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Data Repository: Analytical Methods

U-Pb geochronologic analyses of zircon (Element2 HR ICPMS)

Zircon crystals are extracted from samples by traditional methods of crushing and grinding, followed by separation with a Wilfley table, heavy liquids, and a Frantz magnetic separator. Samples are processed such that all zircons are retained in the final heavy mineral fraction. Approximately 50 high-quality grains are selected and mounted into 1" expoxy mounts with fragments or loose grains of Sri Lanka, FC-1, and R33 zircon crystals that are used as primary standards. The mounts were sanded down to a depth of ~20 microns, polished, imaged, and cleaned prior to isotopic analysis.

Pre-acquisition cathodoluminescence imaging of zircon was conducted at University of Arizona using a Hitachi 3400N SEM and a Gatan CL2 detector system (www.geoarizonasem.org) to provide a guide for locating analysis pits in textural domains (e.g., core and rim overgrowths).

U-Pb geochronology of zircons was conducted by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) at the Arizona LaserChron Center (Gehrels et al., 2006, 2008; Gehrels and Pecha, 2014). The analyses involve ablation of zircon with a Photon Machines Analyte G2 excimer laser equipped with HelEx ablation cell using a spot diameter of 20 microns. The ablated material is carried in helium into the plasma source of an Element2 HR ICPMS, which sequences rapidly through U, Th, and Pb isotopes. Signal intensities are measured with an SEM that operates in pulse counting mode for signals less than 50K cps, in both pulse-counting and analog mode for signals between 50K and 4M cps, and in analog mode above 4M cps. The calibration between pulse-counting and analog signals is determined line-by-line for signals between 50K and 4M cps, and is applied to 4M cps signals. Four intensities are determined and averaged for each isotope, with dwell times of 0.0052 sec for 202, 0.0075 sec for 204, 0.0202 sec for 206, 0.0284 sec for 207, 0.0026 sec for 208, 0.0026 sec for 232, and 0.0104 sec for 238.

With the laser set an energy density of ~5 J/cm2, a repetition rate of 8 hz, and an ablation time of 10 seconds, ablation pits are ~12 microns in depth. Sensitivity with these settings is approximately ~5,000 cps/ppm. Each analysis consists of 5 sec on peaks with the laser off (for backgrounds), 10 sec with the laser firing (for peak intensities), and a 20 second delay to purge the previous sample and save files.

Following analysis, data reduction was performed using an in-house Python decoding routine and an Excel spreadsheet (E2agecalc) that:

1. Decodes .dat files from the Thermo software such individual intensities for measurement are available (routine written by John Hartman, University of Arizona)

2. Imports intensities and a sample name for each analysis

3. Calculates average intensities for each isotope (based on the sum of all counts while the laser is firing)

4. Subtracts ^{204}Hg from the 204 signal to yield ^{204}Pb intensity (using natural ^{202}Hg/^{204}Hg of 4.3). This Hg correction is not significant for most analyses because our Hg backgrounds are low (generally ~150 cps at mass 204).

5. Performs a common Pb correction based on the measured ^{206}Pb/^{204}Pb and the assumed composition of common Pb based on Stacey and Kramers (1975)

6. Calculates measured 206/238, 206/207, and 208/232 ratios

7. Compares measured and known ratios for the three standards to determine fractionation factors for 206/238, 206/207, and 208/232. These correction factors are generally <5% for 206/238, <2% for 206/207, and <20% for 208/232.

8. Determines an overdispersion factor if the standard analyses show greater dispersion than expected from measurement uncertainties

9. Uses a sliding-window average to apply fractionation factors to unknowns (generally averaging 8 standard analyses)

10. Calculates fractionation-corrected 206/238, 206/207, and 208/232 ratios and ages for unknowns

11. Propagates measurement uncertainties for 206/238 and 208/232 that are based on the scatter about a regression of measured values. Uncertainties for 206/207 and 206/204 are based on the standard deviation of measured values since these ratios generally do not change during an analysis. The sum of this uncertainty and any overdispersion factor is reported as the internal (or measurement) uncertainty for each analysis. These uncertainties are reported at the 1-sigma level.

12. Calculates the down-hole slope of 206/238 to highlight analyses in which 206/238 is compromised due to heterogeneity in age (e.g., crossing an age boundary) or intersection of a fracture or inclusion.

13. Calculates concentrations of U and Th for unknowns based on the measured intensity and known concentrations of FC-1.

14. Calculates the external (systematic) uncertainties for 206/238, 206/207, and 208/232, which include contributions from (a) the scatter of standard analyses, (b) uncertainties in the ages of the standards, (c) uncertainties in the composition of common Pb, and (4) uncertainties in the decay constants for ^{235}U and ^{238}U.

15. Determines a “Best Age” for each analysis, which is generally the 206/238 age for <900 Ma ages and the 206/207 age for >900 Ma ages.

16. Provides preliminary filters that highlight analyses with >20% discordance, >5% reverse discordance, or >10% internal (measurement) uncertainty.

17. Creates a publication-ready datatable with concentrations, isotope ratios, and ages for unknowns.

U-Pb geochronologic analyses of monazite (Element2 HR ICPMS)

U-Pb/Th-Pb geochronology of individual monazite crystals was conducted by laser ablation multicollector inductively coupled plasma mass spectrometry (LA-MC-ICPMS) at the Arizona LaserChron Center (Gehrels et al., 2006, 2008). The isotopic analyses involved ablation of monazite with a Photon Machines Analyte G2 excimer laser (□ = 193 nm) coupled to a Nu Instruments HR-MC-ICPMS. Helium carries the ablated material into the plasma source of the ICPMS, which is equipped with a flight tube of sufficient width such that U, Th, and Pb isotopes are measured simultaneously. Analyses were conducted with a 6 micron laser spot diameter using an acquisition routine consisting of one 9 second integration on peaks with the laser off (for backgrounds), 9 one-second integrations with the laser firing, and a 30 second delay to ensure the previous sample is purged from the collector block. Operating parameters for the excimer laser were maintained at ~10 J/cm^2 at 5 Hz in constant energy mode for 65 laser bursts per analysis, producing an ablation depth of ~ 6 microns. Faraday detectors with 3x10^{11} ohm resistors measure ^{238}U, ^{232}Th, and discrete dynode ion counters measure $^{208-204}$Pb, all in static mode.

For each analysis, the errors in determining ^{206}Pb/^{238}U and ^{206}Pb/^{204}Pb result in a measurement error of ~1-2% (at 2-sigma level) in the ^{206}Pb/^{238}U age. The errors in determining ^{206}Pb/^{207}Pb and ^{206}Pb/^{204}Pb also result in ~1-2% (at 2-sigma level) uncertainty in age for grains that are older than 900 Ma; however, they are substantially larger for younger grains due to low intensity of the ^{207}Pb signal. The errors in determining the ^{208}Pb/^{232}Th and ^{208}Pb/^{204}Pb result in a final measurement of ~1-2% (at 2-sigma level) in the ^{208}Pb/^{232}Th age for each analysis.

Common Pb correction is accomplished by using the Hg-corrected ^{204}Pb and assuming an initial Pb composition from Stacey and Kramers (1975). Uncertainties of 1.5 for ^{206}Pb/^{204}Pb and 0.3 for ^{207}Pb/^{204}Pb are applied to these compositional values based on the variation in Pb isotopic composition in modern crystal rocks. Interference of ^{204}Hg with ^{204}Pb is accounted for measurement of ^{202}Hg during laser ablation and subtraction of ^{204}Hg according to the natural ^{202}Hg/^{204}Hg of 4.35.

The 6 micron spot diameter in conjunction with the 9 second analysis routine (65 bursts at a 5Hz repetition) was used to minimize both spatial and at-depth mixing of multiple age domains. Grain selection and spot placements were based on the high resolution BSE images, for grains where there was a large difference in compositional domains multiple analyses were performed.

Following analysis, data reduction was performed using an in-house Python decoding routine and an Excel spreadsheet (E2agecalc) that:

1. Decodes .dat files from the Thermo software such individual intensities for measurement are available (routine written by John Hartman, University of Arizona)

2. Imports intensities and a sample name for each analysis

3. Calculates average intensities for each isotope (based on the sum of all counts while the laser is firing)

4. Subtracts ^{204}Hg from the 204 signal to yield ^{204}Pb intensity (using natural ^{202}Hg/^{204}Hg of 4.3). This Hg correction is not significant for most analyses because our Hg backgrounds are low (generally ~150 cps at mass 204).

5. Performs a common Pb correction based on the measured ^{206}Pb/^{204}Pb and the assumed composition of common Pb based on Stacey and Kramers (1975)

6. Calculates measured 206/238, 206/207, and 208/232 ratios

7. Compares measured and known ratios for the three standards to determine fractionation factors for 206/238, 206/207, and 208/232. These correction factors are generally <5% for 206/238, <2% for 206/207, and <20% for 208/232.

8. Determines an overdispersion factor if the standard analyses show greater dispersion than expected from measurement uncertainties

9. Uses a sliding-window average to apply fractionation factors to unknowns (generally averaging 8 standard analyses)

10. Calculates fractionation-corrected 206/238, 206/207, and 208/232 ratios and ages for unknowns

11. Propagates measurement uncertainties for 206/238 and 208/232 that are based on the scatter about a regression of measured values. Uncertainties for 206/207 and 206/204 are based on the standard deviation of measured values since these ratios generally do not change during an analysis. The sum of this uncertainty and any overdispersion factor is reported as the internal (or measurement) uncertainty for each analysis. These uncertainties are reported at the 1-sigma level.

12. Calculates the down-hole slope of 206/238 to highlight analyses in which 206/238 is compromised due to heterogeneity in age (e.g., crossing an age boundary) or intersection of a fracture or inclusion.

13. Calculates concentrations of U and Th for unknowns based on the measured intensity and

known concentrations of FC-1.

14. Calculates the external (systematic) uncertainties for 206/238, 206/207, and 208/232, which include contributions from (a) the scatter of standard analyses, (b) uncertainties in the ages of the standards, (c) uncertainties in the composition of common Pb, and (4) uncertainties in the decay constants for ^{235}U and ^{238}U.

15. Determines a "Best Age" for each analysis, which is generally the 206/238 age for <900 Ma ages and the 206/207 age for >900 Ma ages

16. Provides preliminary filters that highlight analyses with >20% discordance, >5% reverse discordance, or >10% internal (measurement) uncertainty.

17. Creates a publication-ready datatable with concentrations, isotope ratios, and ages for unknowns.

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