Supplement of Geochronology, 8, 165–189, 2026
https://doi.org/10.5194/gchron-8-165-2026-supplement

Supplement of

Analytical and modelling strategies for thermal histories from in situ (U-Th-Sm) / He data of single apatites

Ann-Kathrin Maier et al.

Correspondence to: Ann-Kathrin Maier (ann-kathrin.maier@helsinki.fi)

Supplementary Material

S1: Technical details for (U-Th-Sm)/He dating at the University of Tübingen, Germany, laboratory

This Supplementary Material provides a detailed description of our ^{4}He measurement procedure. This includes the thermal effects of sample preparation, mass spectrometer calibration, sample loading, measurement sequence design, blank characterisation, and error propagation.

S1.1: Thermal effect of sample preparation

The apatite grains were embedded in Teflon on a hotplate set to 300 °C and kept there for 2 min. We can estimate the fractional helium loss (f) from the grains for a specific heating duration (t) and temperature (T) using Equation 3 from Reiners et al. (2007):

$$f \approx \left(\frac{6}{\pi^{\frac{3}{2}}}\right)\left(\pi^2 \frac{D}{a^2} t\right)^{\frac{1}{2}} - \left(\frac{3}{\pi^2}\right)\left(\pi^2 \frac{D}{a^2} t\right), \quad f \leq 0.85 \qquad \text{(Eq. S1)}$$

with Arrhenius-type diffusion (Equation 2 in Reiners et al., 2007):

$$\frac{D}{a^2} = \frac{D_0}{a^2} e^{-\frac{E_a}{RT}} \qquad \text{(Eq. S2)}$$

where D_0 is the diffusivity, E_a the activation energy, a the grain radius and R the gas constant.

To calculate the fractional helium loss, we use the diffusion parameters determined for the Durango apatite standard ($D_0 = 31.6\ cm^2 s^{-1}$, $E_a = 138\ kJ\ mol^{-1}$; Farley, 2000) and the radii of our analysed grains.

For Apatite-URG with a radius of 175 µm, the fractional helium loss from embedding is ~0.6%, for Apatite-BaF with a radius of 89 µm, the fractional helium loss is ~1.2% and for Apatite-McClure with a radius of 75 µm, the fractional helium loss is ~1.4%.

S1.2: In-situ (U-Th-Sm)/He Analytical Procedure

1. Mass Spectrometer Calibration

Quantification of radiogenic ^{4}He extracted from individual laser pits relies on calibration against an internally monitored ^{4}He reference reservoir ("Q-tank"). The ^{4}He volume of the Q-

tank is known but not constant: it undergoes predictable exponential depletion as Q-shots are consumed during routine analytical work. To maintain accuracy, the Q-tank is recalibrated at regular intervals, typically annually or after several thousand Q-shots, using a secondary reference reservoir ("D-tank"). The D-tank contains an independently certified amount of ^{4}He, externally calibrated by Des Patterson, and serves as the long-term stability anchor for all Q-tank recalibrations. Calibration factors derived from these intercomparisons are used to convert measured ion currents into absolute ^{4}He amounts in sample analyses.

2. Sample Loading and Preparation

Each sample mount is individually placed in an ultra-high-vacuum (UHV) laser cell and evacuated for a minimum of 24 hours to ensure that vacuum levels reach the low-pressure regime required for high-precision He analysis. In fact, our cold cathode was unable to measure the pressure since it reached values below the detection limit 1E-8 torr. Before analysing samples, several initial blank analyses are run until blank ^{4}He/^{3}He ratios reached values <0.0001. These blanks use the identical valve and volume configuration as the subsequent sample measurement, but without firing the laser.

3. Measurement Sequence Design

Because Q-shots yield extremely stable ^{4}He/^{3}He ratios (<1% variability) and analytical sequences are comparatively short (typically 20–40 analyses), each sequence begins and ends with two Q-shots. No Q-shots are included between samples to avoid flushing the line with high amounts of ^{4}He and potential memory effects. The ^{4}He content of Q-shots is 3–4 orders of magnitude higher than the He released during in-situ laser extraction. For this reason, the initial Q-shots are followed by 3–4 blank measurements to allow the system to return to pre-Q-shot blank levels. Sample measurements only begin once these blank values stabilize back to the initial baseline.

Blanks are measured frequently throughout the sequence. For the first three samples, a blank is run immediately before each measurement. As the system stabilizes, the number of sample analyses between blanks is gradually increased (from one, to two, three, four, and finally up to five), while maintaining sufficient blank coverage for accurate correction.

4. Blank Characterization and Correction

For each blank and sample measurement, ^{4}He/^{3}He ratios are calculated. During the Apatite-URG and Apatite-BaF analytical sessions, mean blank ratios and standard deviations were:

- 0.0000677±0.0000034 (Apatite-URG), and
- 0.0000700±0.0000016 (Apatite-BaF).

With a Q-standard ^{4}He content of 10.69 ncc for these sessions, these ratios correspond to mean blank ^{4}He amounts of approximately:

- 0.000700±0.000046 ncc (Apatite-URG), and
- 0.000757±0.000048 ncc (Apatite-BaF).

Blank correction for each sample is performed by subtracting the pre-sample blank ^{4}He/^{3}He ratio from the sample's measured ratio. This ensures that sample-derived radiogenic ^{4}He is isolated from procedural background contributions, and that time-dependent drift in blank characteristics does not introduce bias into the final (U-Th-Sm)/He age calculations.

5. Helium Error Propagation

All measured masses are measured during 10 individual cycles (i) that last in total roughly 50 sec, and the ratio (R43) between masses 4 (M4) and 3 (M3) of each run is calculated with:

$$R43_i = \frac{M4_i}{M3_i} \quad \text{(Eq. S3)}$$

The mean and absolute standard deviation (SD) of the R43 measurement is calculated with:

$$\overline{R43} = \frac{1}{N}\sum_{i=1}^{N} R43_i \quad \text{(Eq. S4)}$$

$$SD_{R43} = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(R43_i - \overline{R43})^2} \quad \text{(Eq. S5)}$$

The ^{4}He content of Q-shot j (number of Q-shot) is derived from the depletion factor (DF = 0.999899832 with SD = 4.26343E-6) and the ^{4}He content (11.098 ncc with SD = 0.011 ncc) determined for Q-shot 1000 with the following equation:

$$^4He_{Q_j} = {}^4He_{Q_{442}} DF^{(j-442)} \quad \text{(Eq. S6)}$$

Accordingly, the absolute standard deviation is:

$$SD\,{}^4He_{Q_j} = {}^4He_{Q_i}\sqrt{\left(\frac{SD\,{}^4He_{Q_{442}}}{{}^4He_{Q_{442}}}\right)^2 + \left((j-1000)\frac{SD_{DF}}{DF}\right)^2} \quad \text{(Eq. S7)}$$

To calculate the ^{4}He content of a sample, we must know the ^{3}He content added to the sample. This is estimated based on the ^{4}He of nearby Q-shots ($^4He_{Qj}$). Accounting for a drift during measuring time, we fit a spline-function to $^4He_{Qj}$. We used a Monte-Carlo approach to take into

account the uncertainty of the $^4He_{Qj}$, and randomly sampled N-times $^4He_{Qj}$ data from a normal distribution (using the mean $^4He_{Qj}$ and corresponding standard deviations). Afterwards, we evaluate the N-times fitted functions at the measuring time of our samples and the resulting $^4He_{Qt}$ and SD at measuring time are:

$$^4He_{Qt} = \frac{1}{N}\sum_{i=1}^{N} f_i(t) \quad \text{(Eq. S8)}$$

$$SD_{\,^4He_{Q_t}} = {}^4He_{Qt}\sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(f_i(t) - {}^4He_{Qt})^2} \quad \text{(Eq. S9)}$$

Blank ratios R_{bl} are calculated with Eq. S3 and used to calculate blank-correct R43 sample measurements (R_{Sbc}) with:

$$R_{Sbc} = \frac{1}{N}\sum_{i=1}^{N}(R_i - R_{bl_i}) \quad \text{(Eq. S10)}$$

Accordingly, the absolute standard deviation is:

$$SD_{R_{Sbc}} = R_{Sbc}\sqrt{\left(\frac{SD_{RS}}{R_S}\right)^2 + \left(\frac{SD_{Rbl}}{R_{bl}}\right)^2} \quad \text{(Eq. S11)}$$

where the sample and blank ratios and standard deviations have been calculated with Eqs. S4 and S5. Finally, the ^{4}He content of a sample at time t is calculated with:

$$^4He_{Sbc_t} = R_{Sbc}\frac{^4He_{Q_t}}{R_{Q_t}} \quad \text{(Eq. S12)}$$

Accordingly, the absolute standard deviation is:

$$SD_{\,^4He_{Sbc_t}} = {}^4He_{Sbc_t}\sqrt{\left(\frac{SD_{R_{Sbc}}}{R_{Sbc}}\right)^2 + \left(\frac{SD_{\,^4He_{Q_t}}}{^4He_{Q_t}}\right)^2 + \left(\frac{SD_{R_{Q_t}}}{R_{Q_t}}\right)^2} \quad \text{(Eq. S13)}$$

6. 4He concentration and uncertainty:

We divide the ^{4}He content ($^4He_{Sbc_t}$) by the ablation pit volume (V_{Pit}) to obtain the ^{4}He concentration (C_{He}). Finally, the pit volume uncertainty (SD_{VPit}) is propagated into the total measurement uncertainty for the ^{4}He concentration (SD_{CHe}) as follows:

$$SD_{CHe} = C_{He}\sqrt{\left(\frac{SD\ {}^{4}He_{Sbc_t}}{{}^{4}He_{Sbc_t}}\right)^2 + \left(\frac{SD_{VPit}}{V_{Pit}}\right)^2} \quad \text{(Eq. S14)}$$

S1.3: AHe age calculation and uncertainties

To calculate the AHe age and uncertainty we use the equations for the non-iterative solution to the age equation by Meesters & Dunai (2005).

References

Farley, K. A. (2000). Helium diffusion from apatite: General behavior as illustrated by Durango fluorapatite. *Journal of Geophysical Research: Solid Earth*, *105*(B2), 2903–2914. https://doi.org/10.1029/1999jb900348

Meesters, A. G. C. A., and T. J. Dunai (2005), A noniterative solution of the (U-Th)/He age equation, *Geochem. Geophys. Geosyst.*, 6, Q04002, doi:10.1029/2004GC000834.

Reiners, P. W., Thomson, S. N., Mcphillips, D., Donelick, R. A., & Roering, J. J. (2007). Wildfire thermochronology and the fate and transport of apatite in hillslope and fluvial environments. 112. https://doi.org/10.1029/2007JF000759