

## **METHODS**

### **SAMPLE PREPARATION**

Zircons were separated by standard techniques including water table, heavy liquids, magnetic separation and final hand picking under a binocular microscope. The zircons were mounted in epoxy and polished to approximately half thickness, and cathodoluminescence (CL) images were obtained with a scanning electron microscope to reveal internal structures such as growth zoning and core-rim relationships.

### **SIMS**

The SIMS (Secondary Ionisation Mass Spectrometry) analyses were performed on the Cameca IMS 1270 SIMS (secondary ion mass spectrometer) at the Nordsim laboratory, Swedish Museum of Natural History, Stockholm, and the SHRIMP II at Curtin University, Perth.

### **NORDSIM**

The analytical method, data reduction, error propagation, and assessment of the results are outlined in Whitehouse et al. (1999) and Whitehouse and Kamber (2005). The analyses were conducted with an O<sub>2</sub>-beam of 4 nA and a spot size of 10–30 μm, calibrated to the Geostandard 91500 reference zircon with an age of 1065 Ma (Wiedenbeck et al., 1995). The error on the U/Pb ratio includes propagation of the error on the day-to-day calibration curve obtained by regular analysis of the reference zircon. A common Pb correction was applied using the <sup>204</sup>Pb concentration and present-day isotopic composition (Stacey and Kramers, 1975).

## SHRIMP II, Curtin Uni.

Standard operating procedures for U, Th and Pb isotopic measurements using the SHRIMP II ion microprobe are detailed in Wingate & Kirkland (2010). The zircon standard 91500 was used for concentration calibration and also U–Pb calibration. Calibration uncertainties are included in the errors of the reported  $^{238}\text{U}/^{206}\text{Pb}^*$  ratios and dates.  $^{204}\text{Pb}$  counts were not observed to decrease during the sputtering process and are not surface related, thus common-Pb corrections were applied to all analyses using contemporaneous isotopic compositions determined according to the model of Stacey & Kramers (1975).

## LA–ICP–MS

LA–ICP–MS (Laser ablation Inductively Coupled Mass Spectrometry) analyses were conducted using two different instrument set-ups.

1) At the Geological Survey of Norway (NGU), the analyses were carried out on an ELEMENT XR single collector, high-resolution ICP–MS, coupled to a UP193–FX 193 nm short-pulse excimer laser ablation system from New Wave Research. The laser was set to ablate single, up to 60  $\mu\text{m}$ -long lines, using a spot size of 20 or 15  $\mu\text{m}$ , a repetition rate of 10 Hz and an energy corresponding to a fluence of 4–5  $\text{J}/\text{cm}^2$ . Each analysis included 30 s of background measurement followed by 30 s of ablation. The masses 202, 204, 206–208, 232 and 238 were measured. The reference material GJ–1 (Jackson et al., 2004) was used for correction of isotopic ratios, whereas 91500 (Wiedenbeck et al., 1995) and an in-house standard (OS–99–14;  $1797 \pm 3$  Ma; Skår, 2002) were used to check precision and accuracy. The data were not corrected for common lead, but monitoring of the signal for 204 allowed exclusion of affected data from further calculations. The data were reduced using the GLITTER® software (Van Achterbergh et al., 2001).

The LA–ICP–MS analyses at NGU were conducted as part of the CAMOC (Centre for Advanced Mineral and Ore Characterisation) collaboration between NGU and the Norwegian University of Science and Technology (NTNU).

2) At the NERC Isotope Geosciences Laboratory, Nottingham, UK, using a Nu Instruments Attom single collector ICP–MS coupled to a New Wave Research 193 nm excimer laser ablation system with a low-volume teardrop-shaped cell. The full instrument conditions, analytical protocol and uncertainty propagation are described in Spencer et al. (2014). Laser conditions were a static 25  $\mu\text{m}$  spot, a fluence of 2–3  $\text{J}/\text{cm}^2$ , a repetition rate of 5 Hz, and a dwell time of 30 s. Data were not corrected for common lead. The reference material 91500 was used for correction of isotopic ratios, and reference materials GJ–1 and/or Plešovice were used for checking precision and accuracy (Jackson et al., 2004; Slama et al., 2008).

The Isoplot program (Ludwig, 2003) was used to calculate and present the U–Pb isotope data from all methods. All U–Pb zircon data are plotted in Tera–Wasserburg diagrams to reveal potential problems with common Pb and fractionation during LA–ICP–MS analysis.

## **ZIRCON TRACE-ELEMENT (Ti) ANALYSIS**

Ti concentrations in zircon were determined at NGU on an ELEMENT XR single collector, high-resolution ICP–MS, coupled to a UP193–FX 193 nm short-pulse excimer laser ablation system from New Wave Research. The laser was operated at a frequency of 8 Hz, laser energy 4.0  $\text{J}/\text{cm}^2$  and a static spot size of 10  $\mu\text{m}$ .  $\text{Ti}^{47}$  was measured in 300 M/ $\Delta\text{M}$  (Low Resolution) together with other trace elements including REE (not presented or discussed in the manuscript). Background counts were measured for 40 s followed by 20 s laser ablation. For data reduction, the GLITTER software package version 4.4.4 (Van Achterbergh et al., 2001) was used. Ti concentration was calibrated using  $\text{Si}^{29}$  as internal standard and NIST SRM 610 as external standard. To control the accuracy and precision of the analyses, NIST

SRM 612 was analysed together with the samples. The concentrations of NIST SRM 610 and 612 were taken from Jochum et al. (2011).

## REFERENCES

- Jackson, S. E., Pearson, N. J., Griffin, W. L., and Belousova, E. A., 2004, The application of laser ablation-inductively coupled plasma-mass spectrometry to in situ U-Pb zircon geochronology: *Chemical Geology*, v. 211, p. 47-69.
- Jochum, K. P., Weis, U., Stoll, B., Kuzmin, D., Yang, Q., Raczek, I., Jacob, D. E., Stracke, A., Birbaum, K., Frick, D. A., Günther, D., andENZWEILER, J., 2011, Determination of Reference Values for NIST SRM 610–617 Glasses Following ISO Guidelines: *Geostandards and Geoanalytical Research*, v. 35, p. 397-429.
- Ludwig, K. R., 2003, *Isoplot 3.00, A Geochronological Toolkit for Microsoft Excel*: Berkley Geochronology Center, Special Publication, v. 4, p. 1-74.
- Skår, Ø., 2002, U-Pb geochronology and geochemistry of early Proterozoic rocks of the tectonic basement windows in central Nordland, Caledonides of north-central Norway: *Precambrian Research*, v. 116, p. 265-283.
- 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.
- Van Achterbergh, E., Ryan, C. G., Jackson, S. E., and Griffin, W. L., 2001, LA-ICP-MS in the Earth Sciences - Appendix 3, data reduction software for LA-ICP-MS, *in* Sylvester, P. J., ed., *Short Course volume 29: St. John's*, Mineralogical Association of Canada, p. 239-243.
- Whitehouse, M. J., and Kamber, B. S., 2005, Assigning dates to thin gneissic veins in high-grade metamorphic terranes: A cautionary tale from Akilia, Southwest Greenland: *Journal of Petrology*, v. 46, p. 291-318.
- Whitehouse, M. J., Kamber, B. S., and Moorbath, S., 1999, Age significance of U-Th-Pb zircon data from early Archaean rocks of west Greenland - a reassessment based on combined ion-microprobe and imaging studies: *Chemical Geology*, v. 160, p. 201-224.
- Wiedenbeck, M., Allé, P., Corfu, F., Griffin, W. L., Meier, M., Oberli, F., Von Quadt, A., Roddick, J. C., and Spiegel, W., 1995, Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analyses: *Geostandards Newsletter*, v. 19, p. 1-23.
- Wingate, M. T. D., and Kirkland, C. L., 2010, *Introduction to geochronology information released in 2010*.