

Dating Late Pleistocene pluvial lake shorelines in the Great Basin, USA using rock surface luminescence dating techniques: developing new approaches for challenging lithologies

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Abstract. This study examines the feasibility of dating pluvial lake beach ridges using rock surface luminescence dating techniques. Dating pluvial lake highstands in the internally drained Great Basin of the United States helps us understand the timing of changes in precipitation and temperature patterns in western North America during the Late Pleistocene. The majority of highstand ages have relied on few radiocarbon ages of shell and/or charcoal sometimes coupled with luminescence dating
15 of sand. Within our study area in the south-central Great Basin, luminescence ages of sand-size particles have successfully dated aeolian influxes of sand during arid intervals, but have not successfully dated the highstand beach ridges, the best preserved of which are largely gravel.

Directly dating when these gravel clasts were last exposed to sunlight via luminescence is ideal but their limestone and volcanic lithologies prove challenging. Initial measurements from these lithologies show that feldspar luminescence signals are suited
20 to single-aliquot regenerative (SAR) dose measurement protocols and show evidence for heterogeneous bleaching of rock surfaces. Polymineral extracts from dissolved limestone clast surfaces from Coal Valley that contain sufficient detrital sediment exhibit infrared signals measured at 50° C (IR₅₀) with low to moderate fading rates. Single-grain ages from detrital sediment from three clasts, calculated using the central dose model, are statistically consistent with the radiocarbon age estimate of the Pluvial Lake Coal highstand.

25 Crushed slices from volcanic clasts from Cave Valley could be dated using a high-temperature (290°C) post-infrared infrared stimulated luminescence (pIRIR) signal with a correction for fading. Many ages obtained from volcanic clast surfaces are observed to be several thousand years younger than the expected age of the ~18-20 ka beach ridge. This suggests that the volcanic rocks have been exposed to light long after the pluvial lake highstand, likely because of bioturbation, and that their most recent burial occurred in response to climatically driven soil formation processes. Comparisons between age-depth profile
30 plateau ages from inside volcanic rocks and independent age control suggest that gravel-sized volcanic rocks were small enough to have been bleached throughout their entire thickness in the pluvial lake beach environment and that pIRIR signals that record the time of beach ridge formation and subsequent soil formation during the Pleistocene-Holocene transition may be preserved within the rock sub-surface.

This study develops novel dating approaches for challenging rock lithologies. Rock surface dating techniques for pluvial lake beach ridges in the Great Basin should be further developed with consideration of local bedrock type(s), clast size, sample collection and preparation methods, gravel bleaching processes in pluvial lake environments and the impact of soil development and bioturbation on study sites.

1 Introduction

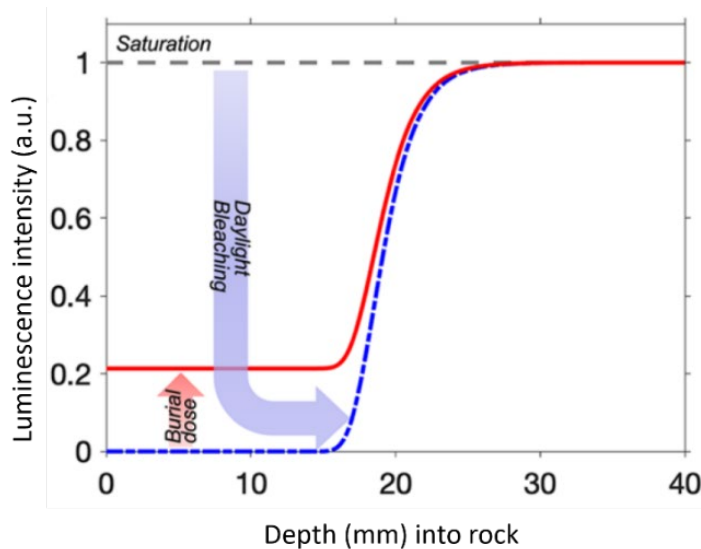
1.1 Dating pluvial lake shorelines

During the Pleistocene epoch, pluvial lakes covered vast areas of the arid western United States, including Nevada, western Utah, southeastern Oregon, and eastern California. Most of the pluvial lake basins are found within the hydrologic Great Basin, an area of closed drainage encompassing about 520,000 km² (Reheis et al. 2014). Great Basin pluvial lake shorelines are invaluable indicators of past hydroclimate conditions and provide baseline data for atmospheric circulation models (Reheis et al., 2014). As such, accurate determinations of the depositional ages of these sediments and related landforms are critical for interpretations of paleoenvironmental conditions. Pluvial lake history is also important for reconstructing the early migration and settlement patterns of people in the western US (Adams et al., 2008). The abundant plants and animals within and near freshwater lakes and marshes have always attracted people. As these systems respond to changing climatic and topographic conditions, their geographic distribution changed in ways that influenced the early mobility of societies (Wriston, 2009). Therefore, reconstructing the timing of pluvial lake highstands, regressions, and expansion and contraction of fringing wetlands, can put archaeological sites into a paleoenvironmental context that helps explain their distribution.

Past studies applying a range of relative and absolute dating techniques to shoreline features have shown serious discrepancies between methods that are difficult to explain (Owen et al., 2007; Redwine et al., 2020). In particular, optically stimulated luminescence (OSL) dating techniques commonly yield inconsistent results with high error (Adams and Rhodes, 2019) or underestimate the expected ages of pluvial highstand beach ridge deposits by thousands of years despite pristine sampling conditions (Owen et al., 2007; Adams, unpublished data). Previous research in Coal Valley, Nevada yielded post-infrared infrared stimulated luminescence 225°C (pIRIR₂₂₅) ages that severely underestimate the expected age of the 1522 m above sea level (asl) Lake Coal highstand, which has been dated to 15,873-16,281 cal yrs BP using radiocarbon dating (Wriston and Adams, 2020). These were 4.34 ± 0.5 ka (1515.6 m asl), 3.69 ± 0.42 ka (1513.8 m asl) and 1.87 ± 0.18 ka (1521.5 m asl) (Wriston and Adams, 2020). We suspect that the medium sands (180-300 μ m) sampled from the beach gravels were actually younger wind-blown sands that translocated into the initially open-work beach ridge gravels after beach ridge formation leading to an age under-estimate. This research investigates the feasibility of dating gravel-size clasts in pluvial beach ridge shorelines to remedy this age discrepancy. Gravels require greater energy to move and should reflect when water flow was strong enough to form the beach ridges. Our project area in Lincoln County, Nevada has beach ridges often comprised of limestone and volcanic rocks. Here, we test and outline novel sample preparation protocols for these materials and investigate the optical properties from polymineral grains extracted from them.

1.2 Luminescence dating rock surfaces

Luminescence dating determines the last time sediments have been exposed to sunlight or heat prior to deposition and burial and provides chronologies for archaeological and geological events (Huntley et al., 1985; Lian and Roberts, 2006; Roberts and Lian, 2015; Woor, 2022). Luminescence dating techniques are commonly applied to silt or sand grains, but in recent years, significant advances have been made in dating rock surfaces (Habermann et al. 2000; Greulich et al., 2005; Vafiadou et al., 2007; Liritzis, 2011; Simms et al., 2011; Sohbati et al., 2012; Freiesleben et al., 2015; Simkins et al., 2016; Khasawneh et al., 2019; Gliganic et al., 2021; Freiesleben et al., 2023, Ageby et al., 2024) as well as mineral grains encased in carbonate deposits, calcarenite and limestone (Rich et al., 2003; Prescott and Habermehl, 2008; Liritzis et al., 2008; 2010; Ageby et al., 2023). Luminescence ages from rocks are important for sites that lack adequate sand/silt for traditional luminescence dating techniques, in addition to sites that are contaminated by mobile fine grain materials that severely post-date (underestimate) the true age of the landform or archaeological site. Rock surface dating approaches applied to natural deposits, such as beach ridges (Simms et al., 2011), flood gravels (Smith et al., 2023), or moraines (Rades et al., 2018; Yang et al., 2024), most commonly target cobble size clasts, which are sampled under dark or dim red light conditions. Sampling may be done at night and/or with a light-safe tarp or tent to block any ambient sun, moon or traffic/city light that may reset the OSL signal. Once in the laboratory, ~10 mm diameter cores are extracted from the rock, which are then sliced into sub-millimeter slices, and the OSL signal is measured from each slice. This produces a luminescence-depth profile that traces the OSL signal intensity from the rock surface to depth, where the depth of light penetration may be inferred from past bleaching episodes (Fig. 1). Figure 1 shows theoretical luminescence-depth profiles that would be measured after: i) light exposure of the rock surface, ii) re-burial a rock surface after an exposure event, and iii) after no light exposure or burial of the rock for extensive time. For a rock that had sufficient sun exposure prior to burial, the time of the most recent exposure event can be calculated from the near-surface plateau of the luminescence-depth profile (red line in Fig. 1).



90 **Figure 1. Luminescence-depth profiles that are expected after i) light exposure of the rock surface (s-shaped blue dashed line), ii) re-burial a rock surface after an exposure event (solid s-shaped red line), and iii) after no light exposure (straight grey dashed line). From Smith et al. (2023).**

Generating an age from rock luminescence-depth profiles requires an estimate of the environmental dose rate at the rock surface, as well as at depth. Dose rate models, therefore, take into account measured dose rates from the rock and surrounding sediments, and use established beta and gamma attenuation factors to calculate dose rates at depth within the rock (e.g., Jenkins et al., 2018; Riedesel et al., 2020).

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2 Study area and sample sites

This study examines the optical and dosimetric properties from clasts collected from pluvial lake beach ridge gravels in Coal, Cave, and Lake valleys, in Lincoln County, Nevada (Fig. 2, Figs S1-S4). In the following discussion, radiocarbon ages are reported in calibrated years before present (cal yrs BP) and are calibrated with IntCal 20.14c using Calib 8.2 (Reimer et al., 2020). Luminescence ages are reported in kilo annum (ka), and synthesized ages (that may include a variety of stratigraphic and archaeological evidence) are reported in years ago. The study area is characterized by an arid to semi-arid climate with ~250 mm of annual precipitation, a quarter of which falls during the summer. Great Basin conifer woodland occupies elevations above 1770 m, while desert scrub, including rabbit brush (*Chrysothamnus nauseosus*, *Chrysothamnus viscidiflorus*) green ephedra (*Ephedra viridis*), horsebrush (*Tetradymia canescens*) and sagebrush (*Artemisia nova*) dominate elevations of 1500 to 2000 m (Spaulding, 1985). Only sheer cliffs and playa floors are devoid of plants.

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During the last major pluvial period in eastern Nevada, pluvial lakes reached their highstand between ~15,000 and ~20,000 years ago (see highstand ages for lakes Carpenter, Spring, Railroad, Jakes, Newark, Diamond, Franklin, Clover and Waring in Munroe and Laabs (2013), their table 2). Coal Valley, Cave Valley, and Lake Valley in Lincoln County, all contained pluvial

lakes (Pluvial Lakes Coal, Cave, and Carpenter, respectively; Mifflin and Wheat, 1979) and marsh systems during the late
110 Pleistocene (cf. Wriston and Adams, 2020). Warming and drying of these lakes between 14,600 and 12,900 years ago (Rhode
and Adams, 2016) was stalled with cooler conditions during the Younger Dryas (ca. 12,900 to 11,700 years ago, Rasmussen
et al., 2006). Beginning ca. 8,000 years ago (Wriston, 2009), extreme drought dried the lakes, marshes, and springs and wind
displaced much of the lake and marsh sediments in the basin floors throughout the Great Basin. The declines in area and
productivity of basin wetlands varied spatially and temporally, likely with decadal or century-level fluctuations. The pace and
115 tempo of the lake and wetland decline to modern conditions has yet to be fully reconstructed.

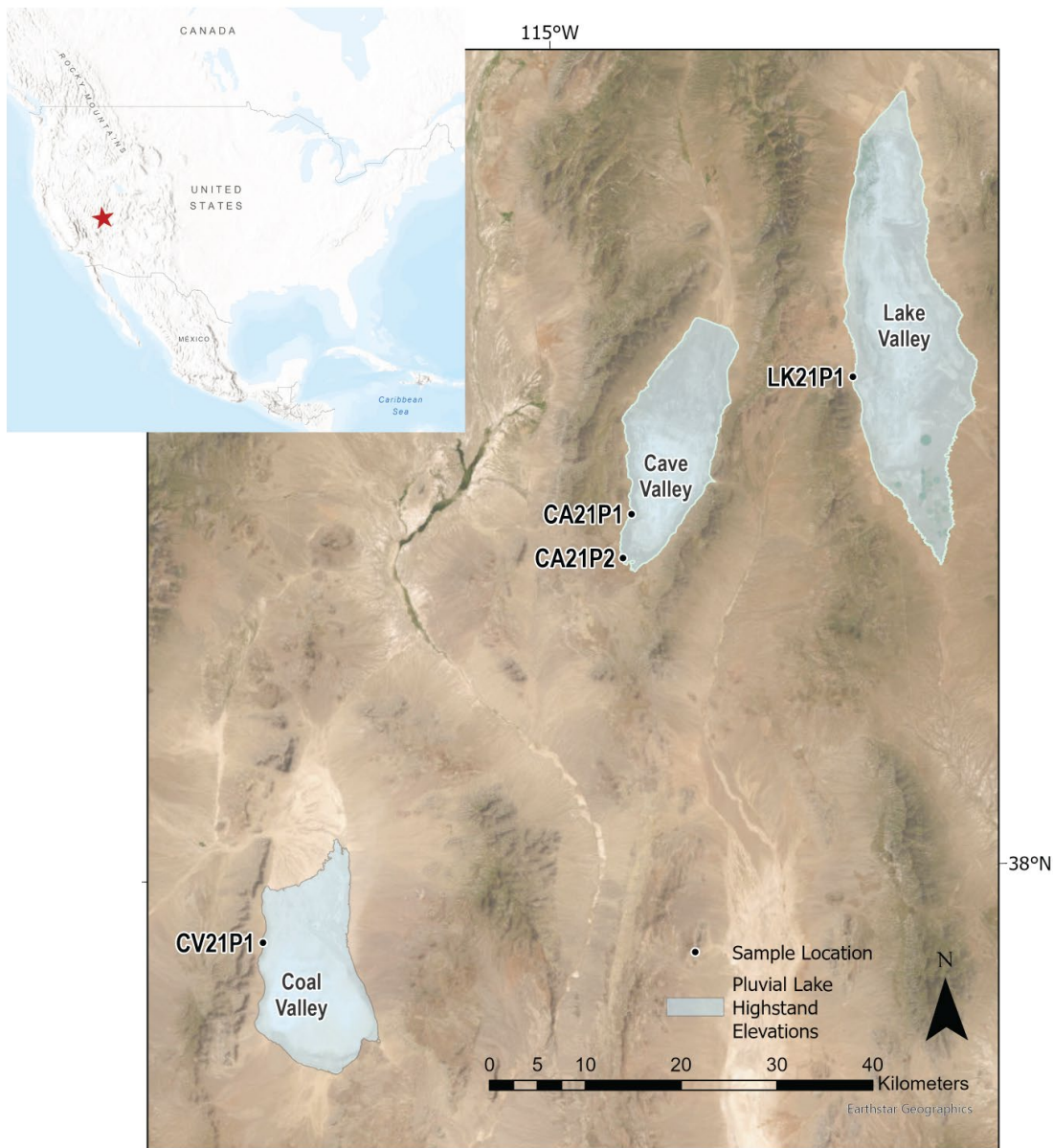


Figure 2. Study site locations in Coal, Cave and Lake valleys, Lincoln County, Nevada in relation to pluvial lake highstand elevations. Basemap: ESRI Maxer, Earthstar Geographics, TomTom, Garmin, FAO, NOAA, USGS, and the GIS User Community.

120 We sampled beach ridges inferred to have been created during the highstand in each basin (Table 1). Geochronological and geomorphic evidence from Coal, Cave, and Lake valleys suggests that pluvial lakes in these basins reached their last highstand sometime between ~16,000 and ~20,000 years ago (Table 1 and Table S1). Wriston and Adams (2020) radiocarbon dated the Coal Valley highstand to 16,088 median cal yrs BP ($13,366 \pm 59$ BP, D-AMS 029792) using a young freshwater Lymnaeidae

Stagnicola sp. shell from backbar lagoonal deposits. The species and size of the dated shell suggest that it is unlikely to suffer from reservoir effects due to the acquisition of old carbon (cf. Pigati et al., 2010). A radiocarbon age of shoreline features from Lake Valley suggests that Lake Carpenter was near its highstand at ca. 17,258 median cal yrs BP (14,210 ± 100 BP, Beta-50773; Currey in Lillquist, 1994; Munroe and Laabs, 2013). Duke and Young (2018) report ages of wetland sediments in the lake floor by 12,685 cal yrs BP (10,656 ± 48 BP, D-AMS 20244), indicating that the lake had receded before this time and further drying occurred before a 9.27 ± 1.87 ka (USU-2529) inset lake shoreline formed.

The highstand at Cave Valley has never been directly dated; however, GIS computer simulations by Duke and King (2014) that predict the relative chronology of lake desiccation using basin geomorphology and hydrology, suggest that Cave Lake desiccated before neighbouring Lake Carpenter in Lake Valley. Therefore, the highstand of pluvial Lake Cave likely occurred between ~18,000 and ~20,000 years ago (Munroe and Laabs, 2013; Duke and King, 2014; Duke and Young, 2018). During recession of Cave Lake, the distributary system entering Cave Valley from the north would have expanded providing patchy wetland environments likely utilized by people during the Paleoamerican period (Duke and Young, 2018). Dated sediments from this distributary system suggest that it dried up ca. 9,422 median cal yrs BP (8,330 ± 30 BP, Beta-440300) after a mesic period and low lake transgression ca. 11.33 ± 1.68 ka (USU-2527) based on luminescence dating of quartz grains below a gravel bar (Duke and Young, 2018).

Table 1. Study site locations in Coal, Cave and Lake valleys.

Study site	Basin	Lat/Long (decimal degrees)	Beach ridge elevation ¹ (m asl)	Lake highstand elevation ¹ (m asl)	Highstand age ²
CV21P1	Coal Valley	37.940906, -115.363785	1520.50	1522	15,873-16,281 cal years BP
CA21P1	Cave Valley	38.337317, -114.914635	1851.66	1852	~18,000-20,000 years ago
CA21P2	Cave Valley	38.296278, -114.925958	1851.68	1852	~18,000-20,000 years ago
LK21P1	Lake Valley	38.461389, -114.646172	1826.82	1826	17,011-17,536 & 17,629- 17,699 cal years BP

¹ Highstand elevations in meters above sea level are interpreted from highest shoreline features in satellite imagery using 1/3 arc second Digital Elevation Model from the USGS National Map (nationalmap.gov) with accuracy of around 0.82 m. The CV21P1 location was surveyed to within 20 cm corrected elevation using a Trimble Nomad during previous work (Wriston and Adams, 2020: 55).

² Radiocarbon ages for sites CV21P1 and LK21P1 are calibrated using IntCal20 in Calib 8.2 (Reimer et al., 2020). The two age ranges reported for site LK21P1 reflect two age interceptions onto the IntCal20 calibration curve. The radiocarbon age dates a time when Lake Carpenter (Lake Valley) was near (4 m below) its highstand.

Bedrock gravel material types in the study area are dominated by limestones and volcanic rock derived from the local bedrock (Figs S5-S7). These lithologies are representative of the bedrock geology of much of Nevada (Crafford, 2007). Limestones, sandstones, and shales formed when Nevada was a broad carbonate shelf during the Paleozoic era. Younger Mesozoic and

Cenozoic volcanic flows, tuffaceous rocks, intrusive igneous and sedimentary rocks formed during a subsequent period of complex tectonic movements, volcanic activity, and terrane accretion (Dickinson, 2006).

2.1 Coal Valley

155 The sampled beach ridge at 1520.5 m above sea level in Coal Valley (site CV21P1) is superimposed on a Quaternary alluvial fan on the western side of Coal Lake basin and is the highest evident shoreline (Fig. S1). This beach ridge was created after the last glacial maximum (Marine Isotope Stage 2) and previously dated to ca. 16,000 years (15,873-16,281 cal yr BP at 2σ calculated using IntCal20; $13,366 \pm 59$ RYBP; D-AMS 029792) by Wriston and Adams (2020). It has been incised by a drainage channel exposing sandy beach gravels overlying lagoonal silts and clays. The soils in this area have been mapped as aridisols and entisols of the Ursine-Cliffdown Association that are typified by thin A horizons of very gravelly sandy loam
160 that grade into B or C horizons of gravelly sandy loam (National Cooperative Soil Survey, USA, 2025). The Golden Gate Range west of the CV21P1 sample location is comprised of Lower Mississippian limestones, such as the Joana, Mercury, and Bristol Pass formations underlain by Pilot shale that tops the upper Devonian Devils Gate limestones belonging to the Guilmette Formation (Rowley et al. 2017; Tschaz and Pampayan, 1961; 1970) (Fig. S5).

2.2 Cave Valley

165 In Cave Valley, two beach ridges were sampled, one on the west side of the basin at 1851.66 m elevation (CA21P1; Fig. S2) and one at the southwestern end of the basin at 1851.68 m elevation (CA21P2; Fig. S3). The pluvial lake highstand is at 1852 m (Table 1). The ridge on the west side of the basin is superimposed on a Quaternary alluvial fan and is the highest of a series of at least six ridges stepping up from the playa. The sampled beach ridge to the south (CA21P2) is wider and flanks a wavecut bench eroded into andesitic and basaltic bedrock. Both localities are dominated by aridisols with gravelly sandy loam A
170 horizons overlying gravelly sandy loam B horizons (National Cooperative Soil Survey, USA). Tschaz and Pampayan (1961; 1970) classify the geology above both sample areas as undifferentiated older volcanics with pockets of Pennsylvanian limestone (Fig. S6). Further west of CA21P1, in the Egan Range, Scotty's Wash quartzite and Chainman shale of upper Mississippian age outcrop and these materials may be found in the pluvial lake reworked alluvial fans near the sample areas (Fig. S6).

175 2.3 Lake Valley

In Lake Valley, the highest observed beach ridge, superimposed on a Quaternary alluvial fan and bisected by HWY 93, was sampled on the west side of the basin at 1826.82 m asl (Table 1; Fig. S4). The pluvial lake highstand averages around 1826 m asl. Soils in the area are dominated by aridisols with gravelly ashy loam and sandy loam A horizons overlying B horizons of a similar texture (National Cooperative Soil Survey, USA, 2025).
180 According to mapping by Tschaz and Pampayan (1961 and 1970), the closest bedrock source to the LK21P1 sample site is middle to upper Devonian Guilmette formation Dolomite and Devils Gate Limestones exposed on the north side of Dutch John

Mountain (northern margin of the Fairview Range) west of the sample area (Fig. S7). Upslope of this formation is an unconformity topped by upper Devonian Pilot Shale that has another unconformity topped by lower Mississippian Bristol Pass limestones and others. To the northwest of the LK21P1, volcanic rocks (older, undifferentiated) and upper Mississippian
185 Scotty Wash quartzite, sandstone, and shale outcrops amongst the Quaternary age alluvium emanating from the Schell Creek Mountain Range (Fig. S7).

3 Methods

3.1 Sampling in the field

190 Sampling at all three sites was conducted at night with the aid of dim red (>660 nm) headlamps. Sample location coordinates were recorded using a survey grade Trimble Geo7X Handheld GNSS System with external antenna. The SW facing natural exposure at Coal Valley was sampled near midnight using a pitched tarp to block light from a partial moon that set at 10:45 pm. The moon shade was also used in the secluded Cave Valley. The sample location in Lake Valley was near a highway and so an open bottomed tent covered with two layers of 6 mil black plastic was used to prevent light exposure.

195 At Coal Valley, the surface of the natural exposure in the drainage was cleaned, photographed and the sedimentary characteristics recorded. Then at night, the exposure was dug back at least 5 cm to expose rocks that were unlikely to have seen daylight since burial, and 30 of the largest observed gravel clasts were collected at measured depths of approximately ~1 m below the top surface of the ridge. These clasts ranged in thickness (i.e., the shortest dimension that was cored in the laboratory) from 18.5 mm to 41.9 mm and were all composed of limestone. A bulk sample (~300 g) of the gravel and matrix was also collected for water content and dosimetry measurements.

200 At Cave Valley, 20 of the largest observed gravel clasts were collected between 38 and 44 cm from the ground surface in an excavated pit at site CA21P1. The clast lithologies were dominated by intermediate volcanic rock and ranged in thickness between ~15 and ~30 mm. A bulk sample (~300 g) of the gravel and matrix was also collected for water content and dosimetry measurements. At site CA21P2, ~100 of the largest observed gravel clasts were collected between 33 and 64 cm depth below the surface, along with another bulk gravel matrix sample for water content and dosimetry. The majority of clasts collected at
205 CA21P2 were a similar size to those at CA21P1 but consisted of darker more mafic or basaltic varieties of volcanic rock.

At Lake Valley, ~100 of the largest observed gravel clasts were collected from ~0.5 m depth below the surface, along with a ~300 g bulk dosimetry sample. These clasts were all composed of limestone and were a similar size range to those collected at Coal Valley. At Cave and Lake Valley sites, the orientation of most rocks were recorded by labelling their top sides with masking tape and marker after wrapping. This was undertaken to allow measurements from the clast upper faces, as these have
210 been shown to record sub-surface bleaching more effectively than the lower face of the clasts (Jenkins et al., 2018). The orientation of some clasts could not be recorded if they fell out of the exposure prior to collection. The sediments in all pits were photographed and their sedimentary characteristics recorded in daylight after sampling was complete.

Given the diversity of rock lithologies present within bedrock surrounding Coal, Cave and Lake valleys, we expected to collect a wider variety of rock types from the sampled beach ridges (e.g., sandstones, siltstones, limestones, intrusive and extrusive igneous rocks). We found, however, that gravels at each beach ridge were dominated by the lithology of the most proximal bedrock outcrop (Figs S5-S7). Thus, our sample processing and dating protocols presented below were developed only for the limestone and volcanic rocks that we collected at Coal and Cave valleys, respectively.

3.2 Sample preparation

Rock sample preparation for limestone and volcanic gravels required different approaches. The volcanic gravel clasts were cored and sliced following the approaches of Jenkins et al. (2018) and Gliganic et al. (2019), however, given that luminescence signals must be measured from quartz or feldspar minerals and not from carbonates, we prepared the limestone rocks in a manner similar to that of Liritzis et al. (2010) (see below). Minerals extracted from both rock types were not further separated to isolate quartz and feldspar (e.g., Aitken, 1998) due to the small quantities of material available.

3.2.1 Limestones

Sample preparation for limestone rocks from Coal and Lake Valley included the following steps:

1. The outer secondary carbonate coatings observed on the surfaces of some limestone rocks were filed away with a file or Stylo-style Dremel tool. These coatings did not cover the entire rock surface but were patchy and thin (generally < 5 mm thick).
2. The outer 1-2 mm of each side of the limestone clast was dissolved in weak (10%) hydrochloric (HCl) acid over several hours while monitoring the change in limestone thickness with repeated caliper measurements.
3. Detrital sediment released from the outer 1-2 mm of dissolved limestone (step 2) was collected, wet sieved to extract available grain size fractions between 63-250 μm , and dried for luminescence measurements.
4. The majority of the remaining limestone was dissolved in high-concentration (~36%) HCl, and the detrital clastic sediment was collected, wet sieved to extract available grain size fractions between 63-250 μm and dried for signal testing and dose recovery tests.
5. The remaining residual limestone rock (~10-20 g) was dried, milled into a fine powder and submitted to ALS Minerals, Reno, NV for U, Th, K and Rb determination using ICP-MS (U, Th, Rb) and ICP-AES (K) (see Section 3.6).

This approach to sample preparation for limestone gravel-sized rocks requires the following assumptions:

1. During beach ridge formation, light penetrated the outer 2 mm or more of the limestone surface to bleach the signals from detrital quartz and feldspar minerals.
2. The entire surface of the limestone rocks in the swash zone of the beach were adequately light exposed prior to burial to bleach near-surface quartz/feldspar detrital grains.

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3. Limestone surfaces experienced limited or no erosion or dissolution after beach ridge formation and prior to sampling.
 4. Micro-beta dosimetry effects that can lead to scatter in measured grain equivalent dose (D_e) and age distributions are minor in the limestone and around it.
 5. Any U-series disequilibrium that may have existed within the limestone during its formation will have corrected itself (i.e., U-238 daughters with half lives up to ~1600 years will have reached a new equilibrium with the parent) since formation of the limestone during the Devonian.
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3.2.2 Volcanics

Volcanic rocks sampled from Cave Valley were prepared using the following approaches. Polym mineral grains (i.e., samples that had undergone no mineral separation) were extracted from volcanic gravel-sized rocks in two ways: by coring and slicing (the traditional method), in addition to removal of the entire outer ~1 mm layer from all sides of each rock. This second approach maximized the amount of material that could be used for D_e measurement:

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1. The outer carbonate coating on the volcanic rocks was removed by setting them in a bath of high-concentration (36%) HCl acid.
2. The rocks were cored using a 10 mm inner-diameter diamond core drill bit. Cores penetrated the entire rock where possible, and where possible, multiple (2-3) cores were extracted from rocks of adequate size. Where the orientation of the rocks were known, rocks were cored on their top surfaces through to their bottom surfaces.
- 260 3. Rock cores were sliced into ~1 mm thick slices using a ~0.3 mm thick wafer blade mounted on a low-speed precision cutting saw. The polym mineral slices were subsequently crushed gently by hand using an agate mortar and pestle and sieved into distinct grain size fractions between 125 and 250 μm for measurement.
4. The outer ~1 mm layer of the remaining rock was removed using diamond bur bits mounted on a variable-speed Dremel tool.
- 265 5. The 32-63 μm polym mineral fraction of the dremeled outer ~1 mm rock layers were extracted using suspension settling and Stokes Law, then dried for measurement.

Our sample preparation protocol for volcanic gravels requires the following assumptions:

1. Minerals extracted by coring and slicing require that at least one side (the top or bottom) of the rock was exposed to sunlight long enough prior to burial to yield accurate ages.
- 270 2. Minerals extracted from the outer ~1 mm layer of all sides of the rock require that all sides of the rock were adequately exposed to sunlight prior to burial.

3.3 Luminescence measurement

In this study polym mineral samples from limestone clasts were measured at the single-grain level, while samples from volcanic clasts were measured using multi-grain aliquots. Multi-grain aliquots of polym mineral fractions were mounted onto 10 mm

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diameter stainless steel discs using silicon oil as an adhesive. Aliquot diameters were 3 mm and their luminescence signals and D_e measurements were measured in one of two Risø TL/OSL-DA-20 readers equipped with a Sr-90/Y-90 beta radiation source. For single-grain measurements, single grains were mounted onto single-grain discs, each containing one hundred 300 μm diameter holes. Single grains were measured in the same Risø readers using IR lasers.

280 Tests were conducted to determine whether or not quartz minerals from the samples yielded a datable luminescence signal (Section 4.2). For these measurements 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 $\sim 80 \text{ mW/cm}^2$ and signals were detected with a bialkali EMI 9235Q photomultiplier tube (PMT) fitted with Hoya U-340 filters that transmit UV light. For feldspar signal measurements from multi-grain aliquots of polymineral sediment, IR stimulation was made with a cluster of Vishay TSFF 5200 IR diodes
285 with peak emission at 870 nm, and maximum power of 115 mW/cm^2 at the sample position. Single-grain feldspar signal measurements were made with an IR laser emitting 830 nm at a maximum power of 150 mW/cm^2 . Corning 7-59 and Schott BG39 filters were used for feldspar signal detection. An additional Schott RG780 filter was mounted in front of the IR laser to reduce background noise during single-grain measurements (Lai et al., 2002).

3.4 Equivalent dose (D_e) determination

290 3.4.1 SAR

The equivalent dose (D_e) was measured using variations of the single aliquot regenerative dose (SAR) protocol (Wallinga et al., 2000; Murray and Wintle, 2000). This protocol measures the sensitivity-corrected natural signal (L_n/T_n) of each grain/aliquot, followed by sensitivity-corrected regenerative dose signals (L_x/T_x) measured after a series of increasing laboratory doses (i.e., the dose response curve). The protocol included the measurement of a repeat-dose point (i.e., one
295 regenerative dose point is measured twice) to measure the recycling ratio, and a zero-dose point (i.e. L_x/T_x is measured after no dose is given) to measure recuperation. Aliquots/grains were rejected from further analysis if the recycling ratio was beyond 10% of unity and if recuperation was greater than 5% of the sensitivity-corrected natural signal. Aliquots/grains were also rejected if their natural test dose signals were less than three times the standard deviation of the background signal.

We applied varying SAR protocols during this study to measure the infrared signal measured at 50 °C (IR_{50} signal) as well as
300 post-infrared infrared (pIRIR) signals measured at a range of temperatures. These protocols are shown in Tables 2 and S2. The IR_{50} protocol includes low-temperature (160 °C) preheats prior to measurement of the regenerative and test doses, as these have been found in the past to reduce or remove recuperation and improve dose recovery test results (e.g., Neudorf et al., 2015). The pIRIR₂₉₀ protocol was adapted from Thiel et al. (2011). Because preheats must be equal to, or higher than pIRIR stimulation temperatures (Murray et al., 2009), this protocol includes a high preheat of 320 °C.

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Table 2. The IR₅₀ and pIRIR₂₉₀ protocols used in this study.

Step	IR ₅₀ (multi-grain)	IR ₅₀ (single-grain)	pIRIR ₂₉₀ (multi-grain)
1	Natural/Regenerative Dose	Natural/Regenerative Dose	Natural/Regenerative Dose
2	Preheat (160°C, 10 s)	Preheat (160°C, 10 s)	Preheat (320°C, 10 s)
3	IR diodes (50°C, 200 s) → L _n , L _x	IR laser (50°C, 2 s) → L _n , L _x	IR diodes (50°C, 100 s)
4	Test dose (~6 Gy)	Test dose (~6 Gy)	IR diodes (290°C, 100 s) → L _n , L _x
5	Preheat (160°C, 10 s)	Preheat (160°C, 10 s)	Test dose (~10 Gy)
6	IR diodes (50°C, 200 s) → T _n , T _x	IR laser (50°C, 2 s) → T _n , T _x	Preheat (320°C, 10 s)
7	IR diodes (180°C, 100 s)	IR diodes (180°C, 100 s)	IR diodes (50°C, 100 s)
8	Return to step 1.	Return to step 1.	IR diodes (290°C, 100 s) → T _n , T _x
9			IR diodes (325°C, 40 s)
10			Return to step 1.

3.4.2 Dose recovery tests

In this study, SAR protocols were tested prior to D_e measurement using dose recovery tests (Aitken, 1994; Roberts et al., 1999). During these tests the sample was bleached using IR or pIRIR stimulation, administered a known laboratory dose to the sample, then measured using the SAR protocol to be tested. The IR₅₀ SAR protocol was tested because, though it can exhibit anomalous fading, it depletes faster during light exposure in nature than other pIRIR signals (Thomsen et al., 2008). pIRIR SAR protocols were also tested, as these exhibit lower fading rates, and may be more appropriate for samples with high anomalous fading rates (Thomsen et al., 2008; Buylaert et al., 2009).

Polymineal grains measured at the single-grain (or micro-hole) level in single-grain discs were first bleached simultaneously for 1000 s using IR diodes, then stimulated individually (in each hole) for 2 s using the IR laser. It was found that this second bleach with the IR laser was necessary as the shadowing effects of the holes in the single-grain discs prevented full depletion of the IR₅₀ signal in all grains by the IR diodes. A similar approach has been adopted for single K-feldspar grains by Feathers et al. (2019). Because the available grain size fraction from the rocks was significantly smaller (63-250 μm) than the width of the single-grain disc holes (300 μm) in the reader, it is likely that several (up to 5) grains within each disc hole contributed to the IRSL signal, and the results should be viewed as small multi-grain aliquot data, or “micro-hole” data as termed by Berger et al. (2013).

For the pIRIR protocols, the pIRIR signal was depleted using a pIRIR L_n measurement (Table 2) in order to target the pIRIR traps and avoid leaving a slowly bleaching part of the signal that can remain if the sample is only exposed to daylight or a solar simulator for a finite period of time (e.g., Li and Li, 2011). A disadvantage of this approach is that the bleaching method (the pIRIR L_n measurement) can lead to sensitivity changes that may lead to inaccurate dose recovery test results. For all SAR protocols tested, the protocol was accepted if the ratio of the measured-to-given dose was within 10% of unity and few aliquots or grains were rejected from analysis due to dim signals, high recuperation or recycling ratios that are not within 10% of unity. However, these results should be viewed with caution until further experiments can be conducted.

330 3.5 Anomalous fading

Fading rates were measured using a SAR procedure modified from Auclair et al. (2003). This procedure entails bleaching the sample, administering a known laboratory dose, preheating the sample to a temperature deemed appropriate by the dose recovery tests, then measuring the sensitivity-corrected signal (L_x/T_x) after a series of delay times ranging from a few minutes to ~10 h.

335 In this study we measured fading rates from several multi-grain aliquots and/or single grains from each sample, where the fading rate from each aliquot/grain is illustrated using a fading plot of L_x/T_x versus delay time in hours (h). Delay time is plotted on a logarithmic scale to account for the fact that feldspar signals fade exponentially. The fading rate of an aliquot/grain is quantified by the slope of the line, termed the g -value, with units of percent per decade, where a decade is a 10-fold increase in delay time (i.e., one increment on a logarithmic scale) (Huntley and Lamothe, 2001). All g -values are normalised to 2 days.

340 3.6 Dosimetry

The dose rates of the sampled clasts and the surrounding bulk matrix samples were determined in two ways: i) using measured concentrations of parent radionuclides Uranium (U), Thorium (Th) as well as Potassium (K) and Rubidium (Rb) using inductively coupled plasma mass spectrometry (ICP-MS) (for U and Th) and inductively coupled plasma atomic emission spectroscopy (ICP-AES) (for K), and ii) using high resolution gamma spectrometry to measure K-40, as well as U-238 and
345 Th-232 radionuclides and their daughter products. This second approach allows us to assess the possibility of secular disequilibrium within the U or Th decay chains that may have led to fluctuations in environmental dose rates through time (Murray et al., 1987; Ivanovich and Harmon, 1992).

Both gravel matrix and gravel rock subsamples from all sites were prepared for ICP-MS and ICP-AES analysis of radionuclide contents by ALS Geochemistry, Reno, NV. These samples were dried and milled to a fine, flour consistency and subsamples
350 used for U, Rb and Th measurement were fused with lithium borate and measured with ICP-MS. K_2O was measured from the bulk sample with ICP-AES and converted to % K.

Gravel matrix samples from sites CV21P1 (Coal Valley) and CA21P1 (Cave Valley) were measured using high resolution gamma spectrometry, as well as one sample containing a mix of subsamples from multiple representative limestone gravel rocks from CV21P1. Gamma spectrometry measurements could not be made for individual limestone gravel rocks, because
355 they did not form large enough samples needed (~50 g) for measurement. Samples were dried, ground to a fine powder consistency, then ashed at 450 °C in a muffle furnace for 24 hours. They were then emplaced in γ Beakers designed by miDose Solutions, Poland to prevent leakage of Rn gas and stored for a minimum of 21 days to allow the sample to reach equilibrium. After storage, samples were placed on top of a broad energy (BE), planar High Purity Germanium (HPGe) detector and the gamma emission was measured for 7 days. The gamma energy spectrum was analysed to calculate the activities of U-238, Ra-
360 226, Th-232, Pb-210, Ra-224, Ac-228 and K-40 in Bq/kg.

Dose rates (Gy/ka) were calculated in the Dose Rate and Age Calculator (DRAC) by Durcan et al. (2015) using the conversion factors of Liritzis et al. (2013) and assumed negligible water contents within the gravels clasts and an average water content of 3.0 ± 1.5 % within gravel matrices. The cosmic dose rate (Gy/ka) was calculated according to Prescott and Hutton (1994) using sample geographic coordinates and depths below the surface. The internal K content of the sediment and rock minerals measured in this study could not be quantified, so grain (or rock mineral) internal dose rates are assumed to be $10 \pm 2\%$ following Smedley et al. (2012). We recognise that this value may be high for our polymineral samples (Maßon et al., 2025), and that future work should investigate the K contents of grains and minerals that contribute significantly to the luminescence signals detected using our detection windows.

4 Results

370 4.1 Beach ridge sedimentology

4.1.1 Coal Valley

Sediments exposed at site CV21P1 reveal two main units including a massive, cohesive blocky silt (Unit 1 – clayey silt) overlain by a weakly bedded gravel with medium-coarse sandy matrix (Unit 2 – sandy beach gravel) (Fig. 3(a), Table 3). Gravel clasts rarely exceed 40 mm along their intermediate (b-) dimension and lithologies are dominated by limestones derived from outcropping Devonian carbonate shelf deposits to the west (Fig. S5). These sediments are interpreted to represent pluvial lake highstand beach gravels overlying fine-grained sediments that were ponded in a lagoon behind the beach ridge shortly before rising waters deposited the gravels over them (Wriston and Adams, 2020) (Fig. 4). In this interpretation, the gravels and underlying lagoonal deposits are contemporaneous (cf. Adams and Wesnousky, 1998). Therefore, the age of radiocarbon dated shell from the lagoonal deposits (15,873-16,281 cal yrs BP) is interpreted to represent the age of the beach gravels that were deposited there during the pluvial Lake Coal highstand.

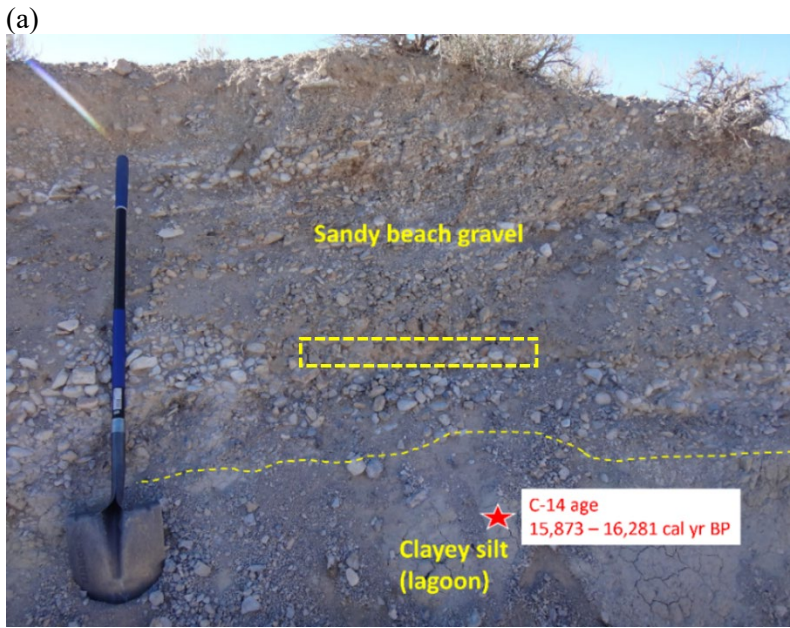
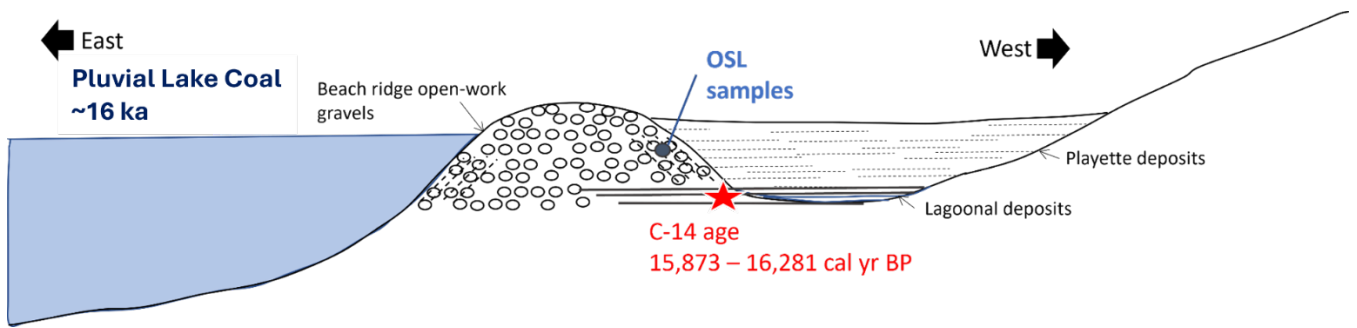


Figure 3. (a) Sampled sedimentary exposure CV21P1 in Coal Valley. The zone of sampling is 1 m below the surface and is indicated by the yellow dashed box. (b) Sampled excavation pit from site CA21P1. The silty sand gravel matrix that forms the soil A horizon is exposed in the trench. Gravel clasts were collected at the base of the trench between 38 and 44 cm depth below surface.



385 Figure 4. Illustration of sampled beach ridge shortly after formation ~16,000 years ago during the Pluvial Lake Coal highstand. Any backwater playette deposits or lagoonal deposits landward of the beach ridge have since eroded away, exposing the beach gravels on the west side of the ridge. Figure not to scale.

Table 3. Sedimentary characteristics of beach ridges at sites CV21P1 and CA21P1.

Site	Depth below surface (cm)	Sedimentary unit	Description	Interpretation	Samples collected
CV21P1 (Coal Valley)	0-120	Unit 2	Weakly bedded gravel with medium-coarse sandy matrix capped by very thin A horizon. Root penetration most prevalent in upper 7 cm. Gravel clasts dominated by rounded-subrounded weakly imbricated limestone with apparent dip N. Gravel shapes are tabular, equant and oblong with b-axes up to ~4 cm. Rare orange andesite gravels present. Moderately sharp lower contact that dips to the N.	Sedimentation in a near-shore, high-energy swash zone of a pluvial lake.	30 gravel-sized clasts from ~1 m below the surface for luminescence testing. One ~300 g bulk sample for dosimetry.
	120- base of exposure	Unit 1	Massive, cohesive carbonaceous blocky silt.	Back-beach lagoonal sediments deposited under low-energy conditions.	Freshwater shell with radiocarbon age of 15,873-16,281 cal yrs BP
CA21P1 (Cave Valley)	44	Unit 1	Matrix-supported silty sand gravel dominated by intermediate volcanic subangular to subrounded gravel clasts with b-axes generally ranging from 2 to 5 cm.	Sedimentation in a near-shore, high energy swash zone of a pluvial lake followed by soil development and accretion following lake recession.	20 gravel-sized clasts from 38-44 cm depth for luminescence testing. One ~300 g bulk sample for dosimetry.

390

4.1.2 Cave Valley

Sediments exposed in excavation pits at sites CA21P1 and CA21P2 include gravel with silty sand matrix. Sedimentary structures were not visible due to the loose silt that quickly covers exposed sections. Both beach ridge gravels are dominated by volcanic rock lithologies derived from outcropping Miocene and Oligocene flows and tuffaceous sedimentary rock to the west and south, with more felsic varieties appearing more common at CA21P1 (Figs 3(b), S6, Table 3). Gravel clasts were subangular to subrounded with their longest dimensions commonly under 10 cm.

4.1.3 Lake Valley

Sediments exposed in the excavation pit of site LK21P1 include gravel with silty sand matrix. Limestone dominates the rock lithologies, which is derived from neighbouring Devonian lithified carbonate shelf deposits (Fig. S7). Gravel clast sizes were similar to those found in Cave Valley. Samples collected from Lake Valley were not datable (see below), so further discussion of the site sedimentology is not reported here.

4.2 Luminescence signals from limestone and volcanic rocks

Luminescence signals were measured from polymineral samples extracted from a subset of limestones from Coal Valley and Lake Valley as well as volcanic rocks from Cave Valley to determine their brightness and suitability for dating. Measurements were made in both continuous-wave (CW-OSL) and linear-modulation (LM-OSL) modes (details in Supplementary Material, Section 3). Because none of the minerals extracted from five Lake Valley limestone gravels had detectable luminescence signals, we focused our efforts on samples from Coal and Cave valleys. Neither limestone samples nor volcanic rocks contained quartz with a datable fast component, but some rocks from both lithologies exhibited IR signals. Volcanic rocks with the brightest IR signals were typically of the felsic (andesitic or rhyolitic) varieties common at site CA21P1, whereas basalts, like those collected from site CA21P2, had no signal.

4.3 Limestone gravels from Coal Valley

4.3.1 Dose recovery test results

Twenty-one limestone rocks from site CV21P1 were processed for IR signal testing and D_e measurement (Table 4). Nineteen of these were clasts excavated within the beach ridge at ~1 m depth, and two were cobbles collected from the ground surface (i.e., modern samples). Minerals extracted from six of these gravels passed dose recovery tests either at the multi-grain or single-grain level, but only three of these (rocks #2, 10, and 18) had sufficient material left for D_e measurement and enough aliquots/grains that passed SAR rejection criteria. Minerals extracted from the two cobbles collected from the ground surface (rocks #2M and 3M, Table 4) did not have a detectable IR or OSL signal.

Table 4. Dose recovery (DRT) and fading test results for polymineral sand grains extracted from limestone rocks from Coal Valley site CV21P1. MG refers to multi-grain analysis and SG refers to single-grain analysis. The IR₅₀ SAR protocol (Table 2) was applied.

Sample	Thickness ¹ (mm) [mass (g)]	Grain size (µm)	MG DRT result ²	SG DRT result ²	MG fading rate (%/decade)	SG fading rate (%/decade)	Notes
Ancient samples							
Rock 1	18.5 (0.043)	90-180	(n=0/4)	0.97 ± 0.07 (n=10/300)	-	-	Dim, not enough grains pass rejection criteria
Rock 2	27.8 (0.229)	63-90	0.98 ± 0.02 (n=4/4)	0.99 ± 0.02 (n=285/800)	3.2 ± 0.2 (n=4)	2.6 ± 0.5 (n=65)	Datable. Lots of material available
Rock 3	26.1 (0.771)	90-125	-	-	-	-	Signal too dim.
Rock 5	32.5 (0.048)	125-180	0.92 ± 0.04 (n=3)	-	-	-	No accepted grains at SG level. Insufficient material
Rock 7	41.9 (0.081)	unsieved	-	-	-	-	No signal
Rock 8	27.8 (0.007)	-	-	-	-	-	Not enough sediment to measure
Rock 9	21.5 (0.171)	125-180	1.00 ± 0.04 (n=4/4)	0.97 ± 0.04 (n=7/500)	9.9 ± 1.0 (n=4)	-	Very low # of accepted grains. Fading rate too high
Rock 10	32.0 (0.800)	125-180	-	0.91 ± 0.03 (n=50/1000)	-	2.6 ± 1.7 (n=28)	Datable. Adequate quantity of material
Rock 11	21.2 (0.890)	125-180	-(n=0/4)	0.99 ± 0.04 (n=9/300)	-	-	Low number of accepted grains. Limited quantity of material.
Rock 13	34.0 (0.163)	unsieved	-	-	-	-	No signal
Rock 14	20.8 (0.007)	unsieved	-	-	-	-	No signal
Rock 16	18.5 (0.030)	<90	-	-	-	-	Dim signal, no grains pass rejection criteria
Rock 17	20.4 (0.633)	90-125	-	-	-	-	No signal
Rock 18	21.9 (0.604)	180-250	-	0.96 ± 0.03 (n=53/1300)	-	2.0 ± 0.7 (n=27)	Datable. Lots of material.
Rock 19	20.8 (0.031)	125-180	-	-	-	-	Not enough grains pass rejection criteria
Rock 20	21.9 (0.392)	unsieved	-	-	-	-	No signal
Rock 21	35.9 (0.012)	180-250	-	-	-	-	Too few grains with a signal
Rock 22	18.5 (0.054)	125-180	-	-	-	-	Too few grains with a signal
Rock 23	34 (1.132)	unsieved	-	-	-	-	No signal
Modern samples							
Rock 2M	46.0 (0.831)	unsieved	-	-	-	-	No signal
Rock 3M	83.3 (0.985)	unsieved	-	-	-	-	No signal

¹Thickness refers to the shortest rock dimension and mass is the mass of polymineral grains extracted from the outer 1-2 mm of the rock. ²n = # grains or multi-grain aliquots that pass rejection criteria / total number of grains/aliquots measured.

The dose recovery test results for Rocks 2 (63-90 μm), 10 (125-180 μm) and 18 (180-250 μm) are plotted in Figure S9, where a laboratory dose of ~ 21 Gy was recovered. Measured sigma-b values were 22%, 18% and 17% for Rocks 2, 10 and 18, respectively, where sigma-b refers to measurement uncertainties that are attributed to instrument reproducibility and grain-to-grain variations in signal properties (Galbraith and Roberts, 2012). Measured-to-given dose ratios were 0.99 ± 0.02 , 0.91 ± 0.03 and 0.96 ± 0.03 for Rocks 2, 10 and 18, respectively suggesting that the IR₅₀ SAR protocol is suitable for these samples, and thus these samples were chosen for dating. The 90-125 μm grain size fraction from Rock 2 was also dated in the following sections, but there was not enough material to perform a dose recovery test prior to dating. Thus, a sigma-b value of 22% has been assumed for this grain size fraction.

4.3.2 Fading test results

Fading measurements for Rocks 2, 10 and 18 were made on the same grains (or small multi-grain aliquots) used for D_e determination. These included single-grain (i.e., “micro-hole”) measurements of the 63-90 μm fraction from Rock 2 using single-grain discs, and single-grain measurements of the 125-180 μm fraction from Rock 10 and the 180-250 μm fraction from Rock 18. This allows each grain (or small multi-grain aliquot in the case of the 63-90 μm fraction) to be corrected for its own fading rate, which can vary significantly from grain to grain or aliquot to aliquot. An average fading rate was also measured from four 2 mm diameter multi-grain aliquots from the 90-125 μm grain size fraction from Rock 2. Representative single-grain/small aliquot IR₅₀ signals and fading plots are shown in Figure S12. The fading rate, or *g*-value, of each grain/small aliquot was measured using multiple prompt and delayed signal measurements. The weighted mean *g*-value for Rock 2 (285 micro-hole measurements of the 63-90 μm fraction) is 2.55 ± 0.52 %/decade, which is slightly lower but within 2 standard deviations of that measured from the 90-125 μm grain size fraction from the same rock (3.19 ± 0.15 %/decade). The weighted mean *g*-value of all single-grain measurements made for Rocks 10 and 18 is 2.65 ± 1.71 and 1.99 ± 0.79 %/decade, respectively.

4.3.3 Radionuclide concentrations

ICP-MS/AES measurements of U, Th, K and Rb were conducted on crushed and milled subsamples of Rocks 2, 10 and 18 as well as a bulk sample of the gravel matrix (Table S8). All radionuclide contents for the limestone gravels are low relative to values typically obtained for bulk non-carbonaceous sediment, and this is expected given the relatively low concentration of silicate minerals within the limestone. The gravel matrix yielded values that are slightly above those of the rocks, reflecting a higher silicate mineral content.

HPGe measurements of the limestone yielded results that are in range of those measured using ICP-MS/AES, while measurements made from the gravel matrix deviate somewhat from the ICP-MS/AES results. To check the calibration of our HPGe detector, measurements were made on a standard prepared by Murray et al. (2015) that was measured by 23 different laboratories (Table S9, Fig. S16 A and B). Our results are within 20% of those previously published.

Radionuclide activity ratios for the U and Th series in the limestone and gravel matrix samples are reported in Table S10 and plotted in Figures S16 (C-F) and S17. The gravel matrix sample from CV21P1 shows elevated Pb-210 activity relative to U-238 and Ra-226, while the Ra-226/U-238 ratio approximates unity and the Ra-224/Ac-228 ratio falls within 20% of unity. Given that the gravel matrix sample was collected from loose, porous medium-coarse sandy gravels ~1 m below the surface, we interpret the elevated Pb-210 values to reflect the translocation of atmospheric Pb-210 that has leached down to the level of sampling during rain events (Murray, 1996). The Ra-226/U-238 and Ra-224/Ac-228 ratios from the gravel matrix otherwise suggest that the deposit approximates secular equilibrium. Radionuclide activity ratios calculated from the limestone rocks are well within 20% of unity, suggesting that our assumption that any U-disequilibrium that may have existed within the limestone during its formation will have corrected itself since Devonian times (Section 3.2.1).

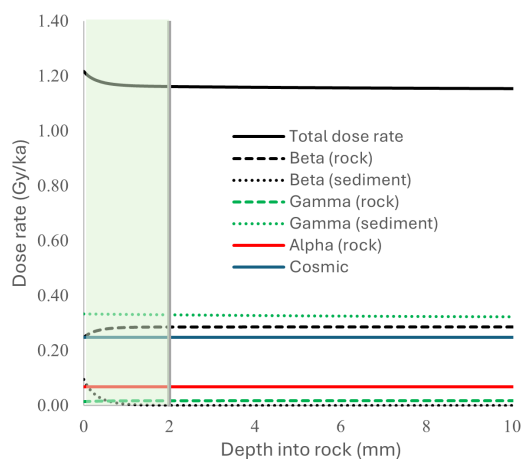
4.3.4 Dose rate modelling

Alpha, beta and gamma dose rates have been calculated for all limestone rocks as well as the gravel matrix from site CV21P1 (Table S13). Dose rates were calculated from both the ICP-MS/AES and HPGe measured radionuclide concentrations. As expected for carbonate-rich materials, all dose rates are low, ranging from ~1.2 Gy/ka to ~1.9 Gy/ka. HPGe measurements from limestone rocks yielded alpha, beta and total dose rates within the range of results obtained from ICP-MS/AES analysis. HPGe measurements of the gravel matrix underestimate the ICP-MS/AES beta and gamma results leading to a ~24% reduction in the total dose rate.

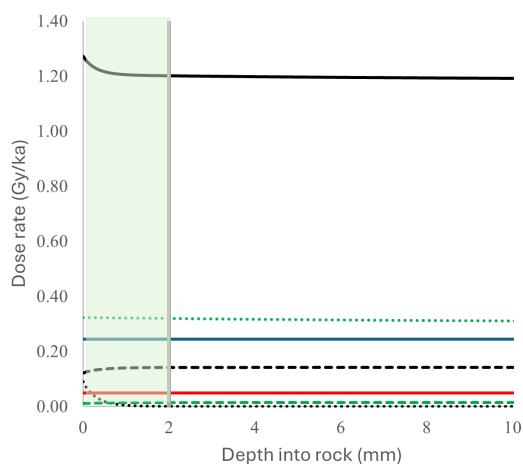
Dose rates were calculated for the outermost ~2 mm of each limestone taking into account the measured alpha, beta and gamma dose rates from each rock, beta and gamma dose rates from the surrounding bulk gravel matrix as well as cosmic rays from outer space. Dose rates for each rock were modelled using the approach of Riedesel and Autzen (2020), which incorporates experimentally derived attenuation factors for granite, assuming that the rock is shaped like a sphere. This approach yields total dose rates that are lower than those calculated using earlier approaches that assume laterally infinite beta and gamma dose rates for both the rock and surrounding sediment (e.g., Jenkins et al., 2018) (see Section 8 of Supplementary Material for a comparison). To check that the beta and gamma attenuation factors of Riedesel and Autzen (2020) are appropriate for our limestone samples, the elemental concentrations of 4 representative limestone subsamples were measured by XRF and used to calculate attenuation factors using Geant4 simulations. These simulations generated attenuation factors that matched those published by Riedesel and Autzen (2020) for granite (Autzen pers. comm.), and so we apply them to our dose rate models in our study.

Modelled alpha, beta and gamma dose rates with depth into Rocks 2, 10 and 18 are shown in Figure 5. These are based on radionuclide concentrations measured by ICP-MS/AES (Section 4.3.3). Similar dose rate with depth models were also calculated using the radionuclide contents determined using HPGe. The total dose rate used for calculating the age of detrital mineral grains within the outer 2 mm layer of each limestone rock is an average value highlighted in green in Figure 5.

(a) Rock 2



(b) Rock 10



(c) Rock 18

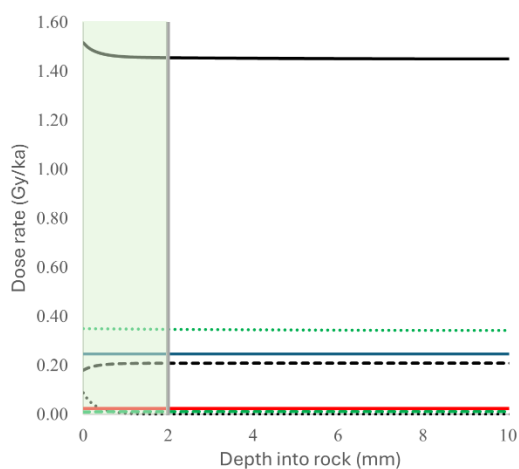


Figure 5. Modelled dose rates for limestone rocks from site CV21P1 following the method of Riedesel and Autzen (2020) and radionuclide concentration data obtained from ICP-MS/AES. The average dose rate for the outer 2 mm of the rocks (green shading) was used for age calculation. The total dose rate includes an internal dose rate component due to an assumed internal K content of $10 \pm 2\%$ after Smedley et al. (2012) for measured feldspar grains.

485

4.3.5 Limestone luminescence age distributions

The D_e and luminescence ages were measured from polymineral grains from Rocks 2, 10 and 18; these rocks passed dose recovery tests (Section 4.3.1). The D_e was measured from two grain sizes from Rock 2 (63-90 μm and 90-125 μm fractions), the 125-180 μm fraction from Rock 10, and the 180-250 μm fraction from Rock 18. D_e was measured using the IR₅₀ SAR protocol in Table 2 for single-grains, and each grain (or small multi-grain aliquot) was corrected for its own fading rate using the correction model of Huntley and Lamothe (2001) for all samples except for the 90-125 μm fraction from Rock 2. All aliquots from the 90-125 μm fraction of Rock 2 were corrected for fading using an average fading rate measured from medium

490

sized multi-grain aliquots (Section 4.3.2). As Figure S13 shows, single-grain fading corrections have the effect of magnifying
495 single-grain age errors for older grains within the distribution, and this is attributed to the limited precision with which we can
measure single-grain fading rates.

The fading-corrected aliquot age distributions are shown in Figure 6 where the cumulative distribution plot is superimposed
on a kernel density estimate (KDE) curve. Overdispersion values (OD) for the fading-corrected data are reported in Table 5.
These have been calculated using the central dose model of Galbraith et al. (1999) after taking into account the measured
500 sigma-b value in dose recovery tests (Section 4.3.1) and record the level of spread in the data that can be attributed to
environmental factors during burial, such as incomplete re-setting of grain signals by sunlight or heterogeneities in the dose
rate field that lead to grain-to-grain variations in acquired dose (Galbraith and Roberts, 2012).

Given that most rock dating studies derive ages from rock (primarily granite, sandstones, quartzites, and volcanics) slices,
rather than single-grains or small aliquots, the relationship between single-grain/aliquot age distribution shapes, OD, and
505 depositional process for rocks is not known and has not been examined in the same way these relationships have been examined
for traditional sediment dating studies (e.g., King et al., 2014). We provide preliminary interpretations of these data that should
be solidified by future testing. The OD of the 90-125 μm fraction of Rock 2 is 30% and it shows a symmetrical KDE curve,
which may indicate that most grains have been sufficiently bleached prior to burial (cf. Arnold et al., 2009, table 4 for quartz).
The OD of all other samples are much higher, ranging from 66% to 99%, and may record incomplete bleaching as well as
510 scatter that is the result of micro-beta dosimetry effects. KDE curves for the fading-corrected data are nearly symmetrical for
Rock 10, possibly indicating that most scatter is due to micro-beta dosimetry effects (Maya et al., 2006), while those of Rocks
2 and 18 show a slight positive skew that may record micro-beta dosimetry effects as well as incomplete bleaching of grains.

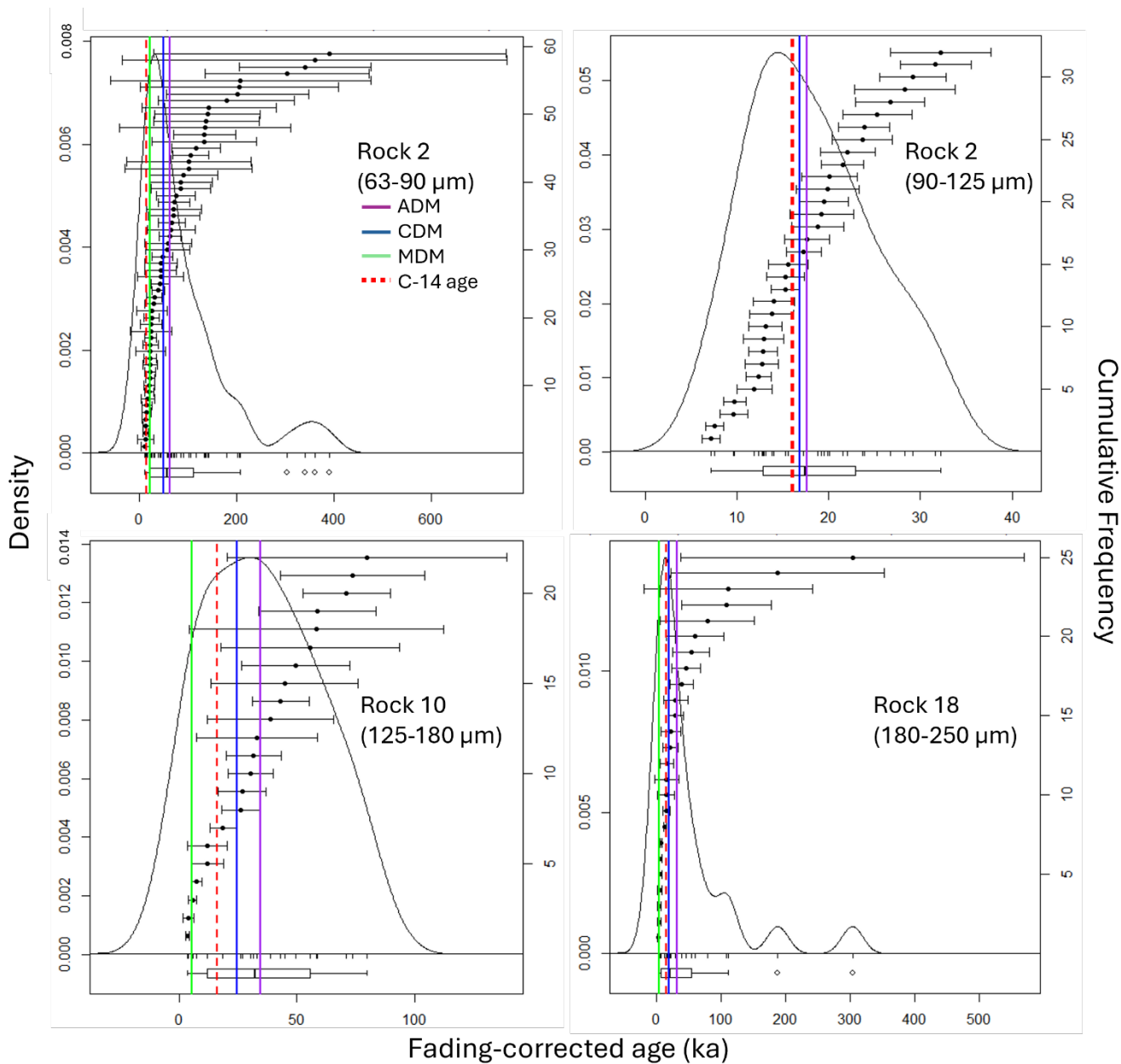
4.3.6 Age models

Statistical models have been developed for calculating a sample D_e value from a distribution of single-grain D_e values (e.g.,
515 Galbraith et al., 1999; Galbraith and Roberts, 2012; Guérin et al., 2017). These models are based on assumptions regarding the
depositional history and composition of the sample analysed. Single-grain ages in this study were calculated using the
minimum dose (MDM) and central dose (CDM) models of Galbraith et al. (1999) as well as the more recently developed
average dose (ADM) model of Guérin et al. (2017). A description of the assumptions behind each model are outlined in the
Supplementary Material, Section 9.

520 4.3.7 Age results

All calculated ages are summarized in Table 5 and plotted in Figures 6 and 7 alongside the radiocarbon age for the freshwater
shell collected from the lagoonal silts (Unit 1). The radiocarbon age plots closest to the CDM ages for limestone Rocks 2 (90-
125 μm , ICP-MS/AES) and 18, overlapping at 1 σ , and agrees with CDM ages calculated for Rock 10 at 2 σ . At 2 σ the
radiocarbon age also overlaps with MDM ages for Rock 2 (63-90 μm), as well as ADM ages for Rock 2 (90-125 μm , ICP-
525 MS/AES) and Rock 18 (Table 5).

Figure 7 shows the relationship between ages determined using ICP-MS/AES dose rates and those determined using HPGe dose rates. These results must be viewed with caution as the HPGe measurements could not be made from each individual limestone sample, but rather were made from one milled sample that combined limestone pieces from multiple rocks from the site. The method of dose rate determination impacted the oldest ages of those compared, where the ICP-MS/AES method resulted in slightly younger age estimates relative to the HPGe method, however ages derived from both dose rate measurement methods agree within 1 σ .



535 **Figure 6.** CDM, MDM and ADM model results superimposed on the age distribution KDE plot. The radiocarbon age for site CV21P1 is indicated as a dashed red line. Ages have been calculated using dose rates determined through ICP-MS/AES. *Single-grain fading-corrections have been applied to all datasets except for the 90-125 μm fraction from Rock 2, where all aliquots have been corrected using an average g -value obtained from multi-grain aliquot fading measurements (Section 4.3.2).

Table 5. Calculated ages (at 1 σ) for limestone gravels from site CV21P1 in Coal Valley. The calibrated 2 σ radiocarbon age obtained from lagoonal deposits underlying the gravel gravels is 15,873 – 16,281 cal yr BP. Luminescence age values that agree with the radiocarbon age within 1 σ are highlighted in bold; those ages agreeing with the radiocarbon age at 2 σ are bold and italicized.

Limestone #	Grain size (μm)	Type of D _e measurement ¹	Type of g-value measurement ¹	Dose rate (Gy/ka)	OD (%)	Modeled age ² (ka)		
						ADM age	CDM age	MDM age
Dose rates determined through ICP-MS & ICP-AES								
Rock 2	90-125	SG	MG	1.24 ± 0.09	30 ± 6	17.6 ± 1.05	16.8 ± 1.2	
Rock 2	63-90	SG	SG	1.17 ± 0.07	66 ± 12	63.9 ± 10.6	51.4 ± 6.4	23.5 ± 5.8
Rock 10	125-180	SG	SG	1.21 ± 0.12	83 ± 17	34.4 ± 5.8	24.4 ± 5.1	5.4 ± 1.6
Rock 18	180-250	SG	SG	1.47 ± 0.17	99 ± 19	32.7 ± 11.4	20.0 ± 4.7	4.8 ± 1.6
Dose rates determined through HPGe gamma spectrometry								
Rock 2	90-125	SG	MG	1.13 ± 0.09	30 ± 6	19.4 ± 1.3	18.6 ± 1.3	
Rock 2	63-90	SG	SG	1.07 ± 0.07	71 ± 12	73.0 ± 13.8	56.6 ± 7.3	26.4 ± 6.0
Rock 10	125-180	SG	SG	1.22 ± 0.12	81 ± 18	33.5 ± 5.1	24.1 ± 5.1	5.7 ± 1.7
Rock 18	180-250	SG	SG	1.40 ± 0.17	94 ± 18	30.6 ± 8.8	19.7 ± 4.4	5.3 ± 1.6

540 ¹ SG = single-grain, MG = multi-grain aliquot. Because the available grain size fraction from the rocks was significantly smaller (63-250 μm) than the width of the single-grain disc holes (300 μm), it is likely that more than one grain within each hole contributed to the IRSL signal, and the results should be viewed as small multi-grain aliquot data, or “micro-hole” data as termed by Berger et al. (2013).

545 ² Sigma-b (σ_b) values (as defined by Galbraith and Roberts, 2012) applied during age modelling were estimated for all samples using dose recovery tests. These are 0.22 for Rock 2 (90-125 μm fraction), 0.22 for Rock 2 (63-90 μm fraction), 0.18 for Rock 10 and 0.17 for Rock 18.

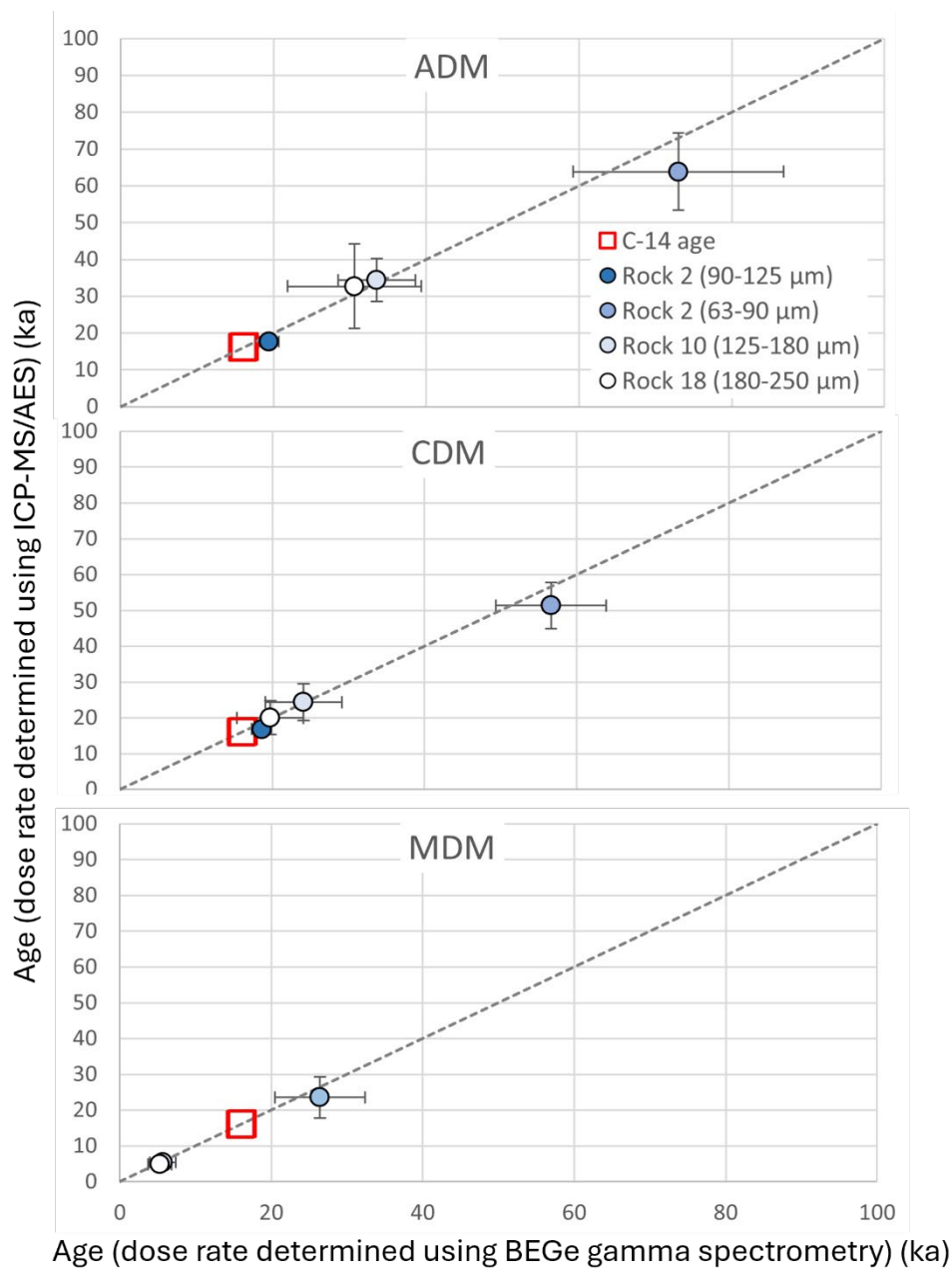


Figure 7. CDM, MDM and ADM ages plotted with the radiocarbon age from Unit 1 that are all from the same Coal Valley profile CV21P1. Ages determined using ICP-MS/AES dose rates are plotted against those determined using HPGe dose rates using a BE Germanium detector (BEGe). The radiocarbon age limits are delineated in red and represent 1σ . Luminescence age error bars are plotted at 1σ .

550

4.4 Volcanic gravels from Cave Valley

4.4.1 Dose recovery test results

Preliminary dose recovery tests were conducted on 3 mm diameter multi-grain aliquots from all volcanic rocks from Cave Valley that exhibited an IR signal. These were all collected from site CA21P1. The rocks were ground and sieved into 32-63 μm grain size fractions prior to measurement. Each aliquot was bleached using a pIRIR L_n measurement (Table 2), then administered a ~ 40 Gy beta dose prior to measurement using SAR. The pIRIR protocols tested included the 180 $^\circ\text{C}$, 225 $^\circ\text{C}$ and 290 $^\circ\text{C}$ protocols (Tables 2, S2), and results obtained from both the IR₅₀ and pIRIR signals from each protocol were plotted (three aliquots per rock) (Fig. S10) and tabulated (Table S6). Despite some outlying values of the measured-to-given dose, most aliquots passed the dose recovery test suggesting that, after signal sensitisation during the L_n measurement, feldspars in these rocks were suitable for SAR.

After assessment of volcanic rock fading rates (Section 4.4.2) and rock characteristics (i.e., the composition of Rock 14, below), additional dose recovery tests were conducted on Rocks 4, 7, 11, 12, 13 and 18 using only the pIRIR₂₉₀ protocol, as this was the only protocol to yield acceptable fading rates (see Section 4.4.2). These tests were conducted on the ground and suspension-settled 32-63 μm grain size fraction (24 aliquots per sample) and were used to estimate a sigma-b value. IR₅₀ and pIRIR₂₉₀ signals from all rocks passed dose recovery tests, yielding measured-to-give dose ratios within 10% of unity and sigma-b values equal to 12 ± 4 % or less (Fig. S11, Table S6). Cutting and coring of Rock 14 revealed that most of this rock was composed of cryptocrystalline quartz (chert). This chert does not have a luminescence signal, so Rock 14 was not processed further.

4.4.2 Fading test results

Measurements of anomalous fading were conducted on the 32-63 μm fractions tested above. As expected, fading rates were the highest for the IR₅₀ signal (up to ~ 40 %/decade), and generally decreased with increasing pIRIR signal temperature (Fig. S15). The pIRIR₂₉₀ signal yielded an average g -value of 3.3 ± 0.7 %/decade, with individual aliquot fading rates varying from 0 to ~ 12 %/decade, while the pIRIR₁₈₀ and IR₅₀ signals yielded average g -values of 10.3 ± 0.9 and 23.1 ± 1.8 %/decade, respectively. We chose the highest temperature pIRIR protocol tested (pIRIR₂₉₀) to minimise fading as much as possible, and thus additional fading tests using the pIRIR₂₂₅ protocol were not pursued.

4.4.3 Radionuclide concentrations

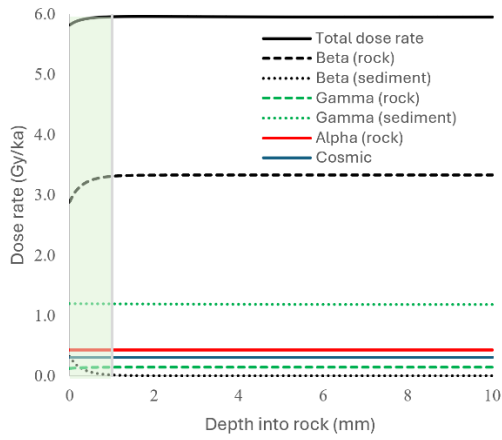
ICP-MS/AES and HPGe measurements of volcanic Rocks 4, 7, 11, 12, 13 and 18 are shown in Table S11. As expected, radionuclide concentrations are much higher than those observed in limestone (Section 4.3.3). The HPGe results in Table S11 agree with the ICP-MS/AES results within 1 σ for U and K, and within 2 σ for Th. Radionuclide activity ratios for U in the gravel matrix collected from site CA21P1 show elevated levels of Pb-210 relative to U-238 that is likely atmospheric and

sourced from recent rain events (Table S12, Fig. S18) (Murray, 1996). All other activity ratios fall within 20% of unity, suggesting that disequilibrium in the U and Th decay chains is limited or negligible.

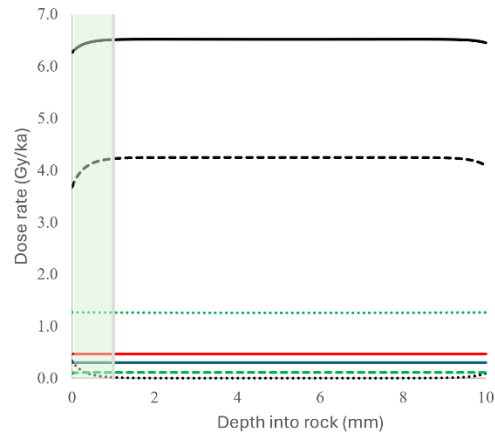
4.4.4 Dose rate modelling

585 The modelled dose rates for the dated volcanic rocks from Cave Valley are shown in Figure 8, and calculated dose rates for both rocks and the gravel matrix are tabulated in Table S14. Calculated dose rates for the gravel matrix from site CA21P1 are high, with a total dose rate equal to ~ 5 Gy/ka (Table S14). Dose rates calculated for all volcanic rocks using ICP-MS/AES radionuclide concentrations are also high, ranging from ~ 6.2 to ~ 7.4 Gy/ka (Table S14).

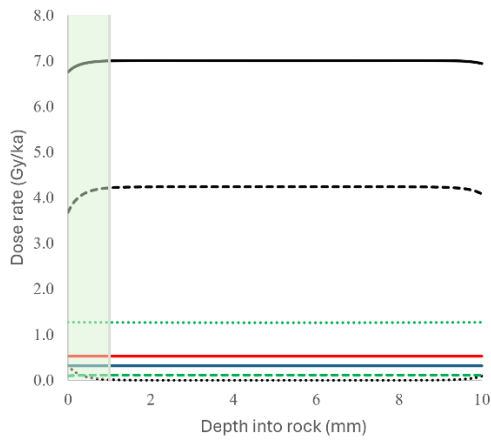
(a) Rock 4



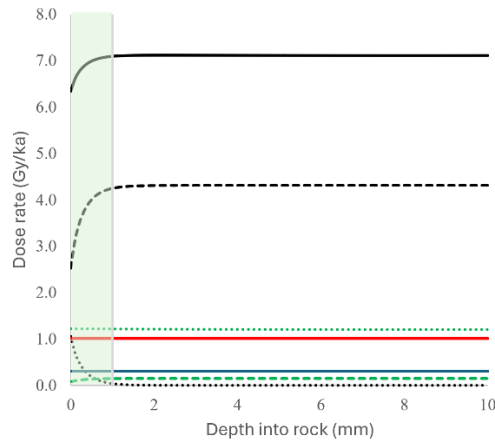
(b) Rock 7



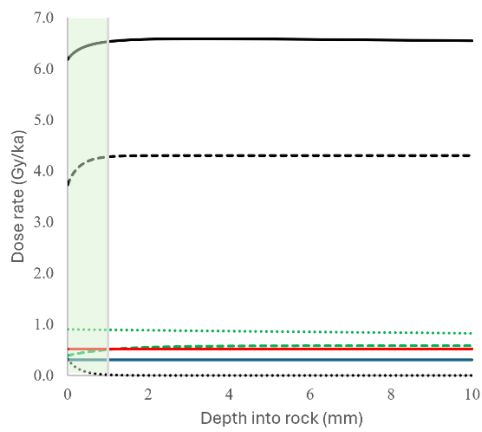
(c) Rock 11



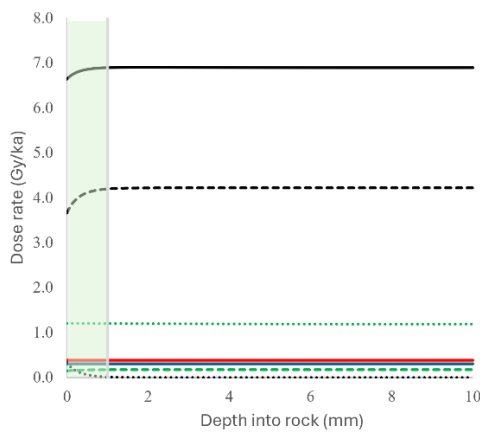
(d) Rock 12



(e) Rock 13



(f) Rock 18



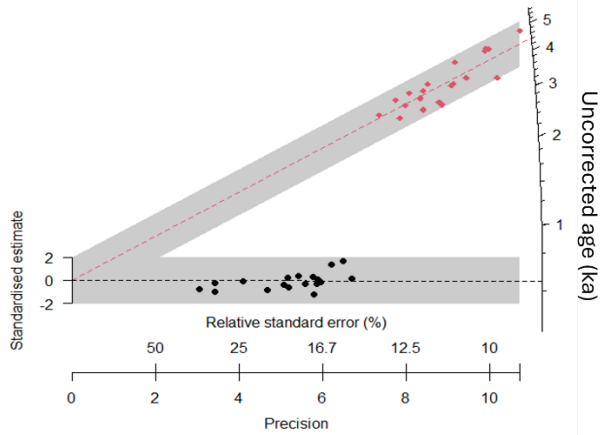
590 **Figure 8. Modelled dose rate with depth into gravel surfaces for rocks from CA21P1 in Cave Valley. Models are based on ICP-MS/AES measurements and the grain size measured from crushed rock slices. The average dose rate for the outer-most slice of the rocks was calculated using the beta, gamma and alpha dose rates within the shaded green region.**

4.4.5 Rock surface luminescence age distributions

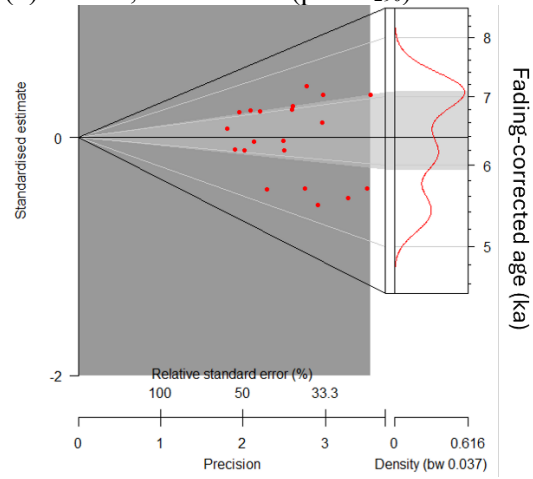
595 The outer ~1 mm layer (dremeled 32-63 μm fraction) of Rocks 4, 7, 11, 12, 13 and 18 was measured for D_e using the pIRIR₂₉₀ protocol for multi-grain aliquots in Table 2. Measurements were made on 3 mm diameter multi-grain aliquots, each containing ~2600 grains. Aliquot D_e values (not corrected for anomalous fading) were divided by the average calculated dose rate within 1 mm of the gravel surface to obtain ages for both the IR₅₀ and pIRIR₂₉₀ signals. The pIRIR₂₉₀ ages were corrected for fading using the model of Huntley and Lamothe (2001) using g -values calculated from four multi-grain aliquot fading measurements from each rock. (Table S7).

600 As expected, IR₅₀ uncorrected ages are significantly younger than pIRIR₂₉₀ uncorrected ages (Figs 9 and 10) and this is attributed to the high rate of fading of the IR₅₀ signal as well as the lower bleaching rate of the pIRIR₂₉₀ signal. Fading-corrected aliquot pIRIR₂₉₀ age distributions for the dremeled outer rock surfaces are plotted in Figures 9 and 10. Weighted mean surface ages for each rock were calculated using CDM and are shown in Figures 9 and 10 as well as in Table 6.

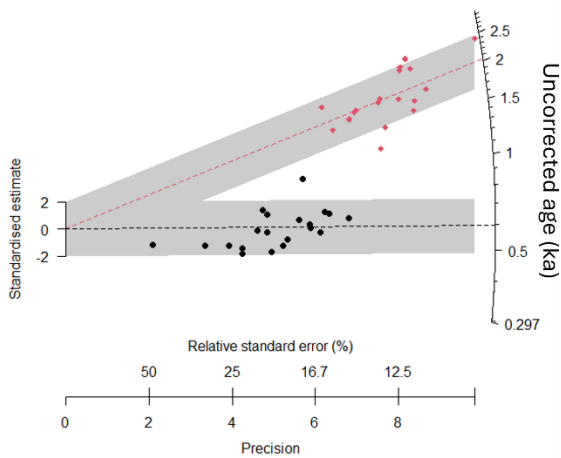
(a) Rock 4
 0.65 ± 0.03 ka (IR₅₀), 4.4 ± 0.1 ka (pIRIR₂₉₀)



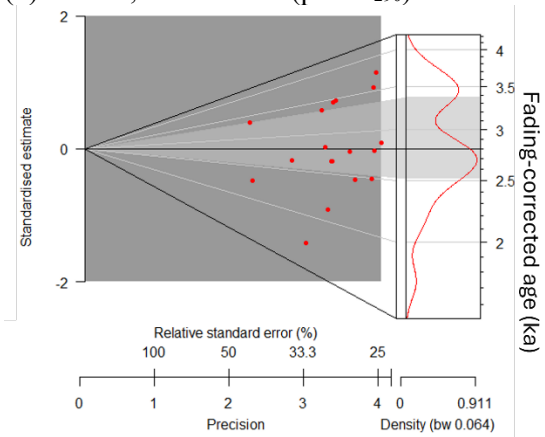
(b) Rock 4, 6.4 ± 3.4 ka (pIRIR₂₉₀)



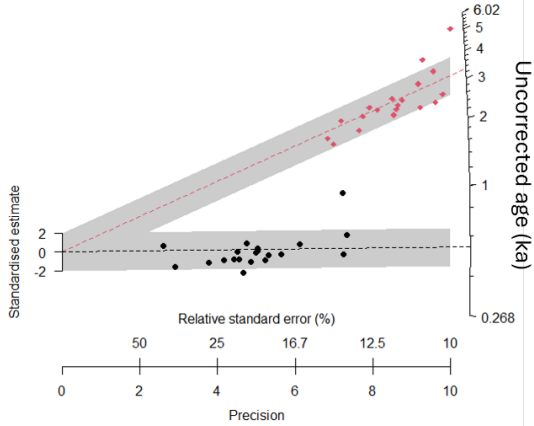
(c) Rock 7
 0.58 ± 0.03 ka (IR₅₀), 2.0 ± 0.1 ka (pIRIR₂₉₀)



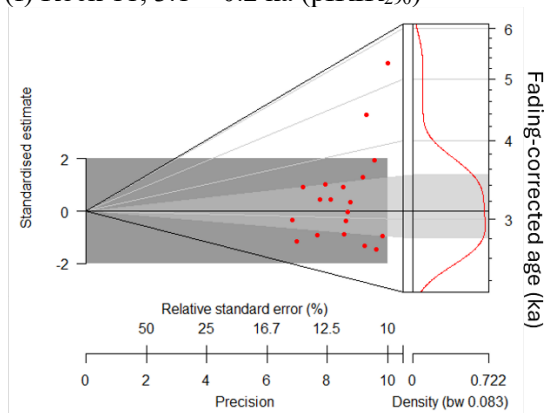
(d) Rock 7, 2.8 ± 0.6 ka (pIRIR₂₉₀)



(e) Rock 11
 0.51 ± 0.04 ka (IR₅₀), 3.3 ± 0.2 ka (pIRIR₂₉₀)

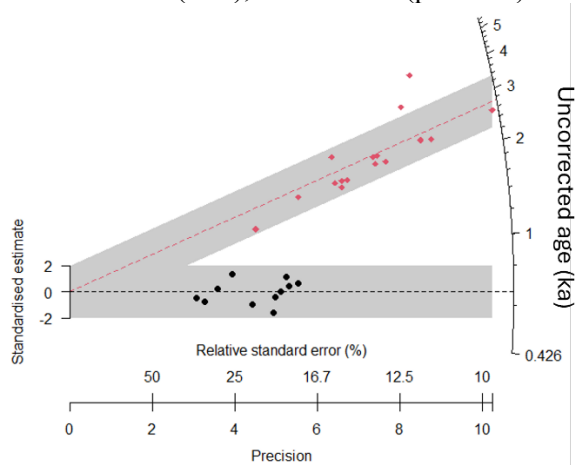


(f) Rock 11, 3.1 ± 0.2 ka (pIRIR₂₉₀)

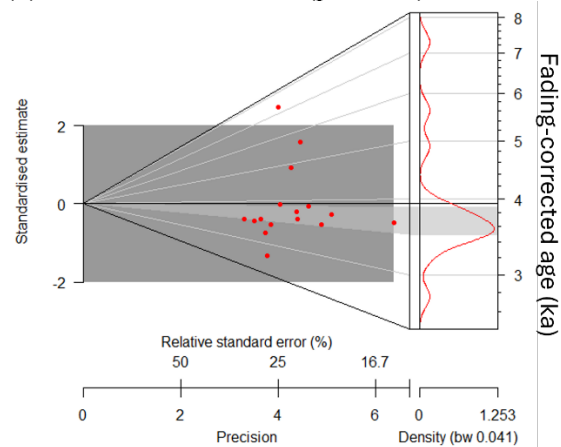


605 **Figure 9. Uncorrected (left) and fading-corrected (right) age distributions from polymineral grains extracted from the outer ~1 mm layer of volcanic Rocks 4, 7 and 11 (~2500 grains per aliquot, 32-63 μm fraction) from CA21P1 in Cave Valley. Corrections apply the fading correction model of Huntley and Lamothe (2001). Black dots are IR₅₀ data (younger age group in each radial plot on the left), red dots are pIRIR₂₉₀ data (older age group in each radial plot on the left, and fading-corrected ages on the right).**

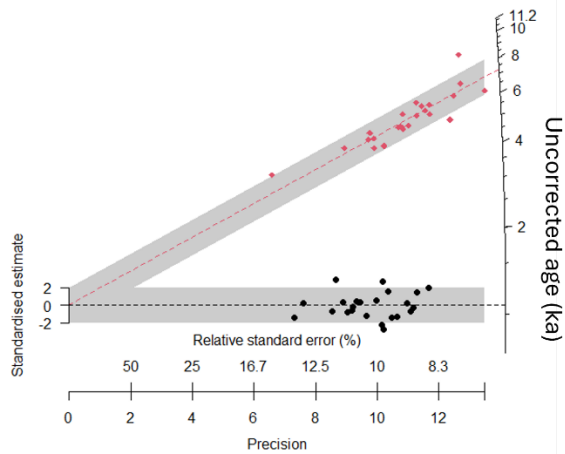
(a) Rock 12
 0.66 ± 0.04 ka (IR_{50}), 2.7 ± 0.2 ka ($pIRIR_{290}$)



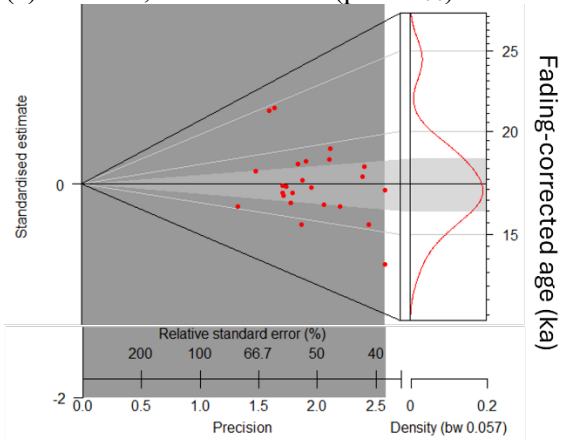
(b) Rock 12, 3.9 ± 0.7 ka ($pIRIR_{290}$)



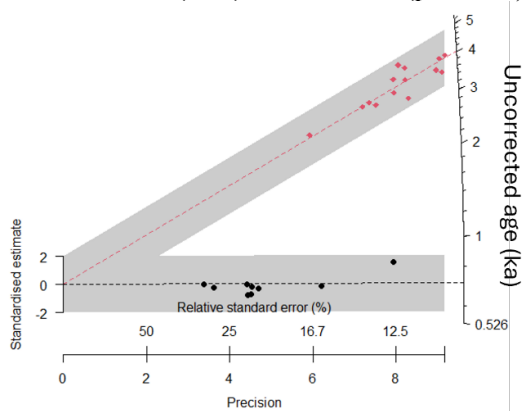
(c) Rock 13
 1.07 ± 0.03 ka (IR_{50}), 7.3 ± 0.2 ka ($pIRIR_{290}$)



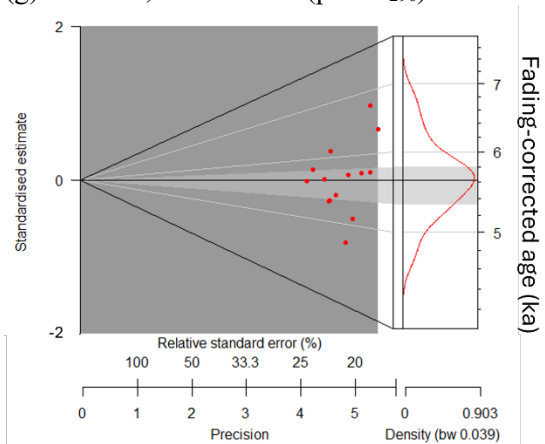
(e) Rock 13, 17.3 ± 10.2 ka ($pIRIR_{290}$)



(f) Rock 18
 0.60 ± 0.05 ka (IR_{50}), 4.0 ± 0.2 ka ($pIRIR_{290}$)



(g) Rock 18, 5.6 ± 1.1 ka ($pIRIR_{290}$)



610 **Figure 10. Uncorrected (left) and fading-corrected (right) age distributions from polymineral grains extracted from the outer 1 mm layer of volcanic Rocks 12, 13 and 18 (~2500 grains per aliquot, 32-63 μm fraction) from CA21P1 in Cave Valley. Corrections apply the fading correction model of Huntley and Lamothe (2001). Black dots are IR₅₀ data (younger age group in each radial plot on the left), red dots are pIRIR₂₉₀ data (older age group in each radial plot on the left, and fading-corrected ages on the right).**

4.4.6 Rock age-depth profiles

D_e was measured from rock core slices using the same pIRIR₂₉₀ SAR protocol that was used for volcanic rock surfaces (Figs S20, S21). Some slices could not be measured due to limited sample material or dim signals and aliquots that failed SAR aliquot rejection criteria. Due to irregular rock surfaces and heterogeneities in rock hardness, slice thicknesses varied.

615 As expected, IR₅₀ D_e values consistently underestimate the pIRIR₂₉₀ D_e values in all rocks, and that includes those D_e values measured from the surface (i.e., top- or bottom-most) slice. Several rock pIRIR₂₉₀ D_e profiles (Rock 4, Rock 7 core 1, Rock 11, Rock 13, Rock 18 core 2) rise with depth into the rock at either the top or bottom side. For example, the Rock 7, core 1 pIRIR D_e profile starts at its lowest point at the bottom surface of the rock (left), then rises with depth into the rock (Fig. S20B). The IR₅₀ D_e profiles of the same rocks typically rise in a similar pattern, but at a much lower rate, yielding much more subdued
620 (flatter) profiles. Similar patterns have been observed in IR₅₀ and pIRIR luminescence-depth profiles reported elsewhere (e.g., Sohbaty et al., 2015; Freiesleben et al., 2015; Jenkins et al., 2018).

Aliquot slice D_e values were divided by depth-attenuated dose rates calculated in Section 4.4.4 to derive age-depth profiles (Figs 11-13). Age-depth profiles generally show similar patterns to those observed in the D_e profiles; here we plot them using a logarithmic scale on the y-axis so that the IR₅₀ age-depth profile shape is vertically enhanced. The uncorrected IR₅₀ and
625 pIRIR₂₉₀ age-depth profiles, as well as the fading-corrected pIRIR₂₉₀ age-depth profiles further support our interpretation above that gravels experienced short-term, heterogeneous light exposure to their surfaces prior to burial. Clear, plateaus that intersect the rock surface suggestive of a long-term bleaching event prior to gravel burial (such as that shown in Figure 1) are absent. Attempts to apply a light bleaching model to the luminescence signal depth profiles to predict pre-burial profile shapes (e.g., Freiesleben et al., 2015; Khasawneh et al., 2019) were unsuccessful; due to the scatter in the data and relative paucity of data
630 points from each rock, model results were overly sensitive to parameter starting values. Therefore, plateaus were detected in the data using the statistical test for homogeneity of Galbraith (2003) following the approach of Gliganic et al. (2021). Using this approach, we identified statistically consistent populations of pIRIR₂₉₀ ages within each slice and rejected outlying values. Then slice ages in each depth profile (each calculated using CDM) were systematically tested against each other to identify plateaus within each age-depth profile (Figs 11-13). Plateau ages were then calculated by applying CDM to all statistically
635 consistent groups of ages (Table 6).

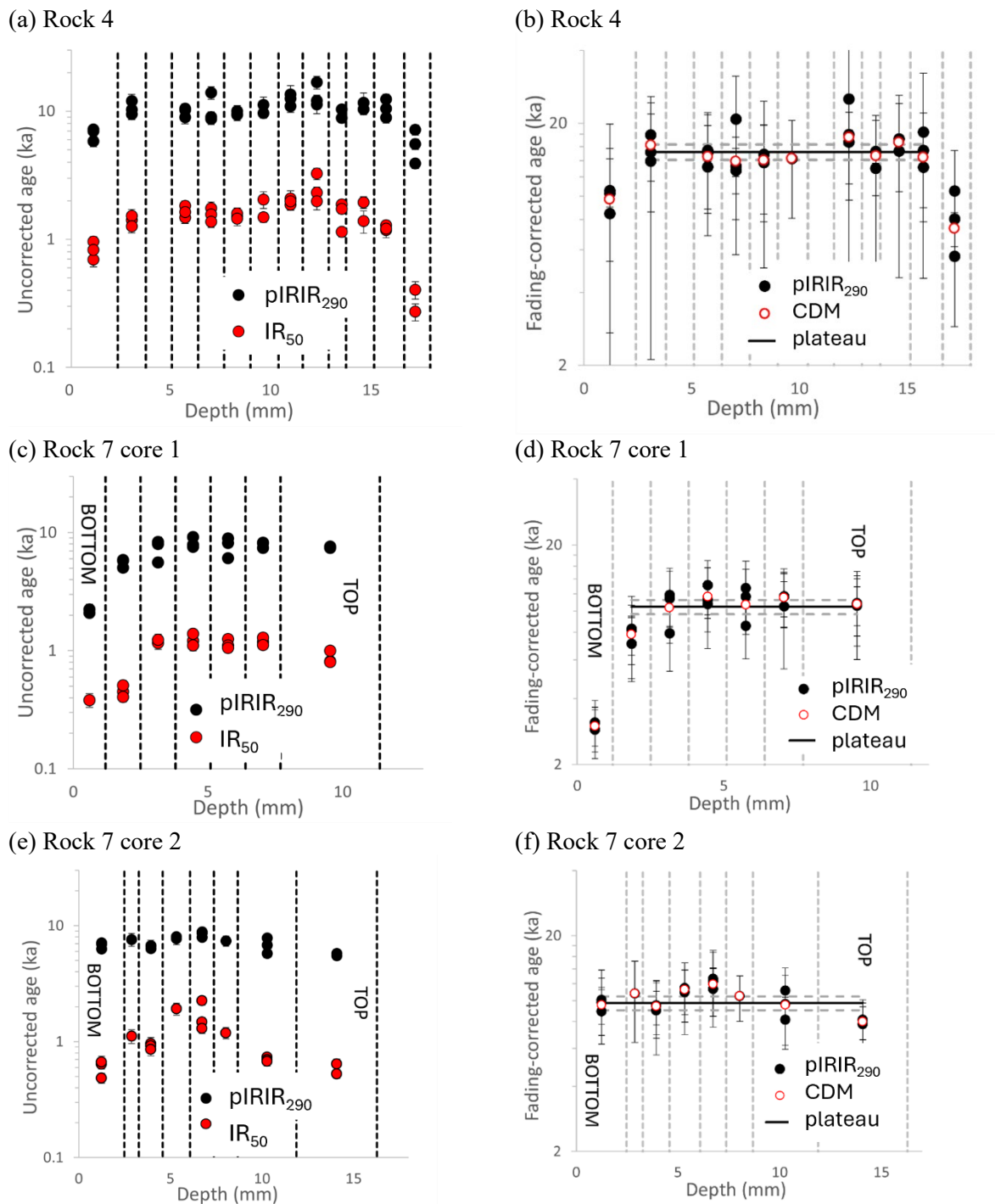
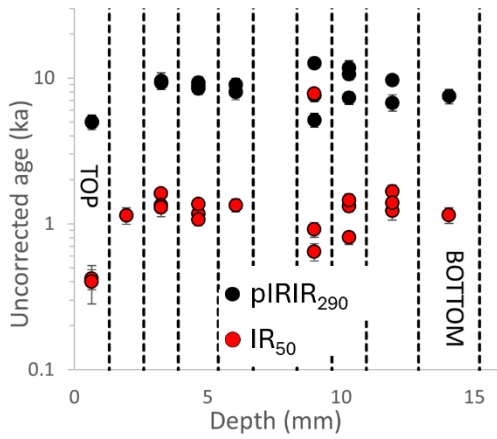
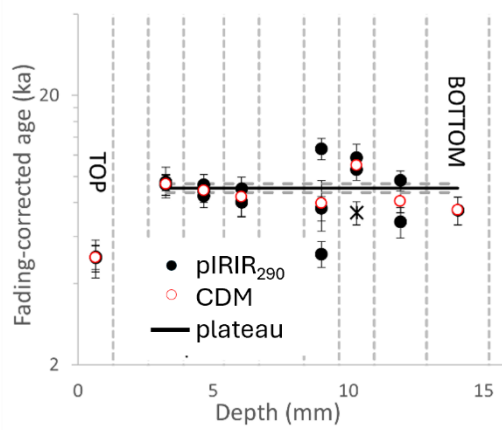


Figure 11. Uncorrected (left) and fading-corrected (right) age-depth profiles for volcanic Rocks 4 and 7 from CA21P1. Vertical dashed lines denote slice boundaries. Aliquots contain ~125 grains each of the 125-250 μm fraction. Top and bottom of the rock indicated for oriented samples. The horizontal lines indicate the plateau age $\pm 1\sigma$. On the right, black dots are pIRIR₂₉₀ aliquot ages and red hollow circles are CDM weighted mean ages for each slice. The y-axes are plotted on a log scale.

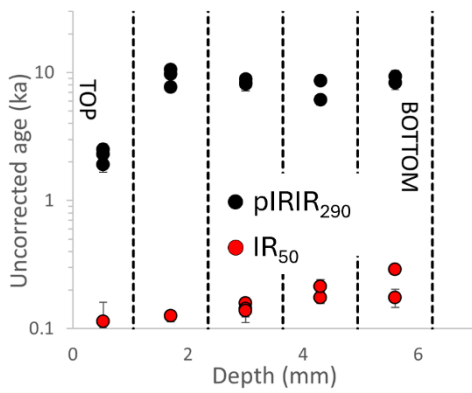
(a) Rock 11



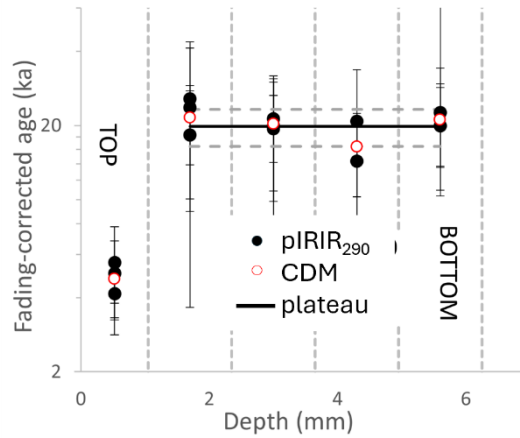
(b) Rock 11



(c) Rock 13

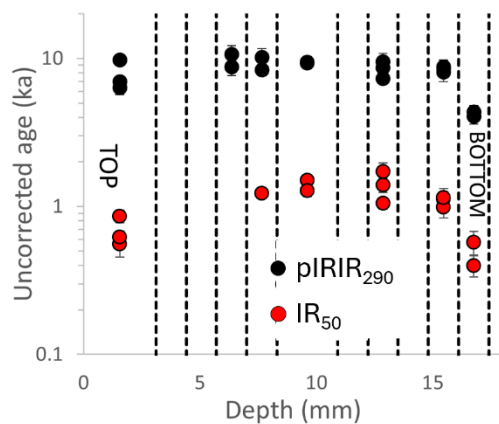


(d) Rock 13

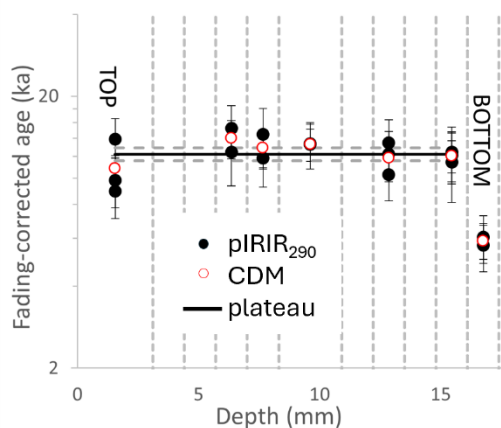


640 Figure 12. Uncorrected (left) and fading-corrected (right) luminescence-depth profiles for volcanic Rocks 11 and 13 from CA21P1. Vertical dashed lines denote slice boundaries. Aliquots contain ~125-165 grains each of the 125-250 μm fraction. The horizontal lines indicate the plateau age $\pm 1 \sigma$. Black dots are pIRIR₂₉₀ aliquot ages, X symbols are rejected aliquots, and red hollow circles are CDM weighted mean ages for each slice. The y-axes are plotted on a log scale.

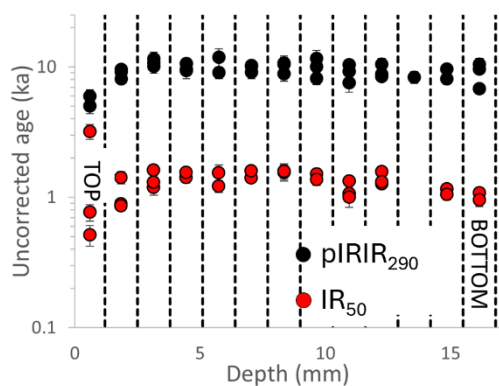
(a) Rock 18 Core 1



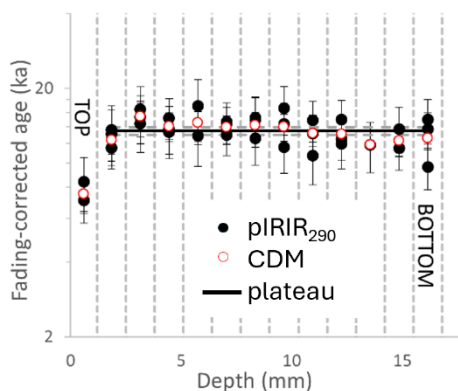
(b) Rock 18 Core 1



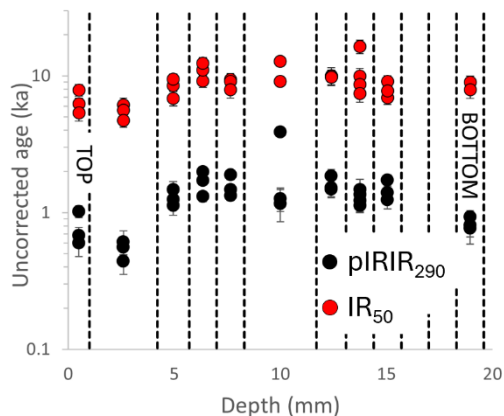
(c) Rock 18 Core 2



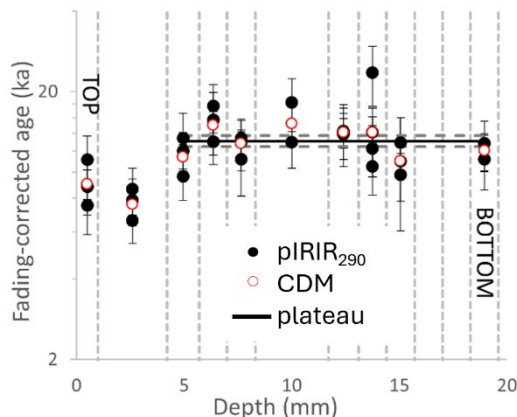
(d) Rock 18 Core 2



(e) Rock 18 Core 3



(f) Rock 18 Core 3



645 **Figure 13.** Uncorrected (left) and fading-corrected (right) luminescence-depth profiles for 3 cores from Rock 18 from CA21P1. Vertical dashed lines denote slice boundaries. Aliquots contain ~125 grains each of the 180-250 μm fraction. Top and bottom of the rock indicated. The horizontal lines indicate the plateau age $\pm 1 \sigma$. On the right, black dots are pIRIR₂₉₀ aliquot ages and red hollow circles are CDM weighted mean ages for each slice. The y-axes are plotted on a log scale.

4.4.7 Dremeled rock surface age results

650 Weighted mean pIRIR₂₉₀ ages for the dremeled outer ~1 mm layer of each volcanic rock (32-63 μm grain size fraction) are summarized in Table 6 and Figure 14(a). The pIRIR₂₉₀ fading-corrected ages range from ~3 to ~17 ka, where all rocks except for Rock 13 dates to the mid-late Holocene between ~3 and 6 ka. The CDM weighted mean of all volcanic rock surface ages, excluding the low-precision, high outlying value from Rock 13, is 3.7 ± 0.4 ka.

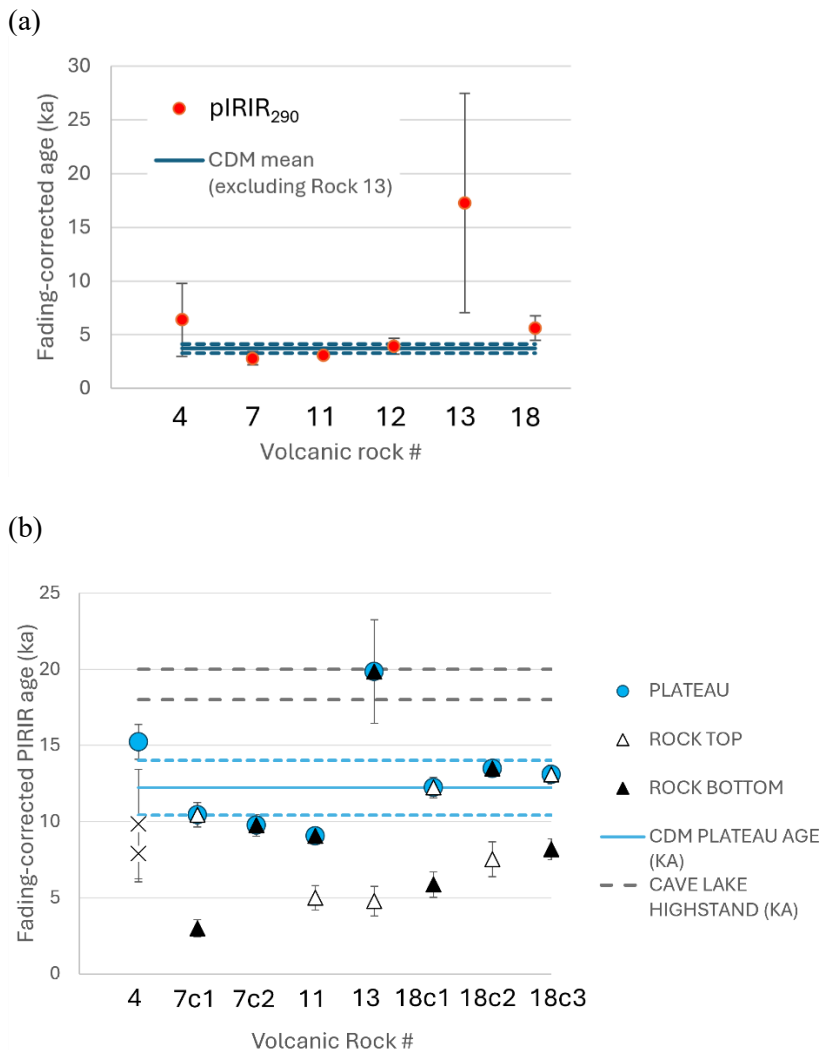
4.4.8 Age-depth profile results

655 Rock surface ages were calculated from the outer-most slices of each rock core (125-250 μm sized grains from the top, bottom or sides of the rocks), as well as from statistically consistent slice ages (the plateaus) within each age-depth profile (Table 6, Fig. 14(b)). Again, fading-corrected surface slice ages vary widely with pIRIR₂₉₀ ages ranging from ~3 to ~20 ka. The rock surface slice ages are generally equal to, or older than the dremeled surface ages discussed in Section 4.4.7 above, suggesting that rock slices may contain unbleached, residual signal as a result of irregular cut rock surfaces. Rock top and bottom age values show significant variability within and between rocks suggesting that sun exposure was not uniform across rock surfaces
660 prior to burial and/or the rocks experienced phases of re-mobilization prior to final emplacement.

Weighted mean ages calculated from age-depth profile plateaus range from ~9 to ~20 ka and show consistency from core to core in Rocks 7 and 18 where more than one core was taken (Table 6, Fig. 14(b)). The CDM weighted mean age of all plateau ages is 12.2 ± 0.9 ka.

665 Table 6. Volcanic rock fading-corrected ages from CA21P1 in Cave Valley. “Oriented” rocks were sampled with their top and bottom surfaces recorded. The expected age of the pluvial lake highstand in Cave Valley is ~18,000-20,000 years, and ages consistent with this at 1 σ are in bold.

Volcanic rock #	Depth (cm)	Core length [Rock dimensions] (mm)	Surface dose rate (Gy/ka)	Dremeled surface pIRIR ₂₉₀ age (ka)	Surface slice(s) pIRIR ₂₉₀ age (ka)	Plateau pIRIR ₂₉₀ age (ka)
Rock 4 (unoriented)	38	16 [55x35x16]	6.22 ± 0.33	6.4 ± 3.4	9.8 ± 3.6 7.9 ± 1.9	15.2 ± 1.1
Rock 7 (oriented)		[70x50x14]]				--
Core 1	44	10	7.33 ± 0.42	2.8 ± 0.6	10.4 ± 0.8 (T) 3.0 ± 0.6 (B)	10.4 ± 0.8
Core 2		14			9.8 ± 0.7 (T, B)	9.8 ± 0.7
Rock 11 (oriented)	36	14 [70x45x14]	7.44 ± 0.42	3.1 ± 0.2	5.0 ± 0.8 (T) 9.1 ± 0.3 (B)	9.1 ± 0.3
Rock 12	42	[70x40x30]	6.86 ± 0.38	3.9 ± 0.7		--
Rock 13 (oriented)	42	6 [70x40x6]	7.31 ± 0.41	17.3 ± 10.2	4.8 ± 1.0 (T) 19.9 ± 3.4 (B)	19.9 ± 3.4
Rock 18 (oriented)		[55x40x16]				
Core 1	42	16	7.23 ± 0.41	5.6 ± 1.1	12.2 ± 0.7 (T) 5.9 ± 0.8 (B)	12.2 ± 0.7
Core 2		16			7.5 ± 1.1 (T) 13.5 ± 0.5 (B)	13.5 ± 0.5
Core 3		16			13.1 ± 0.6 (T) 8.18 ± 0.68 (B)	13.1 ± 0.6



670 **Figure 14. (a)** Weighted mean ages of CA21P1 samples calculated from 32-63 μm polymineral grains extracted from the outer ~ 1 mm of the rock surfaces using a Dremel tool. Each rock age (red dot) is the weighted mean of 5 to 24 multi-grain aliquot ages. The horizontal lines mark the CDM weighted mean (3.7 ± 0.4 ka) of all rock surface ages at $\pm 1\sigma$ excluding the low-precision, high outlying value from Rock 13. (b) pIRIR₂₉₀ rock surface slice ages and plateau ages from CA21P1 samples. Rock 4 was not oriented, so both surface slices are shown as “x” symbols. The top and/or bottom of cores 7c1, 7c2, 11, 13, 18c1, 18c2 and 18c3 are consistent with the plateau age. The 180-250 μm polymineral fraction was measured using 3 mm diameter aliquots (~ 125 grains per aliquot).
 675 Ages are the CDM weighted mean of the measured multi-grain aliquot ages. The blue horizontal lines mark the CDM weighted mean age of all plateau ages $\pm 1\sigma$; this is 12.2 ± 0.9 ka. The horizontal gray dashed lines show the maximum and minimum expected ages of the Cave Lake highstand.

5 Discussion

Luminescence dating approaches applied in this study were highly dependent on the lithology of the rocks available to date. Gravel lithologies in pluvial lake beach ridges are dominated by the lithology of the closest bedrock outcrops that provided rock for shoreline transport. These materials vary significantly from site to site and from valley to valley. In our study this necessitated the development of two very different dating approaches – one for limestone in Coal Valley and one for volcanic rock in Cave Valley. The results obtained from each site are discussed below.

5.1 Coal Valley

5.1.1 Significance of limestone rock ages

Luminescence ages derived from limestone gravels from Coal Valley varied significantly from rock to rock and even showed dependence on measured grain size within the same rock (compare ages for grain size fractions 63-90 μm and 90-125 μm for Rock 2, Table 5). This variability is likely attributed to non-uniform bleaching of rock surfaces, variability of rock light transmission properties (Ou et al., 2018), differences in chemical or physical weathering, as well as micro-beta dosimetry effects that result from non-uniform rock composition (Meyer et al., 2018).

Calculated CDM ages were most likely to agree with the independent age control, while ADM ages tended to overestimate the expected age (Table 5, Fig. 7). The congruency between the CDM ages of Rocks 2 (90-125 μm fraction), 18 and 10 suggest that their surfaces were reasonably well bleached. The anomalously old age obtained from the 63-90 μm grain size fraction from Rock 2 is more difficult to reconcile but may be indicative of incomplete bleaching of finer grains that were more susceptible to clumping than their coarser grain counterparts. MDM ages straddle the radiocarbon age estimate of the deposit, again with the 63-90 μm fraction from Rock 2 yielding the oldest calculated MDM age (Table 5, Fig. 7). MDM ages that appear to post-date the time of beach ridge formation may not be derived from grains inside the limestone at all, but rather, despite our efforts to remove outer carbonate coatings, may be contaminating grains that became cemented onto the limestone surface as part of a pedogenic carbonate coating.

5.1.2 Challenges

The main challenge dating limestone clasts relate to the limited quantity of detrital grains available for dating (Table 4, column 2). Limestone formations in Nevada can vary in their detrital sediment content but are commonly associated with, or interbedded with, sandstones and shales (e.g., Rowley et al., 2017; Hurtubise and Bray, 1988). The concentration of detrital sediment available within limestone rocks for dating varies from location to location, and this necessitates the need for testing for viable luminescence signals from each site, perhaps with some guidance from local geological maps. Quartz signals from polymineral grain extracts exhibited no fast component in any of the limestone gravels tested, so further analysis was restricted

to feldspar. Grains that do have an IR signal, however, were typically well suited to SAR, and had moderate fading rates that are relatively easy to correct for, even at the single-grain level.

5.1.3 The single-grain advantage

710 For limestone gravels, it is preferable to apply the single-grain approach as opposed to measuring larger multi-grain aliquots, primarily because the single-grain approach requires relatively small amounts of material, but also because it produces a high-resolution age distribution for each sample that can be examined for evidence for incomplete bleaching of grains or other sources of scatter. Past research shows that it is common for rocks to experience only partial sun exposure prior to burial on one or more sides (e.g., Meyer et al., 2018; Smith et al., 2023), so single-grain dating, and perhaps the MDM model, should
715 allow us to target grains that are more likely date the most recent bleaching event. However, we must keep in mind that the relationship between single-grain age distribution shapes, overdispersion (OD), rock transport and depositional history, and chemical and physical weathering of limestone rock surfaces, has yet to be examined in detail, and should be the focus of future research.

5.2 Cave Valley

720 5.2.1 Luminescence signals of volcanic rocks

Luminescence signals suitable for dating are most likely to be found in volcanic rocks that are intermediate to felsic in composition and contain a higher concentration of K-rich feldspars. Such types of rock are common in Lincoln County, with intermediate silicic ash flow tuffs and other tuffaceous sedimentary rocks covering more of Nevada than any other type of rock (Crafford, 2007). Intermediate and felsic volcanic rocks are best represented at site CA21P1 in our study.

725 5.2.2 Sources of variability in age-depth profiles

Volcanic rock surface ages and age-depth profiles from site CA21P1 suggest heterogeneous light exposure on rock surfaces. The variability in rock surface ages, where clast tops often appear younger or older than clast bottoms, and the variability between age-depth profiles from within the same rock (Table 6, Fig. 14), are consistent with observations made at other sites (e.g., Rades et al., 2018; Souza et al., 2019; Smith et al., 2023) and are indicative of incomplete bleaching of all rock sides
730 prior to burial. Bleaching rates in rocks are dependent on rock surface aspect, light transmission of the rock, daylight spectrum, intensity, and duration of exposure (Ou et al., 2018; Smedley et al., 2021; Furhmann et al., 2022).

Variability in age-depth profiles may also be attributed to rock composition. Rocks that are light in color, and fine-grained, with a homogeneous mineral composition over the scale of analysis, are anticipated to be better and more uniformly bleached than rocks that are dark in color, coarse-grained, and have a heterogeneous composition over the scale of analysis (Ou et al.,
735 2018; Meyer et al., 2018). Heterogeneities in the dose rate field within gravels and near gravel surfaces could also lead to micro-beta dosimetry effects that are not accounted for in our dose rate models.

5.2.3 Evidence of soil accretion after the pluvial lake highstand

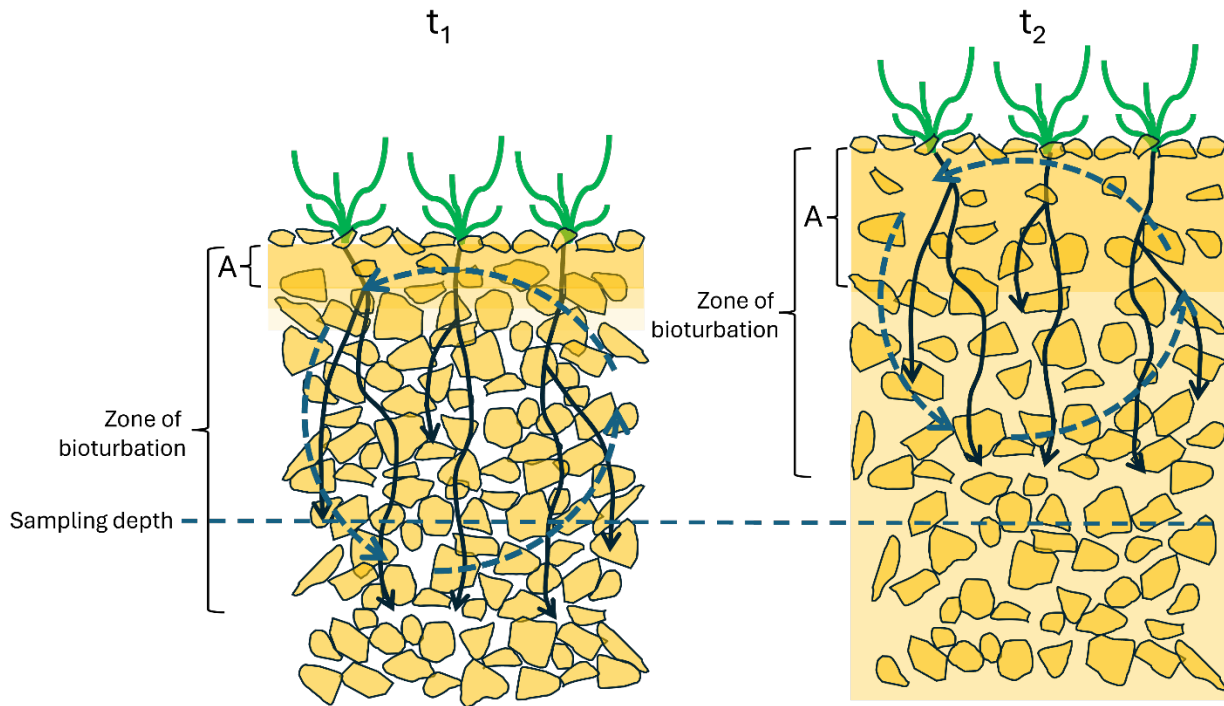
The oldest ages of the dataset from Cave Valley approach ~20 ka (Rock 13) (Table 6). This supports the inference that the beach ridge at site CA21P1 formed no earlier than Marine Isotope Stage (MIS) 2. However, an unexpected finding of this study was the large number of anomalously young ages obtained from volcanic gravel surfaces from site CA21P1. Gravel surface ages obtained from pIRIR₂₉₀ signals suggest that many gravel surfaces were light exposed long after the last pluvial lake highstand in Cave Valley, and even as recently as the late Holocene (Table 6, Fig. 14). Given that our sampling depth at this site was less than 0.5 m below the present-day surface, this must be due, in part, to i) bioturbation, and possibly ii) post-burial light exposure of gravels in what were initially open-work gravels near the beach ridge surface after beach ridge formation.

Soils in dust-influenced arid and semi-arid regions of the American west are thought to form by accretionary processes where dust influxes increase soil volume and inflationary strain (McFadden, 2013). In this accretion-inflationary mode of profile development (also known as AIP), dust becomes trapped by vegetation, desert pavement stones, or irregular, rocky surfaces, then translocates down the soil profile thickening the Bt and Bk horizons. Increases in dust flux during the Holocene have been linked to a 20 cm rise in ancient Pleistocene desert pavement surfaces on lava flows in the Cima Volcanic Field in the Mohave Desert, California (McFadden et al., 1987). Thermoluminescence (TL) ages of ~12-13.5 ka and 5 ka were obtained from Bwk and Av soil horizons, respectively, that are situated on 560 ka lava flows. These ages were attributed to the continuous supply of aeolian materials to the soils during the Late Pleistocene, with an increase in dust flux during the early to middle Holocene (McFadden et al., 1998).

Lake level reconstructions in our study area suggest that Cave Lake receded from its highstand ~18,000-20,000 years ago (Duke and King, 2014), consistent with the plateau age of Rock 13 (Fig. 14). Initially these gravels may have been open-work gravels where large pore spaces allowed light penetration to some depth below the beach surface, allowing for the bleaching of near-surface rocks after ~18,000 years ago. As the climate conditions became more arid from 14,600 to 12,900 years ago (Rhode and Adams, 2016), and again after the Younger Dryas, dust accumulation would have facilitated the establishment of soil and shrubland vegetation (t1 in Fig. 15). Root penetration and burrowing animals would have created a zone of bioturbation below the surface. Given that roots of Great Basin desertscrub species (Spaulding, 1985) can extend several feet below the surface to access water at depth, this depth of disturbance likely extended up to a meter or more and likely pushed gravels at the surface down to the depth of sampling. Plateau ages from inside Rocks 4, 7, 11 and 18 (Fig. 14(b)) coincide with the desiccation of lakes in the region between ~14,600 and ~9,400 years ago (Rhode and Adams, 2016; Duke and Young, 2018) and may record burial of these gravels by newly exposed and wind-transported playa silt and sand. The mid-late Holocene ages derived from rock surfaces (Fig. 14(a) and (b)) likely record more recent temporary heterogeneous light exposure of the rocks as they are re-mobilised in the bioturbation zone of the soil.

During the Middle Holocene Drought (~8-4 ka) (Wriston, 2009; Steponaitis et al., 2015), dust influx to the beach ridge soils likely intensified (cf. McFadden et al., 1992). We infer that accretion-inflationary processes contributed to volume expansion

770 of the soil raising its surface above the original Late-Pleistocene open-work gravel surface level (t_2 , Fig. 15). This implies that
gravel clasts sampled at ~38-44 cm depth in this study, if not bioturbated, would have been several centimetres closer to the
original open-work gravel surface prior to accretion. The youngest volcanic rock surface ages cluster between ~2.8 ka ~6.4 ka.
This suggests that the number of surface gravel clasts pushed down to the depth of sampling decreased substantially after ~3
775 ka as the A horizon thickened and the substrate became less permeable. This implies that by the late-Holocene the soil surface
and the zone of subsurface bioturbation had risen above the level of sampling (t_2 , Fig. 15).



780 **Figure 15. Schematic illustrating the accretionary-inflationary mode of soil profile development on the beach ridge at site CA21P1. Initial establishment of an A soil horizon after beach ridge abandonment in the late Pleistocene-early Holocene is shown as “ t_1 ”. Increases in dust influx ~8-4 ka (t_2) cause accretion of the A horizon and translocation of fines to depth. As the soil surface rises, the zone of bioturbation rises preventing surface clasts reaching the depth of sampling after ~3 ka.**

5.2.4 Significance of volcanic rock plateau ages

All fading-corrected pIRIR₂₉₀ plateau ages from volcanic rocks from site CA21P1 are finite, with no evidence of saturation (Figs 11-13 and 14(b)). In previous studies where cobble-sized rocks are dated, the highest plateau identified near the centre of the rock luminescence-depth profile is typically interpreted to record “saturation” (Fig. 1). “Saturation” refers to the area where
785 bleaching has never occurred, and the luminescence signal has reached an equilibrium, where signal loss due to fading (for feldspars) is in equilibrium with signal gained via irradiation. However, in some cases, if the rocks dated are small enough, and if light exposure is long enough, light can penetrate the entire thickness of the rock, leading to a flattening of the plateau. Lehmann et al. (2018) showed that the inflection point of the IR₅₀ luminescence-depth curve migrates to 3-4 mm depth after

20 to 140 years of sun exposure for coarse grained orthogneiss. Ou et al. (2018) found that after 91 days of sun exposure, the
790 pIRIR₂₂₅ signal was bleached to half of its initial intensity at a depth of ~1.8 mm for a dark grey, fine grained indurated
sedimentary greywacke. Furhmann et al. (2022) depleted the pIRIR₂₂₅ signal to a depth of over 2 mm in granite exposed to the
sun for 108 days. Evidence for internal bleaching of IR₅₀ signals in rocks of thicknesses ranging from ~15 to ~23 mm has been
shown by Souza et al. (2019) for samples collected from a modern beach and a ~2,000-year-old sandy beach ridge in Denmark.
pIRIR signals are known to bleach at a slower rate than IR₅₀ signals, but nonetheless, this begs the question as to whether the
795 Cave Valley beach gravel rocks are small enough such that the pIRIR₂₉₀ signal was completely depleted throughout their
thickness in the swash or near-shore zone of the lake environment prior to burial.

Despite the susceptibility of gravels to re-working in the soil zone, the age range of our rock plateau ages suggests that pIRIR₂₉₀
signals inside gravel rocks may serve as more reliable geochronometers for the time of beach ridge formation and initial soil
development at the late-Pleistocene-Holocene transition than the signals measured at the rock surfaces. Such a scenario could
800 occur if gravels in the swash zone of the lake, or emplaced at or near the surface of an abandoned beach ridge, were sun-
exposed for long enough periods to completely deplete their pIRIR₂₉₀ signals throughout their thickness prior to final burial
(cf. Souza et al., 2019). In this scenario, the pIRIR₂₉₀ signal that accumulates at the centre of the gravels during burial may be
less prone to depletion during subsequent brief periods of sun exposure during bioturbation events, which preferentially
depletes the signal near the surface of exposed rock surfaces.

805 **5.2.3 The impact of soil development**

The ~18,000-20,000 year old Cave Lake highstand is thought to have preceded the Lake Coal highstand 15,873-16,281 cal yrs
BP (Duke and King, 2014; Wriston and Adams, 2020), however despite this, most Cave Lake rock surface and internal plateau
luminescence ages post-date the highstand of Lake Coal as well as the model-equivalent rock surface ages from the Coal
Valley beach ridge (Tables 5 and 6). This is likely due in part to the depth of sampling; at Cave Valley the sampling depth
810 (<0.5 m) was less than that at Coal Valley (1 m) and clearly within the zone of the A/B soil horizon, thus increasing our chances
of sampling bioturbated material. When sampling pluvial lake gravel beach ridges, efforts should be made to sample at depths
of ~1 m or greater. Natural exposures of beach ridge sediments such as those sampled at Coal Valley are rare in the Great
Basin, and so deeper sampling may require the use of mechanical equipment.

Dose rates for Cave Valley were calculated assuming that the present geochemistry of the samples have been consistent during
815 the burial history of the beach ridge. This assumption is problematic given the shallow (<0.5 m) sampling depth within soils
that would have developed shortly after beach ridge abandonment and continued to evolve during the Holocene. Accretion-
inflationary soil profiles are dynamic, characterized by cumulic growth as well as pedogenic modification. As the soil develops,
it increasingly influences infiltration rates, depths of water movement, and thus, rates and processes of carbonate translocation
and accumulation for the entire soil (McFadden et al., 1992; 1998). Future luminescence sampling should ideally focus on
820 primary beach ridge sediments below any soil development until pedogenic effects on time-averaged dose rates are better
understood.

6 Conclusions

This study examined the feasibility of dating pluvial lake beach ridges using rock surface dating techniques. The geology of the Great Basin is dominated by rock lithologies that pose challenges for luminescence dating and the lack of natural exposures make sampling below soils and zones of bioturbation difficult. Tests and measurements from two prominent rock types, limestone and volcanic rock, show promise. Select limestone clasts were found to have adequate quantities of detrital sediment preserved near their surfaces for dating. Polym mineral extracts from limestones exhibited IR₅₀ signals with low to moderate fading rates and properties suited to SAR, and ages calculated using the central dose model tend to agree with the independent age control. Volcanic clasts exhibited IR₅₀ and pIRIR signals with high fading rates and could only be dated using a high-temperature pIRIR₂₉₀ signal with a fading correction. However, comparisons between age-depth profile plateau ages calculated from inside volcanic rocks and independent age control suggests that pIRIR₂₉₀ signals preserved in the rock sub-surface may record the time of beach ridge formation or initial soil development during the late-Pleistocene-Holocene transition.

Comparisons between rock age estimates and independent age control show that both limestone and volcanic gravel-sized clasts are commonly incompletely bleached but also can yield some ages that post-date the time of the pluvial lake highstand. Anomalously young ages from limestones may result from contamination of polym mineral extracts with grains that have adhered to the limestone surface in pedogenic carbonate coatings. Young ages obtained from volcanic rocks suggest that most rock surfaces have been exposed to light long after the pluvial lake highstand, likely because of bioturbation. The youngest volcanic rock surface ages provide a constraint on the timing of pedogenesis and reworking at the sampling depth and perhaps record a climatically driven phase of soil development.

Rock surface luminescence dating techniques for pluvial lake beach ridges in the Great Basin should be further developed to build on this work. We recommend preliminary testing of the optical properties of local rock lithologies comprising the site, followed by mechanical excavation to depths below zones of pedogenesis and bioturbation. Experiments should be designed to examine sample collection and preparation methods, gravel bleaching processes in pluvial lake environments and the impact of soil development and bioturbation on sampled sites.

845 **Data availability**

Data generated during the course of this project is available upon request.

Author contribution

Project conceptualization, fieldwork and sample collection was conducted by CMN and TW. Experiments were designed by CMN with contributions from GTHJ and SH. All experiments and data analyses were conducted by CMN. The manuscript was prepared by CMN with contributions from all authors.

Competing interests

The authors declare that they have no conflict of interest.

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