

CALORIMETRY OF PULSE ELECTRO-MELTING OF PdD_x WIRES

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Several groups[1, 2] have reported anomalous effects in thin PdD_x materials stimulated by different forms of electro-diffusion. The ultimate extrapolation of this technology is the electrical heating of thin PdD_x wires resulting in destructive high-speed melting - "exploding wires". Exploding wire technology has been used for over 150 years to make fine metal particles[3].

Using the techniques of Celani *et al*[4-6] we are loading thin Pd wires electrochemically up to high loading and sealing their surface electrochemically. The wires are immersed in liquid nitrogen in a cryogenic nitrogen boil-off calorimeter. A short duration (~100ms) high current (~100A) electrical pulse is used to instantaneously melt the wire. The energy from the pulse and any excess energy produced from the extremely fast electro-migration inside the PdD_x will boil off a known amount of nitrogen. This nitrogen boil-off mass-flow calorimeter is used to compare the energy released from PdD_x wires to that released by pure Pd or Pt wires. This cryogenic exploding wire technique may yield very high power density anomalous energy releases.

1. E. Del Giudice, et al., "*The Fleischmann-Pons effect in a novel electrolytic configuration*", ICCF8 Conference Proceedings - Italian Physical Society, Vol. 70, pages. 47-54, (2000)
2. C. Manduchi, et al., "*Electric-field effects on the neutron emission from Pd deuteride samples*", Nuovo Cimento della Societa Italiana di Fisica, A Nuclei, Particles and Fields, Vol. 108A, pages. 1187-1205, (1995)
3. M. Faraday, "*Experimental relations of gold (and other materials) to light*", Philos. Trans. Royal Society London, Vol. 147, pages. 145-181, (1857)
4. F. Celani, et al., "*High hydrogen loading into thin palladium wires through precipitate of alkaline-earth carbonate on the surface of cathode: evidence of new phases in the Pd-H system and unexpected problems due to bacteria contamination in the heavy water*", ICCF8 Conference Proceedings - Italian Physical Society, Vol. 70, pages. 181-190, (2000)
5. F. Celani, et al., "*Reproducible D/Pd ratio >1 and excess heat correlation by 1-micro s-pulse, high-current electrolysis*", Fusion Technology, Vol. 29, pages. 398-404, (1996)
6. P. Tripodi, et al., "*Temperature coefficient of resistivity at compositions approaching PdH*", Physics Letters A, Vol. 276, pages. 122-126, (2000)

Triggered Energy Release From Palladium Deuteride

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Outline

- ❖ Research Objective
- ❖ Background: Observations and Correlations
- ❖ Research Plan
- ❖ Electrochemical PdD_x Formation
- ❖ Cryogenic Calorimeter and Reaction Stimulation
- ❖ He Isotopic Ratio Determination
- ❖ Preliminary Results and Calibrations
- ❖ Conclusion
- ❖ Future Work



Research Objective

- ❖ To understand what limits the rate of energy release (power) from the FPE in intentionally destructive experiments employing small, safe samples of ~1:1 PdD in a novel low temperature calorimeter.
- ❖ To search for evidence of potential products of nuclear reaction.
- ❖ To understand underlying reaction processes and mechanisms (theory).
- ❖ To generate, measure, and understand nuclear-level heat effects:
 - in small, safe samples of ~1:1 PdD
 - electrochemically formed from fine, short PdD_x wires with various known He content
 - stimulated electrically and/or by laser pulse
 - measure heat in a novel calorimeter
 - verify nuclear effects by analyzing the wires for changes in their ³He and ⁴He content and ratio.

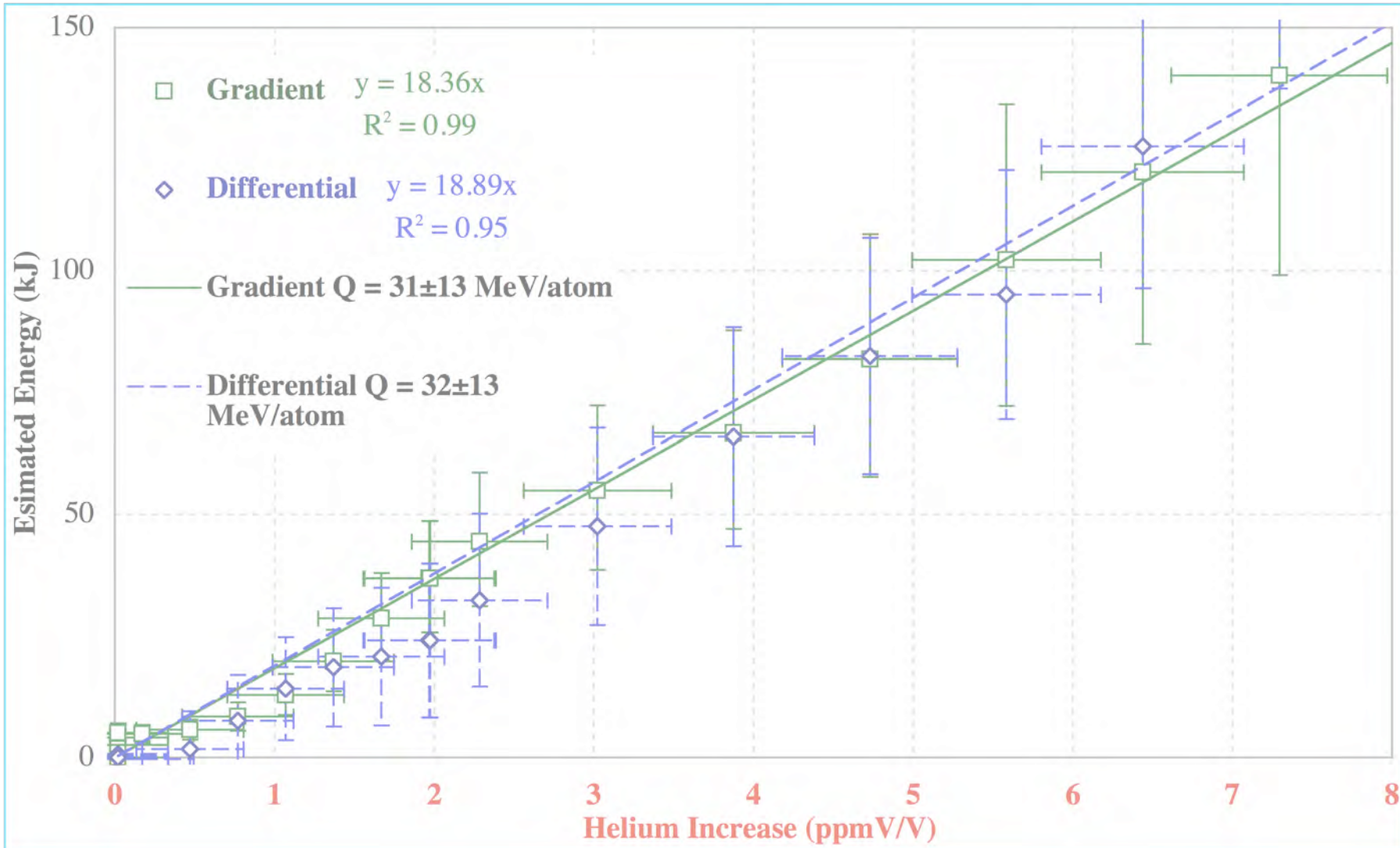


Background: Observations

- ❖ Effect Evidenced on numerous occasions (>70 at SRI)
- ❖ Up to 90σ observation of excess power effect
- ❖ $P_{XS} > 1\text{kW/cm}^3$ (transient)
- ❖ $P_{XS} \sim 150\text{W/cm}^2$ (1 month)
- ❖ $P_{Out}/P_{In} > 50$
- ❖ $E_{Out}/E_{In} > 30$
- ❖ $E_{XS} > 100$ MJ
- ❖ 100 – 10,000 eV/ Pd Atom (100's or 1,000's times known chemical effects)
- ❖ Positive Temperature Coefficient
- ❖ Effect observed up to 650°C
- ❖ Effect has been reported after “mild” electrical stimulation at room temperature



Background: Observation of Energy vs. ^4He



Background: Correlations

- ❖ Necessary conditions:
Maintain High Average D/Pd Ratio
For times $\gg 20-50$ times $\tau_{D/D}$
At electrolytic $i > 250-500 \text{ mA cm}^{-2}$
With an imposed D Flux

(*Loading*)
(*Initiation*)
(*Activation*)
(*Disequilibrium*)

- ❖ Heat correlated with:
 - electrochemical current or current density
 - D/Pd loading or
 - V_{ref} surface potential
 - Pd metallurgy
 - Laser stimulus
- ❖ For 1mm dia. Pd wire cathodes:

$$P_{xS} = M (x-x^\circ)^2 (i-i^\circ) |i_D|$$

$$x = D/Pd, x^\circ \sim 0.875, i^\circ = 50-400 \text{ mA cm}^{-2}, i_D = 2-20 \text{ mA cm}^{-2}, t^\circ > 20 \tau_{D/D}$$



Research Plan

❖ Electrode Preparation:

- ^4He implantation or in-diffusion
- Electrochemical loading
- Surface barrier sealing (transport to calorimeter)

❖ Reaction Triggering:

- Axial current (dc, pulse, sine wave) – 10^5 to 10^7 A cm $^{-2}$
- Surface laser
- Exotic

❖ Heat Measurement:

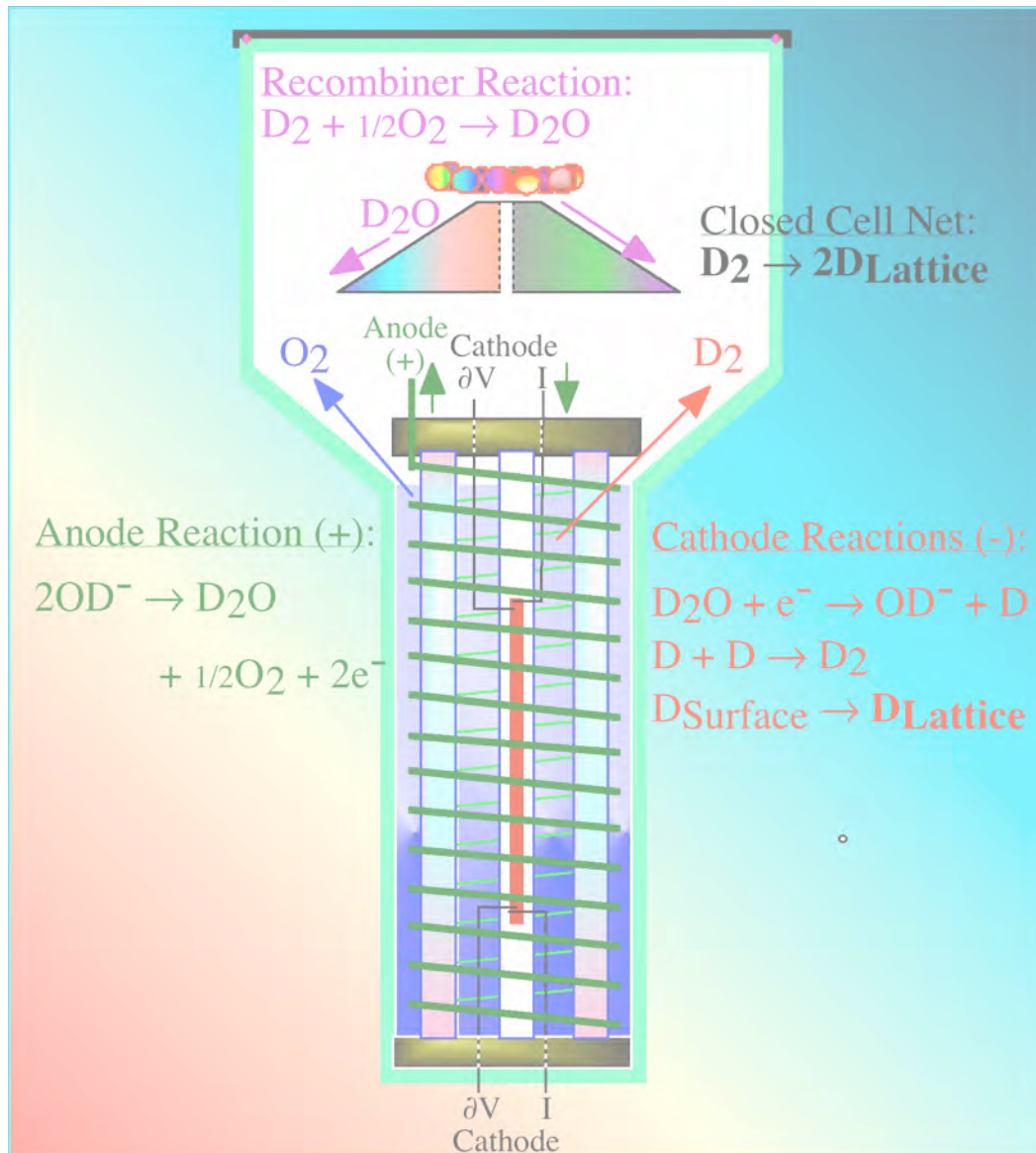
- Mass displacement at low temperature (LN)
- Mass flow at room temperature

❖ Reaction Products:

- Analyze wires and emitted gases for changes in their ^3He and ^4He content and ratio
- Search for isotope effects
- Neutrons and Gammas.



Electrochemical PdD_x Formation



Loading Cell and Reactions.

Wires:

25 – 250 μm in dia.

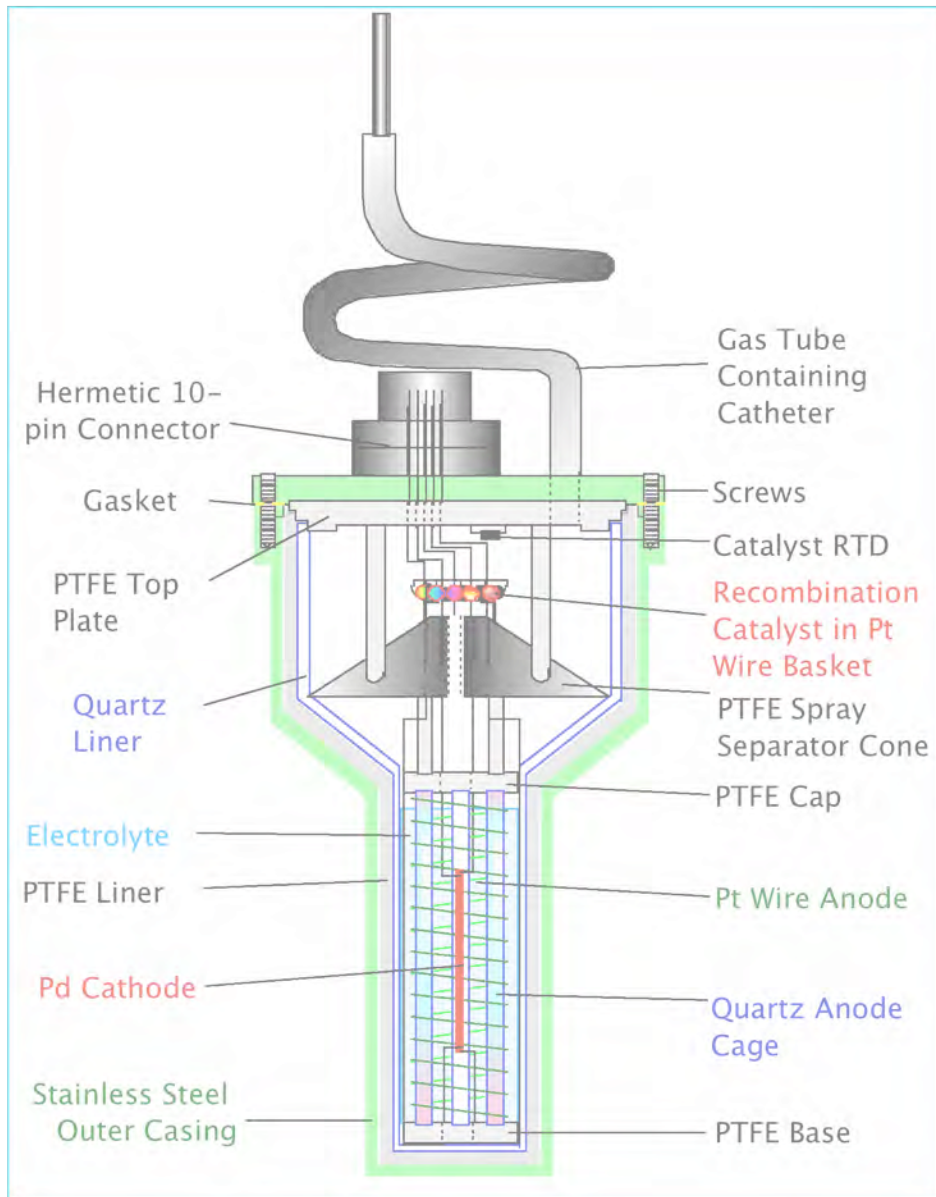
3 – 5 cm in length.

LiOD and low temperature

CD_3OD (or CH_3OD) Electrolytes

Hg (or Pb) to seal loading

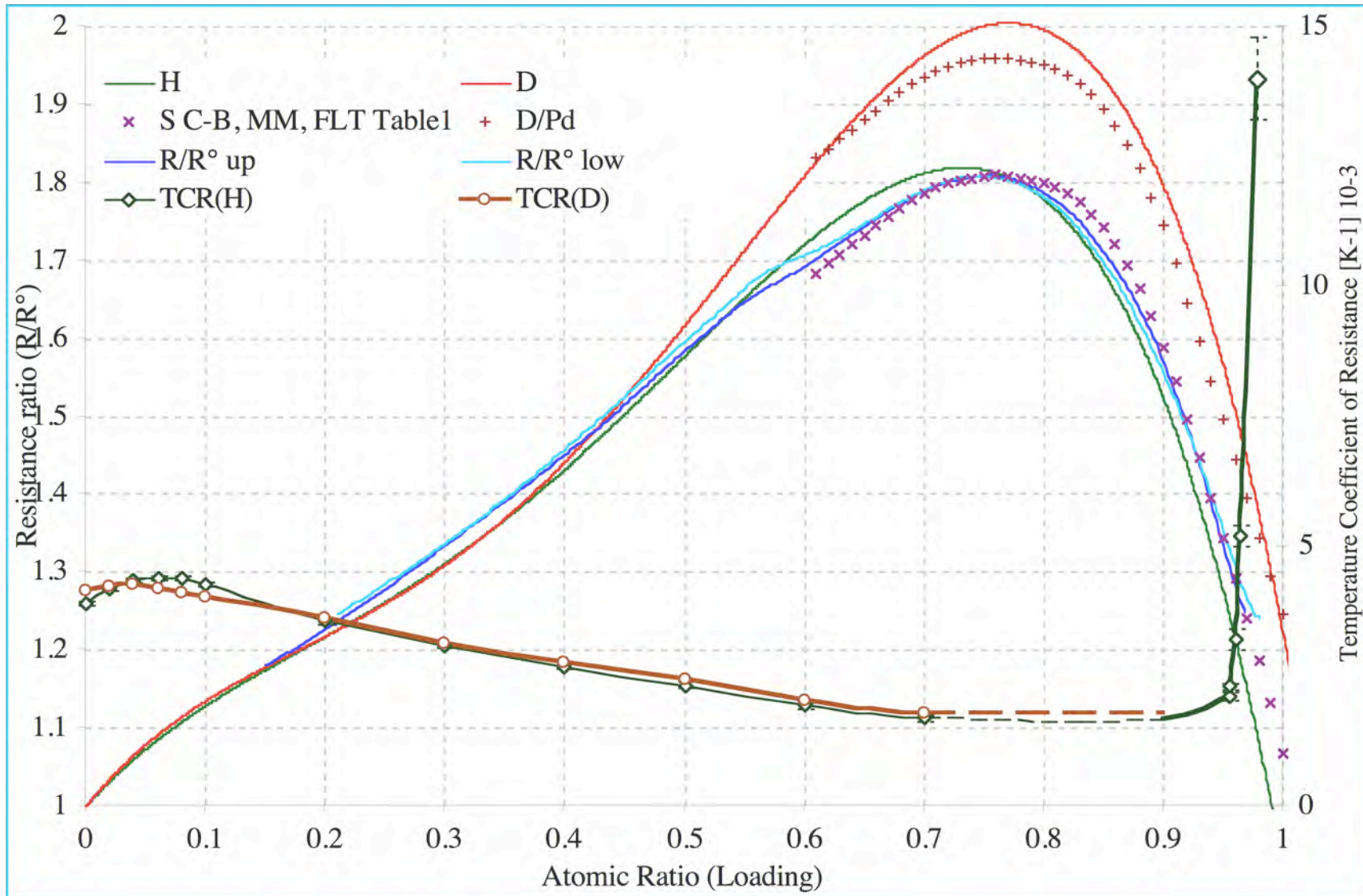
Electrochemical PdD_x Formation



SRI Degree of Loading (DoL) Cell

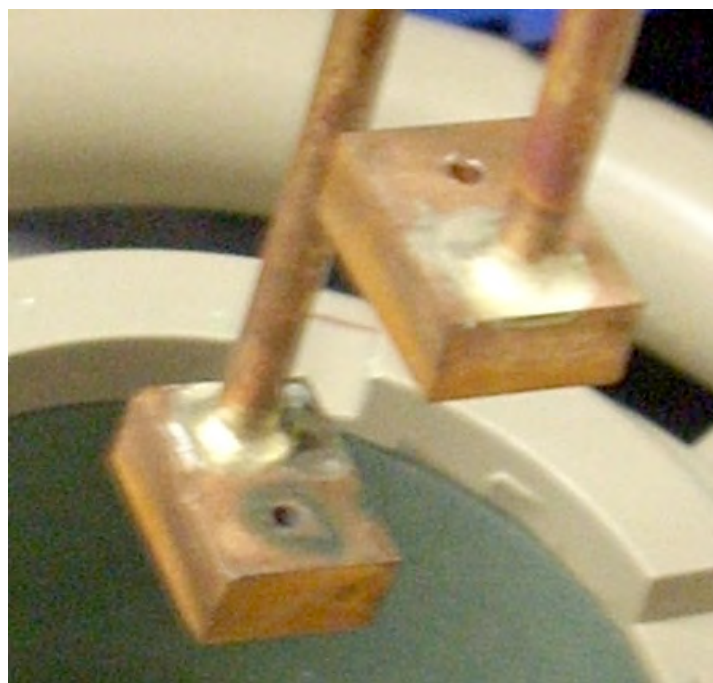


Electrochemical PdD_x Formation Loading and Temperature coefficient of Resistance

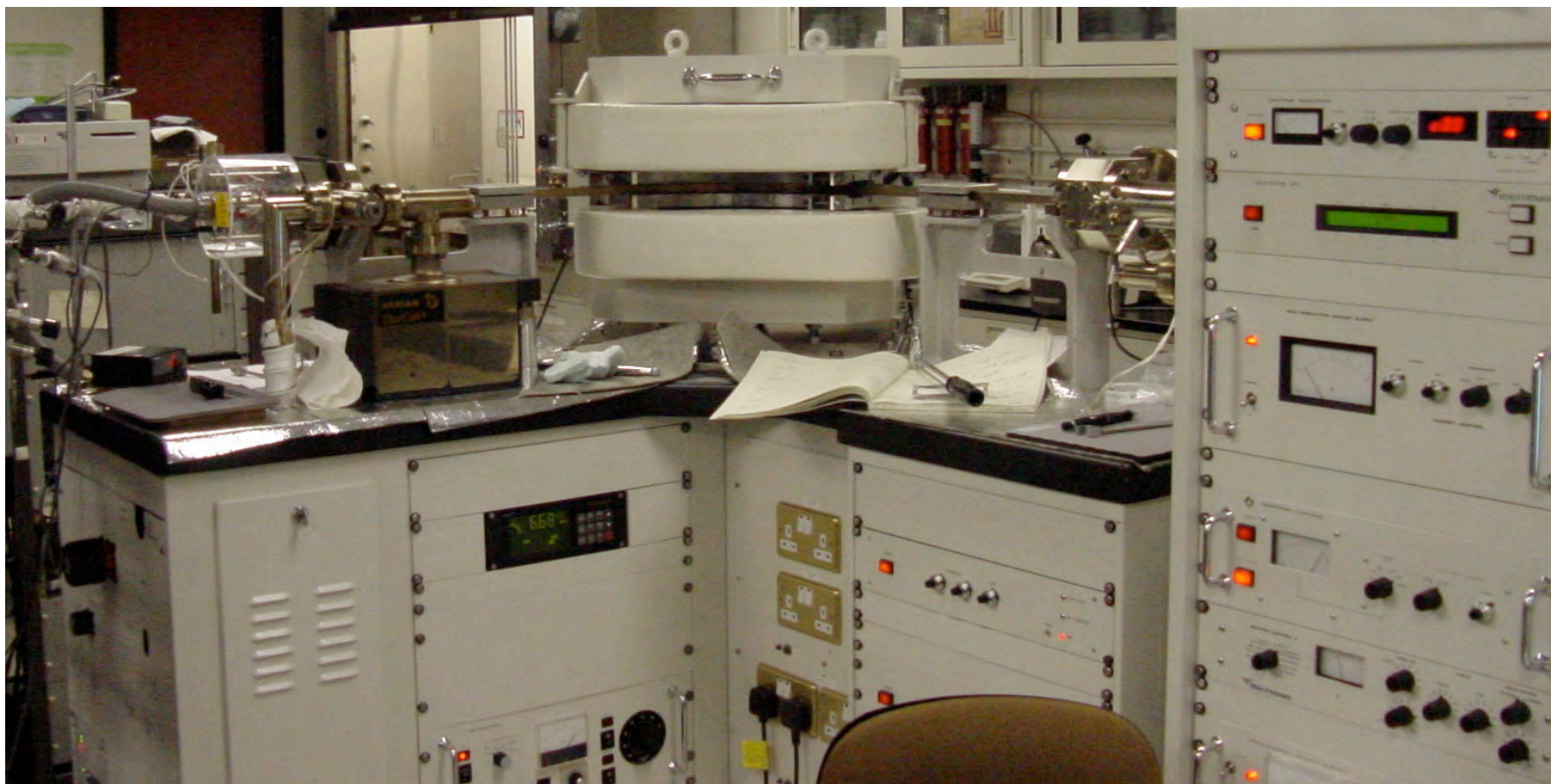


Cryogenic Nitrogen Calorimeter and Reaction Stimulation

- ❖ Hg (or Pb) coated cathodes attached to Current Blocks and dipped in liquid N₂
- ❖ High current ($\leq 125\text{A}$) short pulse destructively stimulates PdD_x to initiate reaction
- ❖ Measure current, voltage, and total N₂ gas evolved
- ❖ Laser or EMF/RF Stimulation in later studies

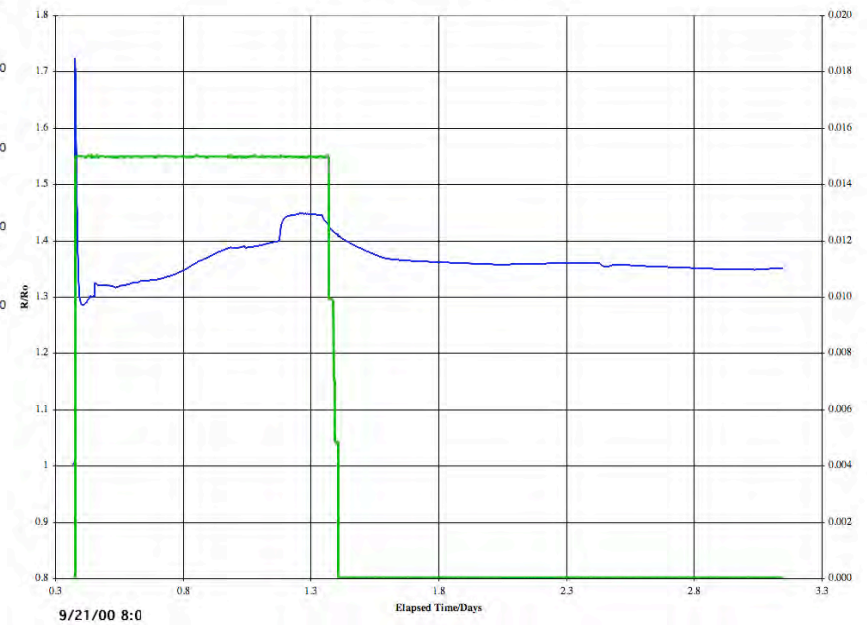
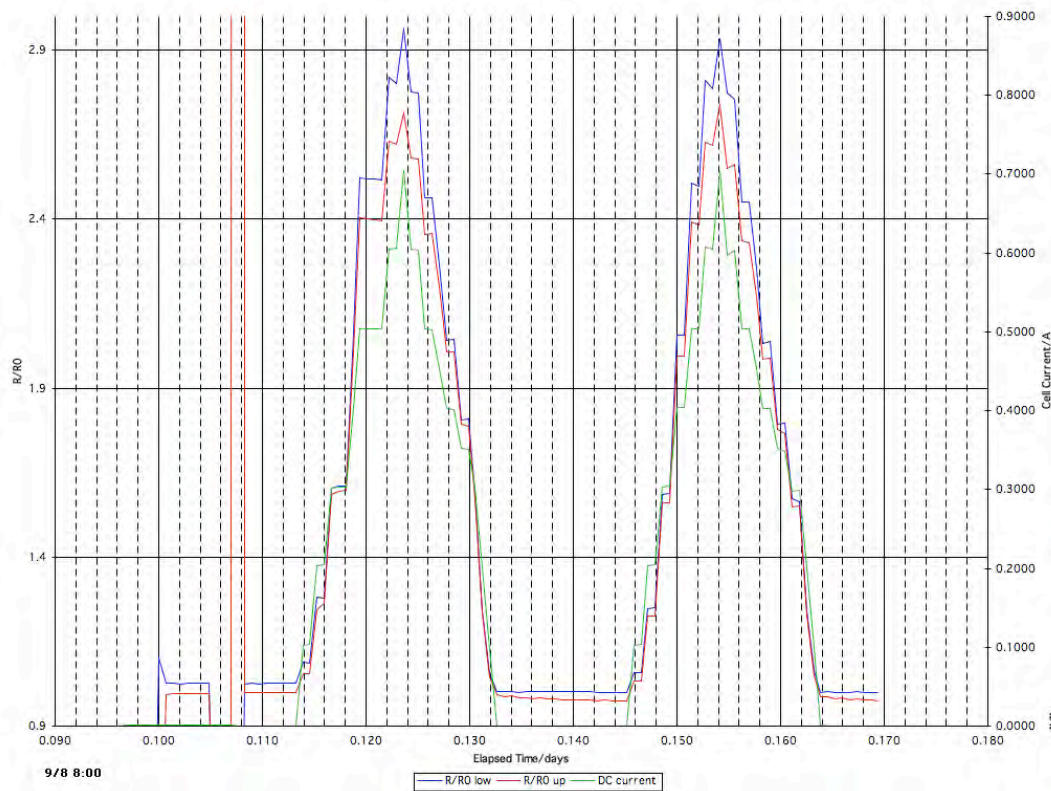


He Isotopic Ratio Determination MicroMass 5400 Noble Gas Mass Spectrometer



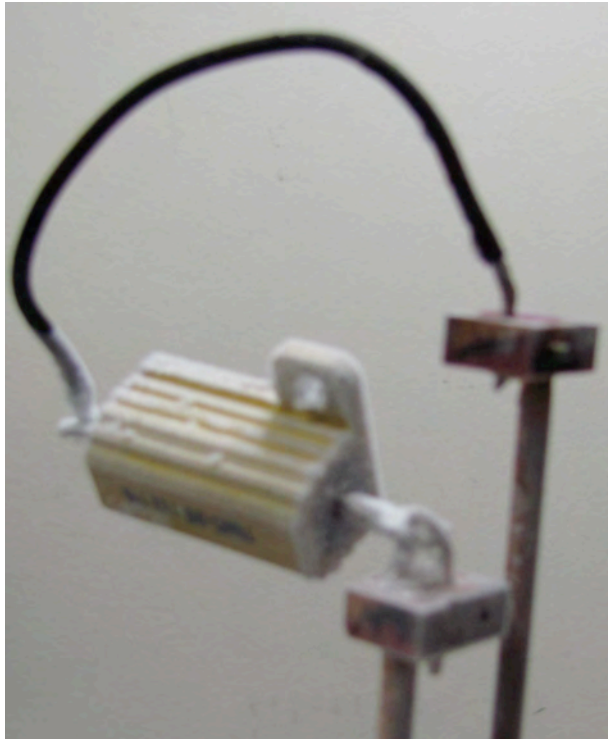
*77K activated charcoal trap/metal getters for hydrogen isotopes
1400°C inlet for He absorbed in Pd*

Preliminary Results: Electrochemical PdD_x Formation In Situ Annealing and Loading Procedure

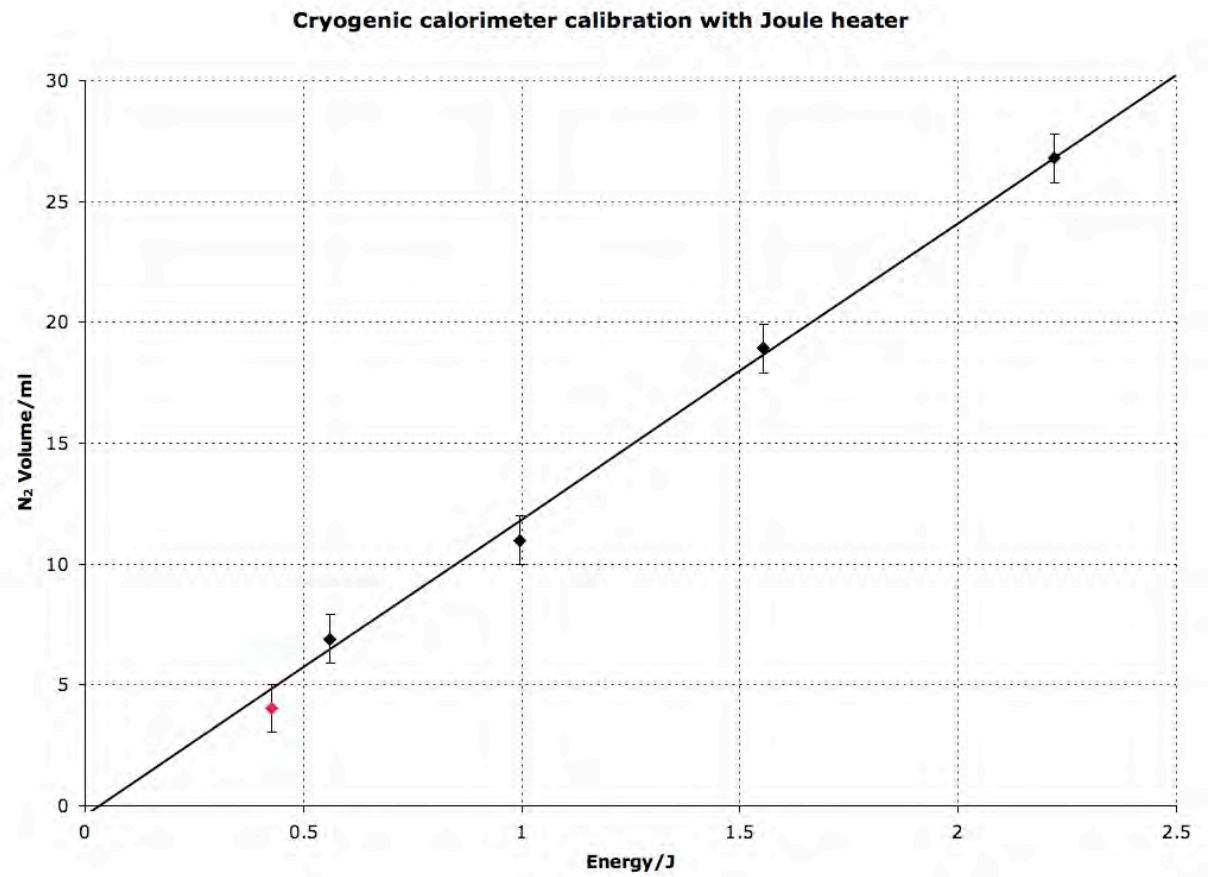


*In situ annealing and high voltage electrochemical loading and sealing as per:
P.Tripodi, et al., Phy.Lett.A, 276, 122-126 (2000).*

Preliminary Results: Cryogenic Calorimeter Calibration



1.008 Ω at 298K, 0.988 Ω at 77K



Red Point is for 50 μ m Pd wire

Preliminary Results: Mass Spectrometer Performance

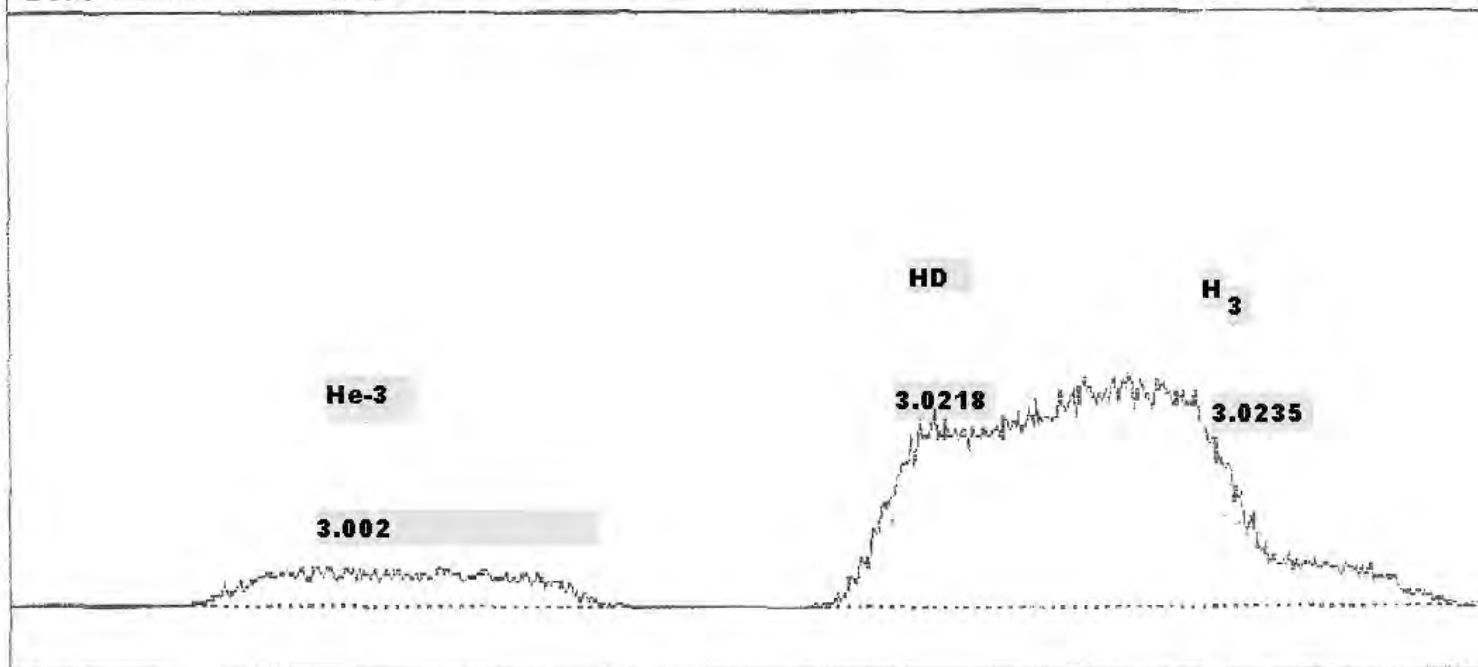
He-3 separation from HD and H₃

Sensitivity: 10⁶ atoms He-4, 10³ atoms He-3
Baseline separation of He-3/H-3 and HD or H₃

Peak Scan

Date : 17 May 2001 Time : 12:11:08

Scale	: 0.00010 Volts	Mass	Collector	: Multipli
Speed	: 3.8	3.012	Mode	: Mass
Integ. Time	: 0.20 Secs	35 CPS	Mark	:
Start Mass	: 2.998		End Mass	: 3.012



Conclusions

- ❖ High energy releases seen in several LENR experiments
- ❖ Some LENR reactions stimulated by axial electrical pulses
- ❖ Pd:D (1:1) wires formed using Tripodi technique
- ❖ Pd:D (1:1) wires can be sealed and transferred to calorimeter
- ❖ Cryogenic calorimeter can detect ~0.4J from “exploding” Pd wire
- ❖ Mass Spectrometer can detect He-4 and He-3 in gas and metal samples



Future Work

❖ Electrode Preparation:

- ^4He implantation or in-diffusion
- More Electrochemical loading

❖ Reaction Triggering:

- More Axial current (dc, pulse, sine wave) – 10^5 to 10^7 A cm^{-2}
- Surface laser
- Ultrasonic(?), TeraHertz(?) stimulation

❖ Heat Measurement:

- More Mass displacement at low temperature (LN)
- Mass flow at room temperature

❖ Reaction Products:

- Analyze wires and emitted gases for changes in their ^3He and ^4He content and ratio
- Search for isotope effects
- Neutrons and Gammas.

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