

1 **Pigati et al – GSA Bulletin**

2

3 **SUPPLEMENTAL INFORMATION**

4 *Radiocarbon dating of terrestrial gastropod shells*

5 Gastropod shells were recovered from Stratum D_{gsc} in the east flank at Stations 8, 9, 17,
6 and 18 and Stratum E_{2c} in the east flank at Stations 1 and 3. The shells were treated with 3%
7 H₂O₂ for 48 hours at room temperature to remove residual organic matter, and rinsed repeatedly
8 with ultrapure water (ASTM Type I, 18.2 MΩ) before being dried in a vacuum oven at 70 °C.
9 We selected several shells at random for X-ray diffraction analysis to verify that only shell
10 aragonite remained prior to preparation for ¹⁴C analysis. We did not find any evidence of calcite
11 in the shells that we analyzed, which indicates that there has not been any recrystallization or
12 addition of secondary calcite to the shell surface. CO₂ was extracted from the shell aragonite by
13 hydrolysis and converted to graphite via catalytic reduction of CO (after Slota et al., 1987).
14 Graphite targets were submitted to the Arizona Accelerator Mass Spectrometry (AMS) Facility
15 for ¹⁴C analysis.

16

17 *Luminescence dating*

18 IRSL analyses were carried out in subdued orange-light conditions at the U.S. Geological
19 Survey's Luminescence Dating Laboratory in Denver, CO using the total-bleach multiple-aliquot
20 additive-dose (MAAD) method (Singhvi et al., 1982; Lang, 1994; Richardson et al., 1997;
21 Forman and Pierson, 2002). Dose rates were determined based on concentrations of K, Rb, U,
22 and Th, which were measured by instrumental neutron activation analysis (INAA). The cosmic-
23 ray dose rate was estimated for each sample as a function of depth, elevation, and geomagnetic

24 latitude, and added to the total dose rate (Prescott and Hutton, 1994). Alpha and beta
25 contributions to the dose rate were corrected for grain-size attenuation as needed (Aitken, 1985).
26 Fading tests were performed on each sample to account for instability in the measured
27 luminescence signal. These tests consisted of measuring the initial luminescence emission and
28 the emission after a set amount of time, usually 21 days after the initial measurement; a ratio of 1
29 indicates a stable luminescence signal (Huntley and Lamothe, 2001). All of the samples
30 measured in this study exhibited a fairly stable signal, requiring corrections on the order of 6-9%.

31

32 *Uranium-series dating of ground-water carbonate*

33 Uranium-series dating of ground-water carbonates relies on the systematic growth and
34 decay of intermediate radioactive daughter products in the ^{238}U decay chain (Ivanovich and
35 Harmon, 1982; Bourdon et al., 2003; Edwards et al., 2003). Uranium that is incorporated into
36 authigenic calcite includes both ^{234}U and ^{238}U in the same ratio that was present in the emergent
37 groundwater (U co-precipitates with Ca). Isotopes of uranium decay at fixed rates, ^{238}U to ^{234}U
38 and ^{234}U to ^{230}Th , which forms the basis for the $^{230}\text{Th}/\text{U}$ geochronometer.

39 Ideally, authigenic carbonates have high U/Th ratios due to the high solubility of U^{6+} as
40 uranyl carbonate complexes in oxidized ground waters and the extremely low solubility of Th^{4+}
41 in most natural waters. However, ground-water discharge (GWD) deposits commonly have
42 appreciable amounts of fine-grained eolian and alluvial material that cannot be physically
43 separated from ground-water carbonate prior to digestion and analysis. As a result, some detrital
44 ^{230}Th may be present initially within the carbonate (i.e., not derived from the *in situ* decay of U)
45 and needs to be accounted for before accurate $^{230}\text{Th}/\text{U}$ age estimates are made. U-series ages
46 presented in Table 4 include a detrital correction that is intended to account for the initial ^{230}Th

47 present at the time of formation (Ludwig and Paces, 2002). Samples with relatively low ^{232}Th
48 abundances (VW9-168 and VW17-170) required smaller corrections and, as a result, have ages
49 that are more precise and likely more accurate than samples with higher ^{232}Th abundances (e.g.,
50 VW22-167 and VW33-169). Age errors were propagated using both analytical uncertainties and
51 uncertainties assigned to the unknown detrital component.

52 Analytical methods follow those described by Ludwig and Paces (2002) and use total
53 digestion of samples to avoid fractionation of U and Th in the laboratory. Purified U and Th
54 salts were run as double and carbon-doped single filament assemblies, respectively, on a Thermo
55 Finnigan Triton thermal ionization mass spectrometer equipped with ion-counting and
56 electrostatic filtering (RPQ) capabilities. Analytical results were normalized using values
57 obtained under the same run conditions using the NIST U-isotope standard SRM4321b (mean
58 $^{234}\text{U}/^{235}\text{U}$ atomic ratio of 0.007306 ± 0.000012 , $2 \times$ standard deviations, over the time period of
59 analyses). Total process blanks were routinely measured and range between 25 and 100 pg for U
60 and Th. An in-house secular equilibrium standard made from uranium ore from the 69-Ma-old
61 Schwartzwalder mine (Ludwig et al., 1985) yielded $^{234}\text{U}/^{238}\text{U}$ and $^{230}\text{Th}/^{238}\text{U}$ activity ratios (AR)
62 of 0.9984 ± 0.0025 and 0.9987 ± 0.0050 , respectively (values for both ratios should be 1.000 if the
63 material is truly in secular equilibrium).

64

65

66
67

Valley Wells sampling locations (refer to Fig. 2)

Station	Latitude (°N)	Longitude (°W)	UTM Easting*	UTM Northing*
1	35.45084	115.67722	620044	3923846
2	35.45024	115.67772	619999	3923778
3	35.45034	115.67892	619890	3923788
4	35.45857	115.68556	619277	3924692
5	35.45828	115.69098	618784	3924655
6	35.45992	115.69424	618486	3924832
7	35.45887	115.68654	619186	3924723
8	35.45406	115.67678	620081	3924202
9	35.45511	115.67769	619995	3924318
10	35.46384	115.68148	619640	3925279
11	35.46942	115.68032	619731	3925901
12	35.45623	115.67385	620341	3924448
13	35.45384	115.67228	620488	3924184
14	35.44247	115.67110	620612	3922924
15	35.44475	115.67104	620613	3923177
16	35.43857	115.66703	620987	3922498
17	35.44945	115.67640	620120	3923692
18	35.44812	115.67453	620292	3923547
19	35.45949	115.69501	618416	3924783
20	35.46629	115.68315	619483	3925551
21	35.45884	115.69096	618785	3924716
22	35.45957	115.69166	618721	3924796
23	35.45035	115.67719	620047	3923791
24	35.45087	115.68067	619730	3923844
25	35.45127	115.67979	619810	3923889
26	35.45156	115.67937	619847	3923922
27	35.45206	115.67856	619920	3923979
28	35.45136	115.67785	619986	3923902
29	35.45864	115.69080	618799	3924694
30	35.45827	115.69104	618778	3924653
31	35.45507	115.69280	618624	3924296
32	35.45691	115.67994	619788	3924515
33	35.46691	115.68171	619612	3925622
34	35.45098	115.67685	620077	3923861
Dug Well	35.46346	115.68217	619575	3925239
Station Well	35.44082	115.67347	620398	3922738

68
69
70
71

* UTM Sheet 11S

72 **References**

- 73
- 74
- 75 Aitken, M. J., 1985, Thermoluminescence Dating: London, Academic Press, 359 p.
- 76 Bourdon, B., Turner, S., Henderson, G. M., and Lundstrom, C. C., 2003, Introduction to U-series
77 geochemistry: Reviews in Mineralogy and Geochemistry, v. 52, p. 1-21.
- 78 Edwards, R. L., Gallup, C. D., and Cheng, H., 2003, Uranium-series dating of marine and
79 lacustrine carbonates: Reviews in Mineralogy and Geochemistry, v. 52, p. 363-405.
- 80 Forman, S. L., and Pierson, J., 2002, Late Pleistocene luminescence chronology of loess
81 deposition in the Missouri and Mississippi river valleys, United States: Palaeogeography,
82 Palaeoclimatology, Palaeoecology, v. 186, no. 1-2, p. 25-46.
- 83 Huntley, D. J., and Lamothe, M., 2001, Ubiquity of anomalous fading in K-feldspars and the
84 measurement and correction for it in optical dating: Canadian Journal of Earth Science, v.
85 38, p. 1093-1106.
- 86 Ivanovich, M., and Harmon, R. S., 1982, Uranium Series Disequilibrium: Applications to
87 Environmental Problems, Clarendon Press, 237 p.
- 88 Lang, A., 1994, Infrared stimulated luminescence dating of Holocene reworked silty sediments:
89 Quaternary Science Reviews, v. 13, no. 5-7, p. 525-528.
- 90 Ludwig, K. R., and Paces, J. B., 2002, Uranium-series dating of pedogenic silica and carbonate,
91 Crater Flat, Nevada: Geochimica et Cosmochimica Acta, v. 66, p. 487-506.
- 92 Ludwig, K. R., Wallace, A. R., and Simmons, K. R., 1985, The Schwartzwalder uranium deposit,
93 II: age of uranium mineralization and Pb-isotope constraints on genesis: Economic
94 Geology, v. 80, p. 1858-1871.
- 95 Prescott, J. R., and Hutton, J. T., 1994, Cosmic ray contributions to dose rates for luminescence
96 and ESR dating: large depths and long-term time variations: Radiation Measurements, v.
97 23, p. 497-500.
- 98 Richardson, C. A., McDonald, E. V., and Busacca, A. J., 1997, Luminescence dating of loess
99 from the northwest United States.: Quaternary Science Reviews, v. 16, no. 3-5, p. 403-
100 415.
- 101 Singhvi, A. K., Sharma, Y. P., and Agrawal, D. P., 1982, Thermo-luminescence dating of sand
102 dunes in Rajasthan, India: Nature, v. 295, no. 5847, p. 313-315.
- 103 Slota, P. J., Jull, A. J. T., Linick, T. W., and Toolin, L. J., 1987, Preparation of small samples for
104 ¹⁴C accelerator targets by catalytic reduction of CO: Radiocarbon, v. 29, no. 2, p. 303-
105 306.
- 106
- 107