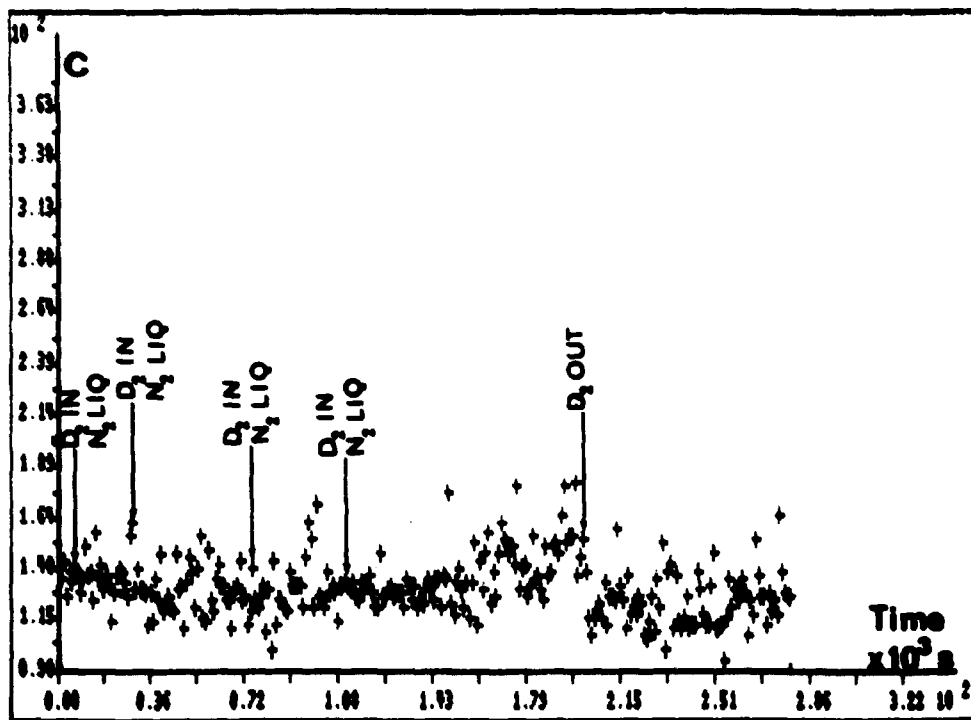


FIG 2

FIGURE 2 - Neutron counting profile from experimental run n<sup>o</sup> 2, during the admission of deuterium gas on titanium. The arrows indicate the restoring of gas pressure and the refill of liquid nitrogen. (see text)

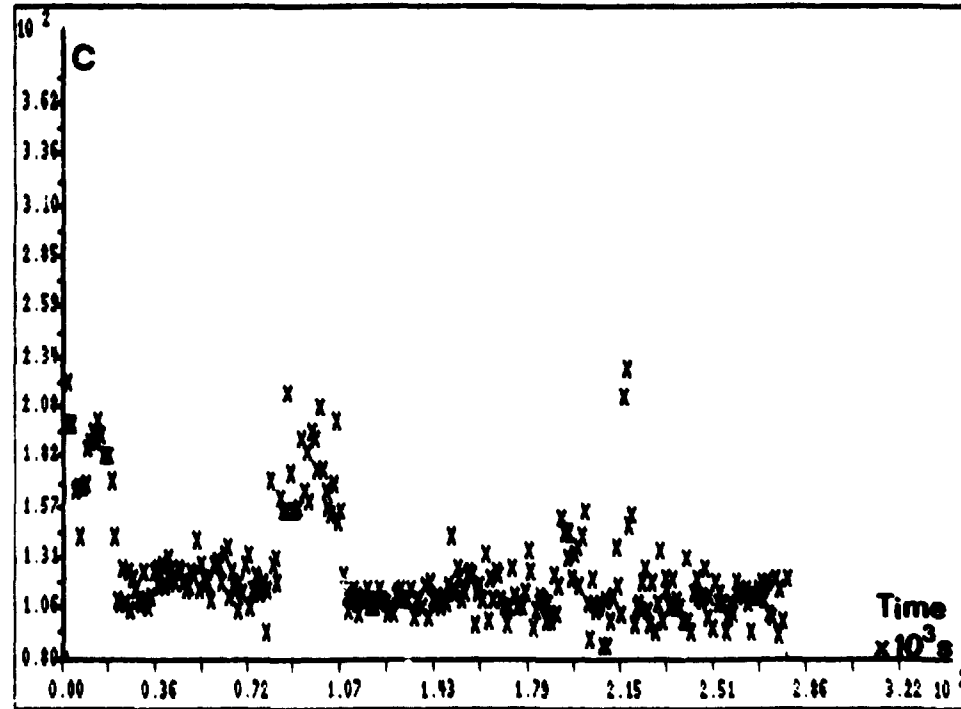


FIG 3

FIGURE 3 - Environmental neutron counting from experiment n<sup>o</sup> 2.

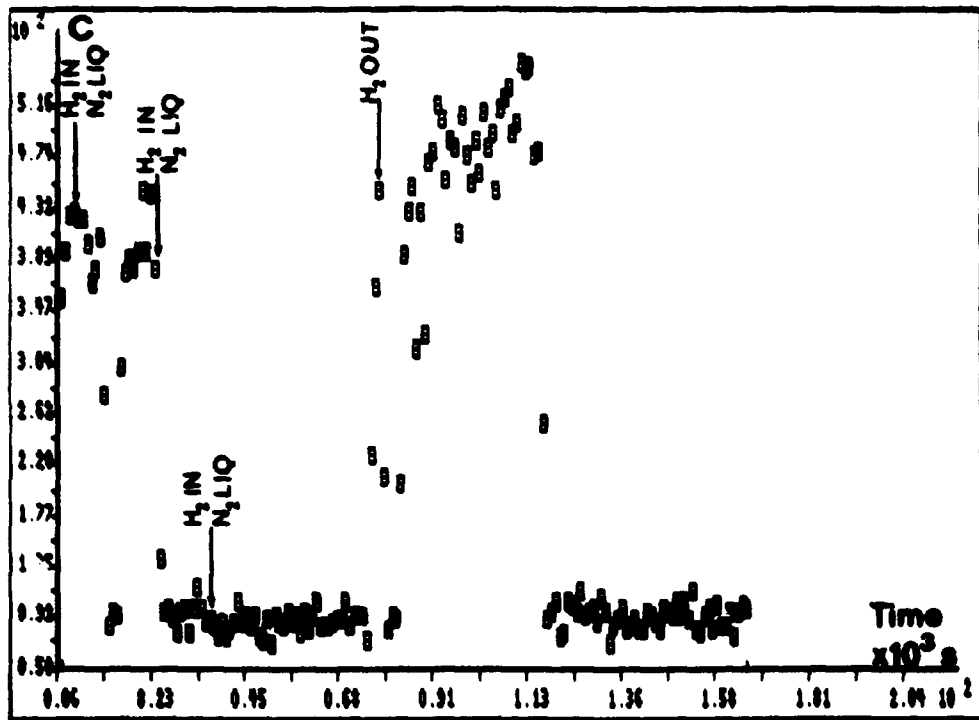


FIG 4

FIGURE 4 - Neutron counting profile from experiment n<sup>o</sup> 5, during the admission of a mixture of deuterium with hydrogen gases into titanium chips. A particular situation where the cyclotron was in operation.

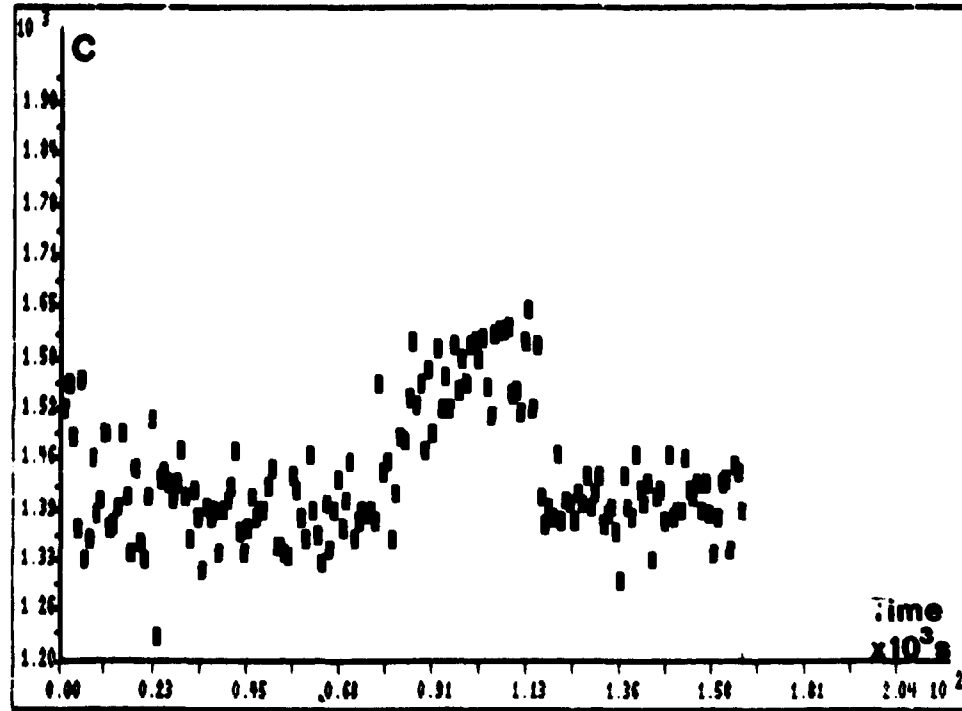


FIG 5

FIGURE 5 - Gamma rays counting profile between 2 MeV and 6 MeV from experiment n<sup>o</sup> 5, during the admission of a mixture of deuterium with hydrogen gases into titanium chips. A particular situation where the cyclotron was in operation.

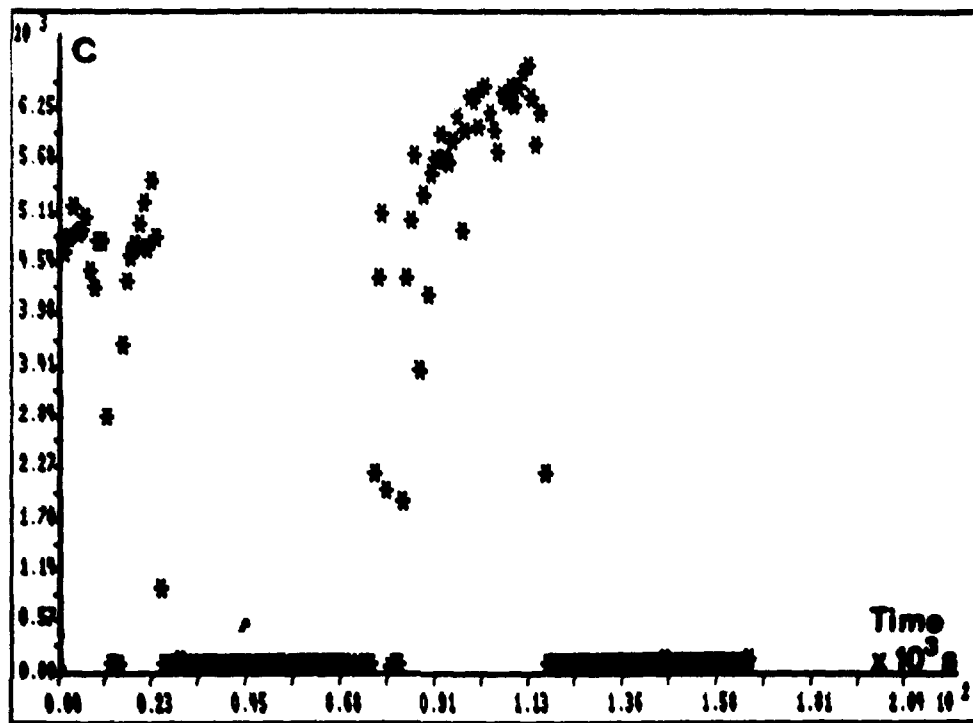


FIG 6

FIGURE 6 - Environmental neutron counting from experiment n<sup>o</sup> 5. A particular situation where the cyclotron was in operation.