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Supplemental Material

Supplement 1. Analytical Methods

Supplement 2. Sample locations for Rattlesnake Creek terrane cover sequence in the southern Klamath Mountains.

Supplement 3. Data Tables: LaMaskin et al. - A crucial geologic test of Late Jurassic exotic collision versus endemic re-accretion in the Klamath Mountains province, U.S.A., with implications for the assembly of western North America.

Supplement 1: ANALYTICAL METHODS

Arizona Laserchron Center

Separates from samples 14CM43, 15KM50, and 19KM1 were mounted in epoxy, polished, and imaged on the Macalester Keck Lab JEOL 6610 LV Scanning Electron Microscope (SEM) before analysis. U-Pb geochronology of igneous and detrital zircon was conducted by laser ablation multicollector inductively coupled mass spectrometry (LA-MC-ICPMS) at the Arizona LaserChron Center (ALC) following the methods outlined in Gehrels et al. (2006). Zircon grains were ablated using a 193 nm ArF laser with a pit depth of ~12 μm and a spot diameter of 20 μm. Fragments of in-house Sri Lanka (SL) and Forest Center (Duluth Complex; FC-1) zircon standards, respectively with isotope dilution–thermal ionization mass spectrometry (ID-TIMS) ages of 563.5 ± 3.2 Ma and 1099 ± 0.6 Ma (2σ), were analyzed once per every five unknown analyses to correct for instrument mass fractionation (Paces and Miller, 1993; Gehrels et al., 2008). A secondary standard R33 (Black et al., 2004) with ID-TIMS age of 418.9 ± 0.4 Ma (2σ) was analyzed once per every fifty unknown analyses yielding a mean age 416.1 ± 8.2 Ma (2σ). Data reduction was done using in-house ALC Microsoft Excel programs and ISOPLOT/Ex Version 3 (Ludwig, 2003). This process included calculation for average intensity, correcting for background interference, calculating isotopic ratios and ages.

California State University Northridge

Uranium-lead ratios were collected using a ThermoScientific Element2 SF-ICPMS coupled with a New Wave/Lambda Physik DUV193 Excimer laser (operating at a wavelength of 193 nm). Prior to analysis the Element2 was tuned using the NIST 612 glass standard to optimize signal intensity and stability. Laser beam diameter was ~30–40 microns at 10 Hz and 75%–100% power. Ablation was performed in a New Wave SuperCell™ and sample aerosol was transported with He carrier gas through Teflonlined tubing, where it was mixed with Ar gas before introduction to the plasma torch. Flow rates for Ar and He gases were as follows: Ar cooling gas (16.0 NL/min), Ar auxiliary gas (1.0 NL/min), He carrier gas (~0.3–0.5 NL/min), Ar sample gas (1.1–1.3 NL/min). Isotope data were collected in E-scan mode with magnet set at mass 202, and RF Power at 1245 W. Isotopes measured include ^{202}Hg, ^{204}(Pb+Hg), ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{232}Th, and ^{238}U. All isotopes were collected in counting mode with the exception of ^{232}Th and ^{238}U which were collected in analogue mode. Analyses were conducted in a ~40 min time resolved analysis mode. Each zircon analysis consisted of a 10-second integration on peaks with the laser on but not ablating (for backgrounds), 20 seconds of integrations with the laser firing on sample, and a 5–10 second delay to purge the previous sample and move to the next sample. Approximate depth of the ablation pit was ~20–30 microns.

The primary standard, 91500, was analyzed every 5–10 analyses to correct for in-run fractionation of Pb/U and Pb isotopes. Second zircon standards, FC-1, Peixe, and Temora-2, were analyzed every ~10 analyses to assess reproducibility of the data. Total uncertainties (analytical + systematic) were determined by Iolite (Paton et al., 2010). U-Pb analysis of FC-1, Peixe, and Temora-2 during all analytical sessions yielded concordant results and error-weighted average ages of 1109 ± 13 Ma, 569 ± 3 Ma, and 419 ± 3 which are within close agreement of the accepted ages for FC-1 (1099 ± 1.2 Ma; Paces and Miller, 1993; Schmitz et al., 2003), Peixe (564 ± 4 Ma; Dickinson and Gehrels, 2003), and Temora-2 (416.5 ± 0.22 Ma; Black et al., 2004).

University of South Carolina

U-Pb detrital-zircon geochronology of aliquots TLKM001 and 16KM003 (sample DUBAKELLA E); aliquot TLKM002 (sample DUBAKELLA W); aliquot 12TL041 (sample 12TL041) and aliquot TLKM003 (sample SALT CREEK) was conducted by laser-ablation high-resolution single-collector inductively coupled plasma mass-spectrometry (LA-HR-SC-ICPMS) at the University of South Carolina's Center for Elemental Mass Spectrometry. Analysis involved grain-ablation with a PhotonMachines (Teledyne) Analyte G2 193 nm (deep ultraviolet) ArF exciplex laser with a circular spot diameter of 25 µm, aimed at the centers of individual sample ('unknown') and reference (aka 'standard') zircon grains, mounted in 1" polished epoxy resin pucks contained within a nine-hole stage nested within a two-volume HelEx sample cell. For each aliquot, unknown zircons were selected randomly via the ribbon method and analyzed in batches of four to five grains each, separated by the analysis of two natural reference zircons of known and well-constrained U-Pb isotope-dilution thermal ionization mass-spectrometry (ID-TIMS) ages. A third reference material was analyzed as a known monitor 'standard' after every ~fourteenth 'unknown' analysis in order to compare the quality of standard-unknown bracketing corrections for each of the two used reference materials.

For all aliquots other than 16KM003, reference materials 91500 (1065.4 ± 0.3 Ma $^{207}Pb/^{206}Pb$ ID-TIMS age (Wiedenbeck et al. 1995); [U] = 43–114 ppm (Wiedenbeck et al., 2004)), and SL2 (563.5 ± 3.2 Ma $^{206}Pb/^{238}U$ ID-TIMS weighted mean age; [U] = ~518 ppm (Gehrels et al., 2008), were analyzed after every four to five 'unknown' analyses. Reference material Plešovice (337.1 ± 0.2 Ma and 339.3 ± 0.3 Ma $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ID-TIMS weighted mean ages respectively; U = 755 ppm and Th/U = 0.10 (Sláma et al. 2008; Horstwood et al. 2016) was analyzed after every third set of four to five unknowns of each aliquot, and used as a monitor reference material to assess and compare the accuracy of corrections based on the 91500 and SL2 reference materials. U-Pb analysis of SL2 during all analytical sessions yielded a mean age of 567 ± 1.1 Ma. For aliquot 16KM003, reference material FC1 (1099 ± 0.6 Ma $^{207}Pb/^{206}Pb$ ID-TIMS weighted mean age, [U] = ~299 ppm (Paces and Miller, 1993) was used in place of reference material SL2 yielding concordant results and a mean age of 1088 ± 8.6 Ma.

Prior to the analytical session, the coupled LA-HR-SC-ICP-MS system was manually tuned to optimize performance using 5 µm/s line scans of large fragments of the SL2 natural zircon reference material, with the laser otherwise set to parameters identical to the analytical session (see below). The tuning optimization routine involved adjusting torch position, then sample and HelEx gas flows to maximize signal (monitored by ^{238}U cps) while minimizing oxide formation (monitored by UO/U) and inter-elemental fractionation (monitored by $^{232}Th/^{238}U$). Optimized signal intensities were ~$2x10^5$ to 10^6 cps for ^{238}U from SL2. UO/U values were less than 1%, Th/U values were ~0.1 (close to the accepted SL2 value of 0.13 (Gehrels et al., 2008).

During each analysis, the ablated material was transported in He carrier gas flowing at ~0.5 and ~0.1 L/minute (LPM), respectively, from internal (MFC1) and external (MFC2) HelEx sample cell mass flow controllers downstream to the mass-spectrometer, where it was mixed with ~1 LPM of Ar sample gas in a PhotonMachines mixing bulb, and injected into the dry plasma source of a Thermo ELEMENT2 high-resolution single-collector mass-spectrometer, where the ablatant was ionized, passed through sample and skimmer cones, and then discriminated using the ELEMENT2's double-focusing magnetic sector field mass analyzer.

Analysis of unknown and reference zircons involved the collection of 6 s of background data acquisition without the laser firing, followed by laser ablation of targeted zircons for 16 s at a laser repetition rate of ~11 Hz and laser fluence of ~11 J cm^{-2}, followed by at least 20 s for signal washout, baseline (blank) stabilization, and data compilation and recording. Signal intensity data were collected for masses 202, 204, 206, 207, 208, 232, and 238 using the ion counting mode of the ELEMENT2's secondary electron multiplier (SEM) detector. Analysis of masses 206, 207, 208, 232, and 238 switched automatically to analog mode above ~$2x10^6$ cps. Mass 235 was determined by dividing the signal intensity of mass 238 by 137.818 (Horstwood et al., 2016). Four data points were measured per isotope (5% mass window; 80 samples per peak). The respective dwell times per isotope varied to optimize counting statistics, and were 21, 31, 81, 114, 10, 10, and 42 ms respectively in order of increasing mass, for a total time per sweep of 309 ms. The analysis of each unknown or reference material — including blank, ablation and washout — involved 73 sweeps over 24 s, and yielding a duty cycle of 94%. This approach enabled the collection of data from ~40 unknown and reference zircons per hour.

Post-acquisition processing of data utilized the *UPbGeochronology3* data reduction scheme of the Iolite software package (Paton et al., 2010) employed in the cross-platform WaveMetrics IgorPro computational environment. Integration windows were selected automatically by trimming 18 s from the end of each data file for baseline integrations, and ~9 s and ~2 s respectively from the start and end of each data file for ablation signals. In addition to removing the baseline signal for the ablatant signal, the latter data trimming and pre-ablation procedure removes any surface contamination incorporated into the mount surface during grinding, polishing and storage. The ablation of epoxy in insufficiently ground zircons and/or small zircons drilled through during standard ablation durations yielded similarly inappropriate windows. Adjustment of these windows was achieved by manual grain-by-grain data inspection, in which case care was taken to ensure that start time of each ablatant integration window was spatially equivalent to those of grains with auto-selected windows in order to optimize the accuracy of down-hole fraction correction models.

Following data import, selection, and inspection of integration windows, Iolite-based data reduction involved: (1) subtraction of background signals from an automatic (best-fit: see Paton et al. 2010) interpolation model; (2) determination of an appropriate downhole-fractionation correction model by separately stacking the $^{206}Pb/^{238}U$, $^{207}Pb/^{235}U$ and $^{208}Pb/^{232}Th$ downhole ratios of each of the ~30 primary reference zircon analyses in each analytical session, calculating best-fit exponential curves to those stacked data sets, and applying the resulting models to transform the isotopic ratios of analyzed 'unknown' zircons, ideally to optimize ratio steadiness; (3) estimation and correction of instrumental age-offsets and drift by comparison of determined (raw) and accepted (i.e., ID-TIMS) isotopic ratios of the primary reference zircon; and (4) calculation of final ages and values, including (a) propagated uncertainties determined from analyses of the primary reference zircon as pseudo-secondary standards, progressively removing them individually from the data set, reprocessing the data, and calculating uncertainty, and (b) error correlations using the IgorPro StatsCorrelation function. See Paton et al. (2010) for further clarification and discussion of methods of Iolite data reduction of U-Pb zircon data.

REFERENCES CITED

Black, L.P., Kamo, S.L., Allen, C.M., Davis, D.W., Aleinikoff, J.N., Valley, J.W., Mundil, R., Campbell, I.H., Korsch, R.J., Williams, I.S., and Foudoulis, C., 2004, Improved Pb-206/U-218 microprobe geochronology by the monitoring of a trace-element-related matrix effect; SHRIMP, ID-TIMS, ELA-ICP-MS and oxygen isotope documentation for a series of zircon standards: Chemical Geology, v. 205, p. 115–140, https://doi.org/10.1016/j.chemgeo.2004.01.003.

Chen, J.H., and Wasserburg, G.J., 1981, Isotopic determination of uranium in picomole and subpicomole quantities: Analytical Chemistry: v. 53, p. 2060–2067, https://doi.org/10.1021/ac00236a027.

Dickinson, W.R., and Gehrels, G.E., 2003, U–Pb ages of detrital zircons from Permian and Jurassic eolian sandstones of the Colorado Plateau, USA: paleogeographic implications: Sedimentary Geology, v. 163, p. 29–66, https://doi.org/10.1016/S0037-0738(03)00158-1.

Gehrels, G., Valencia, V., and Pullen, A., 2006, Detrital zircon geochronology by laser¬ablation multicollector ICPMS at the Arizona Laserchron Center: The Paleontological Society Papers, v. 12, p. 67–76, https://doi.org/10.1017/S1089332600001352.

Gehrels, G., Valencia, V.A., and Ruiz, J., 2008, Enhanced precision, accuracy, efficiency, and spatial resolution of U–Pb ages by laser ablation–multicollector–inductively coupled plasma–mass spectrometry: Geochemistry Geophysics Geosystems, v. 9, no. 3, https://doi.org/10.1029/2007GC001805.

Horstwood, M.S.A., Košler, J., Gehrels, G., Jackson, S.E., McLean, N.M., Paton, C., Pearson, N.J., Sircombe, K., Sylvester, P., Vermeesch, P., Bowring, J.F., Condon, D.J., and Schoene, B., 2016, Community-Derived Standards for LA-ICP-MS U-(Th-)Pb Geochronology - Uncertainty Propagation, Age Interpretation and Data Reporting: Geostandards and Geoanalytical Research, v. 40, no. 3, p. 311–332, https://doi.org/10.1111/j.1751-908X.2016.00379.x.

Irwin, W.P., Yule, J.D., Court, B.L., Snoke, A.W., Stern, L.A., and Copeland, W.B., 2011, Reconnaissance Geologic Map of the Dubakella Mountain 15′ Quadrangle, Trinity, Shasta, and Tehama Counties, California: U.S. Geological Survey Scientific Investigations Map 3149, scale 1:50,000, 1 map sheet, https://pubs.usgs.gov/sim/3149/.

Jaffey, A., Flynn, K., Glendenin, L., Bentley, W., and Essling, A., 1971, Precision measurement of half-lives and specific activities of ^{235}U and ^{238}U: Physical Review C, v. 4, p. 1889–1906, https://doi.org/10.1103/PhysRevC.4.1889.

Ludwig, K.R., 2003, Mathematical–statistical treatment of data and errors for 230Th/U geochro¬nology: Reviews in Mineralogy and Geochemistry, v. 52, p. 631–656, https://doi.org/10.2113/0520631.

Paces, J.B., and Miller, J.D., 1993, Precise UPb ages of Duluth Complex and related mafic intrusions, northeastern Minnesota: Geochronological insights to physical, petrogenic, paleomagnetic, and tectonomagmatic processes associated with the 1.1 Ga Midcontinent Rift System: Journal of Geophysical Research, v. 98, no. B8, p. 13997–14013, https://doi.org/10.1029/93JB01159.

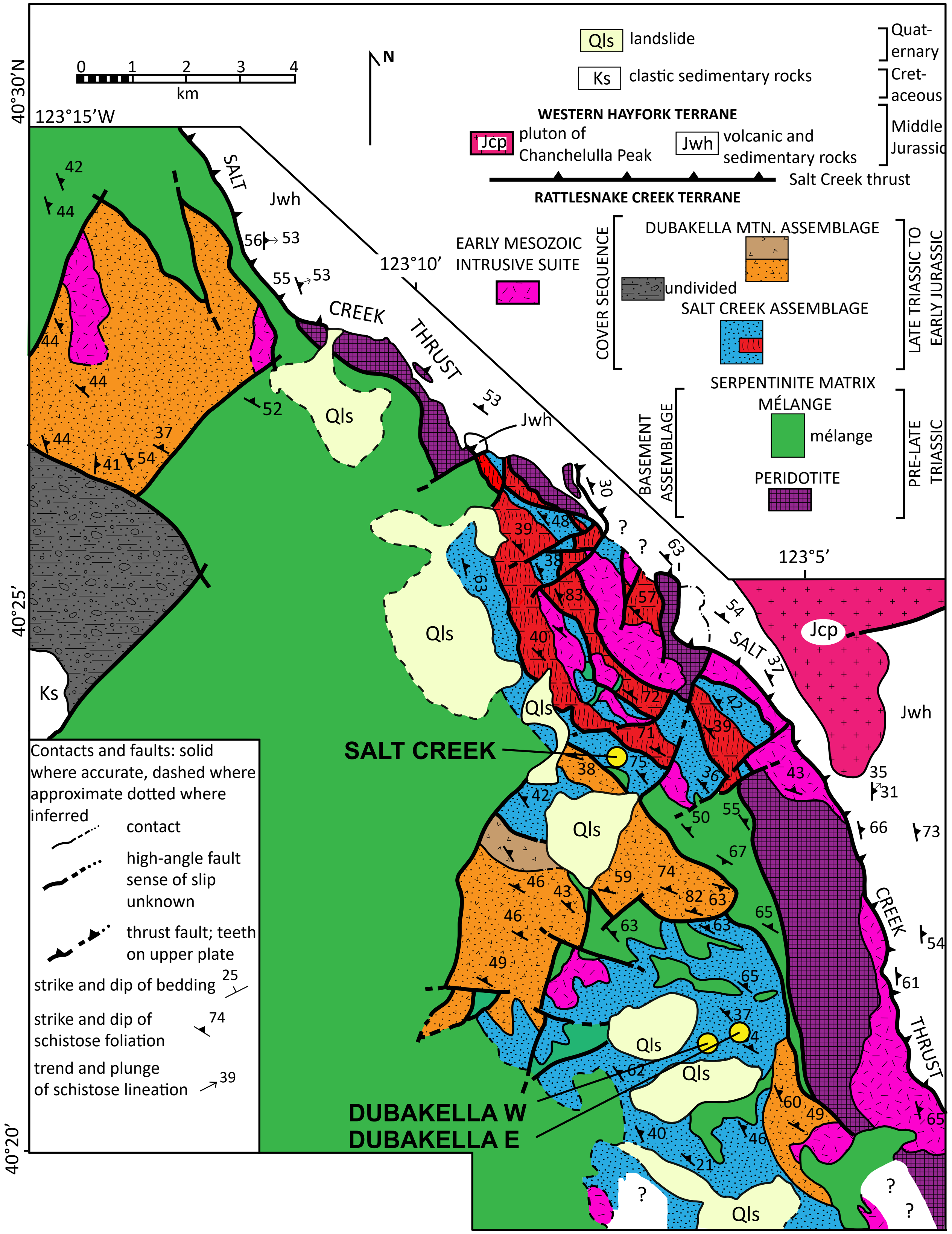
Paton, C., Woodhead, J., Hellstrom, J., Hergt, J., Greig, A., and Maas, R., 2010, Improved laser ablation U-Pb zircon geochronology through robust downhole fractionation correction: Geochemistry Geophysics Geosystems, v. 11, https://doi.org/10.1029/2009GC002618.

Schmitz, M.D., Bowring, S.A., Ireland, T.R., 2003, Evaluation of Duluth Complex anorthositic series (AS3) zircon as a U-Pb geochronological standard: New high-precision isotope dilution thermal ionization mass spectrometry results Geochimica et Cosmochimica Acta, v. 67, p. 3665–3672, https://doi.org/10.1016/S0016-7037(00)00200-X.
Sláma, J., et al., 2008, Plesovice zircon—A new natural reference material for U–Pb and Hf isotopic analysis: Chemical Geology, v. 249, p. 1–35, https://doi.org/10.1016/j.chemgeo.2007.11.005.
Stacey, J.S., and Kramers, J.D., 1975, Approximation of terrestrial lead isotope evolution by a two stage model: Earth and Planetary Science Letters, v. 26, p. 207–221, https://doi.org/10.1016/0012-821X(75)90088-6.
Wiedenbeck, M., Allé, P., Corfu, F., Griffin, W.L., Meier, M., Oberli, F., Quadt, A.V., Roddick, J.C., and Spiegel, W., 1995, Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analyses: Geostandards and Geoanalytical Research, v. 19, no. 1, p. 1–23, https://doi.org/10.1111/j.1751-908X.1995.tb00147.x.
Wiedenbeck, M., Hanchar, J.M., and Peck, W.H., 2004, Further characterisation of the 91500 zircon crystal: Geostandards and Geoanalytical Research, v. 28, no. 1, p. 9–39, https://doi.org/10.1111/j.1751-908X.2004.tb01041.x.

LaMaskin et al., - A crucial geologic test of exotic collision versus endemic re-accretion in the Klamath Mountains province, U.S.A. with implications for the assembly of western North America

Supplement 2 - Sample locations for Rattlesnake Creek terrane cover sequence in the southern Klamath Mountains

Traced and colored map of Wright and Wyld (1994; their Fig. 2) showing sample locations of Salt Creek and Dubakella E and W (this study).



Map of Irwin et al. (2011) showing sample locations of Salt Creek and Dubakella E and W (this study).