Effect of temperature gradient on calorimetric measurements during gas-loading experiments

O. Dmitriyeva[1,2,]*, R. Cantwell[1], M. McConnell[1], and G. Moddel[2]

[1] Department of Electrical, Computer, and Energy Engineering, University of Colorado, Boulder, CO 80309-0425, USA

[2]Coolescence LLC, 2450 Central Ave Ste F, Boulder, CO 80301, USA

**Corresponding author. Tel:+1 7205659690*
E-mail address: olga.dmitriyeva@colorado.edu

Abstract

We studied the influence of the temperature gradient on heat measurements during gas-loading experiments. The quasi isothermal chamber that we conducted our experiments in is built to keep the inside temperature uniform. However, when experimenting at the elevated temperatures above 200˚C nonuniformities induced by the air flow inside the chamber produce local hot and cold spots.

The sample vessel inside the isothermal chamber went through pressurization/depressurization cycles to detect any temperature change due to gas-loading process. The presence of gas changes drastically the heat conduction inside the vessel. Therefore, when under pressure, the temperature probe couples to adjacent hot or cold spots more efficiently than when under vacuum. This coupling effect artificially shifts the measurement baseline up or down, which could be mistaken for excess heating or excess cooling.

We intentionally formed hot and cold spots by placing a resistor or Peltier element on the surface of the sample vessel that was later pressurized with hydrogen or helium. We observed both upward and downward temperature baseline shifts that were independent of gas type. In addition we used a heat sleeve that is sometimes used for gas-loading experiments at elevated temperatures. The heat sleeve also produced temperature baseline shift that in our system configuration resulted in 3% of apparent excess power production.

We conclude by emphasizing the importance of testing the measurement system with inert gas (preferably helium due to its similar value of thermal conduction coefficient to that of hydrogen) to eliminate the measurement error induced by temperature gradients.

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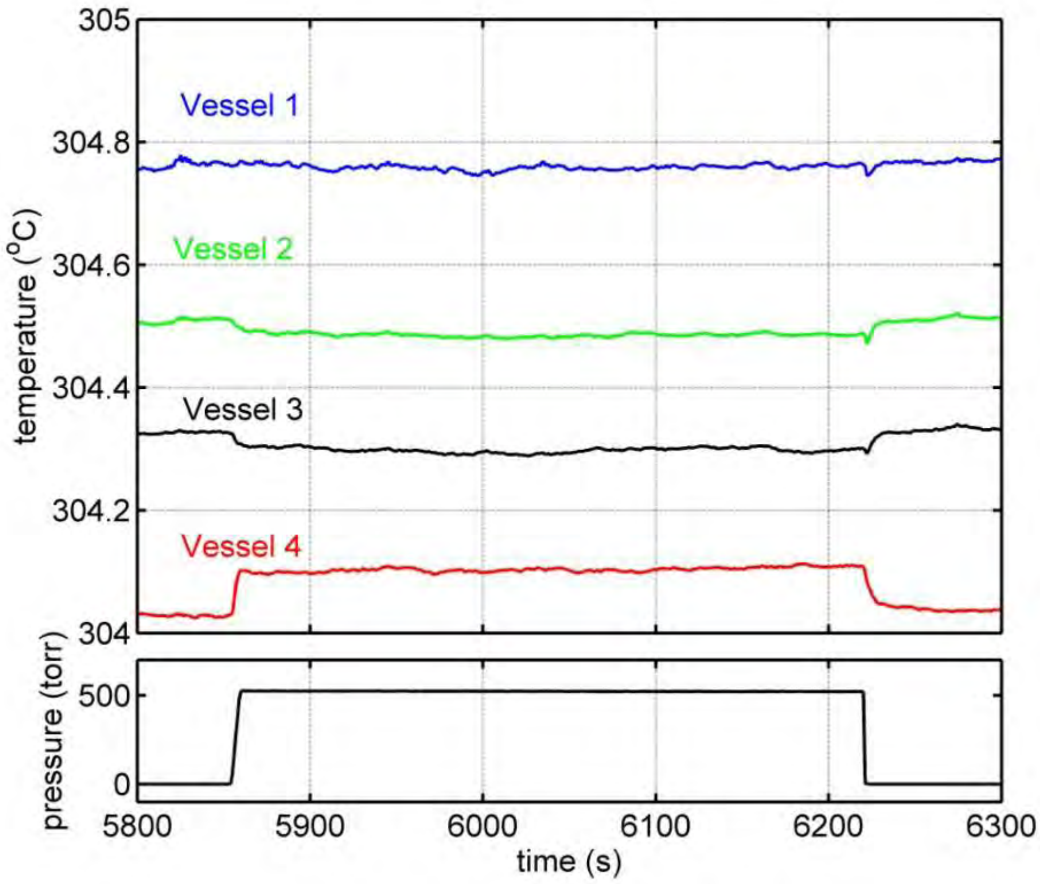
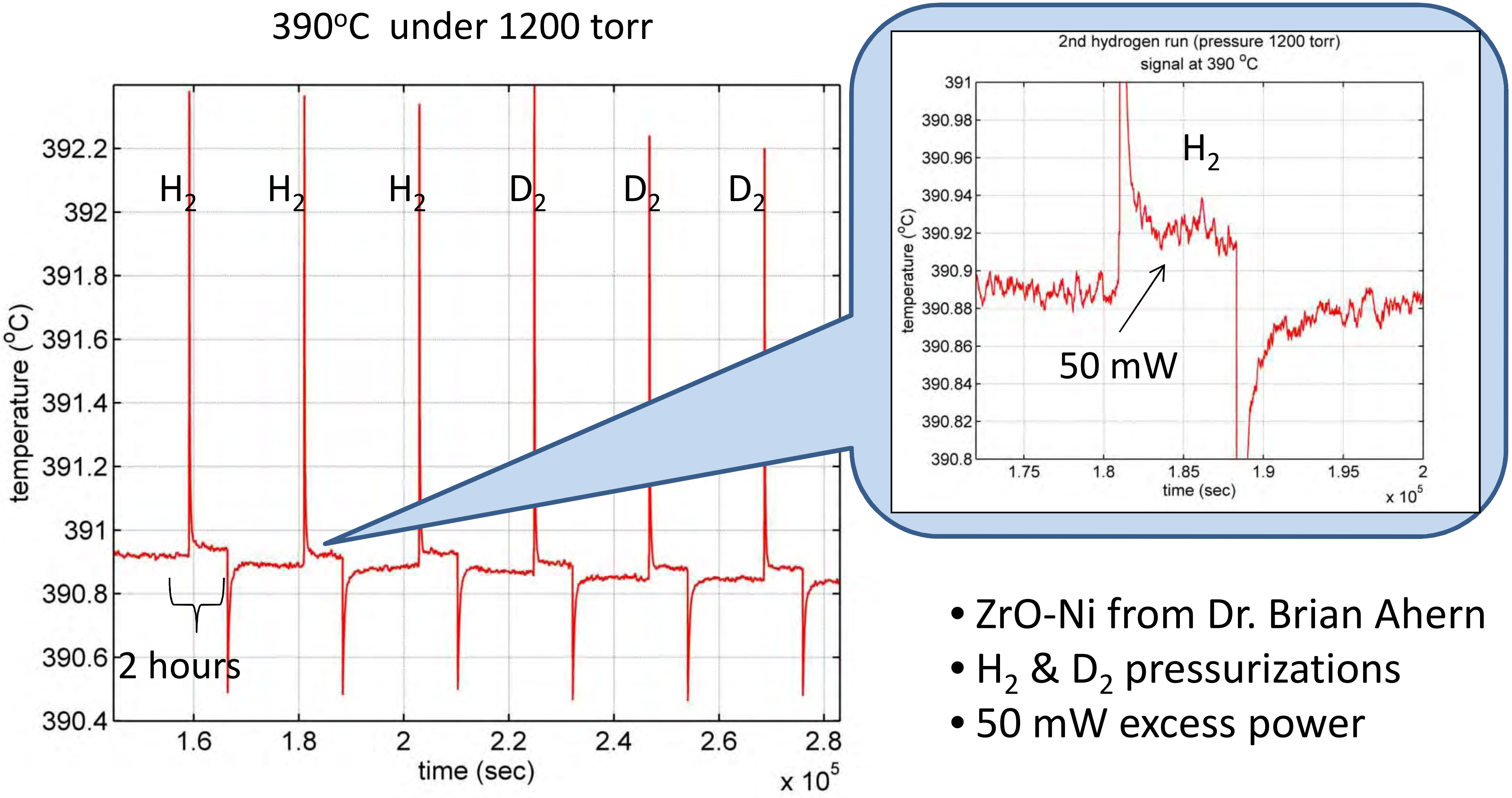
[1] Coolescence, LLC, Boulder, CO, USA
[2] University of Colorado – Boulder, CO, USA

Motivation (June 2011)



- Ni catalyst
- H_2 pressurization
- Cooling & heating observed

Motivation (February 2012)



- ZrO-Ni from Dr. Brian Ahern
- H_2 & D_2 pressurizations
- 50 mW excess power

Motivation

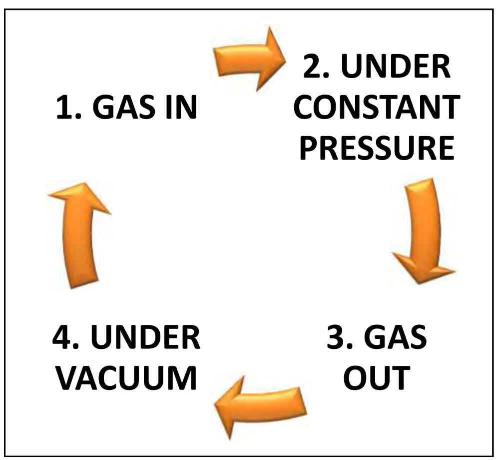
- Excess heat generation:
  - at high temperatures (>250°C)
  - same for H_2 and D_2
  - no decline over time (4 days!)
  - depends on vessel location

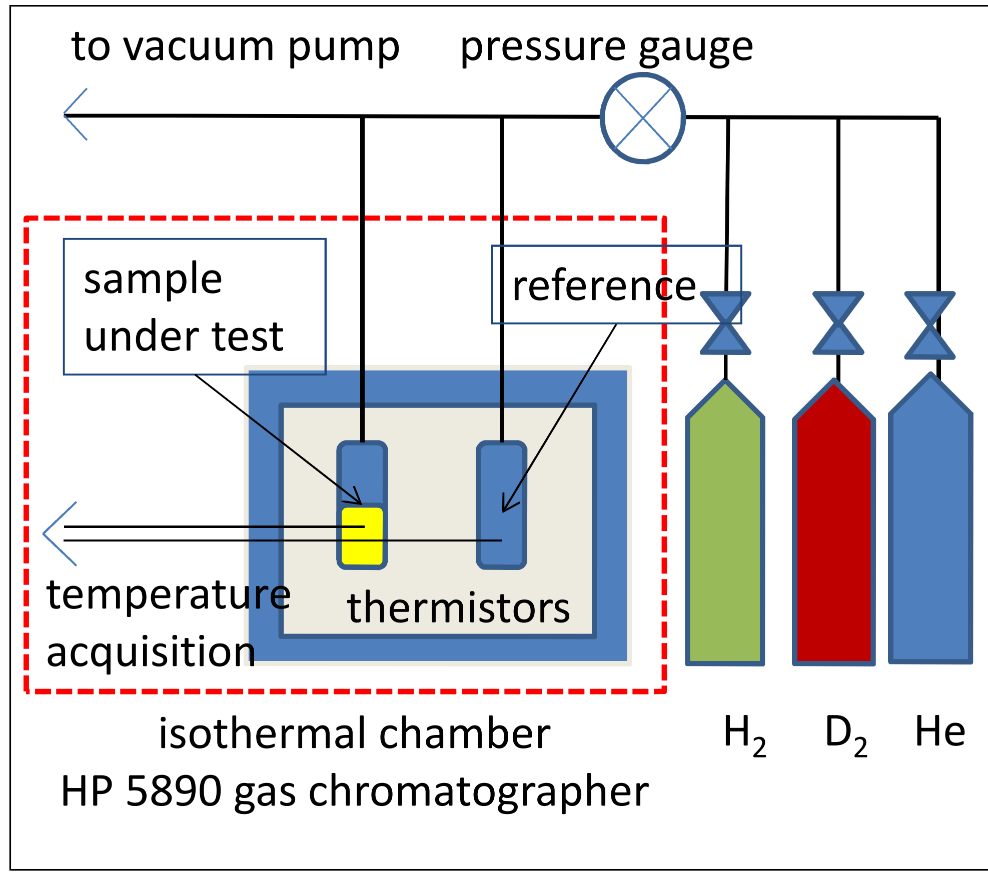
Questions to answer

- What causes baseline shift?
- Can we enhance effect intentionally?
  - excess heat
  - excess cold
- Independent of gas type?
- Applicable to other gas-loading experiments?

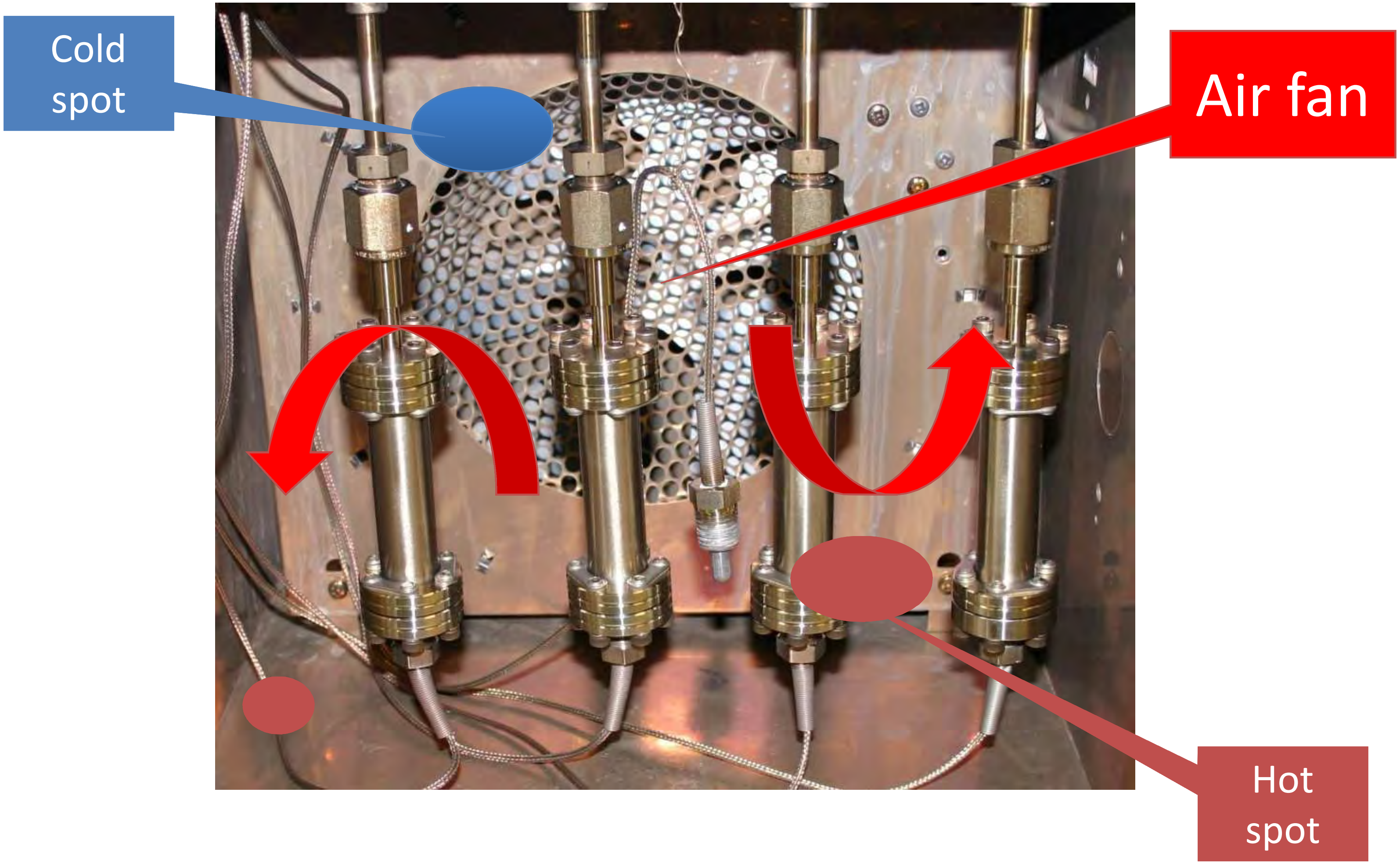
Gas-loading system

- Precision of temperature control 10 mK
- 6g of material
- Pressure up to 1200 torr
- Chamber temperature 40 - 390˚C





Thermal gradient in chamber

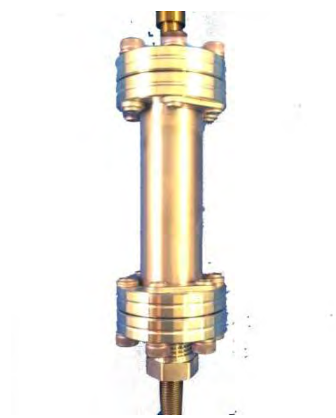


Hypothesis

1. Gas in

2. Increase thermal conductivity

3. Couple thermistor to local hot or cold spot



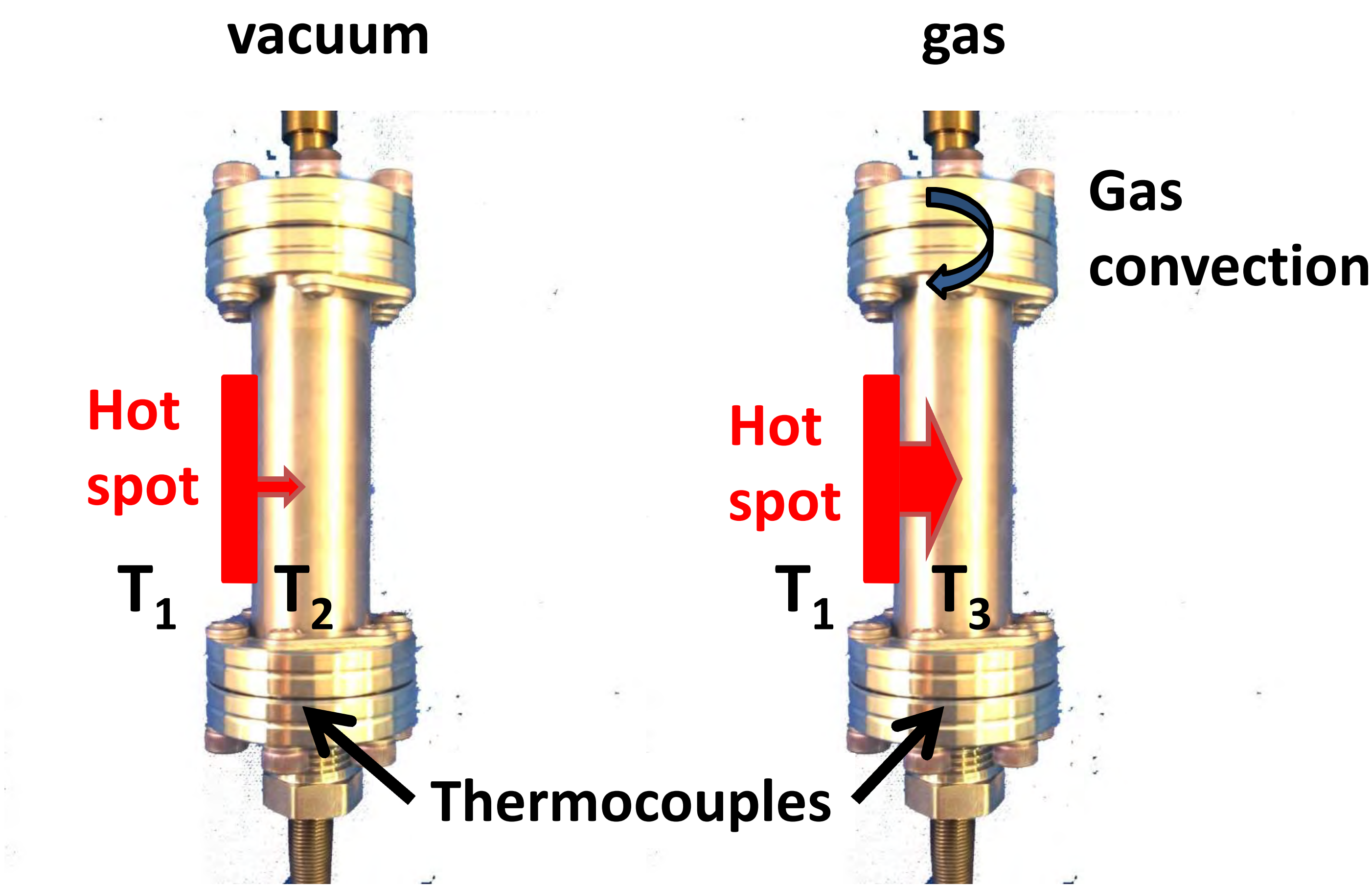
4. Measured baseline shifts

5. Gas out

6. Decrease thermal conductivity

7. Measured baseline shifts back

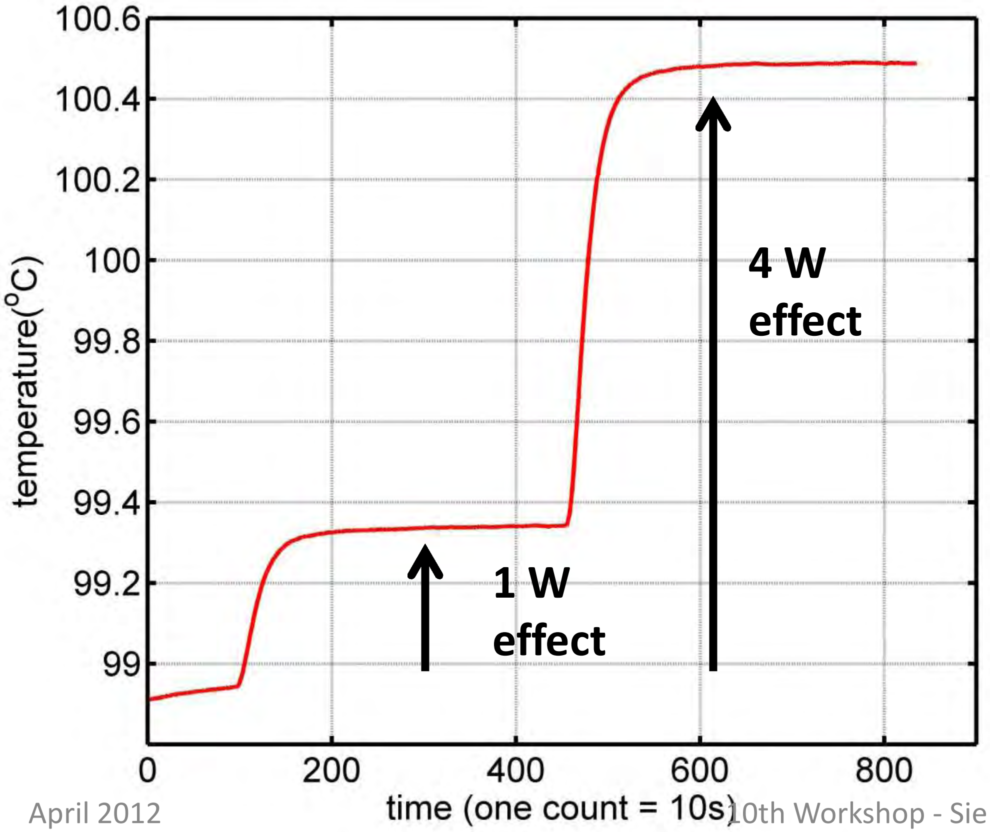
Hot spot coupling

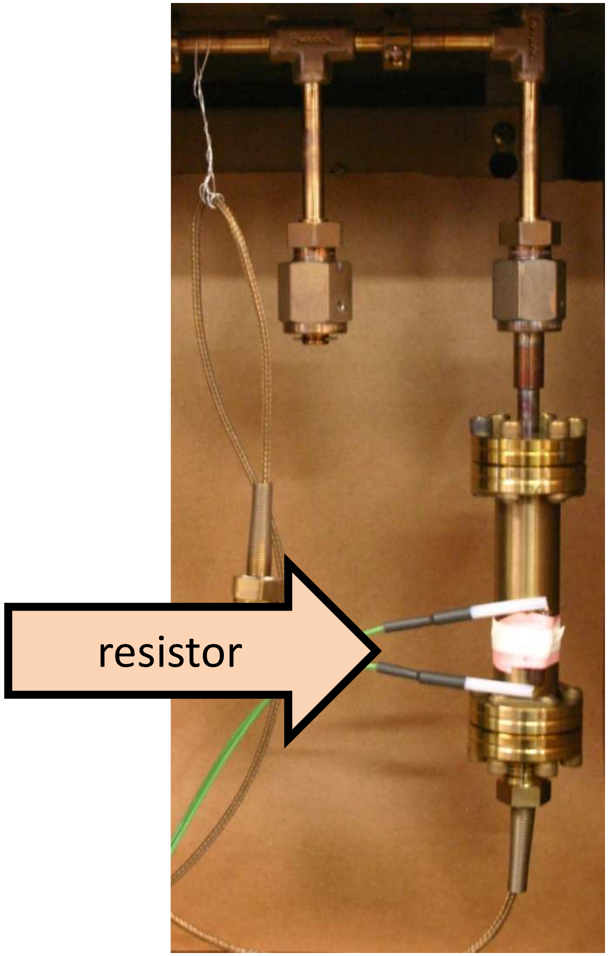


$T_2 < T_3$

Forming hot spot intentionally

- Hot spot: 150 Ohm resistor:
  - 1 watt
  - 4 watt





Effect of hot spot in the presence of gas

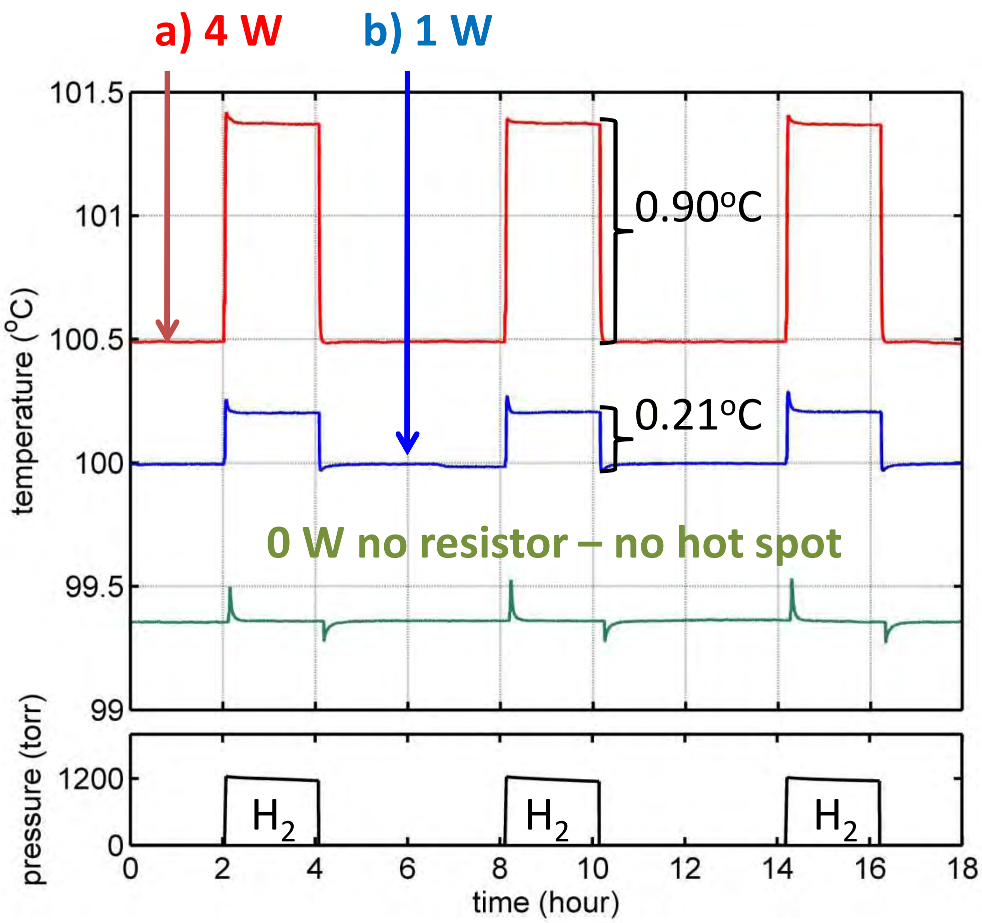
- For our configuration:

  0.90ºC -> 0.8 W

  apparent excess heat 20%

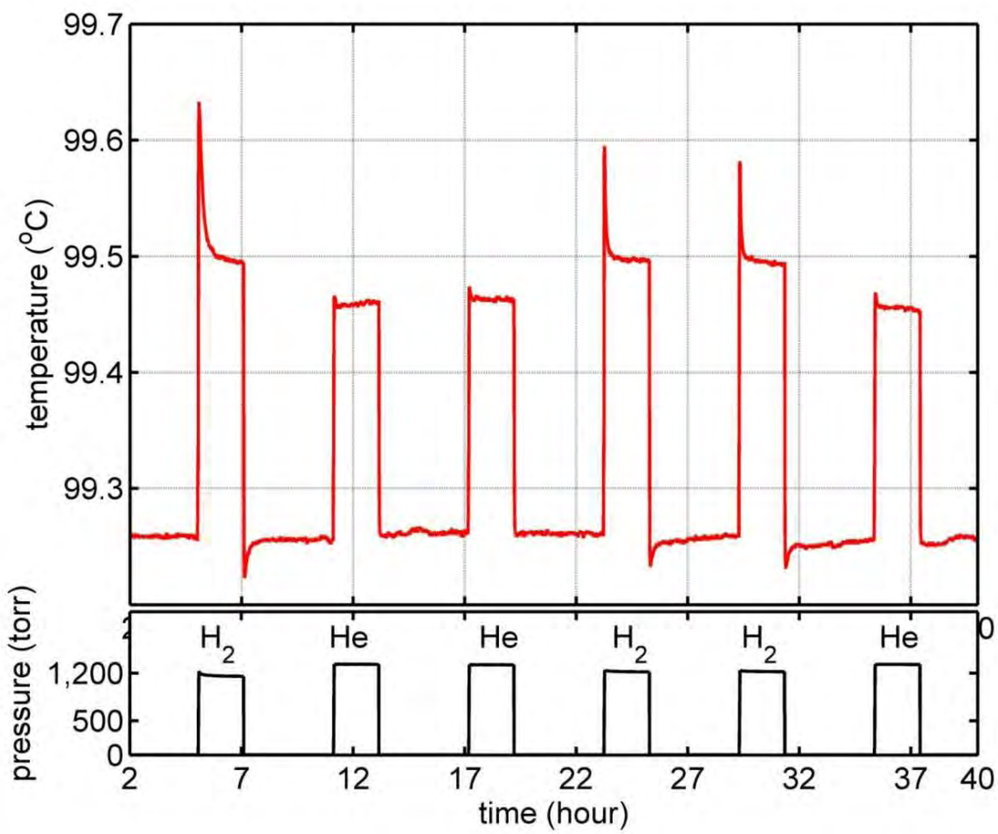
  0.21ºC -> 0.17 W

  apparent excess heat 17%

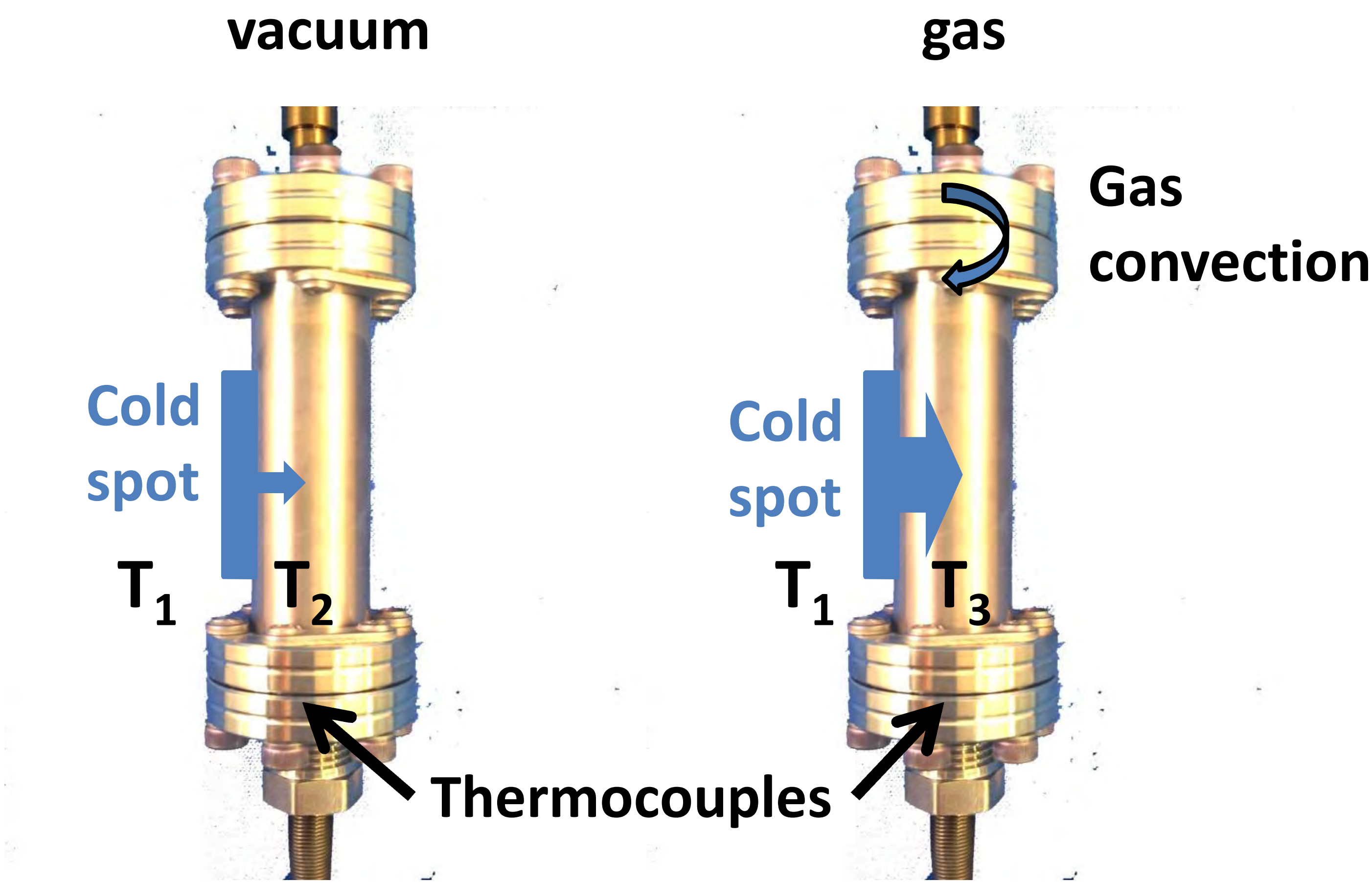


Independent of gas type

- Chamber at 100°C
- H_2, He 1200 torr
- Hot spot 1 watt
- H_2 and He runs show same exothermic shift of the baseline
- H_2 runs show spikes: loading/unloading in Ni

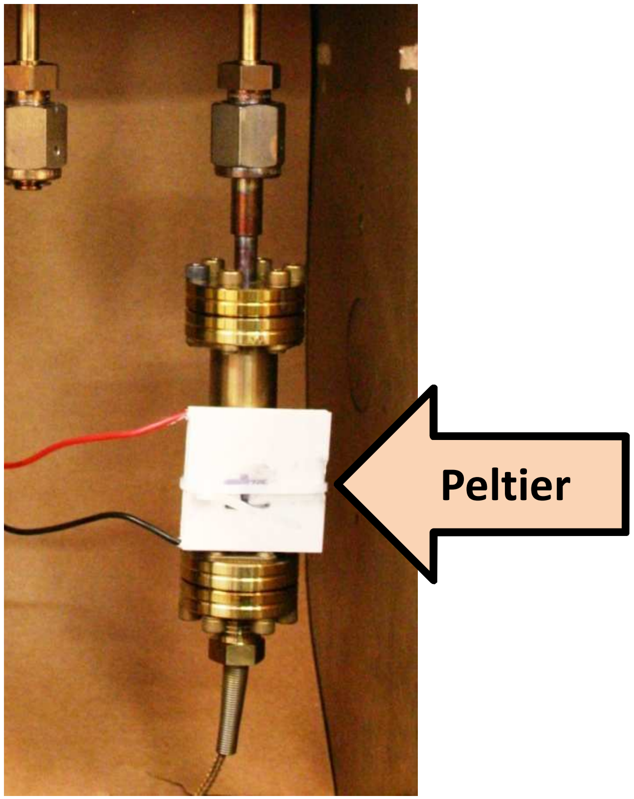


Cold spot coupling

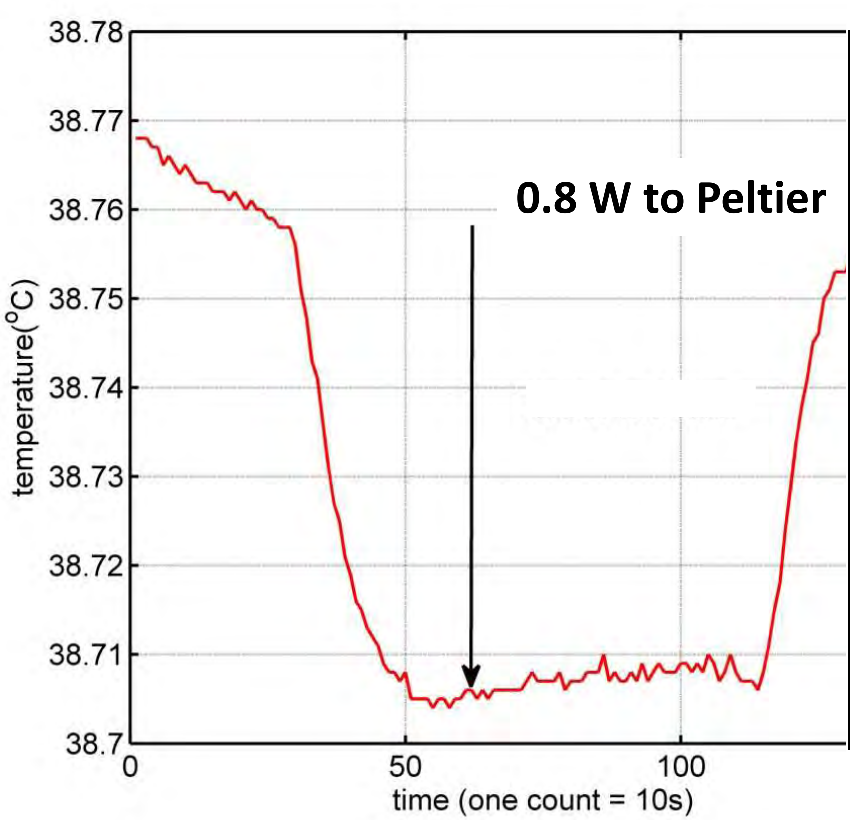


$T_2 > T_3$

Forming cold spot intentionally



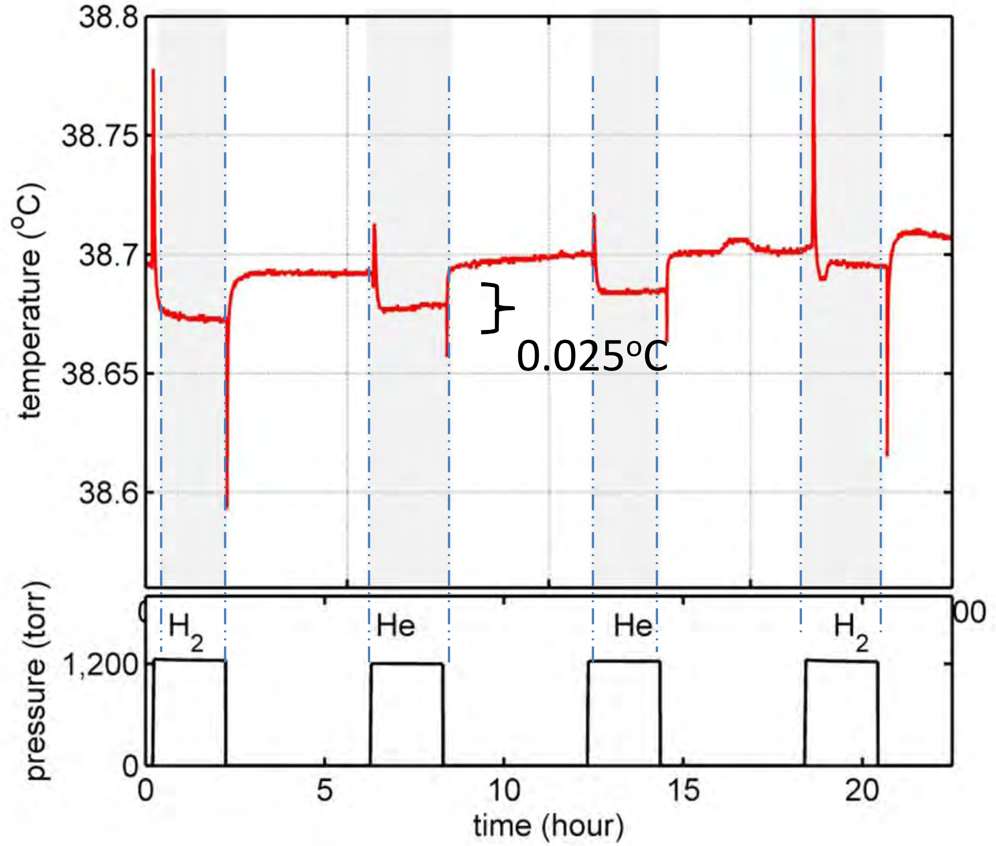
- Peltier element - cooling



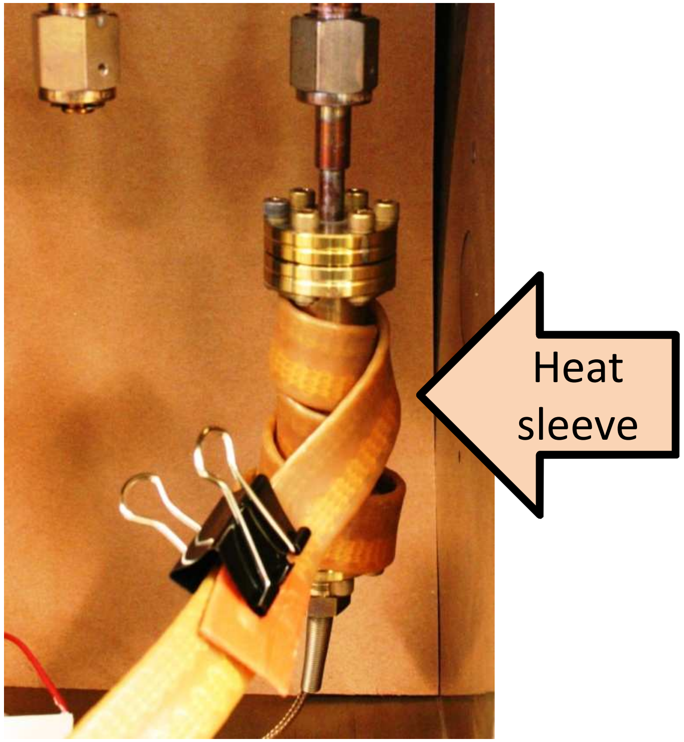
Forming cold spot intentionally

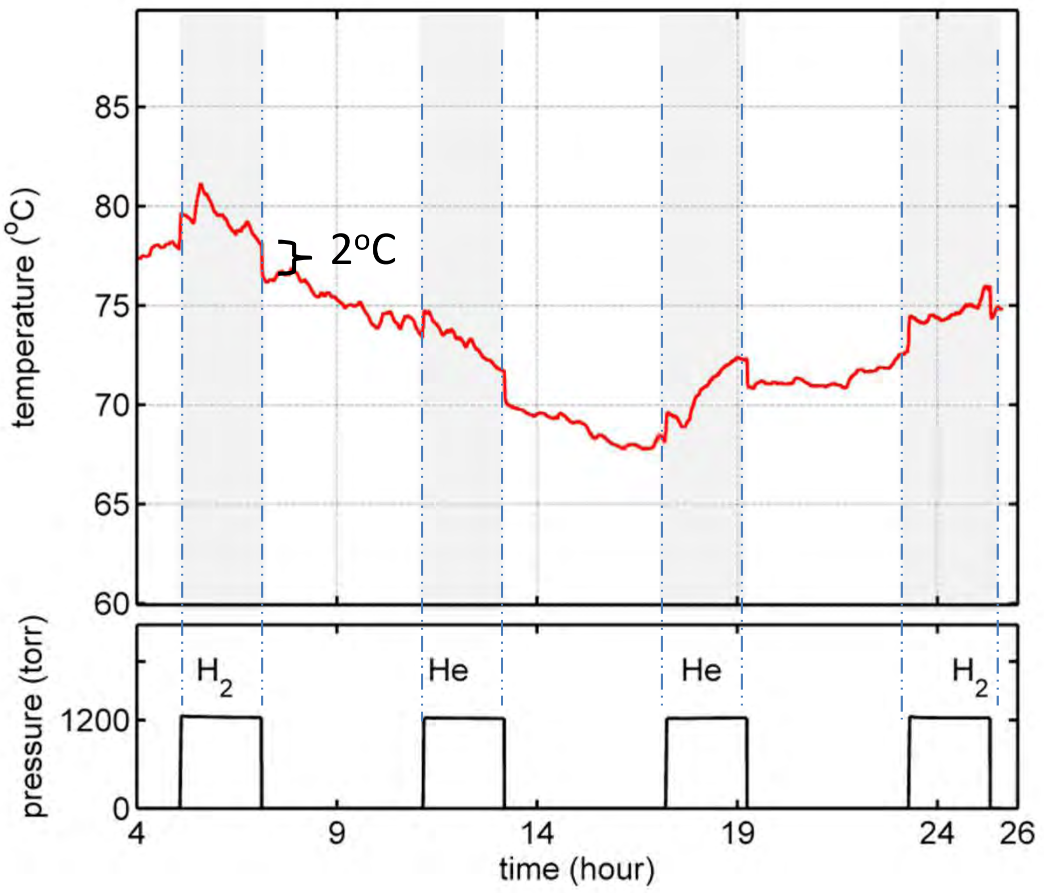
- For our configuration:
  0.025°C -> 0.013 W

  apparent excess cooling 1.6%



Creating hot spot by heat sleeve





For our configuration:

2°C -> 1.2 W

Apparent excess heat 3%

Summary

- Temperature gradient causes measurement artifact
- Conditions were recreated by
  - Resistor – hot spot – baseline shifts upwards
  - Peltier elem. – cold spot – baseline shifts downwards

- Heat sleeve: apparent excess heat 3%

Conclusion

Inert gas must be used to rule out temperature measurements artifacts