http://app.pan.pl/SOM/app67-Davis_etal_SOM.pdf

SUPPLEMENTARY ONLINE MATERIAL FOR

A morganucodontan mammal from the Upper Jurassic Morrison Formation, Utah, USA

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Supplementary Online Material

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U-Pb dating methods and results

Samples and zircon recovery

Three silty mudstone samples (CMQ 190618, CMQ-2, and CMQ-3) from the Cisco Mammal Quarry site were crushed mechanically, and zircons were concentrated by a combination of ultrasonic deflocculation of slurries and Wilfley shaker table separations. Zircons were further purified by standard magnetic separation and heavy liquid flotation of lighter minerals. All three samples contained significant amounts of swelling clay, and one contained significant numbers of zircons (CMQ-2). This sample was purple in color, whereas the 2 samples that yielded very few zircons were greenish gray in color (CMQ 190618 and CMQ-3).

The samples yielded a range of zircon morphologies: dominantly rounded, detrital grains, as well as minor euhedral subpopulations (Figure 1). The euhedral zircon crystals displayed characteristics typical of volcanic origins and ash-fall deposition, such as elongate tips, longitudinal bubble tracks and transverse channels (Figure 2). Some of these euhedral grains had minor adherent glass, which can often indicate a volcanic origin from the most recent eruption. Zircons from the volcanic/ash-fall and euhedral subpopulations were preferentially chosen for dissolution and U-Pb analysis.

Methods

Select zircons were annealed at 850°C for 50 hours, then dissolved in two steps in a chemical abrasion, isotope dilution, thermal ionization mass spectrometric U-Pb dating method (CA-ID-TIMS) modified from Mattinson (2005). The first dissolution step was in hydrofluoric acid (HF) and nitric acid (HNO_3) at 180°C for 12 hours. This removed the most metamict zircon domains in the annealed crystals. Individual grains were then spiked with a mixed ^{205}Pb-^{233}U-^{235}U tracer (ET535), completely dissolved in HF and HNO_3 acids at 235°C for 30 hours, and then converted to chlorides in hydrochloric acid at 180°C for 16 hours. The dissolutions were loaded onto rhenium filaments with phosphoric acid and silica gel without any further chemical processing. Pb and UO_2 isotopic compositions were determined in single Daly-photomultiplier mode on a Micromass Sector 54 thermal ionization mass spectrometer. Total common Pb ranged from 1–7 picograms (Table 1). One picogram of common Pb was assigned to blank as total procedural Pb blanks during the study varied from 1–0.4 picograms; the isotopic composition of the remaining common Pb was estimated by Stacey and Kramers (1975) model. U blanks were estimated to be 0.01 pg based on measured blanks during the

study. Pb fractionation of 0.21±0.10 %/amu was determined from replicate analyses of NIST SRM 981; U fractionation was determined internally from the isotope dilution tracer and corrected for oxide interferences. $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ values were corrected for Th-disequilibrium after Schärer (1984), assuming magma Th/U of 2.2

Data reductions and age calculations used PbMacDat and IsoplotEx, based on the algorithms of Ludwig (1988, 1991, 1998).

Results

Twenty-two single zircons were dissolved and analyzed by CA-ID-TIMS methods. Most of the data overlap Concordia but range in age from 770 to 151 Ma (Table 1, Figure 3). The 4 youngest dates produced a Concordia Age (Ludwig, 1998) of 151.41±0.27 Ma (Figure 3) and a weighted mean $^{206}Pb/^{238}U$ date of 151.50±0.28/0.29/0.33 Ma (Table 1, Figure 4) in the ±X/Y/Z format of Schoene et al. (2006), where X is analytical uncertainty only, Y includes tracer uncertainties, and Z includes decay constant errors. X can be used to compare U-Pb dates from the same lab and tracer, Y compares U-Pb dates between labs that used different tracers, and Z can be used to compare to dates by other methods, such as ^{40}Ar-^{39}Ar, as long as those dates propagated all the systematic errors in their methods.

The weighted mean age of 151.50±0.28/0.29/0.33 is interpreted as the best estimate of the volcanic, ashfall age. The presence of rounded detrital grains and slightly older euhedral zircons however, implies that even the youngest dates could also be from detrital grains; as a result, the weighted mean represents a maximum depositional age.

Table 1. U-Pb CA-ID-TIMS zircon data available at http://app.pan.pl/SOM/app67-Davis_etal_SOM/Table_1.xls

References

Krogh, T.E. 1973. A low-contamination method for hydrothermal decomposition of zircon and extraction of U and Pb for isotopic age determinations. *Geochimica et Cosmochimica Acta* 37: 485–494.

Ludwig, K.R. l988. PBDAT for MS-DOS, a computer program for IBM-PC compatibles for processing raw Pb-U-Th isotope data, version 1.24. *U.S. Geological Survey, Open-File Report* 88-542, 32 pp.

Ludwig, K.R. l991. ISOPLOT for MS-DOS, a plotting and regression program for radiogenic-isotope data, for IBM-PC compatible computers, version 2.75. *U.S. Geological Survey, Open-File Report* 91-445, 45 pp.

Ludwig, K. R. 1998 On the treatment of concordant uranium-lead ages. *Geochimica et Cosmochimica Acta* 62: 665-676.

Mattinson, J.M. 2005. Zircon U-Pb chemical abrasion ("CA-TIMS") method: combined annealing and multi-step partial dissolution analysis for improved precision and accuracy of zircon ages. *Chemical Geology* 220: 47-66.

Parrish, R.R., Roddick, J.C., Loveridge, W.D., and Sullivan, R.D. l987. Uranium-lead analytical techniques at the geochronology laboratory, Geological Survey of Canada, *in* Radiogenic age and isotopic studies, Report 1: Geological Survey of Canada Paper 87-2, p. 3–7.

Schärer, U. 1984. The effect of initial ^{230}Th equilibrium on young U-Pb ages: the Makula case, Himalaya. *Earth and Planetary Science Letters* 67: 191-204.

Schoene, B., Crowley, J.L., Condon, D.J., Schmitz, M.D., and Bowring, S.A. 2006. Reassessing the uranium decay constants for geochronology using ID-TIMS U-Pb data. *Geochemica et Cosmochemica Acta* 70: 426-445.

Stacey, J.S. and Kramers, J.D. 1975. Approximation of terrestrial lead isotope evolution by a two-stage model. *Earth and Planetary Science Letters* 26: 207–221.

Steiger, R.H. and Jäger, E. 1977. Subcommission on geochronology: convention on the use of decay constants in geo- and cosmochronology. *Earth and Planetary Science Letters* 36: 359–362.

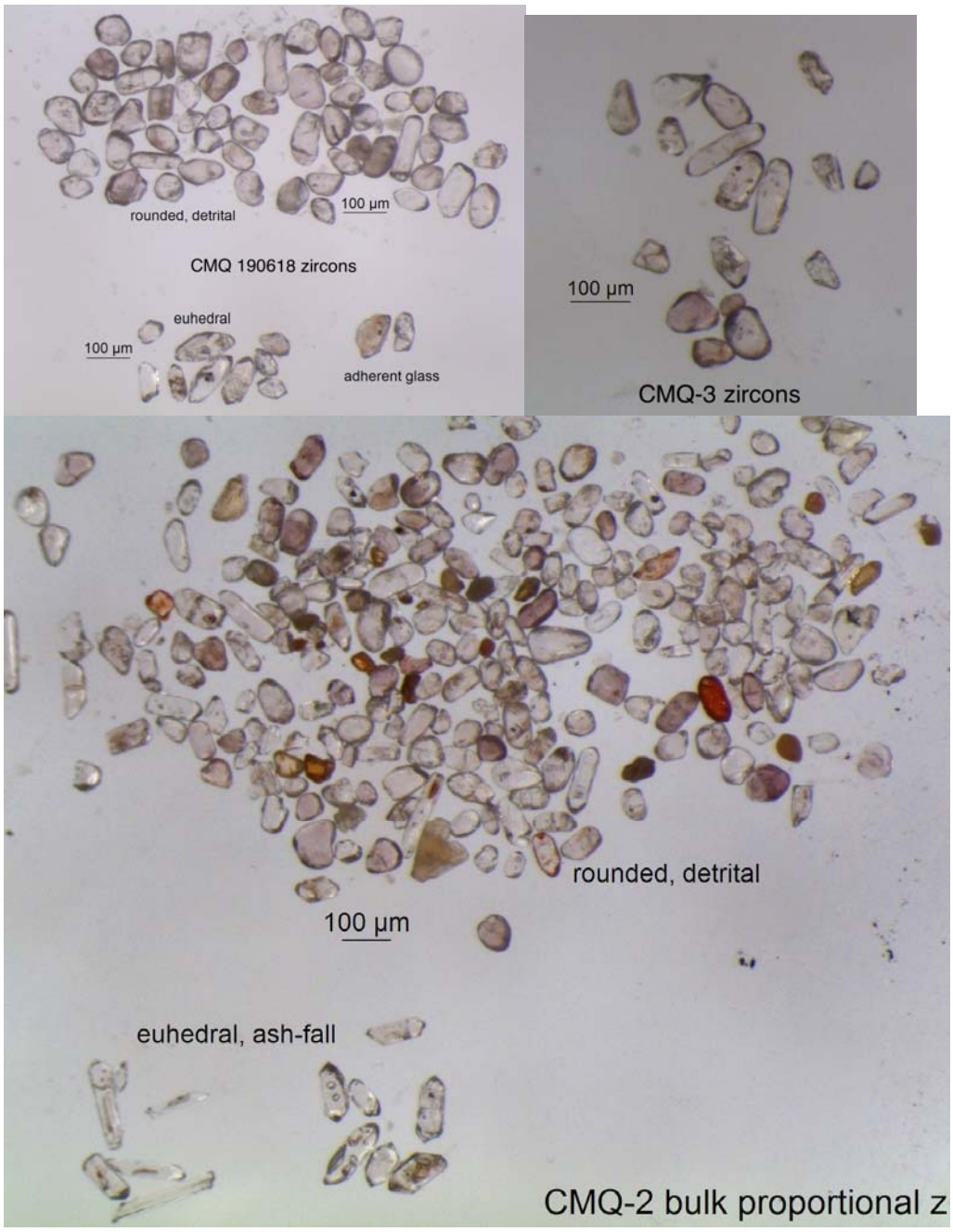

Figure 1. Typical zircon recoveries from 3 tuffs collected from the Cisco Mammal Quarry (Upper Jurassic Morrison Formation, Utah, USA), with subpopulation morphologies in approximate proportions to their abundances. Rounded grains are detrital and were likely entrained during ashfall deposition; they dominate the zircon populations in each sample. Analytical work focused on the euhedral and ash-fall subpopulations.

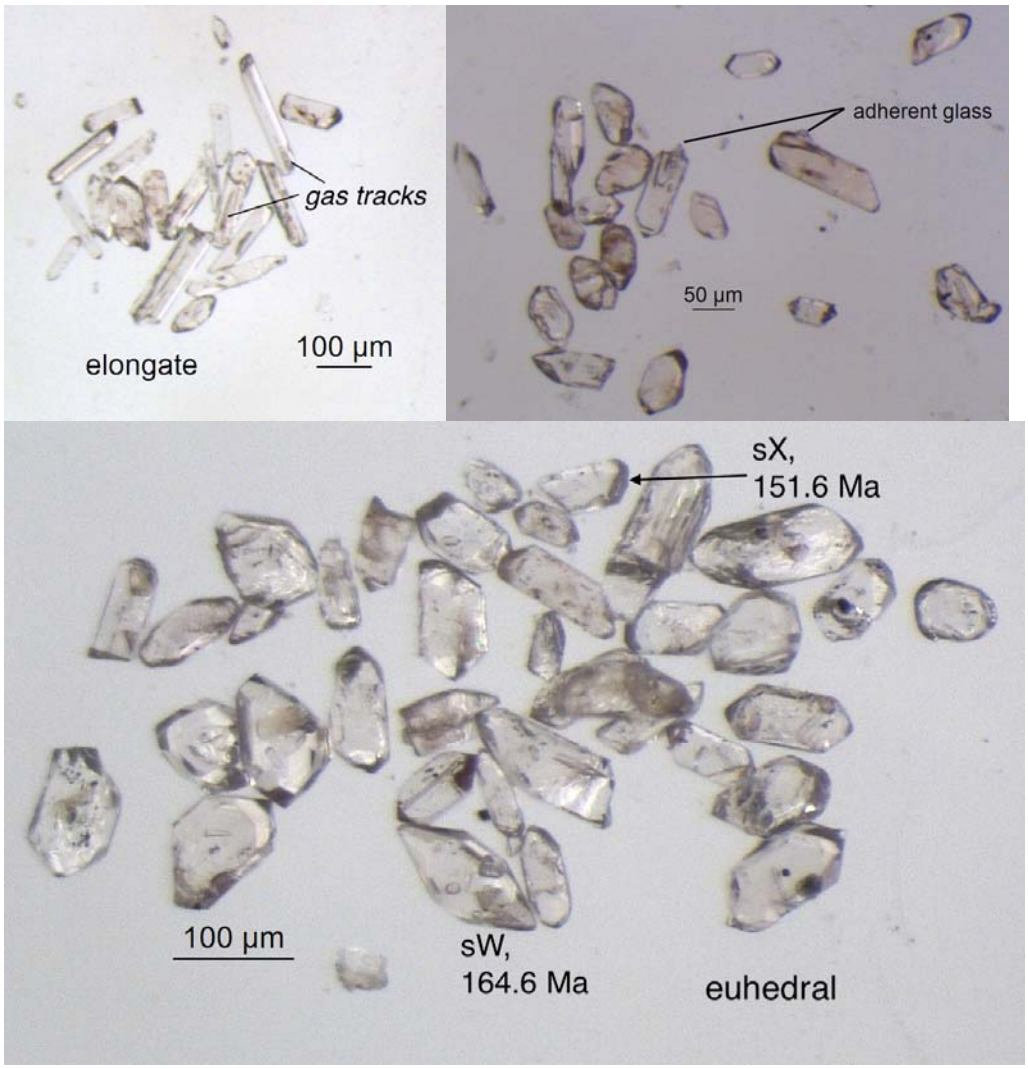

Figure 2. Morphological characteristics of zircons from CMQ-2 (Cisco Mammal Quarry, Upper Jurassic Morrison Formation, Utah, USA), that may indicate a volcanic, ash-fall origin include elongate aspects, longitudinal gas tracks and adherent glass. These subpopulations were dated exclusively by CA-ID-TIMS, but still included some older, pre-eruptive zircons (e.g. euhedral sW 164.6±0.6 Ma).

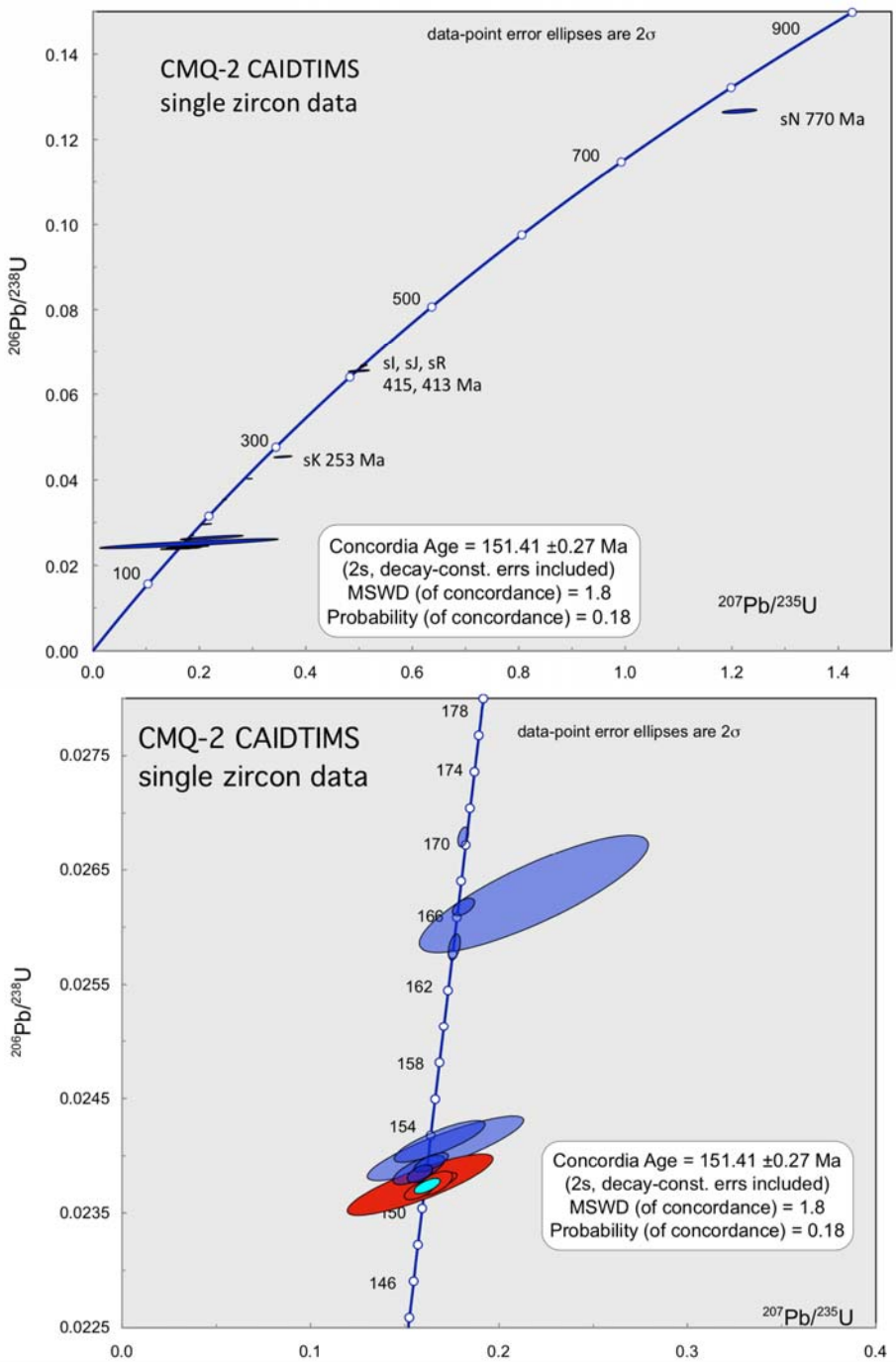

Figure 3. Upper: Concordia plot of CA-ID-TIMS dissolution data from 22 individual zircons from CMQ-2 (Cisco Mammal Quarry, Upper Jurassic Morrison Formation, Utah, USA). Despite their euhedral morphologies, many of the zircon dates are from detrital grains. Lower: close up of concordia plot for zircons with dates <172 Ma. The 4 youngest grains (red ellipses) overlap and produce a Concordia Age of 151.41±0.27 Ma. Grains with older dates are interpreted to reflect antecrystic, pre-eruptive zircons and inherited grains entrained during eruption.

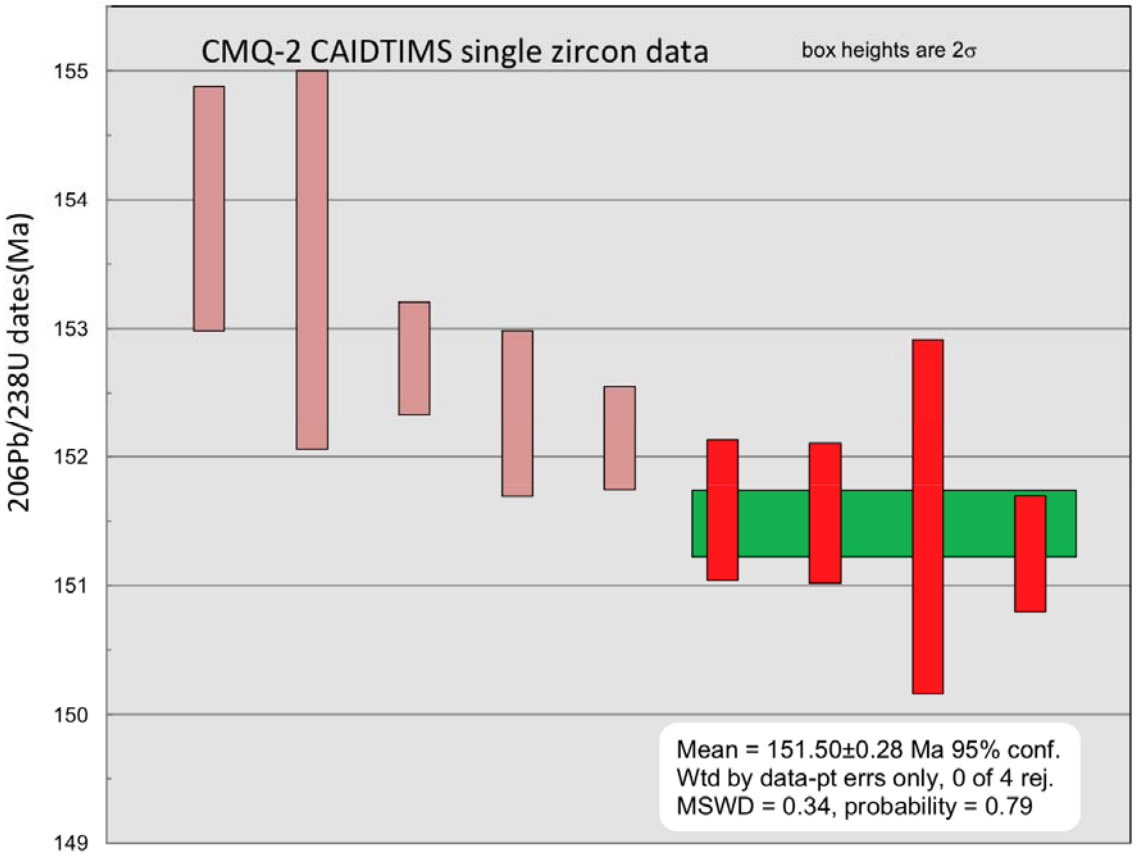

Figure 4. $^{206}Pb/^{238}U$ dates from individual CA-ID-TIMS analyses of zircons from CMQ-2 (Cico Mammal Quarry, Upper Jurassic Morrison Formation, Utah, USA), with dates <155 Ma. Weighted mean date of the four youngest dates is interpreted as the best estimate of the volcanic, ash fall age.