

GSA DATA REPOSITORY ITEM 2003133

APPENDIX: Materials and Methods

Chronology

The radiocarbon chronology is adjusted from a previously published Hall's Cave chronology (Toomey, 1993; Toomey et al., 1993) and is based on a subset of 143 AMS ^{14}C measurements made by T.W. Stafford. Radiocarbon dated materials from the Hall's Cave deposit include cave sediment humins, humic acids, purified gelatin from bone collagen, and charcoal. Conventional (decay counting) radiocarbon ages (y BP) were calculated using a ^{14}C half-life of 5568 y and corrected for isotope fractionation using the $\delta^{13}\text{C}$ value measured for each sample. Calendar ages (y BP) were calibrated using OxCal version 3.8 (Bronk Ramsey, 1995, 2001) with the 1998 atmospheric decadal dataset as a calibration curve (Stuiver et al., 1998). The 2 σ (95.4% probability range) maximum and minimum calendar ages are given in Table DR1. Linear regression of the maximum and minimum age of each stratum as a function of depth in the deposit was used to derive the Hall's Cave chronology and calculate erosion rates.

Materials

Fossil materials from Hall's Cave were collected at 5 cm intervals during excavations conducted by R.S. Toomey in the late 1980's to early 1990's. Specimens used for radiocarbon dating and Sr isotope analyses are archived in the Hall's Cave collection at the Vertebrate Palaeontology Laboratory of the Texas Memorial Museum, The University of Texas at Austin as TMM locality number 41229. Hackberry seeds are identified as *Celtis*.

and include uncatalogued specimens and catalogued specimen numbers TMM 41229-7946, 10012, 4221, 10296, 10297, 5115, 10912, and 10919. Rodent tooth enamel is from pocket gophers identified as *Geomys* (uncatalogued and catalogued specimen numbers TMM 41229-8019, 12195, 12196, 12197, and 12200) and voles identified as *Microtus* (specimen numbers TMM 41229-4604, 4603, and 1090). Modern vegetation (grasses and hackberry), as well as samples of limestone bedrock and soils were collected from thin soils (pits 1 and 2) within ~300 m of the entrance to Hall's Cave and from thick upland soils (pits 3 and 4) in the Kerr Wildlife Management Area, Kerr County, TX, approximately 5 km southeast of Hall's Cave. Soil samples were taken from representative horizons in each soil pit.

Sample Preparation for $^{87}\text{Sr}/^{86}\text{Sr}$ Analysis

Rodent tooth enamel was physically isolated from the associated dentin and bone by rotary drilling or picking with carbide-tipped dental tools. Enamel was sonicated in deionized (DI) H₂O and leached in 5 to 8 % acetic acid for 8 to 10 minutes to remove surface contamination. Aragonite mineralogy of hackberry seeds was determined by using powder x-ray diffraction. With the exception of the 20.8 ka hackberry sample that did not have enough material for x-ray diffraction analysis, only hackberries with aragonite mineralogy were used in this study. Aragonite mineralogy is defined here as those hackberry samples having an aragonite x-ray diffraction peak (hkl 111) intensity greater than the calcite peak (hkl 104) intensity. Aragonite-calcite mixture calibrations suggest these samples are aragonite-calcite mixtures that are 80 to 100 % aragonite. Hackberry seed and limestone bedrock samples were sonicated in DI H₂O to remove surface contaminants and treated with a 10-minute ion exchange in 0.5 N ammonium acetate (buffered to pH 8) to remove exchangeable Sr.

Approximately 0.6 to 0.8 g of grass leaves were sonicated in DI H₂O, dried, and ashed at 500 °C for ~24 hours. Soil samples were dried overnight at 80 °C.

Sr Separation Techniques

Approximately 0.1 to 2 mg of rodent tooth enamel separates were dissolved in concentrated nitric acid. Approximately 1 to 18 mg of hackberry aragonite and 14 to 26 mg of limestone bedrock samples were dissolved in 3 N nitric acid and centrifuged to remove insoluble residues. Leaf ash was dissolved in aqua regia. Exchangeable Sr from 1 to 2 g of dried soils was obtained by ion exchange with 1 N ammonium acetate for ~10 minutes at room temperature. After centrifuging, the supernatant was dried down and dissolved in 7 N nitric acid. All samples were re-dissolved in 3 N nitric acid for Sr separation on ion-specific resin (Sr-Spec) columns.

Analytical Methods

Samples were analyzed for ⁸⁷Sr/⁸⁶Sr ratios by dynamic multi-collection using a Finnigan – MAT 261 thermal ionization mass spectrometer at The University of Texas at Austin (Tables DR2 and DR3). Samples were loaded with 0.3 M phosphoric acid onto a tantalum oxide slurry on zone-refined Re filaments. Total procedural blanks ranged between 18 and 84 pg Sr. The ⁸⁷Sr/⁸⁶Sr ratios reported include statistical data reduction and corrections for ⁸⁷Rb and instrumental fractionation (Banner and Kaufman, 1994). Internal precision for a given analysis ranged from 0.6×10^{-6} to 1.1×10^{-5} for hackberry aragonite, limestone, modern vegetation, and soil leachates and from 1×10^{-5} to 2×10^{-4} for rodent tooth

enamel samples. External 2σ is 1.6×10^{-5} based on the standard deviation of the population of 23 NIST standard NBS 987 analyses for which the mean $^{87}\text{Sr}/^{86}\text{Sr}$ value is 0.710271.

Erosion Rate Uncertainty

To calculate the lower boundary erosion rate of 1-2 cm/k.y., we used the $\sigma_{\text{S.I.R.}/\sigma_h}$ correlation of modern soils and the $\sigma_{\text{S.I.R.}/\sigma_{\text{k.y.}}}$ correlation of fossils (see Figs. 2 and 3 in text). Orthogonal regression of $^{87}\text{Sr}/^{86}\text{Sr}$ ratios through time from 20.8-9.4 ka (excluding the outlier at 18.4 ka) for fossil hackberries and from 16.8-4.4 ka for fossil pocket gophers yields rates of change of $6 \times 10^{-5}/\text{cm}$ and $7 \times 10^{-5}/\text{cm}$ ($\pm 1 \times 10^{-5}/\text{cm}$, 1σ) for the hackberries and pocket gophers, respectively. The $\sigma_{\text{S.I.R.}/\sigma_h}$ of modern soils pits is $5 \times 10^{-5}/\text{cm}$ ($\pm 6 \times 10^{-6}/\text{cm}$, 1σ) (determined from orthogonal regression). Using these regression statistics, we determine an erosion rate range of 1-2 cm/k.y.

The method used to calculate the erosion rate of 11 cm/k.y. relies on soil-depth and time constraints inferred from the isotopic data and paleontological soil-depth indicators. Here, we assign an uncertainty to the soil-depth and time constraints to determine the erosion rate uncertainty. Paleontological records suggest the local extinction of the prairie dog, *Cynomys*, occurs at 12 ka \pm 1 ka. At this time, we use the minimum burrow depth of modern prairie dogs to assume a minimum soil-depth of 100 cm. The stabilization of pocket gopher, *Geomys*, $^{87}\text{Sr}/^{86}\text{Sr}$ ratios occurs at 5 ka \pm 1 ka (Fig. 3 in text). Thus, we interpret that the modern soil thickness of 20 cm \pm 10 cm was established and stabilized by this time. Using these soil-depth and time constraints, we arrive at a minimum erosion rate of 11 cm/k.y. with lower and upper bounds of 8 cm/k.y. and 18 cm/k.y.

References Cited

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TABLE DR1. HALL'S CAVE DEPOSIT CHRONOLOGY

TMM catalog number	Depth (cm)	Material	RC (y BP) (+/- 1 σ)	Cal. max.* (y BP)	Cal. min.* (y BP)
41229-12115	15-20	gelatin	1500 +/- 60	1520	1300
41229-12118	25-30	gelatin	2330 +/- 60	2750	2150
41229-12083	51	charcoal	3190 +/- 70	3580	3240
41229-12117	60-65	gelatin	4000 +/- 60	4850	4250
41229-12162	90-95	gelatin	5320 +/- 60	6280	5930
41229-12099	76-78	humins	5400 +/- 70	6310	5990
41229-12164	105-110	gelatin	7700 +/- 80	8640	8350
41229-12166	120-125	gelatin	8630 +/- 60	9780	9490
41229-12080	145-150	gelatin	10310 +/- 70	12850	11650
41229-12075	155-160	liquified gelatin	11310 +/- 60	13800	13000
41229-12173	165-170	gelatin	11410 +/- 70	13800	13100
41229-N.D.	185-190	gelatin	11550 +/- 70	13900	13150
41229-12176	195-200	gelatin	12110 +/- 90	15350	13650
41229-12073	220-225	gelatin	12570 +/- 80	15550	14150
41229-12179	270-275	gelatin	13940 +/- 100	17350	16150
41229-12177	210-215	liquified gelatin	14400 +/- 80	17850	16650
41229-12076	235-240	gelatin	14700 +/- 90	18250	17050
41229-12137	260	humic acid	15290 +/- 90	18950	17650
41229-12180	295-300	gelatin	16240 +/- 100	20050	18650
41229-12183	315-320	gelatin	16510 +/- 100	20350	18950
41229-12131	338-343	humic acid	16610 +/- 110	20550	19050
41229-12181	300-305	gelatin	16620 +/- 110	20550	19150
41229-12184	345-350	gelatin	16770 +/- 100	20650	19250

Note: Conventional radiocarbon ages (RC) for Hall's Cave. Calendar (cal.) ages calibrated with OXCAL 3.8. Maximum and minimum ages represent 2 (95.4% probability) range.

TABLE DR2. $^{87}\text{Sr}/^{86}\text{Sr}$ OF HALL'S CAVE FOSSILS

Sample	Material	$^{87}\text{Sr}/^{86}\text{Sr}$	Cal. age (y BP)
H18	hackberry aragonite	0.708492	1.8
H17	hackberry aragonite	0.708434	2.7
H16	hackberry aragonite	0.708460	3.5
H15	hackberry aragonite	0.708453	4.4
H14	hackberry aragonite	0.708501	5.2
B9	hackberry aragonite	0.708419	5.2
H13	hackberry aragonite	0.708453	6.0
H12	hackberry aragonite	0.708494	6.9
H11	hackberry aragonite	0.708387	7.7
H10	hackberry aragonite	0.708779	8.6
H10A	hackberry aragonite	0.708372	8.6
H10B	hackberry aragonite	0.708362	8.6
H10C	hackberry aragonite	0.708387	8.6
H10D	hackberry aragonite	0.708378	8.6
H9	hackberry aragonite	0.708540	9.4
H8	hackberry aragonite	0.708409	10.3
H7	hackberry aragonite	0.708422	11.1
H6	hackberry aragonite	0.708747	11.9
H5C	hackberry aragonite	0.708929	12.8
H5D	hackberry aragonite	0.708684	12.8
HPT2	hackberry aragonite	0.708613	12.8
H4	hackberry aragonite	0.708653	13.2
B5	hackberry aragonite	0.708958	14.4
H1	hackberry aragonite	0.708526	15.2
HPT1	hackberry aragonite	0.708571	15.2
HA	hackberry aragonite	0.708680	15.4
HE	hackberry aragonite	0.708983	17.2
HB	hackberry aragonite	0.708808	18.2
B1	hackberry aragonite	0.709513	18.4
HBOLD2	hackberry aragonite	0.709055	19.6
HBOLD3	hackberry aragonite	0.708841	19.6
HBOLD1	hackberry aragonite	0.708963	20.8
G2e	pocket gopher enamel	0.708800	2.3
G3e	pocket gopher enamel	0.708720	4.4
Lyn1	pocket gopher enamel	0.708450	6.0
G4e	pocket gopher enamel	0.708940	6.9
G5e	pocket gopher enamel	0.708616	7.3
G6e	pocket gopher enamel	0.708963	8.6
Lyn2	pocket gopher enamel	0.708890	8.6
Lyn4	pocket gopher enamel	0.709140	11.9
Lyn5	pocket gopher enamel	0.709370	15.0
Lyn6	pocket gopher enamel	0.709280	16.8
1e	vole enamel	0.708938	12.8
2e	vole enamel	0.709108	12.8
3e	vole enamel	0.709211	18.2

Note: Ages are interpolated from the Hall's Cave chronology (Table DR1).

TABLE DR3. $^{87}\text{Sr}/^{86}\text{Sr}$ RATIOS OF MODERN EDWARDS PLATEAU
ECOSYSTEM COMPONENTS

Sample	Location	Material	$^{87}\text{Sr}/^{86}\text{Sr}$	Depth* (cm)
<u>Modern Soils</u>				
Pit 1, A horizon	Hall's Ranch	leachate	0.708143	0-5
Pit 1, B1 horizon	Hall's Ranch	leachate	0.708153	5-14
Pit 1, B2 horizon	Hall's Ranch	leachate	0.708149	14-18
Pit 1, Average	Hall's Ranch	leachate	0.708148	18
Pit 2, A horizon	Hall's Ranch	leachate	0.708408	0-12
Pit 3, A horizon	KWMA	leachate	0.709968	0-4
Pit 3, B1 horizon	KWMA	leachate	0.710122	4-17
Pit 3, B2 horizon	KWMA	leachate	0.710296	17-30
Pit 3, B3 horizon	KWMA	leachate	0.710701	30-39
Pit 3, B4 horizon	KWMA	leachate	0.710781	39-52
Pit 3, Average	KWMA	leachate	0.710374	52
Pit 4, A horizon	KWMA	leachate	0.710120	0-9
Pit 4, B1 horizon	KWMA	leachate	0.710802	9-35
Pit 4, B2 horizon	KWMA	leachate	0.711130	35-52
Pit 4, B3 horizon	KWMA	leachate	0.711066	52-70
Pit 4, Average	KWMA	leachate	0.710780	70
<u>Bedrock</u>				
Pit 1, LS bedrock	Hall's Ranch	limestone	0.707509	18
Pit 4, LS bedrock	KWMA	limestone	0.707652	70
thin soil, LS bedrock	KWMA	limestone	0.707785	12
LS bedrock, near HB tree	KWMA	limestone	0.707927	0
<u>Vegetation</u>				
Hackberry aragonite, near Pit 2	Hall's Ranch	hackberry	0.708450	surface
Pit 1, grass	Hall's Ranch	grass	0.708233	surface
Pit 3, grass	KWMA	grass	0.709824	surface
<i>Note:</i> Samples were collected in Kerr County, Texas from Hall's Ranch, approximately 300 m or less from the entrance to Hall's Cave and the Kerr Wildlife Management Area (KWMA).				
*Sample depth from soil surface.				