

# Studies of Reproducibility in the Field of Condensed matter Nuclear Science

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# Reproducibility

What does it mean?

Of what?

How?

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“Cold Fusion”

“does not exist”

“because the effect is irreproducible”

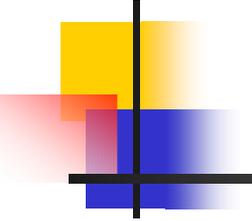
What is “the Cold Fusion Effect”?

How do we test “existence”

What does it mean “to reproduce”?

What protocol(s) should be used to test replicability?

What are the critical parameters of the effect under test?



# Reproducing the F-P Electrochemical Pd/D Heat Effect - a caution

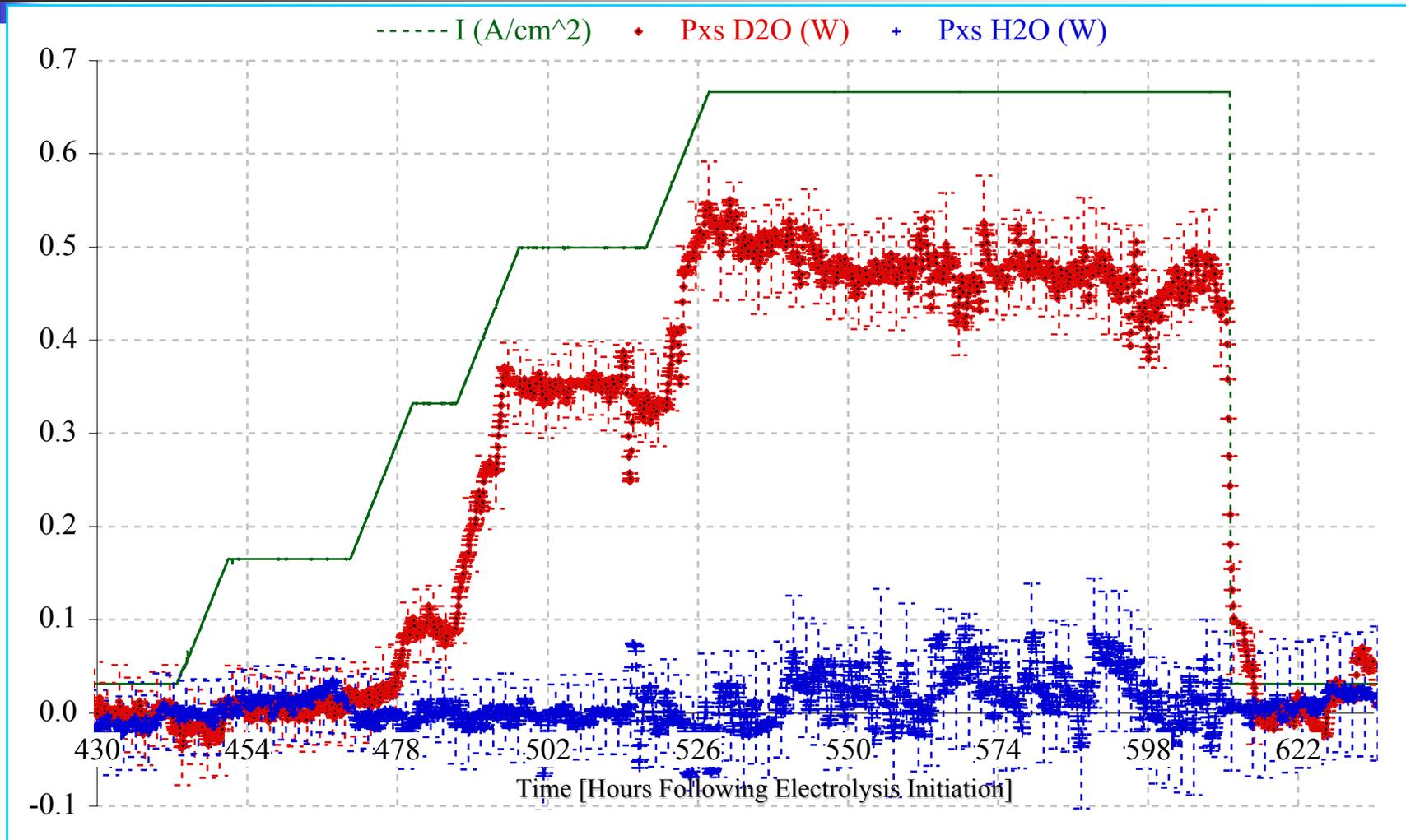
SRI 1989/90: *“An unexpected source of heat can be observed in the D/Pd System when Deuterium is loaded electrochemically into the Palladium Lattice, to a sufficient degree.”*

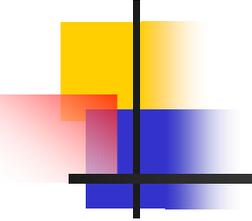
*Based on our experience with this system*

*“What should we do to achieve high loading”?*

- reduced temperature → flow calorimeter
- elevated pressure → resistance measurement of D/Pd
- 1M LiOD → Pt or SiO<sub>2</sub> cells

# Reproducing the F-P Electrochemical Pd/D Heat Effect



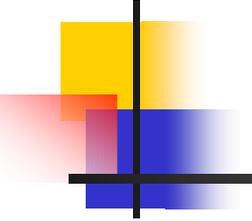


# Reproducing the F-P Electrochemical Pd/D Heat Effect - a caution

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CEA: Lonchamp, G., L. Bonnetain, and P. Hieter.  
*“Reproduction of Fleischmann and Pons Experiments”* in *Proc. ICCF6* Lake Toya, Hokkaido, Japan (1996).

- Various techniques have been utilized to produce excess heat.
  - Numerous results have been obtained [which] seem random, and
  - even sometimes contradictory with each other.
- 
- “simply” reproduce the exact experiments of Fleischmann and Pons
  - to ascertain the various phenomena involved
  - in order to master the experiments.



# Reproducing the F-P Electrochemical Pd/D Heat Effect - a caution

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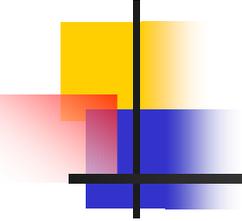
CEA: Lonchamp, G., L. Bonnetain, and P. Hieter.  
*“Reproduction of Fleischmann and Pons Experiments”*

The F-P Calorimeter with precautionary measures taken is:

- *“simple and precise”*
- *“very accurate and well adapted to study cold fusion”*
- *“the maximum error might be in the higher temperature range, and should not exceed 1%”*

The Measured Effect is:

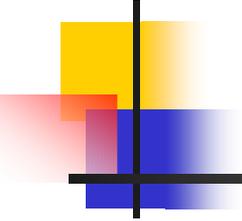
- *“below 70°C, between 0 and 5%”*
- *“between 70°C and 99°C, about 10%”*
- *“at boiling, up to 150%”*



# The SRI Reproduction Protocol

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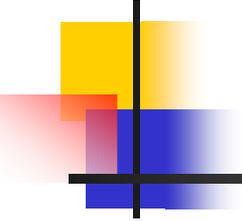
- 1) “Host Hands Off Rebuild” by the Experimenter with simple technical support but no attempted “improvement”
  - phase complete when results are deemed satisfactory by the experimenter.
- 2) Transfer of procedures and performance characteristics.
- 3) Host operation of experiment with added/improved diagnostics as appropriate.



# History

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- Miles/Bush Heat & Helium (1993).
- Srinivasan Ni/H<sub>2</sub>O/CO<sub>3</sub><sup>=</sup> Heat (1994).
- Patterson/Cravens Packed Bed Heat (1995).
- Stringham/George Acoustic Cavitation (1996).
- Arata/Zhang DSC Heat & Helium (1996-1997).
- Celani/Spallone/Tripodi Loading of Fine Wires (1998).
- Stringham Cavitation/Sonoluminescence (1999).
- Case Catalyst Heat & Helium (1998-2002).
- Letts/Cravens Laser Effect (2003).

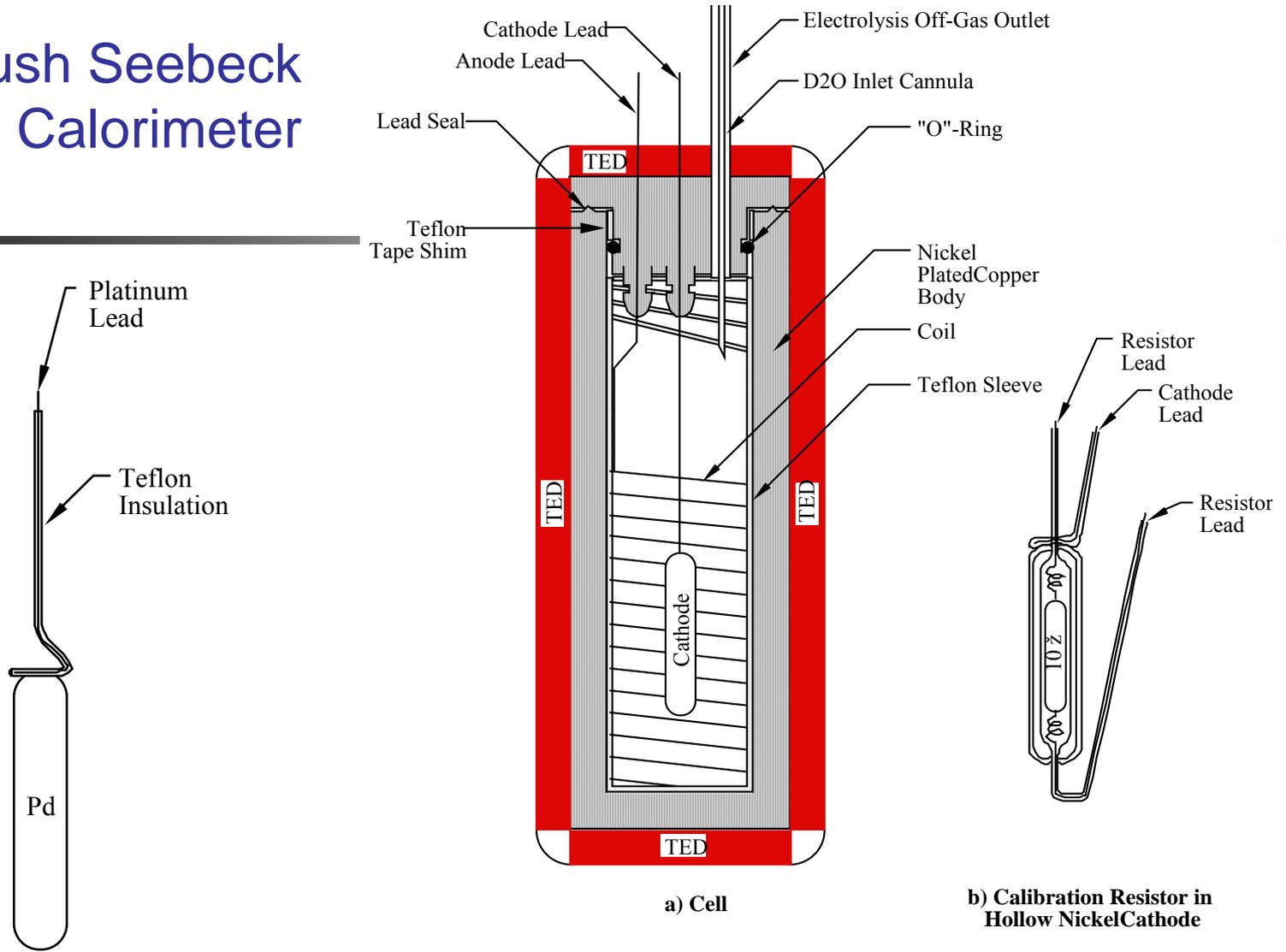


## Miles/Bush

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- Attempt to reproduce the seminal studies by Mel Miles & Ben Bush at China Lake in which  $P_{XS}$  was correlated with effluent  $^4\text{He}$  production in freely electrolyzing cells.
- Ben Bush was invited to SRI to begin a Reproduction effort in 1993.
- Original experiments performed in Borosilicate glass cells.
- Experiment already somewhat “improved” by Bush.

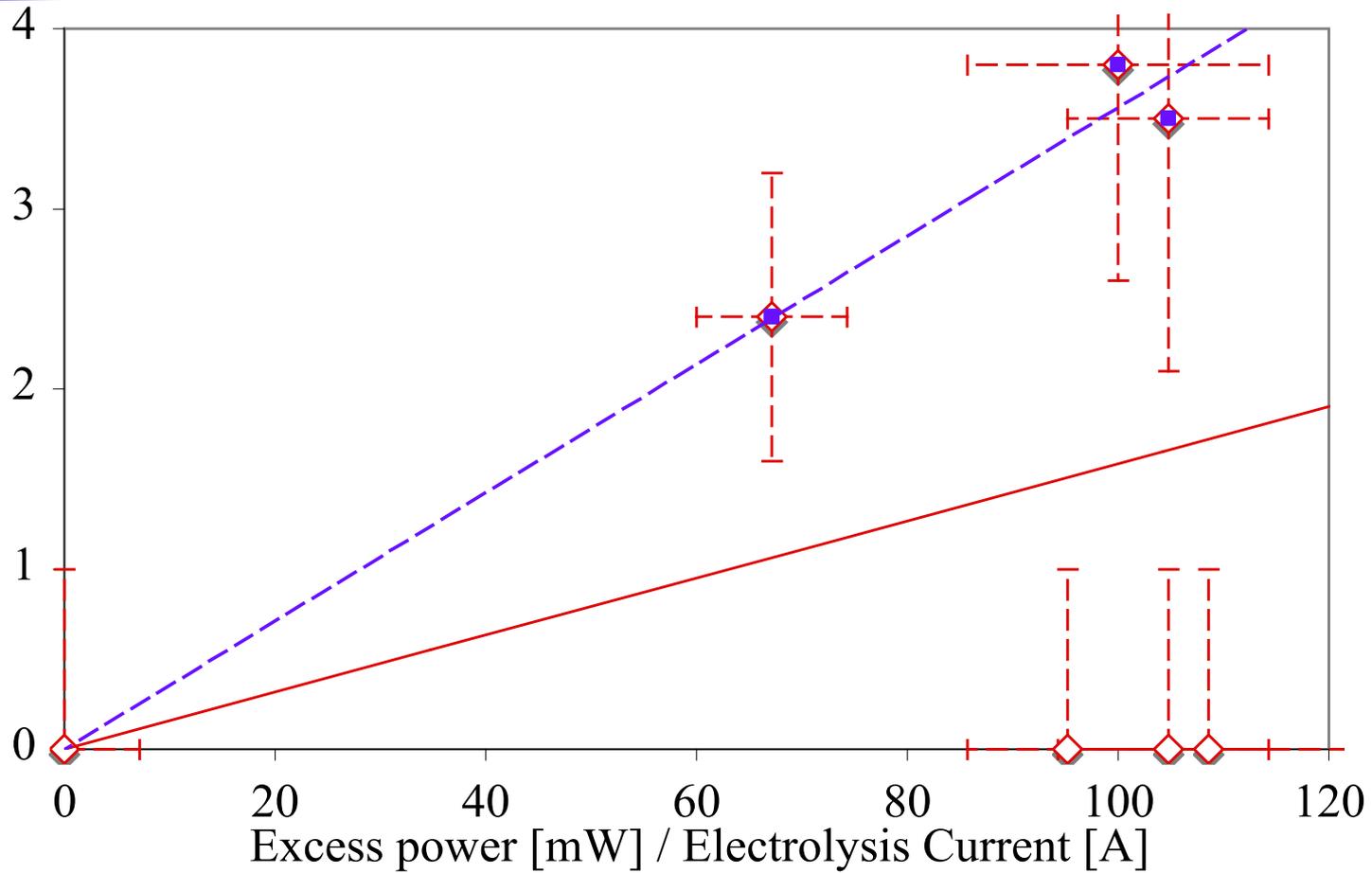
# Bush Seebeck Calorimeter



**Palladium  
Cathode**

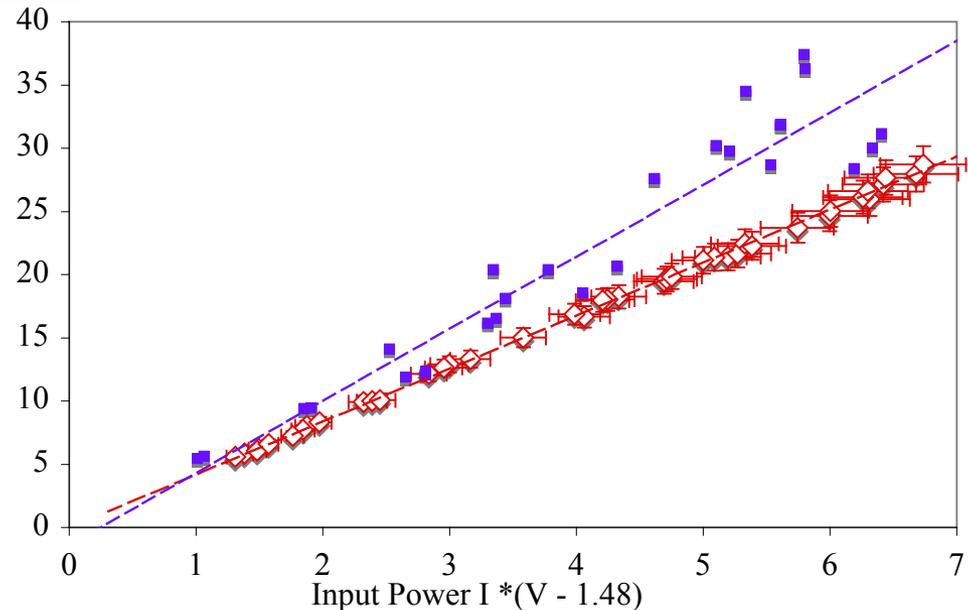
**Figure 2 Seebeck Cell and Calibration Resistor**

# Helium versus Excess Power

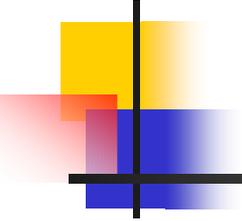


- Large Area Nickel Cathode
- Light Water / Carbonate Electrolyte.
- Open Cells

- $T_{\text{Cell}} - T_{\text{Ref}}$
- $K[I(V - V_{\text{TN}})]?$
- Std. Error
- IV ?



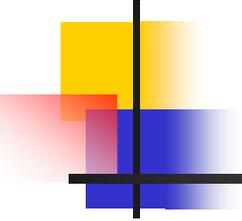
- Release of Electrolysis Gases declined or ceased
- Quantitatively correlated to “Excess”  $\delta T$
- Effect due to RedOx shuttle reaction.



# Patterson/Cravens

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- $T_{\text{Out}} - T_{\text{In}}$  for a flow-through packed bed of Pd(Ni) on plastic spheres  $> IV / [\partial M / \partial t * C_v]$
  - Effect obtained with light? water.
  - Observed independently by Patterson and Cravens?
  - Replication at Motorola?
- 
- Able to reproduce  $\partial T$  effect in freestanding flow loop.
  - When situated isothermally within mass flow calorimeter,  $\partial H$  effect  $\rightarrow 0$ .



# Arata/Zhang

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- Reproducible Excess Heat with D<sub>2</sub>O.
- In DS Cathodes containing nano-crystalline Pd.
- Averaging 15-80 W in different experiments.
- Excess heat ~1.8 times input Energy.
- Initiation time several 100 hours.
- Effect monitored up to 6 months.
- MS observations of <sup>3</sup>He and <sup>4</sup>He in Pd Black.
- No excess heat or helium from H<sub>2</sub>O.

# Arata/Zhang Double Structured Cathode

AZ1 0.3M LiOD, AZ2 0.3M LiOH

Cathodic Current 5 - 7.5A

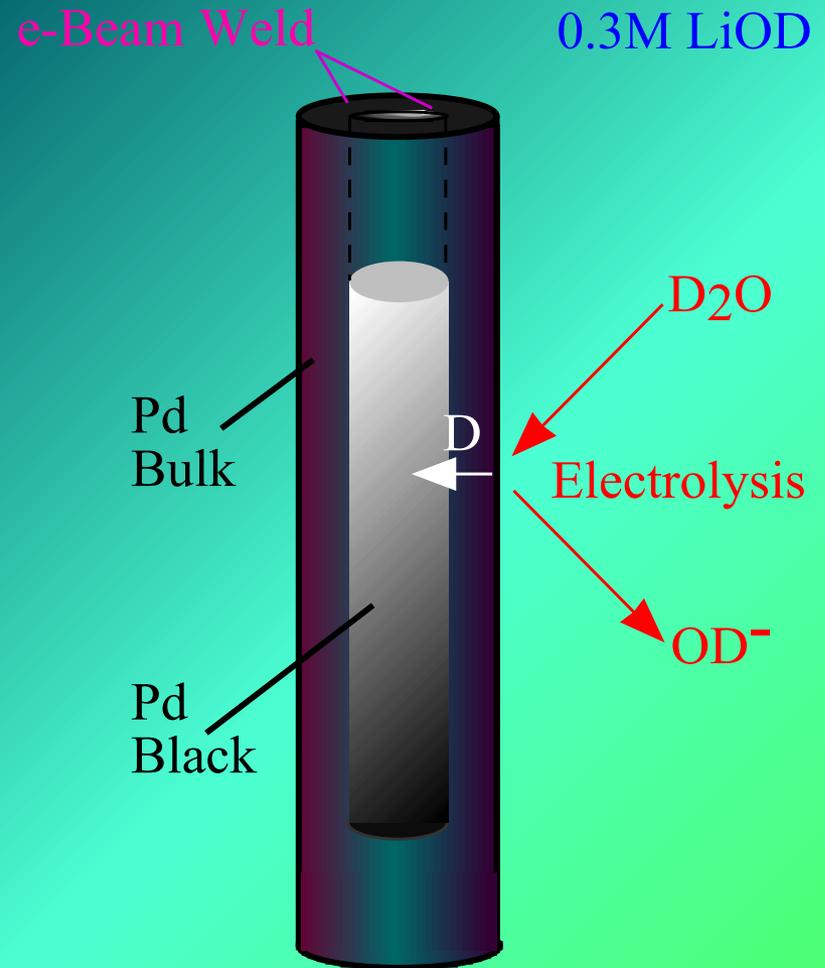
Current Density 170-255mA cm<sup>-2</sup>

P<sub>in</sub> 50-317 W, Duration 120 Days

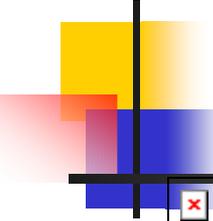
P<sub>xs,Max</sub> = 10 ±1.5%, P<sub>xs</sub> 0 ±1.5%,

Deloaded: open circuit and at 2V

Anodic for a further 100 Days.



Arata/Zhang "DS" Cathode:  
6cm long, 14mm dia., 3.5mm wall

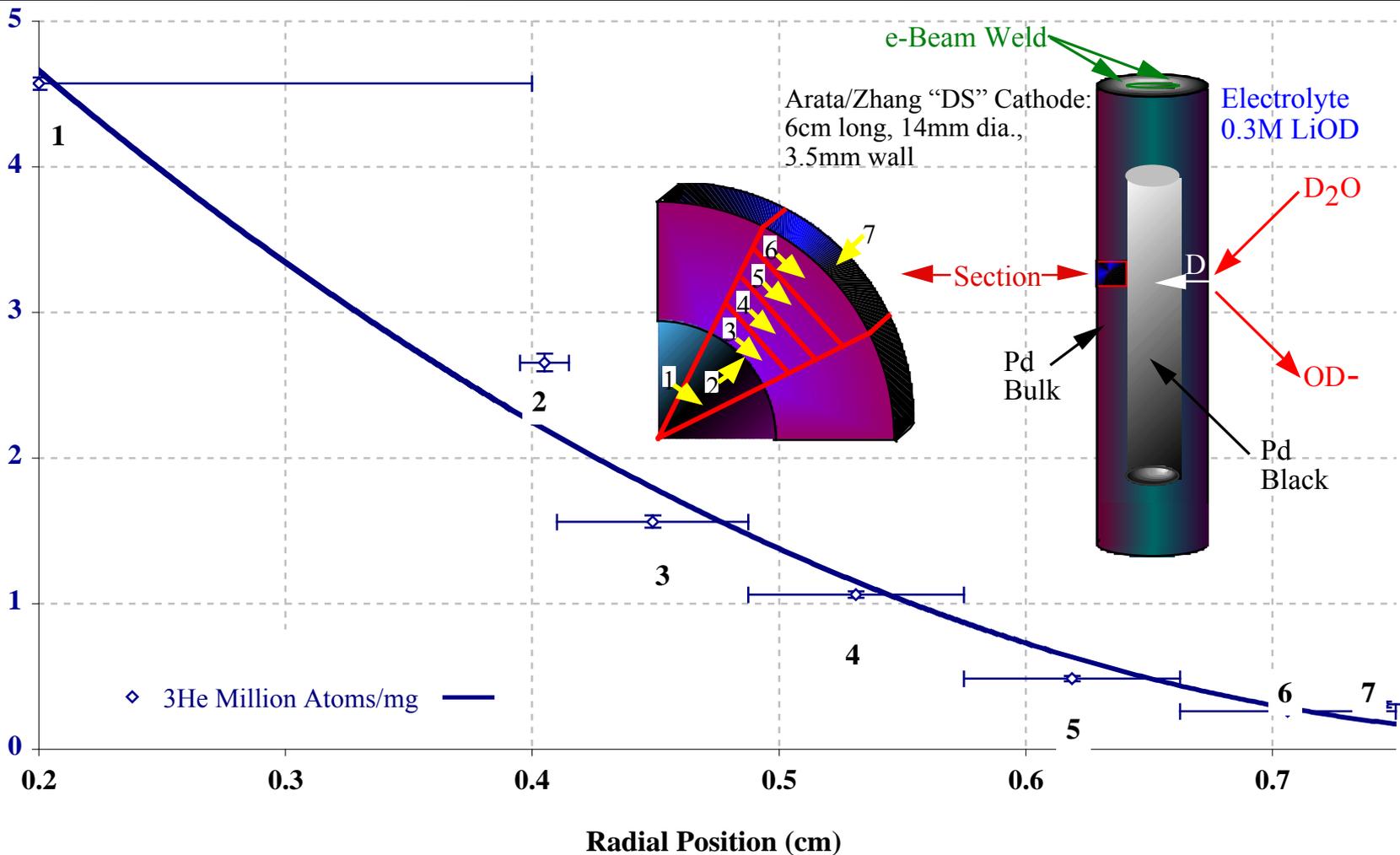


AZ1&2 0.3M LiOH & LiOD with “DS” Cathode  
Effects of Light and Heavy water electrolysis

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# AZ1: Radial Distribution of $^3\text{He}$ and $^3\text{H}$



# Tritium Conclusions

.....New Good News

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Production of Tritium was between  $2 \times 10^{15}$  and  $5 \times 10^{15}$  atoms.

Modeled as a single event, this occurred during cathodic electrolysis.

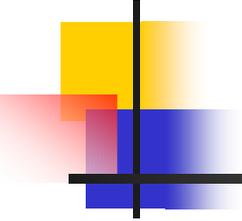
There is definite evidence of excess  $^3\text{He}$  from Tritium decay in all samples of Pd & Pd-black from the  $\text{D}_2\text{O}$  experiment.

Samples of Pd taken from a similar and contemporaneous  $\text{H}_2\text{O}$  electrode show low  $^3\text{He}$  levels consistent with blank Pd.

Measurements of the  $^3\text{He}$  gradient through the 3.5mm wall of the  $\text{D}_2\text{O}$  electrode show that the  $^3\text{He}$  is the decay product of Tritium which diffused from a source inside the electrode.

# Helium Conclusions

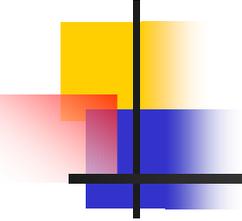
.....New Bad News



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No evidence was obtained for  $^4\text{He}$  quantitatively consistent with excess heat. Possible explanations:

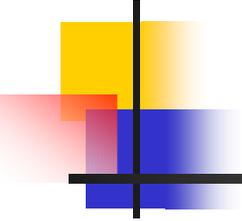
- (1)  $P_{XS}$  measurement substantially in error (at SRI & Osaka).
- (2) Helium was lost before sample collection due to microfractures, which occur in surface-stressed Pd.
- (3) Heat was produced as in F-P electrolytic cells at the cathode Outer Surface where loading, deuterium chemical potential, and stimulation are the greatest.



# Stringham

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- Cavitation of Pd and other alloys in D<sub>2</sub>O.
- Complete Immersion of Oscillator and Reactor in SRI Mass Flow Calorimeters.
- Qualitative Observations of Luminescence.
- Input Power averaging 8W in different experiments.
- Each Experiment last about 24 hours.
- No Excess Heat found at SRI.

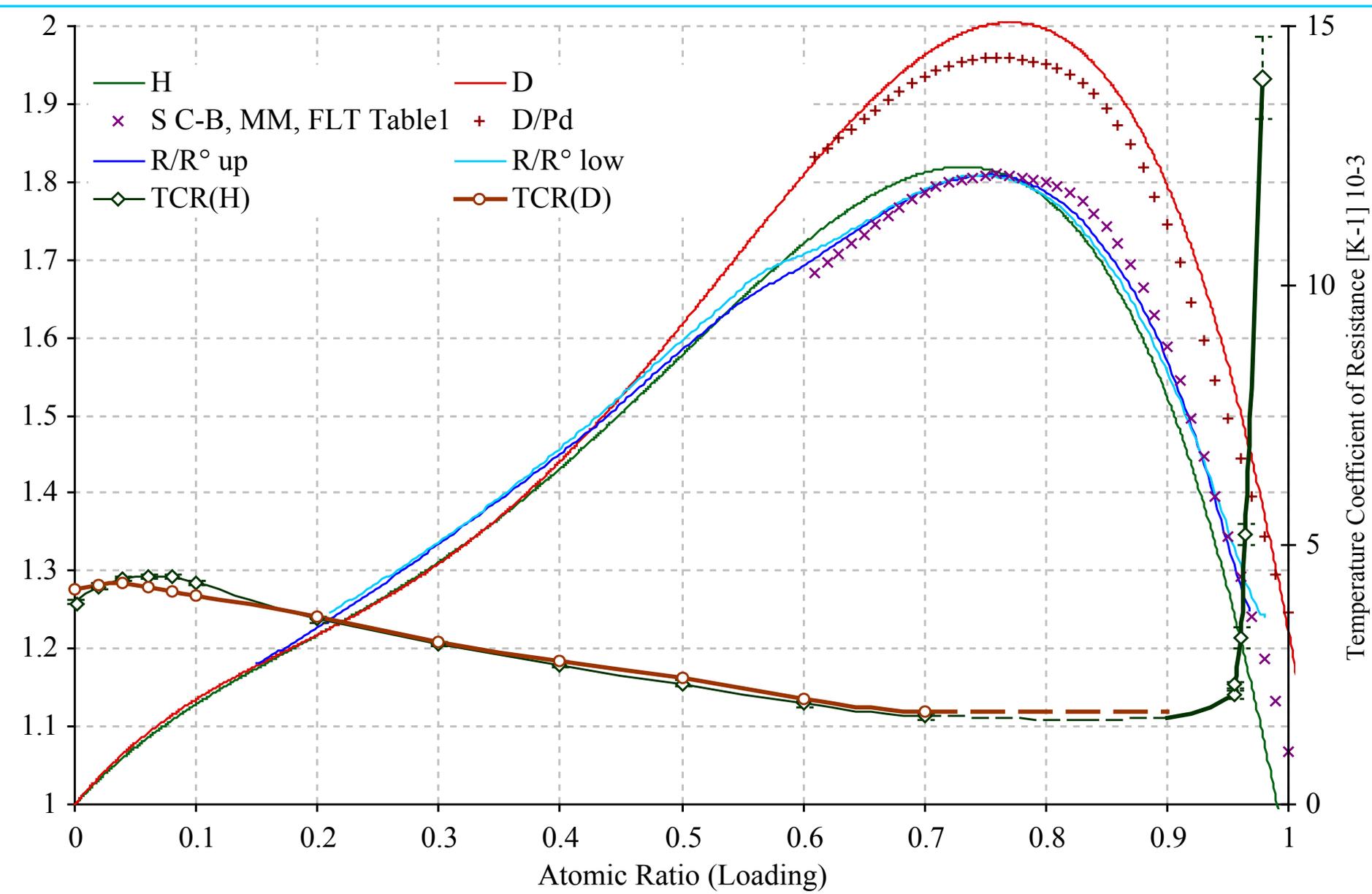


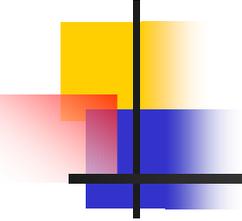
# Celani/Spallone/Tripodi

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- Attained high loading in “long thin wires”
- Very dilute electrolyte:
  - high voltage ?
  - low impurity ?
- Alkaline earth Cation [ $\text{Be}^{++}$ ,  $\text{Mg}^{++}$ ,  $\text{Ca}^{++}$ ,  $\text{Sr}^{++}$ ,  $\text{Ba}^{++}$ ].
- IR “flash” to activate mounted cathode.
- Use of HM’s [ $\text{Hg}$ ,  $\text{Cd}$ ,  $\text{Pb}$ ,  $\text{Sn}$ ,  $\text{In}$ ,  $\text{Tl}$ ] to seal loading.
- Experiments performed by Paolo Tripodi initially using apparatus developed at INFN (Frascati).

# Loading and Temperature coefficient of Resistance



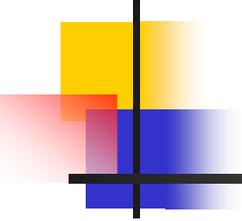


## Case

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L.C. Case, “*Catalytic Fusion of Deuterium into Helium-4*”, Vancouver, Canada (1998).

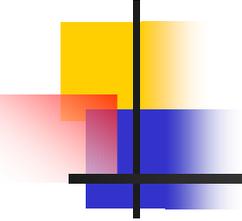
- Excess Temperature Effect ( $D_2$  vs.  $H_2$ )  $> 20^\circ C$ .
- PGM on Carbon Catalyst 0.5 - 1% loading.
- 1 - 3 Atm. gas at 130 - 300°C.
- $\sim 100$  ppm  $^4He$  in  $D_2$ .
  
- 1.6 Liter 304 SS Vessel on heating mantle.
- 50 - 100g Catalyst.



## Case - First Attempt

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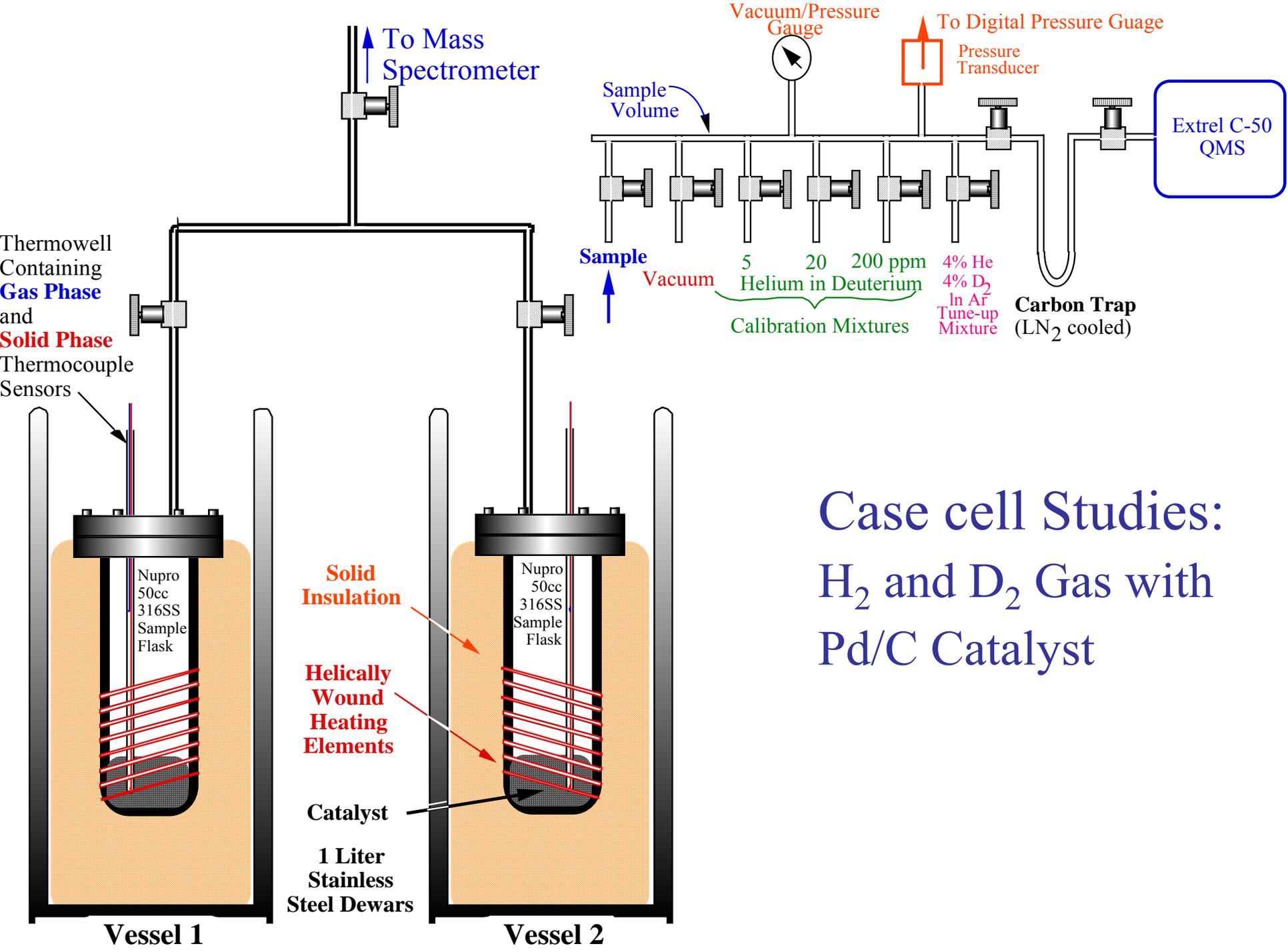
- Working with R. George at SRI.
- Case written and oral procedures.
- Case qualified catalyst (0.4 & 0.5% Pd on C).
- 10g catalyst in 50cc Nupro SS Sample Flask.
- Isothermal Calorimeter and on-line QMS.
  
- No  $\partial T$  Effect!.
- No Evidence of Helium Production!!



## Case - Visit

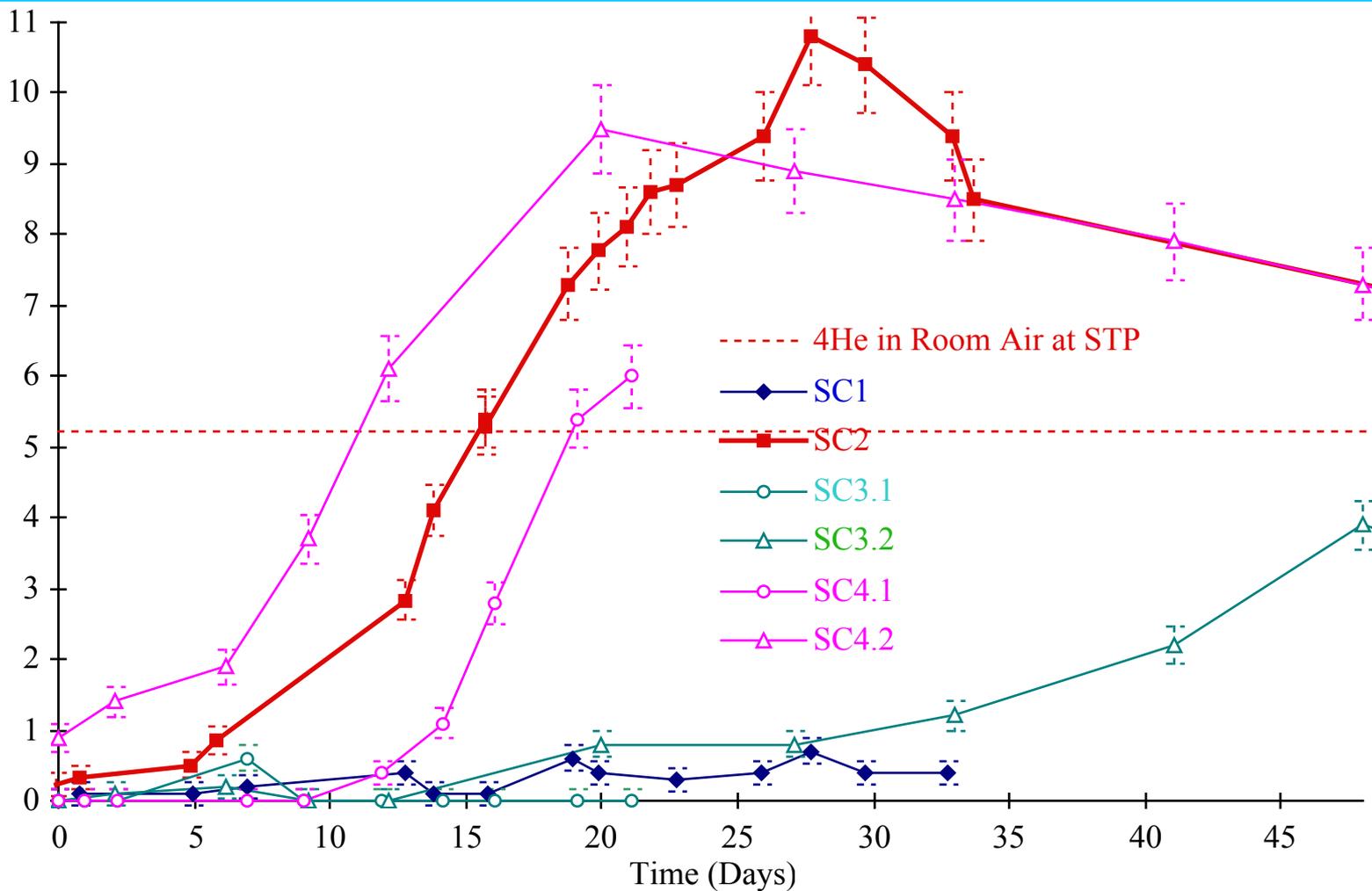
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- Stood beside Case to watch without interference .
- Substantial and subtle differences in interpretation.
- Not all Catalysts were active.
- $\partial T$  Effect was clearly demonstrated!
  
- Some concerns about Heat Quantification.
- No attempt made to measure  $^4\text{He}$ .
- Satisfied that renewed effort was justified.

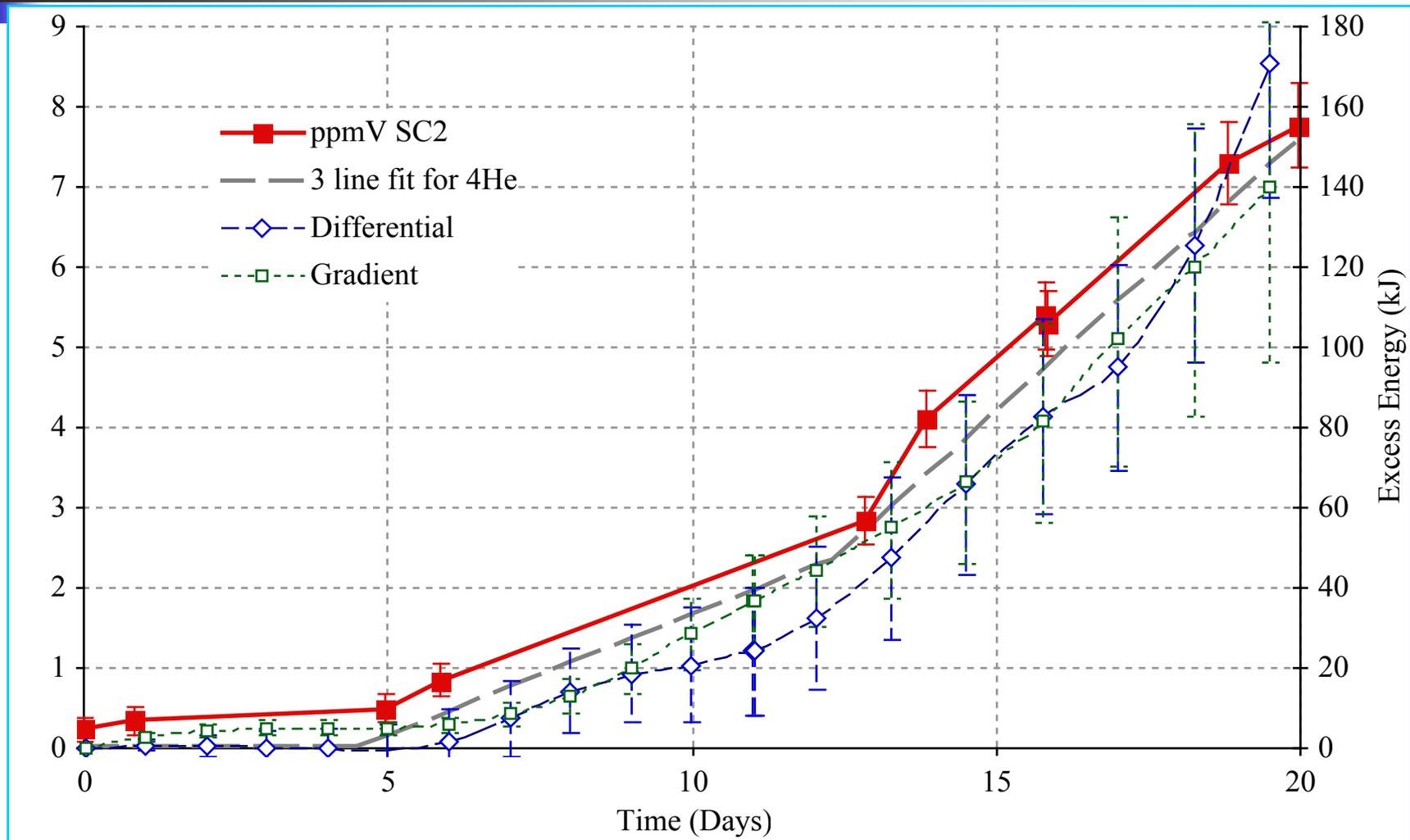


Case cell Studies:  
 H<sub>2</sub> and D<sub>2</sub> Gas with  
 Pd/C Catalyst

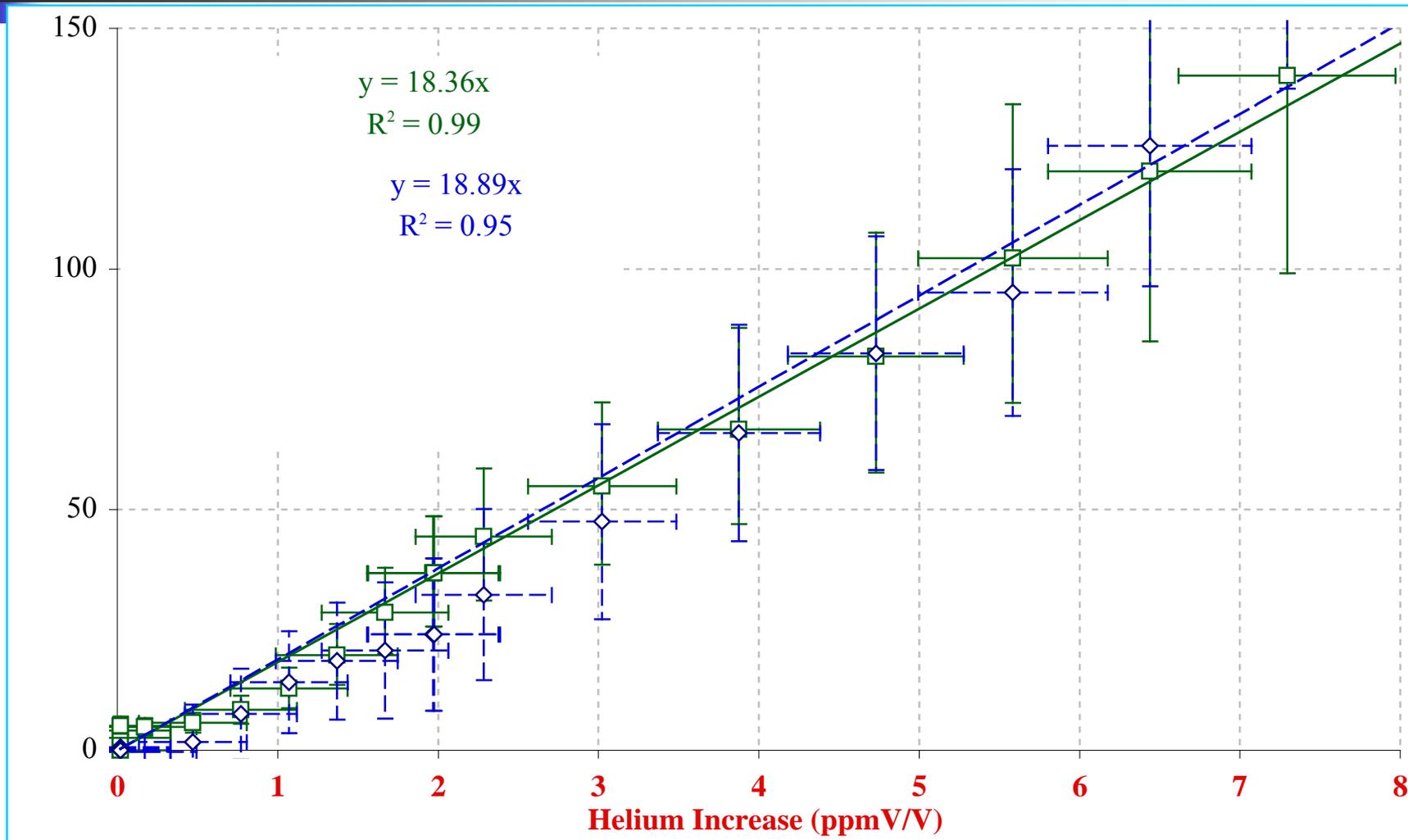
# Case: $^4\text{He}$ vs. time

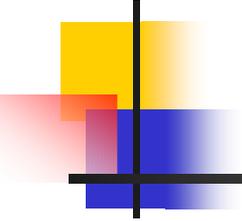


# Case: $^4\text{He}$ and Heat vs. time



# Case: "Q"-Value - Energy vs. $^4\text{He}$

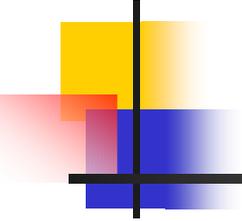




## ENEA Frascati

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- Violante Group at ENEA (Frascati) are presently reproducing the SRI || Case reproduction.
- Assistance from Sued Chemie (parent of UC)
- Preliminary results very encouraging.



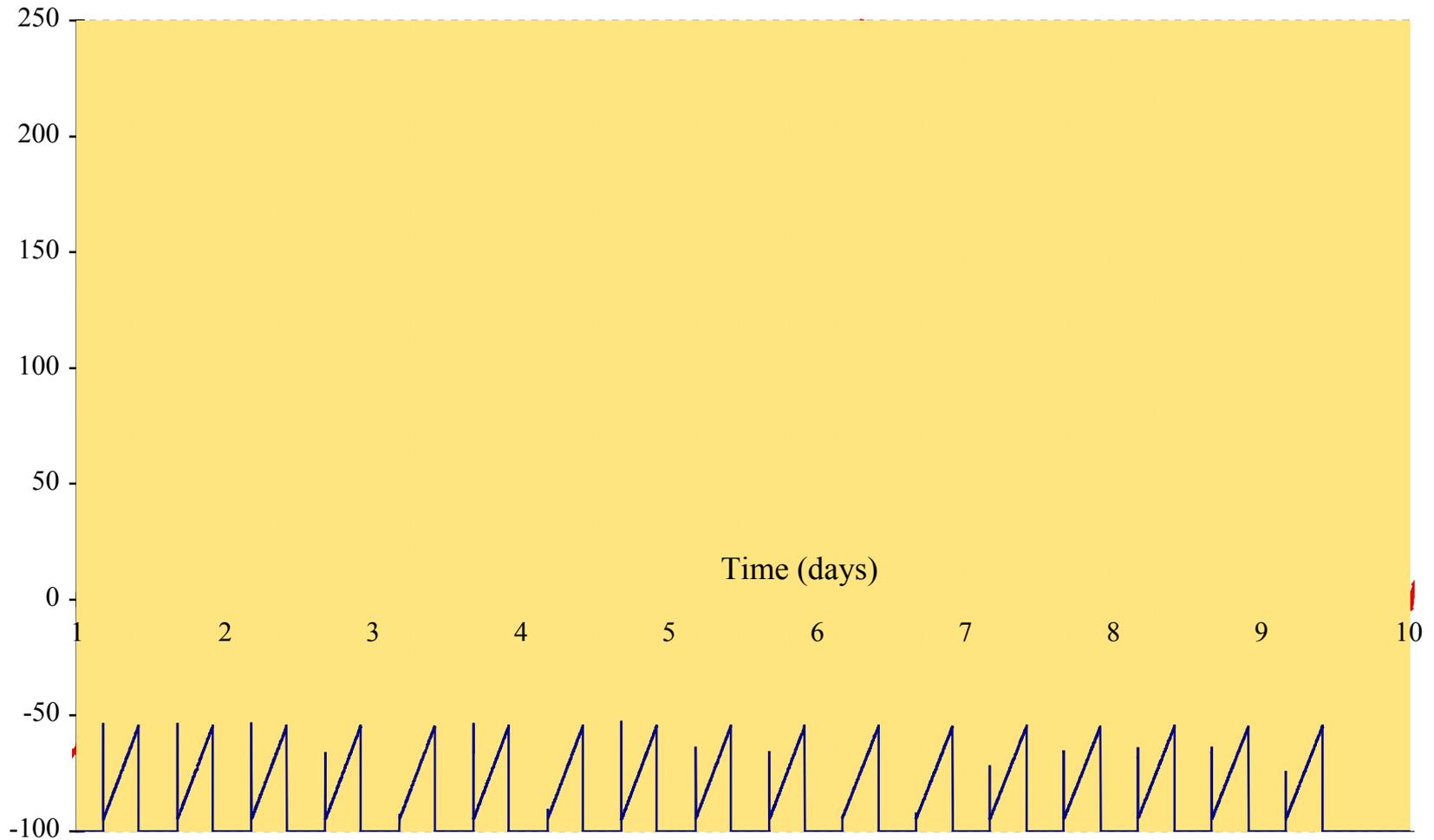
## Letts/Cravens

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- Laser stimulation of excess heat effect
  - Initiation or Amplification ?
- Following loading,
  - and surface modification.
- Power gain 10 - 100.
- Letts visit to SRI May? 1993.
  
- Letts || Cravens, Storms, Cobalt, Hagelstein

# Experiment 3A Result Days 1-10

## Project Cobalt



# Conclusions Experiment 3A

- Experiment 3A yielded between **50 mW** and **250 mW** of excess power starting on day 4 and continuing through day 9 (December 5, 2002)
- Excess Power was produced in apparent correlation with the metal co-deposition and laser stimulation (**17mW** Thermal).
- Given that the volume of the cathode was  $0.00875 \text{ cm}^3$ , the maximum power density was  **$\sim 28 \text{ W cm}^{-3}$** .
- The total amount of excess energy produced,

$$E_{\text{xs}} = 25\text{kJ} (\sim 7\text{Wh}) = \mathbf{320 \text{ kJ / Mole Pd.}}$$

Reproduction can be fulfilling...

even in the attempt

- but far from straightforward.

- The experimenter may not be aware of All critical details.
- Communication is imperfect.
- Postpone the Compulsion to Improve!
  - “*simply*” reproduce the exact experiments of *F* and *P*
  - to ascertain the various phenomena involved in order to master the experiments.

G. Lonchamp *et al* (1996)