

# The high-pO<sub>2</sub> method to prevent U-loss during single-aliquot hematite (U-Th)/He measurement: Development, implementation, and automation

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University of Southern California

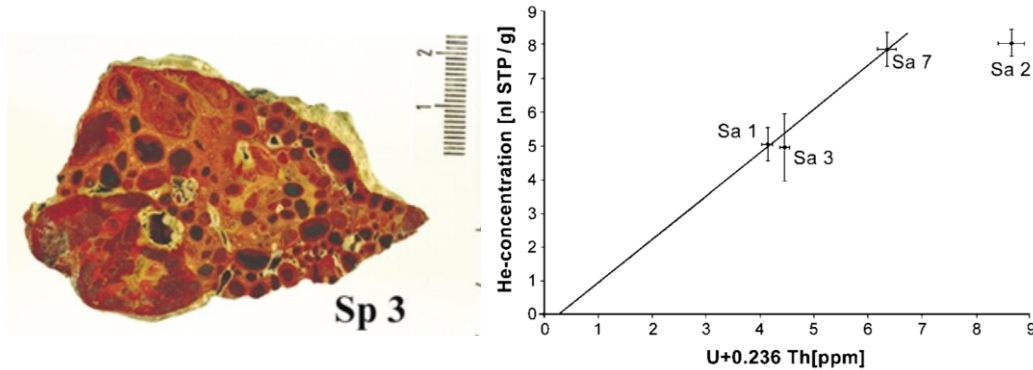
Jonathan Treffkorn  
Ken Farley  
California Institute of Technology

Thermo2021  
September 13, 2021

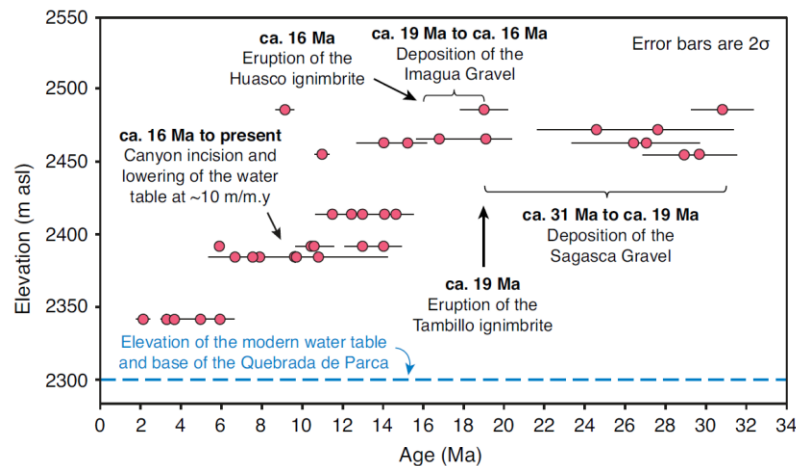
# Utility of hematite (U-Th)/He

## Geochronology

Pidgeon et al. (2004) – formation age of ferruginous nodules from lateritic duricrusts

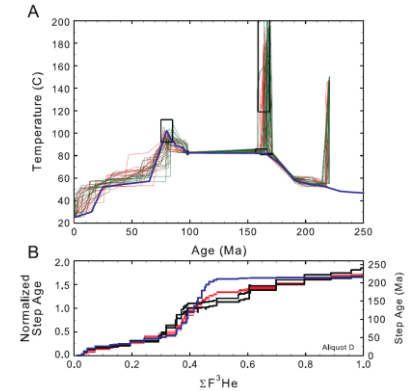


Cooper et al. (2016) – downward migration of water table resulting from incision

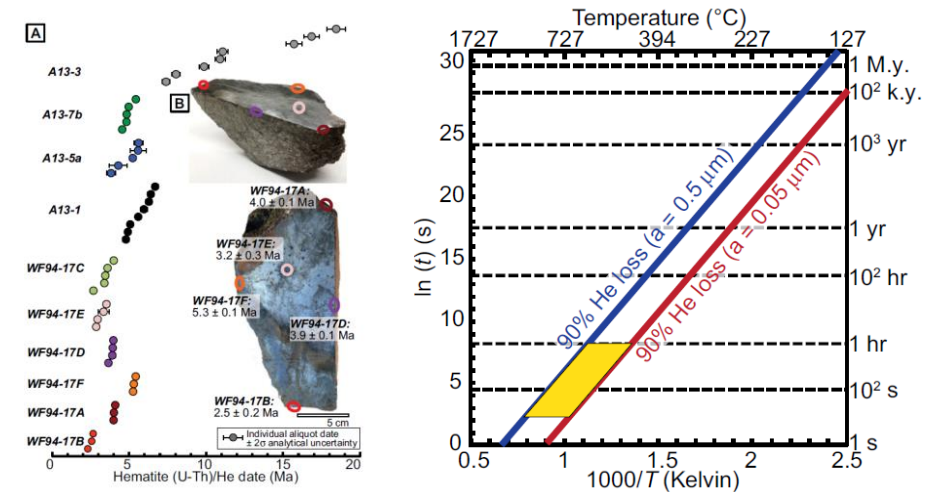


## Thermochronology

Farley and Flowers (2013) – temperature history modeling of polycrystalline aggregates

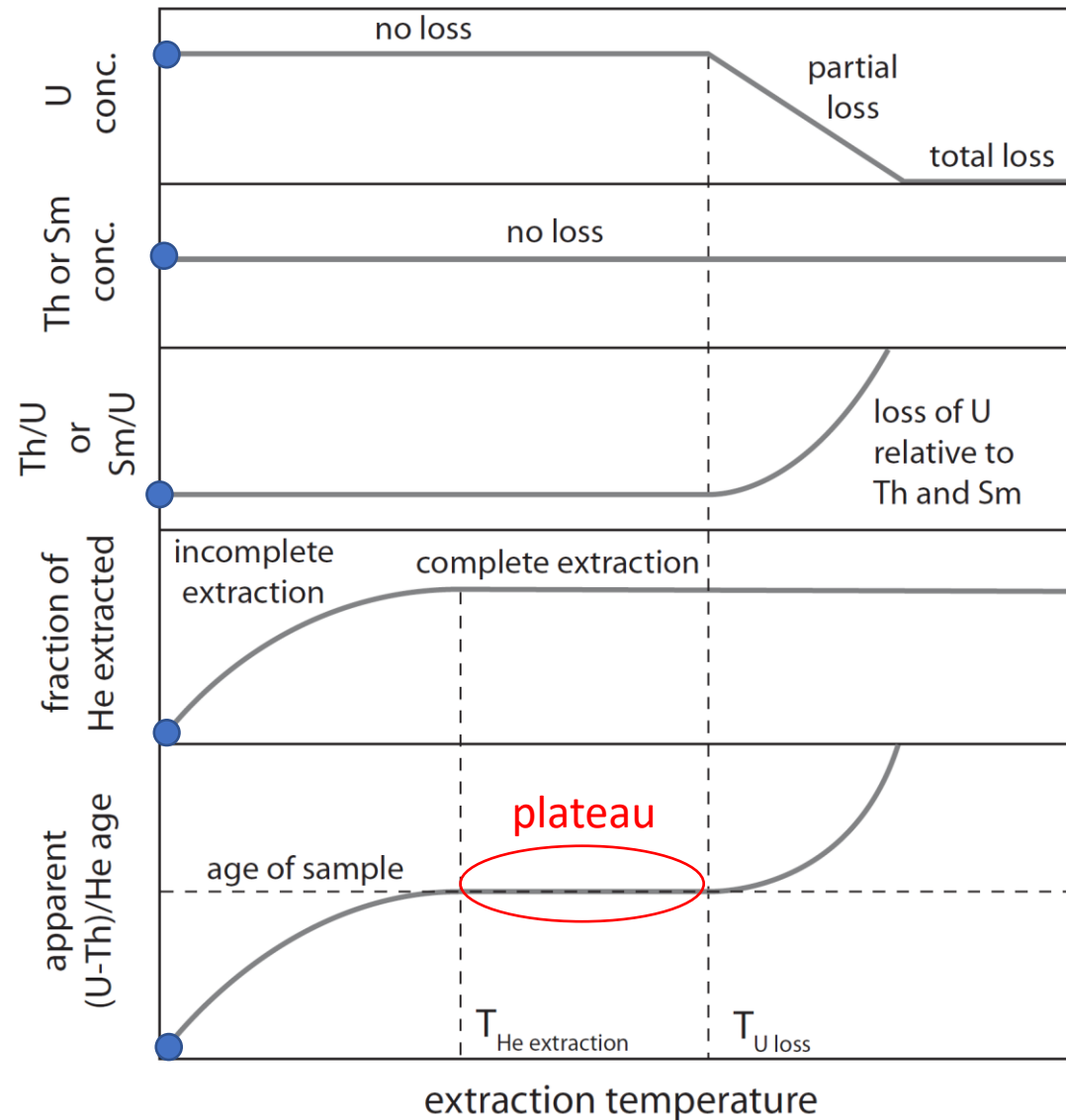


Ault et al. (2015) – resetting of hematite crystallites due to shear-heating

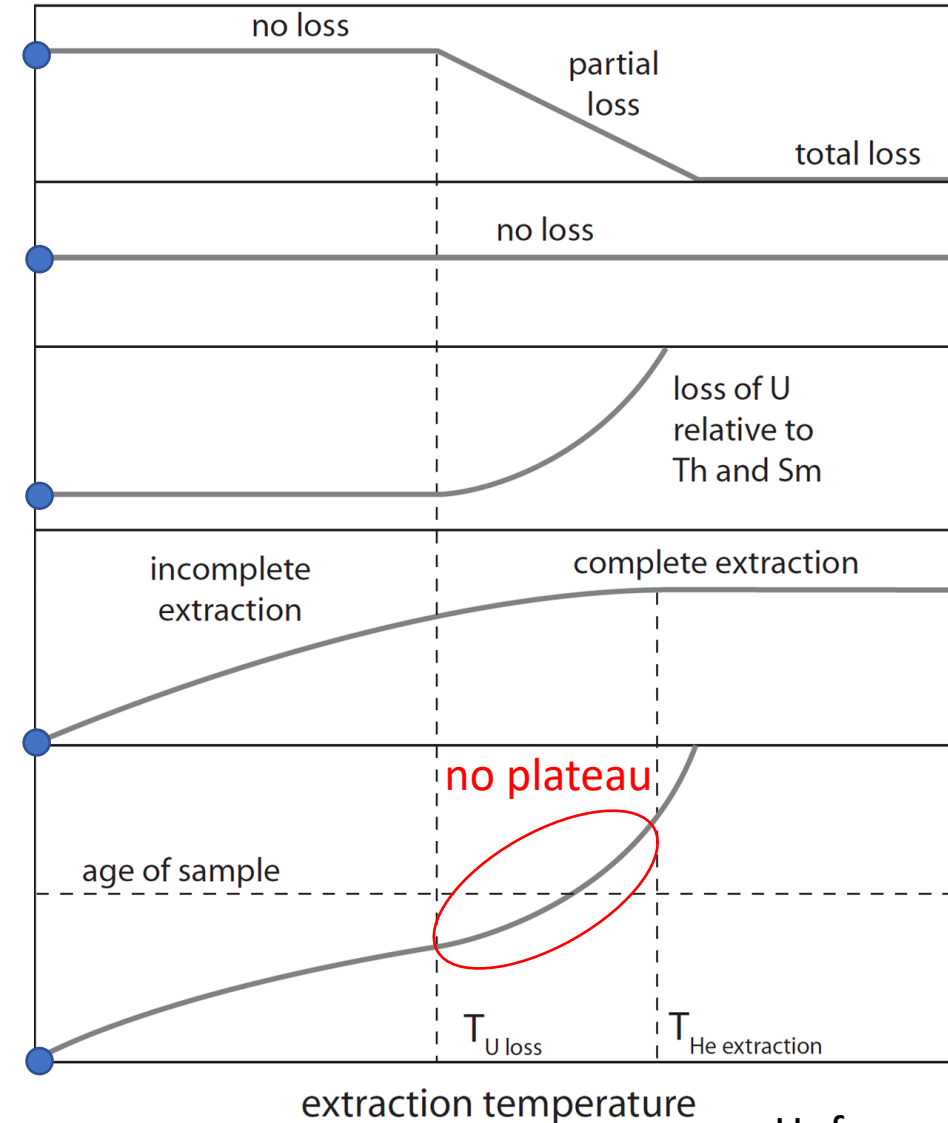


# Loss of U during laser-heating

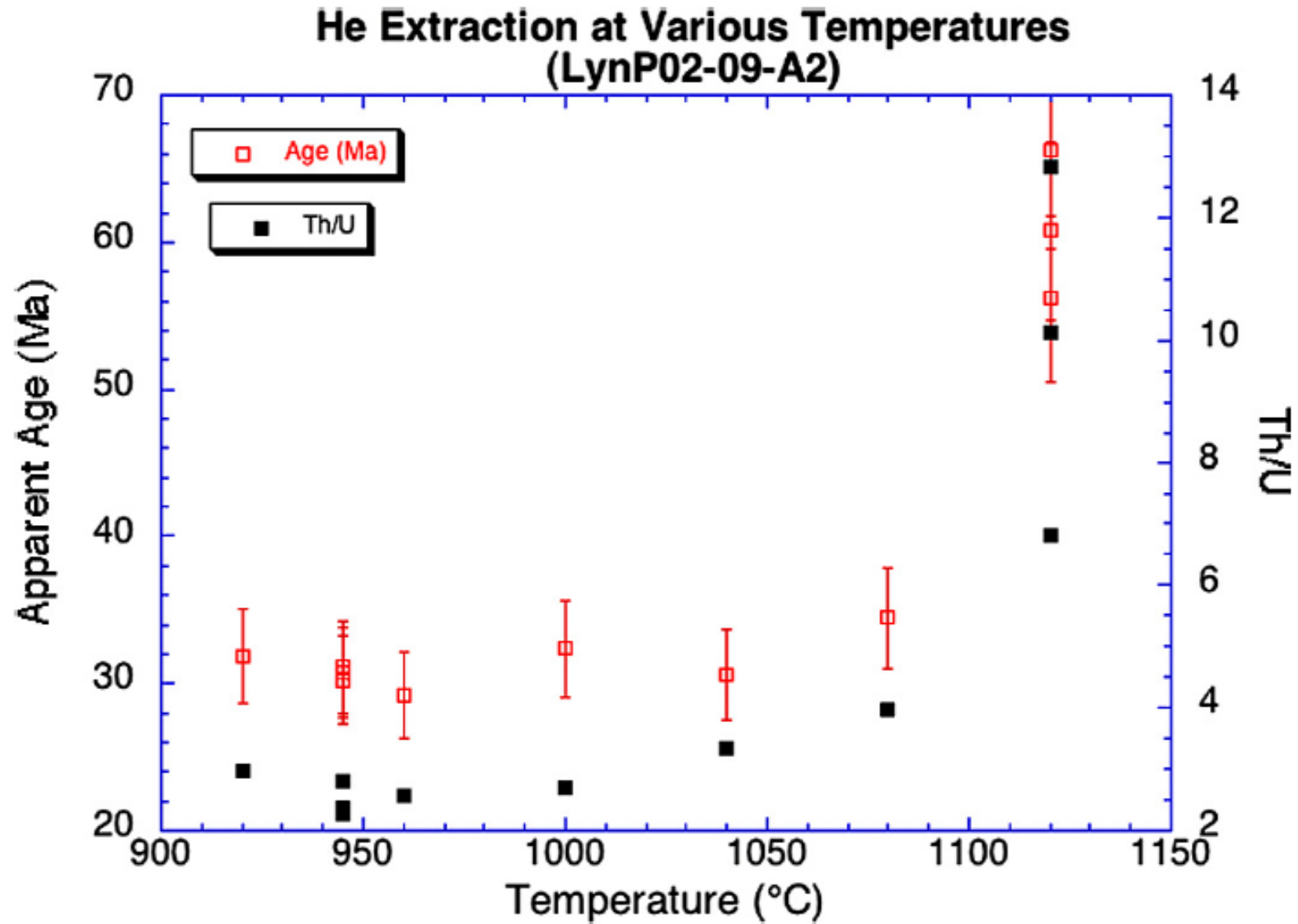
## Ideal case



## Problematic case

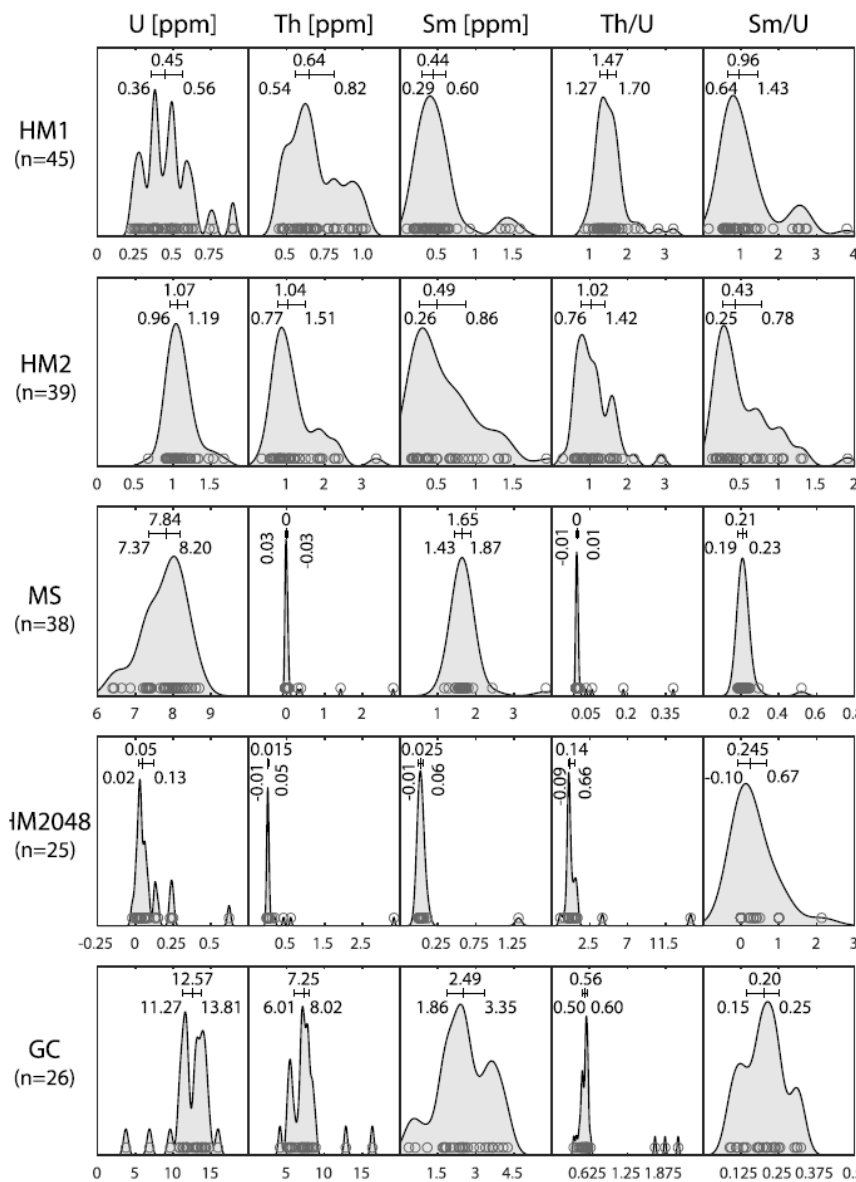


# Loss of U during laser-heating

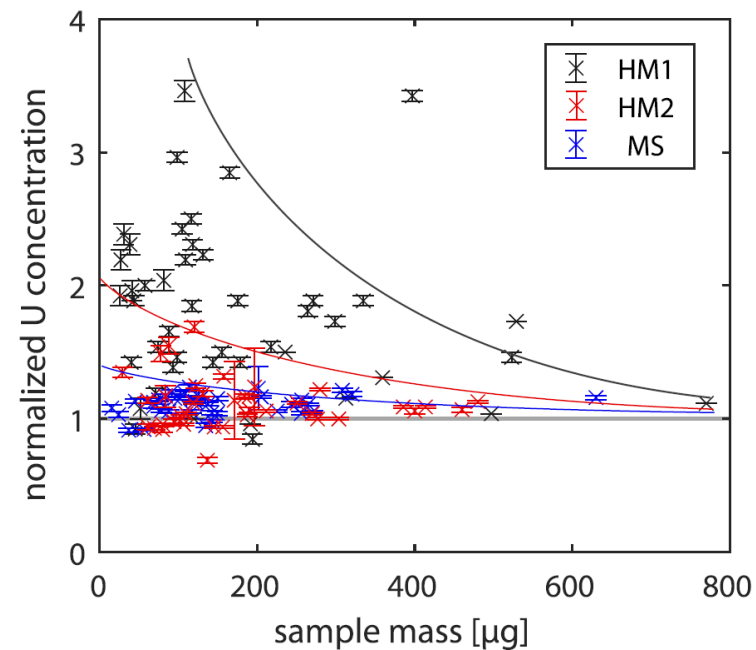
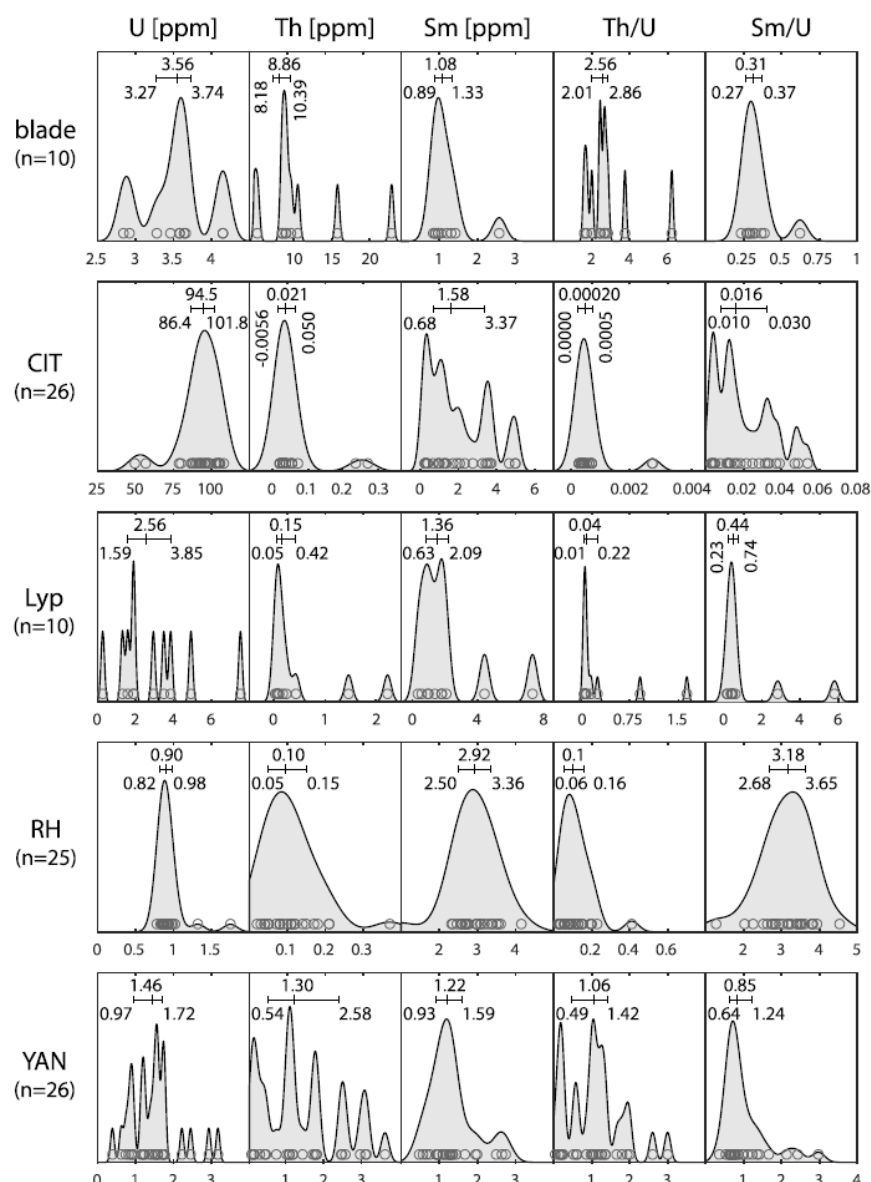


# Natural variability in hematite and goethite samples

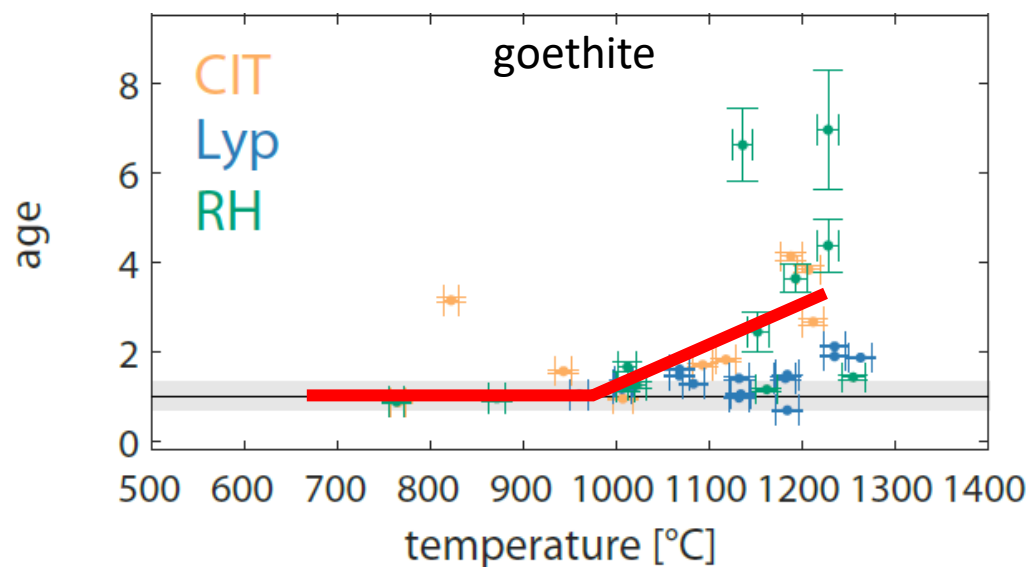
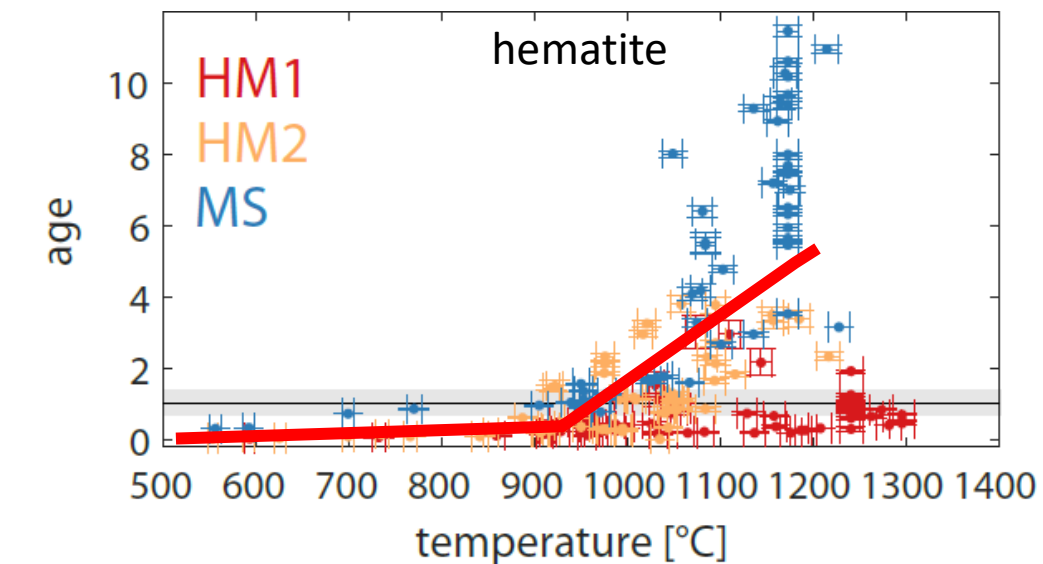
## Hematite



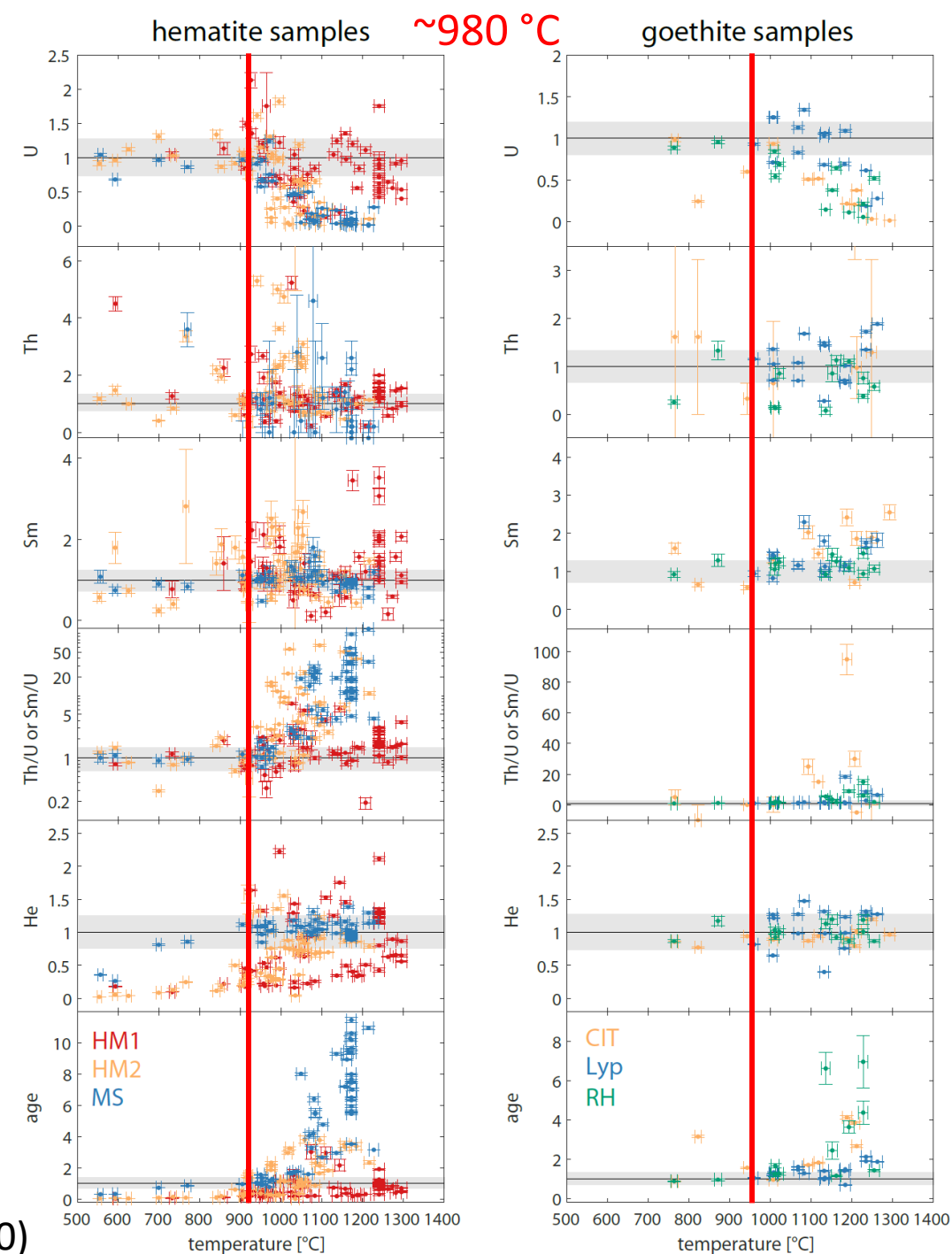
## Goethite



# Heating tests of multiple samples

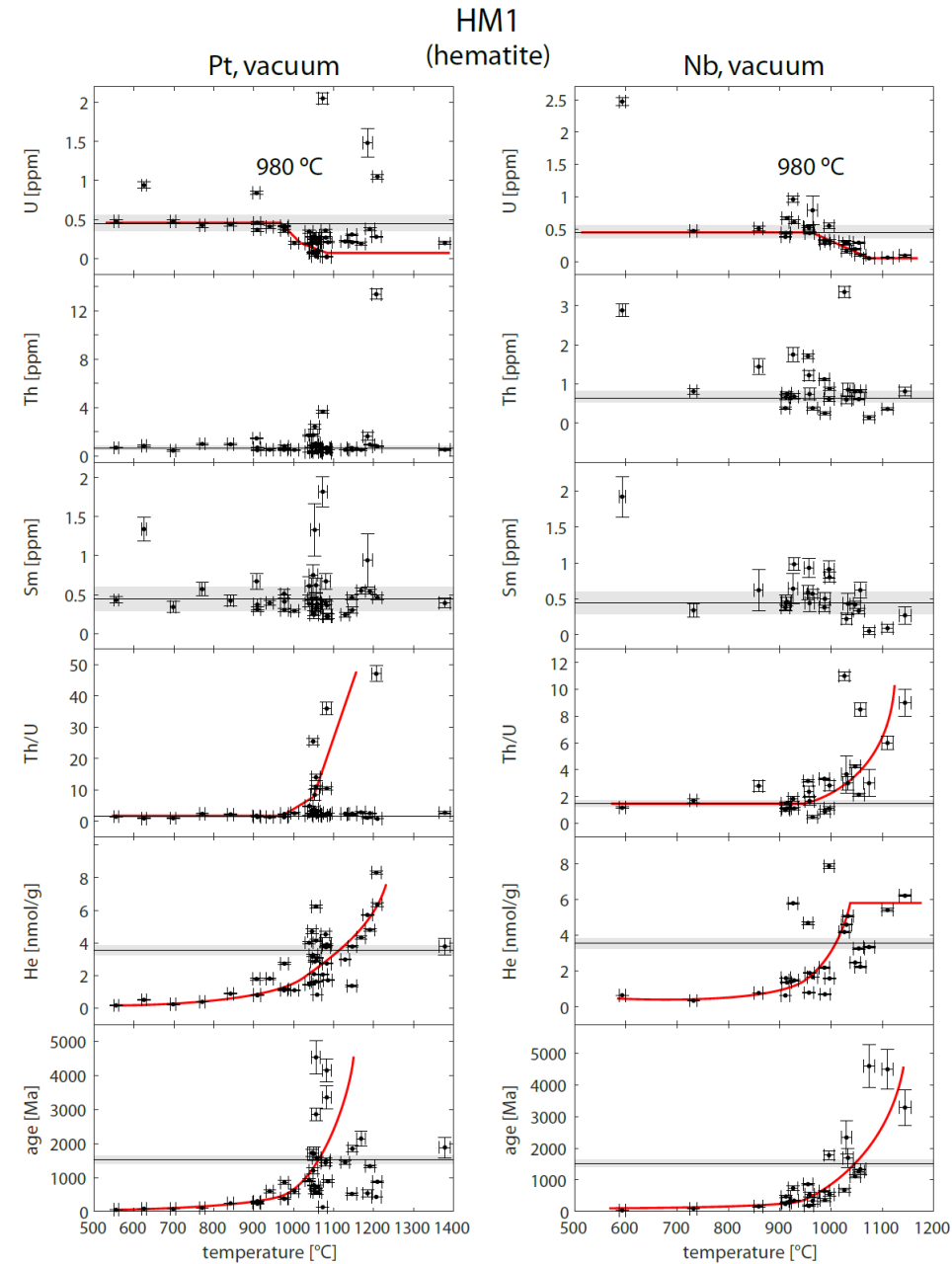


Hofmann et al. (2020)



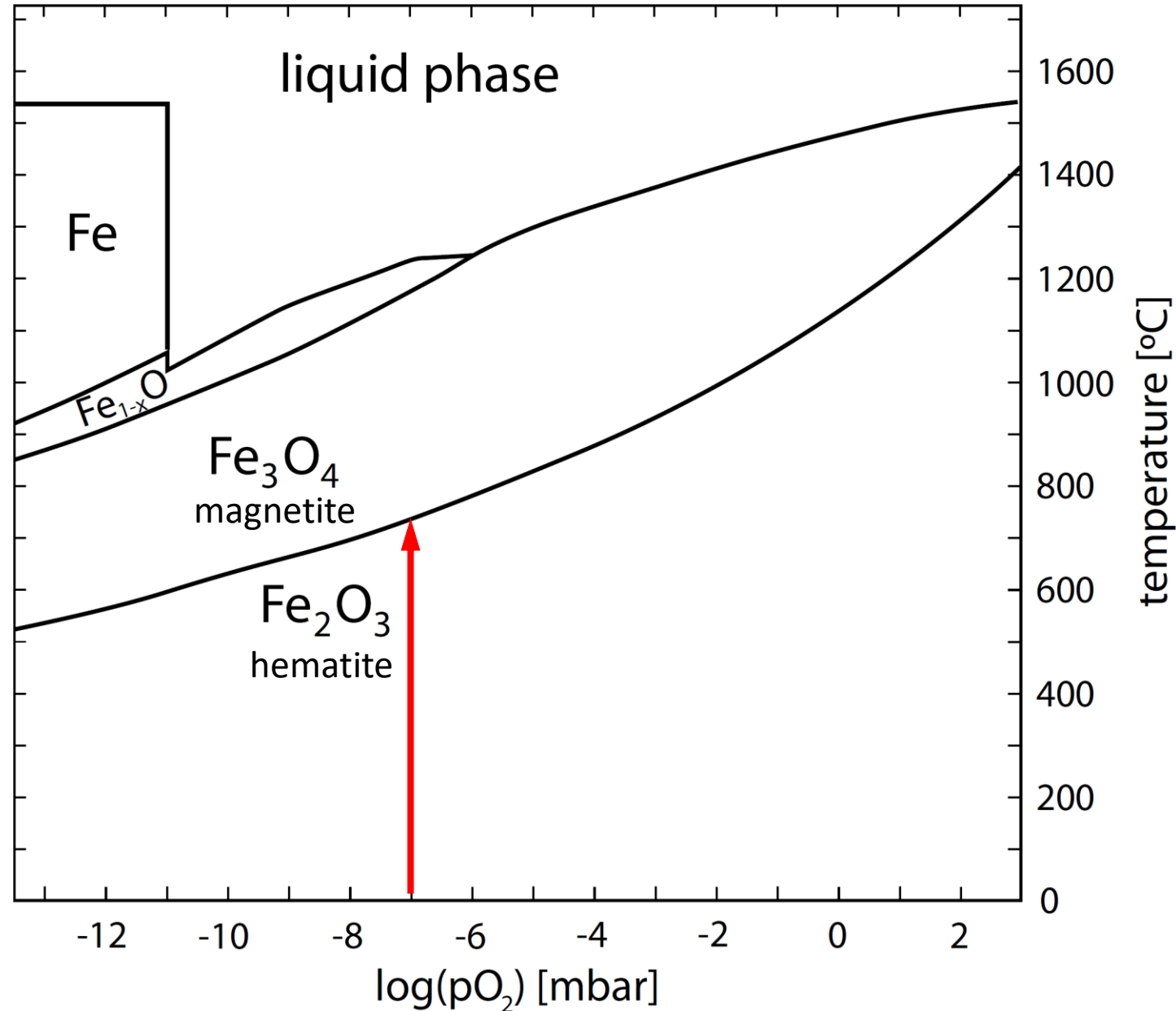


# No difference between Pt and Nb tubes



# What happens during laser-heating?

Reduction of hematite:  
 $3\text{Fe}_2\text{O}_3 \rightarrow 2\text{Fe}_3\text{O}_4 + \text{O}_2$

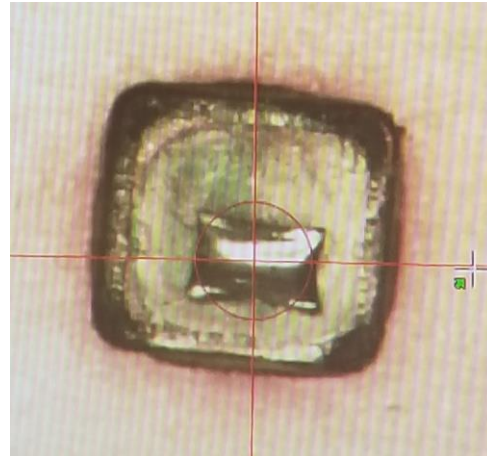
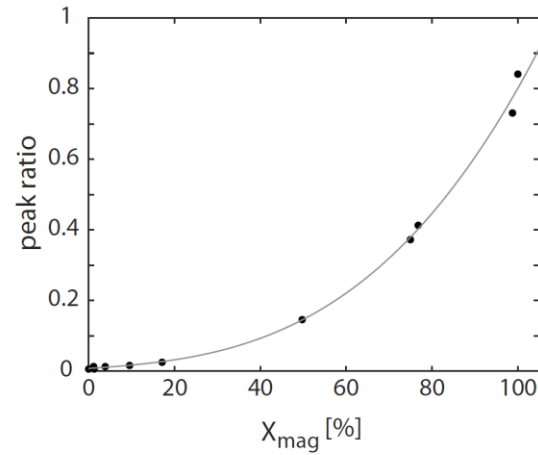
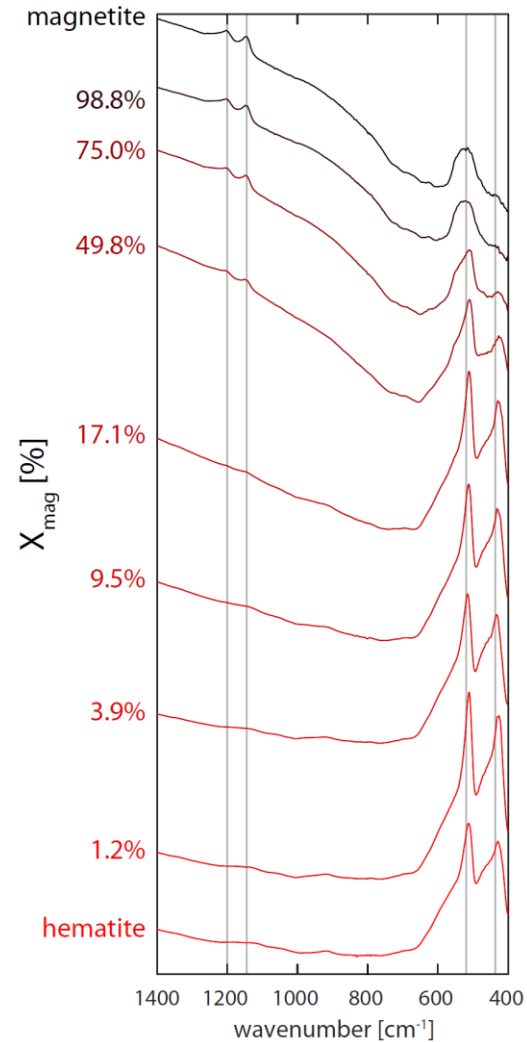


Phase diagram after  
Ketteler et al. (2001)

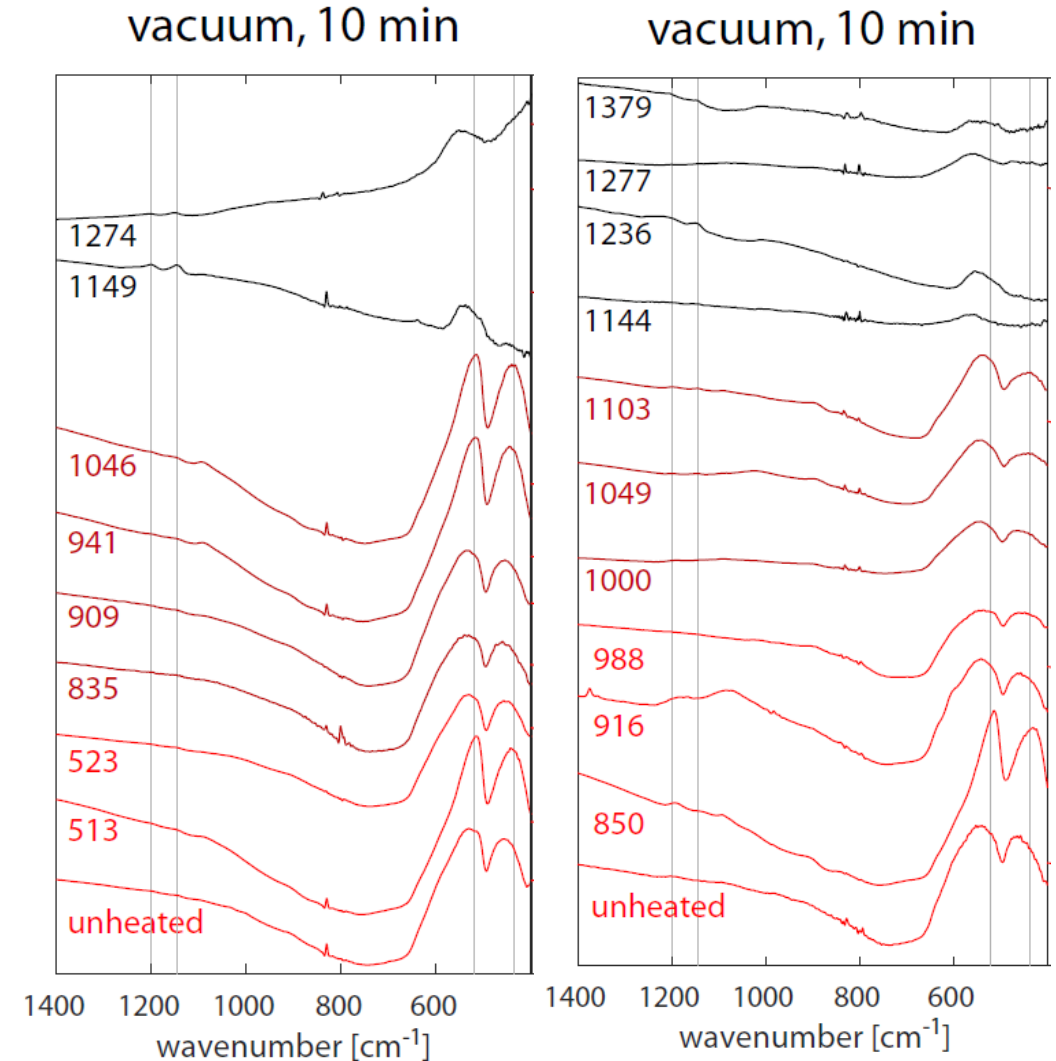


# Monitoring phase changes during laser-heating with FTIR

Calibration of FTIR spectra  
with known mixtures of hematite and magnetite

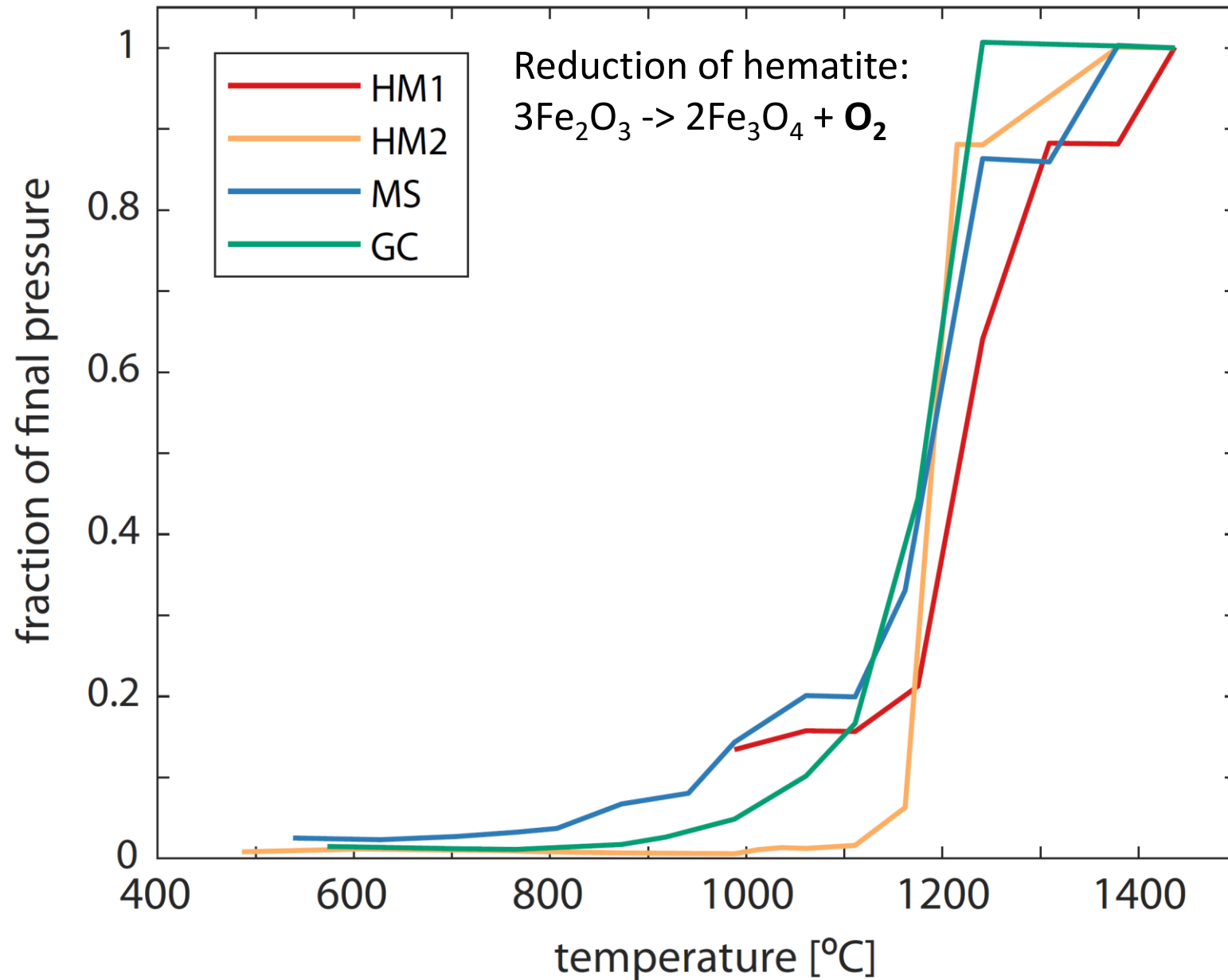


FTIR measurements of heated hematite samples

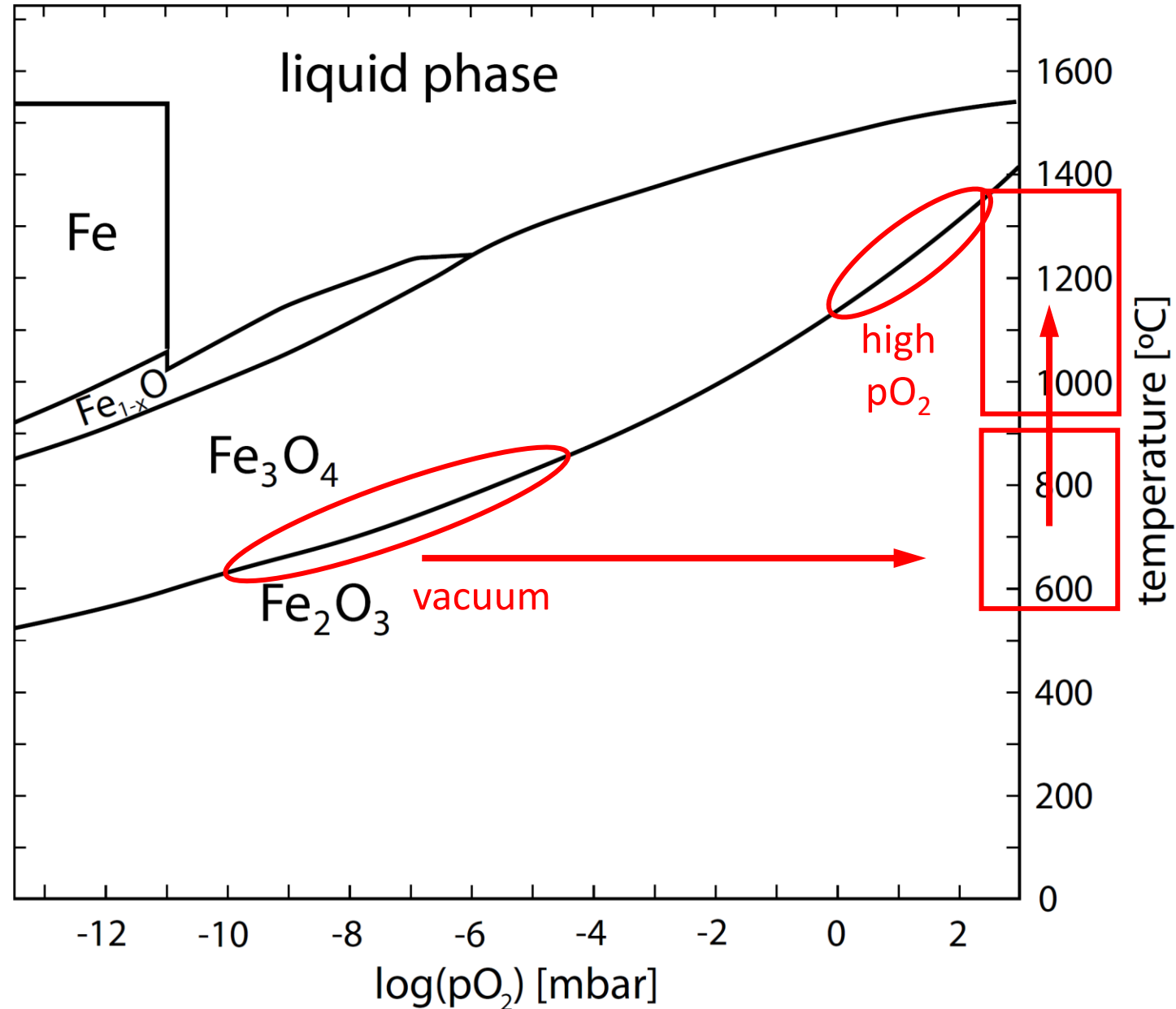


Hofmann et al. (2020)

# Hematite to magnetite transition detected by pressure increase

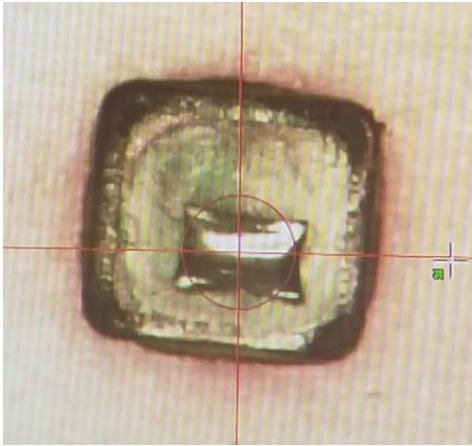


Delaying phase change (and U-loss?) to higher temperatures

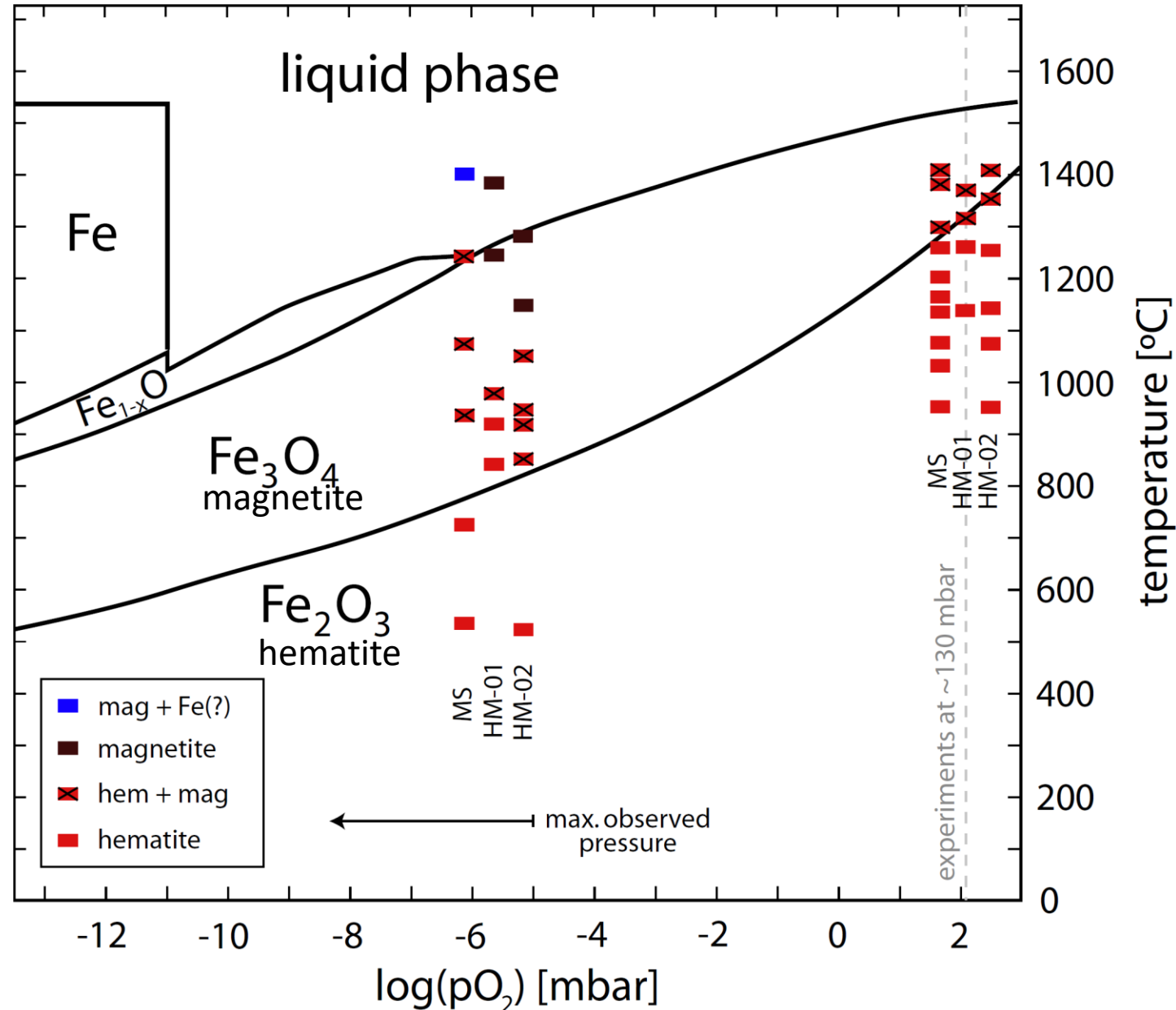


Phase diagram after  
Ketteler et al. (2001)

# Data from laser-heating experiments vs. prediction



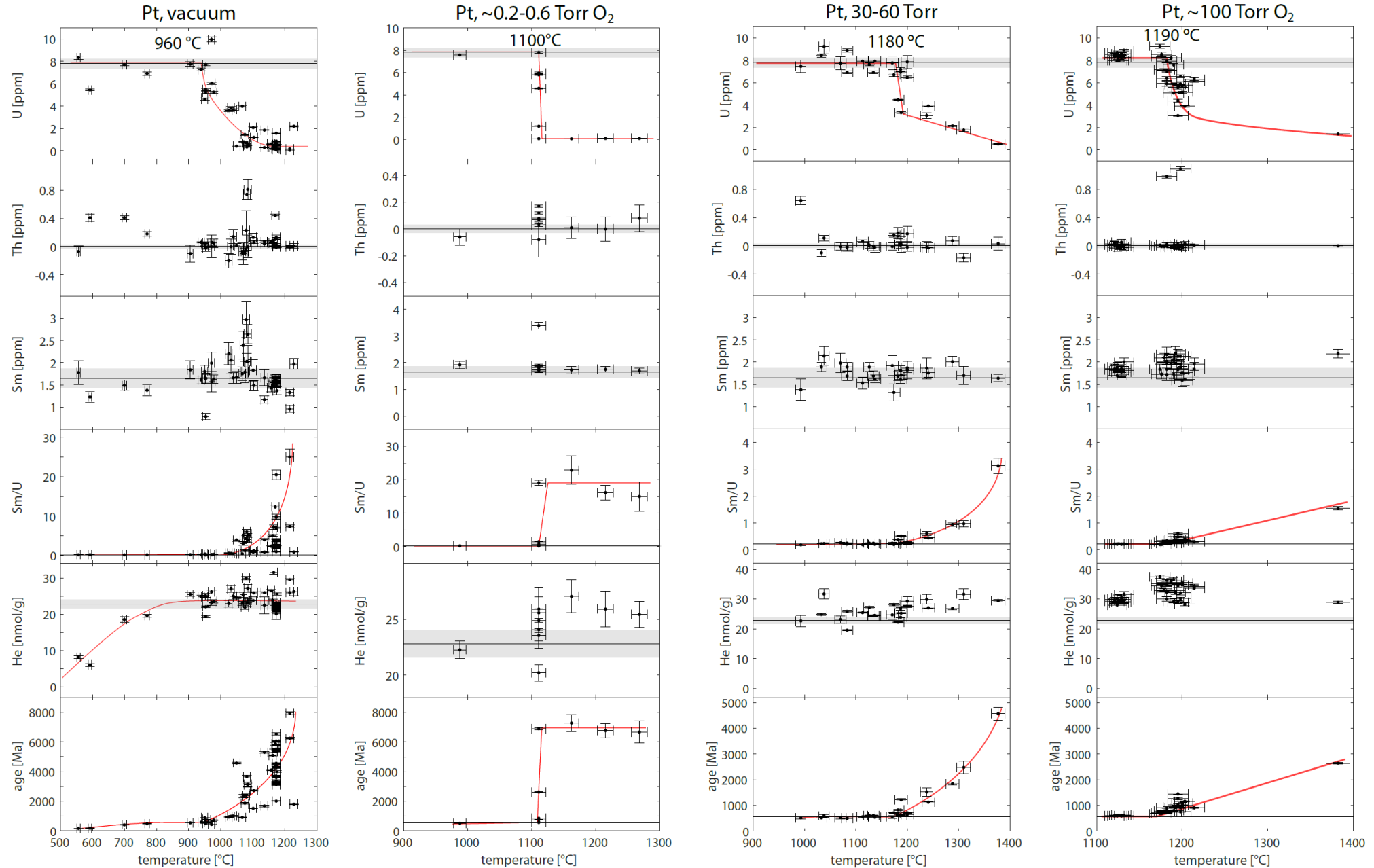
- Laser-heating of Pt packets
- FTIR on powdered sample



Hofmann et al. (2020)

Phase diagram after  
Ketteler et al. (2001)

# Effect of increasing $pO_2$ on U-loss temperature

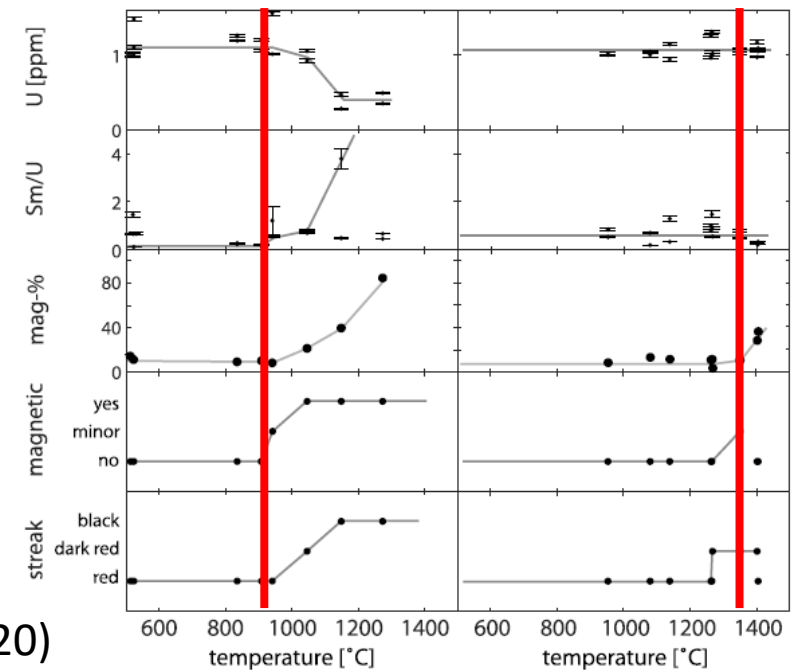
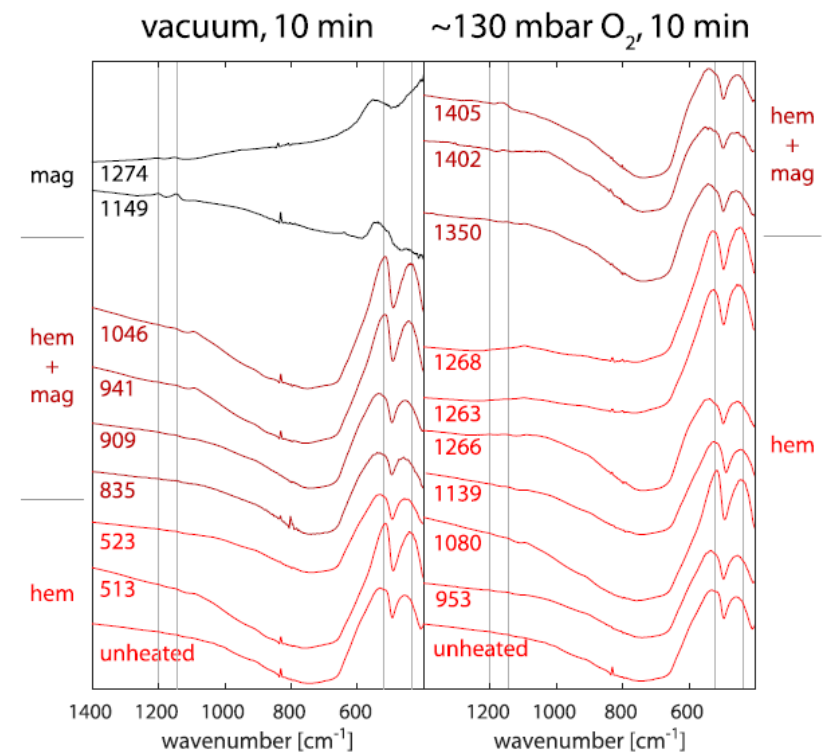
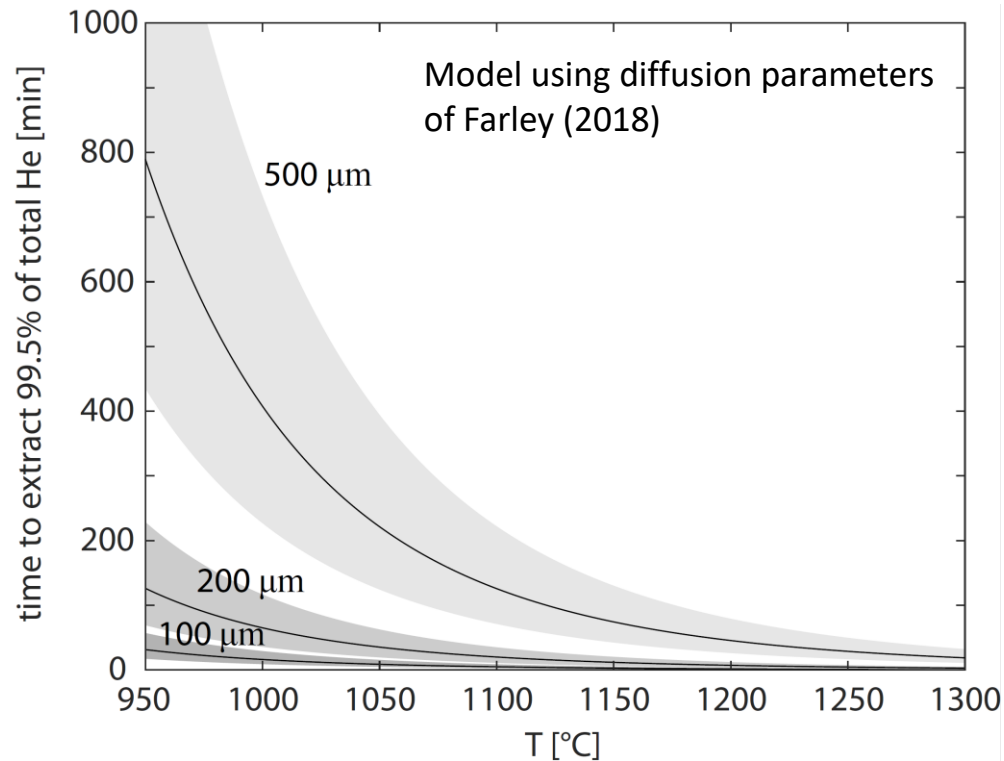


# Correlation of U-loss and phase change

Delay of phase change/U-loss:

~980 °C → 1200-1300 °C

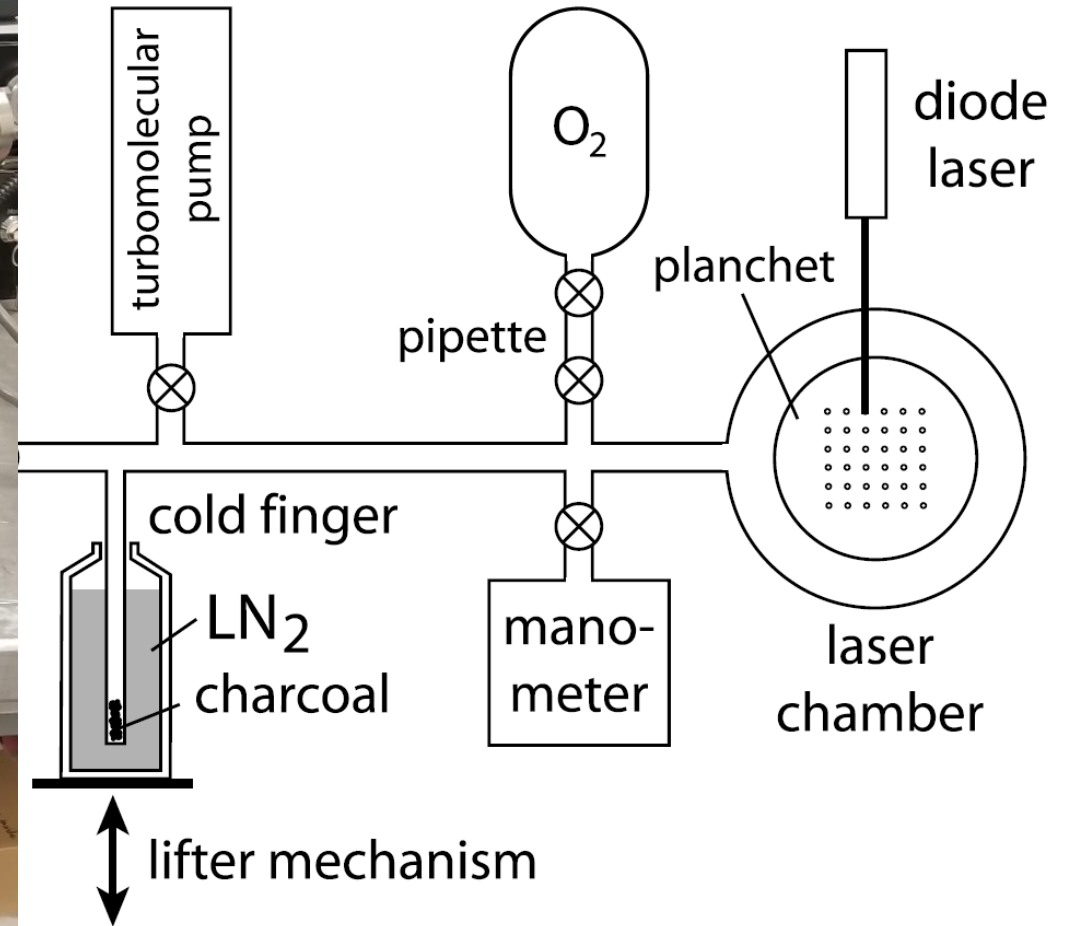
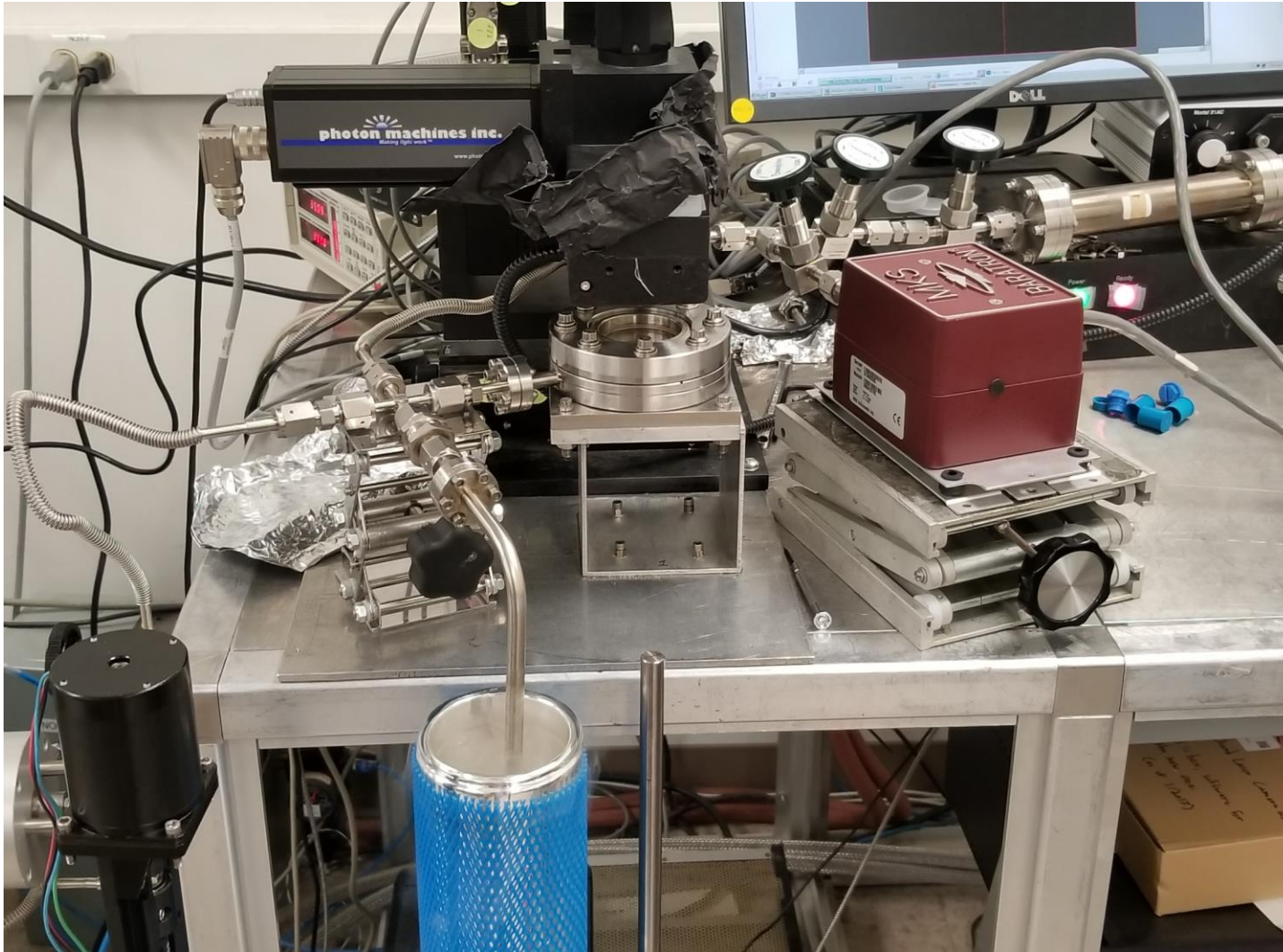
with ~100 Torr (130 mbar) O<sub>2</sub>



Hofmann et al. (2020)

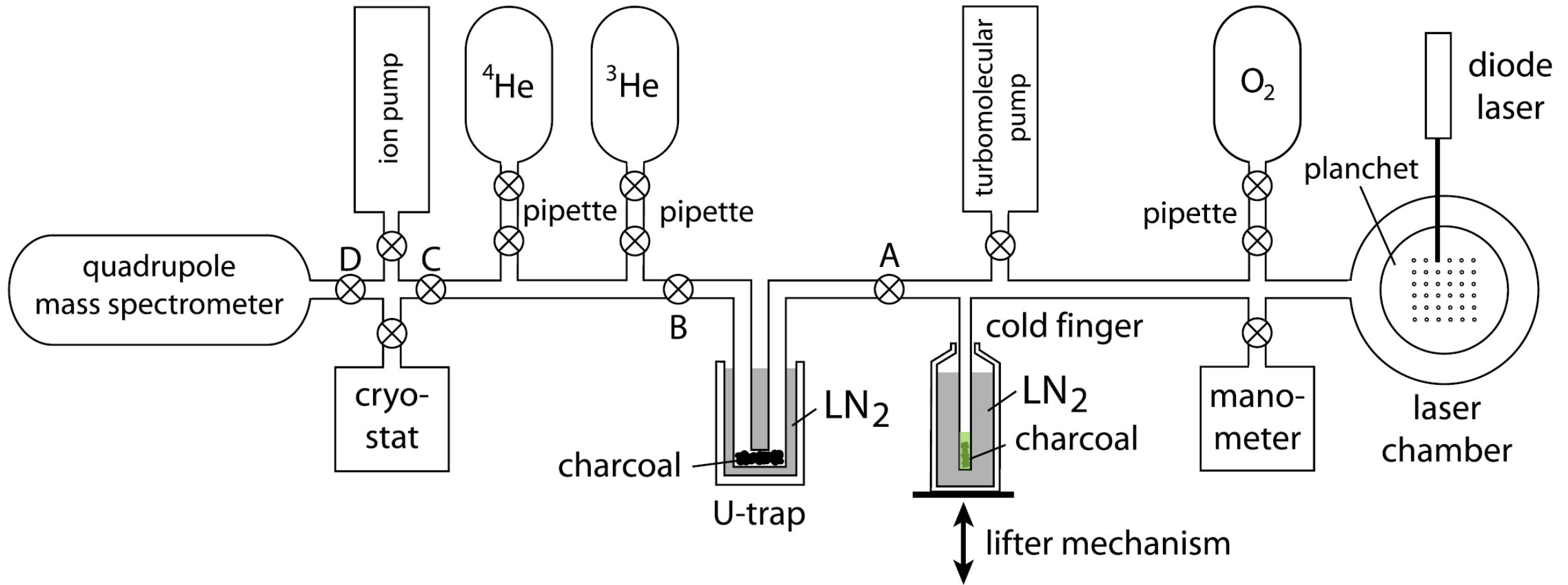


# Modifications to the quad line to automate this method

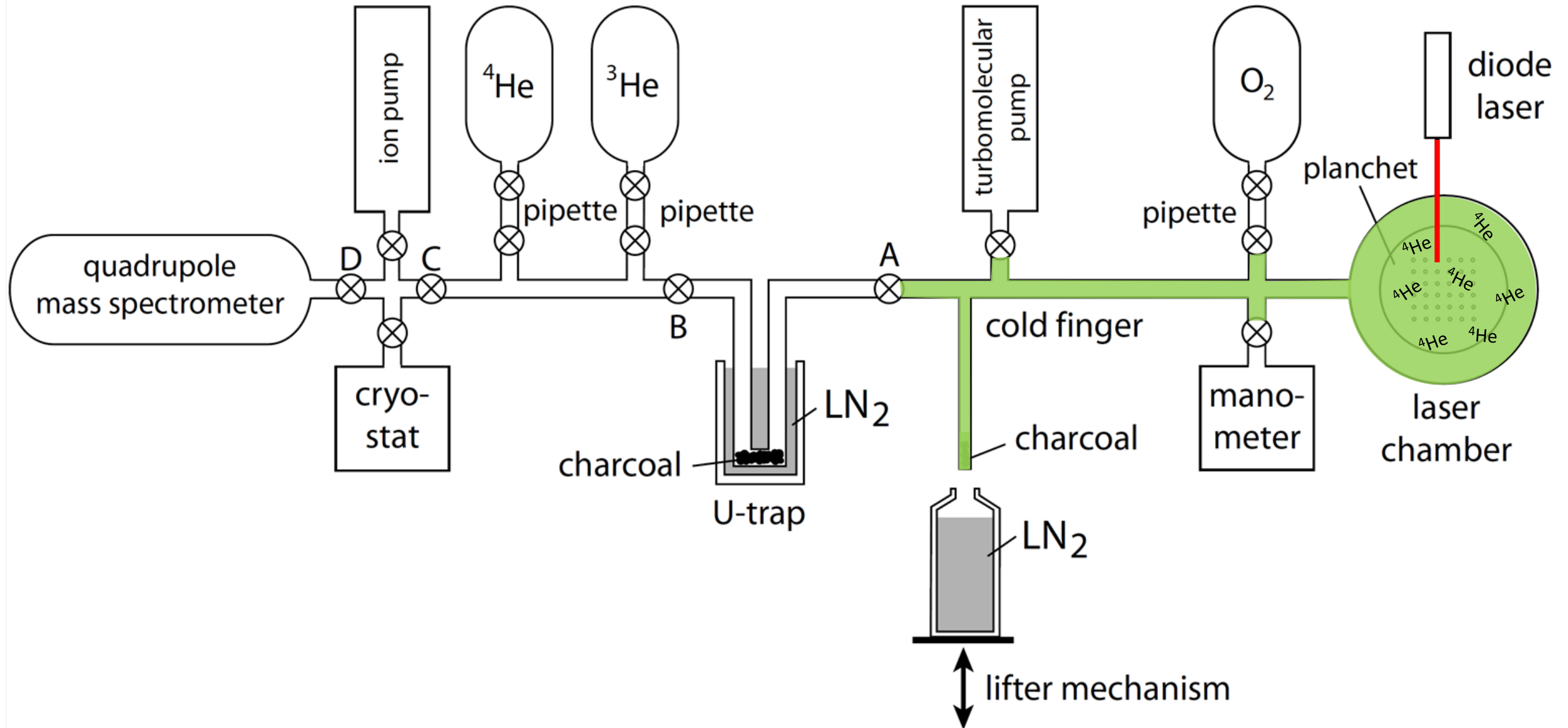




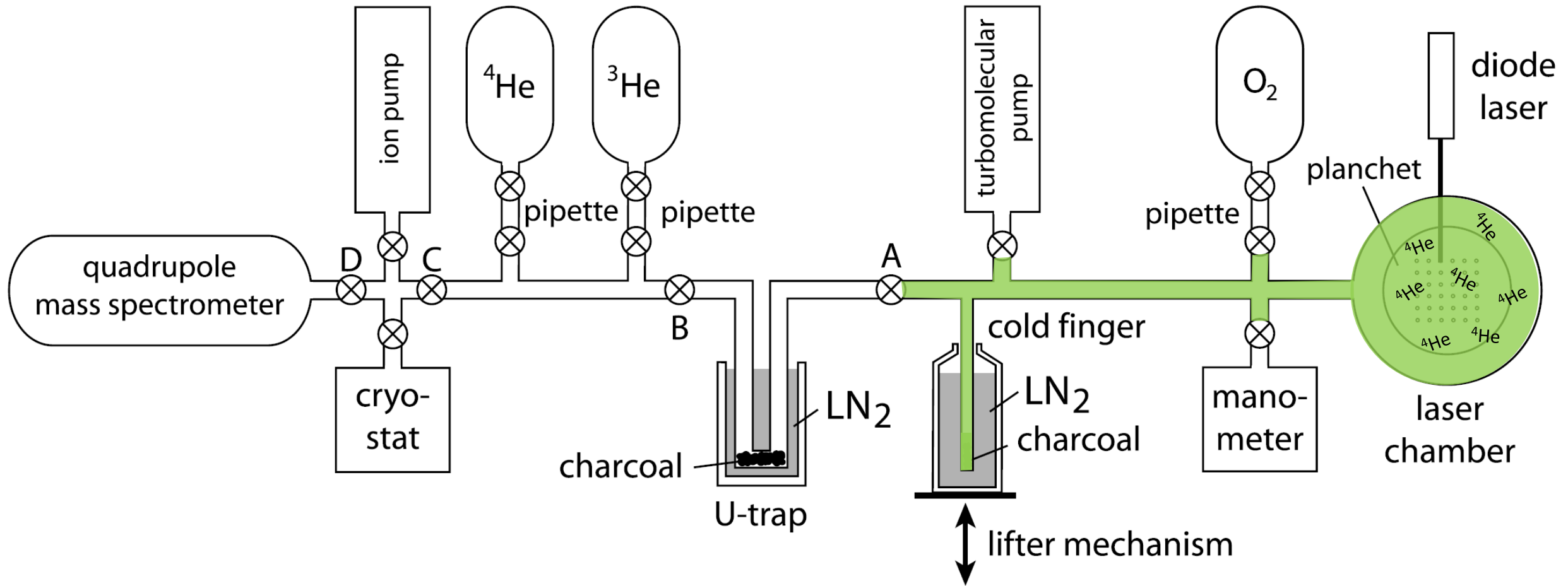
# High-pO<sub>2</sub> degassing procedure



# High-pO<sub>2</sub> degassing procedure

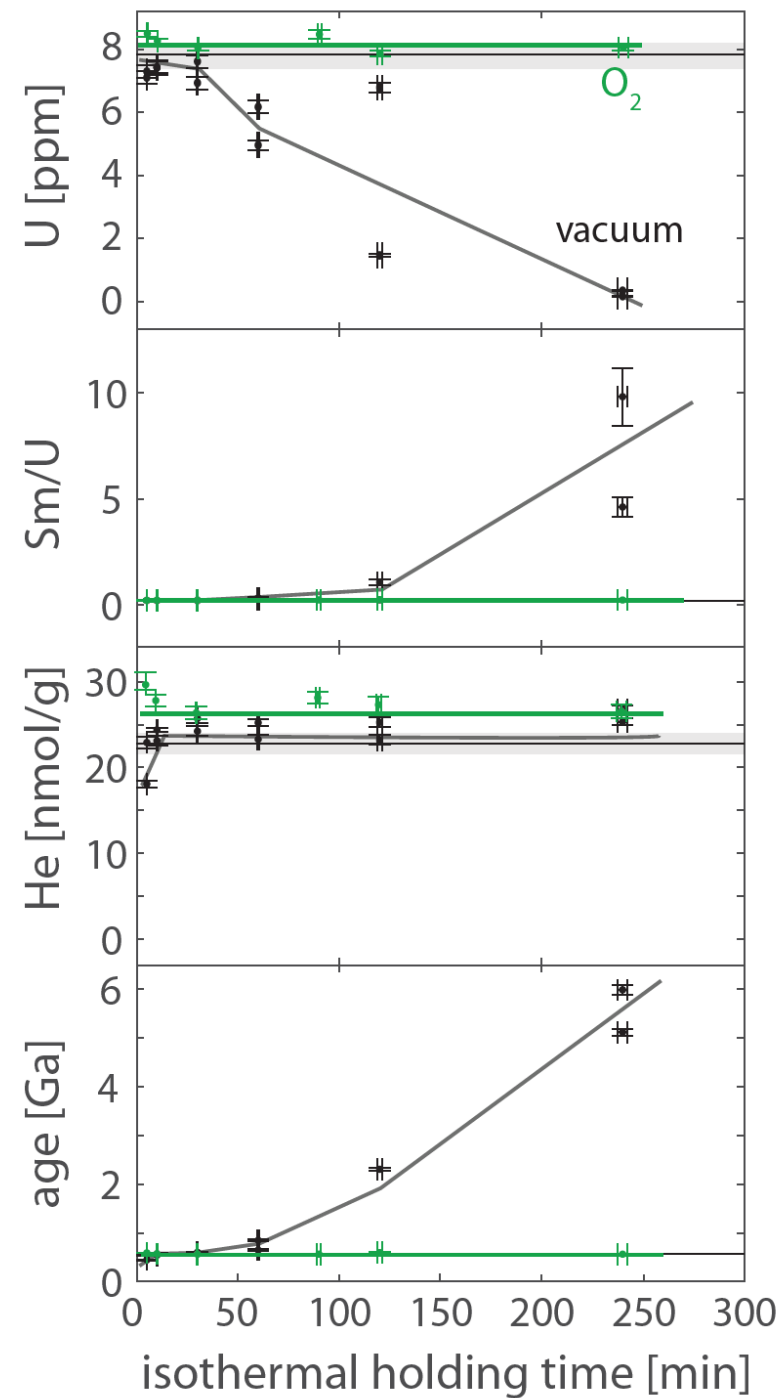


# High-pO<sub>2</sub> degassing procedure

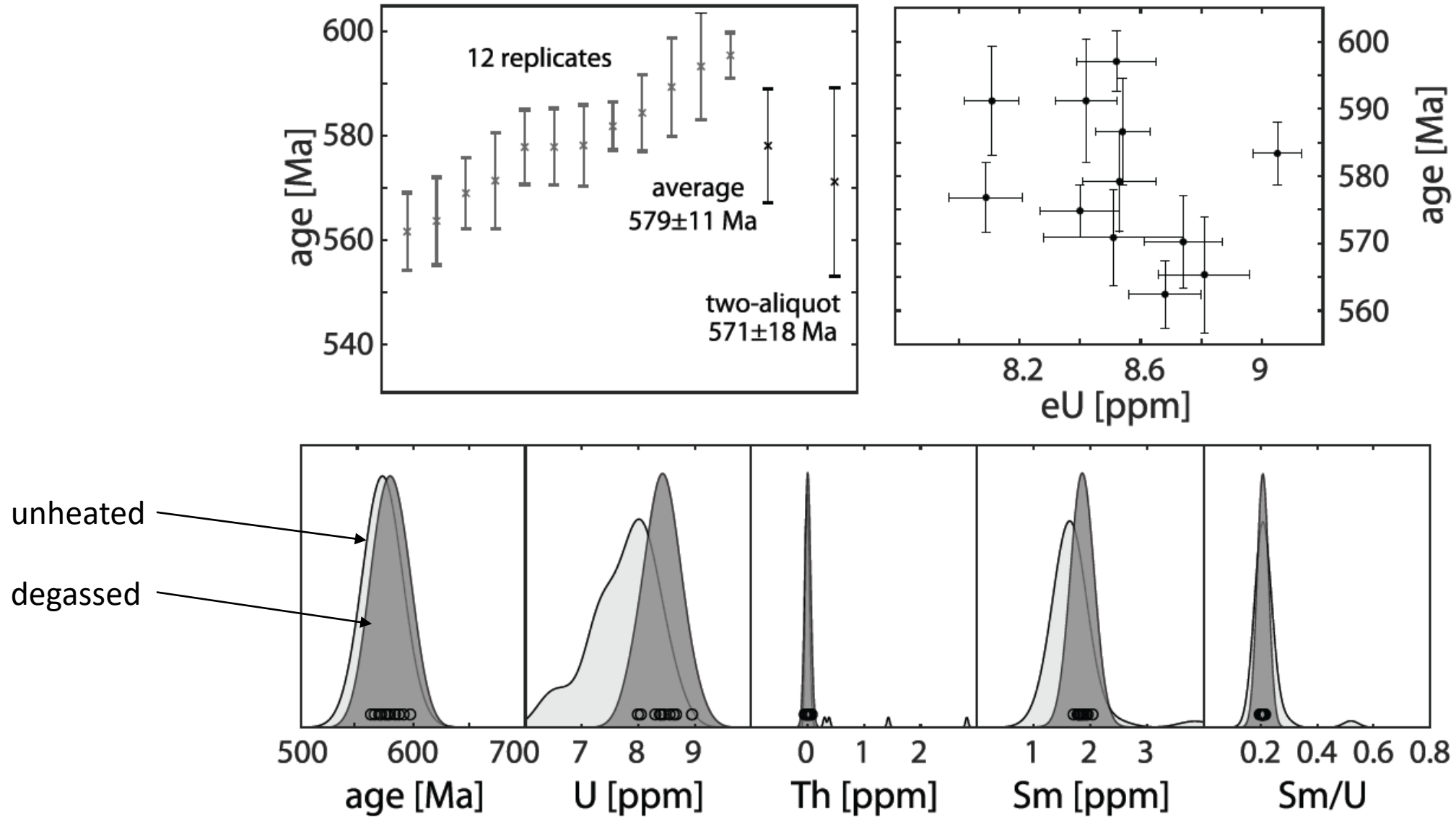


# Isothermal holding at 1000 °C in vacuum and O<sub>2</sub>

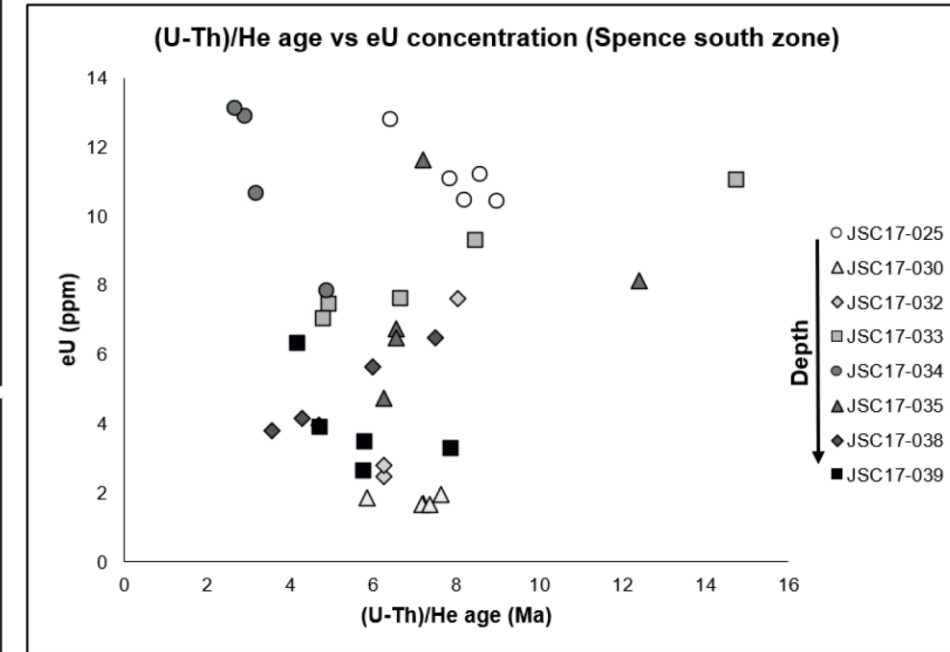
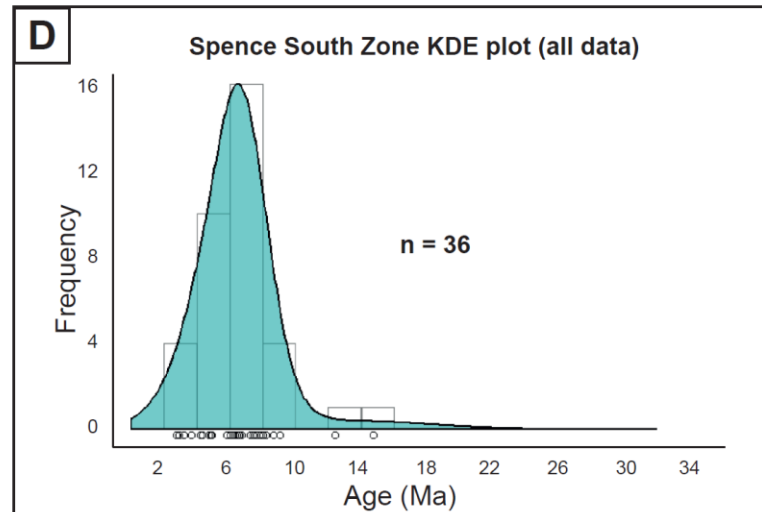
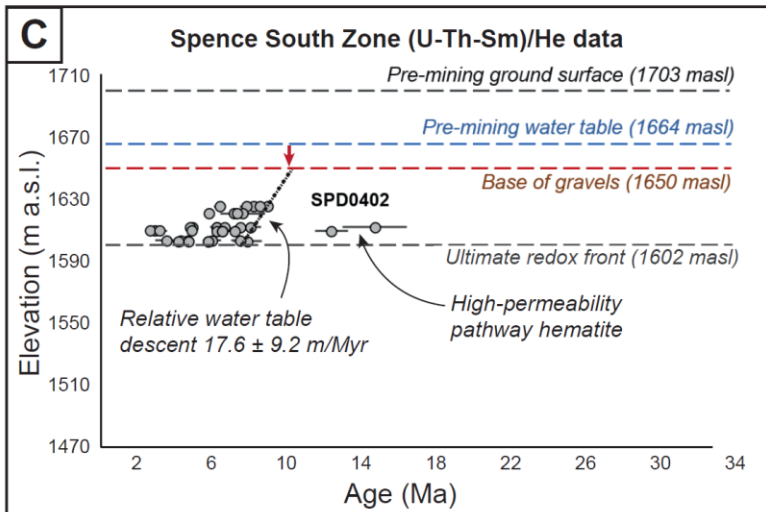
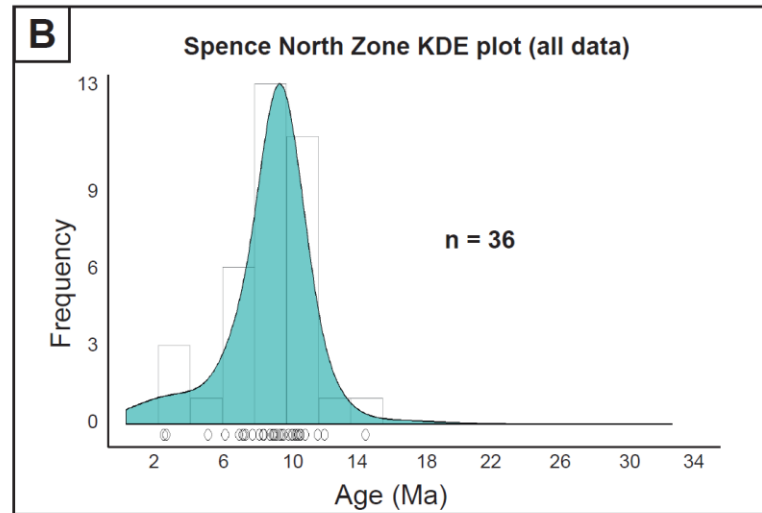
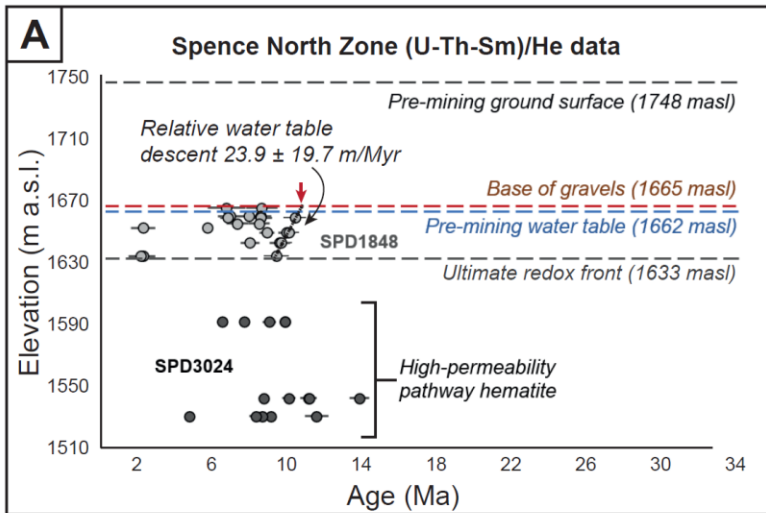
- Loss of U in vacuum increases with holding time
- No detectable loss of U with high-pO<sub>2</sub> at 4 h of holding



# Analysis of highly-retentive hematite sample using high- $pO_2$ method

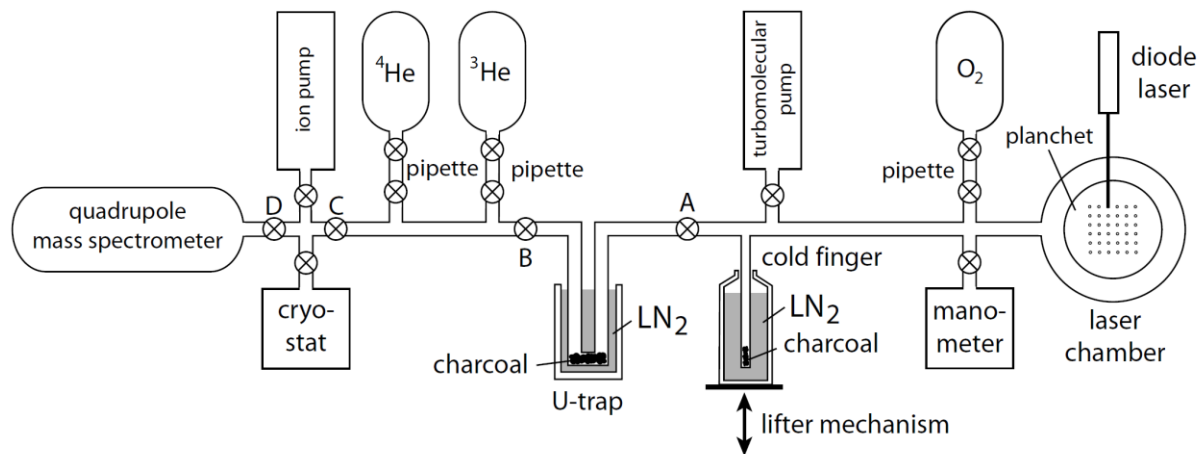


# Recent paper on weathering and groundwater in the Andes using this method



# Conclusions

- Hematite and goethite show detectable U-loss at ~980 °C, massive loss at 1050-1100 °C  
→ Pyrometric feedback during laser-heating is important!
- Th/U or Sm/U can indicate major loss of U, but intra-sample variability (usually 10-20%) can make it hard to detect small but significant loss
- Some hematites must be heated to 1000-1100 °C to be completely degassed
- U-loss correlates with phase change from hematite to magnetite
- This phase change and U-loss can be delayed to higher temperatures with an increased oxygen partial pressure ( $pO_2$ ) during laser-heating
- This procedure can be automated for routine hematite (U-Th)/He dating



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U-loss associated with laser-heating of hematite and goethite in vacuum during (U-Th)/He dating and prevention using high  $\text{O}_2$  partial pressure

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