Hodgin, E.B., et al., 2020, A link between rift-related volcanism and end-Ediacaran extinction? Integrated chemostratigraphy, biostratigraphy, and U-Pb geochronology from Sonora, Mexico: Geology, v. 49, https://doi.org/10.1130/G47972.1

- A link between rift-related volcanism and end-Ediacaran
- 2 extinction? Integrated chemostratigraphy, biostratigraphy, and
- ³ U-Pb geochronology from Sonora, Mexico: SUPPLEMENTAL

4 MATERIAL

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- 6 Eben B. Hodgin^{1*}, Lyle L. Nelson^{2*}, Corey J. Wall³, Arturo J. Barrón-Díaz⁴, Lucy C. Webb²,
- 7 Mark D. Schmitz³, David A. Fike⁵, James W. Hagadorn⁶, Emily F. Smith²
- 8 ¹Deptartment of Earth and Planetary Sciences, Harvard University, Cambridge, MA 02138 USA
- 9 ²Department of Earth and Planetary Sciences, Johns Hopkins University, Baltimore, MD 21218
- 10 USA
- ³Department of Geosciences, Boise State University, Boise, ID 83725 USA
- ⁴Departamento de Geología, Universidad de Sonora (UNISON), Hermosillo, 83000 Mexico
- ⁵Department of Earth and Planetary Sciences, Washington University, St. Louis, MO 63130 USA
- ⁶Department of Earth Sciences, Denver Museum of Nature & Science, Denver, CO 80205 USA
- 15
- 16 ^{*}ebenblake@berkeley.edu, lylelnelson@jhu.edu.
- 17
- 18 This Supplemental Material includes:
- 1) Section S1: Detailed methods (carbonate δ^{13} C and δ^{18} O isotopes, mineral separation 20 and imaging, LA-ICPMS and CA-ID-TIMS U-Pb zircon geochronology);
- and mininging, DATION with OATID TIMES OT 0 Zheon geoemonology),
- 2) Section S2: Diagenetically-altered tuffaceous deposits in marine carbonates;

22	3)	Three supplemental tables (carbonate δ^{13} C and δ^{18} O isotopes, LA-ICPMS U-Pb data,
23		CA-ID-TIMS U-Pb data); and
24	4)	Five supplemental figures (carbonate C-O isotope cross-plot, lithic tuff
25		photomicrographs, U-Pb Concordia and kernel density plots, 540 Ma zircon trace
26		element geochemistry, and zircon CL images).
27		

28 SECTION S1: DETAILED METHODS

29 Carbonate Stable Isotopes

Fist-sized carbonate samples were collected from measured stratigraphic sections, and fresh cut
surfaces were microdrilled for powder along laminations, avoiding alteration textures. Samples
from sections LV1801, LV1802, CC1801, CC1802, and CR1801 were analyzed at the Johns
Hopkins University, while samples from sections CR1705, CR1804, and CR1805 were analyzed
at Washington University in St. Louis.

35 At the Johns Hopkins University Department of Earth and Planetary Sciences Isotope Ratio 36 Mass Spectrometer Laboratory, carbonate samples were analyzed for carbon and oxygen isotopic compositions (δ^{13} C, δ^{18} O) using a GasBench II peripheral device coupled to a Thermo-Finnigan 37 38 MAT253 isotope ratio mass spectrometer (IRMS) in continuous-flow mode. Approximately 0.3 39 mg of sample carbonate powder reacted with 100% phosphoric acid in helium-purged vials at 40 30°C, overnight. Evolved CO₂ gas was then analyzed against tank CO₂ gas and isotopic results 41 normalized to V-PDB (Vienna-Pee Dee Belemnite) per mil (‰) scale using working in house 42 carbonate standards (ICM, Carrara Marble and IVA Analysentechnik, calcium carbonate) that are 43 calibrated against international standards NBS-18 and IAEA-603. A 30°C acid fractionation factor of -1.42‰ was added to normalized dolomite δ^{18} O values (Rosenbaum and Sheppard, 1986; Kim 44

45 et al., 2007). Standard deviation (1 σ) of δ^{13} C and δ^{18} O values for in house standards was <0.02‰ 46 and <0.14‰, respectively.

47 At Washington University in St. Louis, carbonate samples were prepared for carbon and oxygen isotopic analyses by dissolving $\sim 100 \ \mu g$ in 100% phosphoric acid (H₃PO₄) for at least 4 48 49 hours at 70°C. Sample vials were subsequently flushed with He and the evolved CO₂ was measured 50 on a Thermo Finnigan Gasbench II coupled to a Delta V Advantage IRMS. Carbon and oxygen isotopes are expressed in standard delta notation ($\delta^{13}C$, $\delta^{18}O$) in per mil (‰) as a deviation from 51 the V-PDB standard by calibration against NBS 19, NBS 20, and two in-house standards. A 70°C 52 acid fractionation factor of -1.21% was added to normalized dolomite δ^{18} O values (Rosenbaum 53 54 and Sheppard, 1986; Kim et al., 2007). From standard and replicate measurements, the 1σ error on δ^{13} C is <0.15‰ and δ^{18} O is <0.2‰. 55

56

57 Mineral Separation and Zircon Imaging

Zircon grains were separated from rock samples using a sledge, Retsch DM200 disc mill, 500 µm 58 59 sieve, and Wilfley Shaker Table at Harvard University. Separation proceeded using a Frantz 60 magnetic separator and methylene iodide heavy liquid at Massachusetts Institute of Technology. Heavy mineral separates were annealed at 900°C for >48 hours in a muffle furnace. Grains were 61 62 mounted in epoxy and polished. At the University of California Santa Barbara (USCB), a Centaurus cathodoluminescence (CL) detector attached to an FEI Quanta 400f field-emission 63 scanning electron microscope (SEM) was used to obtain CL images of polished and carbon-coated 64 65 grain mounts. At Boise State University (BSU), CL images of zircon were obtained with a JEOL 66 JSM-300 SEM in combination with a Gatan MiniCL.

68 LA-ICPMS U-Pb Zircon Geochronology

69 At UCSB, zircon U-Pb geochronology and trace element analysis was carried out by laser ablation 70 inductively coupled plasma mass spectrometry (LA-ICPMS) using a Photon Machines Excite 193 71 nm Excimer laser coupled with a Nu Plasma 3D MC-ICPMS and Agilent 7700X quadrupole 72 ICPMS, following the methods of Kylander-Clark et al. (2013). For each analysis, a 25 µm spot 73 size was ablated at 4 Hz for 15 seconds with a 20 second washout, following 2 shots of preablation, using a fluence of ~1 J/cm². The primary standards were 91500 (1062.4 \pm 0.4 Ma) 74 75 (Wiedenbeck et al., 1995) and GJ-1 (Jackson et al., 2004) for calibration of age and trace element 76 composition, respectively. Bias and drift in U-Pb ratios were corrected using the 77 program Iolite (Paton et al., 2010) following procedures detailed in Kylander-Clark et al. (2013). Plešovice was used as a secondary age standard $(337.13 \pm 0.37 \text{ Ma})$ (Sláma et al., 2008), and 78 vielded a 206 Pb/ 238 U weighted mean date of 334.8 ± 1.0 Ma (MSWD = 1.8, n = 10). This internal 79 80 uncertainty was not propagated for systematic biases.

81 At BSU, zircon was analyzed by LA-ICPMS using a ThermoElectron X-Series II quadrupole ICPMS and New Wave Research UP-213 Nd:YAG UV laser ablation system. 82 83 Analytical protocols, standard materials, and data reduction software developed at BSU were used 84 for acquisition and calibration of U-Pb dates and a suite of high field strength elements (HFSE) 85 and rare earth elements (REE). Zircon were ablated with a laser spot 16 µm wide using fluence and pulse rates of 5 J/cm² and 10 Hz, respectively, during a 45 second analysis (15 second gas 86 87 blank, 30 second ablation) excavating a pit 25 µm deep. Ablated material was carried to the 88 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 ²⁰²Hg, ²⁰⁴Pb, ²⁰⁸Pb, ²³²Th, and ²³⁸U, 80 89 ms for ²⁰⁶Pb, 200 ms for ⁴⁹Ti and ²⁰⁷Pb, and 10 ms for all other HFSE and REE. Background count 90

91 rates were obtained prior to each spot analysis and subtracted from the raw count rate for each 92 analyte. For concentration calculations, background-subtracted count rates were internally normalized to ²⁹Si and calibrated with the primary standards NIST SRM-610 and -612 glasses. 93 94 Ablation pits that intersected mineral inclusions were identified based on Ti and P excursions, and 95 associated sweeps were discarded. U-Pb dates from these analyses were retained if U-Pb ratios 96 appeared to have been unaffected by the inclusions. Mass 204 signals were typically 97 indistinguishable from zero following subtraction of Hg backgrounds, and dates are thus reported 98 without common Pb correction. The Ti-in-zircon thermometer was calculated using an average 99 TiO₂ activity value of 0.8 in crustal rocks (Watson et al., 2006).

100 Zircon U-Pb data were collected in laser ablation experiments on January 6th, 2019 at UCSB and on August 19th, 2019 at BSU. For U-Pb and ²⁰⁷Pb/²⁰⁶Pb dates, instrumental 101 102 fractionation of the background-subtracted ratios was corrected, and dates were calibrated with 103 respect to interspersed measurements of standards and reference materials. The primary standard 104 Plešovice zircon (Sláma et al., 2008) was used to monitor time-dependent instrumental 105 fractionation based on two analyses for every 10 analyses of unknown zircon. Secondary standards 106 were also analysed twice for every 10 unknowns, and a secondary correction was applied to the ²⁰⁶Pb/²³⁸U dates using Seiland (530 Ma) and Zirconia (327 Ma). 107

108 Radiogenic isotope ratio and age error propagation for all analyses includes uncertainty 109 contributions from counting statistics and background subtraction. These uncertainties are the local 110 standard deviations of the polynomial fits to the interspersed primary standard measurements 111 versus time for the time-dependent, relatively larger U-Pb fractionation factor, and the standard 112 errors of the means of the consistently time-invariant and smaller 207 Pb/ 206 Pb fractionation factor. 113 These uncertainties are 1-5% (average of 2%) (2 σ) for 206 Pb/ 238 U and 0.5-1.5% (average of 1%) (2σ) for ²⁰⁷Pb/²⁰⁶Pb. Errors on dates from individual analyses are given at 2σ, and zircon LAICPMS results are in Table S2.

116

117 CA-ID-TIMS U-Pb Zircon Geochronology

U-Pb dates were obtained by the chemical abrasion isotope dilution thermal ionization mass
spectrometry (CA-ID-TIMS) method from analyses composed of single zircon grains or fragments
of grains, modified after (Mattinson, 2005). Annealed zircon was removed from the epoxy mounts
based on LA-ICPMS data and CL image screening. Single grains or fragments were transferred to
3 ml Teflon PFA beakers, loaded into 300 µl Teflon PFA microcapsules with 120 µl of 29 M HF,
placed in a large-capacity Parr vessel, and partially dissolved for 12 hours at 190°C.

124 The zircon grains or fragments were returned to 3 ml Teflon PFA beakers, HF removed, 125 and rinsed in ultrapure H₂O, immersed in 3.5 M HNO₃, ultrasonically cleaned for one hour, and 126 fluxed at 80°C for one hour. The HNO₃ was removed and grains or fragments were rinsed twice 127 in ultrapure H₂O, then reloaded into Teflon PFA and spiked with Boise State BSU1B tracer solution with a calibration of $^{235}U/^{205}Pb = 77.93$ and $^{233}U/^{235}U = 1.007066$. Zircon was dissolved 128 129 in Parr vessels in 120 µl of 29 M HF at 220°C for 48 hours, dried to fluorides, and re-dissolved in 130 6 M HCl at 180°C overnight. Uranium and Pb were separated from the zircon matrix using an 131 HCl-based anion-exchange chromatographic procedure (Krogh, 1973), eluted together, and dried 132 with 2 μ l of 0.05 N H₃PO₄.

Uranium and Pb were loaded on a single outgassed Re filament in 5 μ l of a silica-134 gel/phosphoric acid mixture (Gerstenberger and Haase, 1997), and U and Pb isotopic 135 measurements made on a GV IsotopX Phoenix multicollector thermal ionization mass 136 spectrometer equipped with an ion-counting Daly detector. Pb isotopes for analyses with smaller

137 amounts of radiogenic Pb were measured by peak-jumping all isotopes on the Daly detector for 138 150 cycles, and corrected for $0.16 \pm 0.06/a$.m.u. (2 σ) mass fractionation. Pb isotopes for analyses 139 with larger amounts of radiogenic Pb were measured by a Faraday-Daly routine that cycles 150-140 200 times between placing mass 204 in the axial Daly collector and masses 205-208 on the H1-141 H4 Faraday detectors to placing mass 205 in the axial Daly and masses 206-208 in the H1-H3 142 Faradays, providing real-time Daly gain correction. These results were corrected for $0.10 \pm$ 143 0.06%/a.m.u. (2 σ) mass fractionation. Transitory isobaric interferences due to high-molecular weight organics, particularly on ²⁰⁴Pb and ²⁰⁷Pb, disappeared within approximately 30 cycles. The 144 ionization efficiency of each Pb isotope averaged 10^4 cps/pg. Linearity (to $\ge 1.4 \times 10^6$ cps) and the 145 146 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 147 for 200-300 cycles, and corrected for isobaric interference of ²³³U¹⁸O¹⁶O on ²³⁵U¹⁶O¹⁶O with an 148 ¹⁸O/¹⁶O of 0.00206. Ionization efficiency averaged 20 mV/ng of each U isotope. Uranium mass 149 fractionation was corrected using the known $^{233}U/^{235}U$ ratio of the tracer solutions. 150

151 CA-ID-TIMS U-Pb dates and uncertainties were calculated using the algorithms of 152 (Schmitz and Schoene, 2007), and U decay constants recommended by (Jaffey et al., 1971). 153 ${}^{206}\text{Pb}/{}^{238}\text{U}$ ratios and dates were corrected for initial ${}^{230}\text{Th}$ disequilibrium using a Th/U[magma] = 154 3.0 ± 0.3 , resulting in an increase in the ${}^{206}\text{Pb}/{}^{238}\text{U}$ dates of ~0.09 Ma. All common Pb in analyses 155 was attributed to laboratory blank and subtracted based on the measured laboratory Pb isotopic 156 composition and associated uncertainty. Uranium blanks were estimated at 0.013 pg.

157 CA-ID-TIMS weighted mean 206 Pb/ 238 U dates were calculated from equivalent dates (pof 158 >0.05) using Isoplot 3.0 (Ludwig, 2003). Errors on the weighted mean dates are given as $\pm x / y /$ 159 z, where x is the internal error based on analytical uncertainties only, including counting statistics, 160 subtraction of tracer solution, and blank and initial common Pb subtraction, y includes the tracer calibration uncertainty propagated in quadrature, and z includes the ²³⁸U decay constant 161 162 uncertainty propagated in quadrature. Internal errors should be considered when comparing our dates with ²⁰⁶Pb/²³⁸U dates from other laboratories that used the same EARTHTIME tracer solution 163 164 or a tracer solution that was cross-calibrated using EARTHTIME gravimetric standards. Errors 165 including the uncertainty in the tracer calibration should be considered when comparing our dates 166 with those derived from other geochronological methods using the U-Pb decay scheme (for example LA-ICPMS). Errors including uncertainties in the tracer calibration and ²³⁸U decay 167 168 constant (Jaffey et al., 1971) should be considered when comparing our dates with those derived from other decay schemes (for example ⁴⁰Ar/³⁹Ar, ¹⁸⁷Re-¹⁸⁷Os). Errors for weighted mean dates 169 170 and dates from individual grains are given at 2σ and shown in Table S3.

171

172 SECTION S2: DIAGENETICALLY-ALTERED TUFFACEOUS DEPOSITS IN MARINE 173 CARBONATES

174 There are millimetric domains preserved within two thin sections of the dated sandy dolostone 175 sample, CC1801-138, that are composed of a combination of finer grained (than the matrix) light 176 green clay minerals, dolomite rhombs, hematite, secondary quartz, and porosity (Fig. S2D-G). 177 These domains are interpreted as intraclasts that contain the same peloidal dolomite textures as 178 the matrix. Therefore, the porosity associated with these intraclasts (evidenced by sparry 179 dolomite and silica crystallization) may be due to dolomite replacement of primary calcite. 180 Alternatively, these features are also consistent with an originally tuffaceous vitroclastic matrix 181 of replaced and devitrified glass and secondary mineral infilling of original porosity. Similar 182 diagenetic features and textures associated with originally tuffaceous components have

183 previously been observed in marine carbonate rocks. For example, biotite rich layers in pelagic 184 carbonate successions from the Eocene-Oligocene of the Mediterranean basin have been 185 identified as mineral relicts of ashfall deposits (Odin et al., 1991; Coccioni et al., 2008; Sahy et 186 al., 2017). Altered clay-size fractions of illite, chlorite, and biotite associated with tuffs have also 187 been documented from beds having undergone burial diagenesis (Ullah et al., 2020). Hematite 188 pigment and neomorphism has also been reported, in association with altered clay in Paleozoic to 189 Mesozoic marine tuffs (Kiipli et al., 2000; Chakraborty et al., 2018). In sample CC1801-138, 190 petrographic evidence for alteration of clay and clay minerals to hematite, and evidence for clay-191 rich intraclasts with original porosity and abundant secondary silica, may thus be related to a 192 minor component of reworked and diagenetically altered silicate ash. Such an interpretation is 193 consistent with the characteristics and quality of the zircon crystals from the dated rock sample that make up a near-depositional population of reproducible 206 Pb/ 238 U dates at 539.40 ± 0.23 194 Ma. 195

196

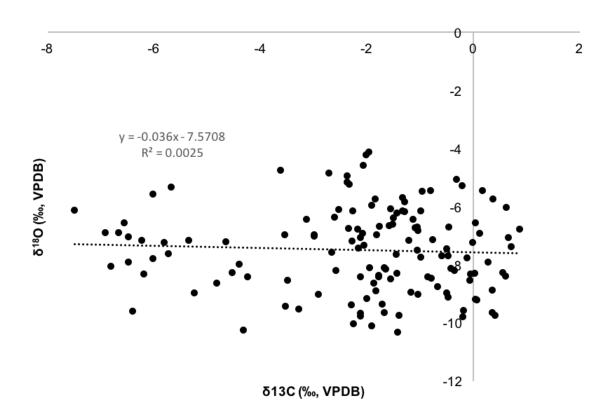
197 SUPPLEMENTAL MATERIAL TABLE CAPTIONS

Table S1. Carbonate δ^{13} C and δ^{18} O isotopic data.

Table S2. LA-ICPMS U-Pb geochronologic analyses and trace element concentrations.

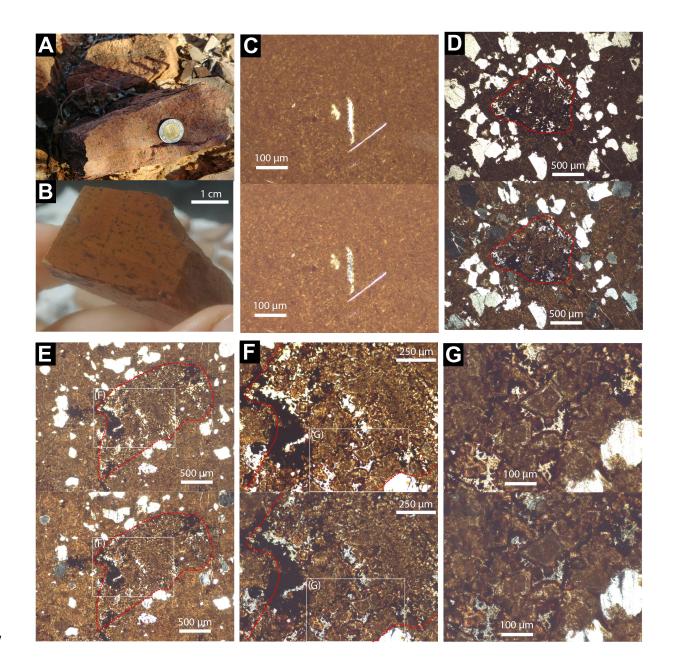
200 **Table S3.** CA-ID-TIMS U-Th-Pb isotopic data.







204 **Figure S1.** Carbon-oxygen isotope cross plot from the La Ciénega Fm. The dotted black line is a linear regression fitted to the data (Fig. 1 and Table S1) with an R² value of 0.0025.



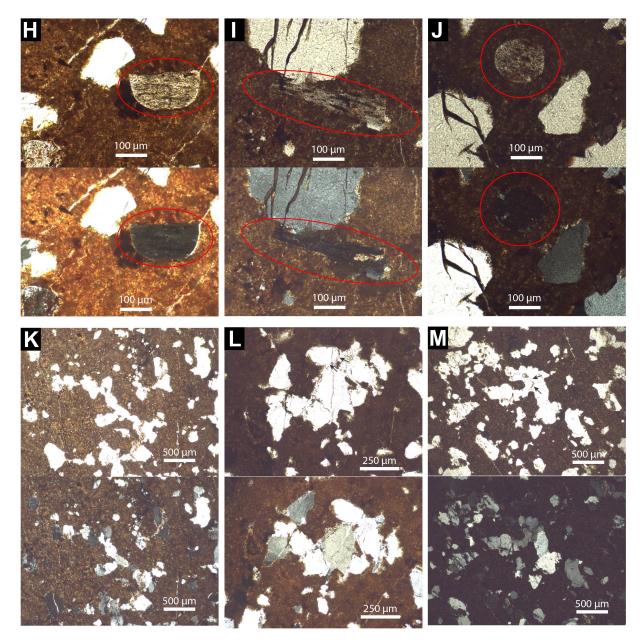




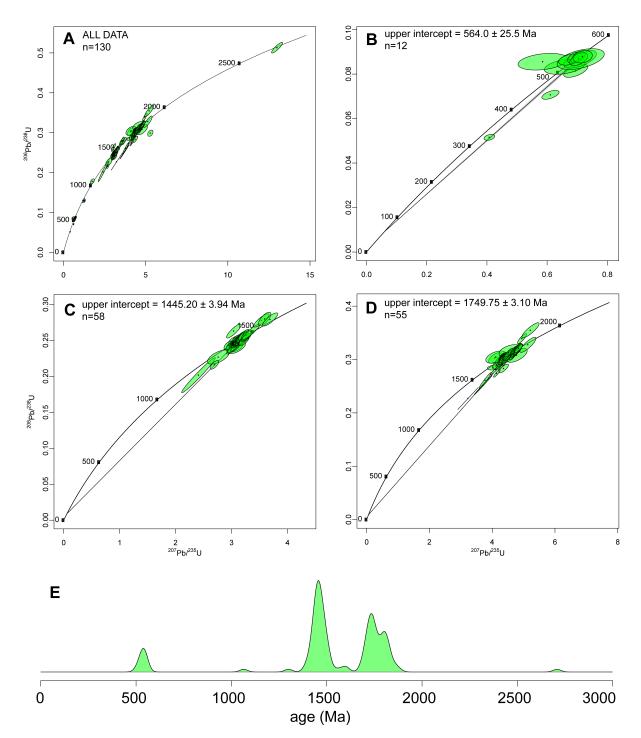
Figure S2. Photographs and Photomicrographs of U-Pb sample CC1801-138. Sets of

- 210 photomicrographs (C-M) are displayed in plane-polarized light (above) and cross-polarized light
- (below). (A) Field photograph of 10-cm thick sandy dolostone bed from which CC1801-138 was
 sampled. Coin for scale, 12.85 mm diameter. (B) Hand sample displaying fine-grained dolomitic
- matrix with well-developed internal lamination. (C) Fine-grained peloidal dolomicrite. (D-G)
- 214 Coarse-grained millimetric intraclasts of peloidal and sparry dolostone with secondary mineral
- infilling of original porosity and hematization of clay minerals, similar to features observed in
- devitrified and diagenetically altered tuffaceous horizons in marine carbonates (Section S2). Red
- 217 dashed line indicates boundary of intraclasts. (H-J) Rounded accidental lithics of basement-
- 218 derived metamorphic rock fragments, identified within red circles. (K-M) Irregularly-shaped
- 219 (amoeboid) clusters of rounded to sub-rounded quartz grains displaying quartz overgrowths with

220 internal dust lines, provide evidence for derivation from older, quartz-rich sedimentary rocks.

221 Dust lines are indicated by black arrows.

222



223 224

Figure S3. CC1801-138 LA-ICPMS U-Pb Concordia and Kernel Density Estimation plots. (A)

U-Pb Concordia plot of all data. (B-D) U-Pb Concordia plots of *circa* 540, 1450, and 1750 Ma

age populations, respectively. Discordia lines are anchored at 60 Ma, when peak metamorphism

- 227 occurred (González-León et al., 2017; Barrón-Díaz et al., 2019). (E) Kernel Density Estimation 228 (KDE) plot of all concordant data (119 of 130 analyses). KDE includes a 900 Ma cutoff for 206 Pb/ 238 U to 207 Pb/ 206 Pb age calculations. 206 Pb/ 238 U dates were excluded when discordance (206 Pb/ 238 U vs. 207 Pb/ 235 U) was >25%. 207 Pb/ 206 Pb dates were excluded when discordance 229 230 $(^{206}\text{Pb}/^{238}\text{U vs.} ^{207}\text{Pb}/^{206}\text{Pb})$ was <-10% or >+15%. Plots generated using *IsoplotR* (Vermeesch, 231 2018).
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- 233

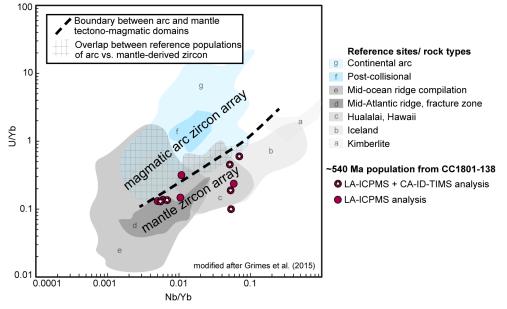
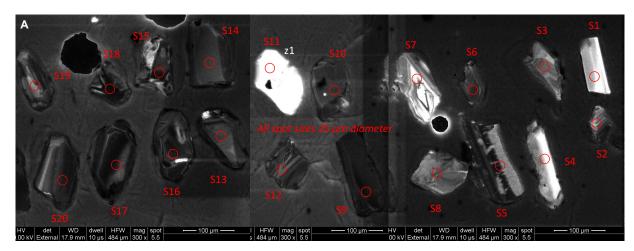


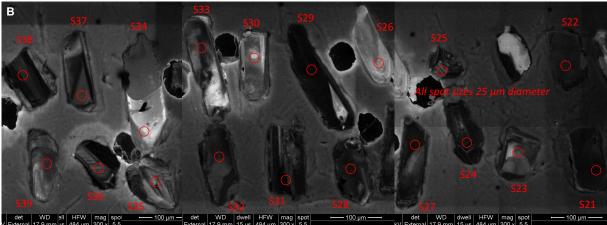


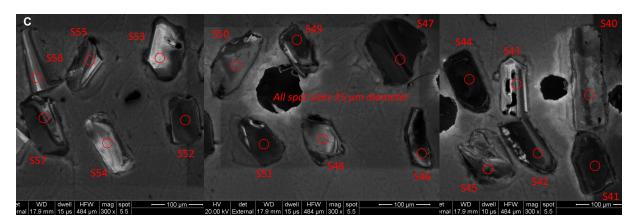
Figure S4. Circa 540 Ma mantle-derived zircon trace element geochemistry. Nb/Yb vs. U/Yb

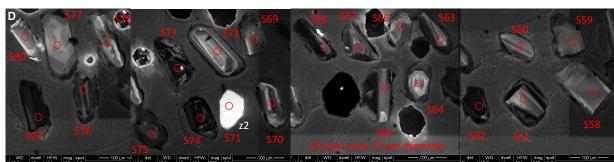
trace element data from the circa 540 Ma population of zircon within the dated sample CC1801-236

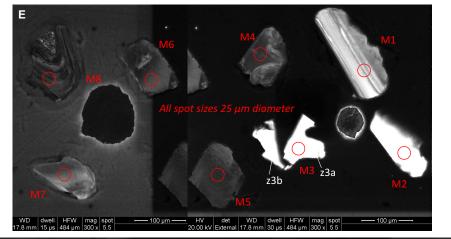
- 237 138, plotted against reference data of known tectono-magmatic setting (Grimes et al., 2015).
- 238
- 239

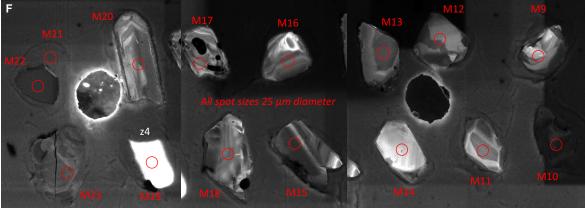










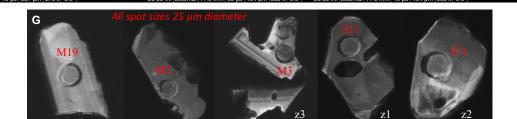


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HFW

ag spot

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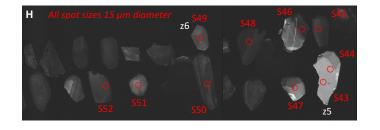
WD

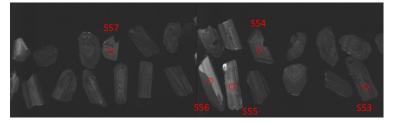
HFW

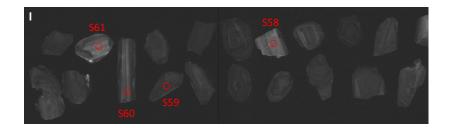
mag spot

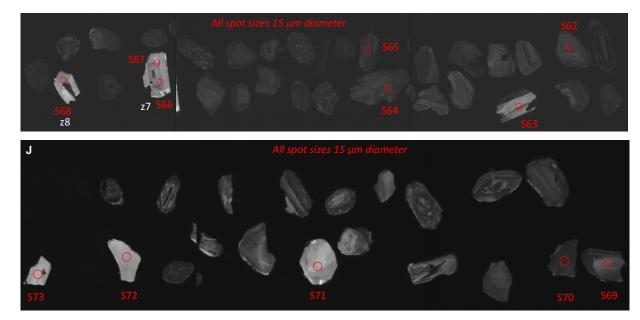
100 µn

246









- **Figure S5.** Zircon Cathodoluminescence (CL) images from dated sample CC1801-138. (A-D)
- Analyses S1-S80. Spot sizes 25 μm. Imaged at UCSB (Jan 2019) from small mount EBH2019.
- 252 (E-F) Analyses M1-M23. Spot sizes 25 μm. Imaged at UCSB (Jan 2019) from medium mount
- EBH2019. (G) Select zircon grains re-imaged at BSU (Jan 2019) from EBH2019 mounts. (H-I)
- Analyses S43-S68. Spot sizes 15 μm. Imaged at BSU (Aug 2019) from small mount
- 255 Blake2019B. (J) Analyses XS69-XS73. Spot sizes are 15 μm. Imaged at BSU (Aug 2019) from
- extra small mount Blake2019B. All labeled laser ablation spots correspond to LA-ICPMS
- analyses in Table S2. Zircons selected for CA-ID-TIMS indicated by z1-z5, and z7.

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