scientific reports

Supplemental Information for

Late Miocene capybara in the Atacama Desert reveals a wetter climate and a transcontinental dispersal pathway

Carolina S. Gutstein[a,b]
Priscilla R. Martinez[c]
Barbara Carrapa[c]
Mark Clementz[d]
Jorge J. Cherem[e]
Mario A. Cozzuol[f]
Caroline A.E. Strömberg[g]
Mario E. Suárez[h]
Catalina Carreño C.[i]
Jacobus P. Le Roux[j]

[a]Escuela de Geología, Universidad Santo Tomás, Santiago, Chile.
[b]Fosfatos de Caldera SpA., Caldera, Atacama, Chile.
[c]Department of Geosciences, University of Arizona, Tucson, AZ, USA
[d]Department of Geology and Geophysics, University of Wyoming, Laramie, WY, USA
[e]Caipora Cooperativa para a Conservação da Natureza, Av. Desembargador Vítor Lima, 260/513, 88040-400, Florianópolis, SC, Brazil
[f]Universidade Federal de Minas Gerais, Instituto de Ciências Biológicas, Departamento de Zoologia, Av. Antônio Carlos, 6627, Pampulha, 31270-910 - Belo Horizonte, MG – Brasil
[g]Department of Biology & Burke Museum of Natural History and Culture, University of Washington, Seattle WA, USA.
[h]Laboratorio de Ontogenia y Filogenia, Departamento de Biología, Facultad de Ciencias, Universidad de Chile, Santiago, Chile
[i]Departamento de Geología, Facultad de Ciencias Físicas y Matemáticas, Universidad de Chile, Plaza Ercilla 803, Santiago
[j]Departamento de Geología, Facultad de Ciencias Físicas y Matemáticas, Universidad de Chile / Centro de Excelencia en Geotermia de los Andes, Plaza Ercilla 803, Santiago

Contents of this file

Text S1. Zircon U-Pb geochronology detailed methods for Element2 HR ICPMS and iCAP RQ Single Quad ICPMS.
Text S2. U-Pb Geochronologic analyses of zircon using E2/iCAPAgeCalcML.
Figure S1. Zircon U-Pb maximum depositional ages (MLA radial plots).
Figure S2. Zircon U-Pb maximum depositional ages (YC2σ, YC1σ, YSG MDA plots).
Figure S3. Representative phytoliths from CB-06.

Additional Supplemental Tables

Table S1. Measurements of MPC3000 tooth specimen compared to published *Cardiatherium* data.
Table S2. Analytical Settings for U-Pb Geochronology at the Arizona LaserChron Center (Element 2 and iCAP Single Collectors) — For detrital zircon sample analyses
Table S3. U-Pb zircon data for sample EMSSTBASE used for maximum depositional age calculation.
Table S4. U-Pb zircon data for sample EM1DZASH used for maximum depositional age calculation.
Table S5. U-Pb zircon data for sample CGSST used for maximum depositional age calculation.
Table S6. Stable isotopic analyses from Atacama soil carbonate nodules from the Caldera basin, northern Chile.
Table S7. Carbon isotope ($\delta^{13}C$) compilation of modern C_3 plant tissue (Kohn et al. 2020; O'Leary 1988), modern western Amazonian rainforest mammal tooth enamel (Tejada et al. 2020), and Miocene Atacama Desert soil carbonate nodules (this study).

Introduction

This supplemental information includes descriptions of the analytical techniques applied in this study and supplemental figures and data tables supporting results and interpretations in the main text.

- Data files herein include text describing analytic methods and approaches, figures supporting analytical results, tables detailing sample information and modeling parameters, and datasets containing detailed analytic data used in this study.
- Data for this study were produced between 2023 and 2025 using multiple laboratory facilities at the University of Arizona, Tucson, Arizona, USA.

Text S1: U-Pb Geochronologic analyses of zircon utilizing the Element2 HR ICPMS

Zircon crystals are extracted from samples by traditional methods of crushing and grinding, followed by separation with a Wilfley table, heavy liquids, and a Frantz magnetic separator. Samples are processed such that all zircons are retained in the final heavy mineral fraction. For detrital analyses, a large split of grains (generally thousands of grains) is incorporated into a 1” epoxy mount together with fragments or loose grains of Sri Lanka, FC-1, and R33 zircon crystals that are used as primary standards. For igneous samples, ~50 high-quality grains are selected and mounted with standards, generally with four samples per mount. The mounts are sanded down to a depth of ~20 microns, polished, imaged, and cleaned prior to isotopic analysis.

Grains of interest are imaged to provide a guide for locating analysis pits in optimal locations, and to assist in interpreting results. BSE and color CL Images are generated with a Hitachi 3400N SEM and a Gatan CL2 detector system (www.laserchron.org).

U-Pb geochronologic analyses are conducted by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) at the Arizona LaserChron Center (www.laserchron.org). Methods for U-Pb geochronology have been described by Gehrels et al. (2006, 2008), Gehrels and Pecha (2014), Pullen et al. (2018), and Sundell et al. (2021). The analyses involve ablation of zircon with a Teledyne/Photon Machines Analyte G2 excimer laser equipped with HelEx2 ablation cell, or an Iridia excimer laser equipped with a Cobalt cell, using a spot diameter of 20-30 microns. The ablated material is carried in helium into the plasma source of an Element2 HR ICPMS and iCAP RQ ICP-MS, which sequences rapidly through the U, Th, Pb, and Hg isotopes. Signal intensities are measured with an SEM that operates in pulse counting mode for signals less than 50K cps, in both pulse-counting and analog mode for signals between 50K and 5M cps, and in analog mode above 5M cps. The calibration between pulse-counting and analog signals is determined line-by-line for signals between 50K and 5M cps, and is applied to >5M cps signals. Because 235U is measured (rather than 238U), it is rare for intensities to be above 5M cps.

With the laser set at an energy density of ~5.6 J/cm2, a repetition rate of 7 Hz, and an ablation time of ~15 seconds, ablation pits are ~15 microns in depth. Sensitivity with these settings is approximately ~8,000 cps/ppm. Each analysis consists of ~5 seconds on peaks with the laser off (for backgrounds), ~15 seconds with the laser firing (for peak intensities), and a ~10 second delay to purge the previous sample.

One intensity is determined on each mass, with dwell times as follows:

- Hg202 0.021 sec
- Hg+Pb204 = 0.031 sec
- Pb206 = 0.081 sec
- Pb207 = 0.150 sec
- Pb208 = 0.010 sec
- Th232 = 0.010 sec
- U235 = 0.150 sec

These dwell times have been established to generate U-Th-Pb ages that are accurate to 2% (2-sigma SE) for a zircon similar in composition and age to FC-1. Total scan time is approximately 0.5 sec including all dwell and settle times.

Data are acquired with an acquisition that consists of 56 scans. These include:

- 11 scans on backgrounds prior to laser firing
- 32 scans with laser firing
- 13 scans after laser is no longer firing (for signal washout)

Data are acquired in Time-Resolved mode, such that data are continuously acquired by the mass spectrometer and the laser sequences through the analyses. The onset of each analysis is set when the 232U signal rises above a threshold intensity (default = 3000 cps) -- this time is referred to as Tzero.

Following analysis, data reduction is performed with a Python decoding routine and a MATLAB program (AgeCalcML; v GGmod68e). The first step is to extract the measured ion intensities from the Thermo output (.dat) files utilizing a decoder that was written by John Dr. Hartman (University of Arizona; Hartman et al., 2017). Details of this decoding routine are available from the Dat File Decoder link on the ALC web page (under Tools). This decoding routine generates .csv and .xls files that include all ion intensities for each scan. These ion intensities and the list of spot names are imported into AgeCalcML_E2_TRA.

Following are the calculations performed by AgeCalcML to reduce U-Th-Pb data.

1. The Browser function first creates the graphical user interface.
2. The Data Import function imports analysis names from the scancsv file (created by the laser software) and all of the raw data from the decoded xls file. Pulse-mode intensities are used for <5M cps signals, whereas analog-mode intensities are used for >5M cps signals. Measured ion intensities are stored in the original "numbers" array.
3. The Reduce Data function performs the initial data reduction and generates the plots shown on the interface. Main steps are as follows:
    a. Sets values for all variables.
    b. Subtracts 204Hg from the measured 204 intensities by using the measured 202Hg signal and the natural 202Hg/204Hg of 4.3. This yields intensities for 204Pb. Note that this Hg correction is not significant for most analyses because our Hg backgrounds are low (<100 cps at mass 202).
    c. Finds Tzero for each analysis by searching for the initial 232Th or 206Pb intensity above 3000 cps (default value).
    d. Populates the AnalysisValues array with values for each analysis. Registration is based on Tzero. 238U intensities are calculated from 235U * 137.818.
    e. Creates arrays for background values and peak values for each analysis. Backgrounds are calculated from the average of seven scans, minus max and min values. Peaks are calculated from the measured ion intensities while the laser is firing, minus the background values.
    f. Calculates preliminary 206/238 ratios determined from the on-peak, background-corrected ion intensities.
    g. Generates a preliminary fractionation factor for 206/238 for each standard by comparing measured 206/238 values with reported 206/238 values from Paces and Miller (1993) for FC-1, Gehrels et al. (2008) for SF, and Black et al. (2004) for R33. Note that ID-TIMS (not CA-TIMS) values are used given that our zircon grains have not been chemically abraded.
    h. Creates a sliding-window average from the ~8 closest values (minus max and min values).
    i. Calculates preliminary 206/238 ages based on the sliding-window average values of fractionation- and background-corrected 206/238.
    j. Preliminary 206/238 ages used to determine the composition of initial Pb based on Stacey and Kramers (1975).
    k. Initial Pb subtracted from 206, 207, and 208, resulting in background- and common-Pb-corrected 206/238, 206/207, and 208/232 ratios.
    l. Preliminary fractionation factors are applied to background- and common-Pb-corrected 206/238, 206/207, and 208/232 ratios, and resulting ratios of standards are compared with known values.
    m. Standard analyses are rejected if the ratios differ from known values by more than 5% (default value).
    n. Accepted standards are used to generate final fractionation factors for 206/238, 206/207, and 208/232. Corrections are generally ~5% for 206/238, <2% for 206/207, and ~20% for 208/232.
    o. A sliding window average (of ~8 closest values) is used to determine the fractionation factors that are used to correct the background- and common-Pb-corrected ratios for standards and unknowns. Sliding-window values and uncertainties for the final fractionation factor are shown on the fractionation plots with a solid line. The initial fractionation factor is shown with a dashed line (to show impact of filtering).
    p. Uncertainties are determined for isotope ratios based on the standard error of the mean of on-peak values for 206/207, 206/204, and 208/204. Uncertainties for 206/238 and

208/232 are calculated from the standard error of a regression through the on-peak values (using the polyfit and polyval functions in MATLAB).

q. Overdispersion factors are applied to the uncertainties for 206/238, 206/207, and 208/232 ratios. These factors have been determined from multiple analyses of secondary standards.
r. Calculates concentrations of U and Th for unknowns based on the measured intensities and known concentrations of FC-1 and R33.
s. A U-Th disequilibrium correction is applied to 206/238 ages based on the methods of Crowley et al. (2007).
t. Ages and uncertainties are calculated from the background-, fractionation-, and common-Pb-corrected 206/238, 206/207, and 208/232 ratios.
u. Determines a "Best Age" for each analysis, which is the 206/238 age for <900 Ma ages and the 206/207 age for >900 Ma ages (default values).
v. Analyses are automatically filtered based on default cutoffs for uncertainty, discordance, reverse discordance, 204Pb counts, 235U intensity, and on-peak signal stability. On-peak stability is based on comparison of eight values while the laser is firing. Analyses that exceed these cutoff values (default value = 5%) are rejected.
w. Error correlations and uncertainty ellipses are generated for 206/238-207/235 and 206/238-208/232 concordia diagrams.
x. Information shown in the list of analysis names (lower left) is generated. This includes a concordia ellipse for each analysis and information about the status of each analysis (whether accepted or rejected, and why rejected).
y. Calculates external (systematic) uncertainties for 206/238, 206/207, and 208/232, which include contributions from (a) the scatter of primary standard analyses, (b) uncertainties in the ages of the primary standard (FC1), (c) uncertainties in the composition of common Pb, (d) uncertainties in the decay constants for 235U, 238U, and 232Th, and (e) long-term variance of secondary standards. These are the systematic uncertainty terms recognized by Horstwood et al. (2016).

4. The Reduce Data function also calls a variety of functions that do all the data-reduction calculations and update all of the plots shown on the user interface.
5. Once initial data reduction has been completed, the operator can analyze the data using various plots and settings. Following is a list of the available plots:
   1 - Concordia All
   2 - Concordia Rejected (with sample names)
   3 - Concordia All - Young/Log
   4 - Concordia All - 206/238 vs 208/232
   5 - Concordia FC-SL-R33
   6 - Concordia FC
   7 - Concordia SL
   8 - Concordia R33
   9 - Offset Plot 206/238
   10 - Offset Plot 206/207
   11 - Offset Plot 208/232
   12 - Weighted Mean Unknowns
   13 - Weighted Mean FC1
   14 - Weighted Mean SLM
   15 - Weighted Mean R33
   16 - Age vs U Concentration
   17 - Age vs Radiation Dosage
   18 - Age vs U/Th
   19 - Age vs Concordance

The suggested flow for data analysis is as follows:

6. Evaluation of standards:
    a. In upper left window, enter higher and lower values for Min 235 (cps) and monitor the number of rejected unknowns and standards. Leave at setting with lowest numbers rejected. Go for minimum value (down to 300 cps) that does not exclude lots of analyses.
    b. Do the same with Max 204 (cps), and leave with lowest numbers of standards and unknowns rejected. Go for minimum value (down to 300 cps) that does not exclude lots of analyses.
    c. Adjust Stability Cutoff value, and leave with just a few unknowns rejected. Go for minimum value (down to 4%) that does not exclude lots of analyses. Note that the timing of each of the eight measurements is shown in the beam trace window.
    d. Select "Concordia Young/Log" Plot in upper right window. Adjust 206/204 factor so that concordance (lower right) ends up close to 100%. This adjusts the 206/204 and 208/204 used for common-Pb correction based on the assumption that young analyses (especially R33 standard) are discordant.
    e. Select "Offset Plot for 206/238" in upper right window. Adjust Low-int and Linear sliders so that standards and unknowns cluster along zero offset line. Standards are shown relative to known values. Unknowns are show relative to concordia. This adjusts the 235U intensity to correct for non-linearities in the measurement of low- and high-intensity signals.
    f. Do the same with 'Offset Plot 207/207" and "Offset Plot 208/232".
    g. Adjust the allowed offsets for 206/238 and 206/207 ages of standards. 5% is the default value. Few standards should be rejected.
    h. In upper-left window, click off Use SL 68, Use SL 67, and Use R33 68 so that only FC is used for fractionation correction. This is because FC-1 is our Primary Standard, and SL and R33 are used as secondary standards.
    i. Go back and fine-tune all settings.
7. Evaluation of unknowns:
    a. Evaluate numbers of unknowns rejected for each of the settings in the upper left window. Note that some analyses may be unacceptable for multiple reasons (e.g., large uncertainty and high discordance). Change settings only if necessary.
    b. Scan down through each analysis in the beam trace window (lower left), evaluating the down-hole pattern of intensities. Reject any analyses that show an unusual pattern (e.g., changes in values, increasing slope). Accept analyses (especially standards) that have been rejected only if there is a good reason. Note that timing is shown for Tzero (red diamond), start and end of the PeakValue array (black diamonds), and the eight stability measurements (vertical lines).
8. Cycle back through adjustments, making sure that each is set correctly.
9. Press Export Tables to save tables with reduced data (in the folder with the raw data files). Tables include:
    a. AnalysisValues Data Table: Array that contains initial ion intensities for each analysis. Values for backgrounds, peaks, Tzero, and stability criteria are indicated.
    b. PeakValues Data Table: Array that contains background-corrected ion intensities for each analysis. Values for peaks, Tzero, and stability criteria are indicated.
    c. Geochron Data Table: Table that can be used to import results into the Geochron Data Base. All information about session is included.
    d. Detailed Data Table: Table that includes all relevant information about each analysis.
10. Press Export Figures to save a copy of all figures. Figures are saved as pdf files in the folder with the raw data files. Close files one-by-one, or save each in a different format before you close them.

References:

Black, L., Kamo, S., Allen, C., Davis, D., Aleinikoff, J., Valley, J., Mundil, R., Campbell, I., Korsch, R., Williams, I., and Foudoulis, C., 2004, Improved 206Pb/238U 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: Chemical Geology, v. 205, p. 115-140.

Crowley, J.L., Schoene, B., and Bowring, S., 2007, U-Pb dating of zircon in the Bishop Tuff at the millennial scale: Geology, v. 35 (12), p. 1123-1126.

Gehrels, G.E., Valencia, V., Pullen, A., 2006, Detrital zircon geochronology by Laser-Ablation Multicollector ICPMS at the Arizona LaserChron Center, in Loszewski, T., and Huff, W., eds., Geochronology: Emerging Opportunities, Paleontology Society Short Course: Paleontology Society Papers, v. 11, 10 p.

Gehrels, G.E., Valencia, V., Ruiz, J., 2008, Enhanced precision, accuracy, efficiency, and spatial resolution of U-Pb ages by laser ablation–multicollector–inductively coupled plasma–mass spectrometry: Geochemistry, Geophysics, Geosystems, v. 9, Q03017, doi:10.1029/2007GC001805.

Gehrels, G. and Pecha, M., 2014, Detrital zircon U-Pb geochronology and Hf isotope geochemistry of Paleozoic and Triassic passive margin strata of western North America: Geosphere, v. 10 (1), p. 49-65.

Hartman, J., Franks, R., Gehrels, G., Hourigan, J., and Wenig, P. 2017, Decoding files from a Thermo Element ICP Mass Spectrometer: Archiv.org (ark:/13960/t3jx4rd3b)

Horstwood, M.S.A., Kosler, J., Gehrels, G., Jackson, S.E., McLean, N.M., Paton, C., Pearson, N.J., Sircombe, K., Sylvester, P., Vermeesch, P., Bowring, J.F., Condon, D.J., Schoene, B., 2016, Community-Derived Standards for LA-ICP-MS U-(Th-) Pb Geochronology – Uncertainty Propagation, Age Interpretation and Data Reporting: Geostandards and Geoanalytical Research, v. 40 (3), p. 311-332.

Paces, J.B., and Miller, J.D., 1993, Precise U-Pb Ages of Duluth Complex and Related Mafic Intrusions, Northeastern Minnesota: Geochronological Insights to Physical, Petrogenetic, Paleomagnetic, and Tectonomagmatic Processes Associated With the 1.1 Ga Midcontinent Rift System: Journal of Geophysical Research, v. 98 (B8), p. 13997-14013.

Pullen, A., Ibanez-Mejia, M., Gehrels, G., Giesler, D., and Pecha, M., 2018, Optimization of a Laser Ablation-Single Collector-Inductively Coupled Plasma-Mass Spectrometer (Thermo Element 2) for Accurate, Precise, and Efficient Zircon U-Th-Pb Geochronology: Geochemistry, Geophysics, Geosystems, v. 19 (10), p. 3689-3705. https://doi. org/10.1029/2018GC007889

Stacey, J.S., and Kramers, J.D., 1975, Approximation of terrestrial lead isotope evolution by a two-stage model: Earth and Planetary Science Letters, v. 26, p. 207-221.

Sundell, K.E., Gehrels, G.E., and Pecha, M.E., 2021, Rapid U-Pb Geochronology by Laser Ablation Multi-Collector: Geostandards and Geoanalytical Research, v. 45 (1), p. 37-57. doi.org/10.1111/ggr.12355

Text S2: U-Pb Geochronologic analyses of zircon using E2/iCAPAgeCalcML

(56 row, IA, 1 int; 235-only; 03 September 2024)

Zircon crystals are extracted from samples by traditional methods of crushing and grinding, followed by separation with a Wilfley table, heavy liquids, and a Frantz magnetic separator. Samples are processed such that all zircons are retained in the final heavy mineral fraction. For detrital analyses, a large split of grains (generally thousands of grains) is incorporated into a 1” epoxy mount together with fragments or loose grains of Sri Lanka, FC-1, and R33 zircon crystals that are used as primary standards. For igneous samples, ~50 high-quality grains are selected and mounted with standards, generally with four samples per mount. The mounts are sanded down to a depth of ~20 microns, polished, imaged, and cleaned prior to isotopic analysis.

Grains of interest are imaged to provide a guide for locating analysis pits in optimal locations, and to assist in interpreting results. BSE and color CL Images are generated with a Hitachi 3400N SEM and a Gatan CL2 detector system (www.laserchron.org).

U-Pb geochronologic analyses are conducted by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) at the Arizona LaserChron Center (www.laserchron.org). Methods for U-Pb geochronology on the Element2 single collector and Nu multicollector have been described by Gehrels et al. (2006, 2008), Gehrels and Pecha (2014), Pullen et al. (2018), and Sundell et al. (2021). The analyses involve ablation of zircon with a Teledyne/Photon Machines Analyte G2 excimer laser equipped with HelEx2 ablation cell, or an Iridia excimer laser equipped with a Cobalt cell, using a spot diameter of 20-30 microns. The ablated material is carried in helium into the plasma source of an iCAP RQ Single Quadrupole ICPMS, which sequences rapidly through the U, Th, Pb, and Hg isotopes. Signal intensities are measured with an SEM that operates in pulse counting mode for signals less than 50K cps, in both pulse-counting and analog mode for signals between 50K and 2M cps, and in analog mode above 2M cps. The calibration between pulse-counting and analog signals is determined line-by-line for signals between 50K and 2M cps, and is applied to >2M cps signals. However, because ^{235}U is measured (rather than ^{238}U), it is rare for intensities to be above 2M cps.

With the laser set at an energy density of ~10.59 J/cm2, a repetition rate of 7 Hz, and an ablation time of ~15 seconds, ablation pits are <15 microns in depth. Sensitivity with these settings is approximately ~8,000 cps/ppm. Each analysis consists of ~5 seconds on peaks with the laser off (for backgrounds), ~15 seconds with the laser firing (for peak intensities), and a ~5 second delay to purge the previous sample.

One intensity is determined on each mass, with dwell times as follows:

- Hg202 0.021 sec
- Hg+Pb204 = 0.031 sec
- Pb206 = 0.081 sec
- Pb207 = 0.150 sec
- Pb208 = 0.010 sec
- Th232 = 0.010 sec
- U235 = 0.150 sec

These dwell times have been established to generate U-Th-Pb ages that are accurate to 2–4% (2-sigma SE) for a zircon similar in composition and age to FC-1.

Data are acquired with an acquisition that consists of 56 scans. These include:

- 11 scans on backgrounds prior to laser firing
- 32 scans with laser firing
- 13 scans after laser is no longer firing (for signal washout)

Data are acquired in Individual-Analysis mode triggered from the iCAP RQ Qtegra software interface, such that data are not continuously acquired by the mass spectrometer. However, data collected on the iCAP is analyzed similar to that of Time-Resolved analyses acquired on the Element 2 HR ICPMS and Nu MC ICPMS in that the onset of each analysis recognized when the ^{232}Th or ^{206}Pb signal rises above a threshold intensity (default = 3000 cps) -- this time is referred to as Tzero.

Following analysis, data is output in nine columns (Start time, Index, 202Hg, 204Pb, 206Pb, 207Pb, 208Pb, 232Th, 235U) and referenced to the standard-sample bracketed G2 .scancsv file using an in house MATLAB program (AgeCalcML; v GGmod75i).

Following are the calculations performed by AgeCalcML to reduce U-Th-Pb data.

1. The Browser function first creates the graphical user interface.
2. The Data Import function imports analysis names from the scancsv file (created by the laser software) and all of the raw data from the xls file. **Pulse-mode intensities are used for <2M cps signals, whereas analog-mode intensities are used for >2M cps signals.** Measured ion intensities are stored in the original "numbers" array.
3. The Reduce Data function performs the initial data reduction and generates the plots shown on the interface. Main steps are as follows:
   a. Sets values for all variables.
   a. Subtracts ^{204}Hg from the measured 204 intensities by using the measured ^{202}Hg signal and the natural ^{202}Hg/^{204}Hg of 4.3. This yields intensities for ^{204}Pb. Note that this Hg correction is not significant for most analyses because our Hg backgrounds are low (<500 cps at mass 202).
   a. Finds Tzero for each analysis by searching for the initial ^{232}Th or ^{206}Pb intensity above 3000 cps (default value).
   a. Populates the AnalysisValues array with values for each analysis. Registration is based on Tzero. ^{238}U intensities are calculated from ^{235}U * 137.818.
   a. Creates arrays for background values and peak values for each analysis. Backgrounds are calculated from the average of seven scans, minus max and min values. Peaks are calculated from the measured ion intensities while the laser is firing, minus the background values.
   a. Calculates preliminary 206/238 ratios determined from the on-peak, background-corrected ion intensities.
   a. Generates a preliminary fractionation factor for 206/238 for each standard by comparing measured 206/238 values with reported 206/238 values from Paces

and Miller (1993) for FC-1, Gehrels et al. (2008) for SF, and Black et al. (2004) for R33. Note that ID-TIMS (not CA-TIMS) values are used given that our zircon grains have not been chemically abraded.

a. Creates a sliding-window average from the ~8 closest values (minus max and min values).
a. Calculates preliminary 206/238 ages based on the sliding-window average values of fractionation- and background-corrected 206/238.
a. Preliminary 206/238 ages used to determine the composition of initial Pb based on Stacey and Kramers (1975).
a. Initial Pb subtracted from 206, 207, and 208, resulting in background- and common-Pb-corrected 206/238, 206/207, and 208/232 ratios.
a. Preliminary fractionation factors are applied to background- and common-Pb-corrected 206/238, 206/207, and 208/232 ratios, and resulting ratios of standards are compared with known values.
a. Standard analyses are rejected if the ratios differ from known values by more than 5% (default value).
a. Accepted standards are used to generate final fractionation factors for 206/238, 206/207, and 208/232. Corrections are generally ~5% for 206/238, <2% for 206/207, and ~20% for 208/232.
a. A sliding window average (of ~8 closest values) is used to determine the fractionation factors that are used to correct the background- and common-Pb-corrected ratios for standards and unknowns. Sliding-window values and uncertainties for the final fractionation factor are are shown on the fractionation plots with a solid line. The initial fractionation factor is shown with a dashed line (to show impact of filtering).
a. Uncertainties are determined for isotope ratios based on the standard error of the mean of on-peak values for 206/207, 206/204, and 208/204. Uncertainties for 206/238 and 208/232 are calculated from the standard error of a regression through the on-peak values (using the polyfit and polyval functions in MATLAB).
a. Overdispersion factors are applied to the uncertainties for 206/238, 206/207, and 208/232 ratios. These factors have been determined from multiple analyses of secondary standards.
a. Calculates concentrations of U and Th for unknowns based on the measured intensities and known concentrations of FC-1 and R33.
a. A U-Th disequilibrium correction is applied to 206/238 ages based on the methods of Crowley et al. (2007).
a. Ages and uncertainties are calculated from the background-, fractionation-, and common-Pb-corrected 206/238, 206/207, and 208/232 ratios.
a. Determines a “Best Age” for each analysis, which is the 206/238 age for <900 Ma ages and the 206/207 age for >900 Ma ages (default values).
a. Analyses are automatically filtered based on default cutoffs for uncertainty, discordance, reverse discordance, ^{204}Pb counts, ^{235}U intensity, and on-peak signal stability. On-peak stability is based on comparison of eight values while the laser is firing. Analyses that exceed these cutoff values (default value = 5%) are rejected.

a. Error correlations and uncertainty ellipses are generated for 206/238-207/235 and 206/238-208/232 concordia diagrams.
a. Information shown in the list of analysis names (lower left) is generated. This includes a concordia ellipse for each analysis and information about the status of each analysis (whether accepted or rejected, and why rejected).
a. Calculates external (systematic) uncertainties for 206/238, 206/207, and 208/232, which include contributions from (a) the scatter of primary standard analyses, (b) uncertainties in the ages of the primary standard (FC1), (c) uncertainties in the composition of common Pb, (d) uncertainties in the decay constants for ^{235}U, ^{238}U, and ^{232}Th, and (e) long-term variance of secondary standards. These are the systematic uncertainty terms recognized by Horstwood et al. (2016).

2. The Reduce Data function also calls a variety of functions that do all the data-reduction calculations and update all of the plots shown on the user interface.
3. Once initial data reduction has been completed, the operator can analyze the data using various plots and settings. Following is a list of the available plots:

1 - Concordia All
2 - Concordia Rejected (with sample names)
3 - Concordia All - Young/Log
4 - Concordia All - 206/238 vs 208/232
5 - Concordia FC-SL-R33
6 - Concordia FC
7 - Concordia SL
8 - Concordia R33
9 - Offset Plot 206/238
10 - Offset Plot 206/207
11 - Offset Plot 208/232
12 - Weighted Mean Unknowns
13 - Weighted Mean FC1
14 - Weighted Mean SLM
15 - Weighted Mean R33
16 - Age vs U Concentration
17 - Age vs Radiation Dosage
18 - Age vs U/Th
19 - Age vs Concordance

The suggested flow for data analysis is as follows:

0. Evaluation of standards:

a. In upper left window, enter higher and lower values for Min 235 (cps) and monitor the number of rejected unknowns and standards. Leave at setting with lowest numbers rejected. Go for minimum value (down to 300 cps) that does not exclude lots of analyses.
a. Do the same with Max 204 (cps), and leave with lowest numbers of standards and unknowns rejected. Go for minimum value (down to 300 cps) that does not exclude lots of analyses.
a. Adjust Stability Cutoff value, and leave with just a few unknowns rejected. Go for minimum value (down to 4%) that does not exclude lots of analyses. Note that the timing of each of the eight measurements is shown in the beam trace window.

a. Select "Concordia Young/Log" Plot in upper right window. Adjust 206/204 factor so that concordance (lower right) ends up close to 100%. This adjusts the 206/204 and 208/204 used for common-Pb correction based on the assumption that young analyses (especially R33 standard) are discordant.
a. Select "Offset Plot for 206/238" in upper right window. Adjust Low-int and Linear sliders so that standards and unknowns cluster along zero offset line. Standards are shown relative to known values. Unknowns are show relative to concordia. This adjusts the ^{235}U intensity to correct for non-linearities in the measurement of low- and high-intensity signals.
a. Do the same with 'Offset Plot 207/207" and "Offset Plot 208/232".
a. Adjust the allowed offsets for 206/238 and 206/207 ages of standards. 5% is the default value. Few standards should be rejected.
a. In upper-left window, click off <u>Use SL 68</u>, <u>Use SL 67</u>, and <u>Use R33 68</u> so that only FC is used for fractionation correction. This is because FC-1 is our Primary Standard, and SL and R33 are used as secondary standards.
a. Go back and fine-tune all settings.

7. Evaluation of unknowns:
a. Evaluate numbers of unknowns rejected for each of the settings in the upper left window. Note that some analyses may be unacceptable for multiple reasons (e.g., large uncertainty and high discordance). Change settings only if necessary.
a. Scan down through each analysis in the beam trace window (lower left), evaluating the down-hole pattern of intensities. Reject any analyses that show an unusual pattern (e.g., changes in values, increasing slope). Accept analyses (especially standards) that have been rejected only if there is a good reason. Note that timing is shown for Tzero (red diamond), start and end of the PeakValue array (black diamonds), and the eight stability measurements (vertical lines).

8. Cycle back through adjustments, making sure that each is set correctly.

9. Press Export Tables to save tables with reduced data (in the folder with the raw data files). Tables include:
a. AnalysisValues Data Table: Array that contains initial ion intensities for each analysis. Values for backgrounds, peaks, Tzero, and stability criteria are indicated.
a. PeakValues Data Table: Array that contains background-corrected ion intensities for each analysis. Values for peaks, Tzero, and stability criteria are indicated.
a. Geochron Data Table: Table that can be used to import results into the Geochron Data Base. All information about session is included.
a. Detailed Data Table: Table that includes all relevant information about each analysis.

10. Press Export Figures to save a copy of all figures. Figures are saved as pdf files in the folder with the raw data files. Close files one-by-one, or save each in a different format before you close them.

References:

Black, L., Kamo, S., Allen, C., Davis, D., Aleinikoff, J., Valley, J., Mundil, R., Campbell, I., Korsch, R., Williams, I., and Foudoulis, C., 2004, Improved 206Pb/238U 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: Chemical Geology, v. 205, p. 115-140.

Crowley, J.L., Schoene, B., and Bowring, S., 2007, U-Pb dating of zircon in the Bishop Tuff at the millennial scale: Geology, v. 35 (12), p. 1123-1126.

Gehrels, G.E., Valencia, V., Pullen, A., 2006, Detrital zircon geochronology by Laser-Ablation Multicollector ICPMS at the Arizona LaserChron Center, in Loszewski, T., and Huff, W., eds., Geochronology: Emerging Opportunities, Paleontology Society Short Course: Paleontology Society Papers, v. 11, 10 p.

Gehrels, G.E., Valencia, V., Ruiz, J., 2008, Enhanced precision, accuracy, efficiency, and spatial resolution of U-Pb ages by laser ablation–multicollector–inductively coupled plasma–mass spectrometry: Geochemistry, Geophysics, Geosystems, v. 9, Q03017, doi:10.1029/2007GC001805.

Gehrels, G. and Pecha, M., 2014, Detrital zircon U-Pb geochronology and Hf isotope geochemistry of Paleozoic and Triassic passive margin strata of western North America: Geosphere, v. 10 (1), p. 49-65.

Hartman, J., Franks, R., Gehrels, G., Hourigan, J., and Wenig, P. 2017, Decoding files from a Thermo Element ICP Mass Spectrometer: Archiv.org (ark:/13960/t3jx4rd3b)

Horstwood, M.S.A., Kosler, J., Gehrels, G., Jackson, S.E., McLean, N.M., Paton, C., Pearson, N.J., Sircombe, K., Sylvester, P., Vermeesch, P., Bowring, J.F., Condon, D.J., Schoene, B., 2016, Community-Derived Standards for LA-ICP-MS U-(Th-) Pb Geochronology – Uncertainty Propagation, Age Interpretation and Data Reporting: Geostandards and Geoanalytical Research, v. 40 (3), p. 311-332.

Paces, J.B., and Miller, J.D., 1993, Precise U-Pb Ages of Duluth Complex and Related Mafic Intrusions, Northeastern Minnesota: Geochronological Insights to Physical, Petrogenetic, Paleomagnetic, and Tectonomagmatic Processes Associated With the 1.1 Ga Midcontinent Rift System: Journal of Geophysical Research, v. 98 (B8), p. 13997-14013.

Pullen, A., Ibanez-Mejia, M., Gehrels, G., Giesler, D., and Pecha, M., 2018, Optimization of a Laser Ablation-Single Collector-Inductively Coupled Plasma-Mass Spectrometer (Thermo Element 2) for Accurate, Precise, and Efficient Zircon U-Th-Pb Geochronology: Geochemistry, Geophysics, Geosystems, v. 19 (10), p. 3689-3705. https://doi. org/10.1029/2018GC007889

Stacey, J.S., and Kramers, J.D., 1975, Approximation of terrestrial lead isotope evolution by a two-stage model: Earth and Planetary Science Letters, v. 26, p. 207-221.

Sundell, K.E., Gehrels, G.E., and Pecha, M.E., 2021, Rapid U-Pb Geochronology by Laser Ablation Multi-Collector: Geostandards and Geoanalytical Research, v. 45 (1), p. 37-57. doi.org/10.1111/ggr.12355

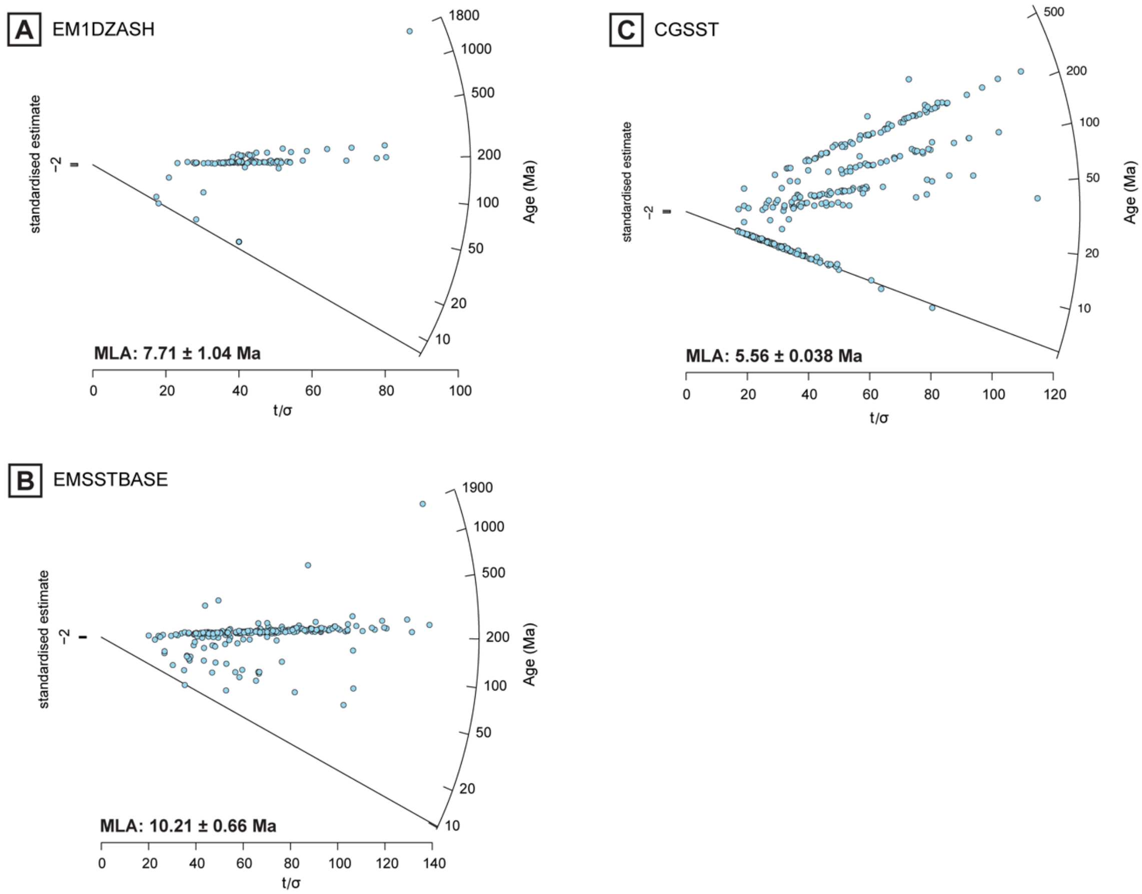

Figure S2. Radial plots showing zircon U‑Pb ages for samples from the Bahía Inglesa Formation at the El Morro locality (**A–B**) and Copiapó Gravels locality (**C**). Line shows 'minimum' discrete age peak defining MLA (Vermeesch, 2021a, 2021b). MLAs and 95% confidence intervals are annotated. Values are reported in Table 3 and appended to stratigraphic columns in Figure 2 of the main text.

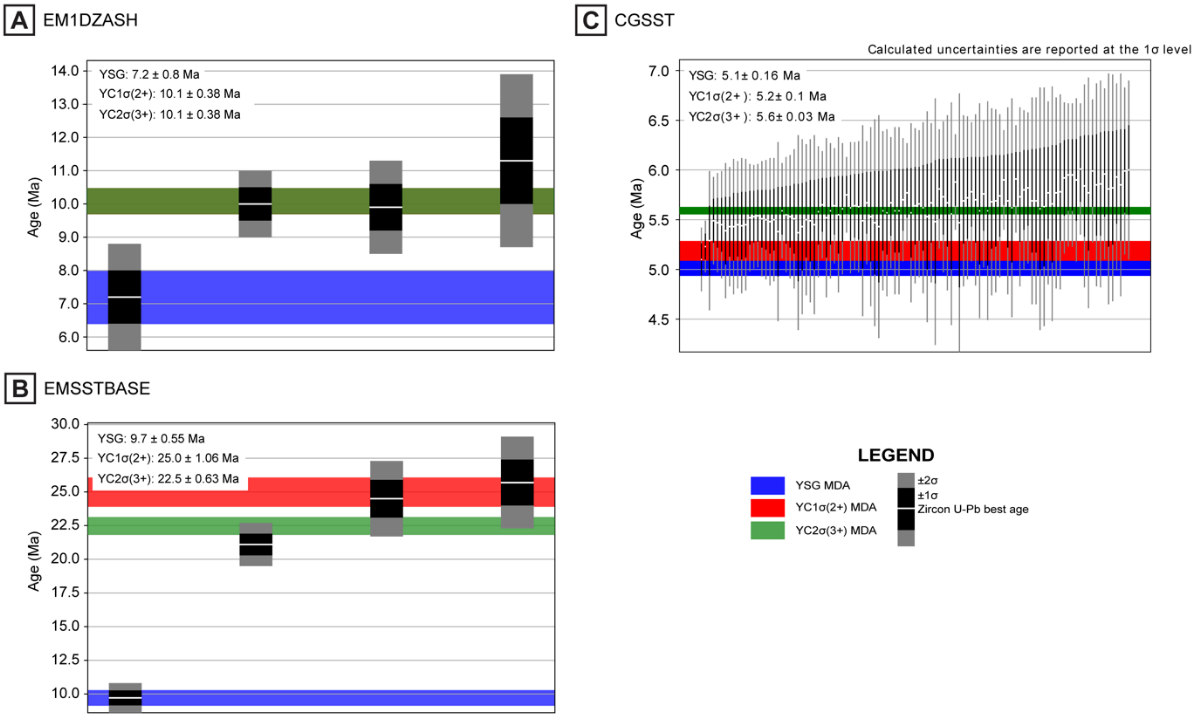

Figure S3. Plots of zircon maximum depositional ages (MDAs) for samples from the Bahía Inglesa Formation at the El Morro locality (**A–B**) and Copiapó Gravels locality (**C**) calculated via the youngest cluster of 3 or more ages overlapping at 2σ uncertainty [YC2σ(3+)], youngest cluster of 2 or more ages overlapping at 1σ uncertainty [YC1σ(2+)], and the youngest single grain. Values are reported in Table 3 of the main text.

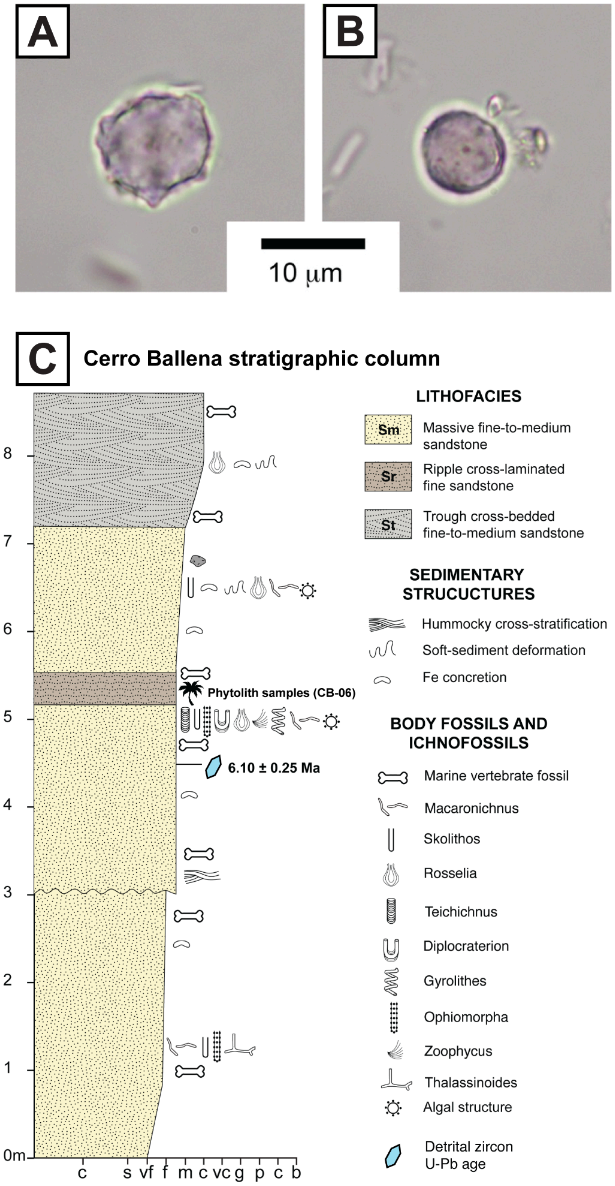

Fig. S4. Representative phytoliths from CB-06. **A.** SPHEROID ECHINATE, diagnostic of palms. **B.** SPHEROID PSILATE, diagnostic of other forest indicators (e.g., woody dicots). **C.** Cerro Ballena stratigraphic column showing location of phytoliths from sample CB-06, modified from Martinez et al. (2025).