GSA Data Repository Item 2015263

Appendix 1. Analytical methods for the various laboratories used in this study.

Appendix 2. LA-ICPMS U-Pb data tables and Isoplot probability plots for samples analyzed at the Arizona Laserchron Center, the Pacific Centre for Isotopic and Geochemical Research (University of British Columbia) and Laurentian University (Microsoft Excel workbook - Appendix 2_Colpron_ALC-UBC-LU_LA-ICPMS_data.xlsx).

Appendix 3. U-Pb geochronologic analyses and trace element concentrations determined by LA-ICPMS at Boise State University (Microsoft Excel workbook - Appendix 3_Colpron_WT_LA-ICPMS data table BSU.xls).

Appendix 4. Compilation of U-Pb zircon igneous ages from pre-Cretaceous (>145 Ma) rocks in the Intermontane terranes of Yukon (Microsoft Excel workbook—Appendix 4_U-Pb_igneous_ages_compilation.xlsx).

Figure DR1. Plots of TuffZirc ages calculated for the youngest peak in ages for samples analyzed in this study.

Figure DR2. Plots of weighted mean ages calculated for cluster of young zircons analyzed in this study.

Figure DR3. Plots of calculated Unmix ages for samples analyzed in this study.

APPENDIX 1—ANALYTICAL METHODS

1. ARIZONA LASERCHRON CENTER

U-Pb geochronology of zircons was conducted by laser ablation multicollector inductively coupled plasma mass spectrometry (LA-MC-ICPMS). The analyses involve ablation of zircon with a New Wave DUV193 Excimer laser (operating at a wavelength of 193 nm) using a spot diameter of 25–50 microns. The ablated material is carried in helium into the plasma source of a Micromass Isoprobe, which is equipped with a flight tube of sufficient width that U, Th, and Pb isotopes are measured simultaneously. All measurements are made in static mode, using Faraday detectors for ²³⁸U, ²³²Th, ^{208–206}Pb, and an ion-counting channel for ²⁰⁴Pb. Ion yields are 0.5–1.0 mv per ppm. Each analysis consists of one 20-second integration on peaks with the laser off (for backgrounds), 20 one-second integrations with the laser firing, and a 30 second delay to purge the previous sample and prepare for the next analysis. The ablation pit is ~15 microns in depth.

For each analysis, the errors in determining ${}^{206}\text{Pb}/{}^{238}\text{U}$ and ${}^{206}\text{Pb}/{}^{204}\text{Pb}$ result in a measurement error of ~1%–2% (at 2-sigma level) in the ${}^{206}\text{Pb}/{}^{238}\text{U}$ age. The errors in measurement of ${}^{206}\text{Pb}/{}^{207}\text{Pb}$ and ${}^{206}\text{Pb}/{}^{204}\text{Pb}$ also result in ~1%–2% (at 2-sigma level) uncertainty in age for grains that are >1.0 Ga, but are substantially larger for younger grains due to low intensity of the ${}^{207}\text{Pb}$ signal. For most analyses, the crossover in precision of ${}^{206}\text{Pb}/{}^{238}\text{U}$ and ${}^{206}\text{Pb}/{}^{207}\text{Pb}$ ages occurs at 0.8–1.0 Ga.

Common Pb correction is accomplished by using the measured ²⁰⁴Pb and assuming an initial Pb composition from Stacey and Kramers (1975) (with uncertainties of 1.0 for ²⁰⁶Pb/²⁰⁴Pb and 0.3 for ²⁰⁷Pb/²⁰⁴Pb). Our measurement of ²⁰⁴Pb is unaffected by the presence of ²⁰⁴Hg because backgrounds are measured on peaks (thereby subtracting any background ²⁰⁴Hg and ²⁰⁴Pb), and because very little Hg is present in the argon gas.

Inter-element fractionation of Pb/U is generally ~20%, whereas fractionation of Pb isotopes is generally ~2%. In-run analysis of fragments of a large zircon crystal (generally every fifth measurement) with known age of 564 ± 4 Ma (2-sigma error) is used to correct for this fractionation. The uncertainty resulting from the calibration correction is generally 2%–3% (2-sigma) for both 206 Pb/ 207 Pb and 206 Pb/ 238 U ages.

The analytical data are reported in Appendix 2. Uncertainties shown in these tables are at the 1-sigma level, and include only measurement errors.

Interpreted ages are based on 206 Pb/ 238 U for <800 Ma grains and on 206 Pb/ 207 Pb for >800 Ma grains. This division at 800 Ma results from the increasing uncertainty of 206 Pb/ 238 U ages and the decreasing uncertainty of 206 Pb/ 207 Pb ages as a function of age. Analyses that are >30% discordant (by comparison of 206 Pb/ 238 U and 206 Pb/ 207 Pb ages) or >5% reverse discordant (in italics in Appendix 2) are not considered further.

The resulting interpreted ages are shown on relative age-probability diagrams (from Ludwig, 2001). These diagrams show each age and its uncertainty (for measurement error only) as a normal distribution, and sum all ages from a sample into a single curve.

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2. ISOTOPE GEOLOGY LABORATORY, BOISE STATE UNIVERSITY

A. LA-ICPMS method at Boise State University

Zircon grains were separated from seven rocks using standard techniques and annealed at 900 °C for 60 h in a muffle furnace. Randomly selected grains were mounted in epoxy and polished until their centers were exposed. Cathodoluminescence (CL) images were obtained with a JEOL JSM-1300 scanning electron microscope and Gatan MiniCL. Zircon was analyzed by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) using a ThermoElectron X-Series II quadrupole ICPMS 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 and a suite of high field strength elements (HFSE) and rare earth elements (REE). Zircon was ablated with a laser spot of 25 μ m wide using fluence and pulse rates of 5 J/cm² and 10 Hz, respectively, during a 45 second analysis (15 s gas blank, 30 s ablation) that excavated a pit ~25 μ m deep. Ablated material was carried by a 1.2 L/min He gas stream to the nebulizer flow of the plasma. Quadrupole dwell times were 5 ms for Si and Zr, 200 ms for ⁴⁹Ti and ²⁰⁷Pb, 80 ms for ²⁰⁶Pb, 40 ms for ²⁰²Hg, ²⁰⁴Pb, ²⁰⁸Pb, ²³²Th, and ²³⁸U and 10 ms for all other HFSE and REE; total sweep duration is 950 ms. Background count rates for each analyte were obtained prior to each spot analysis and subtracted from the raw count rate for each analyte. For concentration calculations, background-subtracted count rates for each analyte were internally normalized to ²⁹Si and calibrated with respect to NIST SRM-610 and -612 glasses as the primary standards. Ablations pits that appear to have intersected glass or mineral inclusions were identified based on Ti and P signal excursions, and associated sweeps were discarded. U-Pb dates from these analyses are considered valid if the U-Pb ratios appear to have been unaffected by the inclusions. Signals at mass 204 were normally indistinguishable from zero following subtraction of mercury backgrounds measured during the gas blank (<1000 cps²⁰²Hg), and thus dates are reported without common Pb correction. Rare analyses that appear contaminated by common Pb were rejected based on mass 204 greater than baseline. Temperature was calculated from the Ti-inzircon thermometer (Watson et al., 2006). Because there are no constraints on the activity of TiO₂ in the source rocks, an average value in crustal rocks of 0.8 was used.

Data were collected in seven experiments between May 2012 and May 2014. For U-Pb and ²⁰⁷Pb/²⁰⁶Pb 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 polynomial fit to the standard analyses yields each sample-specific fractionation factor. A secondary correction to ²⁰⁶Pb/²³⁸U of 0.0%–4.5% (dependent upon experiment) was made based upon the bias indicated by weighted mean dates from the zircon standard Seiland (530 Ma; unpublished chemical abrasion thermal ionization mass spectrometry data, Boise State University), which was treated as an unknown and measured once for every 10 analyses of unknown zircon. 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); because all primary and secondary standards are chemically abraded, bias between standards is not considered to be due to Pb loss.

Radiogenic isotope ratio and age error propagation for all analyses includes uncertainty contributions from counting statistics and background subtraction. Because the detrital zircon analyses are interpreted individually, uncertainties from the standard calibrations are propagated into the errors on each date. 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 ²⁰⁷Pb/²⁰⁶Pb fractionation factor. These uncertainties are 1.0%-2.0% (2σ) for ²⁰⁶Pb/²³⁸U and 0.4%-0.8% (2σ) for ²⁰⁷Pb/²⁰⁶Pb. Age interpretations are based on ²⁰⁶Pb/²³⁸U dates for these zircon grains that are <1000 Ma. Errors on the dates from individual analyses are given at 2σ .

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B. CA-TIMS methods at Boise State University

U-Pb dates were obtained samples TB1 and TB2 by the chemical abrasion isotope dilution thermal ionization mass spectrometry (CA-TIMS) method from analyses composed of single zircon grains (Table 2). Zircon grains were separated from rocks using standard techniques and mounted in epoxy and polished until the centers of the grains were exposed. Cathodoluminescence (CL) images were obtained with a JEOL JSM-1300 scanning electron microscope and Gatan MiniCL. Zircon was removed from the epoxy mounts after laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) and subjected to a modified version of the chemical abrasion method of Mattinson (2005), reflecting analysis of single grains or fragments of grains. Grains were selected for dating based on LA-ICPMS.

Zircon was placed in a muffle furnace at 900 °C for 60 h in quartz beakers. Single grains were then transferred to 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 crystals partially dissolved in 120 μ l of 29 M HF for 12 h at 180 °C. The contents of each microcapsule were returned to 3 ml Teflon PFA beakers, the HF removed and the residual grains 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 the grains were rinsed twice in ultrapure H₂O before being reloaded into the same 300 μ l Teflon PFA microcapsules (rinsed and fluxed in 6 M HCl during sonication and washing of the grains) and spiked with the Boise State University mixed ²³³U-²³⁵U-²⁰⁵Pb tracer solution. These chemically abraded grains were 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 h, dried to fluorides, and then 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. (1 sigma error) mass fractionation. Transitory isobaric interferences due to high-molecular weight organics, particularly on ²⁰⁴Pb and ²⁰⁷Pb, disappeared within ~30 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 monitored by repeated analyses of NBS982, and have been constant since installation. 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.

Weighted mean 206 Pb/ 238 U dates were calculated from equivalent dates using Isoplot 3.0 (Ludwig, 2003). Errors on the weighted mean dates are the internal errors based on analytical uncertainties only, including counting statistics, subtraction of tracer solution, and blank and initial common Pb subtraction. Internal errors should be considered when comparing our dates with 206 Pb/ 238 U dates from other laboratories that used the same Boise State University 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 our dates with those derived from other geochronological methods using the U-Pb decay scheme (e.g., laser ablation ICPMS). These errors are ± 0.11 Ma for sample TB1 and ± 0.11 Ma for sample TB2. 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 acoustidered when comparing our dates methods using the U-Pb decay scheme other decay schemes (e.g., 40 Ar/ 39 Ar, 187 Re- 187 Os). These errors are ± 0.20 Ma for sample TB1 and ± 0.18 Ma for sample TB2. Errors for weighted mean dates and dates from individual grains are given at 2σ .

U-Pb dates and uncertainties were calculated using the algorithms of Schmitz and Schoene (2007), 235 U/ 205 Pb of 77.93 and 233 U/ 235 U of 1.007066 for the Boise State University tracer solution, and U decay constants recommended by Jaffey et al. (1971). 206 Pb/ 238 U ratios and dates were corrected for initial 230 Th disequilibrium using a Th/U[magma] = 3.0 ± 0.3 using the algorithms of Crowley et al. (2007), resulting in an increase in the 206 Pb/ 238 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. U blanks are difficult to precisely measure, but are estimated at 0.07 pg.

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APPENDIX 1—ANALYTICAL METHODS

3. PACIFIC CENTRE FOR ISOTOPIC AND GEOCHEMICAL RESEARCH, UNIVERSITY OF BRITISH COLUMBIA

Detrital zircons were separated from 2 to 5 kg samples using standard rock crushing, Wilfley table, heavy liquid, and magnetic methods. A random portion of each of the zircon concentrates was mounted in an epoxy puck along with several grains of the 337 Ma Plešovice (Sláma et al., 2008) and 1099 Ma FC-1 (Paces and Miller, 1993) standard zircons and brought to a very high polish to expose the interior of the grains. The surface of the mount was washed for 10 min with dilute nitric acid and rinsed in ultraclean water prior to analysis.

Detrital zircons were dated using laser ablation - inductively coupled plasma - mass spectrometry (LA-ICPMS) at the Pacific Centre for Isotopic and Geochemical Research (PCIGR) at the University of British Columbia, Vancouver, British Columbia. Zircons were analyzed with a New Wave UP-213 laser ablation system and Thermo-Finnigan Element 2 single collector, double-focusing, magnetic sector ICP-MS, following operating parameters similar to those described by Chang et al. (2006). Line scans rather than spot analyses were employed to minimize the effects of within-run elemental fractionation. Typically, 35% laser power and a 25 µm laser spot diameter were used. Background levels were measured with the laser off for 25 s. followed by data collection with the laser on for 47 s. The time-integrated signals were analyzed using the GLITTER software package described by Van Achterbergh et al. (2001) and Jackson et al. (2004), which automatically subtracts background measurements, propagates all analytical errors, and calculates isotopic ratios and ages. Corrections for mass and elemental fractionation were made by bracketing analyses of unknown grains with replicate analyses of the standard zircon. A typical analytical session consisted of four analyses of the standard zircon, followed by five analyses of unknown zircons, one standard analysis, five unknown analyses, etc., and finally four standard analyses.

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APPENDIX 1—ANALYTICAL METHODS

4. LAURENTIAN UNIVERSITY

All grains were analyzed at Laurentian University with a 213nm solid source UV laser attached to a Thermo Fisher XSeriesII quadrupole ICP-MS. Ablation occurred in ultra-pure He, with minimal Hg blanks. U/Th/Pb calibration was performed with Geostandards zircon 91500. Zircon analyses were obtained with spot sizes ranging between 15 and 30 microns. The Temora2 standard was analyzed as an unknown. No correction was necessary for mass bias, which was estimated by concomitant analysis of NIST reference material 612. This is further evident from the weighted mean mass-bias uncorrected 207 Pb*/ 206 Pb* age of 1066 ± 8 Ma for Geostandards 91500, which is indistinguishable from the accepted value of 1065.4 ± 0.3 Ma (Wiedenbeck et al., 1995).

Each individual zircon analysis was inspected in an analysis time versus signal intensity diagram. Where distinct zones of different Th/U, 207 Pb/ 206 Pb and/or 204 Pb were found, these were separated ad treated as separate analyses. Therefore, a larger number of dates were obtained than numbers of analytical spots. From the original data, the samples with high common Pb were rejected (206/204 < 1000). The remaining analyses were run through Andersen's (2002) common-Pb routine. The samples, which required common Pb correction, were rejected.

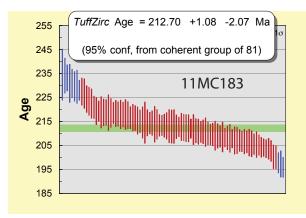
In all cases, the signal intensities of these zircons were comparable or higher than in the Paleozoic zircon standard Temora2. Rather than assigning individual errors from within run statistics, the following conservative 1 sigma uncertainties were applied: $^{207}Pb/^{206}Pb = 1\%$, $^{208}Pb/^{206}Pb = 2\%$, U/Pb = 2% and Th/Pb = 2%. The zircon standard Temora2 was analyzed interspersed as an unknown. This yielded a mean of concordant $^{206}Pb/^{238}U$ dates of 423 ± 9 Ma, easily within error of the accepted ID-TIMS age of 416.8 ± 0.3 Ma (Black et al., 2004). Note that our analysis routine uses extra long dwell times on mass 207. Because we apply a constant relative error, our data are shown to have a constant error correlation in concordia space of 0.89.

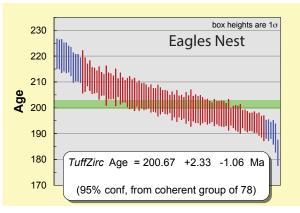
The data for sample 08MC091 are arranged in the following manner in Appendix 2. Concordant dates first, sorted by 238 U/ 206 Pb age. Near-concordant (<10%) data next, sorted by 238 U/ 206 Pb date. Discordant data last, sorted by 207 Pb/ 206 Pb age. For plotting histograms, we recommend using the 207 Pb/ 206 Pb dates for discordant zircon and the 238 Pb/ 206 Pb dates for concordant and near-concordant dates.

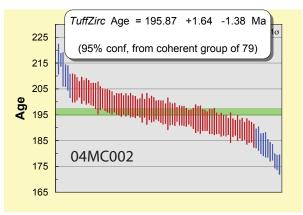
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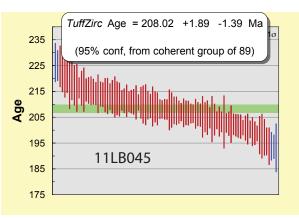
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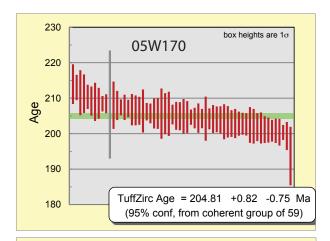
See Excel files.

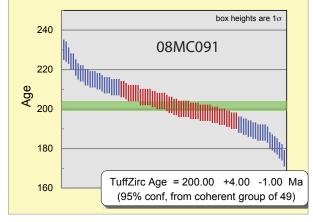


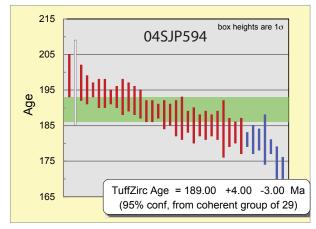


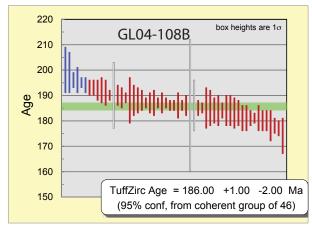




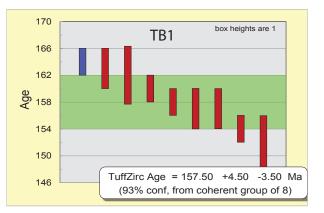


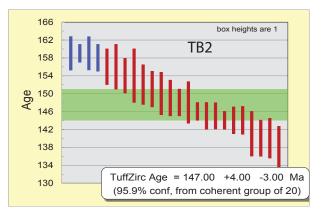


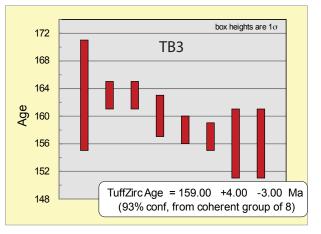


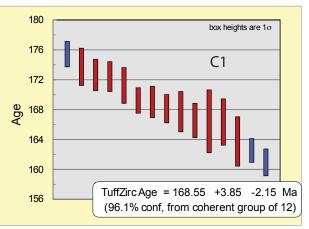


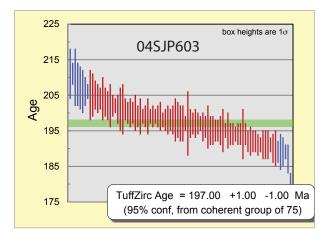
Colpron Fig DR1 TuffZirc Ages from Isoplot

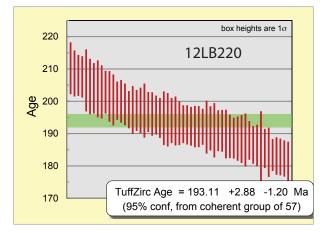


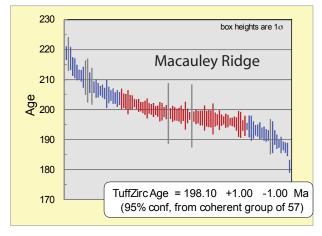


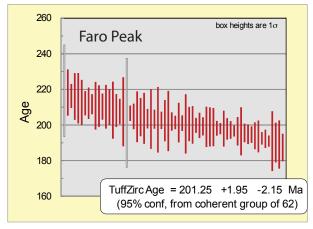




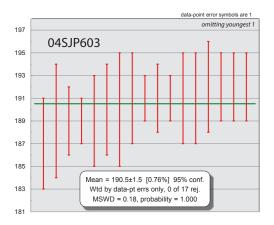


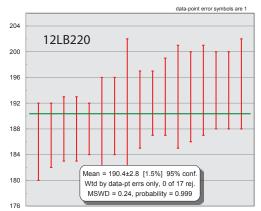


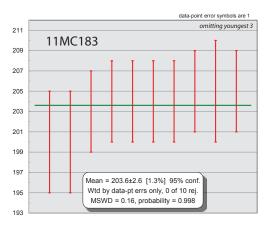


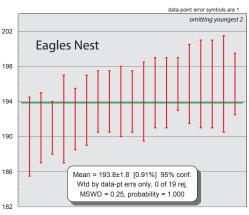


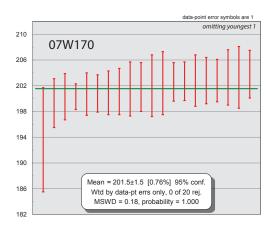
Colpron Fig DR1 (continued) TuffZirc Ages from Isoplot

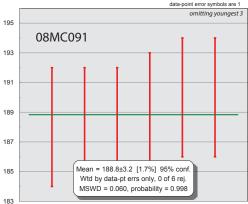


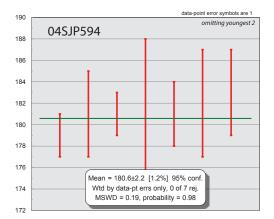


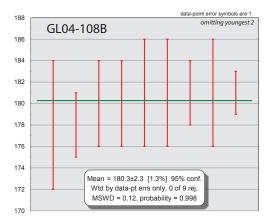




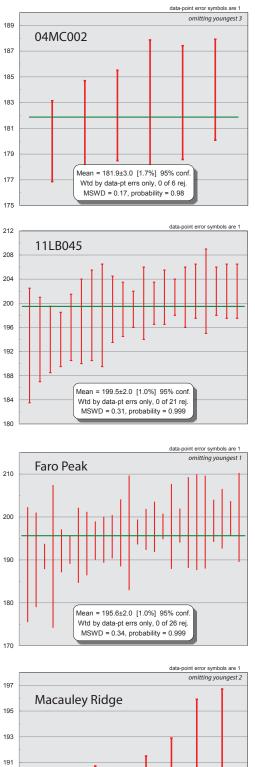


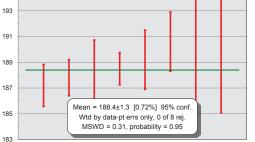




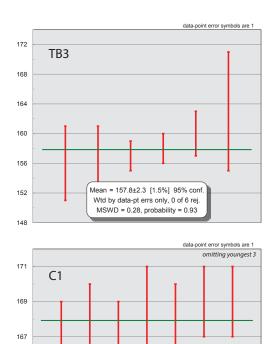


Colpron Figure DR2 Weighted mean ages of youngest cluster at 1o





Colpron Figure DR2 (continued) Weighted mean ages of youngest cluster at 10



Mean = 167.9±1.7 [1.0%] 95% conf.

Wtd by data-pt errs only, 0 of 7 rej. MSWD = 0.24, probability = 0.96

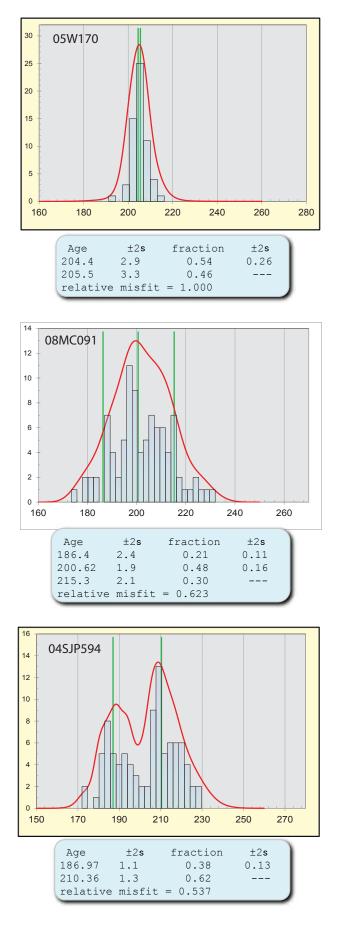


165

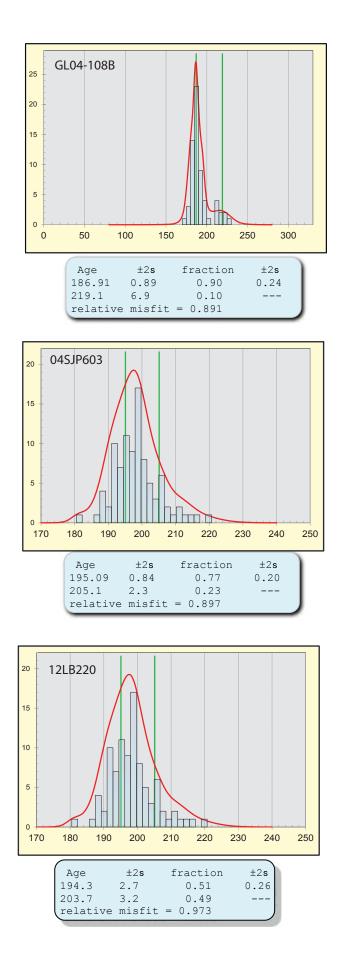
163

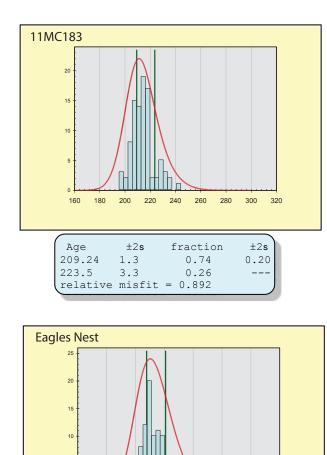
161

Youngest zircons for TB1 and TB2 were dated more precisely by CA-TIMS (see Table 2 and Fig. 9).



Colpron et al. Fig. DR3 Unmix ages from Isolplot





0 **[__** 150

Age

197.74

210.9

170

190

±2s

relative misfit = 0.885

1.3

2.3

210

230

fraction

0.64

0.36

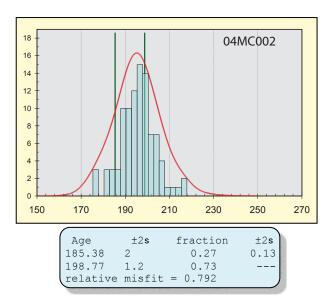
250

270

290

±2s

0.18



Colpron et al. Fig. DR3 (continued) Unmix ages from Isolplot

