Article:

Adams, K.D. and Rhodes, E.J. orcid.org/0000-0002-0361-8637 (2019) Late Holocene paleohydrology of Walker Lake and the Carson Sink in the western Great Basin, Nevada, USA. Quaternary Research, 92 (1). pp. 165-182. ISSN 0033-5894

https://doi.org/10.1017/qua.2018.151

IRSL Data Repository Text: Sample Preparation and Dating Methods

Samples were opened and prepared in the University of California, Los Angeles (UCLA) luminescence laboratory under low-intensity red and amber lighting. Potassium feldspar grains of 175–200µm were separated from the central, unexposed, portion of each sample; following wet sieving to isolate the correct grain size range, samples were treated in dilute HCl to remove carbonate, dried, and the potassium feldspar component floated off using a lithium metatungstate (LMT) solution with a density of 2.58 g/cm^3. After rinsing, samples were treated in 10% hydrofluoric acid for 10 minutes to etch the outer surfaces of each feldspar grain, dried, and sieved at 175µm to remove small fragments.

Between 200 and 600 K-feldspar grains of each sample were measured using a post-IR IRSL SAR (single aliquot regenerative-dose) protocol modified for single grains from Buylaert et al. (2009), documented in Rhodes (2015). Measurements were made in a Risø TL-DA-20CD automated luminescence reader, fitted with an XY single grain attachment incorporating a 150 mW 830 nm IR laser passed through a single RG-780 filter to reduce resonance emission at 415 nm, used at 90% power for 2.5 s. All measurements were made using a BG3 and BG39 filter combination, allowing transmission around 340–470 nm to an EMI 9235QB photomultiplier tube. For the natural measurement, and following each regenerative-dose and test dose application, a preheat of 250º C for 60 s was administered. IRSL was measured (for 2.5 s for each grain) at 50º C, and then subsequently at 225º C (for the post-IR determination). Following a test dose of 9 Gy, an identical preheat, IRSL at 50º C and post-IR IRSL at 225ºC were administered. Each SAR cycle was completed with a hot bleach treatment using an array of Vishay TSFF 5210 870 nm IR diodes at 90% power for 40 s at 290º C. The SAR sequence incorporated measurement of the natural IRSL, between four and six regenerative dose points, a zero dose point to assess thermal transfer, and a repeat of the first regenerative dose point, to assess recycling behavior.

Growth curves were constructed for the post-IR IRSL signal measured at 225º C using an integral of the background-subtracted sensitivity-corrected IRSL from the first 0.5 s, fitted with an exponential plus linear function. For most samples, around 5 to 10% of measured K-feldspar grains provided a useful post-IR IRSL signal, typically providing between 20 and 60 single grain results for each sample; other grains were either insensitive, associated with large uncertainties, or in the case of a few grains, the post-IR IRSL signal was in saturation. Samples typically displayed a uniform minimum equivalent dose value, with other grains displaying higher dose values, interpreted as grains incompletely zeroed before or during transport owing to rapid deposition in water under high energy shoreline conditions. Isolation of a population of grains for age estimation used a “discrete minimum” procedure in which higher values were excluded until the remaining grains were consistent with an overdispersion (OD) value of 15%, based on experience from quartz single grain OSL dating (e.g. Rhodes et al., 2010). No fading correction was applied, as detailed determination of single grain post-IR IRSL fading rates for key samples displayed negligible fading. Fading uncorrected PIR-IR_{225} has been shown to agree with independent age control in several studies (e.g. Rhodes, 2015); in some cases, residuals associated with thermal transfer arising from high pre-heat temperatures can result in age overestimation (Buyalert et al., 2011; Chen et al., 2013; Li et al., 2014). Gamma dose rates were based on in- situ NaI spectrometer measurements; external beta dose rates were calculated from ICP-MS (U, Th) and ICP-OES (K) measurements of sediment from the end of each sample tube, and internal beta dose rate was based on 12.5% internal K content. Cosmic dose rates were based on measured overburden depth, and moisture correction used contemporary water content values.

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