Argonne NATIONAL LABORATORY

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U.S. Department of Energy

THE UNIVERSITY OF CHICAGO

Office of Science U.S. DEPARTMENT OF ENERGY

A U.S. Department of Energy laboratory managed by The University of Chicago

Sample Environment Innovation at the General Purpose Powder Diffractometer

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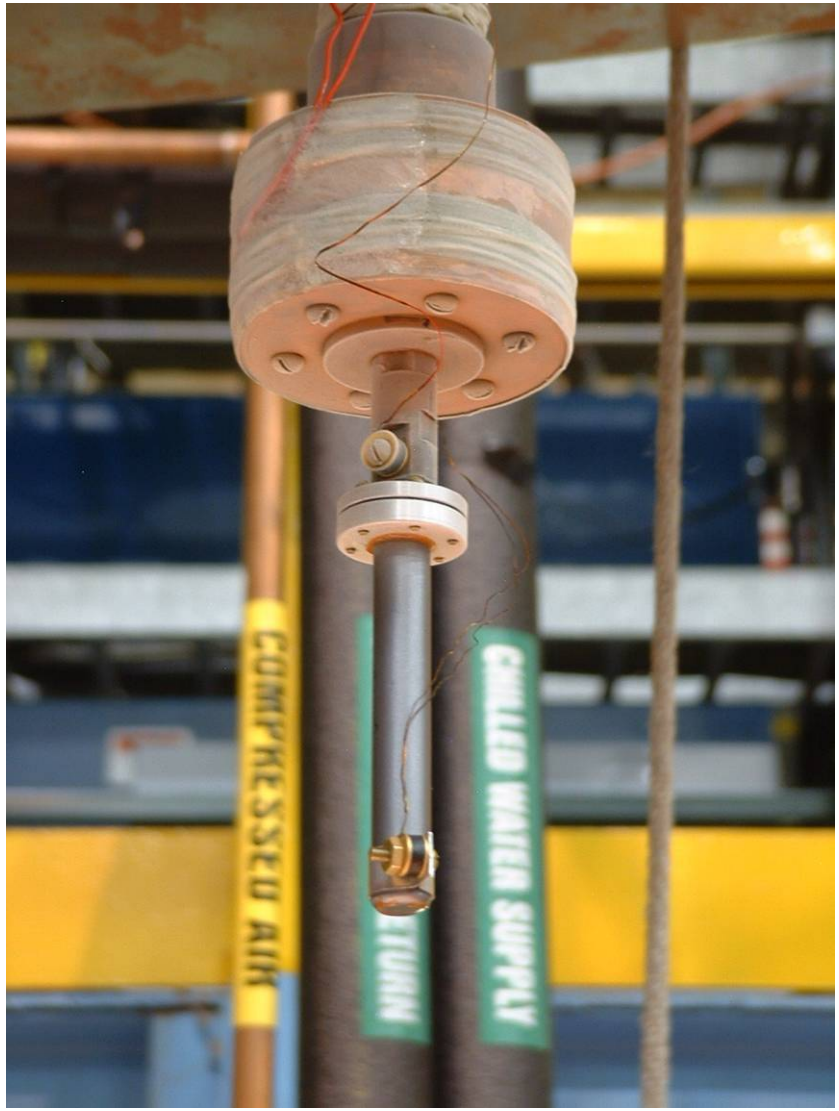
Intense Pulsed Neutron Source, Argonne National Laboratory

Thermal Gradient?

Test

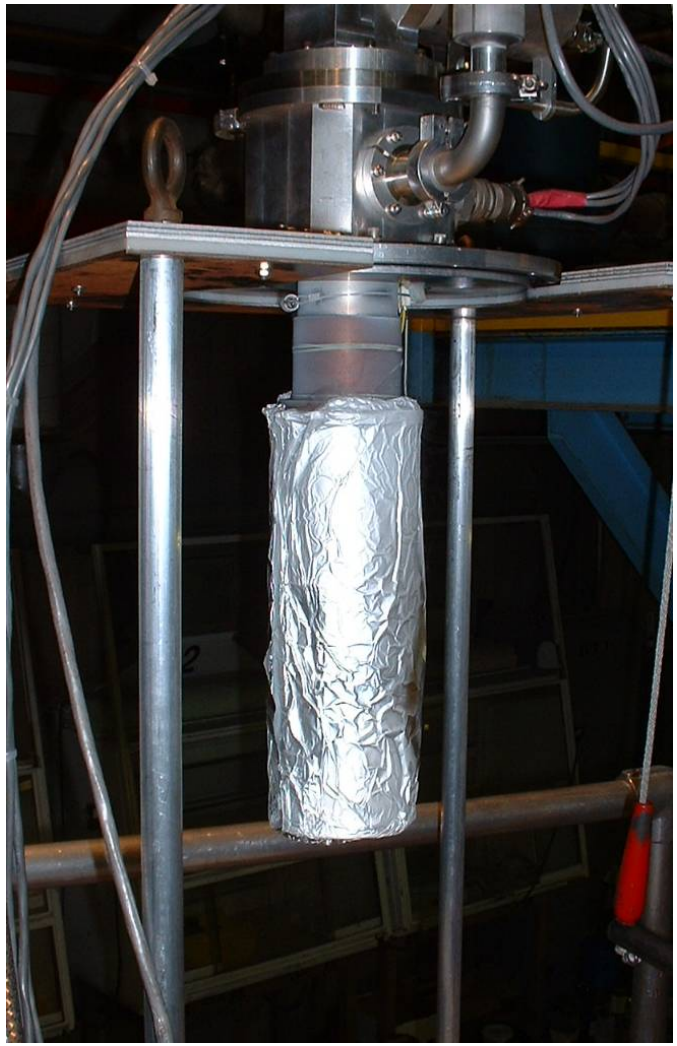
- Long Can
- Al2O3 non thermally conductive “sample”
- Diode at both ends of can

Found: an unacceptable thermal gradient.

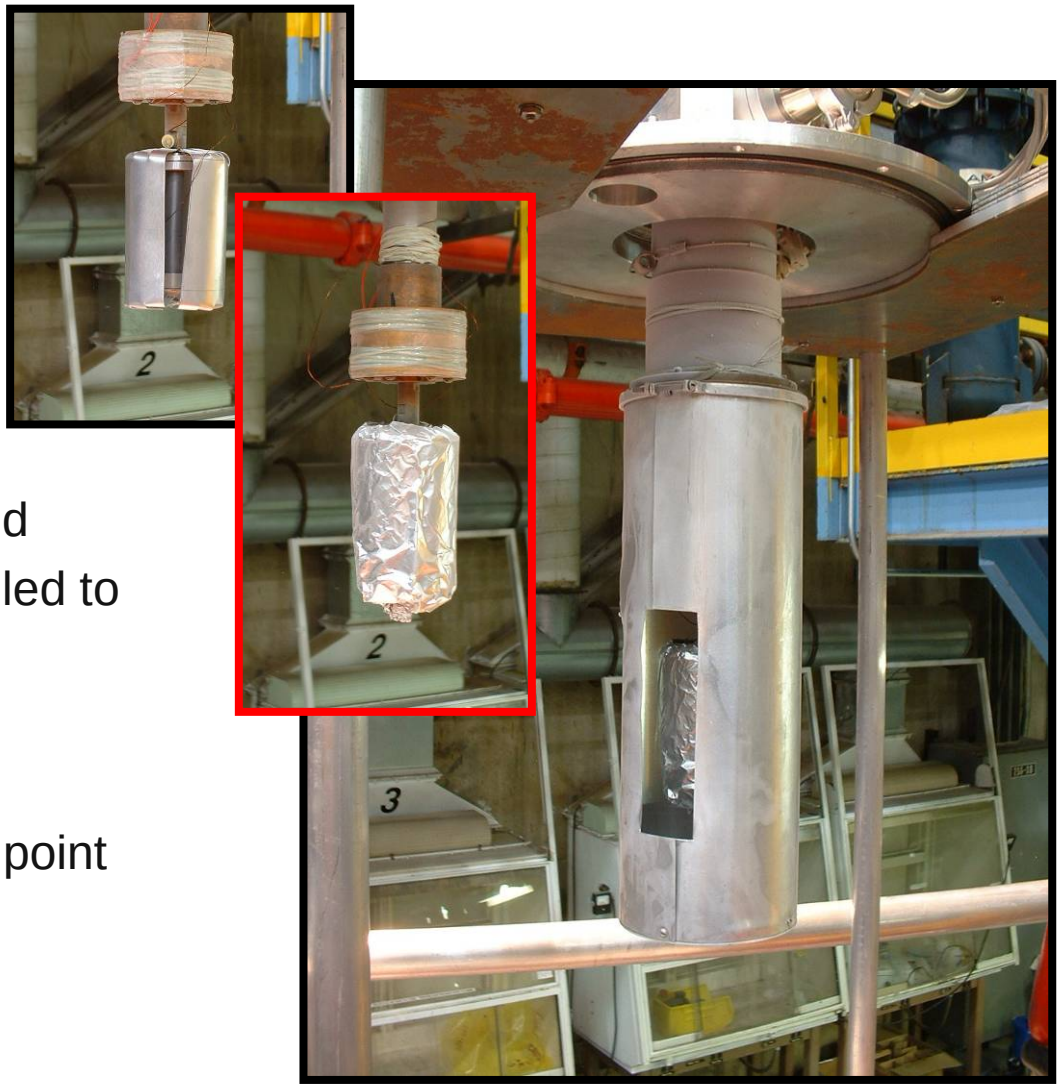


First…

- Covered entire shield with Al foil (this closed the large incident beam openings)
- Fixed the thermal gradient problem at low temperatures
- High temps (closer to RT) still had a gradient across the V-can



Then….



- Simulated an inner heat shield
- Temperature of I.H.S. controlled to sample temperature

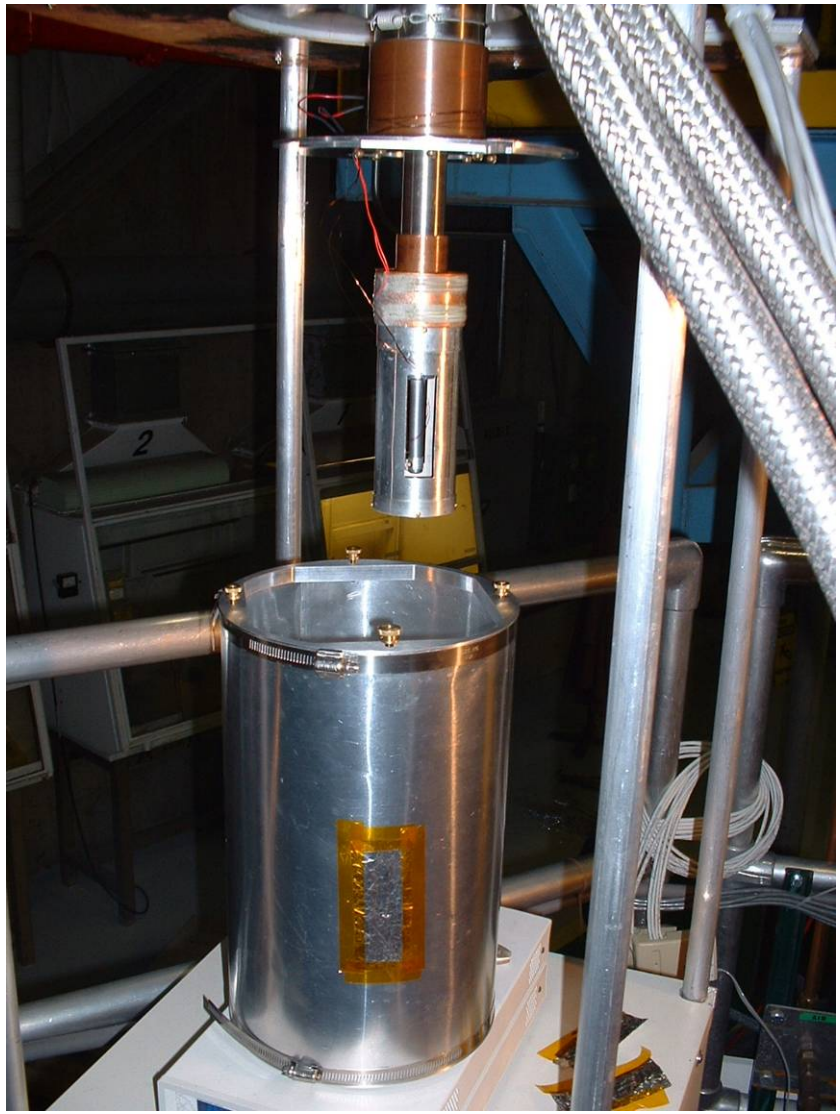
Result:

Eliminated gradients at any set point (RT to 10K)

New heat shields!

- Outer shield from previous IPNS design
- Inner shield is unique
- Both have Aluminized Mylar windows
  (no diffraction from these windows)
- Both designed with “ease of use” features:
  - Keyed mounting for alignment
  - Knurled thumb nuts O.H.S.
  - Loosen to release screws I.H.S.

Result: Works like a dream!



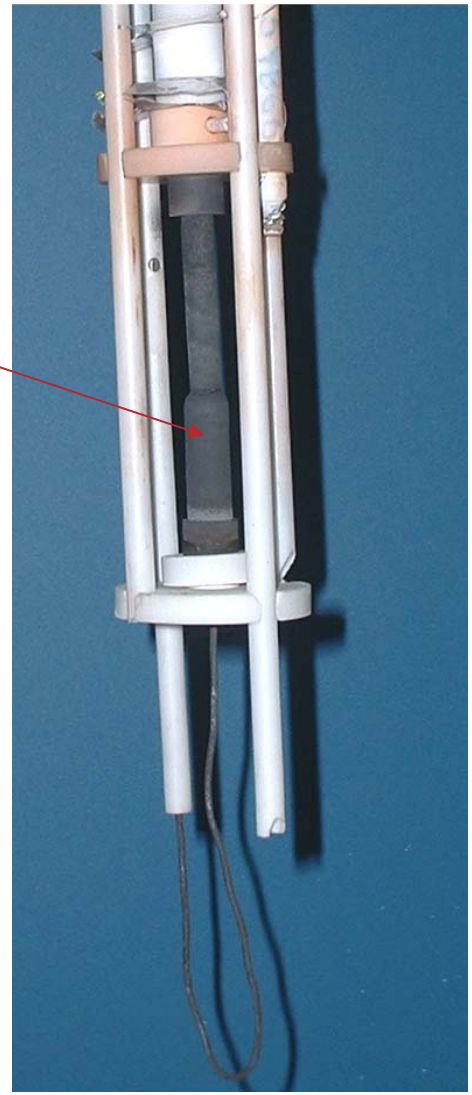
Miller Furnace Tubular Sample Holder

Challenge:

Mount a tubular ceramic membrane in the miller furnace and have a reducing environment on the outside of the sample and an oxidizing environment on the inside of the sample. Temperature: ~ 900° Celsius

Miller Furnace Tubular Sample Holder

- **Accepts Tubular Sample**
  - **hard ceramic**
  - **½” diameter**
- Inconel Tubing
- Uses Compression Spring
  - outside of the hot zone
  - applies ~ 1-2 lbs. pressure
- Gold Gasket



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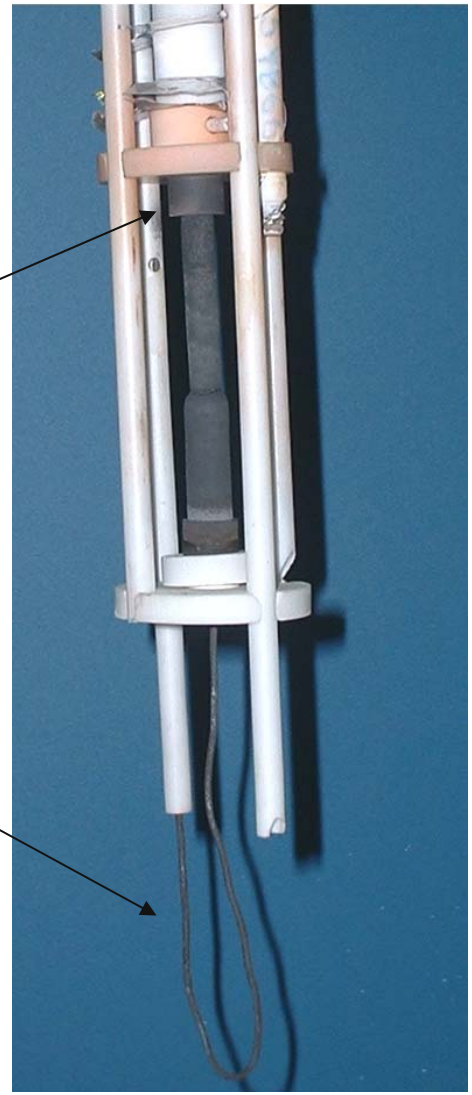

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- Accepts Tubular Sample
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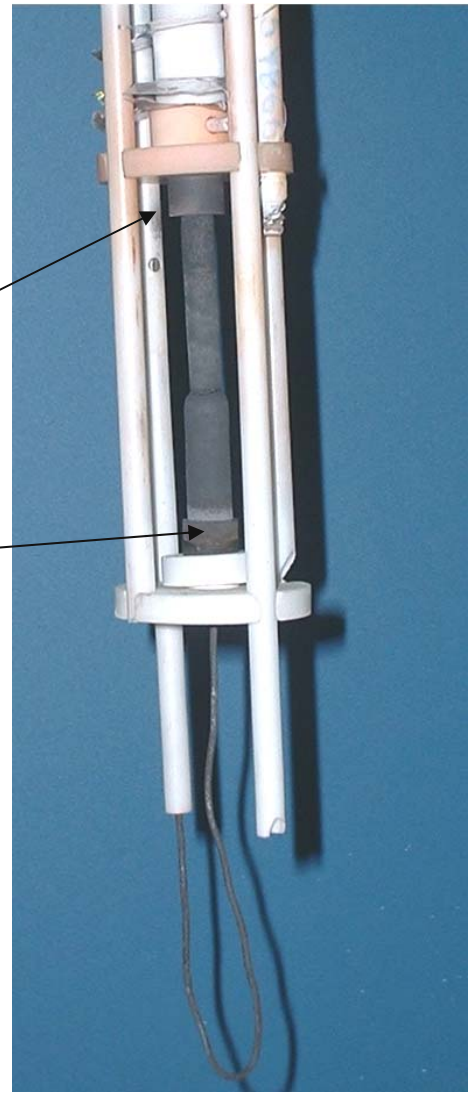

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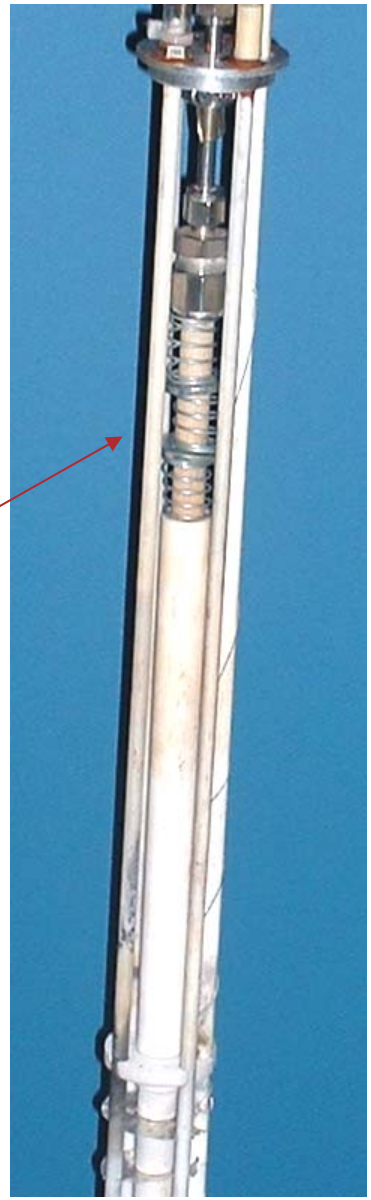


Furnace Adaptation To Study Catalytic Reactions

Sample Environment Challenge:

- Corrosive Ammonia Gas
- High Temperature
- Oxidizing
- Humid
- Sample reacts with Alumina (cannot be done in the Miller Furnace)

Answer: Create and adaptation to the Howe Furnace

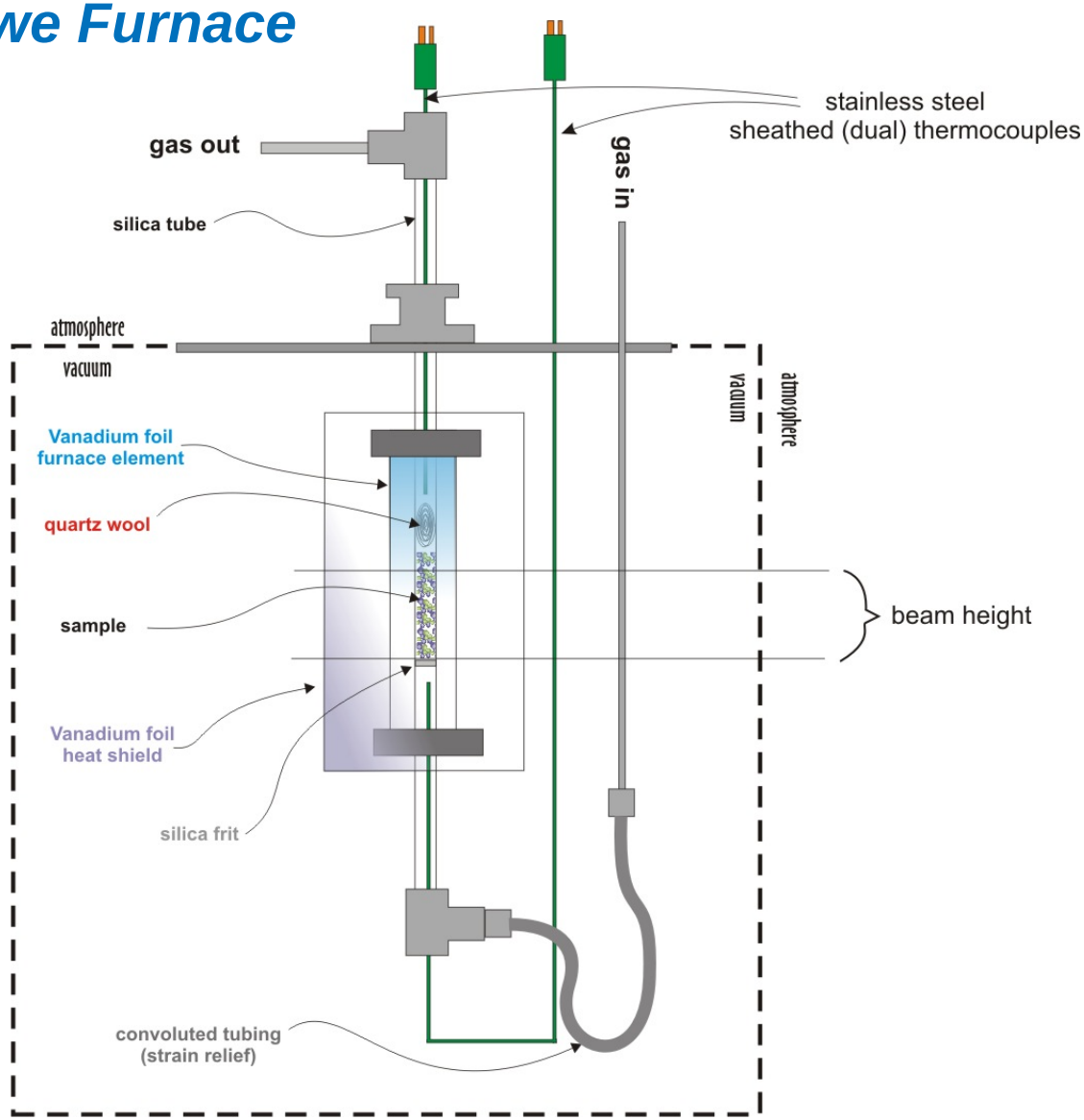
Initial Failure

Steel Can

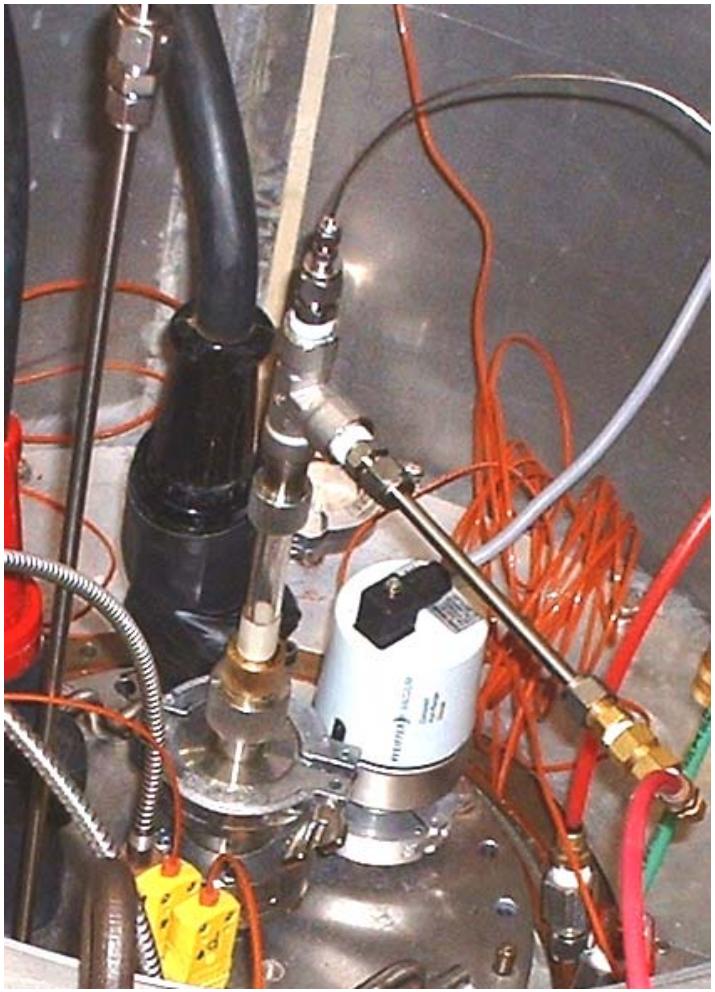
- adverse results in furnace
- scattering from can obscured sample data

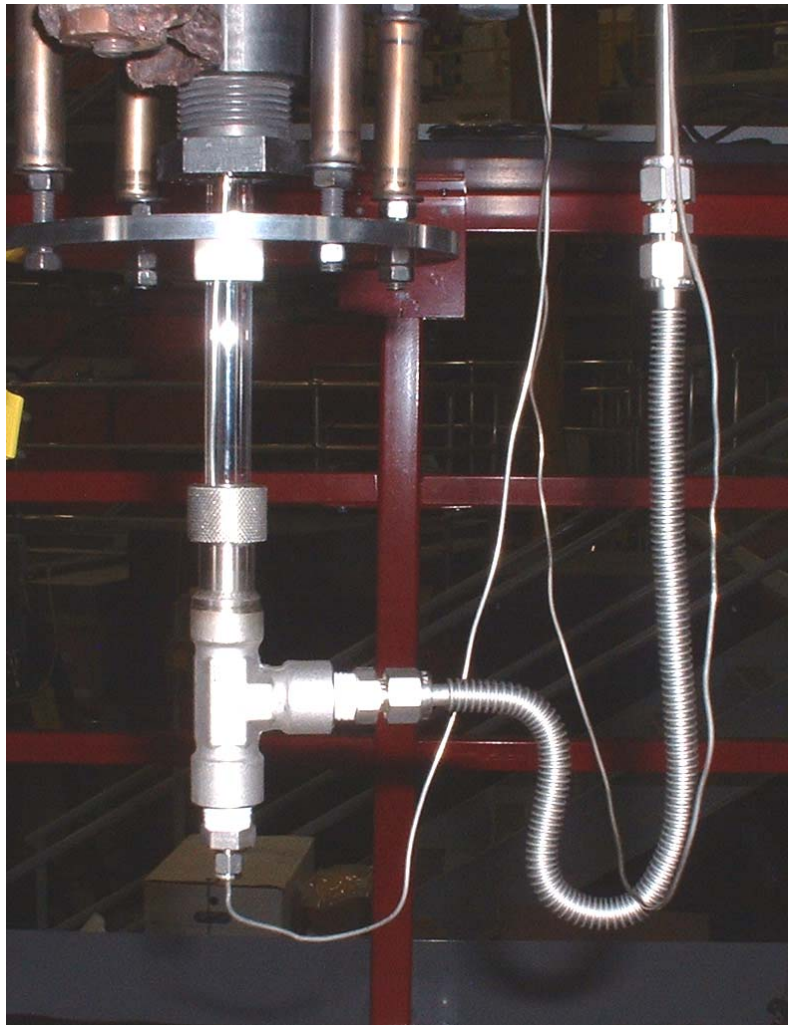
Volin Adaptation to Howe Furnace

- Utilizes a quartz tube
  - does not react with sample
  - adds to background
  - amorphous (adds no real peaks)
  - utilizes a frit to suspend powder sample
  - gas flow
- Allows scattering in all detector banks



Howe Furnace with Volin Adaptor





Humid RT sample environment

Challenge:

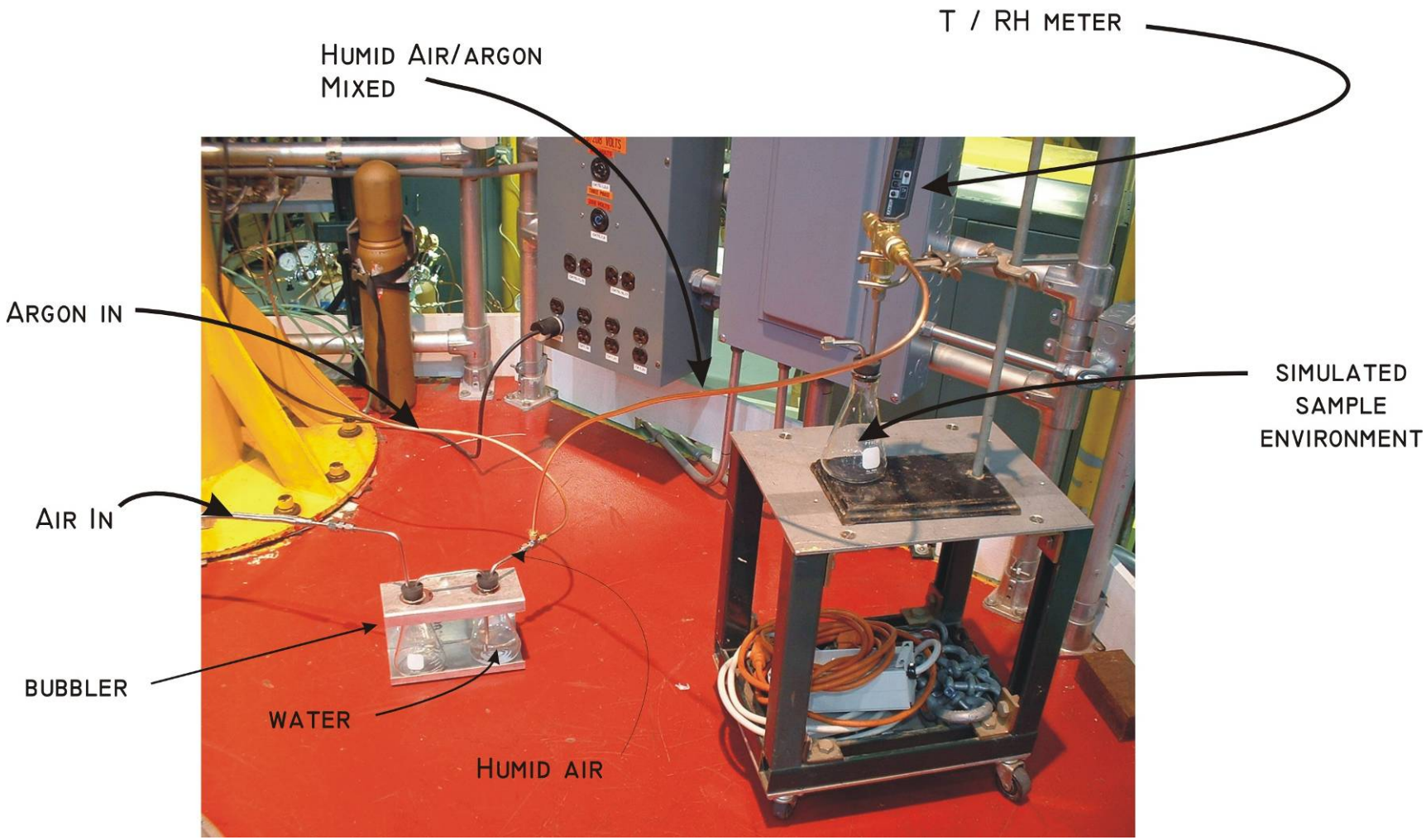
- Accurate
- Controls at around 30%RH
- Costs 0$

Answer: *Utilize existing equipment*

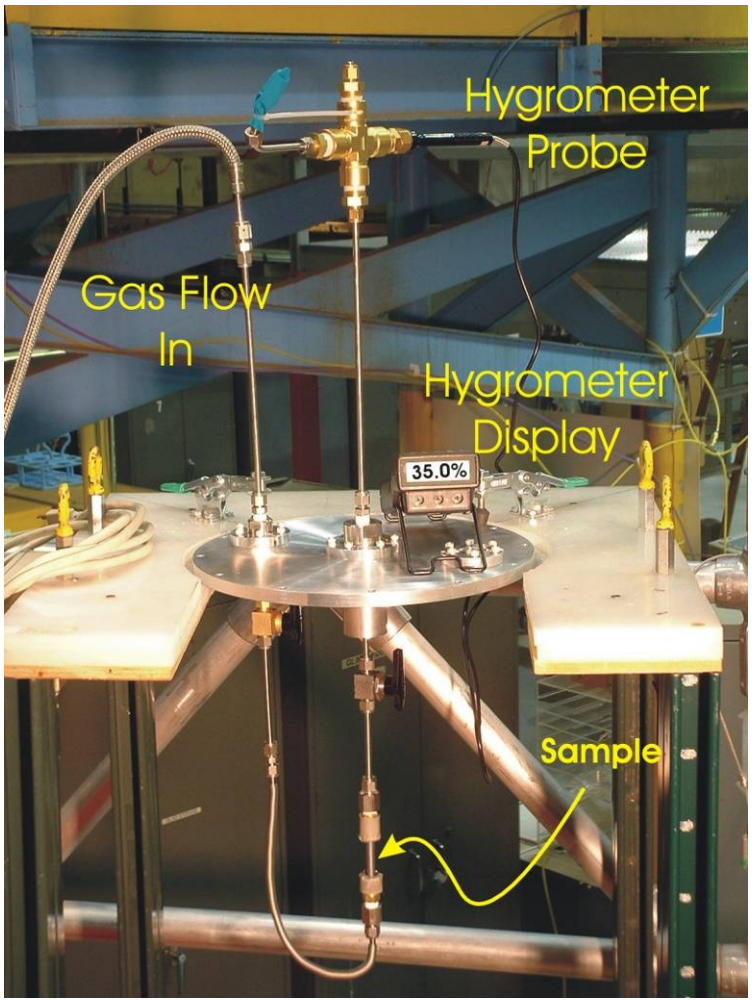
- Mass Flow controllers (mix humid and dry gasses)
- Bubbler
- Hygrometer Pen

Test

Controlled Humidity Gas Delivery Test Set Up



Application



Results:

Evan, Ken and Rich,

Indeed, this was a spectacular success! The users are going away with a wealth of new data, and your device performed perfectly. Based on the technical conversations I overheard between the professor and student, this may be first *in situ* scattering measurement of an inorganic anhydrous-to-anhydrous phase transition promoted by humidity.

Way to go!

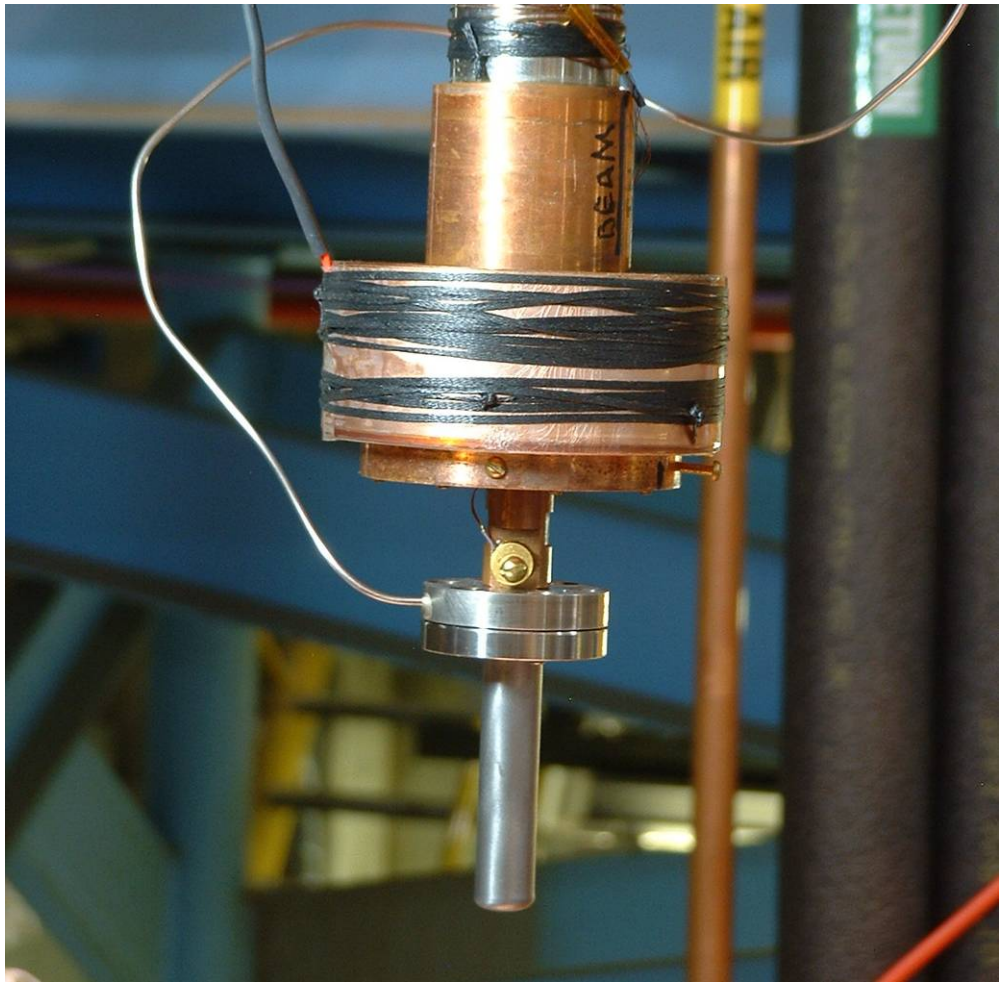
Jim R.

Happy Scientists ☺

O.K., We purchased a new hygrometer probe: $350 (its almost 0$)

Simple and Effective

A “NIST” V can with a modified steel top for low pressure/ low temperature Hydrogen (D2) loading.



First mounted ex-situ to a hot stage Displex and the sample was desiccated at about 150°c

Then mounted to a CCR in-situ and lines vacuum pumped.

Finally a low pressure loading of Deuterium gas

Thank You

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