A possible laser ablation xenotime U-Pb age standard: Reproducibility and accuracy

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With the in-situ U-Pb dating of phosphates by SIMS and LA-ICP-MS new possibilities of linking geochronological and petrological data have been opened up. But wide application of the method is presently hampered by the absence of any accepted xenotime standard for U-Pb dating.

In order to establish such a well defined age standard, xenotime was separated from a Weinsberg granite sample (type Plochwald) from the Bohemian Massif (Austria). This xenotime has a concordant U-Pb TIMS age of 315 ± 2 Ma. No elemental zonation in the crystals was visible using BSE and CL imaging.

Instrumental and analytical setup: New Wave 193 nm solid state laser ablation system with He as carrier gas, Nu Instruments HR multi-collector double-focusing ICP-MS, Nu Instruments DSN-100 desolvating nebulizer. The MS was setup to allow the simultaneous acquisition of the masses ^{238}U-^{233}U-^{232}Th-^{207}Pb-^{206}Pb-^{205}Tl-^{204}-Pb-^{203}Tl. During analysis a ^{233}U -Tl spike solution was added to the ablated material as a dry aerosol.

Data reduction: Raw signal intensities are corrected for gas blank using 40 sec of signal acquisition prior to sample analysis. Power law and $^{205}Tl/^{203}Tl$ and $^{233}U/^{205}Tl$ in the spike solution are used to correct for mass bias of $^{207}Pb/^{206}Pb$ and U/Pb ratios. The U/Pb elemental fractionation is corrected for using an intercept method applying linear regression. The calculated intercept values are corrected for mass discrimination using standard bracketing.

115 measurements on 15 xenotime crystals of 150 to 200 µm size were performed on three consecutive days. Lines with 5µm spot size and 30 µm length were rastered with 5 µm/sec. 12 passes resulted in a total of 72 sec of ablation per analysis.

No significant day-to-day or grain-to-grain variations in age could be detected, nor did the orientation of the laser raster parallel to the prominent {110} cleavage or parallel to crystal faces result in any age discrimination.

Results: All analyses resulted in concordant data points. On average, ages from single line analysis have 2-sigma precisions of 7.6%, 8.3%, and 5.7% for the $^{207}Pb/^{235}U$, $^{206}Pb/^{238}U$, and $^{207}Pb/^{206}Pb$ ages, respectively. The total mean age of all analyses (without rejections) is 313.4 ± 1.9 Ma (2-sigma), in absolute concordance with the conventional TIMS data.

We propose that the investigated xenotime can effectively be used as a standard for in-situ LA U-Pb age dating with a spatial resolution as low as 5 µm.

External accuracy of laser ablation U-Pb zircon dating: Rresults from a test using five different standards

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Using a state-of-the-art 193nm-LA-MC-ICP-MS system and thorough control on analytical procedures, the long term (months) external accuracy and reproducibility for Phanerozoic zircons using standard bracketing is 1% to 2% 2RSD for the $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ages. This is true when using only one standard zircon, such as 91500, for the matrix matched calibration. When using different standards for the calibration (i.e. Plesovice, Sri Lanka, Temora) suspicious systematic shifts in the obtained ages and thus a reduction in the overall accuracy of the dating method become obvious. These shifts are in the range of a few percent of the U-Pb ages and seem to vary unsystematically with age and zircon composition. Two main causes could be made responsible for the effects: 1) matrix and or ablation process-related effects stemming from (subtle?) differences in standard and sample compositions; 2) instrumental effects from the laser system or the mass spectrometer.

In order to test which of the two is responsible, a ‘test of accuracy’ experiment was conducted. Any instrumental effects were reduced as far as possible by analysing five different standard zircons mounted on one single mount and analysed during one session using the identical protocol for all analyses and without changing any instrument parameters and keeping ion beam intensities as identical as possible. Each standard was analysed 8 times with two analyses per grain resulting in a total of 80 measurements. For data reduction, every standard served consecutively as calibration standard, the others were treated as unknowns. The known standard age and the four calculated ages using the respective four other standards for calibration were then compared. Even using such a very strict analytical protocol age shifts were still present. They vary non-systematically and range from -1 Ma to +20 Ma (i.e. 0.3% to 10%) for the investigated age range (1064 Ma to 220 Ma).

Conclusions: Accepting the absence of any instrumental effects (i.e. no memory effects, no effects from dead-time correction, non-linearity of ion counters and inter-detector calibration,…), the observed age shifts have to be attributed to matrix and/or ablation process-related effects. Therefore, to allow the comparison of laser ablation zircon U-Pb ages on an intra- and inter-laboratory basis, more rigorous than usual matrix matching procedures have to be applied, and the resulting reduction in the overall accuracy has to be included into the error propagation schemes for the final age data.