

This Supplemental Material accompanies

White, C.E., Barr, S.M., Crowley, J.L., van Rooyen, D., and MacHattie, T.G., 2022, U-Pb zircon ages and Sm-Nd isotopic data from the Cobequid Highlands, Nova Scotia, Canada: New contributions to understanding the Neoproterozoic geologic history of Avalonia, *in* Kuiper, Y.D., Murphy, J.B., Nance, R.D., Strachan, R.A., and Thompson, M.D., eds., *New Developments in the Appalachian-Caledonian-Variscan Orogen*: Geological Society of America Special Paper 554, [https://doi.org/10.1130/2021.2554\(07\)](https://doi.org/10.1130/2021.2554(07)).

Supplemental Material S1: GEOCHRONOLOGY METHODS

University of New Brunswick (LA-ICP-MS Analyses)

U-Pb age determinations were completed using the laser-ablation – inductively coupled plasma – mass spectrometry (LA-ICP-MS) system at the University of New Brunswick. Zircon grains were analyzed both in-situ in standard polished thin sections (Table S1) and in grain mounts (Table S2) after separation from rock samples using standard techniques and mounted in epoxy and polished until their centers were exposed. Ablation was conducted using a Resonetics M-50-LR 193 nm Excimer laser ablation system. Samples and standards were loaded together into a two-volume low-volume Laurin Technic Pty sample cell that was repeatedly evacuated and backfilled with He to remove traces of air from the cell after each sample exchange. The cell was subsequently pressurized using high purity He cell gas (~800 mL/min) that was mixed downstream of the cell with 750 mL/min Ar and 2.8 mL/min N₂. Gas lines are fitted with VICI Metronics high-flow Hg traps that maintain the ²⁰⁴Hg gas background at <150 cps. A Laurin Technic Pty ‘squid’ smoothing device was placed between the ablation cell and the ICP-MS torch. Laser output energy was set and regulated at 120 mJ corresponding to a fluence of ~5 J/cm² on target. Standards and unknowns were ablated with either 24 µm or 33 µm diameter craters using a repetition rate of either 4.5 or 5 Hz. A single long ablation sequence was constructed for each sample and comprised at least 15 analyses of zircon 91500 standard interspersed with the unknown target grains. Each ablation in the sequence comprised 30 sec of gas background collection followed by 30-35 sec of ablation. Time-resolved intensities were collected using an Agilent 7700x quadrupole ICP-MS configured with dual external rotary pumps. The measured isotopes and dwell times were ⁹⁰Zr (10 ms), ²⁰⁴Pb (50 ms), ²⁰⁶Pb (35 ms), ²⁰⁷Pb (75 ms), ²⁰⁸Pb (10 ms), ²³²Th (10 ms) and ²³⁸U (15 ms) giving a total quadrupole sweep time of ~0.23 seconds. Oxide production monitored as ²⁴⁸ThO/²³²Th on NIST glasses was < 0.4% and ²³⁸U/²³²Th was ~1.05. At the end of the ablation sequence, the laser log file and ICP-MS intensity data file were synchronized using Lolite™ (Paton et al., 2011) running as a plugin for Wavemetrics Igor Pro 6.22™. Laser-induced Pb/U fractionation corrections, corrected isotope ratios, and elemental abundances were calculated offline using Lolite™ version 2.13 (February 1, 2011) and VizualAge™ (Petrus and Kamber, 2012). The exported data were additionally inspected using Isoplot™ v3.75 and 4.15 (Ludwig, 2003, 2012). Ablation of Temora-1 zircon (416.75 ± 0.24 Ma in Black et al., 2003), FC-1 (1099.0 ± 0.6 Ma in Paces and Miller, 1993), and Plesovice (337.13 ± 0.37 Ma in Sláma et al., 2008) under the same analytical conditions was used to assess the accuracy of the method and robustness of the offline data reduction. Errors are reported at 2 sigma.

To determine the youngest age population represented in each sample, we used clusters of 3 or more grains with ages that are less than 10% discordant and that overlap within error. Although diagrams are not shown for weighted mean ages and they are not discussed specifically in the text, they are listed in Supplemental Material Table S5 and in most cases are within error of the concordia age, also shown in Table S5.

Boise State University (LA-ICP-MS Analyses)

Zircon grains were separated from rocks using standard techniques, annealed at 900°C for 60 hours in a muffle furnace, and mounted in epoxy and polished until their centers were exposed. Cathodoluminescence (CL) images were obtained with a JEOL JSM-300 scanning electron microscope and Gatan MiniCL (examples attached). Zircon was analyzed by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) using a ThermoElectron X-Series II quadrupole ICP-MS and New Wave Research UP-213 Nd:YAG UV (213 nm) laser ablation system. In-house analytical protocols, standard materials, and data reduction software were used for acquisition and calibration of U-Pb dates. Zircon was ablated with a laser spot of 25 μm wide using fluence and pulse rates of 5 J/cm^2 and 5 Hz, respectively, during a 45 second analysis (15 sec gas blank, 30 sec ablation) that excavated a pit $\sim 25 \mu\text{m}$ deep. Ablated material was carried by a 1.2 L/min He gas stream to the nebulizer flow of the plasma. Dwell times were 5 ms for Si and Zr, 200 ms for ^{49}Ti and ^{207}Pb , 80 ms for ^{206}Pb , 40 ms for ^{202}Hg , ^{204}Pb , ^{208}Pb , ^{232}Th , and ^{238}U . Background count rates for each analyte were obtained prior to each spot analysis and subtracted from the raw count rate for each analyte. Ablations pits that appear to have intersected glass or mineral inclusions were identified based on Ti and P. U-Pb dates from these analyses are considered valid if the U-Pb ratios appear to have been unaffected by the inclusions. Analyses that appear contaminated by common Pb were rejected based on mass 204 being above baseline.

Data were collected in two experiments in May 2019 and February 2020 (Table S3). 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 zircon standards and reference materials. The primary standard Plešovice zircon (Sláma et al., 2008) was used to monitor time-dependent instrumental fractionation based on two analyses for every 10 analyses of unknown zircon. A secondary correction to the $^{206}\text{Pb}/^{238}\text{U}$ dates was made based on results from the zircon standards FC1 (1098 Ma, unpublished data, Boise State University), Seiland (530 Ma, unpublished data, Boise State University), and Zirconia (327 Ma, unpublished data, Boise State University), which were treated as unknowns and measured once for every 10 analyses of unknown zircon. These results showed a linear age bias of several percent that is related to the ^{206}Pb count rate. The secondary correction is thought to mitigate matrix-dependent variations due to contrasting compositions and ablation characteristics between the Plešovice zircon and other standards (and unknowns).

Radiogenic isotope ratio and age error propagation for all analyses includes uncertainty contributions from counting statistics and background subtraction. The standard calibration uncertainty for U/Pb is the local standard deviation of the polynomial fit to the fractionation factor of Plešovice versus time and for $^{207}\text{Pb}/^{206}\text{Pb}$ is the standard error of the mean of the fractionation factor of Plešovice. The standard calibration uncertainty is the local standard deviation of the polynomial fit to the interspersed primary standard measurements versus time for the time-dependent U/Pb fractionation factor, and the standard error of the mean of the consistently time-invariant $^{207}\text{Pb}/^{206}\text{Pb}$ fractionation factor. These uncertainties are 0.5-0.8 (2σ) for $^{206}\text{Pb}/^{238}\text{U}$ and 0.4-0.7% (2σ) for $^{207}\text{Pb}/^{206}\text{Pb}$. Age interpretations are based on $^{206}\text{Pb}/^{238}\text{U}$ dates. Errors on the dates are at 2σ .

To determine the youngest age population represented in each sample, we used clusters of 3 or more grains with ages that are less than 10% discordant and that overlap within error. Although diagrams are not shown for weighted mean ages and they are not discussed specifically in the text, they are listed in Supplemental Material Table S5 and in most cases are within error of the concordia age, also shown in Table S5.

Boise State University (CA-TIMS Analyses)

A U-Pb date was obtained from one sample by the chemical abrasion isotope dilution thermal ionization mass spectrometry (CA-TIMS) method from analyses composed of single zircon grains (Table S4), modified after Mattinson (2005). Selected zircon grains were removed from the epoxy mounts for dating based on CL images and LA-ICP-MS data.

Zircon was put into 3 ml Teflon PFA beakers and loaded into 300 μ l Teflon PFA microcapsules. Fifteen microcapsules were placed in a large-capacity Parr vessel and the zircon partially dissolved in 120 μ l of 29 M HF for 12 hours at 190°C. Zircon was returned to 3 ml Teflon PFA beakers, HF was removed, and zircon was immersed in 3.5 M HNO₃, ultrasonically cleaned for an hour, and fluxed on a hotplate at 80°C for an hour. The HNO₃ was removed and zircon was rinsed twice in ultrapure H₂O before being reloaded into the 300 μ l Teflon PFA microcapsules (rinsed and fluxed in 6 M HCl during sonication and washing of the zircon) and spiked with the Boise State University mixed ²³³U-²³⁵U-²⁰⁵Pb tracer solution (BSU-1B). Zircon was dissolved in Parr vessels in 120 μ l of 29 M HF with a trace of 3.5 M HNO₃ at 220°C for 48 hours, dried to fluorides, and re-dissolved in 6 M HCl at 180°C overnight. U and Pb were separated from the zircon matrix using an HCl-based anion-exchange chromatographic procedure (Krogh, 1973), eluted together and dried with 2 μ l of 0.05 N H₃PO₄.

Pb and U were loaded on a single outgassed Re filament in 5 μ l of a silica-gel/phosphoric acid mixture (Gerstenberger and Haase, 1997), and U and Pb isotopic measurements made on a GV Isoprobe-T multicollector thermal ionization mass spectrometer equipped with an ion-counting Daly detector. Pb isotopes were measured by peak-jumping all isotopes on the Daly detector for 160 cycles, and corrected for $0.16 \pm 0.03\%$ /a.m.u. (1σ) mass fractionation. Transitory isobaric interferences due to high-molecular weight organics, particularly on ²⁰⁴Pb and ²⁰⁷Pb, disappeared within approximately 60 cycles, while ionization efficiency averaged 10⁴ cps/pg of each Pb isotope. Linearity (to $\geq 1.4 \times 10^6$ cps) and the associated deadtime correction of the Daly detector were determined by analysis of NBS982. Uranium was analyzed as UO₂⁺ ions in static Faraday mode on 10¹² ohm resistors for 300 cycles, and corrected for isobaric interference of ²³³U¹⁸O¹⁶O on ²³⁵U¹⁶O¹⁶O with an ¹⁸O/¹⁶O of 0.00206. Ionization efficiency averaged 20 mV/ng of each U isotope. U mass fractionation was corrected using the known ²³³U/²³⁵U ratio of the Boise State University tracer solution.

U-Pb dates and uncertainties were calculated using the algorithms of Schmitz and Schoene (2007), calibration of BSU-1B tracer solution of ²³⁵U/²⁰⁵Pb of 77.93 and ²³³U/²³⁵U of 1.007066 for, U decay constants recommended by Jaffey et al. (1971), and ²³⁸U/²³⁵U of 137.818 (Hiess et al., 2012). ²⁰⁶Pb/²³⁸U ratios and dates were corrected for initial ²³⁰Th disequilibrium using $D_{Th/U} = 0.20 \pm 0.05$ (1σ) and the algorithms of Crowley et al. (2007), resulting in an increase in the ²⁰⁶Pb/²³⁸U 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. U blanks are estimated at 0.013 pg.

A weighted mean ²⁰⁶Pb/²³⁸U date was calculated from equivalent dates (probability of fit >0.05) using Isoplot 3.0 (Ludwig, 2003). Errors on weighted mean date 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 ²³⁸U decay constant uncertainty propagated in quadrature. Internal error should be considered when comparing our dates with ²⁰⁶Pb/²³⁸U dates from other laboratories that used the same tracer solution or a tracer

solution that was cross-calibrated using EARTHTIME gravimetric standards. Error including the uncertainty in the tracer calibration should be considered when comparing our date with those derived from other geochronological methods using the U-Pb decay scheme (e.g., laser ablation ICPMS). Error including uncertainties in the tracer calibration and ^{238}U decay constant (Jaffey et al., 1971) should be considered when comparing our date with those derived from other decay schemes (e.g., $^{40}\text{Ar}/^{39}\text{Ar}$, ^{187}Re - ^{187}Os). Errors are at 2 sigma.

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