

**Dusel-Bacon, C., Holm-Denoma, C.S., Jones, J.V., III, Aleinikoff, J.N., and Mortensen, J.K., 2017, Detrital zircon geochronology of quartzose metasedimentary rocks from parautochthonous North America, east-central Alaska: Lithosphere, doi:10.1130/L672.1.**

**GSA Data Repository Item 2017332**

**U-Pb Analytical Methods For Data Repository Tables DR3–DR5.**

**Laser Ablation–Inductively Coupled Plasma–Mass Spectrometry (LA-ICP-MS) (University of British Columbia, Vancouver, Canada; J.K. Mortensen analyst)**

Detrital zircons were separated from their host rocks using conventional crushing, grinding, wet shaking table, heavy liquid, and magnetic separation methods, at the Pacific Centre for Isotopic and Geochemical Research (PCIGR) at the University of British Columbia. Zircons from each sample were mounted in thermal-setting epoxy pucks together with several grains of the Plešovice reference zircon standard ( $338 \pm 1$  Ma; Sláma et al. 2008). The mount was loaded into a New Wave “SuperCell” ablation cell and analyzed using a New Wave 213nm Nd-YAG laser coupled to a Thermo Finnigan Element2 high resolution ICP-MS. Analytical protocols were as described by Tafti et al. (2009). A 15 to 25  $\mu\text{m}$  spot with 40 percent laser power was used, employing line scans rather than spot analyses in order to minimize within-run elemental fractionation.  $^{238}\text{U}$  and  $^{232}\text{Th}$  data were acquired in analog mode, whereas  $^{208}\text{Pb}$ ,  $^{207}\text{Pb}$ ,  $^{206}\text{Pb}$ ,  $^{204}\text{Pb}$ , and  $^{202}\text{Hg}$  were acquired using ion-counting mode. Data were reduced using GLITTER software (GEMOC, Macquarie University, Australia; van Achterberg et al., 1999). Time resolved data were carefully examined to identify and avoid portions of the signal that reflected Pb loss and/or the presence of older inherited cores or altered zones in the zircon being analyzed.

Final interpretation and plotting of the analytical results employed the ISOPLOT v. 3.09 software of Ludwig (2003).

**Sensitive High Resolution Ion Microprobe–Reverse Geometry (SHRIMP–RG, jointly operated by the U.S. Geological Survey and Stanford University, California, USA; J.N. Aleinikoff, analyst)**

Zircon was extracted from samples (about 5 kg) collected at outcrops using standard mineral separation techniques including crushing and pulverizing, Wilfley table, magnetic separator, and heavy liquids to obtain a heavy mineral concentrate composed mostly of zircon. Detrital zircon was sprinkled onto double-sided tape, mounted in epoxy, ground to about half thickness, and sequentially polished using 6  $\mu\text{m}$  and 1  $\mu\text{m}$  diamond suspension. All polished grains were imaged in reflected and transmitted light on a petrographic microscope, and in cathodoluminescence (CL) using the USGS-Denver JEOL5800LV scanning electron microscope.

Randomly selected detrital zircon grains were dated by the U-Pb method using the SHRIMP-RG with a primary ion beam of about 20-25  $\mu\text{m}$  diameter. The magnet was cycled through the appropriate mass stations four times to maximize the number of grains analyzed. Measured  $^{206}\text{Pb}/^{238}\text{U}$  ratios for zircon were normalized to the accepted value for standard R33 (419 Ma; Black et al., 2004). SHRIMP data for zircon were reduced using Squid 1 (Ludwig, 2001) and plotted using Isoplot 3 (Ludwig, 2003). All calculated ages are reported with 2- $\sigma$  uncertainties. Concentrations of U and Th are believed to be accurate to about  $\pm 20\%$ , and are only used for comparing analyses. U-Pb data for detrital zircon are screened such that ages that are greater than 10% discordant are excluded from plots that display age distribution.

**Laser Ablation–Inductively Coupled Plasma–Mass Spectrometry (LA-ICP-MS) (U.S. Geological Survey, Denver, USA; C.S Holm-Denoma analyst)**

Mineral separation and mount preparation of detrital zircons in samples 96ADb25A, 97ADb43A, and 98ADb79A were made by Joe Wooden utilizing standard techniques. Zircon

analyses were conducted at the U.S. Geological Survey Central Minerals and Energy Resources Science Center (CMERSC)–Southwest Isotope Research Laboratory (SWIRL) in Denver, CO, using a Nu Instruments AttoM™. Zircon were ablated with a Photon Machines Excite™ 193 nm ArF excimer laser in spot mode (150-200 total bursts for zircon) with a repetition rate of 5 Hz, laser energy of ~5-6 mJ, and an energy density of 8-10 J/cm<sup>2</sup>. The rate of He gas flow to the HelEx cell of the laser was 0.3-0.6 L/min and make-up Ar gas (~0.2 L/min) was added to the sample stream prior to its introduction into the plasma. As suggested by Hu et al. (2008), nitrogen with flow rate of 5.5 mL/min was added to the sample stream, which allowed for significant reduction in ThO<sup>+</sup>/Th<sup>+</sup> (<0.5%) and improved the ionization of refractory Th. The laser spot sizes for zircon were ~25 µm. With the magnet parked at a constant mass, the flat tops of the isotope peaks were measured at the following masses by rapidly deflecting the ion beam: <sup>202</sup>Hg, <sup>204</sup>(Hg+Pb), <sup>206</sup>Pb, <sup>207</sup>Pb, <sup>208</sup>Pb, <sup>232</sup>Th, <sup>235</sup>U, and <sup>238</sup>U with a 30 s on-peak background measured prior to each 30s analysis. Raw data were reduced off-line using the Iolite™ 2.5 program (Paton et al., 2011) to subtract on-peak background signals, correct for U-Pb downhole fractionation, and normalize the instrumental mass bias using external mineral standards, the ages of which have previously been determined by ID-TIMS. Ages are corrected by standard sample bracketing with the primary zircon standard Temora2 (417 Ma; Black et al., 2004) and secondary standards Plešovice (337 Ma; Slama et al., 2008), and an in-house standard WRP-63-08 (1707 Ma; Wayne Premo, personal commun., 2016). Reduced data were compiled into concordia and probability density plots using Isoplot 4.15 (Excel version of procedure explained in version 3.75 manual; Ludwig, 2012).

## REFERENCES FOR ANALYTICAL METHODS AND DR TABLES 3–5

- 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  $^{206}\text{Pb}/^{238}\text{U}$  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.
- Hu, Z., Gao, S., Liu, Y., Hu, S., Chen, H., and Yuan, H., 2008, Signal enhancement in laser ablation ICP–MS by addition of nitrogen in the central channel gas: *Journal of Analytical Atomic Spectrometry*, v. 23, p. 1093–1101.
- Ludwig, K.R., 2001, Squid, version 1.02, A user's manual: Berkeley Geochronology Center Special Publication, no. 2, 21 p.
- Ludwig, K.R., 2003, Isoplot 3.09—A geochronological toolkit for Microsoft Excel: Berkeley Geochronology Center, Special Publication, no. 4, 70 p.
- Ludwig, K.R., 2012, Isoplot 3.75, A geochronological toolkit for Microsoft Excel: Berkeley Geochronology Center Special Publication, no. 5., 75 p.
- Paton, C., Hellstrom, J., Paul, B., Woodhead, J., and Hergt, J., 2011, Iolite: Freeware for the visualisation and processing of mass spectrometric data: *Journal of Analytical Atomic Spectrometry*, v. 26, p. 2508–2518.
- Sláma, J., Kosler, J., Condon, D.J., Crowley, J.L., Gerdes, A., Hanchar, J.M., Horstwood, M.S.A., Morris, G.A., Nasdala, L., Norberg, N., Schaltegger, U., Schoene, N., Tubrett, M.N.,

and Whitehouse, M.J., 2008, Plesovice zircon—a new natural reference material for U-Pb and Hf isotopic microanalysis: *Chemical Geology*, v. 249, 1-2, p. 1–35.

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.

Steiger, R.H., and Jäger, E., 1977, Subcommittee on geochronology: Convention on the use of decay constants in geo- and cosmochemistry: *Earth and Planetary Science Letters*, v. 36, p. 359–362.

Tafti, R., Mortensen, J.K., Lang, J.R., Rebagliati, M. and Oliver, J.L., 2009, Jurassic U-Pb and Re-Os ages for the newly discovered Xietongmen Cu-Au porphyry district, Tibet, PRC: Implications for metallogenic epochs in the southern Gandese Belt: *Economic Geology*, v. 104, p. 127-136.

van Achterberg, E., Ryan, C.G., and Griffin, W.L., 1999, GLITTER: On line intensity reduction for the laser ablation inductively coupled plasma spectrometry [abs.]: 9th Goldschmidt Conference, Lunar and Planetary Institute (LPI), Boston, MA, p. 905.

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