

The Richard's Spur locality

The fissure fills from Richard's Spur (previously termed the Fort Sill deposit) were first discovered in 1932 and have been the subject of many paleontological studies since that time. The fissures are enclosed within steeply dipping beds of the Ordovician Arbuckle Limestone, and were thought to have been open during the Early Permian extending near vertically downward to depths in excess of 30m (Olson, 1991). Detailed studies have proved impossible due to active quarrying at the site and thus the exact morphology of the fissures remains uncertain; it is possible, however, that some may represent subterranean caves (Sullivan et al, 2000). Available evidence indicates that the fissures were gradually filled with a combination of clays and conglomerates containing abundant and well preserved, but generally disarticulated, tetrapod bones. Speleothem deposition is common and sometimes covers the bones (e.g., Fig DR1). The fossils presumably represent some combination of animals that inhabited the fissures, animals that accidentally fell into them, and carcasses or partial carcasses that were washed in from elsewhere. The precise age of the fossiliferous sediments has proved difficult to determine since they are both an isolated occurrence and the variety of species present provide conflicting biostratigraphic correlations (Sullivan & Reisz, 1999). Most previous interpretations have been based on the composition of the terrestrial vertebrate fauna and the fissure fill deposit has thus been considered to be stratigraphically equivalent to the lowermost Clear Fork Group (Leonardian: Lower Permian) of Texas (Reisz, 2005).

U-Pb geochronology

Sawn and polished speleothem slabs were sampled with a dental drill to extract 50-100 mg sub-samples from a single growth horizon. At likely growth rates in the range 5-100 mm/ka, the sampled horizon is likely to represent a growth interval of ≤ 1 ka. All subsequent operations were conducted in a multiple carbosorb and HEPA-filtered clean-room environment. Surficial contamination from sawing and drilling were removed by etching the sample surfaces (2x2-minutes, 1M HCl), followed by rinsing in distilled water and drying in clean air. The dry samples were then weighed into acid-cleaned polymethylpentene beakers, dissolved in dilute HCl and spiked with a mixed ^{233}U - ^{205}Pb tracer. U and Pb were extracted with AG1-X8 and TRU ion exchange resins on small (0.1 ml) teflon columns. Total procedural blanks are estimated to be 10 ± 5 pg for Pb. Separated U and Pb fractions were analysed on a Nu Plasma multi-collector ICPMS using an external mass bias correction for Pb, and the natural $^{238}\text{U}/^{235}\text{U}$ ratio for mass bias correction in uranium. Blank corrections and isotope dilution calculations were performed using the most recent version of the software from Schmitz and Schoene (2007). U-Pb data uncorrected for common Pb were plotted in the Tera-Wasserburg concordia diagram and an isochron age calculated using ISOPLOT Ex.3.5 (Ludwig, 2003). See Table DR1, Figure DR2. Given the antiquity of the sample, corrections for initial disequilibrium in the U-series decay chain were not undertaken.

U-series analyses

A single ~150 mg sub-sample, free from macroscopic cracks, was analyzed for ^{230}Th , ^{234}U , ^{238}U , and ^{232}Th (Table DR1). The sample was dissolved and spiked, with U and Th fractions separated using Fe co-precipitation and ion exchange techniques (Edwards et al., 1987). The isotopic composition of the purified U and Th fractions were determined on a Thermo-Finnigan Element mass spectrometer (Shen et al., 2002). Reported activity ratios are calculated from atomic ratios using half-life values measured or reported in reference (Cheng et al, 2000). The $^{230}\text{Th}/^{234}\text{U}$ value is within error of secular equilibrium. The $^{234}\text{U}/^{238}\text{U}$ value is close to equilibrium, but with a measurable deficit of ^{234}U of about 1.6%, indicating some open-system behavior in the past ~0.5 Ma, likely loss of ^{234}U . To place a maximum limit on the possible affect of ^{234}U loss on the U-Pb age, we can envision an extreme end-member where loss took place continuously throughout the past ~290 Ma. The deficit in radiogenic ^{206}Pb would be ~1.6%, corresponding to a lower age by ~3.6 Ma. The actual likely offset is much smaller as we only have evidence of open system behavior for the last ~0.5 Ma (~0.15% of the age) and, in fact, if uranium had been mobile at this level for any significant portion of the sample's age its observed primary structure would have been obliterated, which is clearly not the case given the elemental imaging results. Even if such ^{234}U loss had persisted over several tens of Ma the age offset would be negligible, thus the uranium-series data broadly support the accuracy of the U-Pb chronology.

Stable isotope analyses

Low resolution traverse

The low resolution $\delta^{18}\text{O}$ profile from speleothem P-1 was measured in the Keck Paleoenvironmental & Environmental Stable Isotope Laboratory in the Department of Geology at the University of Kansas using a Kiel Carbonate Device III and a Finnigan MAT253 isotope ratio mass spectrometer. Samples were drilled by hand with a dental drill in five groups spaced along the growth axis of the polished longitudinal section of P-1. From each sample, ca. 20-80 μg of calcite was roasted under vacuum at 200° C for one hour to release any volatile compounds. Samples and standards were acidified at 75° C using 100% phosphoric acid and isotope measurements were made on the CO_2 evolved from the reaction. Sample results were normalized using measurements of NBS-18 and NBS-19. Analytical precision for $\delta^{18}\text{O}$ is 0.01‰. Data can be provided on request.

High resolution traverse

Stable isotopes were measured on 0.8 +/- 0.1 mg samples extracted using a milling machine operating at 0.2 mm increments, and run on a GV Instruments GV2003 continuous-flow isotope ratio mass spectrometer at the University of Newcastle, Australia. Powders were acidified at 70°C using 105% phosphoric acid and isotope measurements made on the CO_2 evolved from the reaction. Sample results were converted to the conventional 'delta' notation on the Vienna Pee Dee Belemnite scale using an internal standard of Carrara Marble (NEW-1) previously calibrated to NBS-19. Analytical precision for C and O are better than 0.05 and 0.10‰ respectively. Data can be provided on request.

Trace element analyses

Trace element compositions were determined by laser ablation ICPMS using a Varian 810 quadrupole ICPMS coupled to a 193 nm excimer ‘HelEx’ laser ablation system which allows the ablation of highly elongate ‘slits’ rather than spots to achieve maximum spatial resolution in finely banded targets (Woodhead et al, 2005). The long time series were acquired by traversing a slit ~400 x 25 µm across the sample using a 20 µm/second translation rate and 10 Hz repetition rate. Prior to analysis sample surfaces were cleaned by ‘pre-ablation’ at 100 µm/second and 20 Hz. For the high-resolution traverses of ‘annual banding’ a slit of ~100 x 3 µm was translated across the sample at a rate of 1 µm/second. Elemental maps were constructed as described Woodhead et al (2007) from multiple traverses using a 90 µm spot traversing the sample at a rate of 150 µm/second, conducted over a period of around 3 hours, and stitched together using the Iolite software (Hellstrom et al., 2008).

TABLE DR1. U-Th-Pb data

Samples are corrected for analytical blank, spike, and mass fractionation using algorithms of Schmitz & Schoene (2007). Errors are 2-sigma, Pb*/Pbc is the ratio of radiogenic to common Pb.

| Sample/aliquot | U ppm | Pb ppm | Pb*/Pbc | ²³⁸ U/ ²⁰⁶ Pb | % error | ²⁰⁷ Pb/ ²⁰⁶ Pb | % error | ²⁰⁴ Pb/ ²⁰⁶ Pb | %error |
|----------------|-------|--------|---------|-------------------------------------|---------|--------------------------------------|---------|--------------------------------------|--------|
| 1 | 0.24 | 0.015 | 2.38 | 19.3141 | 0.67 | 0.13940 | 3.70 | 0.00594 | 5.94 |
| 2 | 0.20 | 0.012 | 2.60 | 19.6044 | 1.39 | 0.12775 | 8.46 | 0.00516 | 14.33 |
| 3 | 0.15 | 0.010 | 2.17 | 19.1498 | 0.49 | 0.14771 | 2.50 | 0.00652 | 3.88 |
| 4 | 0.17 | 0.023 | 0.62 | 13.7982 | 0.37 | 0.33793 | 0.62 | 0.01959 | 0.74 |
| 5 | 0.18 | 0.016 | 1.10 | 16.8103 | 0.43 | 0.22982 | 1.26 | 0.01216 | 1.63 |
| 6 | 0.20 | 0.026 | 0.65 | 13.9745 | 0.73 | 0.32949 | 1.28 | 0.01892 | 1.53 |
| 7 | 0.22 | 0.020 | 1.01 | 16.4425 | 0.80 | 0.24209 | 2.20 | 0.01297 | 2.81 |
| 8 | 0.23 | 0.013 | 3.37 | 20.0302 | 0.49 | 0.11471 | 3.34 | 0.00427 | 6.16 |
| 9 | 0.24 | 0.013 | 3.55 | 20.1160 | 0.33 | 0.11208 | 2.34 | 0.00411 | 4.37 |
| 10 | 0.25 | 0.013 | 3.88 | 20.2616 | 0.23 | 0.10797 | 1.67 | 0.00380 | 3.27 |
| 11 | 0.18 | 0.015 | 1.22 | 17.2411 | 0.25 | 0.21618 | 0.79 | 0.01118 | 1.05 |

U-series data

| ^{238}U (ppb) | ^{232}Th (ppb) | $^{230}\text{Th}/^{232}\text{Th}$ (atomic, $\times 10^6$) | $\delta^{234}\text{U}$ | $^{230}\text{Th}/^{238}\text{U}$ (activity) |
|------------------------|-------------------------|--|------------------------|---|
| 159.1 ± 0.2 | 53.8 ± 0.5 | 48.0 ± 0.5 | -15.9 ± 1.6 | 0.9866 ± 0.0016 |



Figure DR1. The association of vertebrate fossils and speleothems at the Richards Spur site. Cave deposit flowstone that has precipitated in and around the skull and partial postcranial skeleton of the Early Permian reptile *Captorhinus aguti*. Specimen was found at the Dolese Brothers Limestone quarry, near Richards Spur, Oklahoma, together with numerous other cave speleothems, including stalagmites and stalactites.

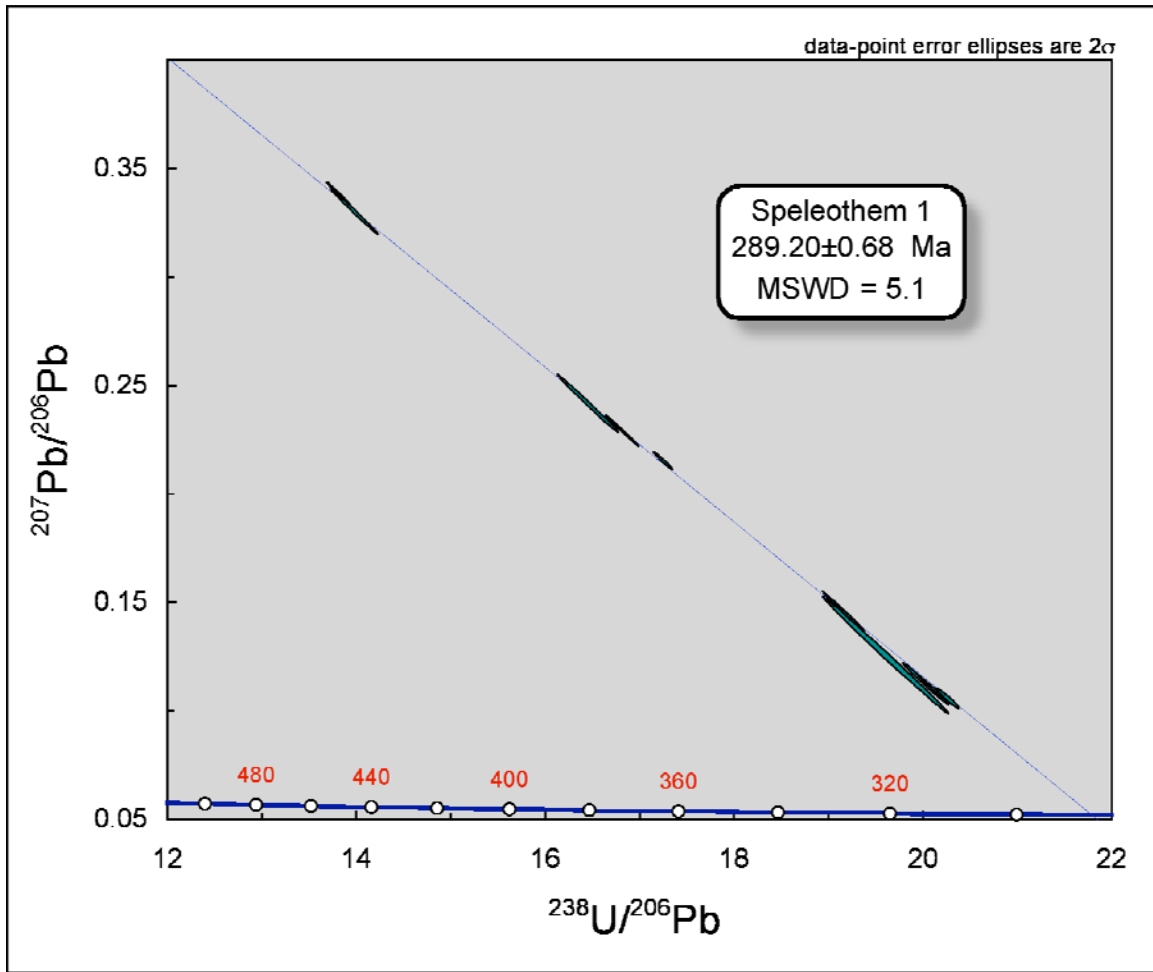


Figure. DR2. Speleothem geochronology. Semi-total U-Pb isochron (‘Tera-Wasserburg’ plot) for sample P-1.

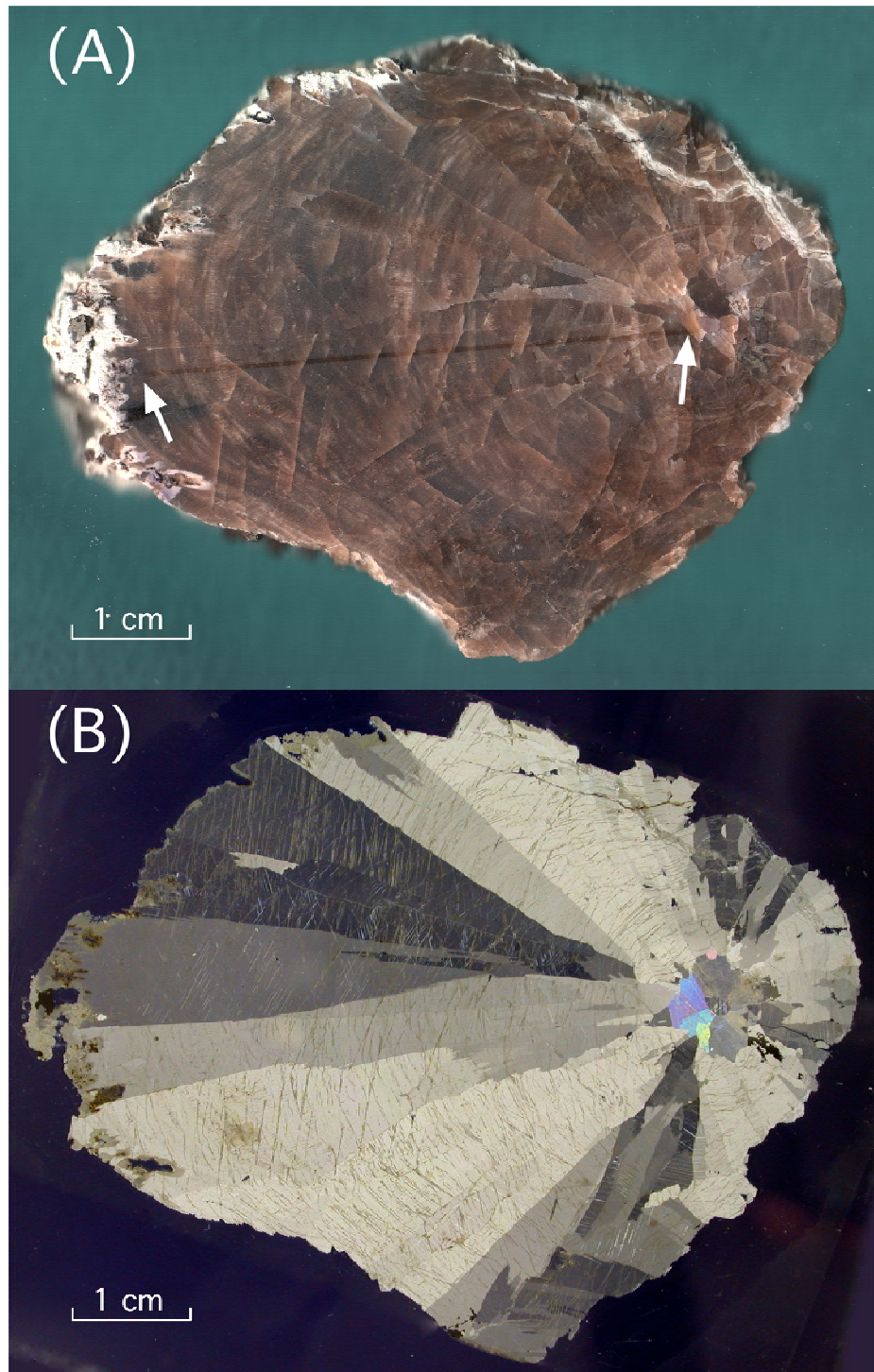


Figure DR3. Sections through Permian speleothem P-2. (A), polished transverse section showing brown calcite and concentric growth layering, with extensive cracking. A single laser traverse can be seen passing from core to rim and is indicated by white arrows at either end. The area milled for stable isotope analysis (Fig 3B-C) was obtained parallel and adjacent to this laser traverse (B) thin section of the same speleothem imaged under

crossed polars. Large columnar calcite crystals emerge from a central area of mosaic calcite.

Supplemental References

- Cheng, H., Edwards, R. L., Hoff, J., Gallup, C. D., Richards, D. A. & Asmerom, Y., 2000, The half-lives of uranium-234 and thorium-230: *Chemical Geology* **169**, 17-33.doi: 10.1016/S0009-2541(99)00157-6.
- Edwards, R.L., Chen, J.H. & Wasserburg, G.J., 1987, ^{230}Th - ^{232}Th systematics and precise determination of time over the past 500,000 years *Earth Planet. Sci. Lett.*, **81**, 175-192.doi: 10.1016/0012-821X(87)90154-3.
- Gradstein, F., Ogg, J. and Smith, A. (Eds), 2004, *A Geologic Time Scale 2004*, Cambridge University Press.
- Hellstrom, J., Paton, C., Woodhead, J. & Hergt, J., 2008, Iolite: software for spatially resolved LA-(quad and MC) ICPMS analysis in *Laser Ablation ICP-MS in the Earth Sciences: Current Practices and Outstanding Issues*, Sylvester, P (Ed) Mineralogical Association of Canada short course series **40**, 343-348.
- Henderson, G.M., 2006, Caving in to new chronologies: *Science* 313, 620-623.doi: 10.1126/science.1128980
- Ludwig, K.R., 2003, *Users manual for Isoplot, a Geochronological toolkit for Microsoft Excel*. Berkeley Geochronology Center Special Publication no.4.
- Olson, E.C.,1991, An Eryopid (Amphibia: Labyrinthodontia) from the Fort Sill fissures, Lower Permian, Oklahoma: *Journal of Vertebrate Paleontology* v. 11, p.130-132.
- Reisz, R.R., 2005, Oromycter, a new caseid from the Lower Permian of Oklahoma: *Journal of Vertebrate Paleontology* v. 25, p.905-910.

- Schmitz, M.D. & Schoene, B., 2007, Derivation of isotope ratios, errors and error correlations for U-Pb geochronology using ^{205}Pb - ^{235}U -(^{233}U)-spiked isotope dilution thermal ionization mass spectrometry. *Geochemistry Geophysics Geosystems* **8**, doi:10.1029/2006GC001492.
- Shen, C.-C., Edwards, R.L., Cheng, H., Dorale, J.A., Thomas, R.B., Moran, S.B., Weinstein, S.E. & Edmonds, H.N., 2002, Uranium and thorium isotopic and concentration measurements by magnetic sector inductively coupled plasma mass spectrometry: *Chemical Geology* **185**, 165-178. doi: 10.1016/S0009-2541(01)00404-1
- Sullivan, C & Reisz, R.R., 1999, First record of Seymouria (Vertebrata: Seymouriamorpha) from Early Permian fissure fills at Richard's Spur, Oklahoma: *Canadian Journal of Earth Sciences* v. 36, p.1257-1266.
- Woodhead, J.D., Swearer, S., Hergt, J. & Maas, R., 2005, In situ Sr-isotope analysis of carbonates by LA-MC-ICP-MS: interference corrections, high spatial resolution and an example from otolith studies: *Journal of Analytical Atomic Spectrometry* **20**, 22-27. doi: 10.1039/b412730g.
- Woodhead, J., Hellstrom, J., Hergt, J., Greig, A. & Maas, R., 2007, Isotopic and elemental imaging of geological materials by laser ablation inductively coupled plasma mass spectrometry: *Journal of Geostandards and Geoanalytical Research*, **31**, 331-343.