U-Th-Pb Geochron analytical procedure used in the SHRIMP IIe/mc instrument of the IBERSIMS lab (UGR)

Hand-picked zircons from the studied samples, several grains of the TEMORA-1 standard (for isotope ratios; Black et al., 2003), one grain of the SL13 zircon standard (for U concentration, Claoué-Long et al., 1995), plus a few grain of the REG zircon (plenty of common lead, for calibrating the masses) are cast on a 3.5 cm diameter epoxy mount (megamount), polished and documented using optical (reflected and transmitted light) and scanning electron microscopy (secondary electrons and cathodoluminescence). After extensive cleaning, mounts are coated with ultra pure gold (8 - 10 nanometers thick) and inserted into the SHRIMP for analysis. The analytical method follows that described by Williams and Claesson (1987). Each selected spot is rastered with the primary beam for 120 s prior to the analysis, and then analysed 6 scans, following the isotope peak sequence $^{196}Zr_2O$, ^{204}Pb, $^{204.1}$background, ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{238}U, ^{248}ThO, ^{254}UO. Every peak of every scan is measured sequentially 10 times with the following total counting times per scan: 2 s for mass 196; 5 s for masses 238, 248, and 254; 15 s for masses 204, 206, and 208; and 20 s for mass 207. The primary beam, composed of $^{16}O^{16}O^{+}$, is set to an intensity of about 5 nA, with a 120 microns Kohler aperture, which generates 17 x 20 micron elliptical spots on the target. The secondary beam exit slit is fixed at 80 microns, achieving a resolution of about 5000 at 1% peak height.

All calibration procedures are performed on the standards included on the same mount. Mass calibration is done on the REG zircon (ca. 2.5 Ga, very high U, Th and common lead content). Every analytical session starts measuring the SL13 zircon, which is used as a concentration standard (238 ppm U). The TEMORA-1 zircon (416.8 ± 1.1 Ma), used as isotope ratios standard, is then measured every 4 unknowns.

Data reduction is done with the SHRIMPTOOLS software (available from www.ugr.es/~fbea), specifically developed for IBERSIMS by F. Bea. This software is a new implementation of the original PRAWN software developed for the SHRIMP, and has been extensively checked against PRAWN and Ludwig's SQUID. SHRIMPTOOLS is platform-independent and runs on any Windows, Mac or Unix computer regardless of language, time, and date system settings. It has been written in the programming language of the STATA commercial package which implements powerful algorithms for robust regression, outlier detection and time-series analysis. The software calculates the intensity of each measured isotope in two steps. First, it uses the STATA letter-value display algorithm to find outliers in the ten replicates measured in each peak during each scan, discarding them and averaging the rest once normalized to the SBM measurements. Then, it calculates the 204/206, 207/206, 208/206, 254/238 ratios using Dodson's (1978) double linear interpolation method. The 206/238, 206/195, 238/195, and 248/254 ratios are calculated by

dividing the value at the mid-time of the analysis of each isotope calculated from the robust regression lines of the peak average of each scan vs the time at which it was measured. Errors for Dodson interpolated ratios are calculated as the standard error of the (scans-1) interpolations for each ratio. Errors for the isotope ratios calculated by regression result from propagating accordingly the standard error of the linear prediction at the mid-point of the analysis. $^{206}Pb/^{238}U$ is calculated from the measured $^{206}Pb^{+}/^{238}U^{+}$ and UO^{+}/U^{+} following the method described by Williams (1998). The error reported for $^{206}Pb/^{238}U$ includes (1) the error in UO^{+}/U^{+} (2) the error in the regression line $\ln(UO^{+}/U^{+})$ vs $\ln(^{206}Pb/^{238}U)$ (3) the standard error in the replicate measurements of the TEMORA zircon. For high-U zircons (U > 2500 ppm) $^{206}Pb/^{238}U$ is further corrected using the algorithm of Williams and Hergt (2000). Though seldom necessary, the software also permits correction for instrumental drift with time using the sequence of replicate measurements of the TEMORA zircon.

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