

The high-pO₂ method to prevent U-loss during single-
aliquot hematite (U-Th)/He measurement:
Development, implementation, and automation

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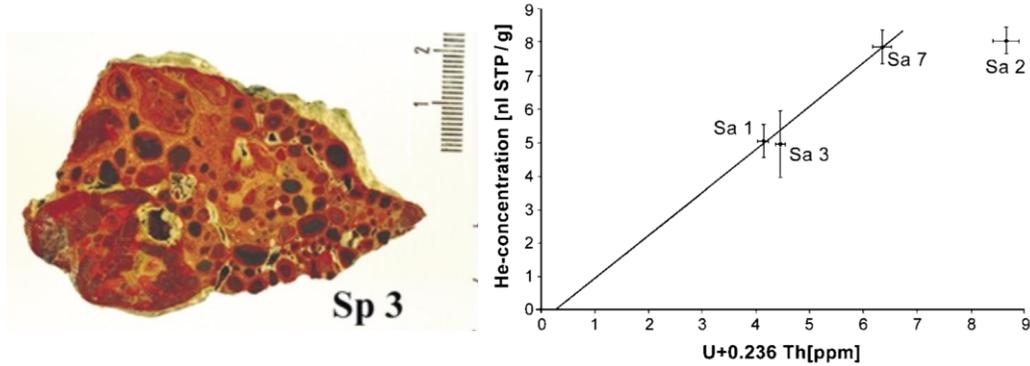
Jonathan Treffkorn
Ken Farley
California Institute of Technology

Thermo2021
September 13, 2021

Utility of hematite (U-Th)/He

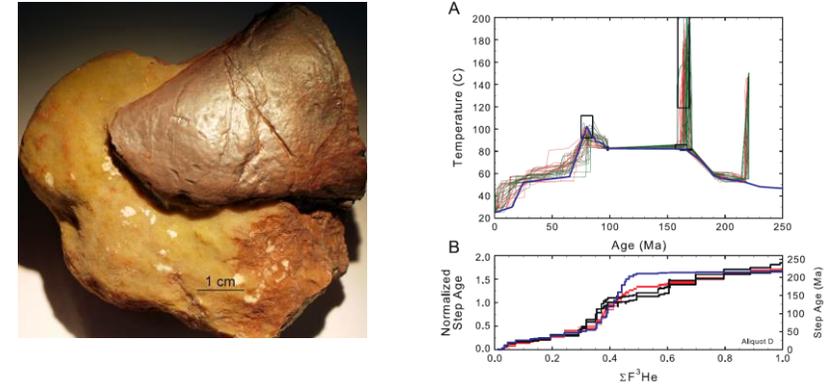
Geochronology

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

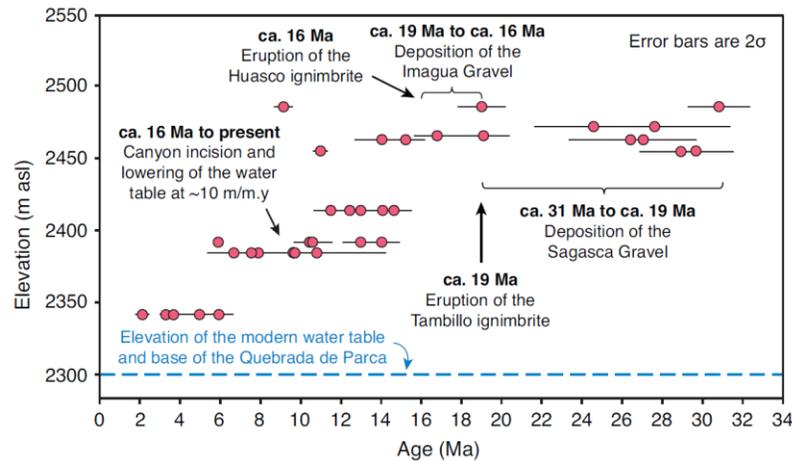


Thermochronology

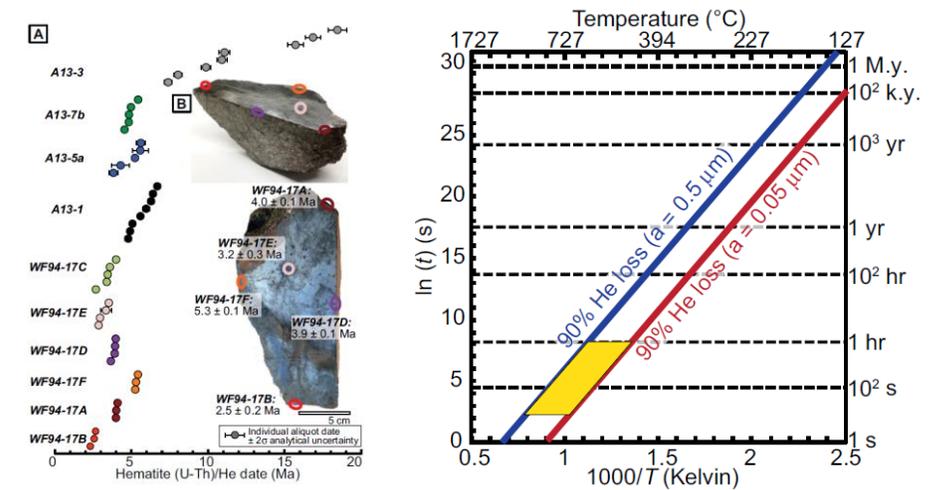
Farley and Flowers (2013) – temperature history modeling of polycrystalline aggregates



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

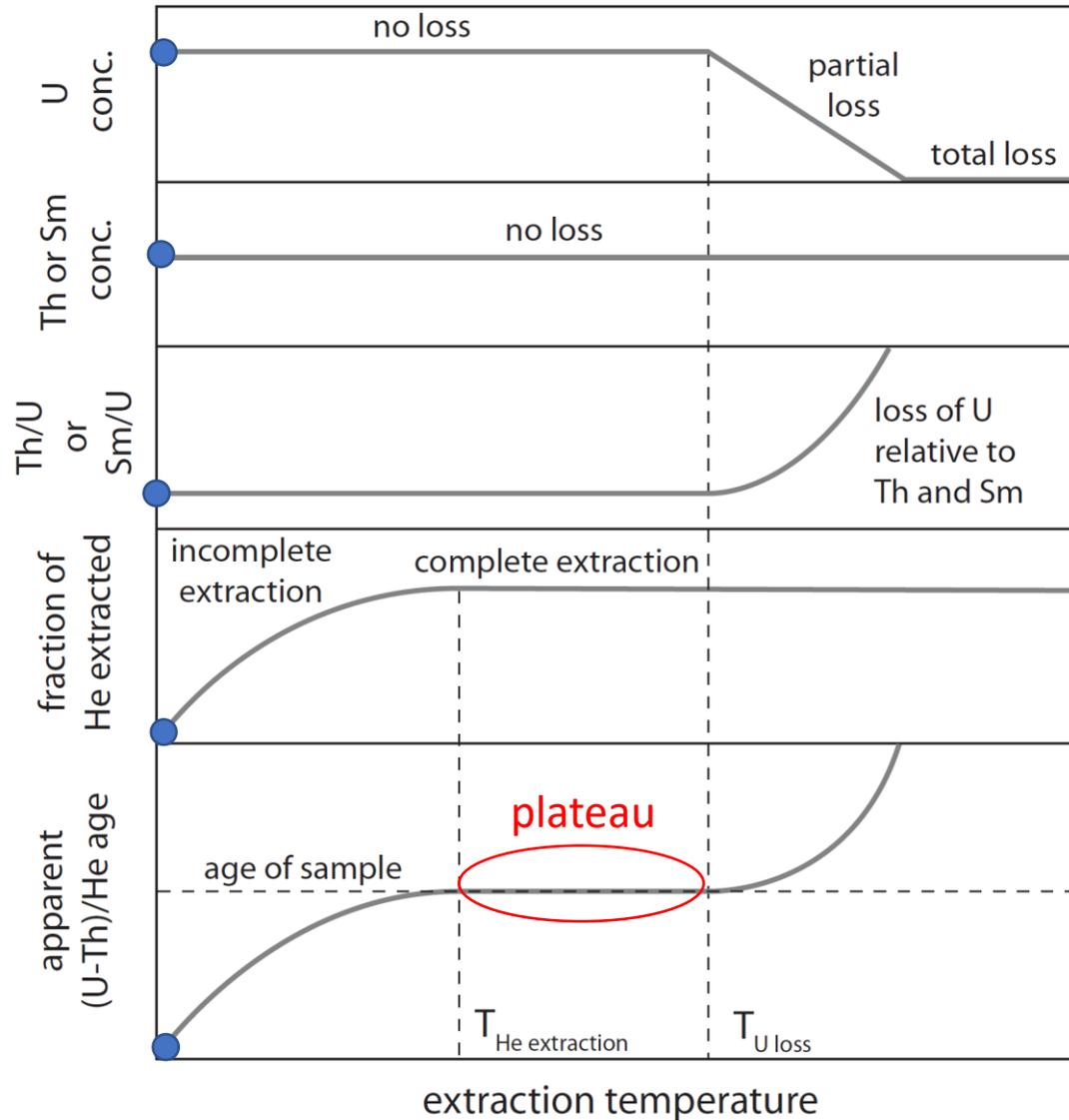


Ault et al. (2015) – resetting of hematite crystallites due to shear-heating

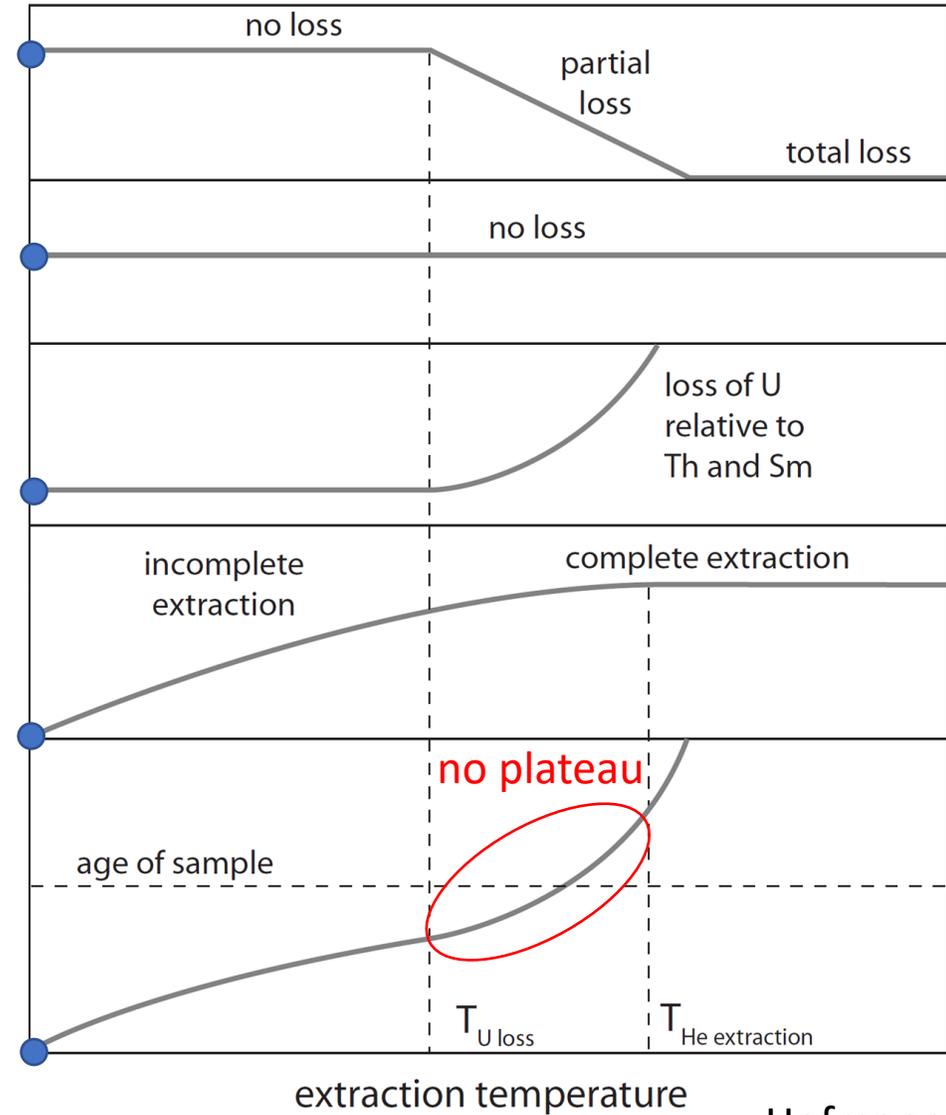


Loss of U during laser-heating

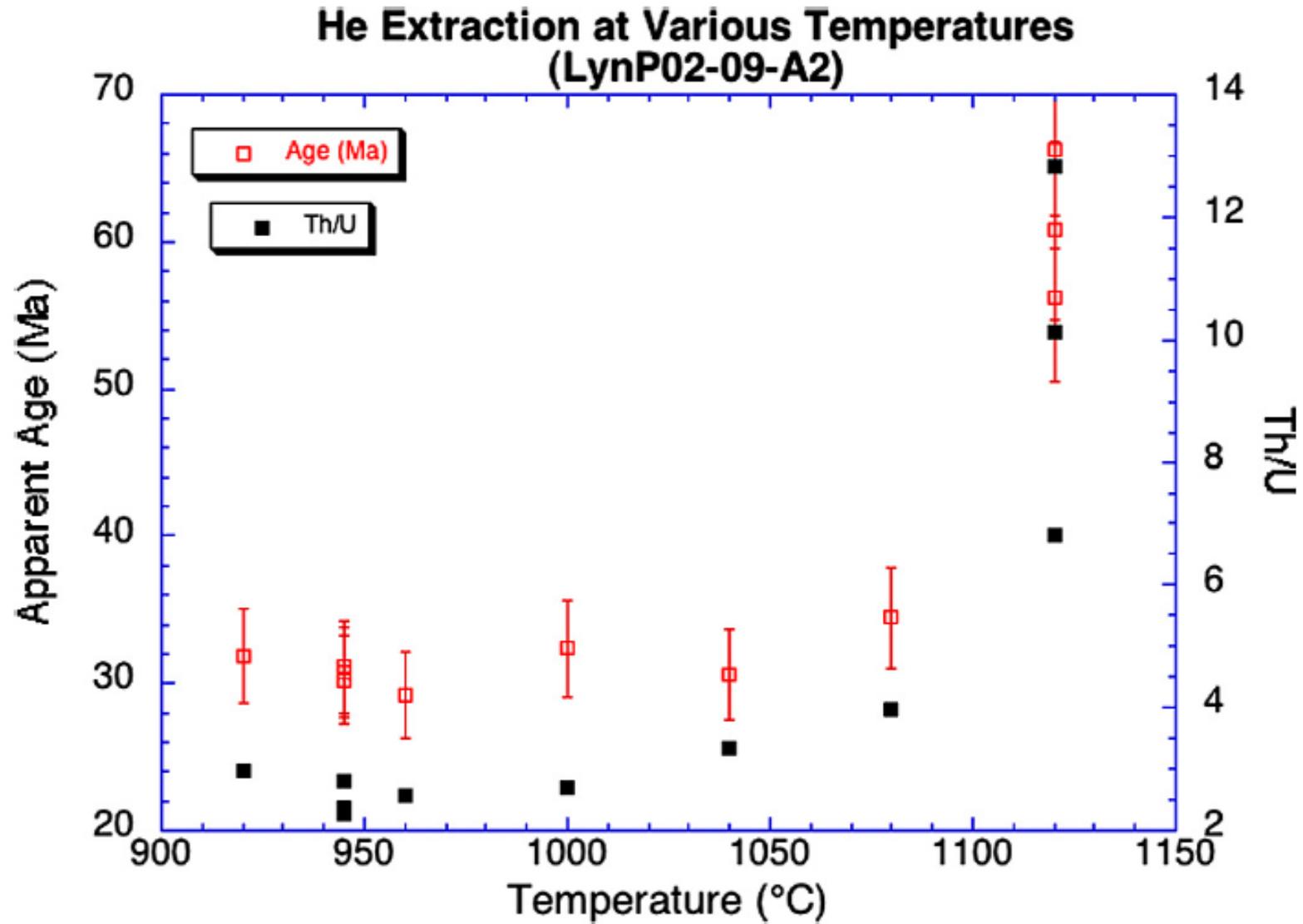
Ideal case



Problematic case

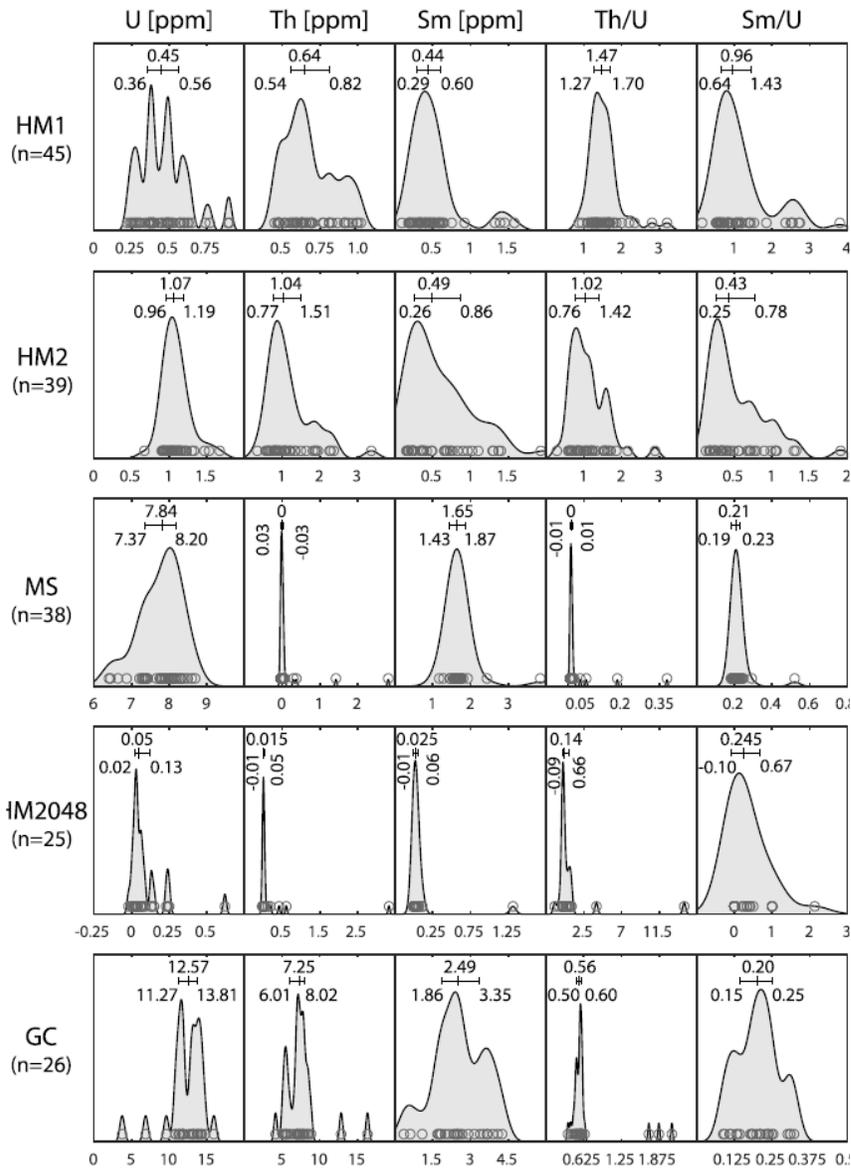


Loss of U during laser-heating

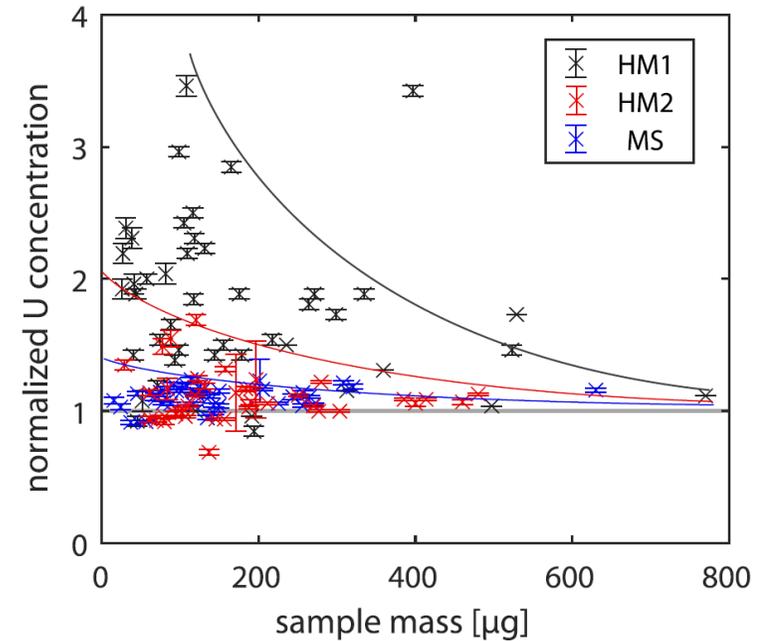
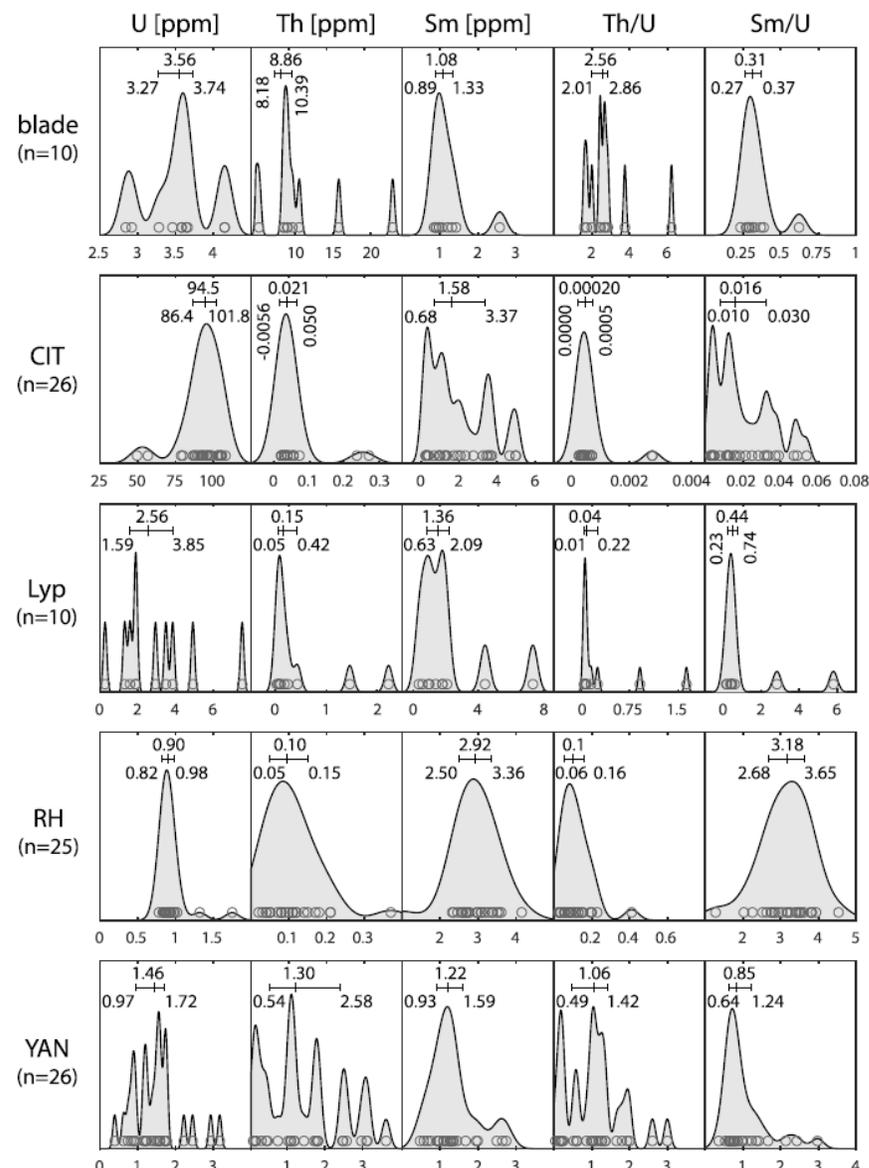


Natural variability in hematite and goethite samples

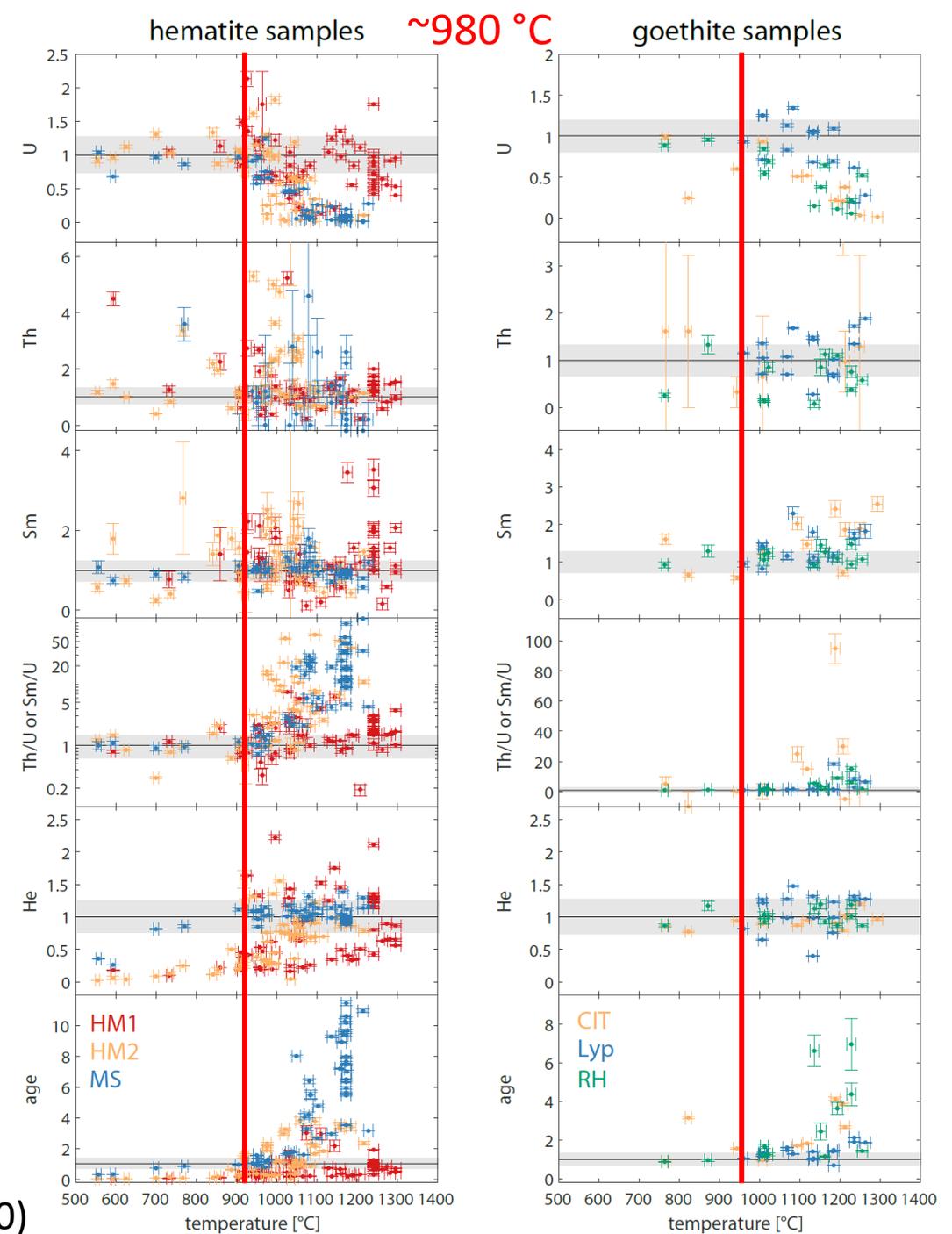
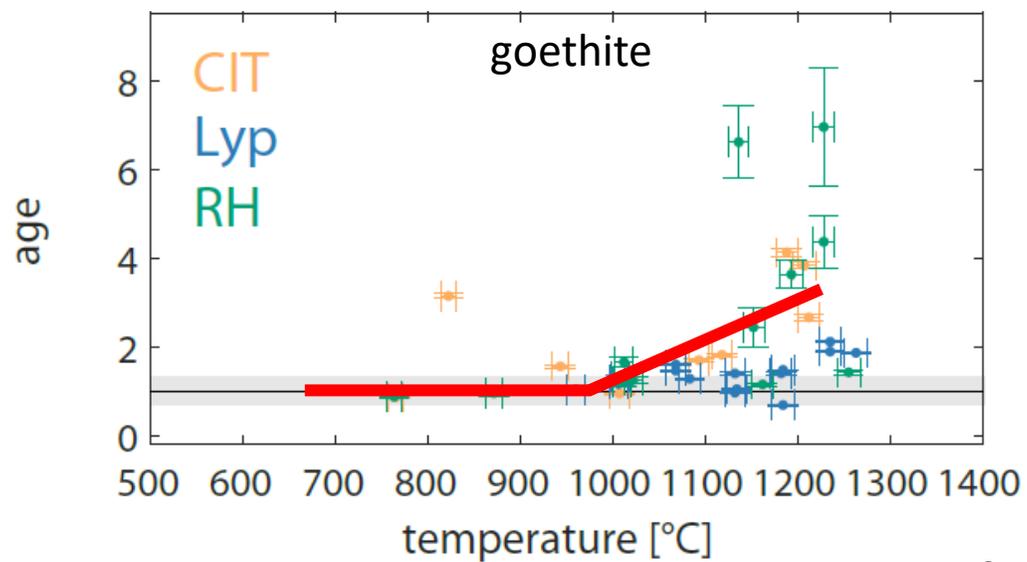
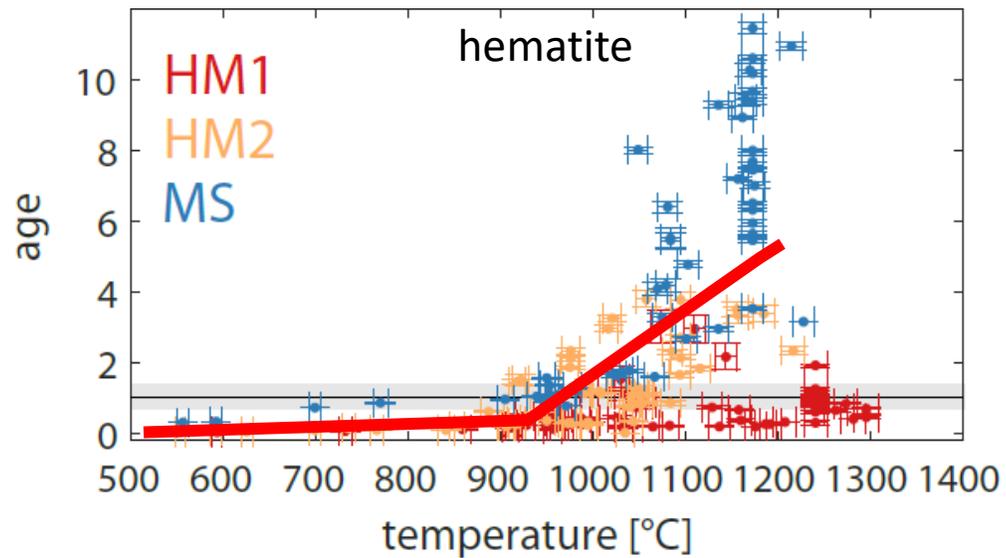
Hematite



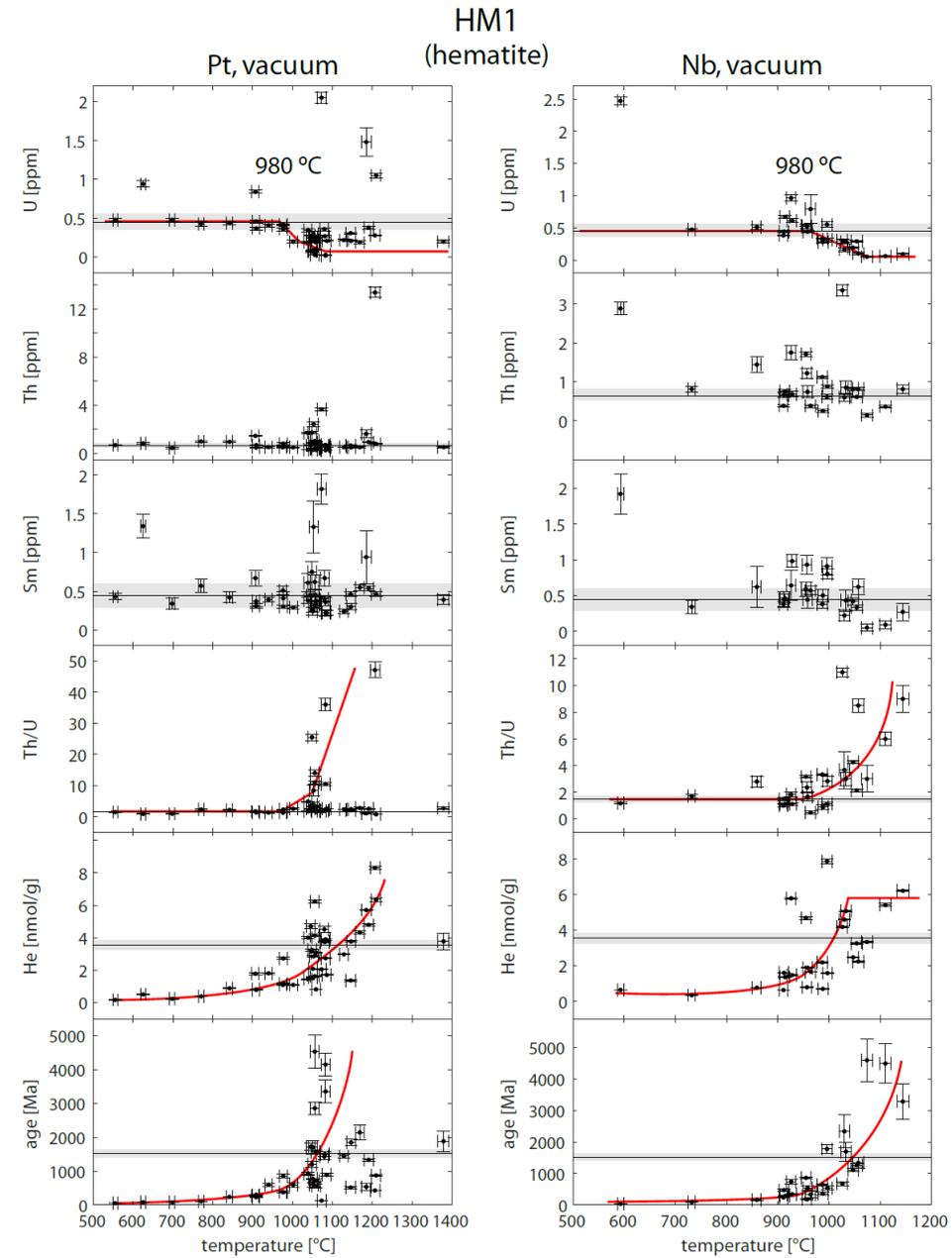
Goethite



Heating tests of multiple samples

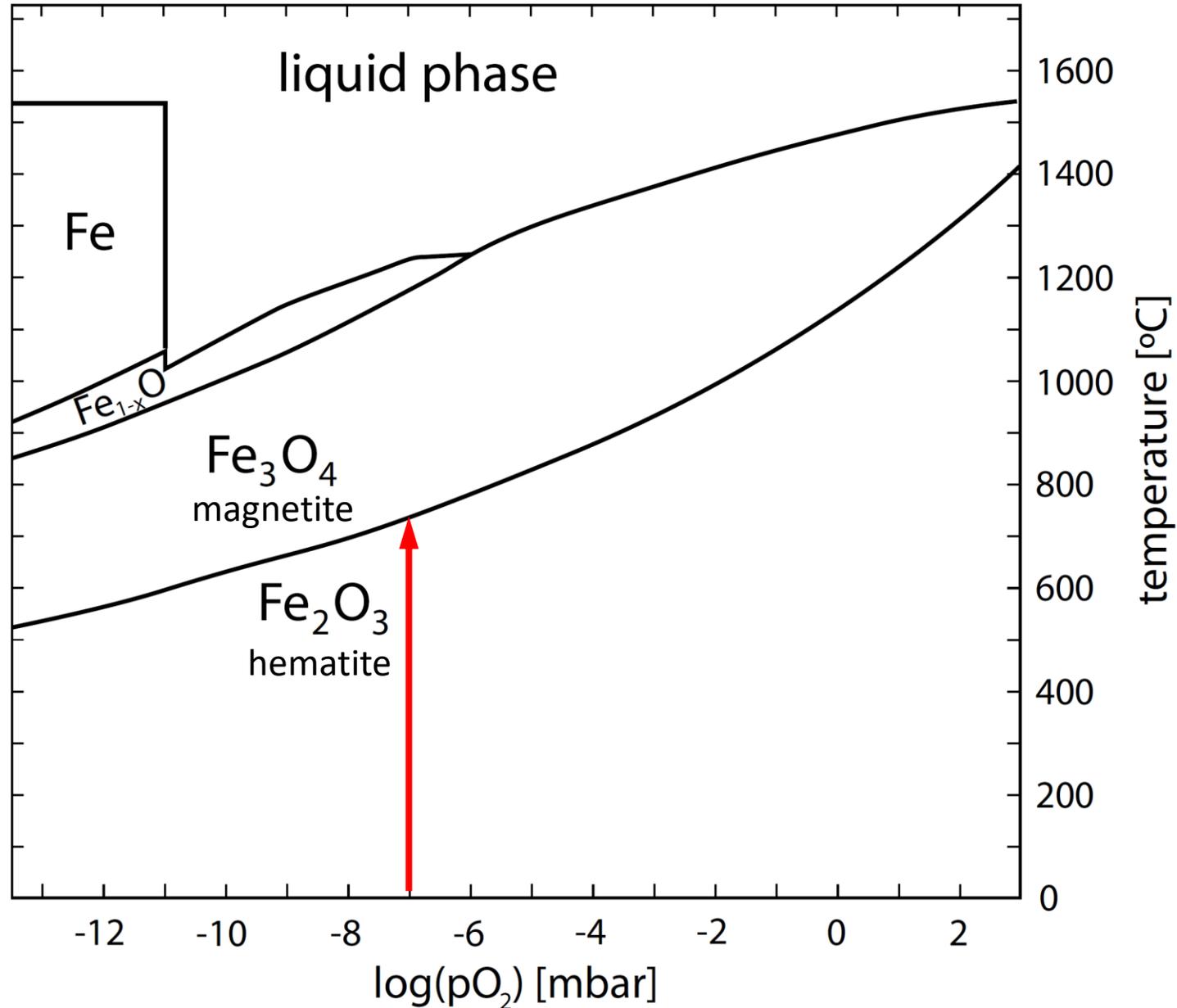


No difference between Pt and Nb tubes



What happens during laser-heating?

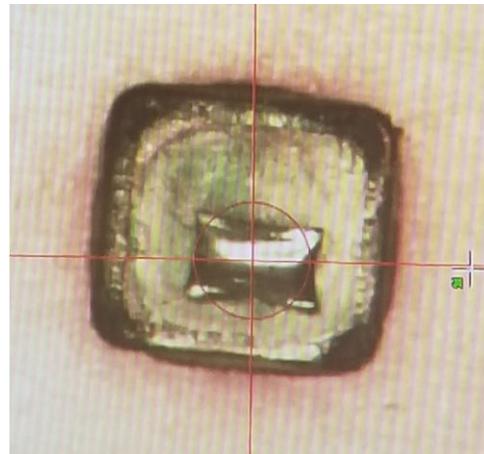
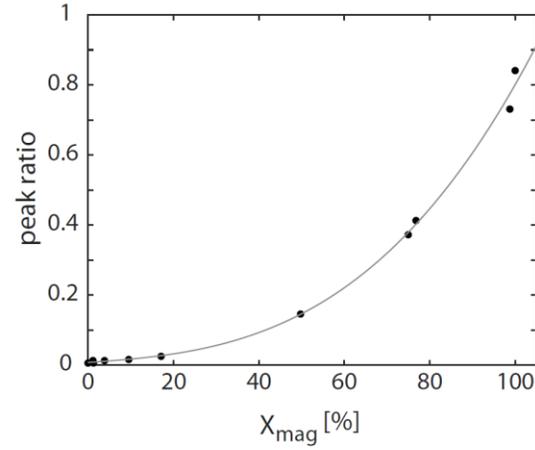
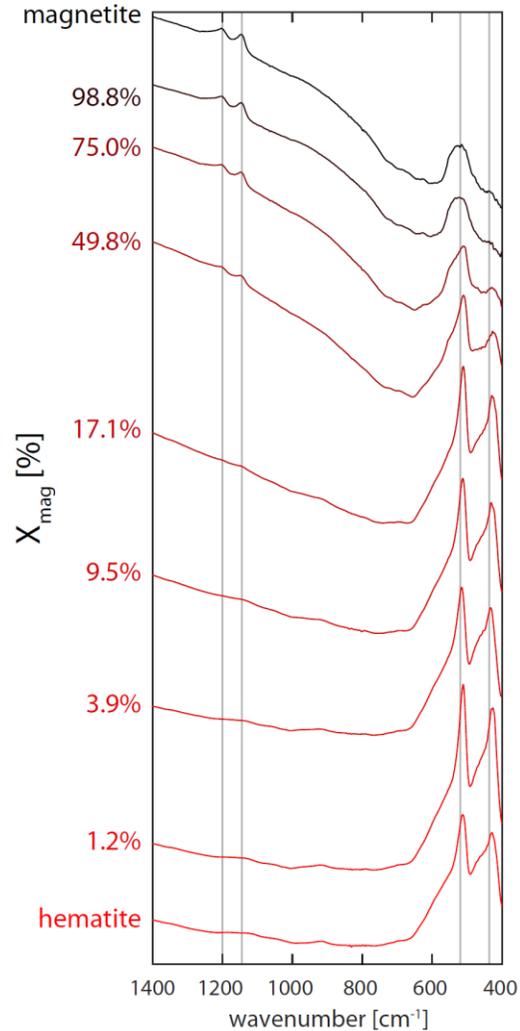
Reduction of hematite:
 $3\text{Fe}_2\text{O}_3 \rightarrow 2\text{Fe}_3\text{O}_4 + \text{O}_2$



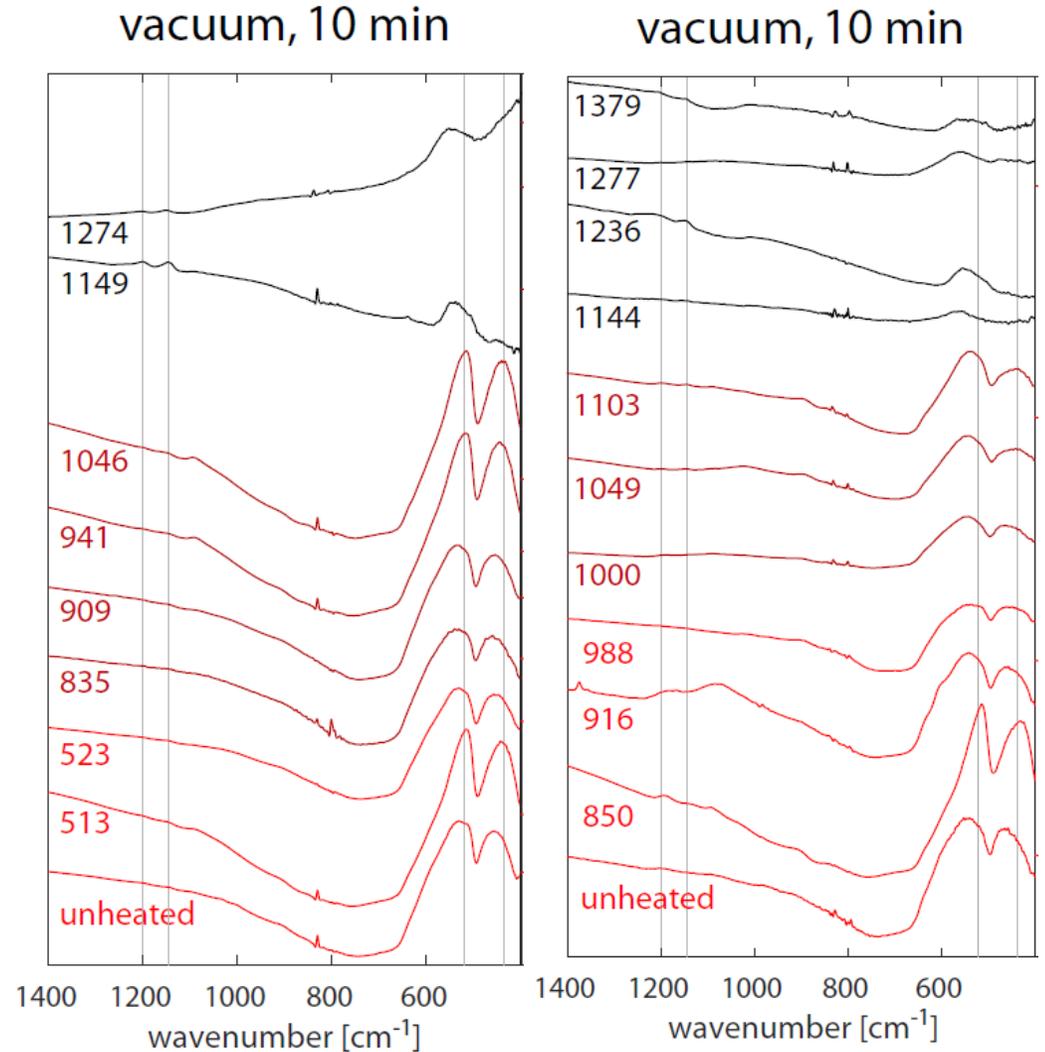
Phase diagram after
Ketteler et al. (2001)

Monitoring phase changes during laser-heating with FTIR

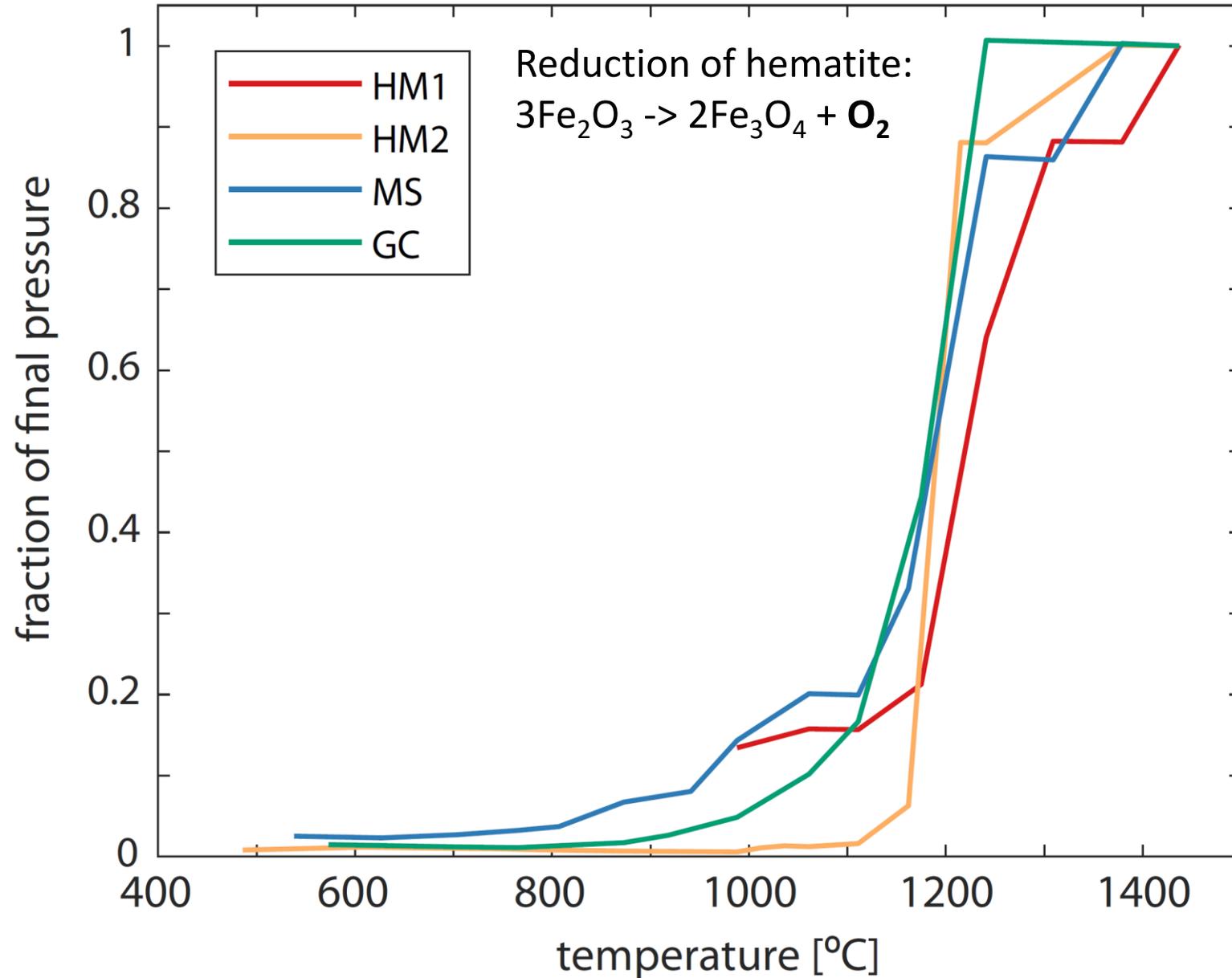
Calibration of FTIR spectra
with known mixtures of hematite and magnetite



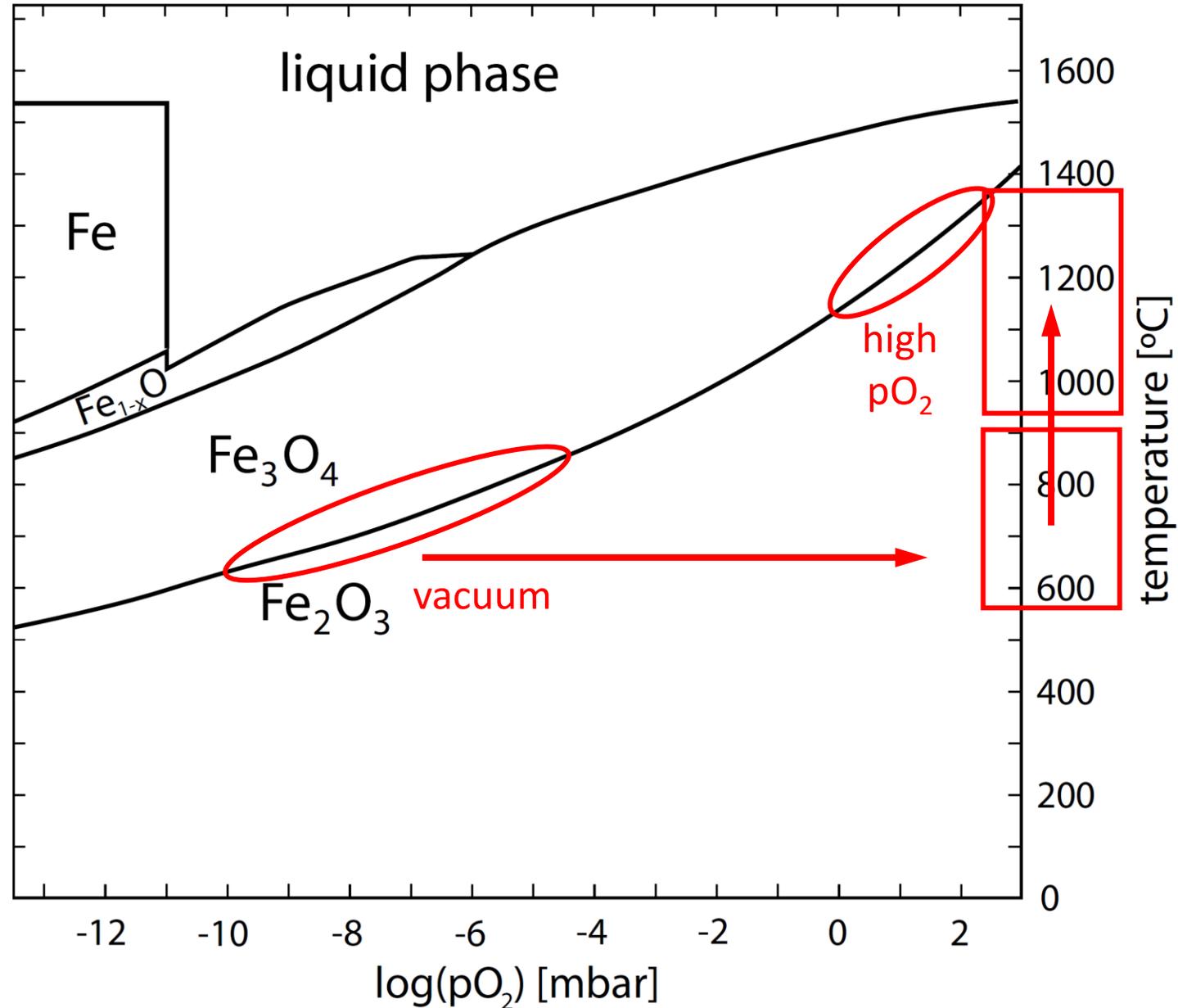
FTIR measurements of heated hematite samples



Hematite to magnetite transition detected by pressure increase

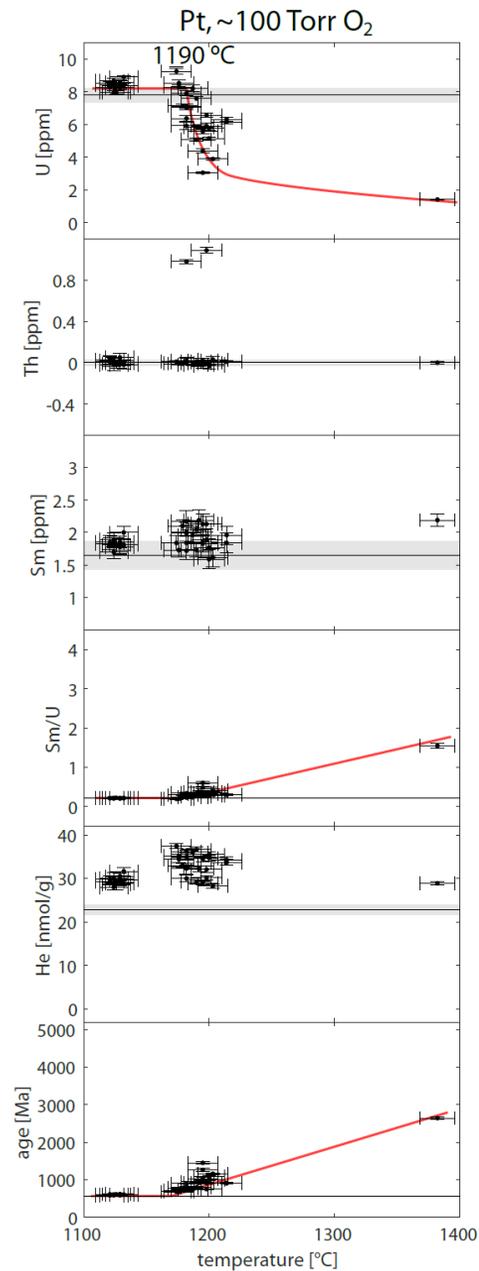
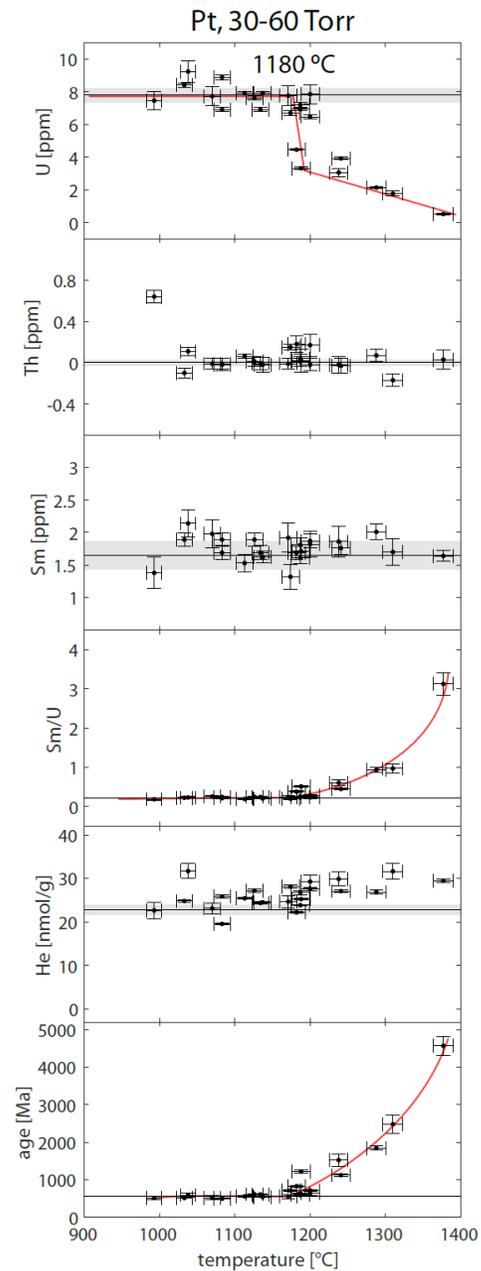
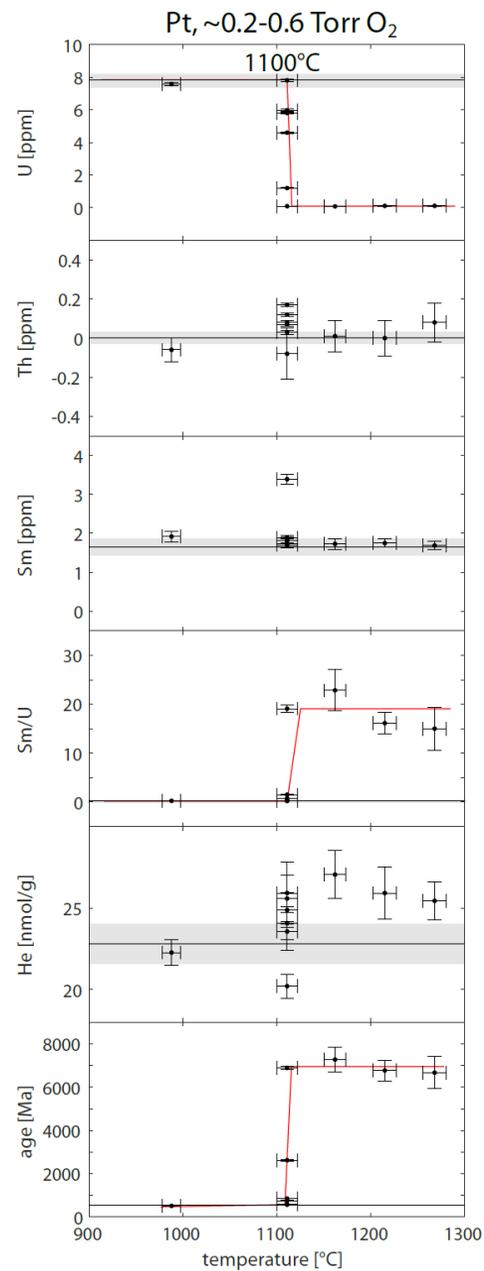
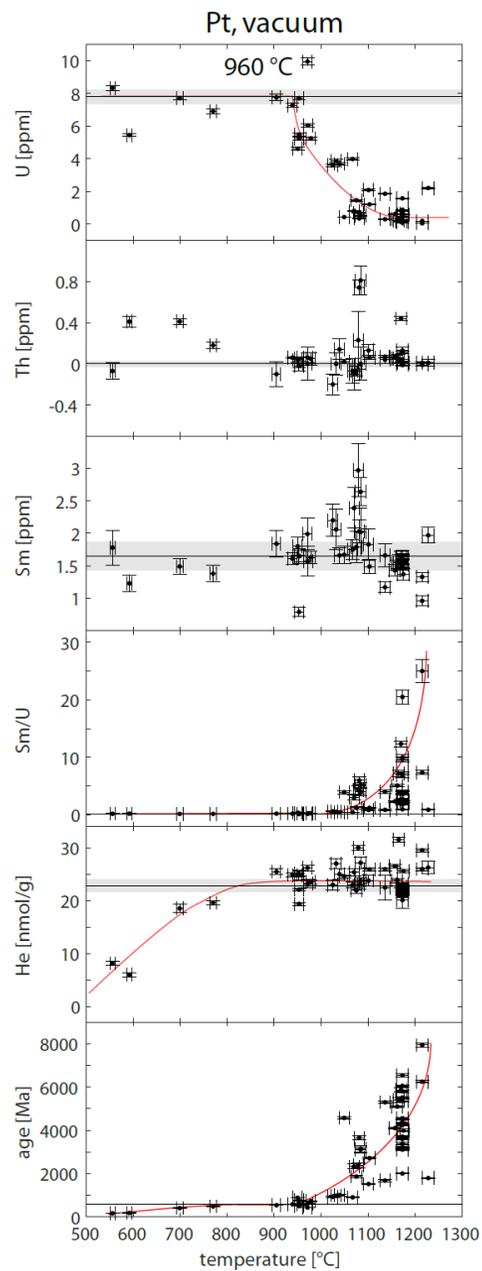


Delaying phase change (and U-loss?) to higher temperatures



Phase diagram after Ketteler et al. (2001)

Effect of increasing pO₂ on U-loss temperature

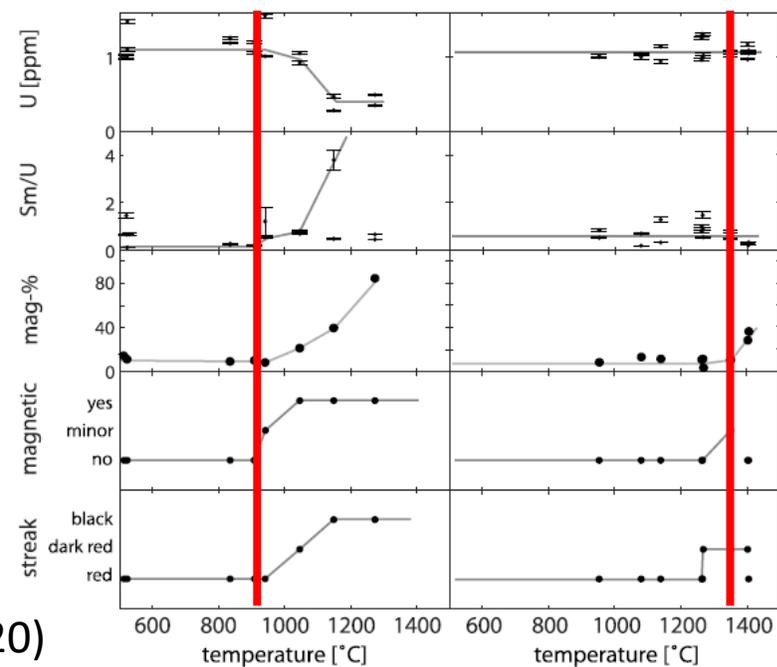
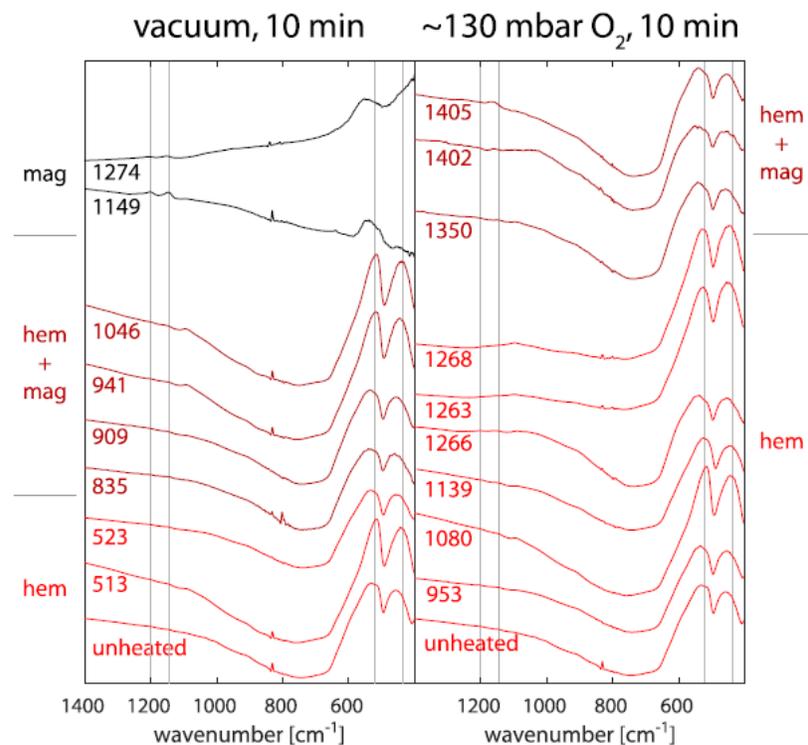
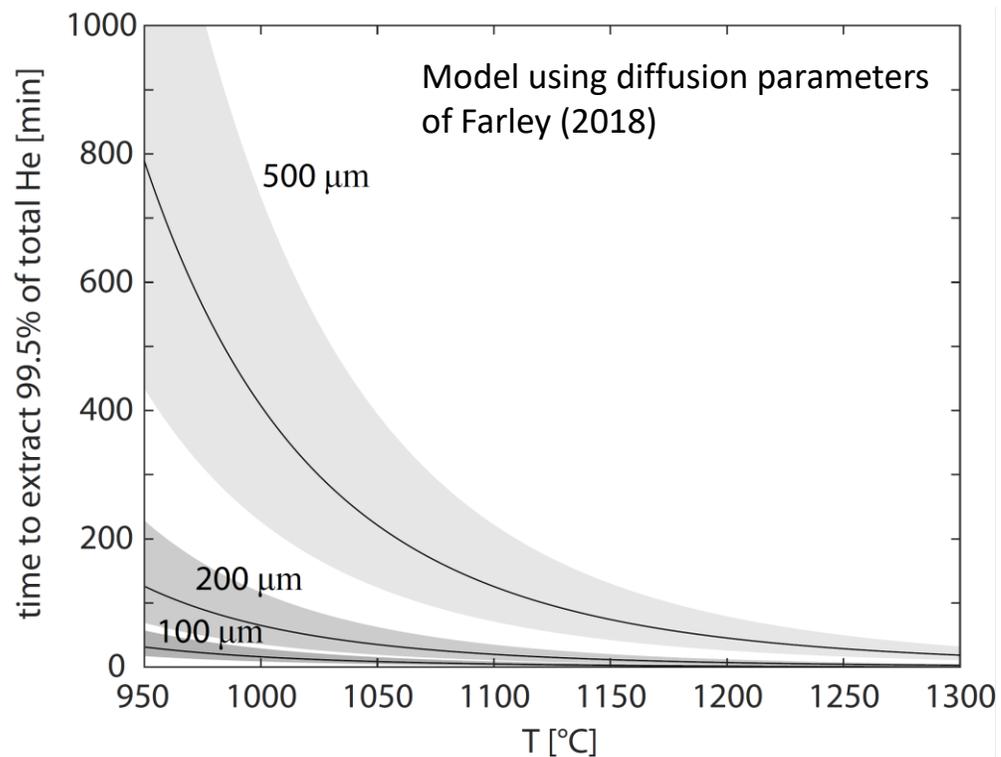


Correlation of U-loss and phase change

Delay of phase change/U-loss:

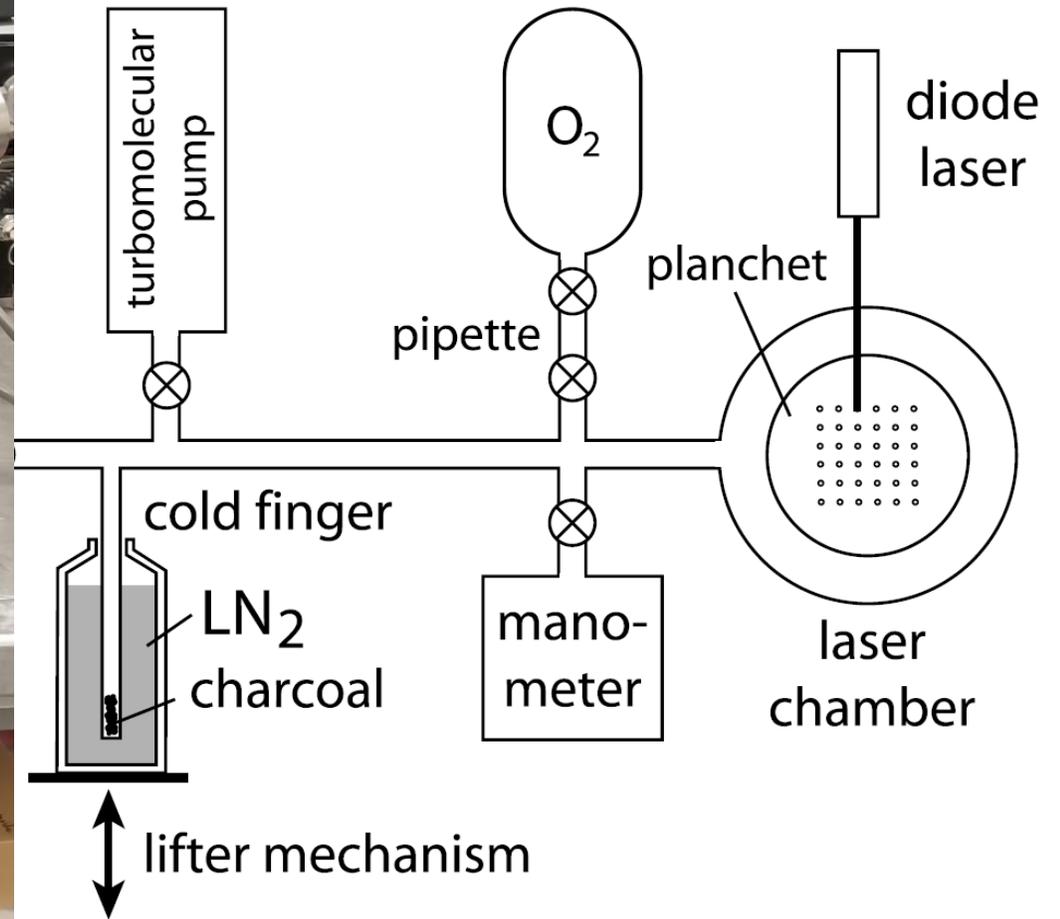
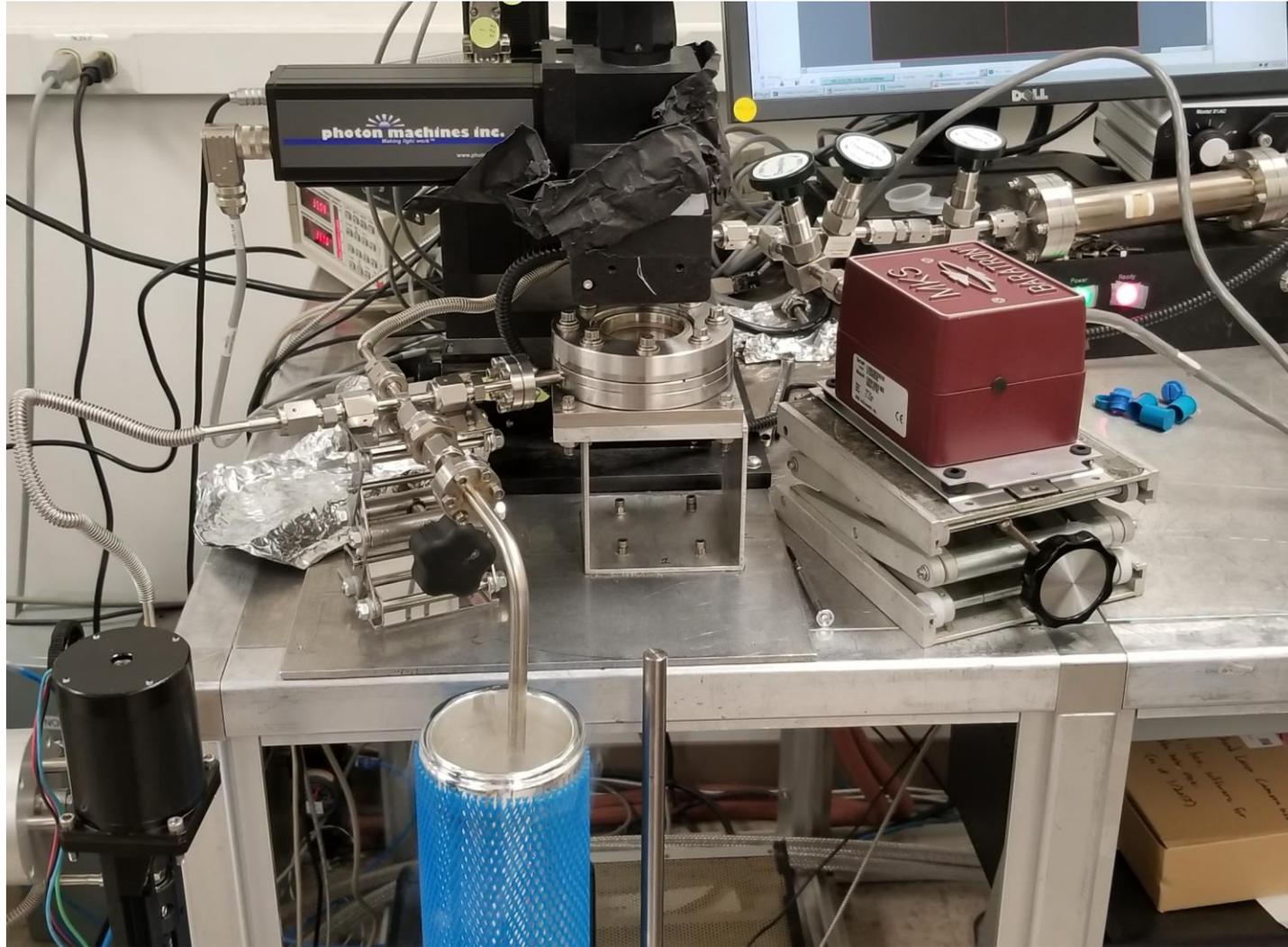
~980 °C → 1200-1300 °C

with ~100 Torr (130 mbar) O₂

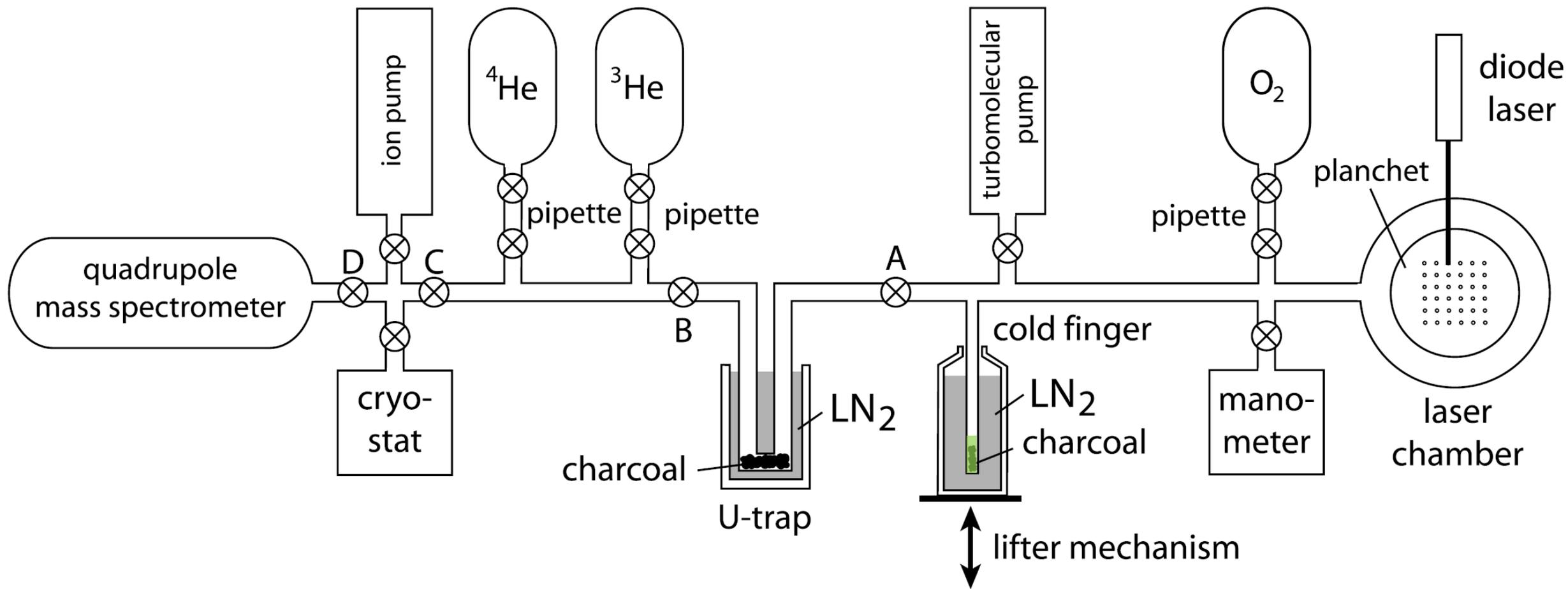


Hofmann et al. (2020)

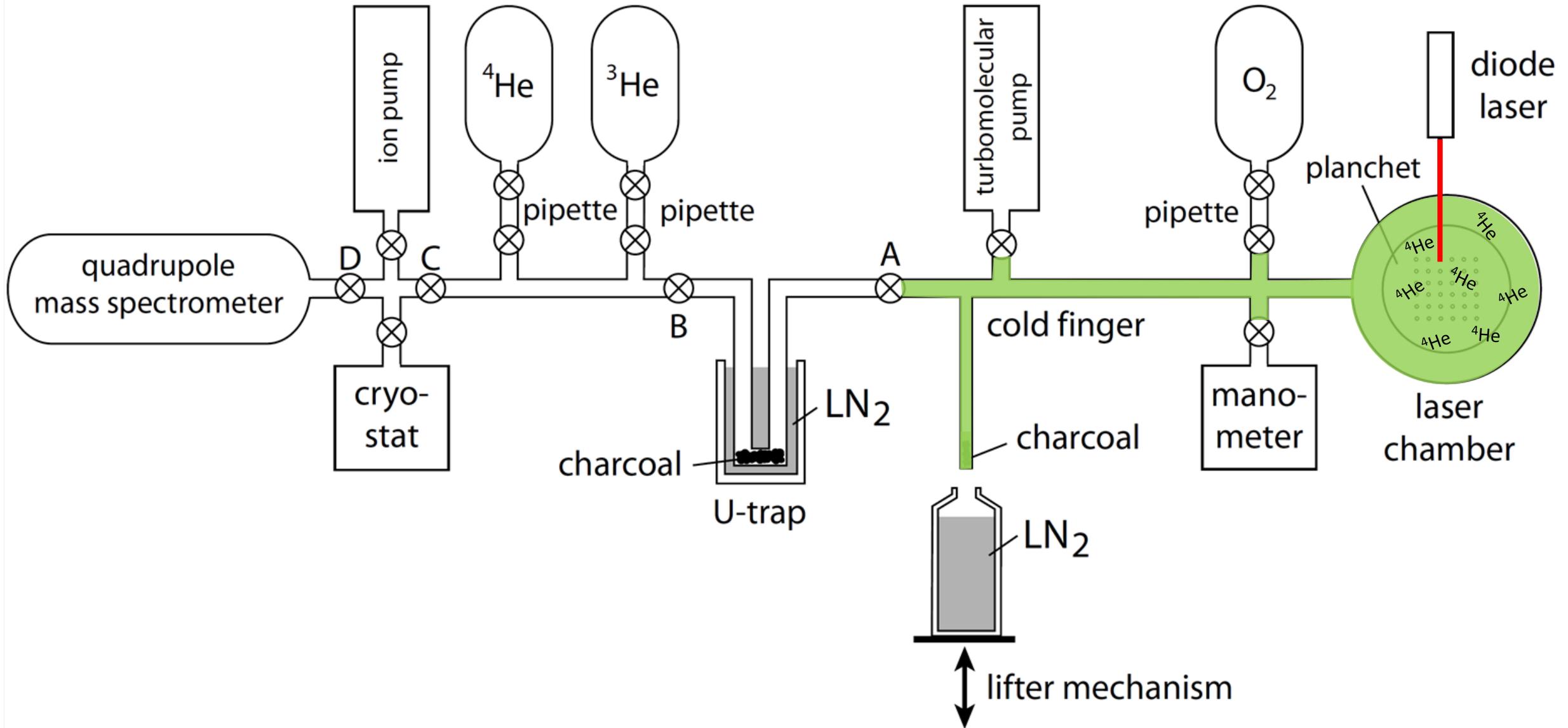
Modifications to the quad line to automate this method



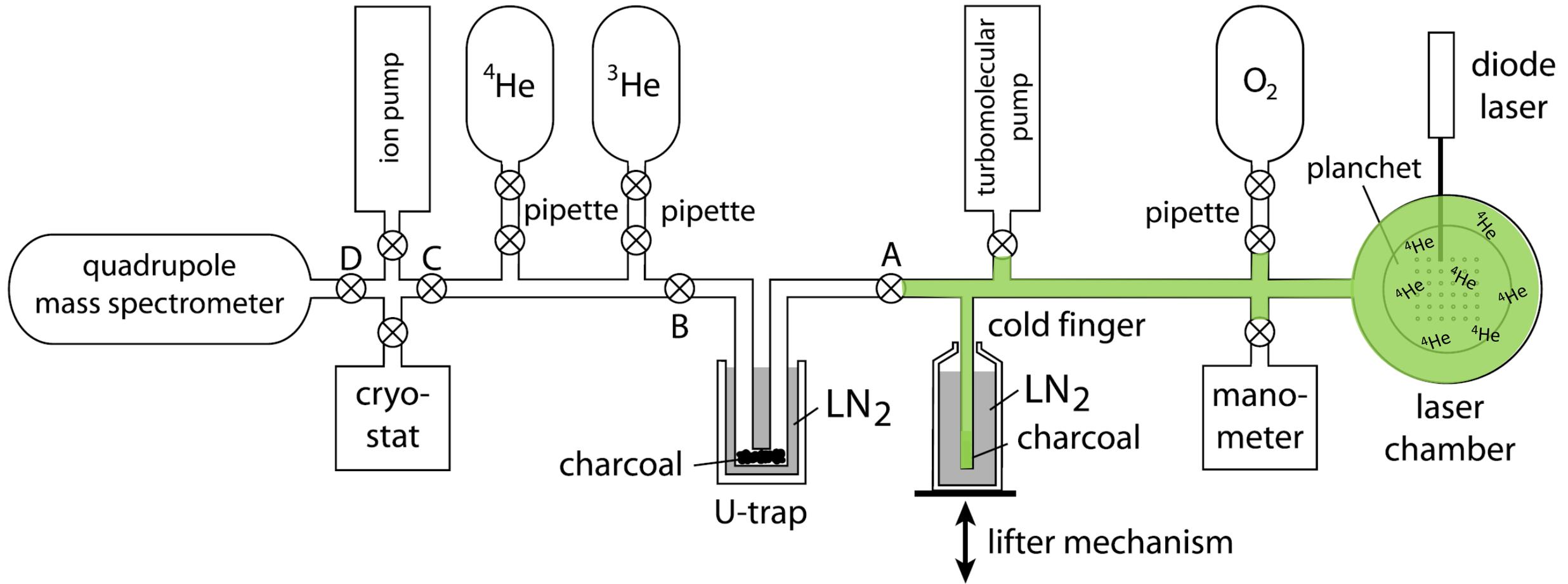
High-pO₂ degassing procedure



High-pO₂ degassing procedure

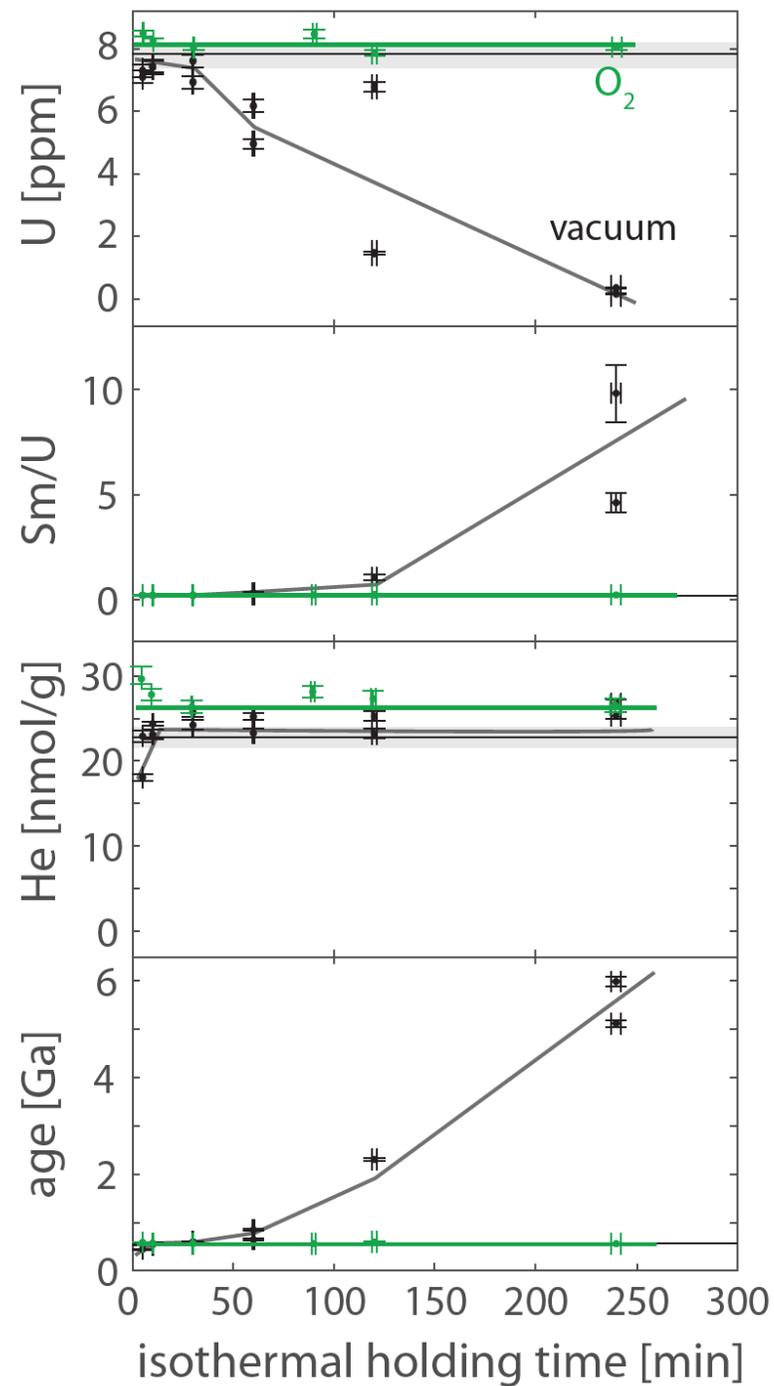


High-pO₂ degassing procedure

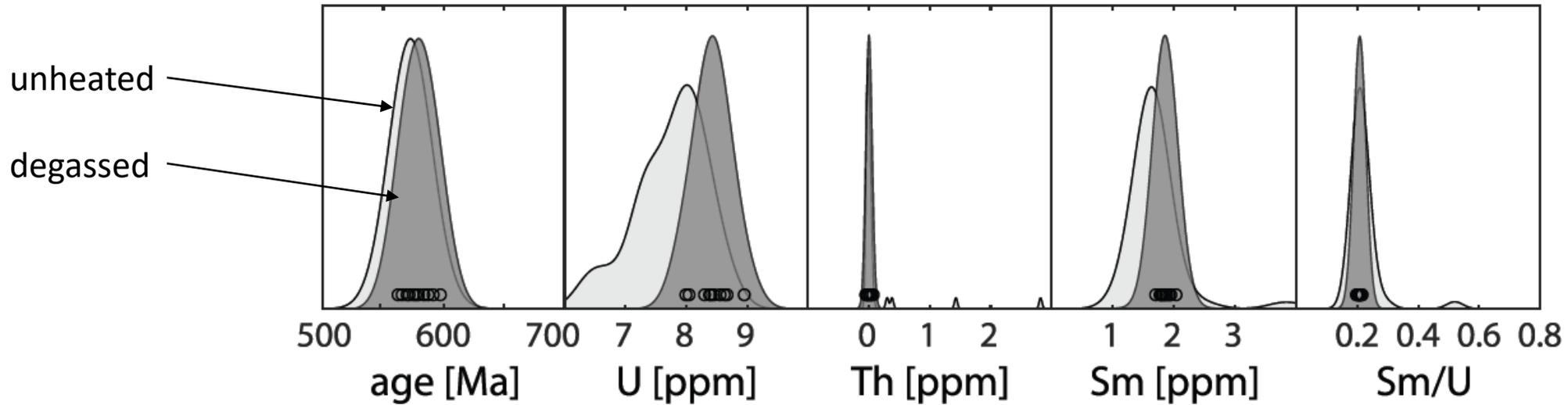
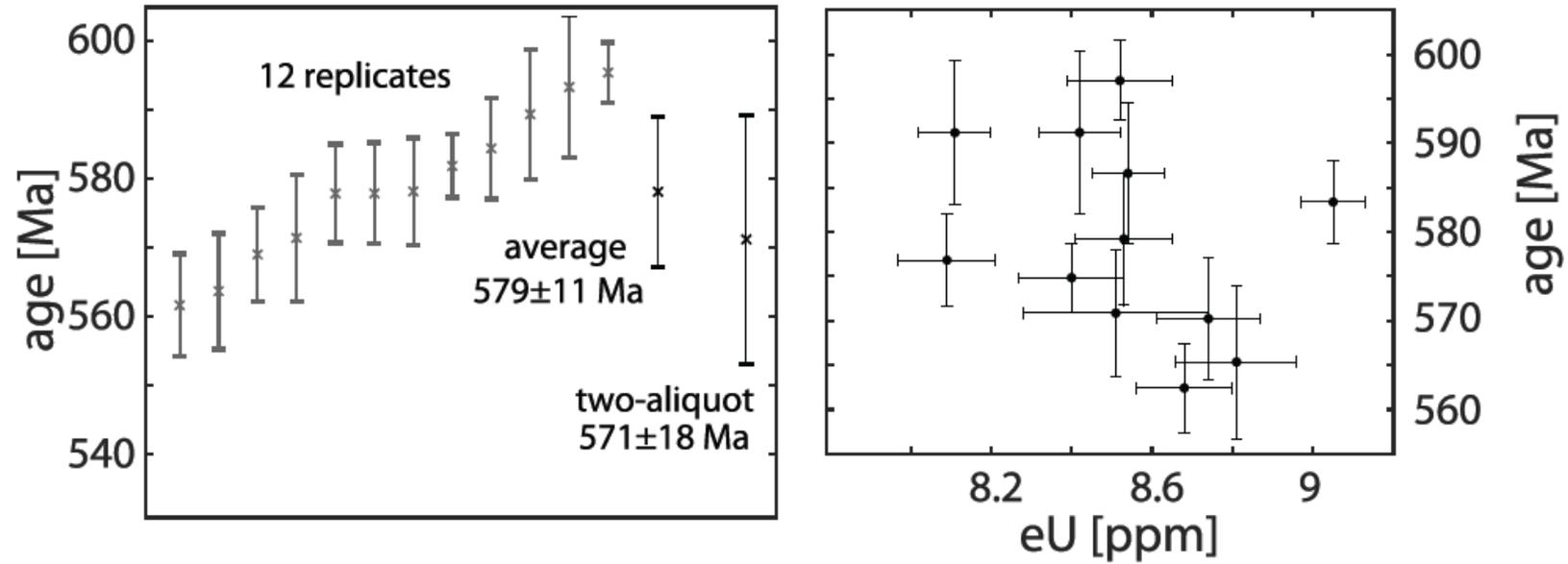


Isothermal holding at 1000 °C in vacuum and O₂

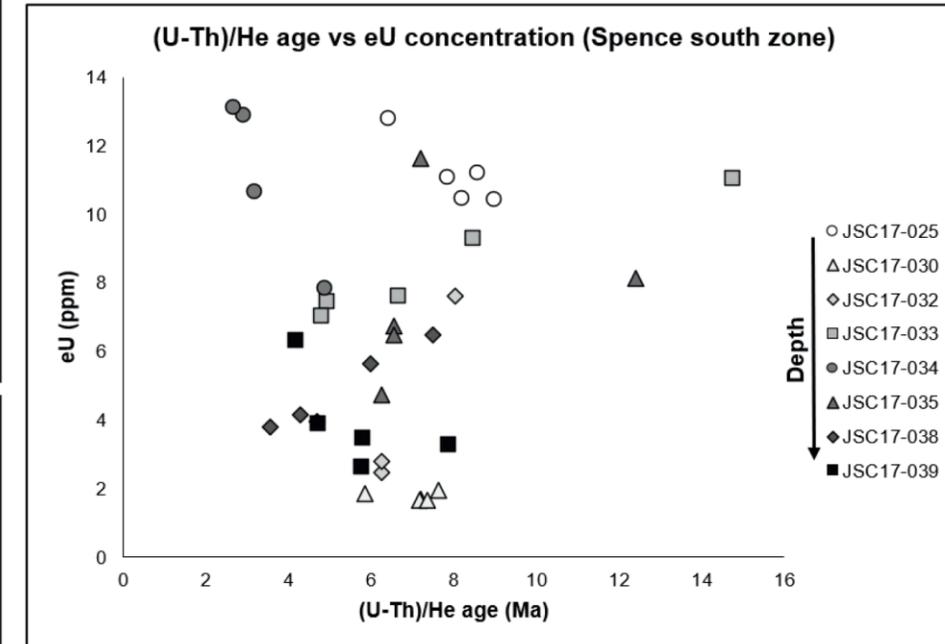
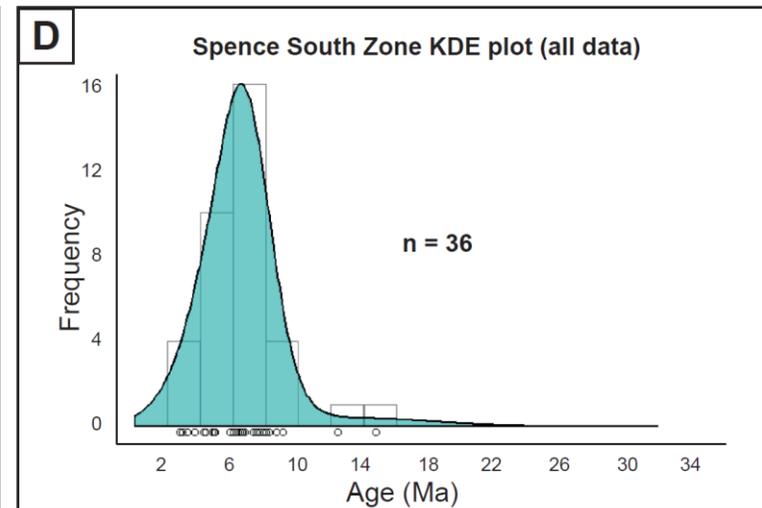
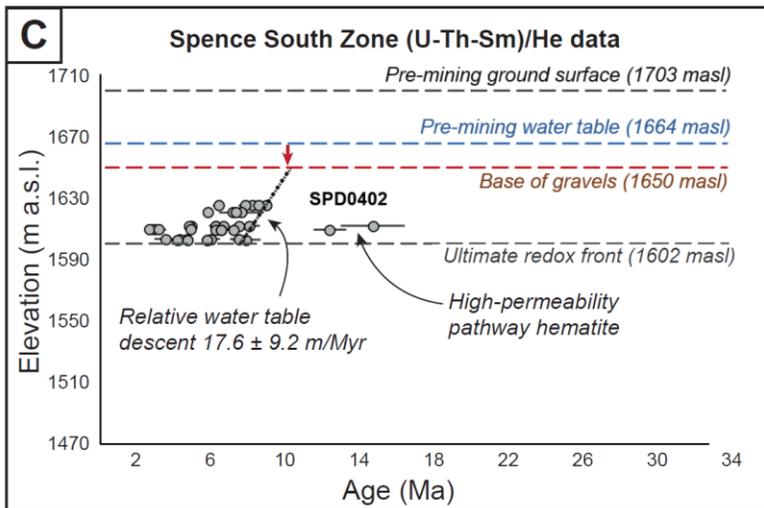
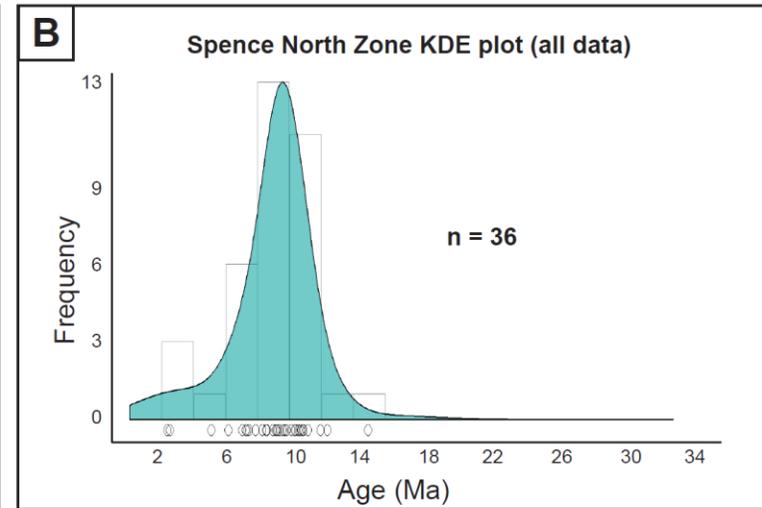
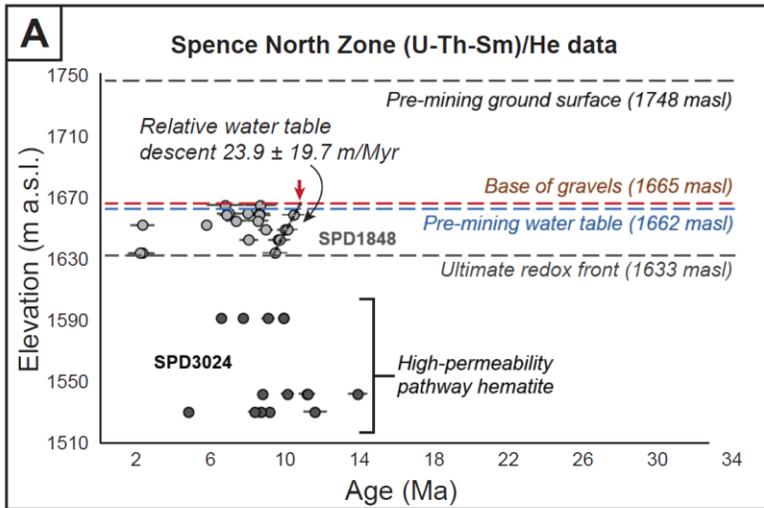
- Loss of U in vacuum increases with holding time
- No detectable loss of U with high-pO₂ at 4 h of holding



Analysis of highly-retentive hematite sample using high-pO₂ method

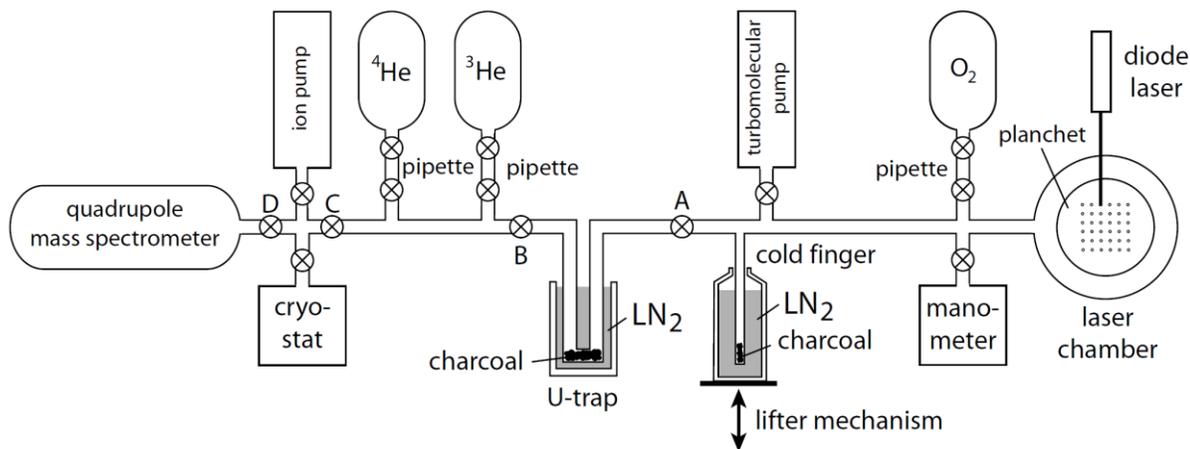


Recent paper on weathering and groundwater in the Andes using this method



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 O_2 partial pressure

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