

Supplemental Material:

A Case Study of Maximum Depositional Ages from Terrestrial Sandstones near the Cretaceous—Paleogene Transition, Western Williston Basin, USA

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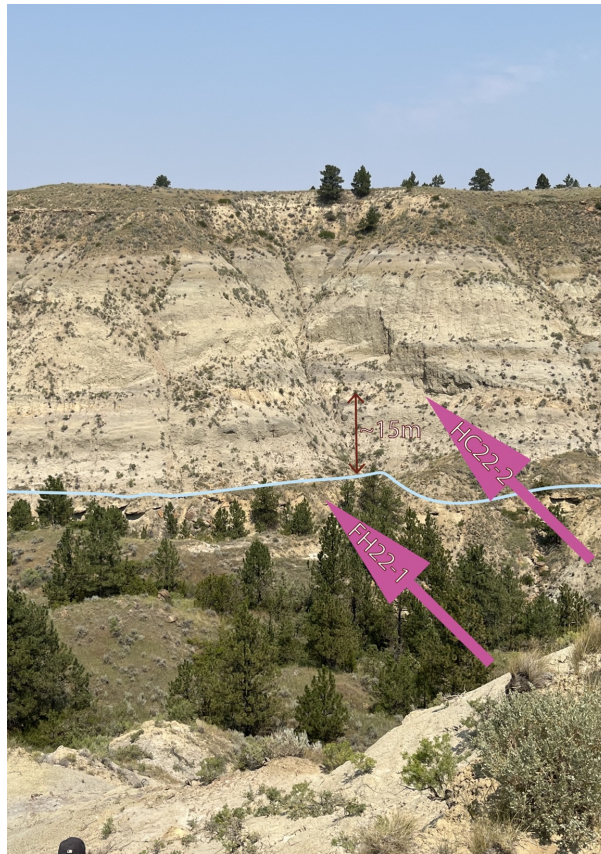


Figure S1. Northerly view of the outcrop spanning the Fox Hills and Hell Creek Formations. Formational contact approximately delineated by the light blue line. Pink arrows show the stratigraphic location of samples FH22-1 and HC22-2. The formational contact was identified by the presence of a fine-grained, relatively resistant, cross-bedded calcareous sandstone at the base of the Hell Creek Formation at this location. This basal lithology was collected as a sample but upon processing did not result in K-feldspar grains large enough to be analyzed reliably. Sample FH22-1 comes from a meter-scale yellowish sand directly underlying the calcareous cross bedded sandstone.

Table S1. Sample Latitudes, Longitudes, and Formations.

Sample	Latitude	Longitude	Formation
BB18-1	47.486217°	−107.067100°	Fort Union
TR21-1	47.666304°	−106.425057°	Hell Creek
HC18-1	47.533339°	−106.934235°	Hell Creek
HC22-2	47.582127°	−106.942529°	Hell Creek
FH22-1	47.581778°	−106.942661°	Fox Hills

Sampling and Processing Procedures

During field sampling, root casts and the surficial ~10 cm of each sample were removed to avoid contamination resulting in ~5,000 cm³ of collected material. Roughly half of each sample was processed to isolate K-feldspar and zircons. Zircons were concentrated via panning and then hand-picked selecting the most euhedral, unabraded grains. The remaining sample aliquot was sieved, magnetically separated, and subject to heavy liquid treatment to concentrate the coarsest K-feldspars. Populations of K-feldspar crystals were then hand-picked to exclude altered grains.

Analytical procedures ⁴⁰Ar/³⁹Ar

K-feldspar samples were co-irradiated in Al disks along with Fish Canyon sanidine fluence monitors in three separate 50 h irradiations in the Cadmium-Lined In-Core Irradiation Tube facility of the Oregon State University TRIGA reactor. All single grain K-feldspar analyses underwent total fusion at 7 W by a CO₂ laser and were analyzed with an Mass Analyzer Products Ltd. (United Kingdom) 215C mass spectrometer at the Berkeley Geochronology Center. Fish Canyon sanidine was used as a fluence monitor with an age of 28.183 Ma and a total decay constant of 5.5042×10^{-10} year⁻¹. Neutron fluence for each sample was determined by planar interpolations between three bracketing standard positions. System backgrounds and pipettes of atmospheric Ar were measured throughout the analytical sessions using the same protocols used to measure standards and unknowns. Data were reduced using MassSpec software v8.229.

Analytical procedures LA-ICPMS

Age screening data for samples TR21-1, HC22-2, and FH22-1 were obtained using a Teledyne-Photon Machines Excite-Analyte (Belgrade, MT, United States) (TM) 193 nm excimer laser and a Nu Instruments Attom (North Whales, United Kingdom) (TM) high-resolution sector field ICPMS at the USGS-Denver G3 Plasma Lab. Zircon grains were placed on sticky tape and laser focus was adjusted for each analysis to account for differences in grain size. Laser ablation spot sizes were 20 µm with a 5 Hz repetition rate and 150 bursts per analysis. Prior to analysis, the system was tuned using National Institute of Standards and Technology (NIST) 610 glass to achieve optimal signal intensity, peak shape, and Th/U ratio. Primary reference material Temora2 as well as secondary reference material Plesovice were analyzed during the run [1,2].

Zircons from samples BB18-1 and HC18-1 were analyzed using an Agilent quadrupole 7500ce at the UC Davis Interdisciplinary Center for Plasma Mass Spectrometry. The system was tuned using NIST 610 glass prior to analysis and primary reference material Plesovice and GJ1 were analyzed during the run [2].

Analytical procedures CA-TIMS

The youngest zircons identified through LA ICPMS were removed from the tape and thermally annealed at 900 °C for 48 h. Zircons were then chemically abraded with concentrated HF in pressurized dissolution capsules at 220 °C for 8 to 12 h. Prior to dissolution the crystals were placed in ultrasonically agitated aqua regia followed by multiple steps of rinsing in clean HNO₃. Zircon were then dissolved by vapor transfer in HF using miniature PTFE capsules at 220 °C for 6 days after being spiked with the EarthTime 535 (ET 535) ²⁰⁵Pb-²³³U-²³⁵U tracer solution. Isotope ratios were determined on a Micromass (United Kingdom) Sector 54 mass spectrometer using a Daly type ion counter

positioned behind a WARP filter with Pb (as Pb^+) and U (as UO_2^+) run sequentially on the same filament. Additional details regarding CA-TIMS analyses are in the Data Repository.

The aim of the model is to determine the Maximum Depositional Age (MDA) from a set of $^{40}\text{Ar}/^{39}\text{Ar}$ single grain fusion analyses. The model approach is to (a) down-weight dubious grains using age and geochemical information (age uncertainty, K/Ca, and $\%^{40}\text{Ar}^*$), (b) build a weighted age prior from the data, and then (c) run a Metropolis-Hastings Markov Chain Monte Carlo (MCMC) that draws a latent age for each grain and records the minimum age and thus the MDA. Ultimately the model attempts to find the collection of youngest grains in a complex environment and return the best guess of the MDA.

1. Removal of outliers
 - a. Drop NaNs, sort data by Age from youngest to oldest
 - b. Force positivity and finiteness (e.g., ages must be greater than 0)
 - c. Force composition of radiogenic to be 0–100%
 - d. IQR Crop on the ages
 - i. Trims outliers from the upper end and keeps all young samples
2. Building the prior
 - a. K/Ca
 - i. Build Kernel Density Estimate (KDE) from the GEOROC database of K/Ca ratios for alkali feldspars
 - ii. For each analysis compute a K/Ca effective density by convolving the GEOROC KDE with the grains measured uncertainty
 - b. Age
 - i. Do same integral weight approach as K/Ca but using the measured uncertainty of the grain
 - c. Radiogenic ($\%^{40}\text{Ar}^*$)
 - i. The scale/steepness of a logistic curve is derived from each samples $\%^{40}\text{Ar}^*$ distribution using the median and interquartile range.
 1. Necessary step to account for the differences across a complete detrital set given the variability of grain mean ages and the age dependence on an ‘acceptable’ radiogenic value.
 - d. Combined weight is then derived by multiplying the weights:
 - i. K/Ca weights * Rad weights * Age weights
 - e. Build the weighted age KDE
 - i. Prior over the true ages
 - ii. Bandwidth determined by leave-one-out cross-validation (LOO-CV) – gives unique bandwidth per sample – reflects heteroscedasticity (- differences in uncertainty across grains) – cannot give same bandwidth to 1 Ma and 1 Ga sample
3. Likelihood
 - a. Likelihood is a normal distribution
 - b. $Y | \theta \sim N(\theta i, \sigma^2)$
 - c. Ties model ages (θ) to the observed value with its analytical uncertainty
4. MCMC
 - a. Samples latent model ages (θ) return MDA-draws
 - b. Model initial state is $\theta = (\theta_1 - \theta_N)$ for N ages
 - c. Perform random walk Metropolis sampling
 - i. Acceptance/rejection based on Metropolis-Hastings Algorithm
 - ii. Adaptation every 1000 steps in the burn to reach target acceptance rate of about 23.4%
 - iii. Runs 50,000 samples with 20,000 burn in
 - iv. Report MDA with 2σ uncertainty – assumes symmetrically of distribution - normally fine – see from plots Figure S6–S10 that this is reasonable in most cases.

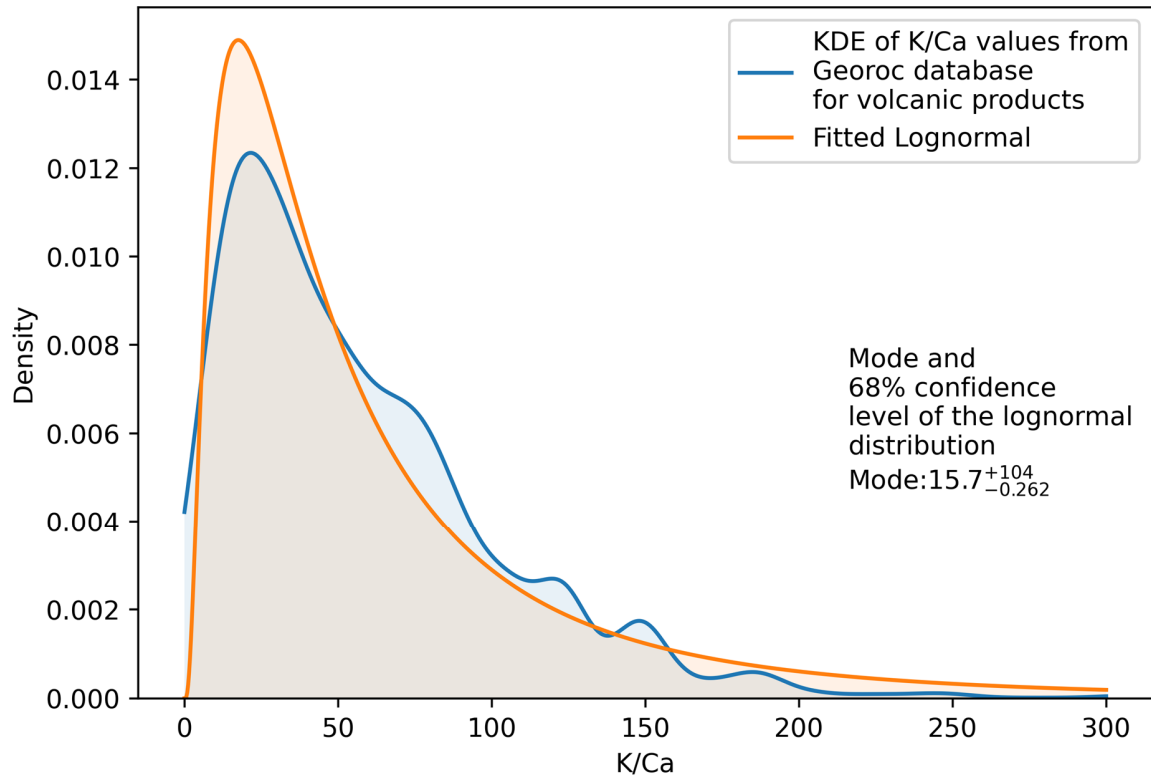


Figure S2. Solid blue curve represents the empirical kernel-density estimate (KDE) of K/Ca values from alkali feldspars compiled in the GEOROC database. Orange solid line corresponds to a fitted lognormal probability distribution to the GEOROC reported KDE. Inset text describes a mode and uncertainty of the “most probable” value for a volcanically derived K-feldspar. The model leverages the empirical KDE (blue curve) to construct K/Ca weights for each individual grain in each sample. Uncertainty in the K/Ca ratio is also accounted for with respect to the empirical KDE, thus determining an effective K/Ca weight that accounts for each analysis.

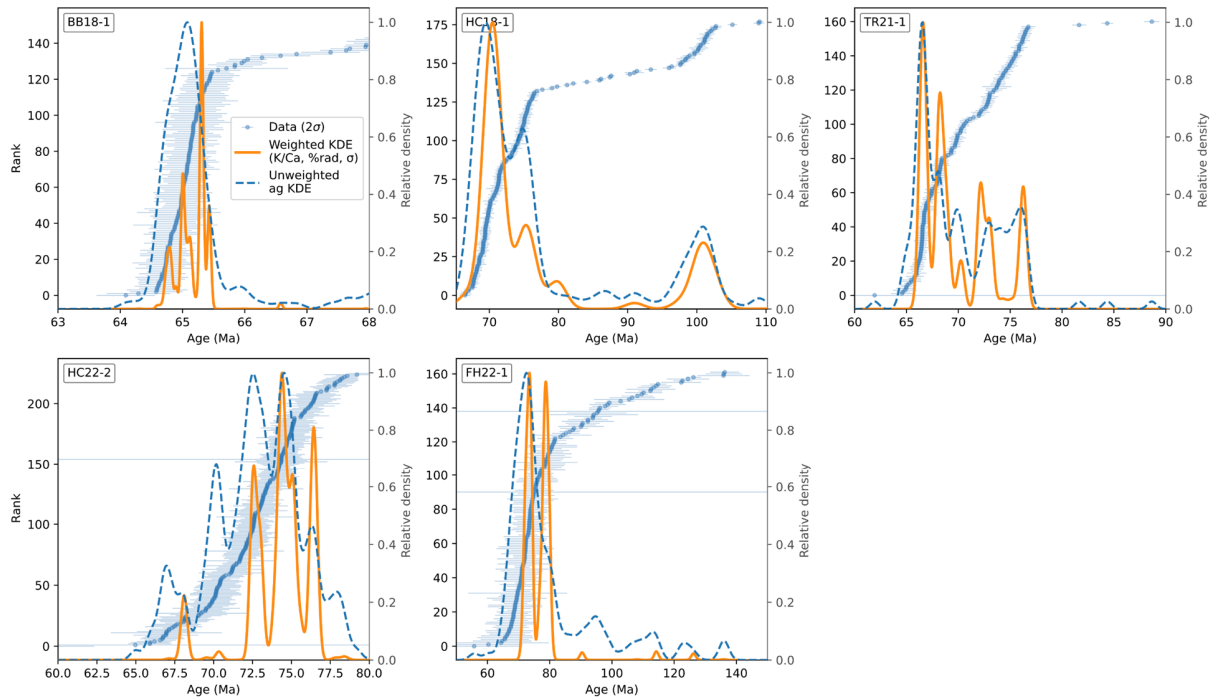


Figure S3. Rank order plot of sample ages in blue with horizontal bars representing $\pm 2\sigma$ analytical uncertainty. For each sample, the dashed blue curve is a KDE based solely on sample age, solid orange curve is the weighted age-density curve for each sample. Right-hand y-axis displays age probability densities. The weighted KDE (orange) peaks indicate likely volcanic modes in the distribution.

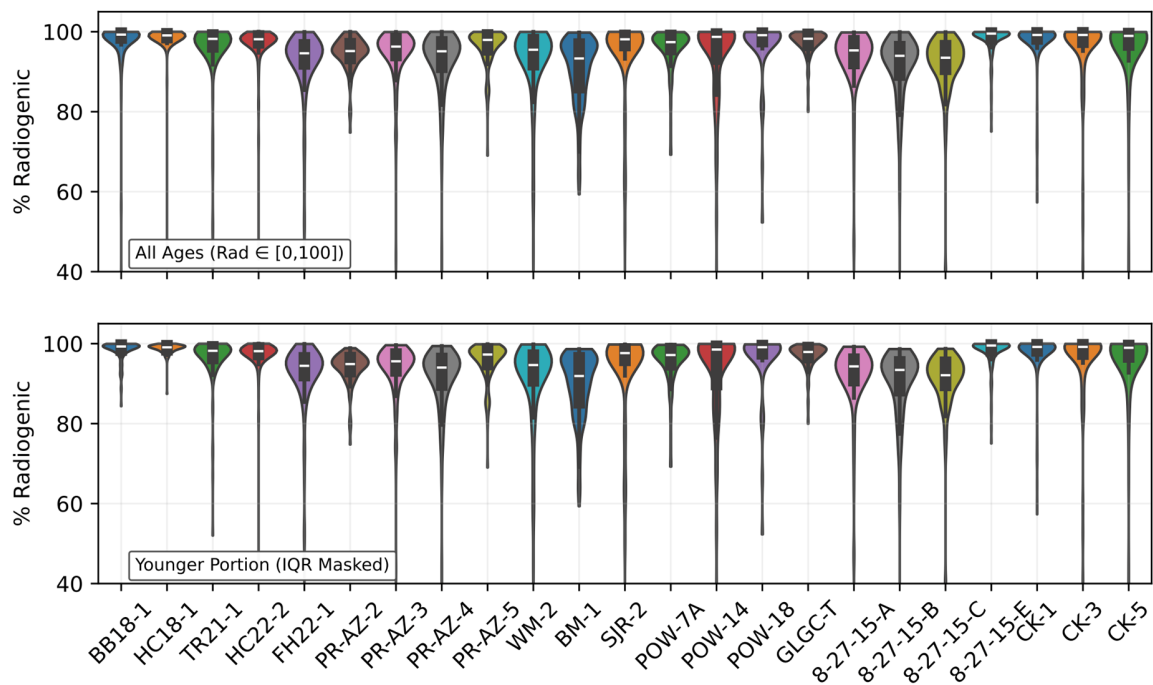


Figure S4. Violin plots depicting radiogenic profiles for each sample from this study (first five from left) and Heizler et al. (2021) (all other sample names) [3]. The inset median is represented by the white horizontal bar, and the 68%

and 95% confidence intervals are the thick and thin vertical black bars, respectively. Top panel shows the complete dataset with the imposed constraint that the % $^{40}\text{Ar}^*$ is between 0 and 100%. Bottom panel shows the youngest portions of the data up to the upper interquartile range mask. Similarity exists across all datasets exhibiting a higher probability of higher % $^{40}\text{Ar}^*$ with tails toward lower values.

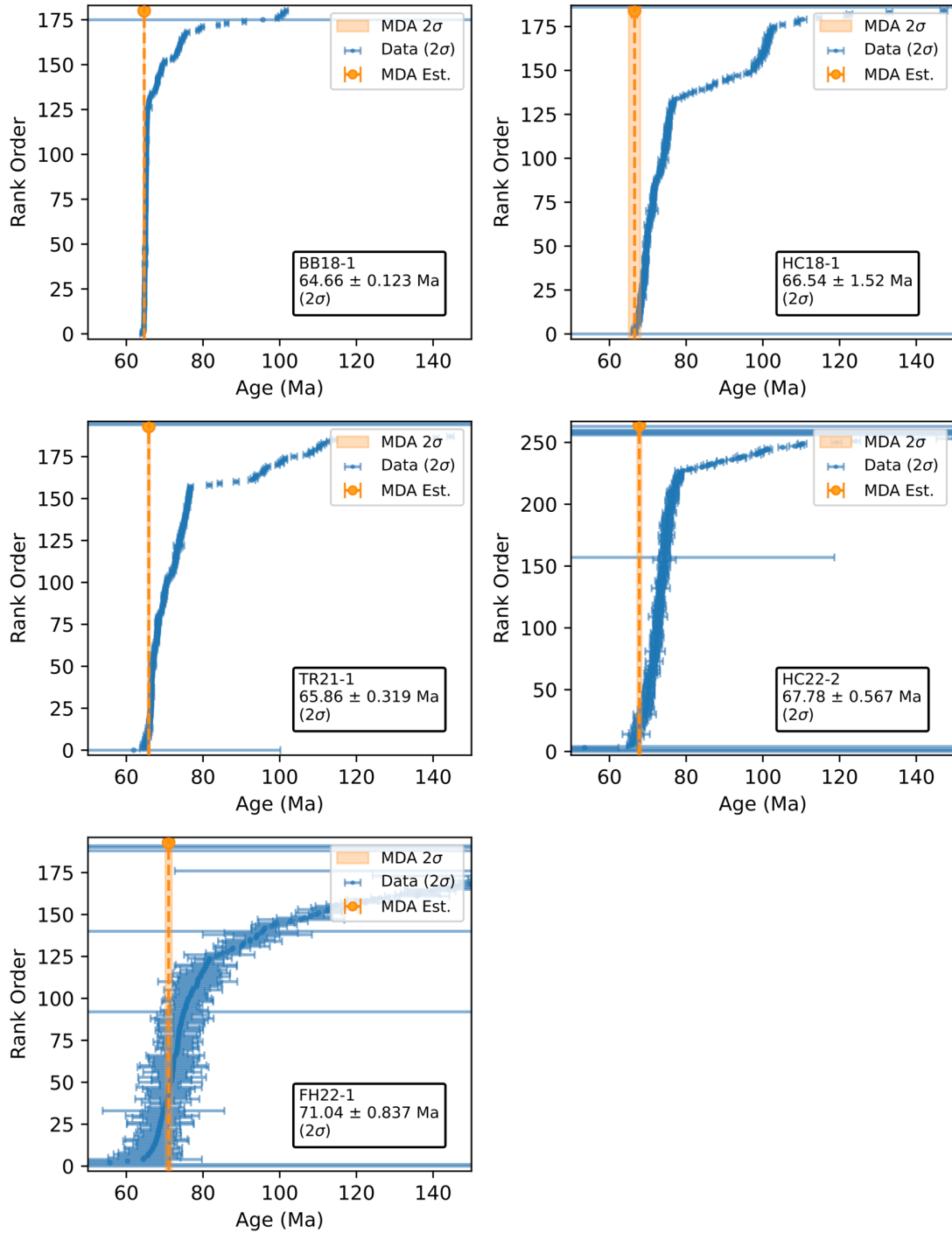


Figure S5. Rank order sample ages (blue) with 2σ analytical uncertainties, with orange vertical dashed line and orange shaded region representing the MDA and 2σ uncertainty. The inset magnifies the region surrounding the MDA (orange symbol) and

those grains falling within this window (blue symbols with black outlines) highlighting proximity to the MDA. Orange shaded region within inset shows a 2σ error envelope for the MDA.

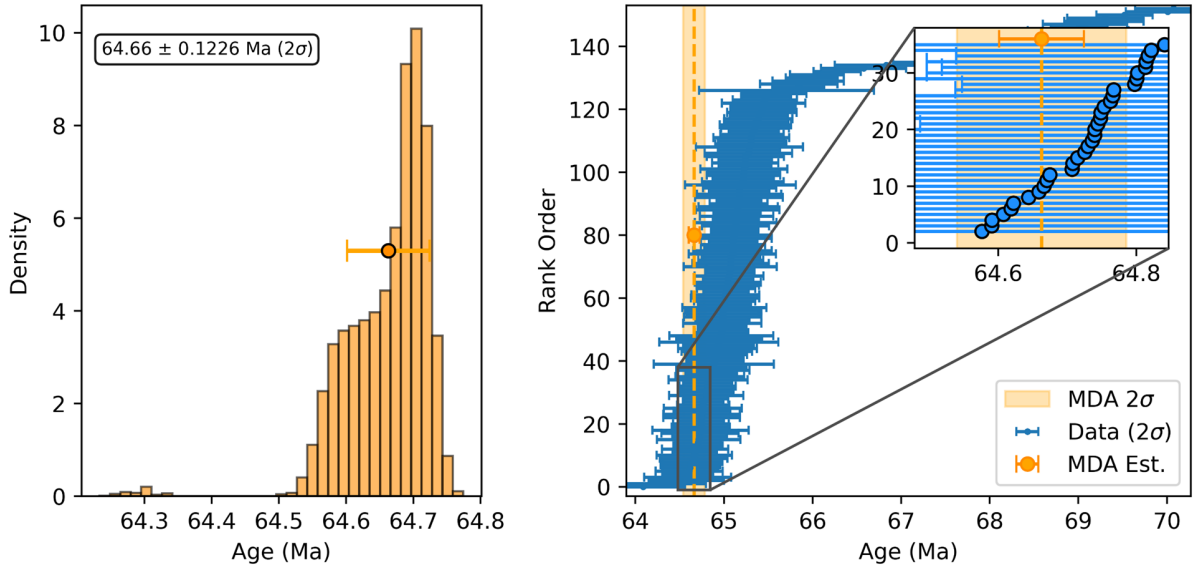


Figure S6. The orange histogram (left) shows the posterior distribution for all analyses post burn-in ($n = 50,000$) for sample BB18-1. Ranked age plot (right) shows individual $^{40}\text{Ar}/^{39}\text{Ar}$ ages and 2σ uncertainties in blue along with the Bayesian mean and 2σ uncertainty in orange as derived from the posterior distribution. The inset within the rank order plot (right) magnifies the region surrounding the MDA (orange symbol) and those grains falling within this window (blue symbols with black outlines) highlighting proximity to the MDA. Orange shaded region within inset shows a 2σ error envelope for the MDA. Connecting lines between the inset panel and main figure panel indicate the portion of the main panel that is magnified.

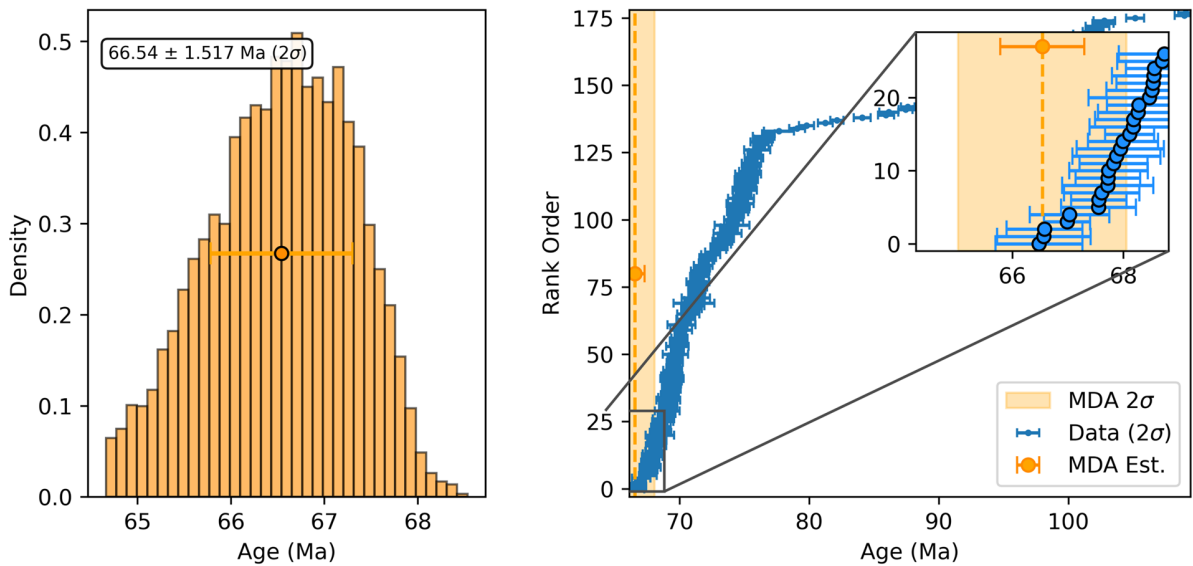


Figure S7. The orange histogram (left) shows the posterior distribution for all analyses post burn-in ($n = 50,000$) for sample HC18-1. Ranked age plot (right) shows individual $^{40}\text{Ar}/^{39}\text{Ar}$ ages and 2σ uncertainties in blue along with the Bayesian mean and 2σ uncertainty in orange as derived from the posterior distribution. The inset within the rank order plot (right) magnifies the region surrounding the MDA (orange symbol) and those grains falling within this window (blue symbols with black outlines) highlighting proximity to the MDA. Orange shaded region within inset shows a 2σ

error envelope for the MDA. Connecting lines between the inset panel and main figure panel indicate the portion of the main panel that is magnified.

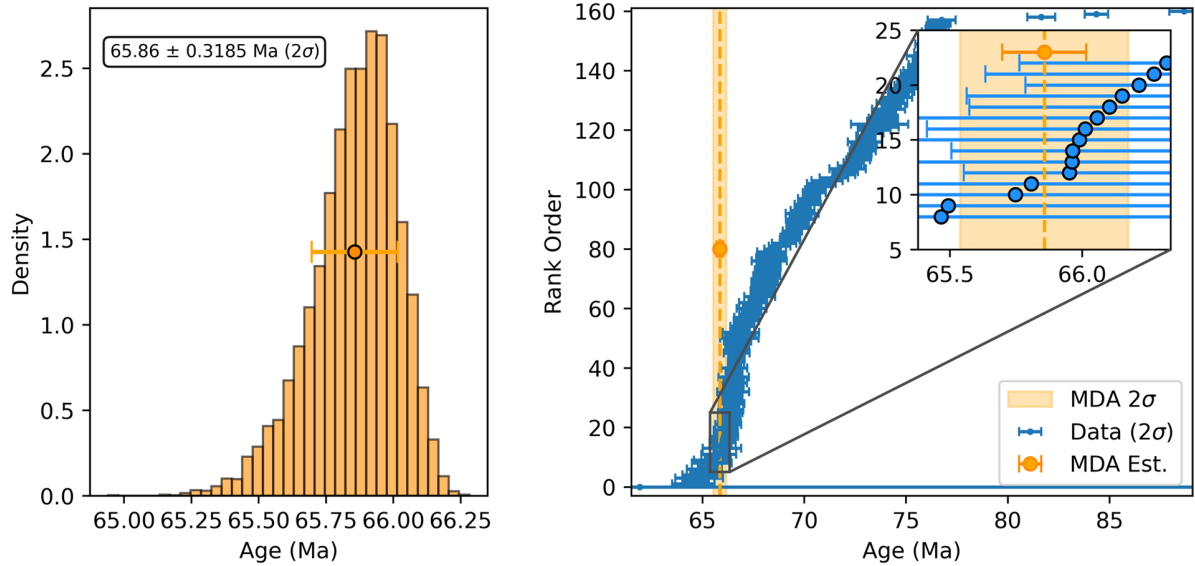


Figure S8. The orange histogram (left) shows the posterior distribution for all analyses post burn-in ($n = 50,000$) for sample TR21-1. Ranked age plot (right) shows individual $^{40}\text{Ar}/^{39}\text{Ar}$ ages and 2σ uncertainties in blue along with the Bayesian mean and 2σ uncertainty in orange as derived from the posterior distribution. The inset within the rank order plot (right) magnifies the region surrounding the MDA (orange symbol) and those grains falling within this window (blue symbols with black outlines) highlighting proximity to the MDA. Orange shaded region within inset shows a 2σ error envelope. Connecting lines between the inset panel and main figure panel indicate the portion of the main panel that is magnified.

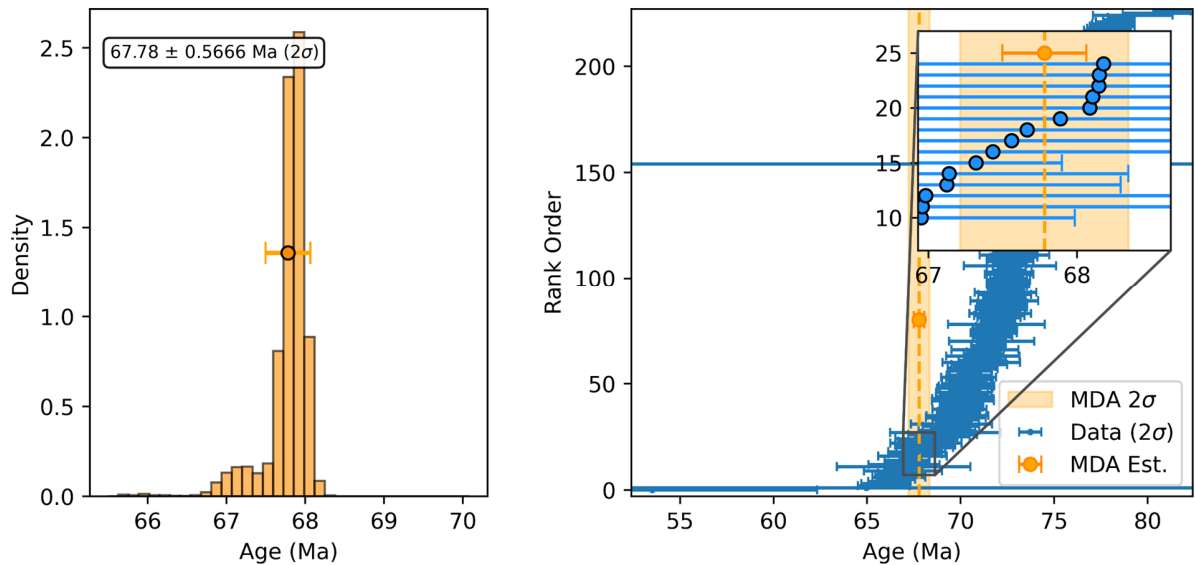


Figure S9. The orange histogram (left) shows the posterior distribution for all analyses post burn-in ($n = 50,000$) for sample HC22-2. Ranked age plot (right) shows individual $^{40}\text{Ar}/^{39}\text{Ar}$ ages and 2σ uncertainties in blue along with the Bayesian mean and 2σ uncertainty in orange as derived from the posterior distribution. The inset within the rank order

plot (right) magnifies the region surrounding the MDA (orange symbol) and those grains falling within this window (blue symbols with black outlines) highlighting proximity to the MDA. Orange shaded region within inset shows a 2σ error envelope. Connecting lines between the inset panel and main figure panel indicate the portion of the main panel that is magnified.

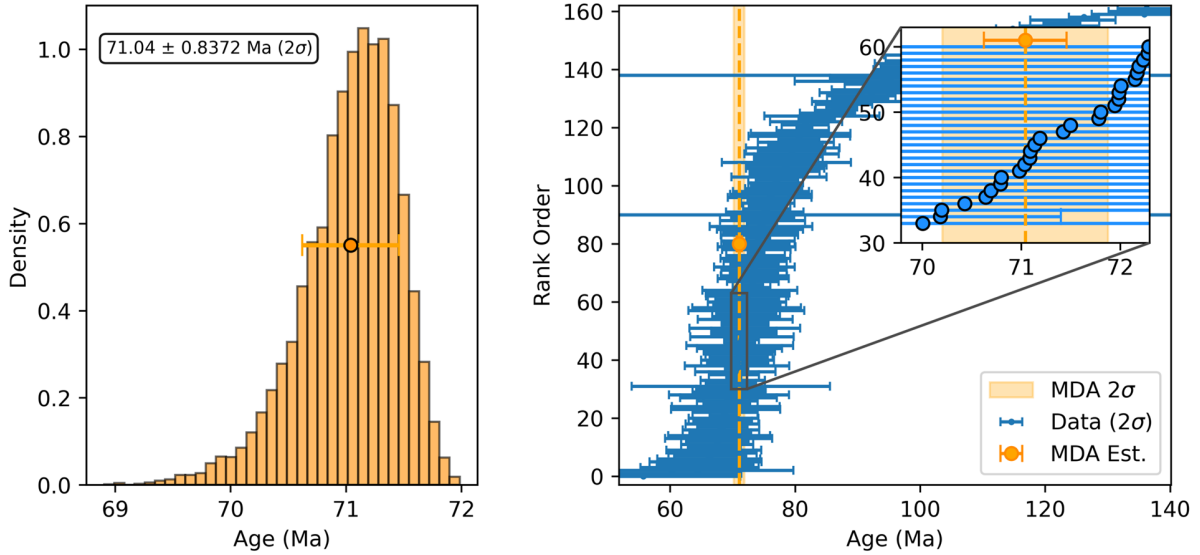


Figure S10. The orange histogram (left) shows the posterior distribution for all analyses post burn-in ($n = 50,000$) for sample FH22-1. Ranked age plot (right) shows individual $^{40}\text{Ar}/^{39}\text{Ar}$ ages and 2σ uncertainties in blue along with the Bayesian mean and 2σ uncertainty in orange as derived from the posterior distribution. The inset within the rank order plot (right) magnifies the region surrounding the MDA (orange symbol) and those grains falling within this window (blue symbols with black outlines) highlighting proximity to the MDA. Orange shaded region within inset shows a 2σ error envelope. Connecting lines between the inset panel and the main figure panel indicate the portion of the main panel that is magnified.

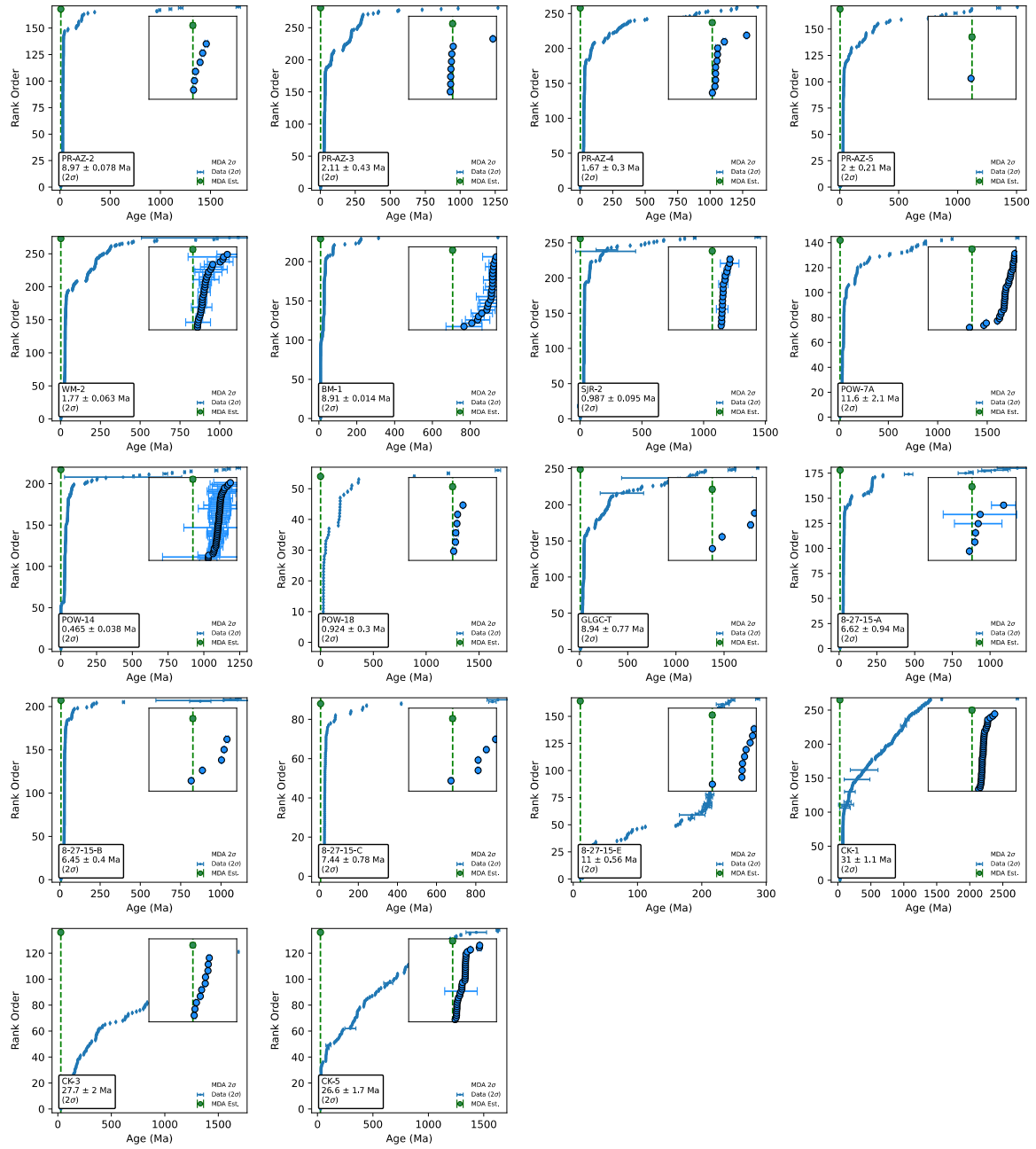


Figure S11. Rank order ages of individual grains with associated 2σ errors (blue) along with an MDA (green dashed vertical line) and 2σ error bounds (green region) from samples as reported in Heizler et al. (2021) [3] using the method reported in this study.

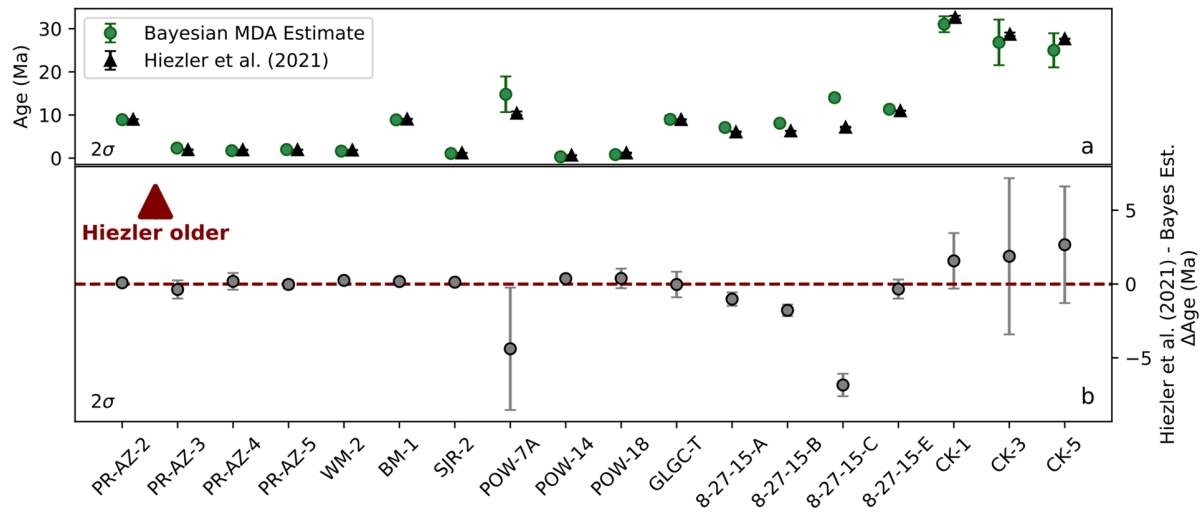


Figure S12. Comparison plots of Bayesian MDAs and reported MDAs in Heizler et al. [3]. Upper panel shows the Bayesian estimate as green circles and the reported Heizler et al. (2021) [3] value in black triangles, all shown with associated 2σ uncertainties. Some uncertainty bounds are smaller than the symbols. The bottom panel highlights the differences between the two values wherein the Bayesian estimated value is subtracted from the reported Heizler et al. (2021) [3] value with uncertainties added in quadrature. Grey circles and error bars denote the mean and 2σ uncertainty of the age difference. A maroon dashed horizontal line is a reference for zero age discrepancy and the triangle indicates the region corresponding to when an Mean Square Weighted Deviates (MSWD) MDA is older than the Bayesian estimate.

References

1. Black, L.P.; Kamo, S.L.; Allen, C.M.; et al. Improved $^{206}\text{Pb}/^{238}\text{U}$ microprobe geochronology by the monitoring of a trace-element related matrix effect; SHRIMP, ID-TIMS, ELA-ICP-MS and oxygen isotope documentation for a series of zircon standards. *Chem. Geol.* **2004**, *205*, 115–140. <https://doi.org/10.1016/j.chemgeo.2004.01.003>.
2. Slama, J.; Kosler, J.; Condon, D.J.; et al. Plesovice zircon-A new natural reference material for U-Pb and Hf isotopic microanalysis. *Chem. Geol.* **2008**, *249*, 1–35. <https://doi.org/10.1016/j.chemgeo.2007.11.005>.
3. Heizler, M.T.; Karlstrom, K.E.; Albonico, M.; et al. Detrital sanidine $^{40}\text{Ar}/^{39}\text{Ar}$ dating confirms < 2 Ma age of Crooked Ridge paleoriver and subsequent deep denudation of the southwestern Colorado Plateau. *Geosphere* **2021**, *17*, 438–454. <https://doi.org/10.1130/GES02319.1>.