

Analytical details of U-Pb zircon geochronometry

Zircon grains from each sample were extracted from samples by traditional methods of crushing and grinding, followed by separation with a Wilfley table, heavy liquids (methylene iodide), and a Frantz magnetic separator. Samples were processed such that all zircons were retained in the final heavy mineral fraction. Subsequently, ~50 or ~1000-2000 grains (for igneous vs. DZ samples, respectively) were incorporated into a 1" epoxy mount together with fragments of our Sri Lanka standard zircon. The mounts were sanded down to a depth of ~20  $\mu\text{m}$ , polished, imaged, and cleaned prior to isotopic analysis. Prior to analysis of igneous sample 08DP04, cathodoluminescence (CL) images were also obtained to assist with laser spot placement and interpretation of zircons with variable age domains.

Subsequently, zircons were analyzed by laser ablation multicollector inductively coupled plasma mass spectrometry (LA-MC-ICPMS) at the Arizona LaserChron Center. Five samples were analyzed with a GVI Isoprobe with a laser spot size of 35  $\mu\text{m}$ ; analyses for the other four samples were performed with a Nu HR ICP-MS, using a spot size of 30  $\mu\text{m}$ .

For samples analyzed using the *Isoprobe*, zircons were ablated with a New Wave/Lambda Physik DUV193 Excimer laser (operating at a wavelength of 193 nm) using a spot diameter of 25-35 microns. The ablated material was carried in helium into the plasma source of the *Isoprobe*, which is equipped with a flight tube of sufficient width that U, Th, and Pb isotopes are measured simultaneously. All measurements were made in static mode, using 10e11 ohm Faraday detectors for  $^{238}\text{U}$ ,  $^{232}\text{Th}$ ,  $^{208}\text{Pb}$ , and  $^{206}\text{Pb}$ , a  $3 \times 10^{11}$  ohm faraday collector for  $^{207}\text{Pb}$ , and an ion-counting channel for  $^{204}\text{Pb}$ . Ion yields were ~1.0 mv per ppm. Each analysis consisted of one 12-second integration on peaks with the laser off (for backgrounds), 12 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 pits were ~12  $\mu\text{m}$  in depth.

For analyses performed using the Nu HR ICPMS, zircons were ablated with a New Wave UP193HE Excimer laser (operating at a wavelength of 193 nm) using a spot diameter of 22-30 microns. Similarly to *Isoprobe* analyses, the ablated material was carried in helium into the plasma source of a Nu HR ICPMS, which is equipped with a flight tube of sufficient width that U, Th, and Pb isotopes are measured simultaneously. All measurements on the Nu HR ICPMS are also made in static mode, using Faraday detectors with  $3 \times 10^{11}$  ohm resistors for  $^{238}\text{U}$ ,  $^{232}\text{Th}$ ,  $^{208}\text{Pb}$ , and  $^{206}\text{Pb}$ , and discrete dynode ion counters for  $^{204}\text{Pb}$  and  $^{202}\text{Hg}$ . Ion yields were ~0.8 mv per ppm. Each analysis consisted of one 15-second integration on peaks with the laser off (for backgrounds), 15 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 pits were ~15  $\mu\text{m}$  in depth.

For each analysis, the errors in determining  $^{206}\text{Pb}/^{238}\text{U}$  and  $^{206}\text{Pb}/^{204}\text{Pb}$  resulted 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 resulted in ~1-2% (at  $2\sigma$  level) uncertainty in age for grains that are >1.0 Ga, but were substantially larger for younger grains due to low intensity of the  $^{207}\text{Pb}$  signal. For most analyses, the cross-over 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 corrections were made using measured  $^{204}\text{Pb}$  and assuming an initial Pb composition according to that predicted by (Stacey and Kramers, 1975); conservative uncertainties are 1.5 for  $^{206}\text{Pb}/^{204}\text{Pb}$  and 0.3 for  $^{207}\text{Pb}/^{204}\text{Pb}$ . With the *Isoprobe*, our measurement of  $^{204}\text{Pb}$  is unaffected by the presence of  $^{204}\text{Hg}$  because backgrounds are measured on peaks (thereby subtracting any background  $^{204}\text{Hg}$  and  $^{204}\text{Pb}$ ), and because very little Hg is present in the

argon gas. Using the Nu HR ICPMS,  $^{204}\text{Hg}$  interference with  $^{204}\text{Pb}$  was accounted for by measurement of  $^{202}\text{Hg}$  during laser ablation and subtraction of  $^{204}\text{Hg}$  according to the natural  $^{202}\text{Hg}/^{204}\text{Hg}$  of 4.35. This Hg correction is not significant for most analyses because our Hg backgrounds are low (generally ~150 cps at mass 204).

Inter-element fractionation of Pb/U in the *Isoprobe* was generally ~20%, whereas apparent fractionation of Pb isotopes was generally ~2%. Inter-element fractionation with the Nu HR ICPMS of Pb/U was generally ~5%, whereas apparent fractionation of Pb isotopes was generally <0.2%. In-run analysis of fragments of a large zircon crystal during analyses on both machines (generally every fifth measurement) with a known age of  $563.5 \pm 3.2$  Ma ( $2\sigma$  error) was used to correct for this fractionation. The uncertainty resulting from the calibration correction is generally 1-2% ( $2\sigma$ ) for both  $^{206}\text{Pb}/^{207}\text{Pb}$  and  $^{206}\text{Pb}/^{238}\text{U}$  ages. Concentrations of U and Th are calibrated relative to our Sri Lanka zircon, which contains ~518 ppm of U and 68 ppm Th.

The analytical data are reported in the Supplemental Table<sup>2</sup>. Uncertainties shown in this table are at the  $1\sigma$  level and include only measurement errors. Interpreted ages are based on  $^{206}\text{Pb}/^{238}\text{U}$  for <1000 Ma grains and on  $^{206}\text{Pb}/^{207}\text{Pb}$  for >1000 Ma grains. This division at 1000 Ma results from the increasing uncertainty of  $^{206}\text{Pb}/^{238}\text{U}$  ages and the decreasing uncertainty of  $^{206}\text{Pb}/^{207}\text{Pb}$  ages as a function of age. Analyses that are >20% discordant (by comparison of  $^{206}\text{Pb}/^{238}\text{U}$  and  $^{206}\text{Pb}/^{207}\text{Pb}$  ages) or >5% reverse discordant (in italics in the Supplemental Table<sup>2</sup>) are not considered further.

Stacey, J.S., and Kramers, J.D., 1975, Approximation of terrestrial lead isotope evolution by a 2-stage model: *Earth and Planetary Science Letters*, v. 26, p. 207-221.