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Analytical details of isotope analyses for Sr and Nd

Samples were digested in a clean room using ultra-clean reagents and analysed by TIMS in a Finnigan Mat 262 spectrometer after chromatographic separation with ion-exchange resins. Normalization values were $^{86}Sr/^{88}Sr = 0.1194$ and $^{146}Nd/^{144}Nd = 0.7219$. Blanks were 0.6 and 0.09 ng for Sr and Nd respectively. The external precision (2σ), estimated from the results of the last 10 replicates of the standard WS-E (Govindaraju et al., 1994), which is routinely analyzed each 10 unknown samples, was better than 0.003% for 87Sr/86Sr, and 0.0015% for 143Nd/144Nd. 87Rb/86Sr and 147Sm/144Nd were directly determined by ICP-MS (Montero and Bea, 1998) with a precision, estimated by analyzing 10 replicates of the standard WS-E, better than 1.2% and 0.9% (2σ) respectively. All analytical procedure was performed using ultra clean reagents. Normalization values were $^{86}Sr/^{88}Sr = 0.1194$ and $^{146}Nd/^{144}Nd = 0.7219$. Blanks were 0.6 and 0.09 ng for Sr and Nd respectively. The external precision (2σ), estimated by analyzing 10 replicates of the standard WS-E (see Montero and Bea, 1998, and references therein), was better than ± 0.003% for $^{87}Sr/^{86}Sr$ and ± 0.0015% for $^{143}Nd/^{144}Nd$. $^{87}Rb/^{86}Sr$ and $^{147}Sm/^{144}Nd$ were directly determined by ICP-MS following the method developed by (Montero and Bea, 1998), with a precision better than ± 1.2% and ± 0.9% (2σ) respectively.

Analytical procedure used in the SHRIMP

Each selected spot is rastered with the primary beam for 120 s prior to the analysis, and then analyzed 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. Then, the blank, measured at 204.1 mass is subtracted from each peak. This may produce negative values in mass 204 when it recorded next to zero counts. Once normalized to the SBM measurements, the software calculates the 204/206, 207/206, 208/206, 254/238 ratios using (Dodson, 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.

Negative ^{204}Pb values may arise from subtracting the blank to the 204-peak measurement when the later is very low, zero, one or two counts per 10 seconds. Zero counts on 204 and 1 count on 204.06 (the blank) result in negative 204/206 ratios. Despite negative isotope ratios have no physical meaning, there is a general agreement amount the SHRIMP users community that 204 negative values should be kept for (1) balancing averages between different measurements (2) to counter-effect anomalously high blank measurements. Given that the blank is subtracted to masses 206 and 207, the negative 204 helps approximating the correct 207/206 ratio.

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