**LUMINESCENCE ANALYSIS OF CERAMICS FROM THAILAND**

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This report presents the results of luminescence analysis of three ceramic samples and a clay “bullet” from two cave sites in Mae Hong Son Province, northwest Thailand. The samples were submitted by Cyler Conrad, University of New Mexico. Provenience data on the ceramics are given in Table 1. Laboratory procedures are given in the appendix. Recently obtained radiocarbon dates are given in Table 2. Oxford University also obtained TL dates on ceramics from Banyan Valley Cave in 1973. These ranged in age from 500 to 900 BC for layer 1 and 2000 BC for layer 2

Table 1. Sample information

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| UW Lab # | Code | Catalog # | Site (English) | material | Unit/Layer | Burial depth  (cm) |
| UW3678 | TL-1 | BVC-C10.1 | Tham Boon Hoong (Banyan Valley Cave) | Clay “bullet” | D5/2 | 10-20 |
| UW3679 | TL-2 | BVC-C20.1 | Tham Boon Hoong (Banyan Valley Cave) | sherd | F5/3 | 40-76 |
| UW3680 | TL-3 | TPC-C18 | Tham Phaa Can (Steep Cliff Cave) | sherd | E3/2 | 10-20 |
| UW3681 | TL-4 | TPC-C20 | Tham Phaa Can (Steep Cliff Cave) | sherd | E2/4 | 40-80 |

Table 2. Radiocarbon dates

|  |  |  |
| --- | --- | --- |
| Site | layer | 14C uncalibrated (years BP) |
| Banyon Valley | 2 | 2850 ± 25 |
| 2 | 3970 ± 25 |
| 3 | 4060 ± 25 |
| 3 | 6180 ± 30\* |
| Steep Cliff | 2 | 7460 ± 30 |
| 2 | 8600 ± 30 |
| 2 | 8180 ± 30 |
| 4 | 8100 ± 30 |
| 4 | 9140 ± 30 |
| 4 | 9960 ± 30 |

\* Provenience same as UW3679

**Dose rate**

The dose rate was measured on each ceramic as described in the appendix, but no associated sediment samples were available to determine the external gamma dose rate. Sediment dose rate information was available from a nearby site, Tham Lod rockshelter. Quartz sediments from this site had concentrations of about 3 ppm U, 17 ppm Th, and 1.1% K. These were used as an estimate for the external dose rate. Using these values, the proportion of the dose rate arising from gamma radiation (which mostly arises from the sediment) was about 15% for UW3679 and UW3681, so some error in the external rate is tolerable. For UW3678 and UW3680, however, the proportion of gamma radiation was about 35%. Here errors in the external dose rate will be more significant. The difference between these two sets is related to the much higher internal dose rate for UW3679 and UW3681.

Concentrations of radionuclides are given in Table 3. Dose rates on the ceramics were mainly determined using alpha counting and flame photometry. The beta dose rate calculated from these measurements was compared with the beta dose rate measured directly by beta counting. Table 3 compares the beta dose rate calculated in these two ways. These do not differ significantly for any sample. Because equivalent dose was measured on coarse-grain potassium feldspars for UW3678, an estimate of internal K content for each grain is required. Based on the moderate sensitivity of the sample, an internal content of 8 ± 3 % was estimated.

Moisture content was estimated as 80±20 % of the saturated value for the ceramics and 15 ± 5% for the sediments at Banyan Valley Cave, reflecting the wet conditions of the cave. For the dry Steep Cliff Cave, the estimate was 60 ± 20 % for the ceramics and 6 ± 2% for the sediments. Cosmic dose rate was determined from Prescott and Hutton (1994), using the latitude, longitude and altitude of the sites and burial depths of the sherds. For the cave settings, adjustment for the cave opening, distance back from the dripline, and over-burden was estimated by dividing the calculated cosmic dose rate by 3. The cosmic dose rate makes up a small portion of the total dose rate, so obtaining further precision is not critical. Table 4 gives the total dose rates.

Table 3. Radionuclide concentrations

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *Sample* | *238U*  *(ppm)* | *233Th*  *(ppm)* | *K*  *(%)* | *Beta dose rate (Gy/ka)* | |
| ß-counting | α-counting/flame photometry |
| UW3678 | 1.75±0.18 | 13.24±1.21 | 1.84±0.04 | 2.69±0.22 | 2.74±0.06 |
| UW3679 | 12.84±0.88 | 46.97±3.68 | 4.37±0.14 | 6.52±0.53 | 6.76±0.20 |
| UW3680 | 2.46±0.22 | 13.83±1.57 | 1.16±0.05 | 1.75±0.16 | 1.69±0.07 |
| UW3681 | 13.81±1.11 | 37.76±3.40 | 3.80±0.16 | 6.52±0.59 | 6.91±0.23 |

Table 4. Dose rates (Gy/ka)\*

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| *Sample* | *alpha* | *beta* | *gamma* | *cosmic* | *total* |
| UW3678 | 0.16±0.07 | 2.10±0.18 | 1.18±0.10 | 0.08±0.02 | 3.51±0.22 |
| UW3679 | 1.76±0.09 | 5.99±0.25 | 1.57±0.11 | 0.06±0.01 | 9.38±0.28 |
| UW3680 | 0.64±0.05 | 1.52±0.08 | 1.29±0.09 | 0.07±0.02 | 3.53±0.13 |
| UW3681 | 4.62±0.22 | 6.29±0.28 | 1.68±0.09 | 0.07±0.01 | 12.66±0.36 |

\* Dose rates for sherds are calculated for OSL. Dose rates will usually be higher for TL and IRSL due to higher b-values. Also the beta dose rate is lower than that given in Table 2 due to moisture correction. Dose rate for UW3678 is for coarse-grained potassium feldspars.

**Equivalent Dose for sherds**

Equivalent dose on the sherds was measured for TL, OSL and IRSL as described in the appendix. The TL plateaus were broad, 80°C or more in breadth (Table 5). There was no sensitivity change between first and second glowouts. TL anomalous fading was only evident for UW3681, but the measured g-value was too high to make a finite correction.

Table 5. TL parameters

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| *Sample* | *Plateau (°C)* | *1st/2nd ratio\** | *fit* | *Fading g-value\*\** |
| UW3679 | 250-360 | 1.0 | linear | none |
| UW3680 | 270-360 | 1.0 | quadratic | none |
| UW3681 | 260-340 | 1.0 | linear | 15.5±1.48 |

\*Refers to slope ratio between the first and second glow growth curves. A glow refers to luminescence as a function of temperature; a second, or regeneration glow comes after heating to 450°C.

\*\* A g-value is a rate of anomalous fading, measured as percent of signal loss per decade, where a decade is a power of 10. Any value over about 14 results in an infinite age correction, implying the fading rate has probably changed through time.

OSL/IRSL was measured on 6 aliquots per sample (Table 6), but some aliquots were rejected because of poor data. Scatter, as measured by over-dispersion, was less than 12% for all samples, although there was one outlier removed from UW3680. An IRSL signal could be measured on all samples, and it was only 2-3 times less intense than the OSL signal. IRSL stems from feldspars, which are prone to anomalous fading. A relatively strong IRSL signal may suggest the OSL signal partly stems from feldspars and therefore may fade, while a weak IRSL suggests the OSL is dominated by quartz. IRSL is generally weaker than OSL in ceramics because heat reduces the IRSL intensity while increasing the OSL intensity. The IRSL signal was relatively strong on these samples, however. Another measure of feldspar contribution is the size of the OSL b-value. The b-value is a measure of alpha luminescence efficiency, and is usually less than 0.7 for quartz and a higher value for feldspar. For these samples the OSL b-value was in the range of quartz for UW3679 and UW3680, but was higher for UW3681. It is possible the OSL signal in this sample partly stems from feldspars and thus the signal may fade some.

As a test of the SAR procedures, a dose recovery test was performed. The recovered dose was within 2-sigma of the administered dose for all samples. Equivalent dose and b-values are given in Table 7.

Table 6. OSL/IRSL data

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sample | # aliquots\* | | OSL Over-dispersion (%) | Dose Recovery (OSL) | |
| OSL | IRSL |  | Given  Dose (sß) | Recovered  Dose (sß) |
| UW3679 | 6 | 6 | 0 | 100 | 104±4.9 |
| UW3680 | 4 | 6 | 11.3±5.6 | 100 | 102±5.9 |
| UW3681 | 6 | 6 | 5.6±2.3 | 100 | 93.0±4.1 |

\* Denotes number of aliquots with measurable signals.

Table 7. Equivalent dose and b-value – fine grains

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| *Sample* | *Equivalent Dose (Gy)* | | | *b-value (Gy µm2)* | | |
| *TL* | *IRSL* | *OSL* | *TL* | *IRSL* | *OSL* |
| UW3679 | 54.1±3.85 | 30.7±1.07 | 40.9±1.36 | 1.01±0.57 | 1.26±0.03 | 0.45±0.01 |
| UW3680 | 23.6±2.70 | 22.6±3.05 | 30.1±2.01 | 0.39±0.89 | 1.07±0.14 | 0.66±0.04 |
| UW3681 | 28.7±1.43 | 16.2±0.74 | 15.1±0.41 | 2.25±0.47 | 1.45±0.03 | 1.02±0.02 |

**Ages for sherds**

Table 8 gives the derived ages for each sample. For both UW3679 and UW3680, the age from OSL statistically agrees with the age from TL. The TL signal showed no fading, but the IRSL age from both samples was younger, indicating some fading with that signal. The OSL and IRSL ages for UW3681 are in statistical agreement but the TL age is slightly older. The TL signal showed fading, but the measured rate was too high to yield a finite correction. The OSL b-value was high, so it is possible both the OSL and IRSL signals fade to some degree. An older age for this sample is therefore possible. Even with the possibility of somewhat older age, this sample is still much younger than any of the others.

Table 8. Ages of sherds

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Sample | Age (ka)\* | % error | Basis for age | Calendar date (years BC/AD) |
| UW3679 | 4.37±0.23 | 5.3 | OSL/TL | BC 2350 ± 230 |
| UW3680 | 8.40±0.67 | 8.0 | OSL/TL | BC 6390 ± 670 |
| UW3681 | 1.16±0.05 | 3.9 | OSL/IRSL | AD 860 ± 50 |
| 1.57±0.22 | 14.1 | TL | AD 450 ± 220 |

\* The base year for ka is 2018.

\*\* High external dose rate refers to use of the ceramic radioactivity for the external dose rate. Low external dose rate uses sediment radioactivity based on 0.5% K, 6 ppm Th and 2 ppm U.

**Equivalent dose and age for clay bullet**

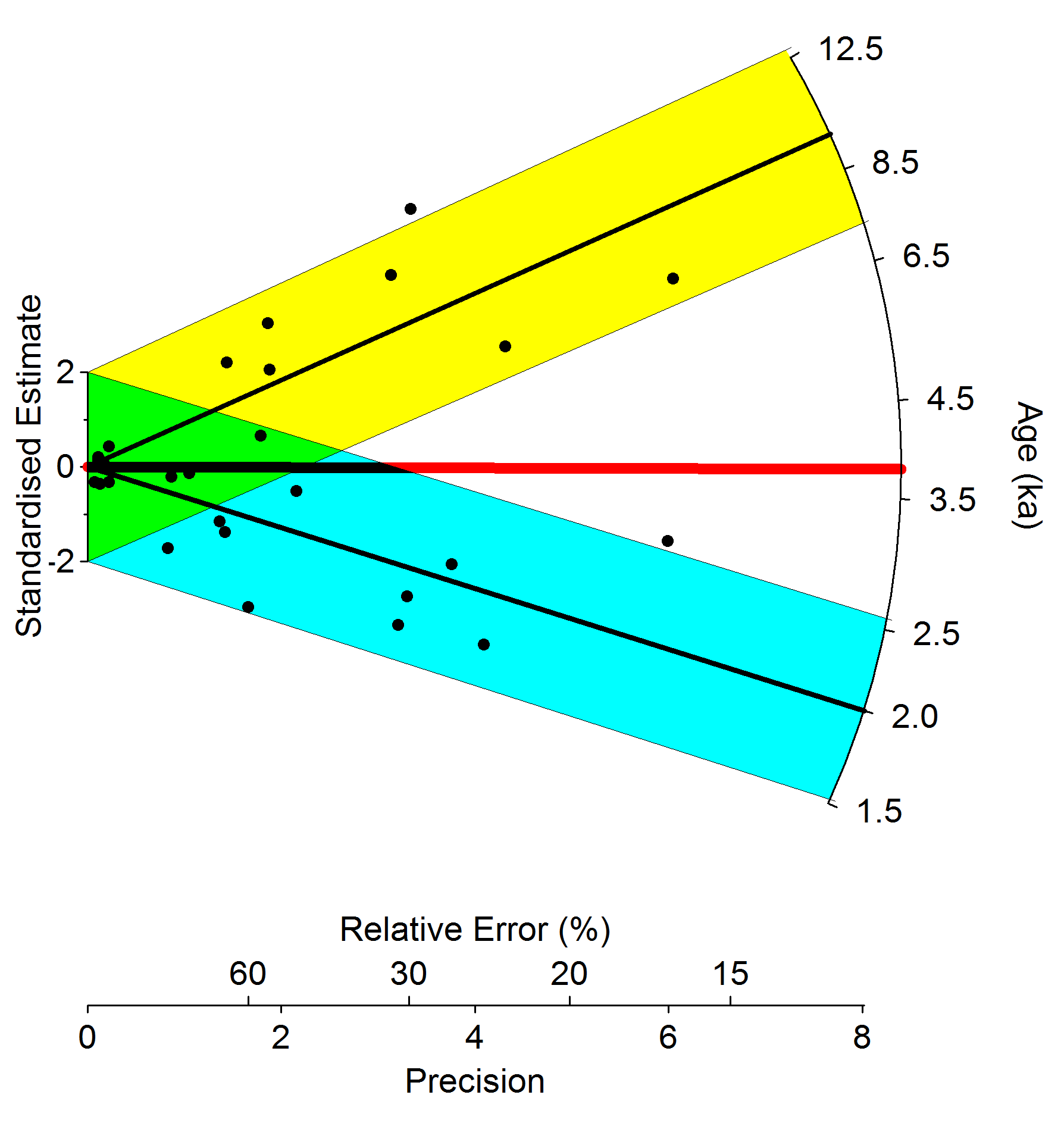
Luminescence was measure on coarse 180-212µm grains for this sample. Quartz was not abundant in this sample. After a density separation, the material less than 2.67 specific gravity was mostly consumed by an HF etch, suggesting most of this material was feldspar. Infrared stimulated luminescence (IRSL) was measured on 180-212µm single-grains of potassium feldspar. Details are in the appendix.

Equivalent dose was measured on 588 grains, of which 37 yielded acceptable values (6.3%) using rejection criteria outlined in the appendix. Most of the rejections were because of poor signal. Additional measurements were not made because of lack of material.

The equivalent dose from the central age model was 11.0 ± 2.03 Gy, with an over-dispersion of 104.1 ± 13.8%. A fading test following Auclair et al. (2003) was administered to each grain. The average fading rate (g-value) was 4.3 ± 2.3%/decade (where a decade is a power of 10), although with high scatter. On 8 grains the fading rate was too high for a finite correction. For the other 29, the average corrected age, from the central age model, was 3.84 ± 0.89 ka, with an over-dispersion of 91± 18%. Because of the high over-dispersion, a finite mixture model was applied using 10% over-dispersion as typical of the single-aged sample. Using 20% over-dispersion produced no significant differences. The sample divided fairly evenly into two components. An older component, accounting for 42% of the grains, gave an age of 9.50 ± 1.14 ka, while a younger component, accounting for 58% of the grains, gave an age of 1.99 ± 0.22 ka. No grains corresponded with the age from the central age model, which shows that the central model age is a simple artifact of averaging between two separated groups. A radial graph of the distribution is shown in Figure 1. A radial graph plots precision against a standardized age, with higher precision to the right. Standardization is by the number of standard errors any point is from a reference. The three references are plotted as the ages from the two components of the finite mixture model and the central age model . The yellow and blue shading encompass all values within two standard errors of the older and younger components respectively. The red line represent the central age model. Any line drawn from the origin through any point intersects the right axis at the derived age.

The figure shows a bimodal distribution with only one point lying between the two components. The reason for the division is probably partial bleaching. Whatever procedure by which clay “bullets” are formed, only some of the grains were bleached at the time of formation. The older grains represent the age of the parent material. A minimum age model, used for partially bleached sediments, was also applied and gave an age of 1.57 ± 0.44 ka, in agreement with the younger component.

**Figure 1. Radial graph**



**Conclusion**

Banyan Valley Cave – The age from the clay bullet is about 1000 years younger than one of the radiocarbon dates from the same level. Of course, the two dating methods might not be dating the same event. Whatever material was radiocarbon dated may have found its way into a younger deposit. The other radiocarbon date is another 1000 years older, not lending much credence to the radiocarbon dates addressing the deposition of the layer. On the other hand, the age of the sherd from Layer 3 (UW3679) closely matches one of the radiocarbon dates from that layer. The other radiocarbon date, from the exact same provenience as UW3679, however, is 2000 years older.

Alternatively, the younger age for the clay bullet could be a function of an incorrect external dose rate. I noted earlier that some error could be tolerated for the external dose rate for UW3679, but not for UW3678. One could thus argue that UW3679 is more accurate.

Steep Cliff Cave – The age of UW3680 is in the same ballpark at the radiocarbon ages for the same layer. (A much younger date reported earlier was incorrect.) This agreement is in spite of the significance of the uncertainly in external dose rate for this sample. UW3681, from layer 4, is very young, possibly an intrusion from later disturbance.

**Appendix:**

**Procedures for Thermoluminescence Analysis of Pottery**

*Sample preparation -- fine grain*

The sherd is broken to expose a fresh profile. Material is drilled from the center of the cross-section, more than 2 mm from either surface, using a tungsten carbide drill tip. The material retrieved is ground gently by an agate mortar and pestle, treated with HCl, and then settled in acetone for 2 and 20 minutes to separate the 1-8 µm fraction. This is settled onto a maximum of 72 stainless steel discs.

*Glow-outs*

Thermoluminescence is measured by a Daybreak reader using a 9635Q photomultiplier with a Corning 7-59 blue filter, in N2 atmosphere at 1°C/s to 450°C. A preheat of 240°C with no hold time precedes each measurement. Artificial irradiation is given with a 241Am alpha source and a 90Sr beta source, the latter calibrated against a 137Cs gamma source. Discs are stored at room temperature for at least one week after irradiation before glow out. Data are processed by Daybreak TLApplic software.

*Fading test*

Several discs are used to test for anomalous fading. The natural luminescence is first measured by heating to 450°C. The discs are then given an equal alpha irradiation and stored at room temperature for varied times: 10 min, 2 hours, 1 day, 1 week and 8 weeks. The irradiations are staggered in time so that all of the second glows are performed on the same day. The second glows are normalized by the natural signal and then compared to determine any loss of signal with time (on a log scale). If the sample shows fading and the signal versus time values can be reasonably fit to a logarithmic function, an attempt is made to correct the age following procedures recommended by Huntley and Lamothe (2001). The fading rate is calculated as the g-value, which is given in percent per decade, where decade represents a power of 10.

*Equivalent dose*

The equivalent dose is determined by a combination additive dose and regeneration (Aitken 1985). Additive dose involves administering incremental doses to natural material. A growth curve plotting dose against luminescence can be extrapolated to the dose axis to estimate an equivalent dose, but for pottery this estimate is usually inaccurate because of errors in extrapolation due to nonlinearity. Regeneration involves zeroing natural material by heating to 450°C and then rebuilding a growth curve with incremental doses. The problem here is sensitivity change caused by the heating. By constructing both curves, the regeneration curve can be used to define the extrapolated area and can be corrected for sensitivity change by comparing it with the additive dose curve. This works where the shapes of the curves differ only in scale (i.e., the sensitivity change is independent of dose). The curves are combined using the “Australian slide” method in a program developed by David Huntley of Simon Fraser University (Prescott et al. 1993). The equivalent dose is taken as the horizontal distance between the two curves after a scale adjustment for sensitivity change. Where the growth curves are not linear, they are fit to quadratic functions. Dose increments (usually five) are determined so that the maximum additive dose results in a signal about three times that of the natural and the maximum regeneration dose about five times the natural.

A plateau region is determined by calculating the equivalent dose at temperature increments between 240° and 450°C and determining over which temperature range the values do not differ significantly. This plateau region is compared with a similar one constructed for the b-value (alpha efficiency), and the overlap defines the integrated range for final analysis.

*Alpha effectiveness*

Alpha efficiency is determined by comparing additive dose curves using alpha and beta irradiations. The slide program is also used in this regard, taking the scale factor (which is the ratio of the two slopes) as the b-value (Aitken 1985).

*Radioactivity*

Radioactivity is measured by alpha counting in conjunction with atomic emission for 40K. Samples for alpha counting are crushed in a mill to flour consistency, packed into plexiglass containers with ZnS:Ag screens, and sealed for one month before counting. The pairs technique is used to separate the U and Th decay series. For atomic emission measurements, samples are dissolved in HF and other acids and analyzed by a Jenway flame photometer. K concentrations for each sample are determined by bracketing between standards of known concentration. Conversion to 40K is by natural atomic abundance. Radioactivity is also measured, as a check, by beta counting, using a Risø low level beta GM multicounter system. About 0.5 g of crushed sample is placed on each of four plastic sample holders. All are counted for 24 hours. The average is converted to dose rate following Bøtter-Jensen and Mejdahl (1988) and compared with the beta dose rate calculated from the alpha counting and flame photometer results. Associated sediments were not available for dose rate analysis. Cosmic radiation is determined after Prescott and Hutton (1994). Radioactivity concentrations are translated into dose rates following Guérin et al. (2011).

*Moisture Contents*

Water absorption values for the sherds are determined by comparing the saturated and dried weights. For temperate climates, moisture in the pottery is taken to be 80 ± 20 percent of total absorption, unless otherwise indicated by the archaeologist. Again for temperate climates, soil moisture contents are taken from typical moisture retention quantities for different textured soils (Brady 1974: 196), unless otherwise measured. For drier climates, moisture values are determined in consultation with the archaeologist.

**Procedures for Optically Stimulated or Infrared Stimulated Luminescence of Fine-grained pottery.**

Optically stimulated luminescence (OSL) and infrared stimulated luminescence (IRSL) on fine-grain (1-8µm) pottery samples are carried out on single aliquots following procedures adapted from Banerjee et al. (2001) and Roberts and Wintle (2001. Equivalent dose is determined by the single-aliquot regenerative dose (SAR) method (Murray and Wintle 2000).

The SAR method measures the natural signal and the signal from a series of regeneration doses on a single aliquot. The method uses a small test dose to monitor and correct for sensitivity changes brought about by preheating, irradiation or light stimulation. SAR consists of the following steps: 1) preheat, 2) measurement of natural signal (OSL or IRSL), L(1), 3) test dose, 4) cut heat, 5) measurement of test dose signal, T(1), 6) regeneration dose, 7) preheat, 8) measurement of signal from regeneration, L(2), 9) test dose, 10) cut heat, 11) measurement of test dose signal, T(2), 12) repeat of steps 6 through 11 for various regeneration doses. A growth curve is constructed from the L(i)/T(i) ratios and the equivalent dose is found by interpolation of L(1)/T(1). Usually a zero regeneration dose and a repeated regeneration dose are employed to insure the procedure is working properly. For fine-grained ceramics, a preheat of 240°C for 10s, a test dose of 3.1 Gy, and a cut heat of 200°C are currently being used, although these parameters may be modified from sample to sample.

The luminescence, L(i) and T(i), is measured on a Risø TL-DA-15 automated reader by a succession of two stimulations: first 100 s at 60°C of IRSL (880nm diodes), and then 100s at 125°C of OSL (470nm diodes). Detection is through 7.5mm of Hoya U340 (ultra-violet) filters. The two stimulations are used to construct IRSL and OSL growth curves, so that two estimations of equivalent dose are available. Anomalous fading usually involves feldspars and only feldspars are sensitive to IRSL stimulation. The rationale for the IRSL stimulation is to remove most of the feldspar signal, so that the subsequent OSL (post IR blue) signal is free from anomalous fading. However, feldspar is also sensitive to blue light (470nm), and it is possible that IRSL does not remove all the feldspar signal. Some preliminary tests in our laboratory have suggested that the OSL signal does not suffer from fading, but this may be sample specific. The procedure is still undergoing study.

A dose recovery test is performed by first zeroing the sample by exposure to light and then administering a known dose. The SAR protocol is then applied to see if the known dose can be obtained.

Alpha efficiency will surely differ among IRSL, OSL and TL on fine-grained materials. It does differ between coarse-grained feldspar and quartz (Aitken 1985). Research is currently underway in the laboratory to determine how much b-value varies according to stimulation method. Results from several samples from different geographic locations show that OSL b-value is less variable and centers around 0.5. IRSL b-value is more variable and is higher than that for OSL. TL b-value tends to fall between the OSL and IRSL values. We currently are measuring the b-value for IRSL and OSL by giving an alpha dose to aliquots whose luminescence have been drained by exposure to light. An equivalent dose is determined by SAR using beta irradiation, and the beta/alpha equivalent dose ratio is taken as the b-value. A high OSL b-value is indicative that feldspars might be contributing to the signal and thus subject to anomalous fading.

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***Age and error terms***

The age and error for both OSL and TL are calculated by a laboratory constructed spreadsheet, based on Aitken (1985). All error terms are reported at 1-sigma.

**Laboratory procedures for dating K-feldspar grains**

The target particles for dating were fine sand-sized single-grain (180-212 µm) potassium feldspars. IRSL was used to determine equivalent dose (De).

Dose rate was determined in the same way as for the sherds, except consideration is also given for internal K of the K-feldspar grains. This was estimated based on the sensitivity of the grains, because sensitivity has been shown to correlate with K content. As the sample was only moderately sensitive, a value of 8 ± 3% was used.

To determine equivalent dose, the outer portion (2 mm) was removed and the internal portion ground in a steel mortar and pestle. The >90 µm fraction was treated with HCl and H2O2, and then dry-sieved to isolate the 180-212 µm fraction. The grains from this fraction were density separated using lithium metatungstate set at 2.58 specific gravity. Luminescence measurements were made on the <2.58 fraction. With feldspars, correction for anomalous fading, which is athermal loss of trapped charge through time, is required.

Single-grain dating was employed for all samples. Single-grain measurements were made using Risø TL/OSL DA-20 reader, with an IR single-grain attachment. Stimulation used a 150 mW 830 nm IR laser, set at 30% power and passed through an RG 780 filter. Emission was collected by the photomultiplier through a blue-filter pack, allowing transmission in the 350-450nm range. IRSL measurements were made at 50°C, and a preheat of 250°C for 1 minute at 5°C/s proceeded each measurement. Exposure for single-grains was for 0.8 s, using the first 0.06 s for analysis and the last 0.15 s for background.

Equivalent dose (De) was determined using the single-aliquot regenerative dose (SAR) protocol (Murray and Wintle 2000), and as applied to feldspars by Auclair et al. (2003). The SAR method measures the natural signal and the signal from a series of regeneration doses on a single aliquot. The method uses a small test dose to monitor and correct for sensitivity changes brought about by preheating, irradiation or light stimulation. SAR consists of the following steps: 1) preheat, 2) measurement of natural signal (OSL or IRSL), L(1), 3) test dose, 4) preheat, 5) measurement of test dose signal, T(1), 6) regeneration dose, 7) preheat, 8) measurement of signal from regeneration, L(i), 9) test dose, 10) preheat, 11) measurement of test dose signal, T(i), 12) repeat of steps 6 through 11 for i regeneration doses. A growth curve is constructed from the L(i)/T(i) ratios and the equivalent dose is found by interpolation of L(1)/T(1). A zero regeneration dose and a repeated regeneration dose are employed to insure the procedure is working properly.

Test doses for the SAR were about 5-6 Gy. Doses were delivered by a 90Sr beta source, which provides about 0.11 Gy/s to 180-212 µm grains, and which was calibrated using quartz irradiated by a gamma source at Battelle Laboratory in Hanford, Washington. The dose delivered to different grains in single-grain disks varied by an order of magnitude from one end of the disk to the other. This variation was taken into account when determining doses to individual grains.

An advantage of single-grain dating is the opportunity to remove from analysis grains with unsuitable characteristics by establishing a set of criteria grains must meet. Grains are eliminated from analysis if they (1) had poor signals (as judged from net natural signals not at least three times above the background standard deviation), (2) did not produce, within 20 percent, the same signal ratio (often called recycle ratio) from identical regeneration doses given at the beginning and end of the SAR sequence, suggesting inaccurate sensitivity correction, (3) yielded natural signals that did not intersect saturating growth curves, or (4) had a signal larger than 10 percent of the natural signal after a zero.

A dose recovery test was not performed because of insufficient material.

Anomalous fading was measured using the procedures of Auclair et al. (2003) on single grains. Age was corrected following Huntley and Lamothe (2001). Storage times after irradiation of up to 3-5 days were employed.

A fading-corrected age was obtained for each suitable grain. Because of varying precision and other factors, the same value is not obtained for each grain even if all are of the same true age. Instead a distribution is produced. The common age model and central age model of Galbraith (Galbraith and Roberts 2012) are statistical tools that were used in evaluation of age distributions. The common age model controls for differential precision by computing a weighted average using log values. The central age model is similar except rather than assuming a single true value it assumes a natural distribution of estimated age values, even for true single-aged samples, because of non-statistical sources of variation that are not accounted for in the estimations, such as variation of luminescence properties among grains or heterogeneity in dose rate. It computes an over-dispersion parameter (σb) interpreted as the relative standard deviation (or coefficient of variance) of the true age estimates, or, in other words, that deviation beyond what can be accounted for by measurement error. Empirical evidence suggests that σb of between 10 to 20 percent for single-grains are typical. Over-dispersion will be higher for samples that are not single-aged because of partial bleaching or post-depositional mixing.

For the single-grain age distributions, a finite mixture model was employed for evaluation. This model (Galbraith and Roberts 2012), which is appropriate for samples where post-depositional processes have mixed grains of different depositional age, uses maximum likelihood to separate the grains into single-aged components based on the input of a given σb value and the assumption of a log normal distribution of each component. The model estimates the number of components, the weighted average of each component, and the proportion of grains assigned to each component. The model provides two statistics for estimating the most likely number of components, maximum log likelihood (llik) and Bayes Information Criterion (BIC). The finite mixture model is most useful for samples that have discrete rather than continuous age populations due to mixing. A minimum age model was also employed (Galbraith and Roberts 2012). This is designed to isolate statistically well-bleached grains from a distribution that includes partially bleached grains. The method assumes a truncated normal distribution, where the truncation represents the fully bleached grains. The over-dispersion estimated to represent a single-age sample is added to the error for each grain in quadrature.

Ages are determined using a laboratory spread sheet based on Aitken (1985). Ages are quoted with 1-sigma errors and using 2019 as the reference for before present designations.

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