Geochronological constraints on Neoproterozoic rifting and onset of the Marinoan glaciation from the Kingston Peak Formation in Death Valley, California: SUPPLEMENTARY MATERIAL

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In this supplement, we detail the analytical methodology used for this study, and provide compiled geochronology data and references from Figure 1, U-Pb geochronology data tables, and field and petrographic photographs.

METHODS

Field work

Geologic mapping of Proterozoic strata throughout the central Panamint Range was conducted between 2015-2018 on 1:10,000 scale topographic maps, and three stratigraphic sections were measured in the vicinity of Pleasant Canyon to create the composite section published here (Figs. 1, 2). Sample L1502 was collected from discontinuous lenses of a 0.2 m-thick light gray fine-grained laminated clastic unit in the Thorndike submember of the Kingston Peak Formation at 36.02063°N 117.08113°W (Figs. 2, SM2C,F,G). Due to its very fine-grained character (mud to silt grain size), it is difficult to interpret petrographically if this is an epiclastic deposit or a reworked tuff bed. The single ID-TIMS age population of the Cryogenian grains from this sample may indicate an eruptive age (Fig. 3A), but it is conservatively interpreted as a maximum depositional age. Sample L1703B was collected from a milky intermediate to felsic horizon containing pillow lava fragment breccia within otherwise mafic volcanic flows in the Limekiln Spring Member of the Kingston Peak Formation at 36.02385°N 117.10602°W (Figs. 2, SM2A,B,D,E).

Mineral separation, annealing, and imaging

Sample L1502 from the Thorndike submember was collected three times due to low zircon yield (L1502, L1502B, L1502C). Weathering rinds were trimmed off of L1502A with a rock saw prior to crushing. Sample L1703B from the Limekiln Spring Member was collected once. For all four samples, zircon grains were separated using a sledge, disc mill or shatterbox, 500 µm sieve, Wilfley shaker table, hand magnet, Frantz magnetic separator, and methylene iodide heavy liquid. Heavy mineral separates were annealed at 900°C for >48 hours in a muffle furnace.

Zircon grains were mounted in epoxy resin and polished. At Boise State University (BSU), zircon grains from all L1502 samples were imaged with a cathodoluminescence (CL) detector using a JEOL JSM-300 scanning electron microscope (SEM) in combination with a Gatan MiniCL. At University of California—Santa Barbara (UCSB), zircon grains from L1703B were imaged using a Centaurus CL detector attached to an FEI Quanta 400f field-emission SEM.

LA-ICPMS analyses

At UCSB, zircon U-Pb geochronology LA-ICPMS analyses of sample L1703B were conducted with a Photon Machines Excite 193 nm Excimer laser coupled with a Nu Plasma 3D MC-ICPMS and Agilent 7700X quadrupole ICPMS, following the methods of Kylander-Clark et al. (2013). Zircons were ablated with a 25 μm laser spot using fluence and pulse rates of 1 J/cm^2 and 4 Hz, respectively, during a 35 second analysis (2 shots pre-ablation, 20 sec wash out, 15 sec ablation). The primary standards were 91500 (Wiedenbeck et al., 1995) and GJ-1 (Jackson et al., 2004) for calibration of age and trace element composition, respectively, and Plešovice was used as a secondary age standard (Sláma et al., 2008). Bias and drift in U-Pb ratios were corrected using *Iolite* (Paton et al., 2010), following procedures detailed in Kylander-Clark et al. (2013).

At BSU, zircon U-Pb geochronology laser ablation-inductively coupled mass spectrometry (LA-ICPMS) analyses of sample L1502 were conducted with a ThermoElectron X-Series II quadrupole ICPMS and New Wave Research UP-213 Nd:YAG UV laser ablation system. Zircons were ablated with a 25 μm (before August 2019) or 16 μm (starting August 2019) laser spot using fluence and pulse rates of 5 J/cm^2 and 10 Hz, respectively, during a 45 second analysis (15 sec gas blank, 30 sec ablation) excavating a 25 μm deep pit. Ablated material was carried to the nebulizer flow of the plasma by a 1.2 L/min He gas stream. Total sweep duration is 950 ms, and quadrupole dwell times were 5 ms for Si and Zr, 40 ms for ^{202}Hg , ^{204}Pb , ^{208}Pb , ^{232}Th , and ^{238}U , 80 ms for ^{206}Pb , 200 ms for ^{49}Ti and ^{207}Pb , and 10 ms for all other HFSE and REE. Background count rates were obtained prior to each spot analysis and subtracted from the raw count rate. For concentration calculations, background-subtracted count rates were internally normalized to ^{29}Si and calibrated with the primary standards NIST SRM-610 and -612 glasses. Ablation pits that intersected mineral inclusions were identified based on Ti and P excursions, and associated sweeps were discarded. U-Pb dates from these analyses were retained if U-Pb ratios appeared to have been unaffected by the inclusions. Mass 204 signals were typically indistinguishable from zero following subtraction of Hg backgrounds, and dates are thus reported without common Pb correction.

For U-Pb and $^{207}\text{Pb}/^{206}\text{Pb}$ dates, instrumental fractionation of the background-subtracted ratios was corrected and dates were calibrated with respect to interspersed measurements of standards and reference materials. Standards were used to monitor time-dependent instrumental fractionation based on 2 analyses every 10 analyses of unknown zircons. Radiogenic isotope ratio and age error propagation for all analyses includes uncertainty contributions from counting statistics and background subtraction. These uncertainties are the local standard deviations of the polynomial fits to the interspersed primary standard measurements versus time for the time-

dependent, relatively larger U-Pb fractionation factor, and the standard errors of the means of the consistently time-invariant and smaller $^{207}\text{Pb}/^{206}\text{Pb}$ fractionation factor. These uncertainties are 0.7-1.7% (average of 1.1%) (2s) for $^{206}\text{Pb}/^{238}\text{U}$ and 0.2-0.8% (average of 0.6%) (2s) for $^{207}\text{Pb}/^{206}\text{Pb}$. Errors on dates from individual analyses are given at 2s, and zircon LA-ICPMS metadata and data are presented in Table SM2. Kernel density estimation and Wetherill concordia plots (Fig. 3) were created using *IsoplotR* (Vermeesch, 2018).

ID-TIMS Analyses

At BSU, single zircon U-Pb geochronology chemical abrasion-isotope dilution-thermal ionization mass spectrometry (CA-ID-TIMS) analyses were conducted on zircon grains from L1502 and L1703B based on LA-ICPMS screening, using methods modified after Mattinson (2005). Single annealed zircons were removed from epoxy mounts and transferred to 3 ml Teflon PFA beakers, loaded into 300 μl Teflon PFA microcapsules with 120 μl of 29 M HF, and partially dissolved for 12 hours at 190°C. Zircons were returned to 3 ml Teflon PFA beakers, rinsed in ultrapure H_2O , immersed in 3.5 M HNO_3 , ultrasonically cleaned for one hour, and fluxed for 1 hour at 80°C. The HNO_3 was removed and zircons were rinsed twice in ultrapure H_2O , then reloaded into Teflon PFA beakers and spiked with BSU1B tracer solution with a calibration of $^{235}\text{U}/^{205}\text{Pb} = 77.93$ and $^{233}\text{U}/^{235}\text{U} = 1.007066$. Zircons were dissolved in 120 μl of 29 M HF for 48 hours at 220°C, dried to fluorides, and dissolved in 6 M HCl for 12 hours at 180°C. Uranium and Pb were separated using an HCl-based anion-exchange chromatographic procedure (Krogh, 1973), eluted together and dried with 2 μl of 0.05 N H_3PO_4 .

Uranium and Pb were loaded on a single outgassed Re filament in 5 μl of a silica-gel/ H_3PO_4 mixture (Gerstenberger and Haase, 1997), and U and Pb isotopic measurements made on a GV IsotopX Phoenix multicollector thermal ionization mass spectrometers equipped with an ion-counting Daly detector. Lead isotopes for analyses with smaller amounts of radiogenic Pb were measured by peak-jumping all isotopes on the Daly detector for 150 cycles, and corrected for $0.16 \pm 0.06\%$ /a.m.u. (2s) mass fractionation. Lead isotopes for analyses with larger amounts of radiogenic Pb were measured by a Faraday-Daly routine that cycles 150-200 times between placing mass 204 in the axial Daly collector and masses 205-208 on the H1-H4 Faraday detectors to placing mass 205 in the axial Daly and masses 206-208 in the H1-H3 Faradays, providing real-time Daly gain correction. These results were corrected for $0.10 \pm 0.06\%$ /a.m.u. (2s) mass fractionation. Transitory isobaric interferences due to high-molecular weight organics, particularly on ^{204}Pb and ^{207}Pb , disappeared within approximately 30 cycles. The ionization efficiency of each Pb isotope averaged 10^4 cps/pg. Linearity (to $\geq 1.4 \times 10^6$ cps) and the associated deadtime correction of the Daly detector were monitored by repeated analyses of NBS982. Uranium was analyzed as UO_2^+ ions in static Faraday mode on 10^{11} or 10^{12} ohm resistors for 200-300 cycles, and corrected for isobaric interference of $^{233}\text{U}^{18}\text{O}^{16}\text{O}$ on $^{235}\text{U}^{16}\text{O}^{16}\text{O}$ with an $^{18}\text{O}/^{16}\text{O}$ of 0.00206. Ionization efficiency averaged 20 mV/ng of each U isotope. Uranium mass fractionation was corrected using the known $^{233}\text{U}/^{235}\text{U}$ ratio of the tracer solutions.

CA-ID-TIMS U-Pb dates and uncertainties were calculated using the algorithms of Schmitz and Schoene (2007) and U decay constants recommended by Jaffey et al. (1971). $^{206}\text{Pb}/^{238}\text{U}$ ratios and dates were corrected for initial ^{230}Th disequilibrium using a $\text{Th}/\text{U}[\text{magma}] = 3.0 \pm 0.3$, resulting in an increase in the $^{206}\text{Pb}/^{238}\text{U}$ dates of ~ 0.09 Ma. All common Pb in analyses was attributed to laboratory blank and subtracted based on the measured laboratory Pb isotopic composition and associated uncertainty. Uranium blanks were estimated at 0.013 pg. CA-ID-TIMS weighted mean $^{206}\text{Pb}/^{238}\text{U}$ dates were calculated from equivalent dates (pof > 0.05) using *Isoplot 3.0* (Ludwig, 2003). Errors on the weighted mean dates are given as $\pm x / y / z$, where x is the internal error based on analytical uncertainties only, including counting statistics, subtraction of tracer solution, and blank and initial common Pb subtraction, y includes the tracer calibration uncertainty propagated in quadrature, and z includes the ^{238}U decay constant uncertainty propagated in quadrature. Internal errors should be considered when comparing dates with $^{206}\text{Pb}/^{238}\text{U}$ dates from other laboratories that used the same EARTHTIME tracer solution or a tracer solution that was cross-calibrated using EARTHTIME gravimetric standards. Errors including the uncertainty in the tracer calibration should be considered when comparing dates with those derived from other geochronological methods using the U-Pb decay scheme. Errors including uncertainties in the tracer calibration and ^{238}U decay constant (Jaffey et al., 1971) should be considered when comparing our dates with those derived from other geochronological decay schemes. Errors for weighted mean dates and dates from individual grains are given at 2s, and U-Pb CA-ID-TIMS data are presented in Table SM3. Weighted mean plots (Fig. 3) were created using *IsoplotR* (Vermeesch, 2018).

SUPPLEMENTARY MATERIAL FIGURE CAPTIONS

Table SM1. Compiled geochronology data from the Cryogenian Period shown in Figures 1 and SM1.

Table SM2. L1502 and L1703B LA-ICPMS U-Pb data.

Table SM3. L1502 and L1703B CA-ID-TIMS U-Pb data.

Figure SM1. Regional and local stratigraphy, and compiled geochronology from the Cryogenian Period, corresponding to Figure 1. Numbered references are provided in Table SM1.

Figure SM2. Photographs of sampled horizons. Lens cap is 5.2 cm in diameter. Hammer is 33 cm in length. A) Pillow lava fragment breccia within a dacitic flow in volcanic horizon of the Limekiln Spring Member (see Stevenson et al., 2009). B) Felsic zone in volcanic horizon of the Limekiln Spring Member with eutaxitic texture (sample L1703B). C) Siltstone or possible reworked tuff bed within dolomite grainstone of the Thorndike submember (sample L1502). D, E) Photomicrographs of L1703B from volcanic rocks in Limekiln Spring Member showing mafic and felsic zonation in (D) plain polarized light and (E) cross polarized light. F) Polished

158 hand sample of L1502 showing fine laminations. G) Photomicrograph of L1502 showing silt-
159 size grains.

160 **Figure SM3.** Field photographs of syn-rift sedimentary features. A) Sedimentary breccia within
161 the lower Limekiln Spring Member composed primarily of clasts of Tonian Beck Spring
162 Dolomite. Geologist for scale. B) Alluvial conglomerate within lower Mountain Girl submember
163 composed of clasts of quartzite and basement gneiss. Lens cap is 5.2 cm in diameter. C)
164 Fanglomerate within Wildrose submember composed primarily of clasts of Beck Spring
165 Dolomite. Hand circled for scale. D) Olistostrome within the Wildrose submember containing
166 olistoliths of Beck Spring Dolomite. Pinyons and junipers are ~1-3 meters.

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Fig. S2

Fig. S3