

High-Resolution Calorimetry for Probing Reactions of Nanopowders with D₂

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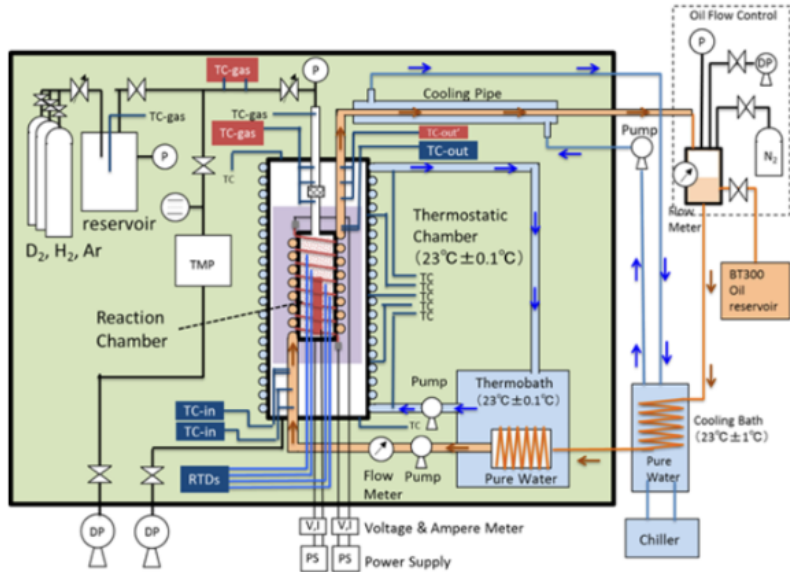
Department of Mechanical Engineering

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Agenda

1. Description of a new microwatt resolution calorimeter
2. Results from two sample runs involving D_2 and a nanopowder

Past reports of anomalous heat outputs



D₂ on PNZ4:

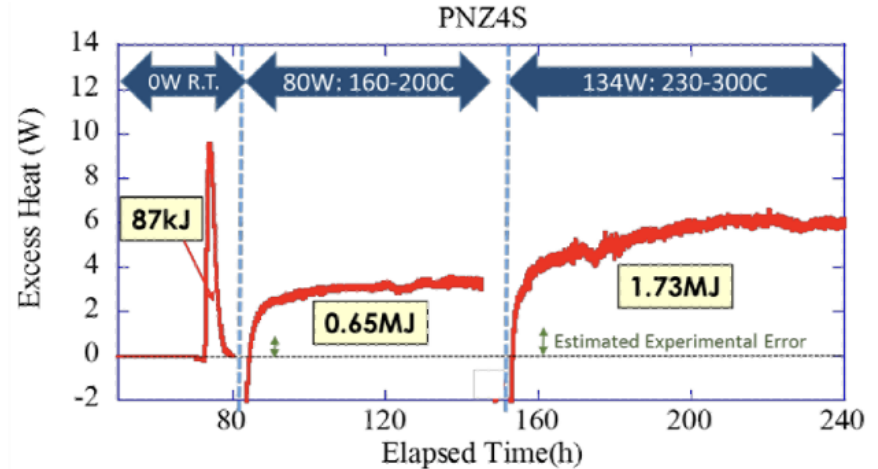


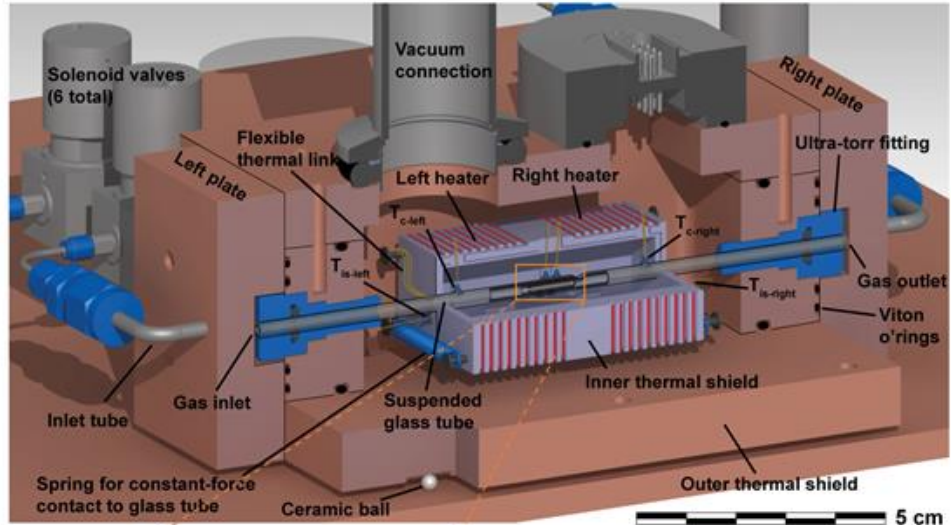
Table 1. Sample composition.

Sample name	Tested at	Weight (g)	Molar fraction (%)				
			Cu	Pd	Ni	Zr	O
PNZ4s	Tohoku University	109.4	---	3.6	25.2	53.4	17.8
PNZ4	Kobe University	109.4	---	3.6	25.2	53.4	17.8

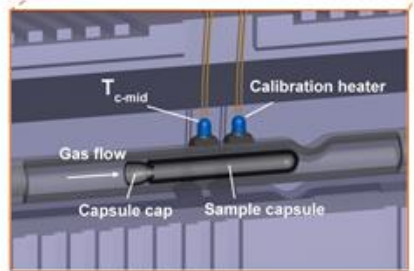
14.9 eV per D atom
Assuming all introduced
Deuterium results in energy
generation

Microwatt resolution calorimetric reactor

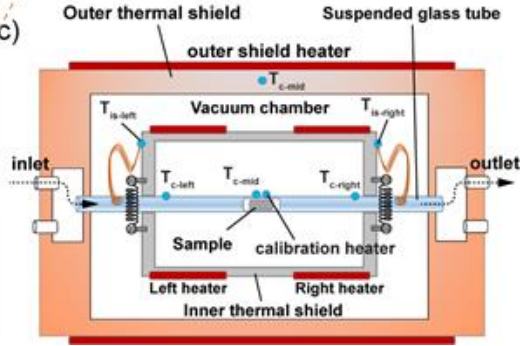
a)



b)



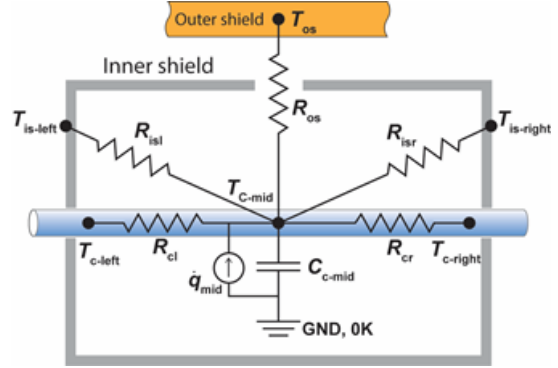
c)



Specifications

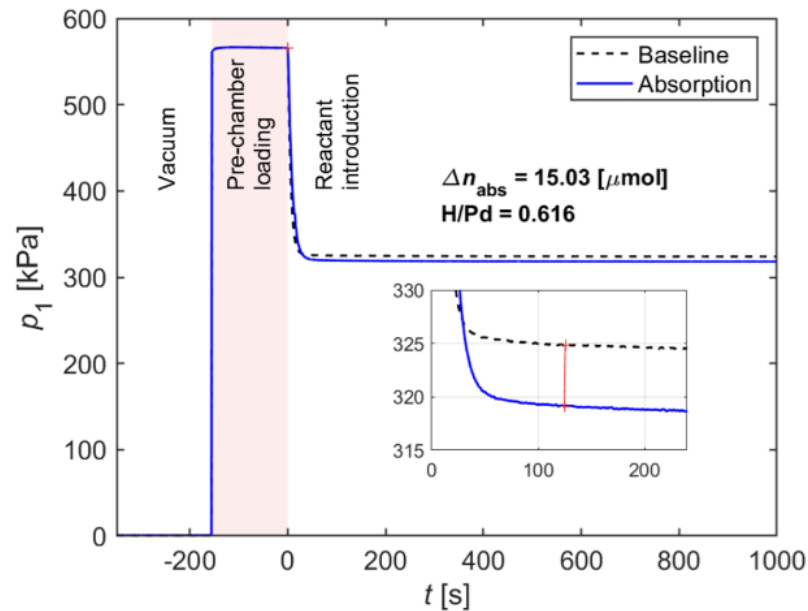
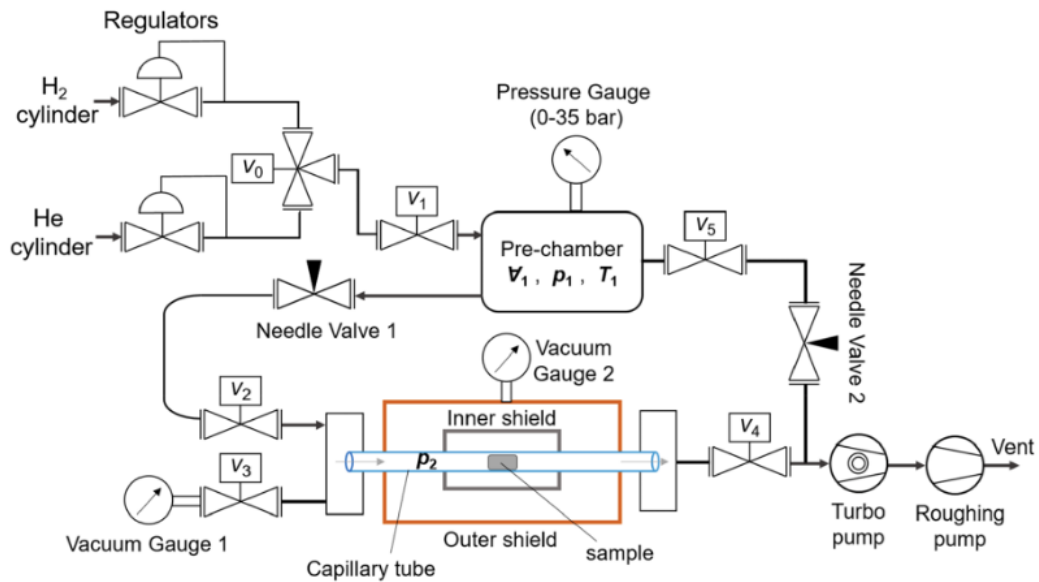
- Temperature range: R.T. – 300 °C
- Pressure range: 10 mbar - 30 bar
- Heat flow resolution: < 3 μW/√Hz
- long-term stability < 4 μW/hour
- Mass balance resolution: 0.1 μmol

Lumped-thermal capacity model:



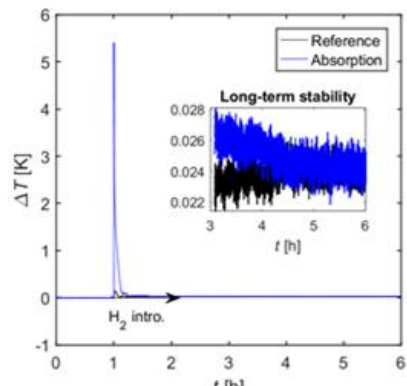
$$\dot{q}_{mid} = \sum_i (T_{c-mid} - T_i) / R_i + C_{c-mid} \frac{dT_{c-mid}}{dt}$$

Gas handling system

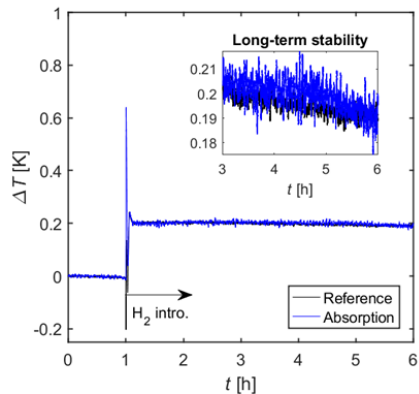


Demonstration of calorimetric capabilities: hydrogenation of Pd nanoparticles

Absorption and baseline

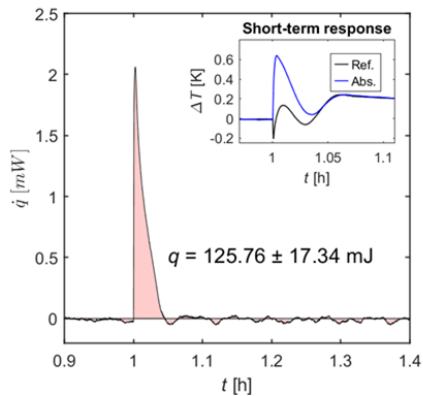
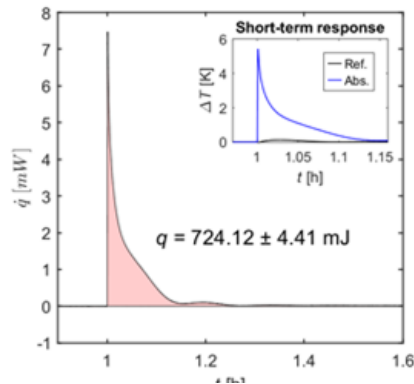


$P_{H_2} = 7$ bar abs
 $T_{RXN} = 30$ °C
 $m_{Pd} = 5.10$ mg

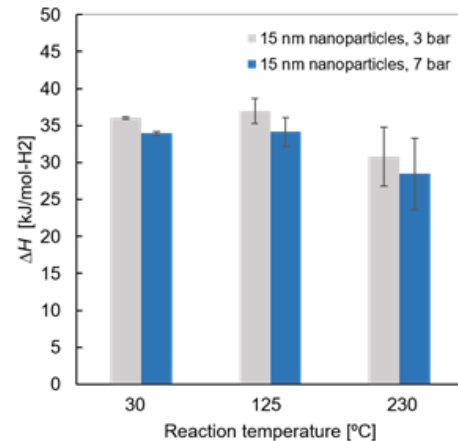


$P_{H_2} = 7$ bar abs
 $T_{RXN} = 230$ °C
 $m_{Pd} = 3.77$ mg

Heat of reaction



Enthalpy of hydrogenation



A. Reihani et al., ACS Sensors (2020)

Preliminary work exploring heat outputs
from metal nanopowders in D₂ environments

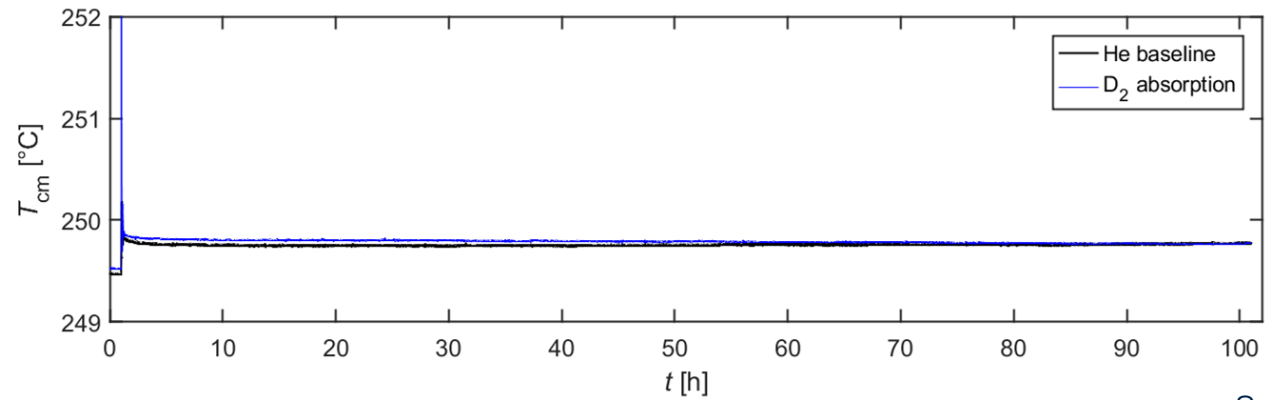
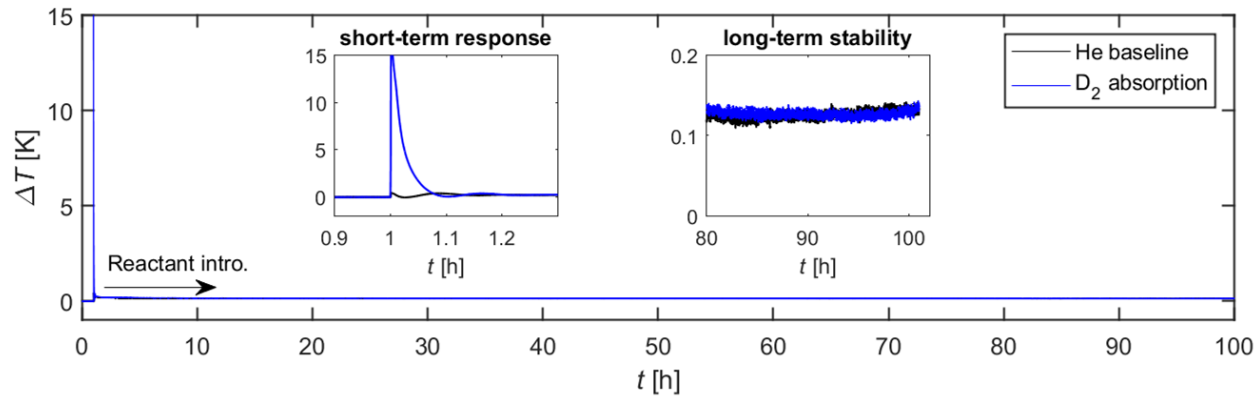
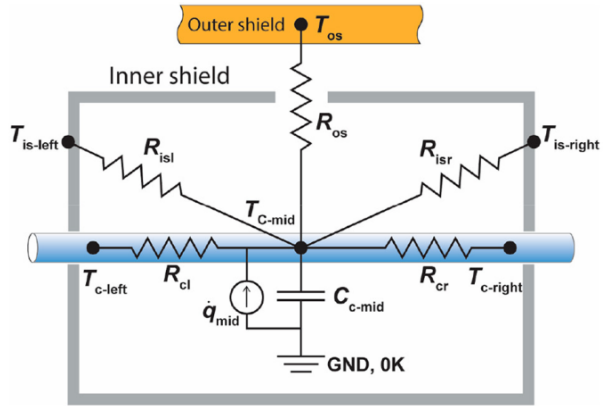
First run: Measurement from a nanopowder

Temperature difference signal is obtained using the thermal network model:

$\Delta T = T_{c\text{-mid}} - T_{c\text{-mid,expected}}$, where

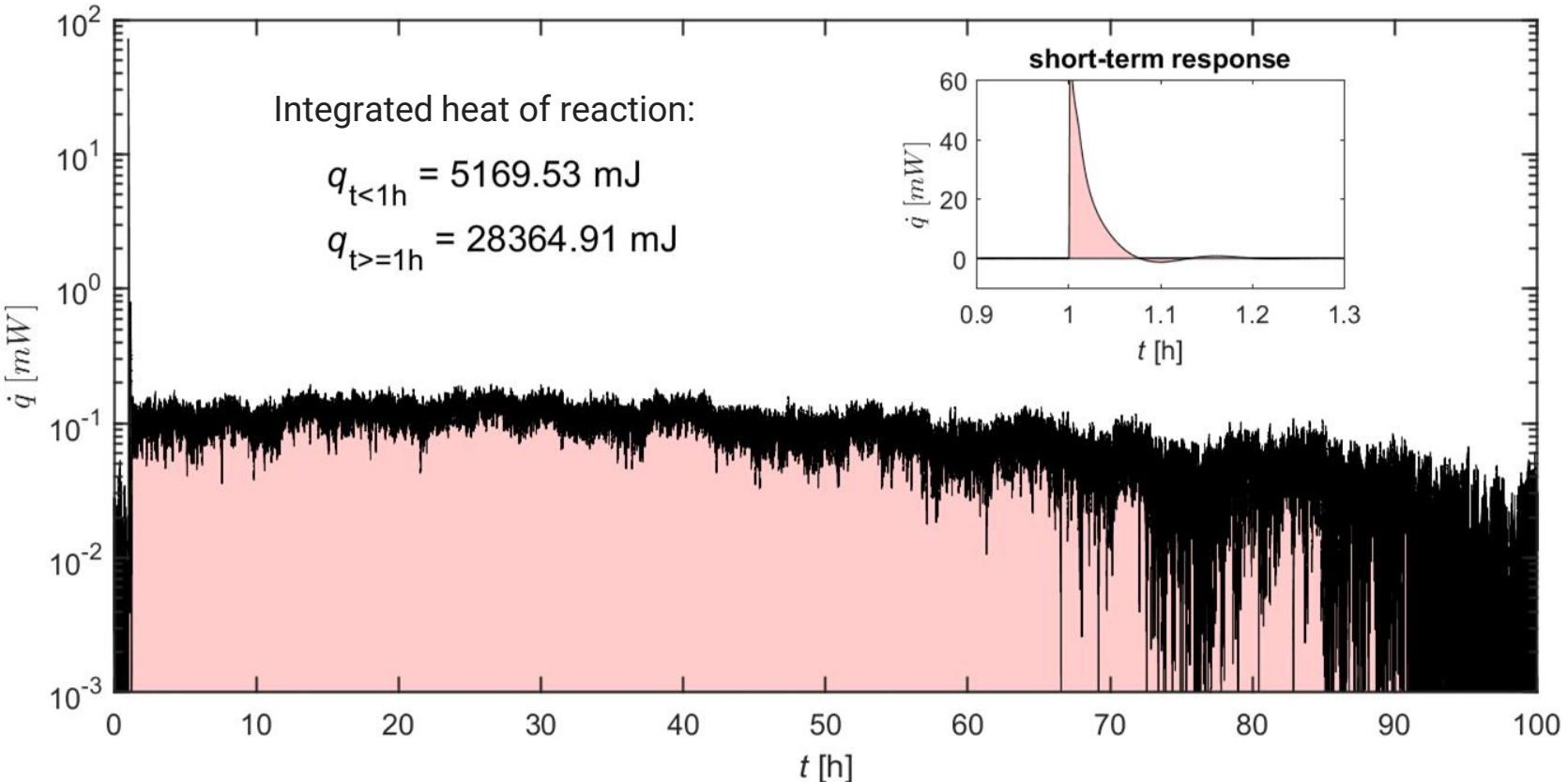
$$T_{c\text{-mid,expected}} = R_{\text{tot}} \sum_i T_i / R_i$$

$$R_{\text{tot}} = \left(\sum_i R_i^{-1} \right)^{-1}$$



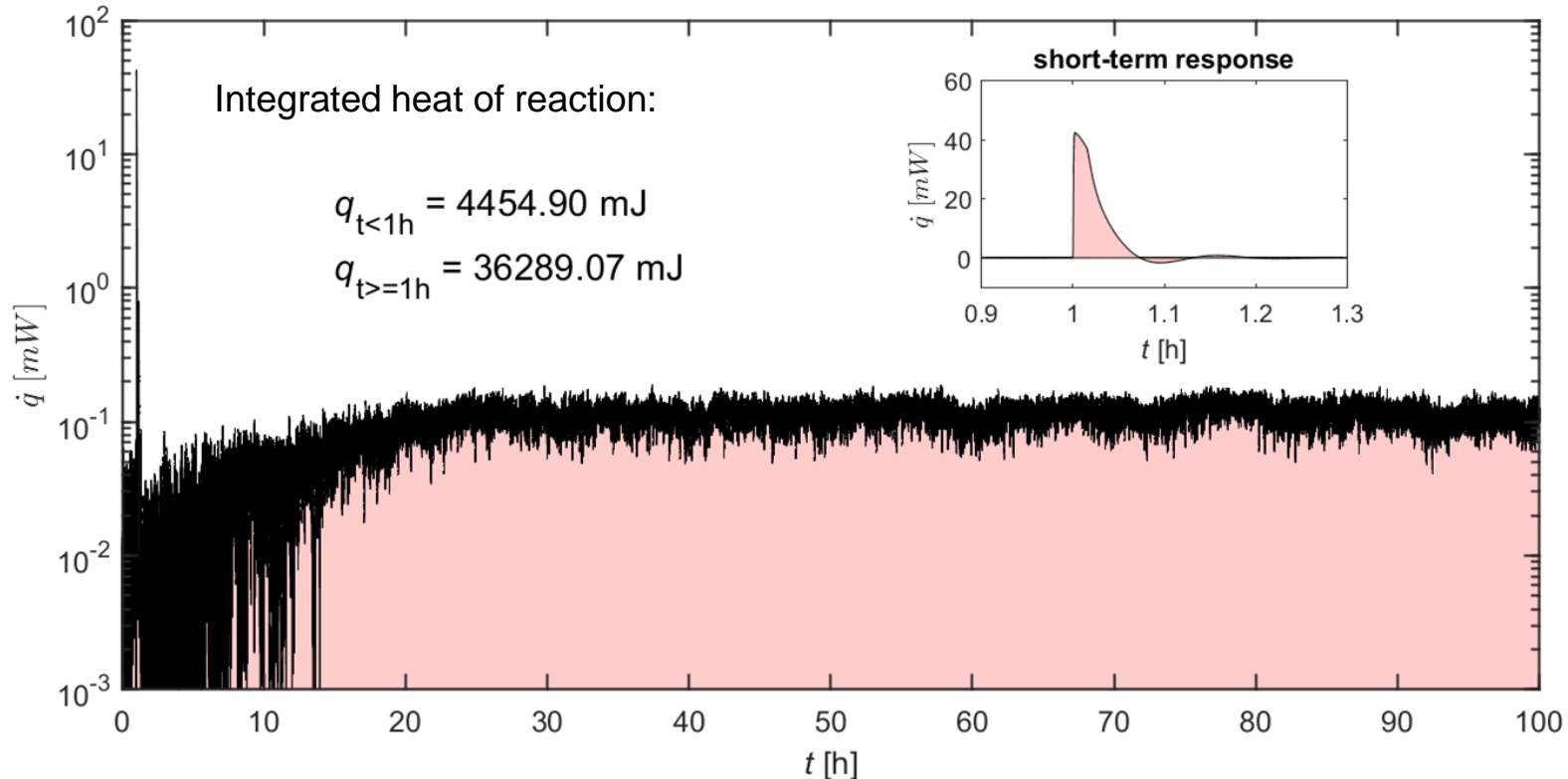
First run: Heat release rate (nanopowder + D₂)

Negative values are not shown in log plot



Second run: Heat release rate (nanopowder + D₂)

Negative values are not shown in log plot



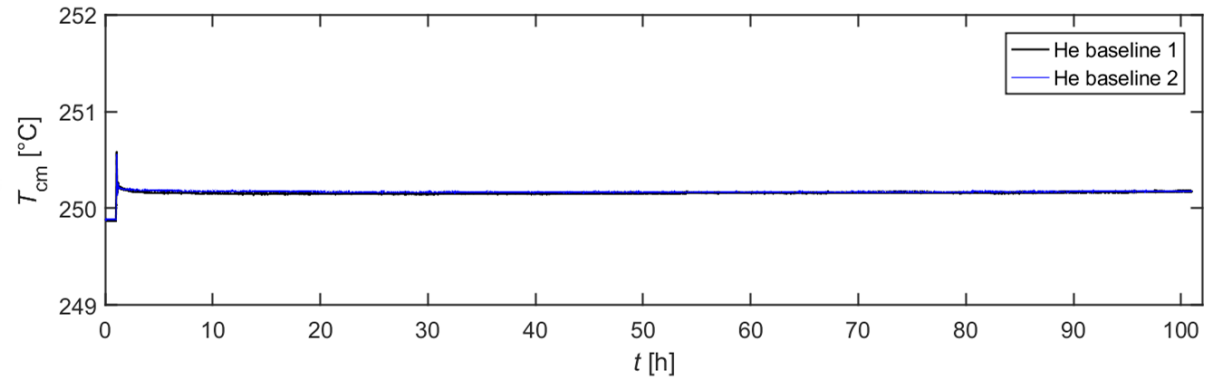
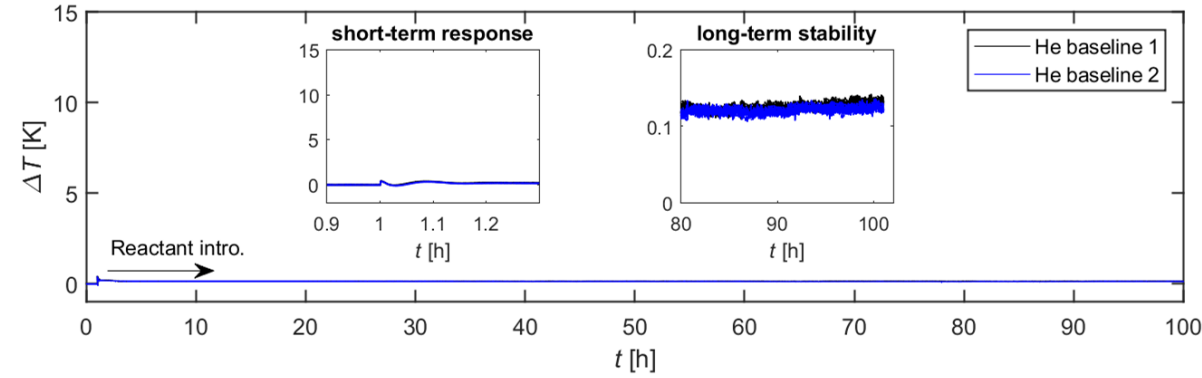
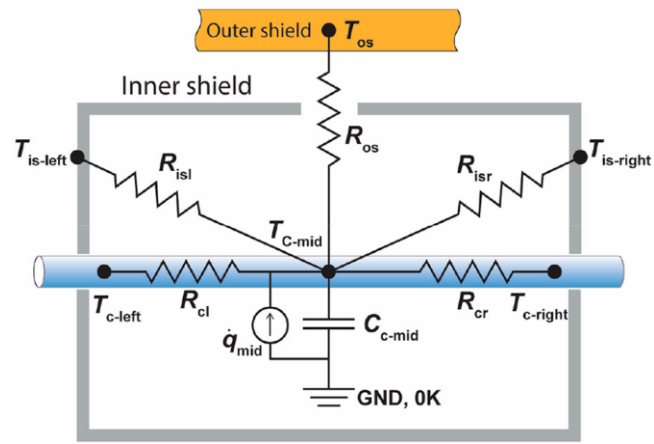
Instrument stability across baseline measurements with He

Temperature difference signal is obtained using the thermal network model:

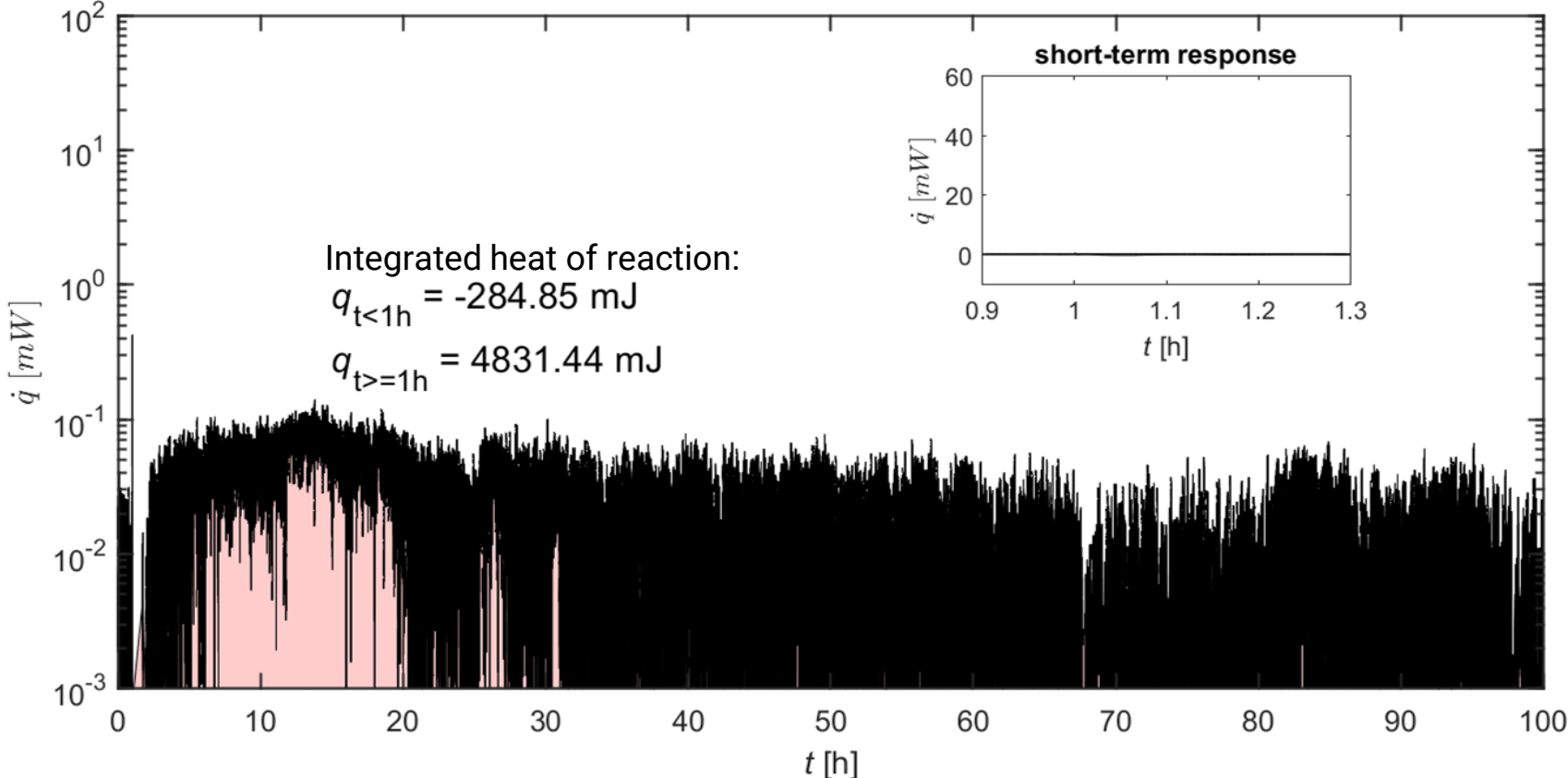
$\Delta T = T_{c\text{-mid}} - T_{c\text{-mid,expected}}$, where

$$T_{c\text{-mid,expected}} = R_{\text{tot}} \sum_i T_i / R_i$$

$$R_{\text{tot}} = \left(\sum_i R_i^{-1} \right)^{-1}$$



Heat release rate in He based control experiments is small



Necessary next steps

- Repeat measurements with a blank sample for both D₂ and He to eliminate the possibility of reactions with adsorbed Oxygen or due to possible leaks into the calorimeter
- Characterize reaction products using mass spectrometry
- Explore the possibility of oxidation of metals as source of exothermic reactions