

# Age of the Laschamp excursion determined by U-Th dating of a speleothem geomagnetic record from North America

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## MAGNETIC METHODS

The slab used for paleomagnetism, with a cross section of 1.7 cm × 0.7 cm, was sectioned into 38 specimens, ~0.5 cm in thickness each. After measuring the natural remanent magnetization (NRM), each specimen was demagnetized in stepwise fashion with a D-Tech 2000 alternating frequency (AF) demagnetizer, using peak fields progressively increasing on a logarithmically-spaced scale from 0.5 mT until the magnetization reached magnetometer noise levels ( $\sim 10^{-11}$  Am<sup>2</sup>). Remanence at each step was independently measured 30 times on each specimen using a 2G Enterprises superconducting quantum interference device magnetometer. Measurements with high noise/signal ratios were excluded from the calculation of the average values. Standard deviations were on the order of 1 nAm<sup>2</sup>/kg. The characteristic remanence was isolated via principal component analysis (Kirschvink, 1980) using PuffinPlot (Lurcock and Wilson, 2012). We adopted the protocol devised by Peppe et al. (2009) for weak specimens, as follows: to define the characteristic remanence component we used either a) the best-fit line through a minimum of three consecutive demagnetization steps that trended toward the origin and had a maximum angular deviation (MAD) less than 20° (for specimens with quasi-linear trajectories, e.g., Figs. DR2, 2C-D), or b) a minimum of 4 consecutive demagnetization steps anchored to the origin, with MAD < 20° (for specimens with directions clustering around one point, e.g., Fig. 2E). Because the speleothem is not azimuthally oriented, declination values were rotated so that average baseline values were situated in the interval [-10°, 30°], corresponding to realistic field declination values at the time in North America (e.g., Lund et al., 2005; Channell, 2006; Böhnel and Molina-Garza, 2002; Negrini et al., 2014).

Mean declination was obtained by averaging adjusted declinations in the considered azimuthal interval at 1° increments. Virtual geomagnetic pole (VGP) coordinates were calculated at each adjusted declination step, and then combined to obtain mean and standard deviation values. After completing the NRM demagnetization, anhysteretic remanent magnetization (ARM) was imparted in the presence of a 0.05 mT bias field superimposed on an AF field decaying from a peak value of 100 mT, at a rate of 5 µT per half cycle. ARM susceptibility ( $\chi_{\text{ARM}}$ ) was calculated by normalizing the ARM to the bias field. Isothermal remanent magnetization (IRM) was imparted in a 1 T direct field using an impulse magnetizer. The ARM/IRM ratio ( $\chi_{\text{ARM}}/\text{IRM}$ ), computed by normalizing the ARM susceptibility by the IRM, is used as a magnetic grain size indicator, with higher values indicating finer bulk magnetic grain size.

Additional discrete (<500 mg) specimens from the speleothem were used for low temperature magnetic measurements to determine magnetic mineralogy. Low temperature saturation IRM was imparted in a 2.5 T field at 10 K following two separate treatments: field cooling in a 2.5 T field and zero-field cooling, from 300 to 10 K. Remanence behavior was then measured in zero field at 5 K intervals upon warming to room temperature. Finally, the behavior of a room temperature saturation IRM imparted in a 2.5 T field was measured in zero field at 5 K intervals while cooling the sample down to 10 K and then warming it back to 300 K.

## REFERENCES CITED

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Table DR1.  $^{230}\text{Th}$  dating results. The error is  $2\sigma$

| Sample depth (mm) | $^{238}\text{U}$<br>(ppb) | $^{232}\text{Th}$<br>(ppt) | $^{230}\text{Th} / ^{232}\text{Th}$<br>(atomic $\times 10^{-6}$ ) | $\delta^{234}\text{U}^*$<br>(measured) | $^{230}\text{Th} / ^{238}\text{U}$<br>(activity) | $^{230}\text{Th}$ Age (yr)<br>(uncorrected) | $^{230}\text{Th}$ Age (yr)<br>(corrected) | $\delta^{234}\text{U}_{\text{initial}}^\dagger$<br>(corrected) | $^{230}\text{Th}$ Age (yr BP) <sup>§</sup><br>(corrected) |
|-------------------|---------------------------|----------------------------|---|--|--|---|---|--|---|
| 1.90 $\pm 0.30$   | 790 $\pm 1$               | 18977 $\pm 380$            | 612 $\pm 12$  | 3507.5 $\pm 3.3$                       | 0.8925 $\pm 0.0015$                              | 23399 $\pm 48$                              | 23252 $\pm 115$                           | 3745 $\pm 4$   | 23189 $\pm 115$   |
| 13.45 $\pm 0.25$  | 688 $\pm 1$               | 4537 $\pm 91$              | 2921 $\pm 59$   | 3079.8 $\pm 3.5$                       | 1.1679 $\pm 0.0022$                              | 35237 $\pm 82$                              | 35193 $\pm 87$                            | 3401 $\pm 4$   | 35130 $\pm 87$  |
| 23.00 $\pm 0.50$  | 778 $\pm 1$               | 941 $\pm 19$               | 16874 $\pm 342$   | 2935.3 $\pm 4.3$                       | 1.2375 $\pm 0.0030$                              | 39250 $\pm 121$                             | 39242 $\pm 121$                           | 3279 $\pm 5$   | 39179 $\pm 121$   |
| 30.95 $\pm 0.35$  | 1003 $\pm 2$              | 835 $\pm 17$               | 24854 $\pm 504$   | 2835.8 $\pm 3.9$                       | 1.2548 $\pm 0.0032$                              | 41102 $\pm 132$                             | 41096 $\pm 132$                           | 3184 $\pm 5$   | 41033 $\pm 132$   |
| 37.80 $\pm 0.30$  | 828 $\pm 1$               | 489 $\pm 10$               | 35313 $\pm 722$   | 2780.2 $\pm 3.3$                       | 1.2656 $\pm 0.0024$                              | 42234 $\pm 101$                             | 42230 $\pm 101$                           | 3132 $\pm 4$   | 42167 $\pm 101$   |
| 49.40 $\pm 0.30$  | 772 $\pm 1$               | 520 $\pm 11$               | 31206 $\pm 639$   | 2641.8 $\pm 2.8$                       | 1.2757 $\pm 0.0024$                              | 44560 $\pm 106$                             | 44556 $\pm 106$                           | 2996 $\pm 3$   | 44493 $\pm 106$   |
| 82.10 $\pm 0.40$  | 580 $\pm 1$               | 1512 $\pm 30$              | 8627 $\pm 174$  | 2451.1 $\pm 2.8$                       | 1.3647 $\pm 0.0022$                              | 51538 $\pm 114$                             | 51518 $\pm 114$                           | 2835 $\pm 3$   | 51455 $\pm 114$   |
| 116.45 $\pm 0.35$ | 648 $\pm 1$               | 5408 $\pm 108$             | 2863 $\pm 58$   | 2441.3 $\pm 3.5$                       | 1.4493 $\pm 0.0032$                              | 55650 $\pm 167$                             | 55587 $\pm 173$                           | 2856 $\pm 4$   | 55524 $\pm 173$   |
| 150.90 $\pm 0.50$ | 550 $\pm 1$               | 36857 $\pm 739$            | 365 $\pm 7$   | 2308.2 $\pm 3.3$                       | 1.4856 $\pm 0.0032$                              | 60317 $\pm 181$                             | 59793 $\pm 412$                           | 2732 $\pm 5$   | 59730 $\pm 412$   |
| 185.80 $\pm 0.80$ | 449 $\pm 1$               | 10149 $\pm 203$            | 1157 $\pm 23$   | 2335.8 $\pm 2.9$                       | 1.5854 $\pm 0.0031$                              | 64768 $\pm 177$                             | 64595 $\pm 215$                           | 2803 $\pm 4$   | 64532 $\pm 215$   |

U decay constants:  $\lambda_{238} = 1.55125 \times 10^{-10}$  (Jaffey et al., 1971) and  $\lambda_{234} = 2.82206 \times 10^{-6}$  (Cheng et al., 2013). Th decay constant:  $\lambda_{230} = 9.1705 \times 10^{-6}$  (Cheng et al., 2013).

\* $\delta^{234}\text{U} = ([^{234}\text{U}/^{238}\text{U}]_{\text{activity}} - 1) \times 1000$ .

<sup>†</sup> $\delta^{234}\text{U}_{\text{initial}}$  was calculated based on  $^{230}\text{Th}$  age (T), i.e.,  $\delta^{234}\text{U}_{\text{initial}} = \delta^{234}\text{U}_{\text{measured}} \times e^{\lambda_{234} \times T}$ .

Corrected  $^{230}\text{Th}$  ages assume the initial  $^{230}\text{Th}/^{232}\text{Th}$  atomic ratio of  $4.4 \pm 2.2 \times 10^{-6}$ . Those are the values for a material at secular equilibrium, with the bulk earth  $^{232}\text{Th}/^{238}\text{U}$  value of 3.8. The errors are arbitrarily assumed to be 50%.

<sup>§</sup>B.P. stands for "Before Present" where the "Present" is defined as the year 1950 A.D.

Table DR2. Paleomagnetic and rock magnetic time series

| Age<br>(yr BP) | NRM<br>(Am <sup>2</sup> /kg) | ARM<br>(Am <sup>2</sup> /kg) | IRM<br>(Am <sup>2</sup> /kg) | NRM/ARM   | X <sub>ARM</sub> /IRM<br>(mm/A) | Declination<br>(degrees) | Inclination<br>(degrees) | VGP<br>Latitude<br>(degrees) | VGP<br>Latitude<br>error<br>(degrees) |
|----------------|------------------------------|------------------------------|------------------------------|-----------|---------------------------------|--------------------------|--------------------------|------------------------------|---------------------------------------|
| 23292          | 5.33E-07                     | 2.56E-06                     | 6.25E-05                     | 0.207922  | 0.515064                        | 28.26                    | 52.67                    | 66.6                         | 10.7                                  |
| 27944.6        | 4.75E-08                     | 8.34E-08                     | 1.29E-06                     | 0.569124  | 0.81343                         | 19.49                    | 49.38                    | 71.3                         | 9.3                                   |
| 33114.1        | 1.42E-07                     | 2.70E-07                     | 6.42E-06                     | 0.524644  | 0.529256                        | 30.46                    | 53.68                    | 65.2                         | 10.7                                  |
| 36423.2        | 8.23E-08                     | 1.69E-07                     | 2.82E-06                     | 0.487076  | 0.75422                         | -15.56                   | 46.81                    | 72                           | 7.9                                   |
| 38542.9        | 8.45E-08                     | 9.81E-08                     | 2.35E-06                     | 0.862019  | 0.524338                        | 34.17                    | 65.11                    | 63.2                         | 9.1                                   |
| 39995          | 1.13E-08                     | 3.68E-08                     | 5.01E-07                     | 0.306445  | 0.922701                        | 84.89                    | 30.47                    | 14                           | 10.6                                  |
| 41103.1        | 3.73E-09                     | 4.71E-08                     | 4.73E-07                     | 0.0791692 | 1.25149                         | 133.79                   | 11.32                    | -28.3                        | 8.8                                   |
| 41910.1        | 5.23E-09                     | 2.96E-08                     | 4.21E-07                     | 0.176279  | 0.884015                        | 28.23                    | 9.82                     | 47.3                         | 7.4                                   |
| 42858.4        | 2.79E-08                     | 4.47E-08                     | 5.58E-07                     | 0.62411   | 1.00707                         | 12.99                    | 56.03                    | 78                           | 8.8                                   |
| 43845.9        | 1.66E-08                     | 3.85E-08                     | 5.42E-07                     | 0.430773  | 0.892482                        | -4.87                    | 57.31                    | 80.8                         | 6.2                                   |
| 44843.8        | 2.33E-08                     | 4.75E-08                     | 8.09E-07                     | 0.490018  | 0.737676                        | 67.55                    | 66.02                    | 41.3                         | 8.8                                   |
| 45897.7        | 5.00E-08                     | 6.30E-08                     | 9.53E-07                     | 0.793532  | 0.831496                        | 10.50                    | 75.71                    | 62.9                         | 2.1                                   |
| 46941          | 3.51E-08                     | 6.10E-08                     | 1.64E-06                     | 0.576282  | 0.465807                        | 33.40                    | 29.22                    | 52.5                         | 9.2                                   |
| 47973.6        | 4.29E-08                     | 5.99E-08                     | 1.10E-06                     | 0.715997  | 0.682593                        | 4.97                     | 53.26                    | 79.3                         | 5.5                                   |
| 49016.9        | 3.09E-08                     | 6.95E-08                     | 1.09E-06                     | 0.444806  | 0.802337                        | 4.76                     | 43.75                    | 73.6                         | 4                                     |
| 50070.8        | 3.19E-08                     | 5.24E-08                     | 8.62E-07                     | 0.608729  | 0.763337                        | 19.49                    | 46.98                    | 70                           | 8.9                                   |
| 51124.8        | 3.62E-08                     | 1.07E-07                     | 1.37E-06                     | 0.33816   | 0.980018                        | -18.46                   | 53.65                    | 74                           | 9.9                                   |
| 51833.8        | 2.75E-08                     | 4.14E-08                     | 8.18E-07                     | 0.664378  | 0.635575                        | 13.16                    | 74.37                    | 64.4                         | 2.9                                   |
| 52402.5        | 4.26E-08                     | 6.41E-08                     | 7.80E-07                     | 0.664964  | 1.03262                         | 17.99                    | 69.03                    | 69.4                         | 5.6                                   |
| 52988.8        | 1.59E-07                     | 2.20E-07                     | 4.79E-06                     | 0.722937  | 0.576672                        | 0.00                     | 47.33                    | 76.2                         | 3.7                                   |
| 53584.1        | 2.03E-07                     | 3.06E-07                     | 8.19E-06                     | 0.66483   | 0.469031                        | -1.36                    | 50.51                    | 78.2                         | 4.3                                   |
| 54170.5        | 7.73E-08                     | 1.60E-07                     | 2.63E-06                     | 0.483591  | 0.762092                        | 9.07                     | 56.13                    | 79.5                         | 7.4                                   |
| 54742.1        | 1.53E-07                     | 3.63E-07                     | 6.29E-06                     | 0.421753  | 0.726309                        | 35.50                    | 56.02                    | 61.9                         | 10.7                                  |
| 55322.5        | 1.52E-07                     | 2.27E-07                     | 5.05E-06                     | 0.668087  | 0.56533                         | 11.71                    | 36.99                    | 67.8                         | 5.5                                   |
| 55908.5        | 1.54E-07                     | 2.01E-07                     | 6.96E-06                     | 0.766462  | 0.361779                        | -13.45                   | 31.26                    | 64                           | 5.5                                   |
| 56494.6        | 2.60E-07                     | 4.25E-07                     | 1.76E-05                     | 0.6121    | 0.30387                         | -12.93                   | 35.84                    | 66.7                         | 5.7                                   |
| 57099          | 4.21E-07                     | 7.48E-07                     | 3.20E-05                     | 0.563701  | 0.293534                        | -11.50                   | 52.44                    | 77                           | 7.6                                   |
| 57691.2        | 8.23E-07                     | 2.43E-06                     | 0.00012813                   | 0.338623  | 0.238231                        | -2.55                    | 57.84                    | 81                           | 5.4                                   |
| 58283.3        | 7.06E-07                     | 1.98E-06                     | 0.00012026                   | 0.35683   | 0.206741                        | 11.93                    | 55.31                    | 78.2                         | 8.3                                   |
| 58878.6        | 3.56E-07                     | 1.19E-06                     | 5.83E-05                     | 0.298329  | 0.257438                        | -9.26                    | 65.56                    | 75.4                         | 4.8                                   |
| 59437.1        | 3.13E-07                     | 1.24E-06                     | 6.88E-05                     | 0.252824  | 0.225952                        | 12.31                    | 33.21                    | 65.4                         | 5.3                                   |
| 60057          | 6.65E-07                     | 2.36E-06                     | 0.00013179                   | 0.28189   | 0.225067                        | -16.21                   | 45.98                    | 71.2                         | 7.9                                   |
| 60769          | 6.73E-07                     | 1.02E-06                     | 5.44E-05                     | 0.65775   | 0.23625                         | 34.71                    | 58.18                    | 63                           | 10.5                                  |
| 61477.7        | 4.66E-07                     | 1.20E-06                     | 6.84E-05                     | 0.388535  | 0.220435                        | 24.26                    | 41.07                    | 64                           | 9.1                                   |
| 62172.5        | 2.96E-07                     | 6.18E-07                     | 2.78E-05                     | 0.478151  | 0.279665                        | 0.44                     | 38.51                    | 70.7                         | 2.9                                   |
| 62888.1        | 3.04E-07                     | 7.64E-07                     | 3.37E-05                     | 0.398166  | 0.284751                        | -10.61                   | 39.09                    | 69.4                         | 5.4                                   |
| 63596.7        | 3.51E-07                     | 1.03E-06                     | 4.18E-05                     | 0.339652  | 0.311065                        | 12.76                    | 47.12                    | 73.4                         | 7                                     |
| 64284.7        | 1.64E-07                     | 9.09E-07                     | 3.12E-05                     | 0.180789  | 0.366401                        | 5.28                     | 58.74                    | 80.5                         | 5.9                                   |

Table DR3. Incremental chronology

| Depth (mm) | Age (yrs BP) | Growth rate (mm/kyr) | Depth (mm) | Age (yrs BP) | Growth rate (mm/kyr) |
|------------|--------------|----------------------|------------|--------------|----------------------|
| 22.500     | 39192        | 4.24                 | 29.168     | 40770        | 6.54                 |
| 22.623     | 39221        | 4.24                 | 29.522     | 40812        | 8.41                 |
| 22.777     | 39266        | 3.41                 | 29.772     | 40849        | 6.78                 |
| 23.187     | 39392        | 3.25                 | 29.859     | 40863        | 6.21                 |
| 23.316     | 39434        | 3.07                 | 30.312     | 40917        | 8.38                 |
| 23.592     | 39551        | 2.37                 | 30.410     | 40928        | 8.91                 |
| 23.623     | 39564        | 2.37                 | 30.724     | 40964        | 8.72                 |
| 24.053     | 39742        | 2.42                 | 30.860     | 41001        | 3.68                 |
| 24.178     | 39763        | 5.95                 | 30.952     | 41014        | 7.10                 |
| 24.368     | 39813        | 3.80                 | 31.149     | 41040        | 7.56                 |
| 24.436     | 39833        | 3.37                 | 31.423     | 41076        | 7.63                 |
| 24.475     | 39847        | 2.80                 | 31.448     | 41081        | 5.00                 |
| 24.542     | 39870        | 2.91                 | 31.669     | 41116        | 6.32                 |
| 24.624     | 39894        | 3.40                 | 31.771     | 41131        | 6.78                 |
| 24.690     | 39909        | 4.43                 | 32.016     | 41166        | 7.00                 |
| 24.725     | 39917        | 4.41                 | 32.282     | 41209        | 6.19                 |
| 24.976     | 39977        | 4.18                 | 32.368     | 41223        | 6.16                 |
| 25.188     | 40017        | 5.29                 | 32.578     | 41258        | 5.99                 |
| 25.366     | 40048        | 5.76                 | 32.648     | 41270        | 5.85                 |
| 25.408     | 40057        | 4.67                 | 32.740     | 41287        | 5.37                 |
| 25.654     | 40109        | 4.73                 | 32.922     | 41318        | 5.87                 |
| 25.858     | 40156        | 4.34                 | 33.086     | 41348        | 5.48                 |
| 26.116     | 40214        | 4.44                 | 33.460     | 41408        | 6.23                 |
| 26.203     | 40229        | 5.82                 | 33.767     | 41452        | 6.98                 |
| 26.267     | 40242        | 4.96                 | 33.963     | 41485        | 5.95                 |
| 26.380     | 40261        | 5.91                 | 34.192     | 41522        | 6.18                 |
| 26.503     | 40281        | 6.18                 | 34.359     | 41547        | 6.66                 |
| 26.762     | 40331        | 5.18                 | 34.610     | 41592        | 5.59                 |
| 26.977     | 40373        | 5.11                 | 34.688     | 41608        | 4.84                 |
| 27.201     | 40417        | 5.09                 | 34.760     | 41624        | 4.52                 |
| 27.270     | 40432        | 4.62                 | 34.793     | 41631        | 4.75                 |
| 27.492     | 40466        | 6.51                 | 34.898     | 41652        | 4.96                 |
| 27.563     | 40482        | 4.45                 | 34.977     | 41668        | 4.94                 |
| 27.801     | 40523        | 5.80                 | 35.281     | 41716        | 6.34                 |
| 27.896     | 40552        | 3.29                 | 35.503     | 41747        | 7.15                 |
| 27.986     | 40569        | 5.26                 | 35.607     | 41767        | 5.22                 |
| 28.198     | 40611        | 5.05                 | 36.266     | 41869        | 6.46                 |
| 28.312     | 40630        | 6.00                 | 36.410     | 41889        | 7.16                 |
| 28.442     | 40646        | 8.16                 | 36.478     | 41900        | 6.20                 |
| 28.614     | 40677        | 5.52                 | 36.573     | 41917        | 5.62                 |
| 28.708     | 40694        | 5.54                 | 37.191     | 42013        | 6.43                 |
| 28.788     | 40708        | 5.70                 | 37.623     | 42084        | 6.08                 |
| 28.902     | 40728        | 5.71                 | 37.766     | 42111        | 5.30                 |
| 29.083     | 40757        | 6.26                 | 38.100     | 42167        | 5.97                 |

Fig. DR1. Confocal micrograph showing fluorescent annual layering in the studied speleothem.

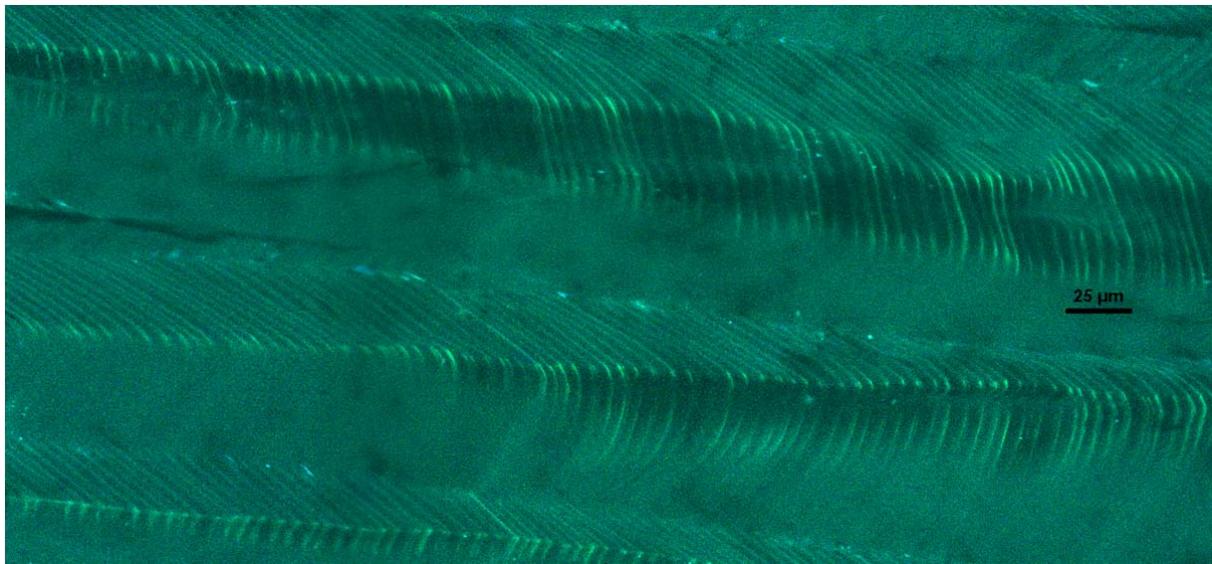


Fig. DR2. Orthogonal projection and azimuthal equidistant plot of the specimen from 62 ka BP. Full (open) squares represent the horizontal (vertical) projections of the demagnetization vectors (grey arrows). Magnetometer noise level is  $\sim 10^{-11}$  Am $^2$ .

