

SUPPLEMENTARY METHODS 2: CARBON-14, BERYLLIUM-10, AND ALUMINUM-26 MEASUREMENTS

Quartz isolation and purification (all samples)

We prepared purified quartz samples for cosmogenic nuclide analysis at both Tulane University and Imperial College London; quartz samples measured for in situ ^{14}C were prepared at the former, whilst purified quartz samples measured for ^{10}Be and ^{26}Al were prepared, in full or in part, at both. First, each ~10 cm bedrock core subsample was disaggregated using a jaw crusher and disk mill, and then sieved to isolate the 125 - 710 μm grain size fraction. We opted for a large grain size range given the small size of the samples. Next, the sieved samples were magnetically separated to isolate the nonmagnetic grains. The nonmagnetic fraction of samples were split; one split for in situ ^{14}C remained at Tulane University (see below) and the other split for ^{10}Be and ^{26}Al was sent to Imperial College London.

Quartz isolation and purification for ^{10}Be and ^{26}Al sample splits

In the CosmIC Laboratory at Imperial College London, we first used froth flotation (Herber, 1969) to remove feldspars and micas, and concentrate quartz, from the ^{10}Be and ^{26}Al splits. After froth flotation, the quartz concentrate fractions were further purified using the methods of Kohl and Nishiizumi (1992). Quartz concentrate fractions were etched three times overnight (9 h) in an ultrasonic bath in 1 % HF/HNO_3 at 90 $^{\circ}\text{C}$ to preferentially dissolve non-quartz minerals and remove meteoric ^{10}Be adsorbed to the outside of quartz grains. The final quartz samples were assayed using an Agilent 5100 SVDV inductively coupled plasma optical emission spectrometer (ICP-OES) to verify mineral purity and quantify the expected cation load and, in turn, optimize the performance of ion exchange chromatography during Be and Al extraction and purification. Al concentrations ranged from 13-264 ppm and Ti concentrations ranged from 8-30 ppm in the final quartz samples; the total cation load estimated for the samples ranged from 0.3-0.7 meq, which was approximately 10 % of the total capacity of the cation exchange chromatography resin (see below).

Beryllium and aluminum extraction and purification

We performed isotope dilution chemistry for ^{10}Be and ^{26}Al analyses in the CosmIC Laboratory at Imperial College London using methods similar to Corbett et al. (2016). We dissolved between 9 and 23 g of pure quartz for beryllium and aluminum extraction and purification (Tables S3, S4). Approximately 0.25 mg of low-background ^9Be carrier ($775.23 \pm 3.16 \mu\text{g Be g}^{-1}$; $^{10}\text{Be}/^9\text{Be} = 2.6 \times 10^{-16} \pm 6.9 \times 10^{-17}$) and 2200 mg of ^{27}Al carrier ($1033.56 \pm 8.30 \mu\text{g Al g}^{-1}$; $^{26}\text{Al}/^{27}\text{Al} = 1.3 \times 10^{-15} \pm 3.5 \times 10^{-16}$) was added to each sample prior to dissolution (Tables S3, S4). Al carrier was added to achieve ~2.5 mg of total Al (i.e., total of native Al and carrier) in each sample. Quartz was dissolved using concentrated HF and HNO_3 in the presence of the carriers. Two procedural blanks (carrier only) and a CRONUS-A reference material replicate were processed for every 9 core samples (see below). After the quartz samples were completely dissolved, which ensured that the isotopic ratios of $^{10}\text{Be}/^9\text{Be}$ and $^{26}\text{Al}/^{27}\text{Al}$ equilibrated between the quartz and carriers, a ~2% aliquot of the bulk dissolution solution was removed and its fraction of the total solution gravimetrically calculated. We prepared this aliquot for ICP-OES analysis to quantify the total amount of Al and Be in the solution and verify that native Be was not present in the samples. The sample for ICP-OES analysis was matrix matched to the standard solutions, and the analyses calibrated using a 6 point calibration that spanned the full range of concentrations for the aliquot samples. After the aliquot

was removed, the remaining dissolution solution was dried down to volatilize silicon fluoride, and fumed to dryness three times with HClO_4 to evaporate residual fluorides and convert to soluble chlorides. Beryllium and aluminum were isolated and purified using successive anion and cation ion exchange chromatography following methods similar to Corbett et al. (2016), but using 2 ml of anion resin, 20 ml of cation resin, and eluting a third cation column fraction to remove Mg from the Al fraction. The final purity and yield of the Be fractions was verified by ICP-OES. The Be and Al fractions were then dried down, precipitated with ammonia hydroxide at pH8 as a beryllium and aluminum hydroxide complex, and ignited to beryllium and aluminum oxide at 750°C in a muffle furnace. Finally, these beryllium and aluminum oxides were mixed with niobium and silver powder, respectively, in a 1:1 molar ratio, and loaded into copper cathodes prior to isotopic measurement.

Accelerator Mass Spectrometry (AMS) of ^{10}Be and ^{26}Al

$^{10}\text{Be}/^9\text{Be}$ and $^{26}\text{Al}/^{27}\text{Al}$ isotope ratios were measured at the Centre for Accelerator Science, Australian Nuclear Science and Technology Organisation (ANSTO) (Wilcken et al., 2022). $^{10}\text{Be}/^9\text{Be}$ AMS measurements were normalized to the primary standard KN-5-3 with a nominal value of 6.320×10^{-12} (Nishiizumi et al., 2007). $^{26}\text{Al}/^{27}\text{Al}$ AMS measurements were normalized to the primary standard KN-4-2 with a nominal value of 3.096×10^{-11} (Nishiizumi, 2004). Two secondary standards were run as unknowns to confirm the linearity, accuracy, and precision of the measurements for both isotopes. The $^{10}\text{Be}/^9\text{Be}$ and $^{26}\text{Al}/^{27}\text{Al}$ measured ratios were reduced to the number of total ^{10}Be and ^{26}Al atoms in each sample using the total amount of beryllium and aluminum in each sample from gravimetric and ICP-OES measurements, respectively.

We measured the $^{10}\text{Be}/^9\text{Be}$ ratio for 37 samples across all bedrock cores (19-KP-H1, 4, 5, and 6) and seven procedural blanks (Table S4). An additional repeat measurement was made for splits of H6-surface and H1-105-114 (H6-surfaceR and H1-105-114R, see below). We focused on core H1 for the $^{26}\text{Al}/^{27}\text{Al}$ ratio measurements, with 10 samples (and five procedural blanks), as well as repeat measurements for splits of the same two aforementioned samples (Table S3).

Blank correction

Beryllium procedural blanks (carrier-only samples processed with the rest of the samples) range from approximately 18,000 to 39,000 total ^{10}Be atoms. We also measured the $^9\text{Be}/^{10}\text{Be}$ ratio of two ~2.5 mg aliquots of ^9Be carrier (precipitated prior to sample drydown and ignition without any column chemistry). Be carrier blank measurements (mean 4546 ± 3040 (1 SD) atoms) are significantly lower than the Be procedural blanks (Table S4), which indicates that the ^{10}Be background is sourced from the laboratory process (e.g. exposure to the air in the laboratory or the chemical reagents used) and not solely from the Be carrier. Therefore, ^{10}Be backgrounds were corrected using the arithmetic mean and standard deviation (30287 ± 8456 atoms) of the procedural blanks, with uncertainties in the sample and average blank propagated in quadrature. Aluminum procedural blanks range from ~26,000 to 90,000 total ^{26}Al atoms (mean 48225 ± 24268 (1 SD) atoms). We also measured the $^{26}\text{Al}/^{27}\text{Al}$ ratio of two ~2.5 mg aliquots of ^{27}Al carrier (Table S3). Al carrier blank measurements (mean 75882 ± 31493 (1 SD) atoms) are indistinguishable from Al procedural blanks, indicating that our ^{26}Al blank is sourced solely from the Al carrier (unlike ^{10}Be procedural blanks). Thus, we corrected ^{26}Al backgrounds using the weighted mean (39017 atoms) and standard error (5606 atoms) rather than the arithmetic mean and standard deviation, with uncertainties in sample and weighted mean blank propagated in quadrature. The choice of background correction method for either nuclide does not significantly alter the concentrations, and thus any inferences made, beyond measurement uncertainties.

Reproducibility of ^{10}Be and ^{26}Al analyses

In addition to the inherent reproducibility test of depth profiles of cosmogenic nuclide measurements, we verified the reproducibility of our ^{10}Be and ^{26}Al analyses in two ways. First, we replicated measurements of ^{10}Be and ^{26}Al in splits of the single sample collected from borehole 19-KP-H6 ("H6-surface"). As noted in the main text, cosmogenic-nuclide concentrations in the H6 sample are not unambiguously interpretable because it is not known whether this sample is bedrock or an englacial clast, so they are not discussed further. However, it has identical lithology to bedrock cored at the other sites, so was used as a sample for reproducibility tests. The two H6-surface replicates are indistinguishable within uncertainties: ^{10}Be concentrations vary by 5.2 % and ^{26}Al concentrations by 7.5 % (Tables S3, S4).

Second, we replicated multiple samples of the reference quartz material CRONUS-A (Jull et al., 2015). A ~0.5 g aliquot of CRONUS-A quartz was processed alongside each batch of core samples. To avoid contaminating other samples, CRONUS-A aliquots were the final sample in each batch to undergo each step of the isotope dilution chemistry procedure. We measured ^{10}Be in five ($3.52 \times 10^7 \pm 5.01 \times 10^5$ (1 SD) atoms g^{-1}) and ^{26}Al in three ($1.46 \times 10^8 \pm 1.70 \times 10^6$ (1 SD) atoms g^{-1}) CRONUS-A replicates (Tables S3, S4). Our ^{10}Be and ^{26}Al measurements are ~3 and 2 % different than the reported CRONUS-A values, respectively, and within the reported uncertainties on the reference value ($^{10}\text{Be} = 3.42 \pm 0.1 \times 10^7$ atoms g^{-1} , $^{26}\text{Al} = 1.43 \times 10^8 \pm 7 \times 10^6$ atoms g^{-1} , Jull et al., 2015), which verifies the reproducibility of our methods and ^{10}Be and ^{26}Al measurements and gives us confidence that our uncertainty estimates are reliable.

Quartz isolation and purification for in situ ^{14}C sample splits

To avoid any possible modern carbon contamination from froth flotation (c.f. Nichols & Goehring, 2019), we began etching our samples immediately after magnetic separation. Samples were etched for at least one period (24 h) on a shaker table in 5% HF/ HNO_3 and then in an ultrasonic bath in 1% HF/ HNO_3 until pure quartz remained (5-11 days, depending on the sample). Upon inspection of dry samples under microscope, we observed pyrite that persisted through sample etching, and used an additional 24-48 hour soak in concentrated HNO_3 to rid samples of all pyrite.

Carbon extraction, IRMS, AMS

We used 5 g of clean quartz from each sample to extract in situ ^{14}C using the Tulane University Carbon Extraction and Graphitization (TU-CEGS) system. Here we followed modified methods of Goehring et al. (2019), by which quartz was step heated (500°C for 30 min, 1100°C for 120 min) in a lithium metaborate (LiBO_2) flux under a high-purity O_2 atmosphere. Following combustion, gas was released from the tube furnace and converted to CO_2 via hot-quartz-bed oxidation. All CO_2 produced in this process was cryogenically purified, quantified manometrically, and diluted with ^{14}C -free CO_2 to a measurable sample size (115 $\mu\text{g C}$). A small aliquot (2-3 $\mu\text{g C}$) was removed for $\delta^{13}\text{C}$ analysis by GasBench-IRMS at the UC Davis Isotope Facility, and all results are reported relative to VPDB (Table S5). Remaining CO_2 was graphitized using standard H_2 reduction methods over an Fe catalyst. We sent all graphite samples to the National Ocean Sciences Accelerator Mass Spectrometry (NOSAMS) facility for $^{14}\text{C}/^{12}\text{C}$ ratio measurements. AMS data reporting does not account for normalizations needed for in situ ^{14}C data reporting, so we follow data reporting and standardization methods outlined in Hippe and Lifton (2014) to convert $^{14}\text{C}/^{12}\text{C}$ ratios to $^{14}\text{C}/\text{C}_{\text{total}}$ (Table S5).

Blank correction

During the extraction of samples included in this study, we measured 50 procedural blanks to characterize the level (^{14}C atoms) and repeatability of the TU-CEGS process blank following methods described in Goehring et al. (2019). The observed distribution of blanks is positively skewed, so a conventional blank correction approach assuming a normal uncertainty distribution would (i) underestimate the likelihood of observing anomalously high concentrations in samples, and (ii) imply a significant probability that the blank is less than zero, which is impossible. Occasional measurements of impossible ^{14}C concentrations above production-decay equilibrium values in published data sets (e.g., Nichols et al., 2019; Akçar et al., 2020; Hillebrand et al., 2021) also imply a long-tailed process blank distribution. Thus, we approximated the frequency distribution of blanks using a lognormal distribution, which has the desired properties of being positively skewed, long-tailed, and always greater than zero, fitted to the observed distribution (Fig. 1). We then used a Monte Carlo simulation to perform a blank correction for sample measurements. We first generate a large number of normally distributed values for the total number of ^{14}C atoms observed in a sample analysis (N_{obs}), with standard deviation given by propagated uncertainties from AMS measurement and manometry. We then draw an equal number of values for the process blank (N_b) from the fitted lognormal distribution, and compute the true number of atoms in the sample $N_{true} = N_{obs} - N_b$. Values of N_{true} less than zero are discarded as unphysical. The resulting distribution for N_{true} is thus also not normal, so we describe the resulting blank-corrected ^{14}C concentrations by the median bound by 68% and 95% confidence intervals.

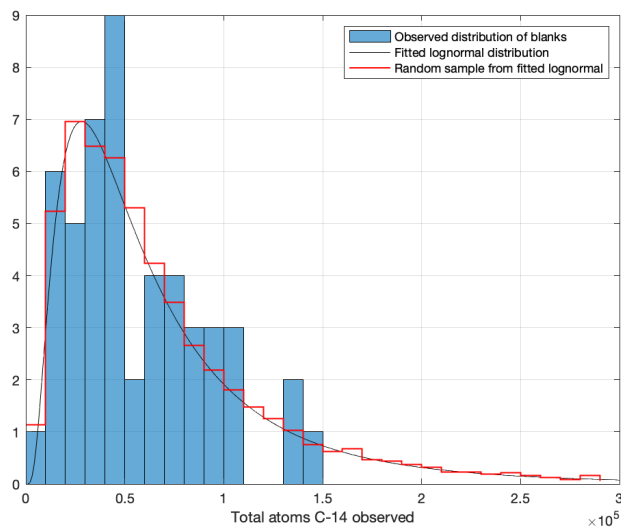


Figure 1. Distribution of 50 full process blanks from Tulane ^{14}C extraction line. The black curve is a lognormal distribution fitted to the observed distribution for purposes of blank subtraction from sample measurements. The red line is a histogram of a representative random sample drawn from the lognormal distribution.

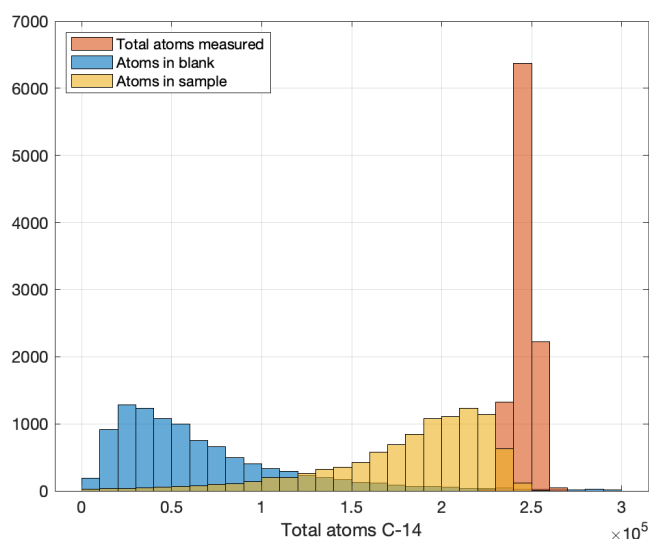


Figure 2. Example of Monte Carlo procedure for blank correction. The red histogram is a random sample ($n = 10,000$) drawn from a normal distribution representing total atoms measured. The blue histogram is a random sample drawn from the lognormal distribution fit to the measured process blank distribution. The yellow histogram is the difference of these two, which represents the uncertainty distribution for the true number of atoms in the sample.

References

- Corbett, L.B., Bierman, P.R. and Rood, D.H., 2016. An approach for optimizing in situ cosmogenic ^{10}Be sample preparation. *Quaternary Geochronology*, 33, pp.24-34.
- Goehring, B.M., Wilson, J. and Nichols, K., 2019. A fully automated system for the extraction of in situ cosmogenic carbon-14 in the Tulane University cosmogenic nuclide laboratory. *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, 455, pp.284-292.
- Herber, L.J., 1969. Separation of feldspar from quartz by flotation. *American Mineralogist: Journal of Earth and Planetary Materials*, 54(7-8), pp.1212-1215.
- Hippe, K. and Lifton, N.A., 2014. Calculating isotope ratios and nuclide concentrations for in situ cosmogenic ^{14}C analyses. *Radiocarbon*, 56(3), pp.1167-1174.
- Jull, A.T., Scott, E.M. and Bierman, P., 2015. The CRONUS-Earth inter-comparison for cosmogenic isotope analysis. *Quaternary Geochronology*, 26, pp.3-10.
- Kohl, C.P. and Nishiizumi, K., 1992. Chemical isolation of quartz for measurement of in-situ-produced cosmogenic nuclides. *Geochimica et Cosmochimica Acta*, 56(9), pp.3583-3587.
- Nichols, K.A. and Goehring, B.M., 2019. Isolation of quartz for cosmogenic in situ ^{14}C analysis. *Geochronology*, 1(1), pp.43-52.
- Nishiizumi, K., Imamura, M., Caffee, M.W., Southon, J.R., Finkel, R.C. and McAninch, J., 2007. Absolute calibration of ^{10}Be AMS standards. *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, 258(2), pp.403-413.
- Nishiizumi, K., 2004. Preparation of ^{26}Al AMS standards. *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms*, 223, pp.388-392.

Wilcken, K.M., Codilean, A.T., Fülöp, R.H., Kotevski, S., Rood, A.H., Rood, D.H., Seal, A.J. and Simon, K., 2022. Accelerator mass spectrometry of ^{10}Be and ^{26}Al at low nuclide concentrations. *Geochronology*, 4(1), pp.339-352.