APPENDIX

Tests on $\Delta_{47}$ Clean Up

Subambient and ambient (room) column temperatures were tested for optimal sample clean-up efficiency. Column performance was verified using 1000 °C heated gases, CO$_2$ standard gases, and CO$_2$ derived from the acid digestion of clean (Carrara Marble) and dirty (modern non-living coral fragments; impure limestone) calcite. The column as configured above was found to clean samples effectively and provide throughput yields of CO$_2$ indistinguishable from 100% when processed at room temperature and allowing a 35–45 min collection time, depending on sample size. Samples were analyzed isotopically within 24 h of digestion and purification.

Corrections and Quality Control Checks on the MAT253

The analytical run structure employed a pressure baseline correction described previously (He et al., 2012). Our run was divided into 5 or 6 acquisitions with pressure balancing in between each acquisition to achieve a 16V signal on the m/z 44 detector (Huntington et al., 2009). Cumulative on-peak signal integration time was generally 1000 – 1600 seconds for each of sample and reference gases, and instrumental uncertainty was at or near that calculated from counting statistics (around 10 ppm in $\Delta_{47}$).

We performed a number of quality control checks on our analytical system. These included monitoring the $\Delta_{47}$ versus cycle number for individual runs to ensure stable instrument performance. The m/z 48 signal was monitored for excess amplitude that would indicate inadequate cleaning of the sample gas. None of our samples gave anomalous $\Delta_{48}$ values. Nevertheless, we passed a subset of our samples through the chromatographic clean-up column a second time in order to ensure that isobaric interferences had been removed; analyses before and after the second column clean-up gave $\Delta_{47}$ values within uncertainty of each other. As a further test for isobaric interferences, we equilibrated splits of CO$_2$ from a few large samples at 26 °C or 60 °C with water, or roasted at 1000 °C without water. These test equilibrations yielded $\Delta_{47}$ values that were on our equilibrated gas lines within uncertainty, indicating that sample gases were free of isobaric interferences.

Equilibrated Gases and Calcite Standard Materials

Tibetan samples were weighed out to yield roughly 100–200 µg CaCO$_3$ equivalent. Comparable size ranges of the isotopic standards NBS-18 ($\delta^{13}$C = −5.01‰ VPDB scale; $\delta^{18}$O = −23.20‰ VPDB), NBS-19 ($\delta^{13}$C = 1.95‰ VPDB; $\delta^{18}$O = −2.19‰ VPDB), and periodically LSVEC ($\delta^{13}$C = −46.6‰ VPDB) were interspersed with the Tibetan samples.

We measure $\Delta_{47}$, $\delta_{47}$, $\delta^{13}$C, and $\delta^{18}$O relative to an Oztech (Oztech Trading Co., Safford, AZ, USA) isotopic standard gas (UOC 1766, $\delta^{13}$C = −3.61 VPDB; $\delta^{18}$O = 24.99 VSMOW; VSMOW used in $\Delta_{47}$ calculations) used as the working reference gas. The $\Delta_{47}$ values measured
relative to the working gas were corrected to an acid digestion temperature of 25 °C using the
temperature dependent acid fractionation factor for calcite computed by Guo et al. (2009). These
Δ_{47} results are then converted from the working gas reference frame to the carbon dioxide
equilibrium scale (CDES; Dennis et al., 2011) using results from routine analyses of CO₂ heated
to 1000 °C (HG) and CO₂ equilibrated through reaction with water at 4 °C, 25 °C, and 60 °C
(EG).

Arrays of HG and EG analyses are used to generate an empirical transfer function (ETF),
following the steps detailed in He et al. (2012) and the approach of Dennis et al. (2011). All data
are pressure baseline (PBL) corrected (He et al., 2012). In contrast to the negative slopes
encountered with PBL-corrected HG and EG lines in He et al. (2012), we observed small
positive slopes in the analytical period in which Oiyug and Penbo samples were measured
(−3.092e-03), signifying a slight under-correction of the pressure baseline. Nevertheless, the HG
and EG slopes were reduced by a factor of 4 and there is no evidence for a change in the PBL-
corrected HG and EG line slopes over the measurement interval.

We interspersed sample runs with digestions and analyses of reference carbonates
(Tables A1 and A2). These included Carrara Marble (our own sample and CM-2 from D. Schrag,
Harvard University) and the suite of ETH-1, ETH-2, ETH-3, and ETH-4 (S. Bernasconi, ETH
Zurich). Carrara Marble analyses, using both our sample and CM-2, are generally ca. 20–30 ppm
higher in Δ_{47} than those results reported in Dennis et al. (2011), but fall within the range
measured by other labs. Our ETH results are within uncertainty of the mean results from a
preliminary interlaboratory comparison reported by S. Bernasconi at the 4th and 5th International
Clumped Isotope Workshops, held in August 2014, Zurich, Switzerland, and January 2016, St.
Petersburg, Florida. We include the ETH standards because we expect that they will become
widely used as international standards for clumped isotope analysis in the coming years.

Paleoaltimetry Model Parameters

Paleoelevation estimates are calculated using a modified version of Rowley’s (2007)
isotopic lapse rate model. We first use the Δ_{47} temperature estimate coupled with measured δ^{18}O_c
in order to calculate the δ^{18}O_w of the water with which the carbonate equilibrated (Kim and
O’Neil, 1997):

\[
\delta^{18}O_{water} = \delta^{18}O_{calcite} - 18.03 \left( \frac{10^3}{T(\Delta_{47})} \right) + 32.42, \quad (1)
\]

with all δ^{18}O values on VSMOW scale (converting the carbonate data using \( \delta^{18}O_{VSMOW} =
1.03086 \times \delta^{18}O_{VPDB} + 30.86 \% \)); Friedman and O’Neil, 1977). The paleowater δ^{18}O_w was then
used to estimate model elevation, Z:

\[
Z(m) = -0.0129 \left( \Delta \left( \delta^{18}O_p \right) \right)^4 -1.1213 \left( \Delta \left( \delta^{18}O_p \right) \right)^3 -38.214 \left( \Delta \left( \delta^{18}O_p \right) \right)^2 -715.22 \left( \Delta \left( \delta^{18}O_p \right) \right) \quad (2)
\]

where \( \Delta \left( \delta^{18}O_p \right) \), a model parameter, is the difference between the oxygen isotopic composition
of precipitation at elevation and that near sea level along the same mean storm trajectory.
Licht et al. (2014) report fresh water gastropod carbonate and tooth enamel phosphate oxygen isotopic compositions and corresponding reconstructed surface waters in late Eocene (~40 Ma) strata from the central Burma basin with values of ~-7.2‰ to ~-8.4‰, respectively. Although they discount a high elevation contribution, it would only require the precipitation-weighted hypsometric mean elevation to be between ~500 and ~1500 m assuming the ~-6.6‰ normalization value adopted here to account for these slightly more depleted values. Given the detrital zircon data from this same area (Robinson et al., 2014) and our conclusion described below regarding the paleoelevation of the Linzizong arc, we view it likely that their (Licht et al., 2014) estimates are not sea level values.

Using the modern isotopic composition of low-latitude, near sea level precipitation (~-3.6 ± 1.6 ‰; Rowley and Currie, 2006), our calculated mean elevation is boosted to 4.3 ± 0.4 km or 4.8 ± 0.4 km, using the Dennis and Schrag (2010) or Zaarur et al. (2013) thermometers, respectively. The Zaarur et al. (2013) paleothermometer, derived from analyses of inorganic CaCO₃ and supported by biogenic CaCO₃ analyses, conducted in individual phosphoric acid reactors at 25 °C, is more appropriate for our sample digestion conditions and has been applied to other recent clumped isotope paleoaltimetry in Tibet (Huntington et al., 2015). Even if we were to use the warm endmember Δ47 paleothermometer from Dennis and Schrag (2010) (derived from analysis of inorganic CaCO₃ precipitates processed in a 90 °C common phosphoric acid bath), the calculated paleotemperatures would be 25–29 °C. The conservative elevation calculation, using these warmer temperatures and the oldest Siwaliks to constrain lowland δ¹⁸O₉, still implies that the Penbo Basin would have been in excess of 3 km at the onset of the Indo-Eurasian collision. We emphasize that our first-order conclusion regarding high topography is independent of the paleothermometer calibration and/or near-sea level δ¹⁸O₉ used.

The ± 2 σ error envelope for the elevations uses the equations that characterize the 2σ error on the empirically determined modern precipitation-elevation relationship updated from Rowley (2007). The T(Δ47) errors are propagated through the equation for the calcite-water fractionation factor to determine Δ(δ¹⁸O₉) 2σ error.

REFERENCES CITED


Figure A1. Inverse isochron plot of potassium feldspar ⁴⁰Ar/³⁹Ar age.

Figure A2. Effects of evaporation on siderites (dark red square), calcites (dark red circles) and leaf wax (blue-green diamonds) from the same sedimentary horizon. Inferred paleo-lake and soil water hydrogen and oxygen isotope compositions are plotted for sedimentary horizons 621 (Oiyug Fm) and 618 (Gazhacun Gp). The δ¹⁸O_w values are calculated from δ¹⁸O_c measurements on carbonate minerals, and δD_w values are calculated from leaf waxes, as described in the main text. The δD_w and δ¹⁸O_w axes are aligned using the Rozanski et al. (1993) Global Meteoric Water Line equation (δD = 8.20 × δ¹⁸O+11.27). This alignment means that if the paleo-water isotopic composition lay on the modern Global Meteoric Water line, the corresponding carbonate and leaf wax samples would yield inferred δ¹⁸O_w and δD_w values in the same vertical position. Lacustrine siderite 621F has experienced much greater evaporative enrichment than pedogenic calcites 621H and 621I from the same strata. This could be indicative of a period of greater aridity or deposition in a closed playa lake rather than an open system, or a change in atmospheric circulation over the Lhasa Block in the late Cenozoic. The difference between the isotopic
compositions of leaf waxes and siderite in the Miocene Gazhacun Formation is negligible within uncertainty.

Table A1. Summary of Δ47 measurements on standard calcite materials run during the same analytical periods as the samples reported in this paper (2014–2016).

Table A2. Isotopic measurements (δ¹³C, δ¹⁸O, Δ₄₇,S-G,WG, δ⁴⁷, Δ₄₈) of all standard calcite materials run during the same analytical periods as the samples reported in this paper (2014–2016).

Table A3. locations and lithologies of samples analyzed by the U/Pb zircon LA-ICP-MS method and their interpreted ages
Inverse Isochron: K-feldspar W0628-4
Oiyug Basin, Rigongla Formation

$^{40}\text{Ar}/^{36}\text{Ar}$ Intercept = 463.2 ± 32.2
Inverse Isochron Age = 28.90 ± 1.52 Ma
MSWD = 0.06
Kinetic isotope effects of evaporation on carbonate and leaf wax from the same strata

Proxy material for paleo-water

Gazhacun Gp.  
Siderite  
Calcite  
Leaf Wax

Oiyug Fm.  
Siderite  
Calcite  
Leaf Wax

$\delta^{18}O_{\text{water}}$ (‰)  
$\delta^D_{\text{water}}$ (‰)

$\delta^{18}O$  
$\delta^{18}O$  
$\delta D$

$n=4$  
n=5
TABLE A1. SUMMARY OF $\Delta_{47}$ MEASUREMENTS OF STANDARD CALCITE MATERIALS MEASURED WITH SAMPLES.

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<tr>
<th>Standard Material</th>
<th>$\Delta_{47},$CDES</th>
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<td>Carrera Marble</td>
<td>0.437 ± 0.004</td>
<td>34</td>
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<tr>
<td>ETH-1</td>
<td>0.349 ± 0.029</td>
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<tr>
<td>ETH-2</td>
<td>0.319 ± 0.010</td>
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<td>0.724 ± 0.011</td>
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<tr>
<td>ETH-4</td>
<td>0.582 ± 0.011</td>
<td>5</td>
</tr>
<tr>
<td>NBS-19</td>
<td>0.444 ± 0.015</td>
<td>4</td>
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### TABLE A2. ISOTOPIC COMPOSITIONS OF STANDARD CALCITE MATERIALS ANALYZED DURING ANALYTICAL PERIODS IN WHICH SAMPLES FROM THE OIYUG AND PENBO BASINS WERE ANALYZED.

<table>
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<th>Sample</th>
<th>(^{13}\text{C})</th>
<th>(^{18}\text{O})</th>
<th>(^{47}\text{SG-WG})</th>
<th>(^{47}\text{C})</th>
<th>(\Delta_{48})</th>
<th>(\Delta_{47,\text{CDES}})</th>
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**Average ± s.e.m.**

**ETH-1**

- 2.0 ± 0.003
- 38.9 ± 0.011
- 0.486 ± 0.001
- 18.9 ± 0.011
- 0.435 ± 0.001

**Average ± s.e.m.**

**ETH-2**

- -10.1 ± 0.07
- 21.8 ± 0.05
- -0.527 ± 0.009
- -9.85 ± 0.07
- 0.319 ± 0.010

**Average ± s.e.m.**

**ETH-3**

- 1.8 ± 0.05
- 39.5 ± 0.07
- -0.209 ± 0.045
- 19.4 ± 0.702
- 9/17/2014

**Average ± s.e.m.**

**ETH-4**

- -10.1 ± 0.05
- 21.8 ± 0.07
- -0.322 ± 0.037
- -9.8 ± 0.006
- 0.551 ± 0.031

**Average ± s.e.m.**

**NBS-19**

- 1.9 ± 0.05
- 38.9 ± 0.49
- -0.494 ± 0.450
- 18.8 ± 1.762
- 1/12/2016

**Average ± s.e.m.**

- 1.95 ± 0.01
- 38.99 ± 0.03
- -0.500 ± 0.015
- -18.85 ± 0.03
- 0.444 ± 0.015

---

Note: *s.e.m. (Standard Error of the Mean) used to estimate analytical error for samples with more than 3 measurements. For samples with <3 measurements, standard deviation is reported.*
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<th>Sample number</th>
<th>Rock type</th>
<th>Method</th>
<th>Formation</th>
<th>Longitude</th>
<th>Latitude</th>
<th>Elevation (m)</th>
<th>Age (Ma) ± Error (2σ)</th>
<th>n (# crystals)</th>
<th>Standards</th>
<th>Source</th>
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<td>Volcanic tuff</td>
<td>zircon U/Pb</td>
<td>Linzizong upper Pana</td>
<td>E91° 8.9'</td>
<td>N30° 0.57'</td>
<td>4205</td>
<td>50.5 ± 2.4 (1σ)</td>
<td>23</td>
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<td>2009TL973</td>
<td>Volcanic tuff</td>
<td>zircon U/Pb</td>
<td>Linzizong upper Pana</td>
<td>E91° 11'</td>
<td>N30° 0.57'</td>
<td>4308</td>
<td>48.4 ± 1 (1σ)</td>
<td>29</td>
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<td>MW16</td>
<td>Dacitic lava flow</td>
<td>sanidine feldspar 40Ar/39Ar</td>
<td>Gazhacun</td>
<td>E89° 35'</td>
<td>N29° 42'</td>
<td>14.03</td>
<td>0.37 ± 0.03</td>
<td>?</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>MW15</td>
<td>Dacitic lava flow</td>
<td>sanidine feldspar 40Ar/39Ar</td>
<td>Gazhacun</td>
<td>E89° 35'</td>
<td>N29° 42'</td>
<td>15.25</td>
<td>0.4 ± 0.04</td>
<td>?</td>
<td></td>
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</tr>
<tr>
<td>MW8</td>
<td>Volcanogenic sediments</td>
<td>phlogopite mica 40Ar/39Ar</td>
<td>Gazhacun</td>
<td>E89° 35'</td>
<td>N29° 42'</td>
<td>15.03</td>
<td>0.11 ± 0.01</td>
<td>?</td>
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<tr>
<td>Mafic Dike</td>
<td>hornblende 40Ar/39Ar</td>
<td></td>
<td>Linzizong Nianbo</td>
<td>E91° 8'</td>
<td>N29° 57'</td>
<td>52.9</td>
<td>0.4 ± 0.04</td>
<td>?</td>
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</tr>
</tbody>
</table>

Calibrated Pb/U and Th/U fractionation by analyzing Sri Lanka zircon with known concordant age of 564±4Ma (2σ)*

*Gehrels et al., 2006
†Slama et al., 2008
§Cai et al., 2012

TABLE A3. LOCATIONS AND LITHOLOGIES OF SAMPLES ANALYZED BY THE U/PB ZIRCON LA-ICP-MS METHOD AND THEIR INTERPRETED AGES

He et al., 2007
Ding et al., 2014
Spicer et al., 2003
Yue & Ding, 2006
No clear methods section with mineralogy or other sample metadata; no mention of standards used in measurements
No available English translation