

## **ANALYTICAL METHODS – ZIRCON ISOTOPIC ANALYSES**

### **A.1. Sample preparation and imaging**

The samples were processed with conventional crushing, grinding and screening methods at the LOPAG-DEGEO laboratory of Universidade Federal de Ouro Preto (UFOP), Brazil and at the Instituto de Geociências of Universidade Estadual Paulista (UNESP), in Rio Claro, Brazil. After the screening, the heavy fraction concentration of 60-250 mesh was pre-concentrated by panning. After the concentration, samples were sieved and washed to remove clay and silt size. The 60-250 mesh pre-concentrate was purified using heavy liquid (LST, lithium heteropolytungstate or bromoform) to remove all light minerals. Then a Frantz LB1 magnetic separator was used to separate the less magnetic minerals, such as non-metamict zircons. Zircon was handpicked and organized in an epoxy mount, which was polished and carbon-coated for SEM (Scanning Electron Microscope) study using BSE that were taken using a TESCAN VEGA3 at the Centre for Microscopy, Characterisation and Analysis (CMCA) in The University of Western Australia (UWA). Imaging of the zircon with BSE is critical for identifying internal features, such as the core and rims, and to avoid areas with high common lead content (inclusions, fractures, and metamict areas).

### **A.2. Major and trace elements**

The chemical composition of zircon was obtained using the SEM JEOL8530F EPMA electronic microprobe, at 25 kV accelerating voltage and 80 nA current, at the CMCA, in UWA, Perth, Australia. Zircon was analyzed with electron microprobe (EMP) for Si, Ca, Ce, Dy, Er, Fe, Gd, Hf, Lu, Mg, Na, Nb, Nd, O, P, Pb, Ta, Th, Ti, U, Y, Yb, Zr. The used standards were the following: Zircon for Si, Zr, Hf; Periclase for Mg; Jadeite for Na; Wollastonite for Ca; Thorium oxide for Th; pure U; Crocoite for Pb; YPO<sub>4</sub> for Y and P; rutile for Ti; magnetite for Fe; CePO<sub>4</sub> for Ce; GdPO<sub>4</sub> for Gd; NdPO<sub>4</sub> for Nd; YbPO<sub>4</sub> for Yb; REE<sub>2</sub> for Lu; ErPO<sub>4</sub> for Er; DyPO<sub>4</sub> for Dy; Manganotantalite for Ta and pure Nb.

## A.2. U–Pb geochronology

Epoxy mounts were coated with gold for SHRIMP (Sensitive High-Resolution Ion Microprobe) analyses conducted in the laboratory of John de Laeter Centre for Isotope Research at Curtin University, Western Australia. Most SHRIMP analytical spots were in the diameter range of 20–30  $\mu\text{m}$  but the analyses of U-rich (hydrothermalized) zones used a spot size of only 10  $\mu\text{m}$ . Six scans were used for each spot analysis of magmatic zircons and five for detrital zircons. The following masses were analyzed for zircon:  $^{196}\text{Zr}_2\text{O}$ ,  $^{204}\text{Pb}$ , background,  $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$ ,  $^{208}\text{Pb}$ ,  $^{238}\text{U}$ ,  $^{248}\text{ThO}$ ,  $^{254}\text{UO}$ . The calibration of Pb/U ratios was conducted using the zircon standards M257 (561.3 Ma - Nasdala et al. (2008)) and BR266 (559 Ma, 903 ppm U - Stern, (2001)). Data with common lead correction greater than about 1% were rejected during the first scan. The uncertainties of individual ages are quoted at the  $1\sigma$  level, whereas the plotted ages are calculated at  $2\sigma$  levels (about 95% confidence). SHRIMP data were reduced using SQUID software (Ludwig, 2001) and plots were prepared using ISOPLOT/Ex (Ludwig, 2003).

## A.3. Lu–Hf isotope analyses

Lu–Hf isotopes in zircon were measured in the GeoHistory Facility in the John de Laeter Centre of the Curtin University, Perth, Australia. Analyses were placed over SHRIMP spots on grains with concordant ages. Spatially, U–Pb and Lu–Hf spot analyses were as close together as possible in order to analyze domains of the zircon grain with the same isotopic characteristics. Instrumentation includes a Resonetics *RESolution* M-50A excimer laser, coupled to a Nu Plasma II multi-collector inductively coupled plasma mass spectrometer (LA-MC-ICPMS). Following two cleaning pulses and a 55s period of background analysis, samples were spot ablated for 30 s at a 10Hz repetition rate using a 50 or 33  $\mu\text{m}$  beam and laser energy at the sample surface of  $2.2 \text{ J cm}^{-2}$ . An additional 15s of baseline was collected after ablation. The sample cell was flushed with ultrahigh purity He ( $320 \text{ mL min}^{-1}$ ) and N<sub>2</sub> ( $1.2 \text{ mL min}^{-1}$ ) and high purity Ar was employed as the plasma carrier gas. All isotopes ( $^{180}\text{Hf}$ ,  $^{179}\text{Hf}$ ,  $^{178}\text{Hf}$ ,  $^{177}\text{Hf}$ ,  $^{176}\text{Hf}$ ,  $^{175}\text{Lu}$ ,  $^{174}\text{Hf}$ ,  $^{173}\text{Yb}$ ,  $^{172}\text{Yb}$ , and  $^{171}\text{Yb}$ ) were counted on the Faraday collector

array. Time resolved data was baseline subtracted and reduced using Iolite (DRS after Woodhead et al., 2004), where  $^{176}\text{Yb}$  and  $^{176}\text{Lu}$  were removed from the 176 mass signal using  $^{176}\text{Yb}/^{173}\text{Yb} = 0.7962$  and  $^{176}\text{Lu}/^{175}\text{Lu} = 0.02655$  with an exponential law mass bias correction assuming  $^{172}\text{Yb}/^{173}\text{Yb} = 1.35274$  (Chu et al., 2002). The interference corrected  $^{176}\text{Hf}/^{177}\text{Hf}$  was normalized to  $^{179}\text{Hf}/^{177}\text{Hf} = 0.7325$  (Patchett et al., 1982) for mass bias correction. Zircons from the Mud Tank carbonatite locality were used as primary standard for all standard corrected Hf ratios (Woodhead and Hergt, 2007). The R33 zircons were used as primary standard for standard corrected Lu/Hf ratio (Fisher et al., 2014). All Analytical ratios of  $^{176}\text{Yb}/^{177}\text{Hf}$ ,  $^{176}\text{Lu}/^{177}\text{Hf}$ , and  $^{176}\text{Hf}/^{177}\text{Hf}$  are reported with  $2\sigma$  error. For all analyzed grains were used the decay constant of  $1.867 \times 10^{-11} \text{ year}^{-1}$  for  $^{176}\text{Lu}$  (Söderlund et al., 2004). The chondritic ratios of  $^{176}\text{Hf}/^{177}\text{Hf}$  (0.282785) and  $^{176}\text{Lu}/^{177}\text{Hf}$  (0.0336) of Bouvier et al. (2008), and the present day depleted mantle of  $^{176}\text{Lu}/^{177}\text{Hf}$  (0.0388) and the  $^{176}\text{Hf}/^{177}\text{Hf}$  (0.28325) of Andersen et al. (2009) were adopted in calculations. We also calculated two distinct crustal model ages  $T_{\text{DM}}$ , assuming that the parental magma of the zircons was produced from an average continental crustal source with  $^{176}\text{Lu}/^{177}\text{Hf} = 0.015$  (Goodge and Vervoort, 2006) or  $^{176}\text{Lu}/^{177}\text{Hf} = 0.0125$  (Chauvel et al., 2014).

#### **A.5. $^{18}\text{O}$ and $^{16}\text{O}$ isotope analyses**

Oxygen isotope ratios of zircon grains were measured using a Cameca IMS 1280 multi-collector ion microprobe at the CMCA, UWA. The sample mounts were cleaned with detergent, distilled water and ethanol in an ultrasonic bath and coated with gold (30 nm in thickness) prior to secondary ion mass spectrometry (SIMS) analyses. Analytical conditions were similar to those outlined in detail by Kita et al. (2009). Oxygen analyses were performed using a 3nA Gaussian  $\text{Cs}^+$  beam with an impact energy of 20 keV and was focused to a 20 mm spot. Secondary ions were sputtered using a 10  $\mu\text{m}$ -raster and were introduced into the double focusing mass spectrometer within a 110  $\mu\text{m}$  entrance slit and focused in the center of a 4000  $\mu\text{m}$  field aperture (x 130 magnification). Secondary ions were energy filtered using a 40 eV band pass with a 5 eV gap toward the high-energy side.  $^{16}\text{O}$  and  $^{18}\text{O}$  were collected simultaneously in Faraday cup detectors. A normal incidence electron gun was used

for charge compensation. Each analysis spot was pre-sputtered for 10 s, and analyses consisted of 20 s x 4 cycles, which gave an average internal precision of better than  $\pm 00.2\%$  (2 standard deviations or SD). Instrumental mass fractionation (IMS) was corrected using Temora 2 following the procedure described in Kita et al. (2009). Corrected  $^{18}\text{O}/^{16}\text{O}$  ratios are reported in  $\delta^{18}\text{O}$  notation, in per mil variations relative to Vienna standard mean ocean water (VSMOW). Uncertainty on each  $\delta^{18}\text{O}$  spot has been calculated by propagating the errors on instrumental mass fractionation determination, including the error on the reference value of the standard and standard deviation of the mean oxygen isotope ratio measured on the primary standard during the session, and internal error on each sample data point.

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