SUPPORTING MATERIAL:

LUMINESCENCE ANALYSIS OF CERAMICS

Five sherds were collected from red earth and gravelly loess layers on several terraces Table S1 lists the samples.

Table S1. Samples

UW Lab #	Sample designation
UW1725	07JZGASN:28.1
UW1726	07JZGASN:37
UW1727	07JZGASN:16.1
UW1728	07JZGASN:17.2
UW1729	07JZGASN:20.5

Sample preparation

Luminescence analysis was done on fine-grained material. The sherds were broken to expose a fresh profile and material drilled from the center of the cross-section using a tungsten carbide drill tip. The drilled material was taken more than 2 mm from any surface and used for luminescence measurements. It was ground gently in an agate mortar and pestle, treated with HCl and settled in acetone for 2 and 20 minutes to isolate the 1-8 μm fraction. This was settled as an approximate mono-layer onto a maximum of 72 stainless steel disks for measurement. The outer 2 mm material was ground to a flour consistency and used for dose rate measurements.

Dose rate

The dose rate was measured on each ceramic and an associated sediment sample, which was also ground to flour consistency. No sediment sample was available for UW1726, so an average of the other four sediments were used for that sample.

Dose rate was measured by alpha counting, beta counting and atomic emission for K. Samples for alpha counting were packed into plexiglass containers with ZnS:Ag screens, and sealed for one month before counting. The pairs technique was used to separate the U and Th decay series. For beta counting, about 0 .5 g of crushed sample was placed on each of four plastic sample holders and counted for 24 hours, using a Risø low level beta GM multicounter system. The average was converted to beta dose rate following Bøtter-Jensen and Mejdahl (1988) For atomic emission measurements, samples were dissolved in HF and other acids and analyzed by a Jenway flame photometer. K concentrations for each sample were determined by bracketing between standards of known concentration. Conversion to ^{40}K was by natural atomic abundance. As a check for consistency and as a possible indicator of deviation from secular equilibrium in the decay chains, the beta dose rate from beta counting was compared to that calculated from alpha counting and flame photometer results. Cosmic radiation was determined after Prescott and Hutton (1994). Radioactivity concentrations were translated into dose rates following

Guérin et al. (2011). For moisture contents, water absorption values for the sherds were determined by comparing the saturated and dried weights. Given the temperate climate, moisture was was taken to be 80 ± 20 percent of total absorption. Moisture content for the sediments was estimated to be 15 ± 5 percent. Average burial depth over time was difficult to estimate. Presumably the sherds were on or near the surface for some time before being covered by a land slide of unknown age. Average burial depth was estimated 20 ± 10 cm, recognizing that anything over 30 cm makes little difference for the gamma dose rate.

Relevant dose rate concentrations are given in Table S2. The table also compares the beta dose rate calculated in the two ways mentioned. There were no significant differences for any sherds. Table S3 gives total dose rates for each sample. Dose rates are similar for all the ceramics.

Table S2. Radionuclide concentrations

Sample	*^{238}U (ppm)*	*^{233}Th (ppm)*	*K (%)*	*Beta dose rate (Gy/ka)*	
				ß-counting	α-counting/flame photometry
UW1725	2.95±0.24	13.88±1.49	1.88±0.17	2.12±0.18	2.32±0.14
Sediment	2.71±0.22	11.12±1.41	1.75±0.15		
UW1726	4.43±0.29	10.51±1.31	1.95±0.05	2.53±0.22	2.50±0.07
UW1727	5.22±0.35	14.36±1.62	2.46±0.26	2.91±0.24	3.12±0.22
sediment	2.25±0.20	10.60±1.35	1.88±0.23		
UW1728	5.04±0.30	8.12±1.00	2.23±0.14	2.49±0.21	2.75±0.12
Sediment	1.99±0.17	10.38±1.12	2.08±0.13		
UW1729	4.87±0.30	7.48±1.21	1.91±0.06	2.64±0.23	2.45±0.07
Sediment	1.65±0.18	11.83±1.32	2.04±0.13		

Table S3. Dose rates (Gy/ka)*

Sample	*alpha*	*beta*	*gamma*	*cosmic*	*total*
UW1725	0.74±0.07	2.09±0.14	1.09±0.10	0.30±0.06	4.22±0.19
UW1726	1.54±0.11	2.19±0.09	1.08±0.07	0.30±0.06	5.11±0.17
UW1727	1.58±0.52	2.75±0.21	1.09±0.10	0.30±0.06	5.72±0.57
UW1728	1.13±0.06	2.42±0.13	1.07±0.09	0.30±0.06	4.92±0.18
UW1729	1.52±0.09	2.17±0.09	1.07±0.09	0.30±0.06	5.06±0.17

* Dose rates for ceramics are calculated for OSL. They 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 S2 due to moisture correction.

Equivalent Dose

Equivalent dose (D_e) was measured using TL, OSL and IRSL.

TL was measured on a Daybreak reader using a 9635Q photomultiplier with a Corning 7-59 blue filter, in N_2 atmosphere at 1°C/s to 450°C. A preheat of 240°C with no hold time preceded each measurement. Artificial irradiation was given with a ^{241}Am alpha source and a ^{90}Sr beta source, the latter calibrated against a ^{137}Cs gamma source. Disks were stored at room temperature for at least one week after irradiation before glow out. Data were processed by Daybreak TLApplic software.

D_e for TL was determined by a combination additive dose and regeneration methods (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 D_e 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 was determined by calculating the D_e at temperature increments between 250° and 450°C and determining over which temperature range the values do not differ significantly. This plateau region was compared with a similar one constructed for the b-value (alpha efficiency), and the overlap defined the integrated range for final analysis.

Several discs were used to test for anomalous fading in TL. The natural luminescence was first measured by heating to 450°C. The discs were 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 were staggered in time so that all second glows were 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 showed fading and the signal versus time values could be reasonably fit to a logarithmic function, an attempt was 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.

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

Optically stimulated luminescence (OSL) and infrared stimulated luminescence (IRSL) were carried out on single aliquots following procedures adapted from Banerjee et al. (2001) and Roberts and Wintle (2001. D_e was 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. A preheat of 240°C for 10s, a test dose of 3.1 Gy, and a cut heat of 200°C was used. The luminescence was 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 was through 7.5mm of Hoya U340 (ultra-violet) filters. The two stimulations were used to construct IRSL and OSL growth curves, so that two estimations of D_e were 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 feldspar signal.

A dose recovery test was performed for OSL/IRSL by first zeroing the sample by exposure to light and then administering a known dose. The SAR protocol was then applied to see if the known dose could 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). Results from several samples from different geographic locations show that OSL b-value is less variable and centers around 0.5. IRSL and TL b-values are more variable and higher than that for OSL. The b-value for IRSL and OSL was measured by giving an alpha dose to aliquots whose luminescence was drained by exposure to light. A D_e is determined by SAR using beta irradiation, and the ratio of beta D_e and the known alpha dose 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.

The TL plateau (Table S4) was more than 70°C in breadth for all samples except UW1726. This suggests the ceramics were all fired sufficiently, including UW1726 where the plateau was at high temperature. Only two samples showed a sensitivity change with heating. TL anomalous fading was evident in all samples. Anomalous fading rates, or g-values, were high (>10%) for two samples, so high for UW2727 that correction produced an infinite date. Either the measurement was in error or the fading rate changed through time for this sample.

Table S4. TL parameters

Sample	*Plateau (°C)*	*1st/2nd ratio**	*fit*	*Fading g-value***
UW1725	250-430	1.0	linear	12.5±3.4
UW1726	300-350	0.85±0.08	quadratic	4.7±0.9
UW1727	250-330	1.0	linear	15.1±2.0
UW1728	270-340	1.0	quadratic	5.3±1.4
UW1729	250-320	2.10±0.20	linear	5.6±1.6

*Refers to slope ratio between the first and second glow growth curves. A glow refers to luminescence as a function of temperature; a second 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.

OSL/IRSL was measured on 5-7 aliquots per sample (Table S5). Scatter was low (less than 11% over-dispersion) for all samples. An IRSL signal could be measured on all samples, but was weaker than the OSL signal by 2 to 4 times. This is not atypical. Ceramics were heated when made and that has a tendency to increase the quartz signal and reduce the feldspar signal. IRSL stems from feldspars, which are prone to anomalous fading. A relatively large 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. Another measure of feldspar contribution is the size of the OSL b-value. For these samples the b-value was higher than the range of quartz for all samples (it was not measured for UW1727, for which an average of the others was used). This suggests feldspars are making some contribution to the OSL signal, which thus may fade. Fading was only measured on the TL signal, because of lengthy machine time required to measure fading for OSL and IRSL. As a test of the SAR procedures, a dose recovery test

was performed. The recovered dose was within two-sigma of the given dose for all samples, but UW1729. D_e and b-values for TL, OSL and IRSL are given in Table S6.

Table S5. OSL/IRSL data

Sample	# aliquots*		OSL Over-dispersion (%)	Dose Recovery (OSL)	
	OSL	IRSL		Given Dose (sß)	**Recovered Dose (sß)**
UW1725	6	6	6.7±7.8	50	**49.1±2.3**
UW1726	6	6	10.6±3.7	60	**64.6±3.0**
UW1727	**5**	**5**	**3.0±3.2**	**60**	**58.7±2.9**
UW1728	**6**	**6**	**4.6±7.5**	**50**	**51.0±2.5**
UW1729	**7**	**7**	**8.8±3.1**	**60**	**70.6±4.6**

* Denotes number of aliquots with measurable signals.

Table S6. Equivalent dose and b-value

Sample	*Equivalent Dose (Gy)*			*b-value (Gy µm^2)*		
	TL	*IRSL*	*OSL*	*TL*	*IRSL*	*OSL**
UW1725	10.2±0.99	7.28±0.38	6.82±0.22	2.39±0.41	1.38±0.07	0.71±0.06
UW1726	6.12±0.78	7.42±0.18	8.65±0.41	0.97±0.08	1.33±0.09	1.42±0.07
UW1727	10.1±0.35	8.58±0.21	11.4±0.29	1.43±0.15		
UW1728	11.9±0.32	8.87±0.45	9.64±0.25	1.36±0.09	1.36±0.04	1.03±0.03
UW1729	8.54±0.73	9.00±0.23	11.1±0.43	1.27±0.09	1.33±0.08	1.45±0.05

Ages

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

Table S7 gives the derived ages for each sample. Two samples, UW1726 and UW1729 showed agreement between the OSL age and the fading-corrected TL age. These are the most reliable ages. UW1725 had agreement between OSL, IRSL and uncorrected TL. Such agreement would not be likely if fading were an issue. The OSL b-value was also the lowest of the five samples, suggesting the OSL signal was mostly from quartz and not likely to fade. The measured TL fading rate of 12% is probably an over-estimate. UW1728 had agreement between OSL and uncorrected TL. The OSL signal could fade some, so this age may be slightly underestimated. For UW1727, the TL and IRSL ages are both younger than the OSL age. Their signals likely fade. The OSL age is the best estimate but it also could be a slight underestimate. The three most reliable dates, UW1725, UW1726 and UW1729 date around AD200-400. The other two, which may be underestimates, are somewhat older.

Table S7. Ages

Sample	Age (ka)*	% error	Basis for age	Calendar date
UW1725	1.56±0.07	4.3	OSL/IRSL/uncorrected TL	AD 440 ± 70
UW1726	1.69±0.09	5.6	OSL/corrected TL	AD 320 ± 100
UW1727	2.00±0.21	10.5	OSL	AD 10 ± 210

UW1728	2.08±0.08	4.0	OSL/uncorrected TL	BC 70 ± 80
UW1729	2.22±0.11	5.1	OSL/corrected TL	AD 210 ± 110

* The base year for ka is 2015.

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Preparation and analysis of sediment samples HY37-90, 37-300, 50-135, 49-175, 49-6.5, 37-200 at the Desert Research Institute.

Preparation

We used typical approaches to remove carbonates and organic material through treatment with 10% v.v. dilution of HCl and 30 volumes H_2O_2. We chose a grain size of 63-90 μm to maximize the potential for grains to be in situ since deposition. This fraction is considered 'fine grained' in luminescence practice. Preparation of fine grained polymineral samples typically involve treatment in hydrofluorosilicic acid (HFS) doped with quartz (Berger, 1991; Roberts, 2007). We found that this did not sufficiently isolate quartz with both quartz and feldspar signals appearing in initial tests. Through a series of experiments, we found that using both a HFS treatment and a short hydrofluoric acid (HF) treatment successfully isolated quartz and eliminated the feldspar signal in most measurements. Samples were treated with refrigerated quartz-doped 25% hydrofluorosilicic acid H_2SiF_6 (HFS) for 7 days. Then an etch in hydrofluoric acid (HF) involved treatment in a 1:4 ratio of 32-38% hydrochloric acid (~12 N HCl) to 48% hydrofluoric acid (HF) for 10 minutes. Samples were then re-sieved to remove grains that were reduced to <63 μm by the HF etch. We then mounted 4 mm diameter aliquots of quartz grains onto 9 mm aluminum steel discs using silicone spray. Discs were not re-used to limit cross-contamination with fine grained particles.

Instrument configuration for analysis

All measurements were made on Risø TL/OSL readers model DA-20, serial #243 or #244 using continuous wave measurements of the luminescence signal. Infrared stimulation was made with a cluster of infrared (IR) diodes (Vishay TSFF 5210) with peak emission at 870 nm and a maximum power of 145 mW/cm^2 at the sample position. Blue light stimulation was made with a cluster of blue LEDs (NICHIA type NSPB-500AS) with a peak emission at 470 nm and a total power of 80 mW/cm^2.

Analysis

A dose recovery at varied pre-heat temperatures was conducted on (7 mm) aliquots of quartz using an applied dose of ~12 Gy, which was chosen to be relatively close to be the estimated equivalent dose of the samples. A preheat temperature of 180 °C was found to produce the most reproducible results and was used for subsequent measurements. Internal overdispersion (termed σ_b) was measured using a dose recovery test at 180 °C and found to be = 0.09 (typically reported as %). This value was used for modeling equivalent dose (D_e). Owing to some IR signal appearing in initial tests and the likelihood of contaminant feldspar in fine-grained samples, we used a modified SAR (Murray and Wintle, 2000; 2003) approach to measuring D_e. We used post IR-OSL (aka 'double SAR') (Roberts and Wintle, 2001; 2003) measurements with an IR depletion ratio check on multigrain, 4 mm aliquots of quartz for all 6 samples to measure D_e.

In the post-IR OSL SAR approach used, an initial IR stimulation is applied before OSL stimulation to attempt to deplete the signal from any potential contaminant feldspar (Spooner, 1994). The signal emitted from the subsequent blue light stimulation appeared to be dominated by luminescence from quartz and was used for D_e determination. The dating experiments were conducted with the following procedural details that were determined on the basis of initial dose recovery and preheat tests:

- Preheat: 180°C
- Cut heat: 160°C
- IR stimulation using IR diodes for 40 s at 50°C
- OSL measurements using blue diodes for 40 s at 125°C
- Test Dose: 40 s (~5 Gy)
- At least five regenerative doses that span the range of aliquot D_e values of each sample (regenerative doses ranged from 0-100 Gy).
- Instrument beta source calibration: 0.121 Gy/s

An exponential fit was used to fit regeneration curves. Routine screening criteria included rejection of aliquots that exhibited the following behavior and characteristics:

- Poor signals as judged from net natural signals less than three standard deviations above the background.
- Natural signals that did not intersect saturating growth curves.
- Failure to produce, within 10% the same signal ratio from identical regeneration doses given at the beginning and end of the SAR sequence, which suggests inaccurate sensitivity correction (recycling test).
- Maximum test dose error of 20%.
- Maximum paleodose error of 20%.
- Maximum recuperation of %10

At least 48 aliquots were measured for each sample and for most samples we measured >100 aliquots. For most samples, a minimum of 20 accepted D_e measurements were used in age modeling. The total equivalent dose or burial dose (D_b) for each sample was modeled using the central age model (CAM) (Galbraith et al., 1999) that models the central tendency taking into account the internal overdispersion of the sediment measured with σ_b and the error associated with each aliquot measurement. Kernal density estimates and radial plots were used to further assess the distribution of D_e.

Dose rate and age calculations

Samples for dose rate were dried and milled to a fine, flour consistency and sent to ALS Geochemistry in Reno, NV for geochemical analysis of U, Th, and K_2O. U and Th samples were fused with lithium borate and measured with ICP-MS. Because the samples are fine grained, U and Th were also measured in the DRILL using α-counting. Results were similar to those from geochemistry. We used results from α-counting in the dose rate calculation. K_2O was measured at ALS on bulk sample with ICP-AES and converted to % K. Dose rates (Gy/ka) were calculated using the conversion factors of Liritzis et al., 2013 and are shown to 2 decimal places; ages were calculated prior to rounding. A water content of 3 ± 1.5 % (expressed as the percentage of the mass of dry sediment) was used for all samples. The water content was estimated for the lifetime of burial based on the measured water content of samples. For coarse grained samples, etching removes contribution of α particles to the dose rate, but for fine grained samples, the α efficiency should be evaluated. In dose rate calculations, we use an a-value (referring to the α-efficiency) of 0.035 ± 0.003 following Lai et al., 2008 and Feathers et al., 2012, which both measure loess from the same region as the samples in this study. Central values are given for dose rates and errors

are incorporated into that given for the total dose rate. Cosmic dose rates (Gy/ka) are calculated according to Prescott and Hutton (1994). Dose rate and final age calculations were made using DRAC (Durcan et al., 2015). Ages are expressed as thousands of years before A.D. 2011 and rounded to the nearest 10 years. Error on ages is 1σ (Table S8).

Table S8. Luminescence dating results for sediment samples HY37-90, 37-300, 50-135, 49-175, 49-6.5, 37-200 prepared and analyzed at the Desert Research Institute Luminescence Laboratory (DRILL)

DRILL Sample number	Field Sample ID	Depth (m)	N accepted (N analyzed)[a]	Over-dispersion (%)	D_b (Gy)[b]	U (ppm)[c]	Th (ppm)[c]	K (%)[c]	External beta dose rate wet (Gy/ka)	External gamma dose rate wet (Gy/ka)	Cosmic dose rate (Gy/ka)[d]	Total dose rate (Gy/ka)[e]	Age (ka)[f]
DRILLJZG001	HY37-90 cm	0.9	34 (132)	30	33.31 ± 1.82	2.89	9.08	1.93	2.07	1.20	0.27	3.69 ± 0.17	9.02 ± 0.65
DRILLJZG002	HY37-300 cm	3.0	42 (143)	27	37.66 ± 1.68	2.93	9.66	1.86	2.03	1.22	0.21	3.62 ± 0.17	10.40 ± 0.67
DRILLJZG003	HY50-135 cm	3.6	42 (48)	0	36.41 ± 0.62	3.47	10.83	2.09	2.31	1.38	0.20	4.08 ± 0.19	8.93 ± 0.44
DRILLJZG005	HY49-175 cm	4.3	7 (142)	7	38.63 ± 3.25	2.82	9.20	1.93	2.06	1.20	0.18	3.60 ± 0.18	10.73 ± 1.04
DRILLJZG006	HY49-6.5 m	0.5	12 (96)	28	4.96 ± 0.46	2.52	10.86	1.90	2.04	1.24	0.30	3.74 ± 0.18	1.32 ± 0.13
DRILLJZG007	HY37-200 cm	2.0	27 (143)	26	32.49 ± 1.78	2.75	11.09	1.93	2.10	1.28	0.24	3.79 ± 0.18	8.58 ± 0.62

[a] N is the number of D_e determinations accepted after screening; in parentheses are the total number of aliquots measured.

[b] The burial dose, D_b, and associated error were modeled with the central age model (CAM) (Galbraith et al., 1999).

[c] U and Th were measured using α-counting in the DRILL. K_2O was measured on bulk sample with ICP-AES and converted to % K.

[d] Cosmic dose rates (Gy/ka) are calculated according to Prescott and Hutton (1994). The location for all samples is 33.2357°N; 103.9043°E, altitude 2535 masl.

[e] Dose rates (Gy/ka) were calculated using the conversion factors of Liritzis et al. 2013 and are shown rounded to two decimal places; ages were calculated using values prior to rounding; central values are given for dose-rates and errors are incorporated into that given for the total dose-rate. Water content of 3 ± 1.5 % was used for all dose rate calculations.

[f] Luminescence ages were calculated using DRACv1.2 (Durcan et al., 2015) and are expressed as thousands of years before 2011 (year collected) and rounded to the nearest 10 years. Error is 1 sigma.

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