How old are the Jack Hills really? The case for contamination of bedrock by zircon grains in transported regolith.

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Supplemental Data

Figure S1

A: Zircon U–Pb age spectra for individual samples generated from subsets of ‘n’ near-concordant analyses, variable vertical scale; B: Multi-dimensional scaling diagram of detrital zircon Concordia ages; C: Kernel density plots of the near-concordant age distributions.

Analytical Methods

Sample selection and preparation

Alluvium samples were collected by hand from minor modern drainage channels at the locations shown in Figures 1B and 1C. Sample 37C was obtained by gathering the loose soil particles attached to a meta-conglomerate rock sample (location shown in Fig. 1B), liberated with a clean nylon brush.

Zircon grains were separated using a Jasper Canyon Research concentrating table, followed by standard magnetic and heavy liquid separation. The entire (>20 µm) non-magnetic heavy mineral fraction of each sample was dump-mounted and polished in one-inch epoxy rounds, without any pre-selection or hand-picking of individual grains. The mount surface was polished to approximately half grain thickness.

TIMA and spot selection

The composition of each heavy mineral fraction was characterized using the TESCAN Integrated Mineral Analyzer (TIMA) at the John de Laeter Centre, Curtin University, Perth, Australia. The TIMA uses the energy dispersive spectrum (EDS) of individual grains referenced against a library of known spectra to identify individual minerals automatically.

Following TIMA characterisation, the mounts were loaded into the ICP-MS sample holder and between 580 and 1230 individual zircon grains per sample were selected at random for analysis. No imaging of zircon grains was undertaken prior to analysis to characterise internal zoning. The very high number of analyses per sample coupled with the absence of grain pre-selection and imaging was a deliberate strategy to eliminate sampling bias in the resulting age spectra.

Data acquisition

Fast laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) was performed at the GeoHistory Facility, John de Laeter Centre, Curtin University, Perth, Australia. Ablations utilized an ASI RESOLUTION-SE 193 nm excimer laser controlled by GeoStar µGIS™ software. Laser fluence was calibrated above the sample cell using a hand-held energy meter, and subsequent analyses were performed in constant energy mode. The Laurin Technic S155 sample cell was flushed by ultrahigh purity He (320 mL/min) and N₂ (1.2 mL/min), both of which were passed through inline gold sand Hg traps. High purity Ar was used as the ICP-MS carrier gas (flow rate ~1 L/min). All measurements were performed using an Agilent 8900 QQQ quadrupole ICP-MS operated in single quad mode. Each analytical session consisted of initial gas flow and ICP-MS ion lens tuning for sensitivity with the signal smoothing device ('squid') connected. Additional tuning adjusted flow parameters for robust plasma conditions ($^{238}\text{U}/^{232}\text{Th} \sim 1$; $^{206}\text{Pb}/^{238}\text{U} \sim 0.2$; and $^{238}\text{UO}/^{238}\text{U} < 0.004$). Finally, pulse-analog (P/A) conversion factors were determined on NIST 610 reference glass by varying laser spot sizes and/or laser repetition rate to yield 1–2 Mcps per element. After tuning, the signal smoothing device was removed and the laser was connected to the mass spectrometer via a shorter (~60 cm) length of Teflon tubing. Using this setup, signal wash-in and wash-out times were typically around 200 and 600 ms, respectively. Following Chew et al. (2019), laser parameters were chosen as 20 µm spot diameter, 50 Hz repetition rate, and on-sample laser energy of 2 Jcm⁻². The Geostar software sequence for fast pulse analysis consisted of a 3 s delay after the TTL pulse for baseline acquisition, 4 s ablation, and 7 s delay for the 8900 to complete data handling and return to a 'wait for trigger' state. ⁹¹Zr, ²⁰⁶Pb, ²⁰⁷Pb, ²⁰⁸Pb, ²³²Th, and ²³⁸U were collected with dwell times of 3, 15, 20, 6, 6, and 9 ms, respectively, for a total acquisition time of 10s, and a duty cycle (data acquisition time/total time) of about 66%.

Data reduction and statistics

Analytical sessions consisted of up to 2175 unknown zircon analyses, 250 reference zircon analyses and 40 analyses of NIST612 reference glass. The time-resolved mass spectra were reduced using the 'U–Pb Geochronology' data reduction scheme in Lolite 4.3.5.3 (Paton et al., 2011 and references therein). The dating reference materials used in this study were primary reference zircons GJ-1 (608.53 ± 0.37 Ma; Jackson et al., 2004) and OG1 (3465.4 ± 0.6 Ma; Stern et al., 2009); secondary reference zircons 91500 (1062.4 ± 0.4 Ma; Wiedenbeck et al., 1995) and Maniitsoq (3008.7 ± 0.72 Ma; Marsh et al., 2019). Given the wide age range of age for the analyzed unknown zircons, the data were reduced using GJ-1 for those with $^{207}\text{Pb}/^{206}\text{Pb}$ age < 2.5 Ga, and OG1 for those with $^{207}\text{Pb}/^{206}\text{Pb}$ age > 2.5 Ga, the ablation response of OG1 being more appropriate for the crystal state of Archean age zircon. The mean age ratios of the secondary reference analyses for each session are within 2% of the accepted ID-TIMS ages, as per the following table.

Table of analyses of secondary reference materials

<i>Session</i>	<i>Samples</i>	<i>Pb/U age using GJ-1 as 1° std</i>	<i>Pb/Pb age using OG1 as 1° std</i>
1	10, 25	91500 1062 ± 20 Ma MSWD 0.57, n = 50/50	Maniitsoq 3000 ± 27 Ma MSWD 0.68, n = 50/50
2	28, 35	91500 1057 ± 19 Ma MSWD 0.54, n = 50/50	Maniitsoq 3005 ± 34 Ma MSWD 0.92, n = 50/50
3	37C	91500 1041 ± 23 Ma MSWD 1.15, n = 20/21	Maniitsoq 3006 ± 33 Ma MSWD 0.60, n = 24/25

Because the fast pulse protocol utilised here does not include pre-ablation cleaning pulses to remove possible surface contamination, an assessment was undertaken previously by Brennan et al. (2021), comparing the Pb isotope data from fast pulse analyses to a corresponding 'conventional' LA-ICP-MS data set collected on the same grains, reference materials and instrumentation. This comparison of reference zircon data in inverse Concordia space did not reveal any discernible systematic trends to indicate the presence of additional surface-derived common lead associated with the fast pulse protocol (Brennan et al., 2021), giving confidence that the method is well suited to characterizing the age distribution of a large sample pool. Statistically, the fast ablation sample data were somewhat more scattered, but with no change to the indicated amount of discordance (Brennan et al., 2021).

Data filtering

Prior to production of age spectra, each dataset was treated with the following methodology to ensure consistency, reproducibility, and comparability. The data were first filtered for isotopic discordance, expressed as $((^{207}\text{Pb}/^{206}\text{Pb} \text{ age} - ^{206}\text{Pb}/^{238}\text{U} \text{ age}) / (^{207}\text{Pb}/^{206}\text{Pb} \text{ age}) * 100)$, with only those data falling within the $\pm 10\%$ discordance range used in the generation of kernel density estimation plots. Between 55 and 70% of analyses were discarded due to their discordance. Additionally, the counts per second on the ^{91}Zr peak were assessed, with any spots displaying low count rates assumed to be not 100% zircon and thus removed from subsequent calculations. All age calculations use Concordia ages as defined by Ludwig (1998), to avoid swapping between ratios over the wide age range of material considered.

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