# U-Pb inter-laboratory calibrations using zircon samples: Application of the new CA-TIMS technique

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U-Pb inter-laboratory calibrations using natural zircon standards are perhaps the most useful and meaningful type of inter-calibration, but also the most demanding: they test not only isotopic tracer calibrations, but also sample homogeneity and analytical techniques, especially complete removal of all zircon domains with Pb loss.

A new zircon U-Pb geochronology method, CA-TIMS, combines pre-dissolution high-temperature annealing of natural radiation damage followed by multi-step dissolution. Early partial dissolution steps preferentially remove domains that have lost Pb. Later partial dissolution steps sample zircon that has behaved as a perfect closed system with respect to U and Pb. This yields highly precise and accurate crystallization ages for zircons that lack any inheritance, and also is very sensitive to detecting the presence of inheritance or any other isotopic complexities.

CA-TIMS was applied to two recent, but widely used zircon standards: R33 and TEMORA-2. An aliquot of each zircon standard was annealed in air at 1,000°C for 48 hrs, then digested in 16 partial digestion steps of gradually increasing intensity. In each case, the first 4 steps removed disturbed zircon. Both samples showed perfect closed-system behavior for the remaining 12 steps, each yielding a set of statistically identical $^{206}Pb^*/^{238}U$ ages and defining a plateau on a plot of age versus 238U released. The plateau for R33 yields an age of 419.96 ± 0.15 (MSWD = 0.96); TEMORA-2 yields an age of 417.82 ± 0.06 (MSWD = 0.52).

In terms of mass balance, the plateau for R33 was reached after removal of 15% of the total U, but only 5% of the zircon mass. The plateau for TEMORA-2 was reached after removal of 31% of the total U, but only 3.4% of the zircon mass, reflecting the strong U zoning of the zircons, and also demonstrating the high selectivity of the CA-TIMS steps.

Both samples yielded $^{207}Pb^*/^{206}Pb^*$ ages for the plateau steps that are concordant with the $^{206}Pb^*/^{238}U$ ages within analytical and decay constant uncertainties.

The CA-TIMS analyses demonstrate that both R33 and TEMORA-2 have "clean" isotopic systematics, and that only a very small volume of high-U rim material has Pb loss; both are excellent standards for U-Pb calibration purposes.

# Progress report on the U-Pb interlaboratory experiment

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Uncertainty in the Pb/U ratio of tracer solutions utilized in U-Pb isotope-dilution thermal ionization mass-spectrometry (ID-TIMS) geochronology, typically estimated at 0.1 to 0.25%, is one of the largest sources of uncertainty in the comparison of data from different laboratories. In order to better assess the degree of agreement among the various ID-TIMS U-Pb laboratories, an interlaboratory experiment involving natural zircon standards (R33 and TEMORA) and common mixed U-Pb gravimetric solution(s) is now underway as an outgrowth of the EARTHTIME project (http://www.earth-time.org/). Published ID-TIMS data sets for these two standards indicate that concordant and equivalent clusters of data can be obtained with uncertainties in the $^{206}Pb/^{238}U$ date of ca. 0.1-0.2% [1]. These studies also demonstrate that techniques employed to minimize Pb-loss (e.g., air abrasion) are critical for producing equivalent datasets. Initial datasets from TEMORA and R33 zircons that were presented at the EARTHTIME II workshop indicate up to 1% scatter in the U-Pb dates between various laboratories. However, given variation in pre-treatment techniques (i.e., degree of air-abrasion etc.) and variability in blank contribution, it is impossible to assess how much of the variation is attributable to tracer solution calibration. In order to minimize such effects, batches of uniformly pre-treated zircons (both air-abraded and annealed/leached) were prepared and distributed. Such high-*n* ID-TIMS data sets, combined with tracer solution calibrations against multiple mixed U-Pb gravimetric solutions, offers the potential for interlaboratory calibration at the 0.1% level or better. Data on these standards and solutions will be presented. Elimination of interlaboratory biases is a crucial first step before the geochronology community will be able to systematically (1) compare and/or integrate U-Pb and $^{40}Ar/^{39}Ar$ data sets from multiple labs and, (2) assess systematic variation between $^{206}Pb/^{238}U$ and $^{207}Pb/^{235}U$ dates and uncertainties in the $^{235}U$ and $^{238}U$ decay constants.

## Reference

[1] L. P. Black *et al.*, *Chemical Geology* **205**, 115-140 (APR 30, 2004).