Single-grain optically stimulated luminescence (OSL) dating of terminal Pleistocene and early Holocene human occupation sites in the Dhofar region of Oman

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By examining three Nejd Leptolthic rock shelters within western Oman (Ghazal, Khumseen and Al-hatab) and providing accurate age estimates using pseudo single-grain optically stimulated luminescence (OSL) dating to determine the age of the sediments, we were able to provide ages for all three sites. The OSL ages are given between ~7-10 ka for Ghazal, ~8-10 ka for Khumseen and ~2-20 ka for Al-hatab.

The implications of the ages are discussed in relation to two current palaeoanthropological models; whether the leptolithic assemblages in Oman represent the development of an in situ human population with unique stone tool industries and genetic signatures, or whether it was the result of a population expansion from the western Mediterranean (the Levant) and/or an out of Africa expansion (Bab al Mandab strait).

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Single-grain optically stimulated luminescence (OSL) dating of terminal Pleistocene and early Holocene human occupation sites in the Dhofar region of Oman

Lauren Patricia Linnenlucke

“A thesis submitted in part fulfilment of the requirements of the Honours degree of Bachelor of Science in the School of Earth and Environmental Sciences, University of Wollongong”

October 2012
Abstract

The Arabian Peninsula is situated within a crossing zone for the movement of pastoralist communities. Current interest regarding the development from hunter-gather to pastoralists is due to the discovery of the Nejd Leptolithic Tradition located within western Oman. The use of Nejd Leptolithic assemblages as an archaeological marker is considered important, as they are only found within Southern Arabia and are considered a defining point in determining when humans transformed into a more prominent pastoralist society.

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‘Some men give up their designs when they have almost reached the goal; While others, on the contrary, obtain a victory by exerting, at the last moment, more vigorous efforts than ever before’.

- Polybius-

The Histories
Book 16:28
Chapter 1
Chapter 1  Background & Study Design

1.1 Introduction

Over the past two decades interest in the archaeology of the Arabian region has peaked, causing an increased awareness of the parameters controlling human evolution in the Terminal Pleistocene and Early Holocene within southern Arabia. Current research from fields such as palaeoanthropology, human genetics, palaeoclimatology (Rose and Bailey, 2008; Maher; 2009; Marks 2009) and palaeolithic archaeology will be discussed within this study. Optically stimulated luminescence (OSL) age estimates, combined with these fields of study, will endeavour to answer questions about the development of modern human societies in western Oman from hunter-gatherers to pastoralists (Fig 1.1).

Suitable sedimentary material for OSL dating in association with archaeological remains have been identified from a number of stratified sites that contain evidence for the Terminal Pleistocene and Early Holocene periods in western Oman. Excavations carried out by the Dhofar Archaeological Project (DAP) and Central Oman Research Project (CORP) in southern Arabia, have identified a number of stratified sites that contain a unique Nejd Leptolithic (Late Palaeolithic) stone tool assemblage (Hilbert., et al, 2012). The term ‘Late Palaeolithic’ refers to the period of stone tool development by modern humans. Southern Arabian Late Palaeolithic assemblages have been dated to between 75 and 8 thousands of years (ka) (Rose and Usik, 2009; Rose and Bailey, 2008). Nejd Leptolithic assemblages in this study will focus on the most recent part of this time period - 14 to 7 ka (Rose and Usik, 2009; Hilbert et al., 2012).

The use of Nejd Leptolithic assemblages as an archaeological marker combined with OSL age estimates will be used to outline the transitional period from hunter-gatherer to pastoralists. Leptolithics are important as they are only found within southern areas of Arabia. Specifically, Nejd Leptolithics will be studied from areas of western Oman in order to define the pastoralists’ transitional period.
In this study, OSL dating will be applied to construct a chronology for stratified sites in which leptolithic assemblages were found. This will help to constrain the leptolithics in southern Arabia as both an archaeological and chronological marker. Current research on human migration patterns throughout Arabia, initially the ‘from Africa into Arabia’ scenario, will be discussed in relation to previous climatic shifts during the Pleistocene.

An outline for the structure of study is presented below.

Chapter 1 reviews current literature on Arabian pastoralist movements, human settlements and the significance of leptolithic assemblages in Oman. In focusing on the development of human settlements within Arabia, three migration patterns are outlined and discussed in relation to the palaeoclimate of the region. This will provide environmental contexts for patterns of human movement leading up to and including the Terminal Pleistocene and Early Holocene. The aims and significance of this study will also be provided.

Chapter 2 outlines the OSL dating methodology used in this study, including an explanation of the methods carried out in sample collection and preparation, instrumentation, data collection and analysis. Descriptions will be provided of the measurement and analytical methods used in the single-aliquot regenerative-dose (SAR) procedure as applied to the samples dated in this study.

Chapters 3, 4 and 5 will provide contextual information for each of the three archaeological sites dated in this study. The results of the pseudo single-grain OSL analysis for each sediment samples will be discussed in detail. Previous dating will also be discussed for all sites. The comparison will form the basis for a discussion of the benefits of single-grain OSL over single-aliquot OSL. The pseudo single-grain $D_e$ distributions will be interpreted within the contextual framework of all three individual sites in order to obtain final and reliable age estimates from each of the samples.

Chapter 6 will discuss the chronology in relation to the two proposed models:

(1) Whether the leptolithic assemblages in Oman represent the development of an in situ human population with unique stone tool industries and genetic signatures, or
(2) Whether it was the result of a population expansion from the western Mediterranean (the Levant) and/or an out of Africa expansion (Bab al Mandab strait).

This chapter will discuss how this study relates to current research and how applying accurate OSL ages will assist in future research.

1.2 Site Study: Nejd plateau, Oman

The site study consists of three Nejd Leptolithic archaeological sites which are located on the Nejd plateau, in south-western Oman, Dhofar (Fig 1.1). The three rock shelters known as Ghazal, Khumseen and Al-hatab (Fig 1.2) have provided stone tool assemblages which have so far been classified under the ‘Nejd Leptolithic Tradition’ (NLT) (Rose, 2006; Rose and Usik, 2009). Two of these sites have given single-aliquot OSL age estimates between approximately 14 and 7 ka, representing the first human occupation sites during the Terminal Pleistocene within southern Arabia (Hilbert et al., 2012). The focus for this study will be on the Terminal Pleistocene and Early Holocene boundary; this is the suspected range for ages of the Leptolithic assemblages within the three sites, based on previous dating (refer to site-specific chapters).

Southern Arabia’s coastal plain is characterised by two plateaus, the Hadramawt (Yemen) and the Nejd (Oman). The focus will be on OSL dating for three Nejd Leptolithic sites in Oman. The Nejd plateau extends north from the Dhofar escarpment onto sedimentary beds of an elevation of 1000 m above sea level, and northwards onto the Nejd (incised Tertiary limestone plateau) (Parker and Rose, 2008). The Nejd plateau has a south to north dip of 120 km extending from the Dhofar mountain chain to the Rub’ al-Khali desert. The plateau, as it extends northward, incorporates highly eroded arid landscape which are dissected by small wadis, and evolves into a flat gravel plain cut by myriad Pleistocene riparian systems (high-energy fluvial systems) (Hilbert et al. 2012). The riparian system occurs along rivers and streams that periodically break their levees, resulting in flooding; this may also occur around meandering channels. The ridge of the Dhofar escarpment marks the southwards-flowing drainage system, which is seasonally active.
under monsoonal conditions. During pluvial cycles the magnitude of the monsoon can be sufficient to produce riparian systems (Parker and Rose, 2008). Contextual information for each site will be discussed within the site-specific chapters.

### 1.3 Standardisation of Leptolithic stone tools

Two debates have arisen as to the origins of Nejd Leptolithics in Oman. These refer to either an advancement of stone tools in response to an *in situ* development of pastoralists in southern Arabia, or if they were the impending result from groups dispersing into the area from different access points in Africa (see section 1.5). Due to the Leptolithic taxonomy, the variation in ages range between approximately 75 and 8 ka, however our primary focus is the Terminal Pleistocene and Early Holocene boundary (Rose and Usik, 2009). For this reason the term ‘Nejd Leptolithic’ was initially proposed by Rose (2006), in reference to a series of surface scatters that were located across the Nejd plateau, Oman (Fig 1.3a) (Rose and Usik, 2009). This assemblage has been used to regionally categorise the Nejd Leptolithic assemblages which have been currently dated to between 14 and 7 ka (Rose and Usik, 2009; Hilbert et al., 2012). Nejd Leptolithic stone tools are only found within southern areas of Arabia and are used as chronological markers in determining the transitional period from hunter-gatherer to pastoralists.

The blade technology of southern Arabia is considered to belong to a separate ‘techno-typological’ family due to the bladed reduction strategy (Rose & Usik, 2009). The production of Leptolithics was manufactured using localised chert; this is found within close proximity to most of the sites (Fig 1.2) (Hilbert et al., 2012; Rose & Usik, 2009; Jagher, 2009). The NLT is divided into early and late; by differentiating between the two, a more analytical approach can be conducted into the modalities that exist within the NLT of the three sites investigated. A further discussion will address the relationships between the three sites (Ghazal, Khumseen and Al-hatab), see section 1.3.1.
1.3.1 Core reduction modalities for NLT

Archaeological assemblages belonging to the Early and Late Nejd Leptolithics are found within sites located on the Nejd plateau. The Early Nejd Leptolithics are established between 14 and 10 ka, and the Late Nejd Leptolithics were dated to between 10 and 7 ka (Hilbert et al., 2012). An outline of the three distinct reduction modalities will be discussed specifically in relation to Ghazal, Khumseen and Al-hatab. It should be noted that refits are repeated across multiple cores (Hilbert et al., 2012). For examples of modalities refer to Fig 1.4.

1. This core reduction strategy is found in both Early and Late Nejd Leptolithic assemblages, located in Al-hatab (GH5-GH7) and Ghazal (GH4). Modality 1 employs a single platform unidirectional-parallel strategy which is also used in modality 2 (Hilbert et al., 2012).

2. Modality 2 appears in both Early and Late Nejd Leptolithic assemblages from Al-hatab (GH5-GH7) and Khumseen (GH5a-GH5b). Key features include the volumetric reduction of elongated blanks across multiple working surfaces of the core from a single platform. This process results in the formation of a convex and convergent place of removal for the blade. It should be noted that modalities 1 and 2 produce the same result (an elongated blank with unidirectional-parallel scar patterns) (Hilbert et al., 2012, Rose and Usik, 2009).

3. Modality 3: The Late Nejd Leptolithic is specific to modality 3, located among sites Al-hatab (GH5-GH7) and Khumseen (GH5a-GH5b). This technique produces a single end product, resulting in the preferential removal of an elongated, diamond-shaped blank. (Crassard, 2008a, 2008b),

1.4 The Levant, Mediterranean

The Mediterranean is a diverse cultural area which has been studied across a variety of fields, specifically in regards to examining a ‘from Africa into Arabia’ crossing. The Levant is considered important when studying Arabia for it is at the centre of where three major
continents meet, all with vast archaeological histories and expanding human refugia (Egypt, Syria, Mesopotamia and Asia Minor). When comparing demographic and lithic industries in the Levant, it has been assumed that Upper Pleistocene lithic industries tend to follow the same geographic patterning (Africa into Arabia) (Parker and Rose, 2008). Studies in phylogenetic reconstructions of human mitochondrial DNA (mtDNA) indicate a fundamentally exclusive use of the Levantine corridor in human dispersals between the Upper Palaeolithic and the Neolithic (Rowold et al., 2007; Fernandes, 2009).

Chronological markers are located across the Levant as lithic artefacts found at archaeological sites enable researchers to determine when change occurred within human population groups as they began to develop into socialised agricultural communities. Lithic artefacts have been a primary focus for the past few decades, leading to a highly refined cultural chronological framework. As a result of the abundance of archaeological evidence, lithic artefacts within the Levant have become easier to categorise, both temporally and typologically (Maher, 2009; Rose et al., 2011). The Levantine region shows a diverse Epipalaeolithic hunter-gatherer society being present between 23 and 12 ka. Archaeological evidence and current research indicates that encampments occupied by the Epipalaeolithic societies varied geographically and temporally, i.e. mixing of local Upper Palaeolithic and Epipalaeolithic traditions found among lithic sites in eastern Jordan and northern Arabia (Rose et al., 2011; Maher, 2009). However this is not assumed to extend throughout Arabia.

The Levant holds significance in changes from hunter-gatherer to pastoralists, as many cultural complexes have been identified (i.e. Negev and Sinai) on the basis of different toolkits and other associated site features (Olszewski, 2001, 2006). By approximately 12 ka, lithic examples from the Levantine region of archaeological sites indicate that humans in the Arabian Peninsula changed to incorporate methods of basic agriculture. Examples include base camps, including the construction of more permanent dwellings and basic implementations of agriculture to intensify plant and animal exploitations, along with the adoption of mobile art and personal ornamentation (Maher, 2009; Rose et al., 2011). These complex transitions occurred within a small geographical area and resulted in the production of a large number of well-excavated and well-dated sites. The documentation of these has led to the key defining features of Levantine lithic assemblages.
1.4.1 Levant stone tools

The production of the Levantine blade is distinguished by the pyramid-shaped cores and made of either localised chert or flint (Maher, 2009). The Levantine features are characterised by a microlithic (blade) toolkit consisting of bone tools, ground stone, art, personal ornamentation, burials and sites. This represents an advanced internal spatial organisation. Current research indicates that the Levantine toolkit is extensive in comparison to the Late Pleistocene within southern Arabia (see section 1.3). The development of regional taxonomy within Arabia is too dependent on the identification of Levantine toolkits, due to the abundance of lithic assemblages and less suitable material reliable for dating methods.

1.5 Migration Patterns

1.5.1 The first model

The first model proposes that the Leptolithic assemblages in Oman represent the development of an in situ human population with unique stone tool industries and genetic signatures. This is discussed by the role of climate, in particular the effect of the Indian Ocean Monsoon system, on western Oman. The irrigation effect caused by the Rub’ al-Khali desert and the contrasting highlands result in excessive runoff into the Dhofar region, thereby creating a more humid environment suitable for human settlement (Rose, 2010; Thompson, 2000). The model discusses the internal influence of cultural development of stone tools through the role of climate in response to Leptolithic assemblages having no outside influence (i.e. Africa and Levant); specifically during the transitional period from hunter-gatherer to pastoralists (Rose and Petraglia, 2009; Thompson, 2000).

As a result of the climatic changes over the course of the Quaternary, the development of agricultural communities in southern Arabia may be associated with topographic relief and localised climatic regimes (Parker and Rose, 2008). The model suggests that during arid phrases, human refugia settled within western Oman that and during periods of oscillating climatic shifts moved into isolated areas such as the Dhofar
mountains, Yemen highlands, the Arabo-Persian Gulf Basin, or the now submerged coastal lowlands of the southern Red Sea Basin (Rose & Bailey, 2008; Marks, 2009; Maher, 2009).

1.5.2 The second model

The second model discusses whether or not the development of Leptolithic assemblages were the result of a population expansion from the western Mediterranean (the Levant), and/or an out of Africa expansion (Bab al Mandab strait). The second model proposes three possible external influences for the development of the NLT specific to the transition period of hunter-gatherer to pastoralists within western Oman. Listed below are the three proposed migration patterns along with current archaeological and palaeoclimatological evidence (Fig 1.5).

(1) River Nile

The first movement proposes human migration from Africa into the Eurasian continent, along the River Nile and through the Sinai and into the Levant (Bar-Yosef, 1987; Preusser, 2009). The Leptolithic assemblages have noted varying degrees of affinity with archaeological evidence from the Levant (Crassard, 2009; Rose & Usik, 2009).

(2) Interior of Arabia

The second theory suggests that human migration occurred through the interior of Arabia via the Rub’ al-Khali desert during periods of increased humidity (McClure, 1976; Maher, 2009). Southern Arabia served as bridge during pluvial systems and as a barrier during arid phases. This again indicates similarities with bladed technology from the Levant (Crassard, 2009; Rose & Usik, 2009).

(3) Horn of Africa

The third migration pattern indicates human movement through the narrow Bab al Mandab Strait and continues into the south-western corner of Arabia. This may have occurred during MIS 2 (12-24 ka), when sea levels were at their lowest point during the Last Glacial Maximum (~18 ka) (Rose, 2010).
1.6 Significance & Innovation

The purpose of this study is to use techniques to place Leptolithic assemblages on an accurate time scale. This will assist with narrowing down the transition of humans within western Oman from hunter-gatherer to pastoralists. By applying pseudo single-grain OSL dating methods to the archaeological site Ghazal, Khumseen and Al-hatab, lithic assemblages will be compared to OSL ages presenting new chronological evidence for the region.

The significance of this study will be to provide accurate pseudo single-grain OSL dates for the archaeological sites used in this study to assist with obtaining accurate time scales for the NLT of western Oman.

1.7 Aims

The aims of this study are;

(1) to provide accurate OSL ages for three archaeological sites - Ghazal, Khumseen and Al-hatab,
(2) to use the pseudo single-grain OSL method to place Leptolithic assemblages within a narrowed timescale, improving our understanding of the timing and duration of the transition from hunter-gather to pastoralism in western Oman
(3) to discuss whether or not the Leptolithic in Oman represents the development of an in situ human population with unique stone tool industries and genetic signatures, or
(4) whether it was the result of a population expansion from the western Mediterranean (the Levant) and/ or an expansion out of Africa (Bab al Mandab Strait).
1.8 Chapter Summary

This chapter summarises the aims and significance of this study by establishing a chronological framework for Nejd Leptolithic assemblages through OSL dating methods. The archaeological significance of lithic assemblages from western Oman is discussed in relation to their possible influences through human migration movements. Pseudo single-grain OSL ages will be used to date three Leptolithic archaeological sites. The age of these three sites will determine the age of the Leptolithics. Since Leptolithics represent the transitional period from ‘hunter-gatherer to pastoralists’, this will then provide an accurate age for the transitional period within western Oman.
Fig 1.1 a) Map indicating the Arabian Peninsula and the Dhofar region, showing the site location for this thesis. b) Site localities of all three rock shelters i) Al-hatab (N 17.313417° E 54.061050°), ii) Ghazal (N 17.314483° E 54.056617°) and iii) Khumseen (N 17.313517° E 54.042111°).
Fig 1.2 Leptolithic stone tools found within southern Arabia.

Fig 1.3 Representing different strike directions for stone tools from the Levant (Crassard, 2009).
Fig 1.4 Schematic showing different examples of blade production in Oman: Modality 1: characterised by the creation of a core work surface with single strike; Modality 2: marked by the volumetric reduction of the cores volume; Modality 3: characterised by a short cycle of reduction favouring the production of triangular to sub-triangular blades and bladelets (Taken from Hilbert et al., 2012).
Fig 1.5 Map indicating areas of potential dispersal patterns into southern Arabia. 1) Migration from Africa into the Eurasia continent, via the Nile River. 2) Migration through the Rub‘ al-Khali desert. 3) Migration through the Bab al Mandab Strait.
Chapter 2
Chapter 2  Dating Methods & Procedures

2.1  Introduction

Dating methods such as OSL are used to provide accurate ages for archaeological sites (Jacobs and Roberts, 2007). Dating of organic matter may be difficult within extreme conditions (extreme heat and fluvial deposits), as there may be little left (e.g. southern Arabia) (Rittenour, 2008). The association of dated material and human occupation is always a key question when applying chronometric dating methods to establish the age of archaeological sites (Jacobs and Roberts, 2007). This chapter will discuss the methods and procedures applied to dating archaeological deposits for single-grain analysis. A structural outline will be given following the theoretical process applied to a typical procedure, as well as the application process undertaken for samples in this study. This chapter will provide the methodology section for the site-specific chapters (3-5).

2.2  Luminescence dating

Numerical dating methods such as luminescence are based on measuring the cumulative effect of ionising radiation on any given material, typically quartz and feldspar (Aitken, 1998; Jacobs and Roberts, 2007). These methods are based on the fact that natural minerals can absorb and store ionizing energy (as trapped electrons) over geological time-scales. This energy is released in the form of luminescence (photon emission), if a mineral is exposed to a sufficient amount of light (OSL) or heat (TL) (Lian, 2007; Wintle, 2008). The irradiation of a mineral grain occurs through the decay of naturally occurring radioisotopes present in the surrounding sediments. These include uranium ($^{238}\text{U}$ and $^{235}\text{U}$), thorium ($^{232}\text{Th}$) and their daughter products, potassium ($^{40}\text{K}$); with minor contributions from cosmic radiation. The energy emitted by these radioisotopes causes the accumulation of electronic charge at defect sites or ‘traps’ in the crystalline structure of mineral grains (Feathers and Bush, 2003; Duller, 2004). Therefore, the mineral grains act as natural dosimeters and record the incoming alpha ($\alpha$), beta ($\beta$) and gamma ($\gamma$) radiation (in the form of trapped
electrons) which is released over time during the process of radioactive decay (Aitken, 1998). When these minerals are stimulated by either light or heat in the laboratory the electrons are evicted from the traps with a corresponding, detectable emission of photons. The intensity of this emission is proportional to the amount of stored energy since the traps were last emptied, and the age can be estimated in conjunction with an assessment of the rate of delivery (i.e. the dose rate). There are three conditions for luminescence dating which may be applied to sedimentary deposits, these include;

1) the presence of a natural dosimeter that will adequately record radiation,
2) sources of natural radioactivity, and
3) a zeroing event.

2.3 OSL dating

The OSL method is dependent on the emission of light where the energy is stored as electrons within the crystalline structure of the mineral grain (Lian, 2007). The energy absorbed is known as the radiation dose, which is measured in Grays (Gy). The term Grays (Gy) refers to the amount of radiation energy absorbed per kilogram of matter (Lian, 2007). OSL dating relies upon the comparison of natural luminescence resulting from environmental radiation, and the luminescence produced by calibrated radiation doses in the laboratory. In the environment, the energy absorbed by the mineral grain comes from the radiation emitted by the radioactive elements of potassium (K) and the decay series of uranium (U) and thorium (Th), and from cosmic rays (Lian, 2007; Jacobs and Roberts, 2007).

Quartz and potassium feldspars act as natural dosimeters and absorb the incoming alpha (α), beta (β) and gamma (γ) radiation emitted during the process of radioactive decay (Aitken, 1998) and incoming cosmic rays. The intensity of the luminescence signal is dependent on the time since each mineral grain underwent repeated cycles of erosion, transport and deposition (Huntley et al., 1985; Duller, 2008). During transport, grains will be exposed to sunlight, releasing the electron charge from within the crystalline structure of the mineral grain. This will return the luminescence signal to zero. Following deposition, natural radiation results in the movement of electrons into the crystalline structure of
mineral grains, causing a build up in the latent luminescence signal; refer to Fig 2.1 (Duller, 2004; Lian, 2007). The luminescence is proportional to the number of trapped charged electrons accumulated within the mineral grain. To measure the luminescence signal, the equivalent dose \((D_e)\) must be found. The \(D_e\) is the radiation dose received by the grain over the period of burial, assuming that complete zeroing has taken place initially. By projecting the natural luminescence signal onto the dose response curve, the \(D_e\) can be estimated (Duller, 2004; Jacobs and Roberts, 2007). This is done by usually giving a set of three regenerative laboratory doses and a test dose. Once results are received, a saturated exponential or a saturating exponential function with an extra linear term is fitted to the data points that pass the rejection criteria (see section 2.6.2). To calculate the depositional age of a sample, the following age equation is used:

\[
(1) \quad \text{Burial Age (ka)} = \frac{\text{Equivalent Dose } (D_e) \text{(Gy)}}{\text{Environmental Dose Rate (Gy/ka)}}
\]

### 2.4 Sample collection

Sample collection was carried out using the same process to ensure that each sample was collected in the same way. Over the period of two field seasons (2010-2011), twenty samples were collected, and ten of these were prepared for OSL dating purposes. Two sediment samples were collected from each stratigraphic layer, adjacent to one another, to ensure that a sufficient quantity of quartz was available for dating. Samples were collected from all sites using opaque, plastic tubing that was hammered by a mallet into the cleaned face of the pit. The tubes were then sealed with duct tape before being double bagged in black plastic for safe transport to Australia. Refer to chapters 3-5 for details of sample locations at each site.
2.4.1 Sample preparation

Samples were prepared in the laboratory using standard OSL procedures under subdued red light conditions to ensure that ‘zeroing’ of the OSL signal does not occur. This was done to avoid inadvertent bleaching of the unexposed sediments. The tube end sediments were retained for either water content determinations or dosimetric purposes. If separate samples had not been collected for this, the middle section of the tube (light-safe sediment) was used following the removal of carbonates with a 10% solution of hydrochloric acid (HCl) and the organic fraction of the sediment oxidised with a 15% solution of hydrogen peroxide (H$_2$O$_2$). Each individual sample was wet sieved to isolate fractions between 90-125 μm and 180-212 μm in diameter and left to dry in a 50 °C oven. Feldspar and heavy minerals were separated from the quartz fraction using sodium polytungstate (SPT) solutions, at a density of 2.62 and 2.70 g/cm$^2$. All samples, except TH50 (1 and 2), underwent an additional density separation at 2.58 g/cm$^2$, to separate the potassium feldspars (K-feldspars) from the sodium feldspars (Na-feldspars). TH50-1 and TH50-2 were not subjected to this additional treatment due to the small amount of material left following the initial 2.62 and 2.70 g/cm$^2$ density separations.

The quartz fraction was etched in 40% hydrofluoric (HF) acid for 45 minutes to remove the outer rind of each grain. The K-feldspars were etched in 10% HF for 10 minutes. Both quartz and feldspar fractions were then sieved again. Due to the small fraction size of the quartz grains, only the 90-125 μm fraction size was used for pseudo single-grain dating. It should be noted that normally the size fraction for single-grain analysis is done using 180-212 μm grains, but due to the abundance of finer sediment in these samples the size fraction used for this study is 90-125 μm. This may result in 3-5 grains in each hole. Such analyses are commonly referred to as ‘pseudo single-grain’ measurements, and some effects of grain-averaging may occur as a result of more than one luminescent grain occupying each hole (Arnold et al., 2012).
2.5 The SAR procedure

The single-aliquot regenerative-dose (SAR) procedure was first established as a method that involves making repeated OSL measurements onto a single-grain or a single-aliquot, which is composed of more than one grain. The natural intensity is then projected onto a dose response curve and the $D_e$ is then established following the sensitivity corrected changes; these are determined by doing test doses (Galbraith et al., 1999; Murray and Wintle, 2000). The construction of a SAR dose response curve typically requires a minimum of three regenerative doses ($R_X$). These will provide the bracket for the expected $D_e$ estimate; a zero dose ($R_X=0$) is provided to monitor for recuperation. The values $L_N/T_N$ and $L_X/T_X$ make up the dose response curve; where $L_N$ is the natural dose, $L_X$ is the regenerative dose, and $T_N$ and $T_X$ are the test dose signals. A repeat regenerative dose is used to monitor for any sensitivity corrected changes that may occur within the test dose ($T_N$ and $T_X$) signals (Galbraith et al., 1999; Murray and Wintle, 2003; Jacobs and Roberts, 2007). An infrared (IR) sensitivity check is done at the end of the sequence to ensure no contamination by feldspar inclusions (Duller, 2004). An outline of a typical SAR procedure may be seen in Fig 2.2.

2.6 Single-grain OSL

The single-grain OSL method has an inherent number of benefits over the simultaneous measurement of multiple grains. By measuring one grain at a time and receiving an OSL signal from this, interpretations from the measurement of many thousands of grains are processed in a routine manner to generate a statistically representative number of independent estimates of $D_e$.

The single-grain OSL method has a number of inherent benefits, these include:

(1) The ability to assess the stratigraphic integrity of the site, including the intrusion of younger grains into older deposits or the reverse, or contamination by roof-spall (i.e. decomposition of unbleached cave rock into well-bleached sediments) (Roberts et al., 1998; Jacobs et al., 2011a).
(2) The ability to check the adequacy of the bleaching event prior to burial (Duller, 2008; Olley et al., 1999, 2004).

(3) The ability to identify and discard any grains with abnormal OSL behaviours prior to the $D_e$ and age estimations (e.g., Jacobs et al., 2003, 2006a,c). The inclusion of such aberrant grains would increase the overdispersion or spread (scatter) in the data and can thereby produce underestimates or overestimates of the true burial dose (Jacobs et al., 2006a, 2006c).

(4) The ability to tell the difference in the beta-dose received by individual grains in their burial environment. This may be very common in archaeological sites that may contain a range of different types of materials (Roberts et al., 1999; Jacobs et al., 2011a).

Single-grain analysis allows suitable material to be selected for dating, thereby allowing depositional and post-depositional history to be assessed from the $D_e$ distributions.

2.6.1 OSL equipment

The Risø TL/OSL-DA-15/20 readers fitted with single-grain laser attachments were used for dating purposes for pseudo single-grain analysis in this study. Pseudo single grains were loaded onto aluminium discs; each disc is 9.7 mm in diameter and 1 mm thick, with 100 individual holes where the grains are placed. Bøtter-Jensen et al. (2000) gives the dimensions for separate chambers at 300 µm deep and 300 µm in diameter, and spaced 600 µm apart, to keep ‘cross illumination’ (from laser scattered light) to a minimum (Bøtter-Jensen et al., 2000). Along the perimeter of the disc there are three location holes (500 µm in diameter), which are used to orientate the disc inside measurement chamber (Fig 2.3).

Ten discs were prepared as pseudo single grains for each individual sample and then placed in alternating positions on a 48 position sample carousel (i.e. disc numbers 1, 3, 5, 7, 9…) to stop “cross talk” between adjacent discs. Optical stimulation of pseudo single grains was achieved using a ~50 W/cm² diode pump green laser (532 nm) at a setting of 90% power. The beam is directed onto a ~10 µm spot using a set of two mirrors and three lenses to steer and focus the laser within the chamber; these are motor driven (Bøtter-Jensen et
al., 2000). The heater plate is used to preheat the single-grain disc in order to eliminate thermally unstable electron traps within the grains. An Electron Tubes Ltd. 9235QA photomultiplier tube fitted with two 3 mm thick Hoya U-340 optical filters is used to detect the ultraviolet OSL emission from pseudo single grains (Bøtter-Jensen et al., 2000).

2.6.2 Data rejection criteria

A set of rejection criteria was used for samples in this study based on criteria established by Jacobs et al. (2003, 2006b). These criteria are required because of the variability of behavioural characteristics seen to be observed in individual grains from the same sample (e.g. Roberts et al., 1999; Duller et al., 2000). Inappropriate grains, based on the characteristics listed below, may affect the \( D_e \) distribution of the sample. By applying a set rejection criteria; this will systematically eliminate those grains from the distribution, thereby excluding them from the final age determination (Jacobs et al., 2006a).

Grains were rejected in this study for the following reasons;

1) if the OSL signals are weak (i.e. test signals \( T_N \) less than 3 times the instrumental background),

2) if the recuperation is high (i.e. \( L_X/T_X \) for 0 Gy dose is greater than 5% of \( L_N/T_N \)),

3) if the recycling ratio is poor (i.e., more than 2 standard errors away from unity). The recycling ratio can be defined as using a repeat of the first regeneration dose to check whether the sensitivity-corrected OSL \( (L_X/T_X) \) was reproducible (Jacobs et al., 2006a, 2006b),

4) if the sensitivity-corrected natural signal \( (L_N/T_N) \) is greater than any of the sensitivity-corrected \( L_X/T_X \) ratios (i.e., it does not intersect the dose-response curve), or

5) if exposure to infrared stimulation causes significant loss of OSL signal (i.e. OSL-IR depletion ratios smaller than unity by more than two standard errors),
Following this process of elimination, the finally accepted grains were used to calculate the final age for the sample. The $D_e$ values were applied to appropriate age models and represented in a visual manner, i.e. radial plot, for graphical display.

2.7 OSL signal characteristics

OSL signal characteristics are seen using dose response curves and decay curves. These are known to vary based on grain to grain variation, across all sites (Bailey et al., 1997; Arnold et al., 2009). Although the reason for variability is not well understood, some of the differences may be the result of the depositional and erosional history of the sample or post-depositional circumstances such as intense heating of grains in fireplaces. For example, the more cycles of erosion and deposition, the more sensitive or ‘brighter’ the signal (Jacobs et al. 2008).

2.8 Single-grain $D_e$ distribution analysis

2.8.1 Radial plots

The use of radial plots provides a means of visually evaluating the $D_e$ distribution of a sample by examining the overall patterning and precision of the individual grains (Galbraith 1988, 1990). A series of independent $D_e$ values obtained from individual grains are displayed in an easily identifiable manner that allows the $D_e$ values to be compared with the target event. Galbraith et al. (1999) notes that radial plots also allow the spread in the $D_e$ values to be represented quantitatively.

A basic understanding of reading a radial plot is as follows. The $D_e$ value for a grain is read by extending a straight line from the origin of the standardised estimate axis through the data point and onto the radial axis on the right-hand side; the intercept is known as the $D_e$ value (Galbraith, 1999). The range of precision can be easily observed as the imprecise grains fall to the left and the higher precision grains fall towards the right; this is read along the relative error axis bar on the bottom of the plot. The term “overdispersion” is used for
the spread in $D_e$ values that is larger than can be explained by the $D_e$ measurement errors alone. If the $D_e$ values for each grain are consistent within the measurement error then 95% of them should fall within the 2 standard error margin. If this should occur, then a single-age population can be assumed (Galbraith et al., 1999). It should be noted that relative precision reflects the errors of dose response curve fitting, in addition to photon counting statistics, background subtraction, and an instrumentation reproducibility uncertainty of 2% per OSL measurement (Duller, 2007).

### 2.8.2 Overdispersion

Overdispersion, also termed ‘scatter’, is known to vary in samples from geological and archaeological deposits (Arnold et al., 2009). The overdispersion of $D_e$ values has been interpreted to result from differences in the beta dose received by individual grains as well as the inadequate bleaching of sediments prior to burial (Olley et al., 2004; Arnold et al., 2009). The grain to grain variation in luminescence can also affect the overdispersion of samples. By using the single-grain process, this removes grains with abnormal characteristics prior to determining a final age. This might otherwise result in over- or under-estimates of the true burial age (Jacobs et al., 2006a, 2006b, 2006c; Galbraith et al. 2005). Following the removal of aberrant grains, $D_e$ values may be calculated and any residual overdispersion is assumed to be the result of depositional or post-depositional processes. Based on Jacobs et al. (2006a, 2006b, 2006c) there are four types of $D_e$ distribution:

1) $D_e$ distributions for grains that have been well bleached prior to deposition and have remained undisturbed since then.

2) Partially bleached $D_e$ distributions occur when grains are insufficiently bleached prior to burial and contain a residual charge. Grains with the smallest $D_e$ values are assumed to be the best bleached.

3) ‘Mixed’ $D_e$ distributions are those that display one or more discrete dose components. This is common in samples where well bleached grains with different burial histories from adjacent layers have been mixed after deposition. This is
observed in archaeological sites, and ages can be calculated assuming, for example, that the dose population represented by the greatest proportion of grains are representative of the target event.

4) ‘Scattered’ $D_e$ distributions display a range of $D_e$ values, explicable in terms of beta-dose heterogeneity. This is typical of samples that have been fully bleached prior to deposition and exposed to variable beta-dose rates after burial (Jacobs et al., 2008).

The ideal $D_e$ distribution pattern is when majority of grains fall within 2 standard errors of the mean (the 95% confidence interval). This indicates a typical well bleached sample. When considering the depositional environment of the site, it is best to have knowledge in regards to site stratigraphy and formation, to properly interpret the single-grain $D_e$ distributions.

### 2.8.3 Age models

When integrating site processes with the estimate of overdispersion it is important to decide which age model to use. The age model is necessary to combine all the individual $D_e$ values in an appropriate fashion to obtain a single $D_e$ estimate to be used to obtain an age estimate for the sample. The Central Age Model (CAM) is most appropriate for well bleached samples (2.8.2) (Galbraith et al., 1999; Rhodes, 2011). This method of analysis assumes all $D_e$ values are centred on the weighted mean. For samples with high overdispersion values, e.g. >10%, an indication of grain mixing or incomplete zeroing may be assumed. The appropriate age model will vary depending upon the sample; this will be discussed within the site-specific chapters (3-5).

### 2.9 Single-grain procedure used on samples in this study

All samples in this study used a modified version of SAR, tailored to the individual sample requirements, for pseudo single-grain dating purposes (Table 2.1). Changes to the single-grain procedure incorporates a change in the test dose preheat temperature from
200°C/10 s to 180°C/10 s following all test dose measurements, see Table 2.2 (Jacobs et al., 2006b,c).

An initial preheat of 180°C/10 s was used for all samples prior to the measurement of the natural (L_N) and regenerative dose (L_X) signals. Not all samples underwent the same set of regenerative and test doses, due to the difference in grain to grain variability among the three sites (see individual site chapters). All samples received a cutheat of 180°C/5 s, before being stimulated by a green laser at 125°C for 2 s at 90% power. To ensure that there was no feldspar contamination, at the end of the sequence all grains were given a repeat regenerative dose and then exposed to 40 s of infrared radiation at room temperature before optical stimulation with the green laser. The resulting L_X/T_X was compared with the previous L_X/T_X, allowing the OSL-IR depletion ratio to be calculated (Duller, 2003). Refer to Table 2.1 for pseudo single-grain procedure used in this study.

By applying the SAR procedure to single grains, dose response curves were fitted to the regenerative-dose data by using the following equation:

\[ I = I_0 + I_{max} (1 - e^{-D/D_0}) + k.D \]

This equation is used to fit dose response curves to a saturated exponential or a saturating exponential function with an extra linear term (Aitken, 1998).

### 2.10 Environmental dose rate determinations

To gain high-quality age estimates an estimation of the environmental dose rate is necessary. The environmental dose rate is produced from natural sources of ionizing radiation, which comes in four forms. These are; i) alpha (α) from the decay chain elements \(^{238}\text{U}\) and \(^{235}\text{U}\), and \(^{232}\text{Th}\), ii) beta (β) from \(^{238}\text{U}\) and \(^{235}\text{U}\), \(^{232}\text{Th}\) and \(^{40}\text{K}\) and \(^{87}\text{Rb}\), iii) gamma (γ) from \(^{238}\text{U}\) and \(^{235}\text{U}\), \(^{232}\text{Th}\) and \(^{40}\text{K}\) and iv) cosmic radiation. The radioactive elements \(^{238}\text{U}\),
$^{235}$U and $^{232}$Th, also have a series of radioactive daughter products. The environmental dose rate of a sample can be divided into either internal or external dose rates:

1) The internal dose rate represents radiation coming from within the sedimentary grains themselves as a result of alpha and beta radiation. The internal dose rates of quartz sediments are generally low, with low levels of uranium, thorium and potassium (Rhodes, 2011).

2) The external dose rate is derived from alpha, beta and gamma radiation in the bulk sediment matrix surrounding the grain (and also from cosmic rays). This component is dominant in quartz and may be calculated using various techniques such as thick-source alpha counting (TSAC), X-ray fluorescence (XRF), high resolution gamma spectrometry (HRGS), instrumental neutron activation analysis (INAA) and Geiger-Müller beta counting (GMBC) (Rhodes, 2011).

TSAC and XRF provide radionuclide concentrations that need to be converted into dose rates, whereas beta counting provides a direct measurement of the external beta dose rate. Each form of nuclear radiation has a different penetration range. Alpha particles ionise heavily and result in a rapid loss of energy within ~0.02 mm of the emitting nucleus (Aitken, 1998). The external alpha radiation contribution may be removed by etching the outermost 0.02 mm rind of the mineral grain using hydrofluoric (HF) acid. Ionising radiation in the form of gamma and beta rays penetrates approximately 30 cm and 2-3 mm respectively, as compared to cosmic-ray radiation flux which can penetrate tens of metres.

The methods used in calculating the total dose rate is as follows: GM-25-5 beta counting in conjunction with in situ gamma spectrometry gives a direct measurement of the beta and gamma dose rates. The conversions of TSAC and XRF concentrations provide estimates on U, Th and K (Adamiec and Aitken, 1998; Guerin et al., 2011). Beta counting plus TSAC will give U, Th and K (via subtraction), while TSAC plus XRF give U, Th and K. Multiple, independent measurements of U, Th and K are also obtained from these various methods.
2.10.1 Thick-source alpha counting (TSAC)

The method of TSAC focuses on the combined contribution of alpha particles from the elements U and Th; this is used to calculate the beta and gamma dose rates however an independent estimate of K is required (Aitken, 1985; Feathers, 2012). A flattened alpha-thick layer of prepared sample material is placed on top of a zinc sulphide (ZnS) phosphor screen in a perspex sample holder. The perspex holder fits on top of a photomultiplier tube (PMT) within the alpha counter. As alpha particles from the sediment sample strike the ZnS screen, scintillations are produced. The scintillations cause emissions of photoelectrons, which are detected by the PMT. The total number of scintillations per unit area of ZnS screen is reflected to the U and Th concentration per sample. Around 3% of the counts in the $^{232}$Th decay series are termed ‘pairs’, which are compared with the total number of alpha particles counted (Huntley and Wintle, 1981; Aitken, 1985). From this, the separate Th and U concentrations in the sample can be determined and the beta and gamma dose rates established (Huntley and Wintle, 1981; Aitken, 1985).

2.10.2 High-resolution gamma spectrometry (HRGS)

Five sediment samples were measured using high-resolution gamma-ray spectrometry (HRGS); this was done to investigate the equilibrium status of the decay chain elements $^{238}$U and $^{232}$Th and obtain an independent estimate of $^{40}$K. Estimates will be compared against beta and gamma rates. Olley et al. (1996, 1997) describes the activities of $^{238}$U, $^{226}$Ra and $^{210}$Pb in the $^{238}$U decay chain, $^{228}$Ra and $^{228}$Th in the $^{232}$Th decay chain, and the procedures and equipment for measuring $^{40}$K as dried powdered subsamples of sediment.

2.10.3 GM-25-5 beta counting

Beta counting results in the measurement of U, Th and K, using a Risø GM-25-5 beta counter (Bøtter-Jensen and Mejdahl, 1985, 1988). The equipment contains five Geiger-Müller (GM) detectors and a common guard. Five sample positions are located on a sample
slide directly underneath each of the GM detectors. To ensure outside interference is minimised, the unit is covered by lead shielding, which also reduces the background radiation from cosmic rays.

The sample emits a beta particle, which produces a pulse that is recorded by the GM detector. The beta dose rate was calculated manually by subtracting the counts obtained from the magnesium oxide (Mg0) from the beta pots and the Nussi (i.e. a standard with a known beta dose rate), to obtain a background-corrected count rate for each of the replicate beta pots. The background-corrected count rate for each sample was then divided by the background-corrected count rate of the Nussi, and then multiplied by the known beta dose rate of the Nussi to obtain a direct estimate of the beta dose rate for the sample.

2.10.4 In situ gamma spectrometry

Archaeological sites generally contain artefacts within multiple stratigraphic layers. These layers commonly have different levels of radioactivity, which is when in situ (or field) gamma spectrometry is most useful (Aitken, 1985). In situ gamma spectrometry measures the gamma-ray flux from a 60 cm diameter of sphere, around the drilled hole.

The gamma detector is fitted into holes made for OSL sample collection. In situ gamma emissions were measured using an Ortec Digidart gamma spectrometer with an attached detector consisting of a Nal crystal one inch in diameter. To obtain an accurate gamma dose rate, measurements were made for one hour using the ‘threshold’ calibration technique outlined by Mercier and Falguères (2007).

2.11 Dose rates

Dose rates were calculated using a variety of methods, as discussed within the individual site-specific chapters (3-5). Beta and gamma dose rates were calculated using direct beta counting and in situ gamma spectrometry for sites Al-hatab and Ghazal. A
combination of TSAC and beta counting was used for Khumseen, as field measurements for the gamma dose rate were not available.

2.11.1 Moisture content corrections

Water within a sedimentary deposit is an important variable when determining the dose rate, for it absorbs high amounts of radiation (Jacobs and Roberts, 2007). A 1% decrease in water content produces an increase in dose rate of approximately 1%, which will in turn decrease the age by a 1% estimate (Jacobs and Roberts, 2007). To determine the correction for beta and gamma absorption, an entire moisture content of the sample over the entire burial history is required. This is done by estimating the present day water content of the sample and the effect that long-term climatic variations may have induced on the site and/or area (Cohen et al., 2011; Jacobs and Roberts, 2007).

In calculating the moisture content, the dry weight is subtracted from the wet weight and the difference is then divided by the dry weight to obtain the moisture content. The present-day field water content of the samples must be adjusted to account for longer-term climatic variability. In order to account for the annual monsoonal rainfall of the region, the samples in this study were given a value of 3%, with a relative error of 50% at 1σ (i.e. 3.0 ± 1.5%) to allow for the fact that the samples were collected during the dry season. The 3.0 ± 1.5% value, therefore, covers the range of measured field value (0.1-2.6%), as well as the likely range due to modern-day and past climatic changes.

2.11.2 Grain size corrections

When calculating the beta dose rate, corrections must be made for the size of the individual grain. Grains are sieved to either 90-125 µm or 180-212 µm in diameter. Size must be taken into account when determining the different levels of radiation imposed upon a grain. Large grains (180-212 µm or >212 µm) do not allow complete penetration of the beta particles, therefore HF etching was used to remove the outer rind of each quartz grain. As a
result of these effects, a small modification to the beta dose rate is required. The attenuation factors used for the correction of the beta dose rate were obtained from Mejdahl (1979).

2.12 Cosmic-ray dose rate

The cosmic-ray dose rate is dependent on the sample depth below the ground surface, sediment density, altitude, geomagnetic latitude, and water content, and can be estimated using the formulae and methods of Prescott and Hutton (1994) and Readhead (1987). The contribution of cosmic rays to the environmental dose rate is 10% or so of the total dose rate (Aitken, 1985; Prescott and Hutton, 1988). The in situ rate at which cosmic rays are absorbed decreases with increasing depth below the ground surface, but site altitude, longitude and latitude also have an effect. The depositional rate of sediment at the site is important when calculating the cosmic ray dose rate; the site stratigraphy either determines a slow deposition or rapid deposition. The samples burial depth is also used when calculating the cosmic-ray dose rate (Lian et al., 1995; Munyikwa, 2000).

2.13 OSL age determination

Once the internal alpha and external beta, gamma and cosmic-ray dose rates have been obtained, they are then combined and applied to the OSL age equation given earlier (Equation 1), along with their corresponding $D_e$ values, in order to obtain the burial age of the sediment grains. The age is considered the time elapsed since the grains last saw sunlight. The final dose rates and age determinations for all samples will be discussed in the site-specific chapters.
2.14 Methods used for samples in this study

2.14.1 Thick-source alpha counting (TSAC)

All measurements were made at the University of Wollongong, using a Daybreak-538 thick-source alpha counter. Firstly a measurement of the alpha background count rate was made by placing two ZnS screens face to face in the perspex holder for a total of 24 hours. Immediately after this, a background screen is used for the sample, with the background count rate for each screen taken as half the measured value of the pair (Huntley and Wintle, 1981). A flattened alpha thick layer of prepared sample material is evenly distributed on top of the ZnS screen and measured as an ‘unsealed’ sample, enabling air to get into the perspex holder and resulting in the release of radon gas, which when sealed and unable to escape may lead to significant over-counting (Aikten, 1985). All samples were measured to obtain a count of least 2000.

2.14.2 GM-25-5 beta counting

The preparation of beta pots for beta counting followed a set laboratory procedure. Measurements were made on three pots of the same sample, each pot containing equal amounts of evenly distributed sediment. The sample was prepared by being dried in a 100°C oven and placed in a ball mill and ground into a fine powder. These are covered with polyethylene film and placed in the detectors. All measurements were made over a period of 24 hours (24 hr count cycles of 1 hour each), along with a standard of known beta dose rate (Nussi) and a sample of Mg0 to estimate instrumental background.

2.14.3 In situ gamma spectrometry

In situ gamma spectrometry was conducted by Richard Roberts at Al-hatab (ALH) and Ghazal (TH47). Field measurements could not be made, however, so beta counting and TSAC were used to estimate the gamma dose rates for the TH50 samples.
2.14.4 Moisture content determinations

Moisture contents were determined from the small bags of sediment collected from the back of each OSL sample hole. The process was started by weighing an empty beaker to ‘zero’ the scales. The sample was then weighed before being saturated with water and weighed again to obtain the ‘saturated weight’. Following this, the sample was placed in a 100˚C oven until dry, and then weighed to measure the ‘dry weight’. The mass of water in the field and the saturated conditions were then calculated by subtraction. For moisture calculations and corrections see section 2.11.1

2.15 Chapter summary

This chapter discussed the methods and procedures used when carrying out OSL dating. All samples in this study were prepared in the same systematic manner as described in this chapter for pseudo single-grain analysis. Details of OSL dose response curves, decay curves and environmental dose rates are discussed in the individual site-specific chapters (3-5). The HRGS data were not used to calculate the OSL ages, and are therefore summarised in Appendix 1 for all three sites.
**Fig 2.1** Latent luminescence signal as a function of time. Diagram shows ionising radiation building up as energy within a crystalline material that is a quartz grain. Upon stimulation by light (OSL), energy is released in the form of luminescence in a measurable intensity that is proportional to age (Rhodes 2011).
Fig 2.2 a) Flow chart of the conventional SAR procedure for single (multiple-grain) aliquots (Taken from Murray and Wintle, 2000). b) A hypothetical sensitivity-corrected dose response curve using the SAR procedure (Jacobs et al., 2006c)
Fig 2.3 a) Schematic of a typical OSL/IRSL detection system, based on the Risø TL-DA-20 luminescence reader. The sample is held at raised temperatures not exceeding 400°C and stimulated blue or IR light emitting diodes (LEDs). UV luminescence emission is detected through a U340 filter by the photomultiplier tube (PMT) (Rhodes, 2011). b) Example of a single-grain disc containing 10 x 10 holes. Each hole is 300 µm deep and 300 µm in diameter. This is then loaded below the photomultiplier tube and used to measure the intensity of the luminescence signal.
### MODIFIED SAR PROCEDURE FOR SINGLE GRAINS USED IN THIS STUDY

<table>
<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Give dose (0 Gy if Natural)</td>
<td>–</td>
</tr>
<tr>
<td>2</td>
<td>Preheat at 180°C for 10 s</td>
<td>–</td>
</tr>
<tr>
<td>3</td>
<td>Stimulate using focused green (532 nm) laser at 125°C for 2 s</td>
<td>$L_N$ or $L_X$</td>
</tr>
<tr>
<td>4</td>
<td>Test dose ($T_0$)</td>
<td>–</td>
</tr>
<tr>
<td>5</td>
<td>Preheat to 180°C for 5 s</td>
<td>–</td>
</tr>
<tr>
<td>6</td>
<td>Stimulation at 125°C for 2 s using green laser light</td>
<td>$T_N$ or $T_X$</td>
</tr>
<tr>
<td>7</td>
<td>Regenerative dose ($R_X$)</td>
<td>–</td>
</tr>
<tr>
<td>8</td>
<td>Return to step 1 and repeat until all $R_X$ have been given</td>
<td>–</td>
</tr>
<tr>
<td>9</td>
<td>Give zero dose ($R_X=0$) and repeat steps 1-6</td>
<td>Check for recuperation</td>
</tr>
<tr>
<td>10</td>
<td>Repeat dose point ($R=0$) and repeat steps 1-6</td>
<td>Check recycling ratio</td>
</tr>
<tr>
<td>11</td>
<td>Repeat dose point ($R=R_{in}$)</td>
<td>–</td>
</tr>
<tr>
<td>12</td>
<td>IRSL at 50°C for 40 s using infrared diodes</td>
<td>–</td>
</tr>
<tr>
<td>13</td>
<td>Repeat steps 1-5</td>
<td>Check for OSL-IR</td>
</tr>
</tbody>
</table>

*Table 2.1* The modified SAR procedure used to measure quartz grains (90-125 µm in diameter) from Ghazal, Khumseen and AL-hatab in this study.
### DRT

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Bleached/Natural</th>
<th>Preheat Temp</th>
<th>Cutheat Temp</th>
<th>Given dose (sβ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ALH-2(SG)DRT (1-19) Run 1</td>
<td>Bleached 90-125µm</td>
<td>180°C/10 s</td>
<td>180°C/10 s</td>
<td>400 sβ</td>
</tr>
<tr>
<td>ALH-2(SG)DRT (21-39) Run 2</td>
<td>Bleached 90-125µm</td>
<td>200°C/10 s</td>
<td>160°C/10 s</td>
<td>400 sβ</td>
</tr>
</tbody>
</table>

**Table 2.2** Dose recovery test (DRT) preheat and cutheat temperatures given to ALH-2.

1. Bleached dose using natural sunlight exposure to stimulate the grains prior to a given known laboratory dose (Murray and Roberts, 1998; Roberts et al., 1999).

2. Preheat temperatures remove charge from thermally unstable but optically sensitive traps.

3. Cutheat temperatures is generally high enough to remove signals that may interfere with the OSL measurement.

4. Given dose should be as close as possible to the expected natural signal.
Chapter 3
Chapter 3  Ghazal (TH47)

3.1 Introduction

A brief introduction to the depositional history of the site will be provided in this chapter, with geological and archaeological units, and previous dating methods. The single-grain OSL procedure will be used and the data will be presented in the form of decay curves, dose response curves and radial plot of $D_e$ values. This chapter will also discuss the effect of operator variance on $D_e$ values. Following this, the $D_e$ distributions obtained after the rejection process will be examined and according to the shape and contextual information (e.g. overdispersion value), the $D_e$ distribution will be interpreted and an appropriate statistical model applied (i.e. Central Age Model). The latter will be used to combine the individual $D_e$ values obtained for each sample into a single representative $D_e$ value that will be used to calculate the final age of each sample. The environmental dose rates will be provided also be provided. This will be discussed in regards to site interpretation and how the final ages compare against previous independent age controls.

3.2 Background information

Ghazal rock shelter, also known as TH47, is located approximately 0.5 km west of Al-hatab (Fig 1.1b). The trench was first excavated in 2010 by Dhofar Archaeological Project (DAP), with work continuing in 2011 (Fig 3.1). The trench was divided in a systematic manner, i.e. in a grid fashion, with dimensions of 1 m$^2$. All sediments were sieved using a 5 mm mesh (Hilbert et al., 2012). A total of 15 squares were excavated down to bedrock, exposing six geological horizons (GH1-6) from top to bottom (Fig 3.2b).
3.2.1 Lithic analysis

Lithic assemblages were located in stratigraphic layers GH2 and GH4. The manufacturing of blades occurred using local chert, showing similar blade formation to the NLT of Al-hatab (Level 2). These are important as they depict a different method of blade production categorised under the NLT (Hilbert et al., 2012). They represent parallel blade formation, rather than the typical convergent blade formation (Fig 3.3).

3.3 Stratigraphy

A geological summary of the sedimentary succession is provided in Table 3.1. The roof of the rock shelter is formed predominately by calcrete capping on an uphill slope. This extends over part of the rock shelter and is approximately 30-50 cm thick. There is evidence of calcrete blocks that have broken off from the roof and fallen into the depression. In situ decomposition of blocks may be of some concern when examining sediment mixing in regards to OSL dating; contamination of samples by roof spall may be possible (see section 2.6). In such circumstances, single-grain methods provide a means of identifying the contaminated grains and rejecting them before age determination.

3.4 OSL dating at Ghazal

3.4.1 Sample collection and preparation

A total of four sediment samples were collected for OSL dating (TH47-1, TH47-2, TH47-3 and TH47-4). Three of the samples were collected from the excavation trench in the first field season (2010), and sample TH47-4 was collected the following year.

Two plastic tubes of each sample were collected (see section 2.4.1) on the eastern side of the trench, adjacent to one another and separated laterally by 9 cm (TH47-1), 8 cm (TH47-2), 8 cm (TH47-3) and 11 cm (TH47-4). The OSL samples were collected from the following geological units and depths.
TH47-4: 20 cm depth below ground level; geological Unit GH1
TH47-1: 35 cm depth below ground level; geological Unit GH3
TH47-3: 40 cm depth below ground level; geological Unit GH5
TH47-2: 56 cm depth below ground level; geological Unit GH5

The OSL samples were collected from the units that did not contain the artefacts (Units GH2 and GH4). Two samples were collected from Unit GH5, which was not continuous across the site; this can be seen in Fig 3.2a.

For each sample, a bag of sediment was collected for beta counting and moisture content, following the procedure outlined in section 2.4. These samples were collected during the dry season. The field moisture content was measured and gave values of 0.1% to 5.1%. A value of 3.0 ± 1.5% was used for dose rates and age calculations; the uncertainty covers the likely variation in water content between the dry season, the monsoon season, and longer term variations over the period of the site’s depositional history.

In situ gamma spectrometry measurements were made at the location of sample TH47-3. Due to a gamma spectrometer malfunction in the first field season, new holes were made in 2011 on the southern wall of the excavation pit. In this section the stratigraphy was still intact and very similar to the original face, and measurements were made here of the gamma dose rates for TH47-1, TH47-2 and TH47-3. Sample preparation followed the same procedures as presented in section 2.4.

3.4.2 Single-aliquot measurements

Previous dating for this site has been done using the single-aliquot method as outlined in section 2.5. Single-aliquot measurements were made on multi-grain aliquots by Richard Roberts from the University of Wollongong. These data sets have been re-analysed for this study in order to compare them against the pseudo single-grain data sets and to indicate why single-grain analysis is needed at this site.

The data sets that were re-analysed are those for samples TH47-1 and TH47-3. Both samples accepted 10 out of 39 single aliquots measured; Fig 3.4a shows OSL decay curves
for both samples. Both samples show some amount of spread in the $D_e$ values (Fig 3.4b) with overdispersion values of $8.4 \pm 0.6\%$ (TH47-1) and $11.2 \pm 0.9\%$ (TH47-3). Due to the paucity of material that passed the rejection criteria (as outlined in section 2.6.2), these data sets were not considered to be reliable for age determination and the potential remained for roof spall contamination to inflate these ages. From these results it was considered necessary to do pseudo single-grain analysis in order to obtain a more reliable age.

### 3.4.3 Dose recovery data

Due to the proximity of Ghazal to Al-hatab and the same source material of the sediments, we did not conduct dose recovery tests on grains from this site, but instead used the same temperature combination as was determined appropriate for Al-hatab ($180^\circ C/10\ s$ and $180^\circ C/5\ s$).

### 3.5 Single-grain measurements

#### 3.5.1 OSL decay curves

OSL decay curves show the variation in grain-to-grain brightness and signal luminescence intensity. The shape of the decay curves indicate that they are dominated by the ‘fast’ component of quartz OSL for which the SAR procedure was designed (Bailey et al., 1997; Jacobs and Roberts, 2007). Two typical examples of decay curves are given in Fig 3.5 for a bright grain and a dim grain. All decay curves were measured either following a preheat temperature of $180^\circ C/10\ s$ (natural and regenerative dose) or a cutheat temperature of $180^\circ C/5\ s$ (test dose). The duration of optical stimulation was $2\ s$ (Fig 3.5a). It can be seen that the relative proportion of the fast component decreases with increasing bleaching time (i.e. time that the grain is exposed to light). The curve will therefore become flatter as the fast-component electron traps are emptied (Bailey et al., 1997). The fast component is the component of choice in OSL dating, as the trapped electrons only take a
few seconds of sunlight exposure to be emptied, so the SAR procedure primarily focuses on the fast component in quartz grains. This component is also stable on geological time scales (Jacobs and Roberts, 2007).

### 3.5.2 OSL dose response curves

OSL dose response curves were fitted with either a saturating exponential or a saturating function with an extra linear term. As discussed in section 2.7, individual grains do not respond to doses in an identical fashion, therefore grains were measured with a variety of regenerative doses and a fixed test dose of 20 Gy. Sample TH47-1 was given regenerative doses of [40, 20, 40, 80, 10, 0 and 40 Gy]. TH47-2 was given doses of [40, 20, 40, 80, 120, 0 and 40 Gy], and sample TH47-3 and TH47-4 were given doses of [13.3, 6.7, 20, 40, 0 and 13.3 Gy]. The error on the $D_e$ was determined by using Monte Carlo stimulation in Analyst MC; this was used for all samples in this study.

### 3.6 $D_e$ determination

The determination of $D_e$ values for single-grain analysis followed the procedure set out in section 2.5. The pseudo grains that passed the set of rejection criteria outlined in section 2.6.2 showed the characteristics of a typical bright grain (Fig 3.5a), while those that were rejected showed similar properties to the dull grain in Fig 3.5b. Initially, 4000 grains were measured from all four samples, and a total of 3324 grains were rejected (Table 3.2). The majority of grains were rejected based on the inability of the OSL to be discerned above background noise. For TH47-4, the rejection process had been modified after none of the grains survived the criteria. By removing the ‘recuperation’ criterion, 191 grains were accepted for $D_e$ estimation.

A cumulative light sum plot was used to show the distribution of signal intensity among the grains (Duller et al., 2000). Grains that passed the rejection criteria are plotted in Fig 3.6, which shows the proportion of the total light sum that originates from the specified
percentage of grains from all four OSL samples. Fig 3.6 indicates that 50% of the grains account for 90% of the OSL intensity, so these samples are not dominated by the OSL, but from a few bright grains. It should be remembered that in this study, each hole contained between 3 and 5 grains which may explain the higher proportion of grains contributing to the total light sum. The cumulative light sum plot does not account for the effects of pseudo single grains as it reflects the grain-averaging effects of having more than one grain in each hole, and therefore is an increased probability of each hole containing at least one luminescent grain. True ‘single-grain’ studies indicate only 10% or fewer of the grains account for 90% of the emitted OSL (Arnold et al., 2012), but large variations depending on the geographical location and transport history of the grains have been reported previously.

3.7 Analysis and interpretation of $D_e$ distributions

The analysis of pseudo single-grain $D_e$ distributions were displayed in the form of radial plots (Fig 3.7). A comparative study of $D_e$ determination by the author (LL) and Zenobia Jacobs (ZJ) was done to assess the extent of operator variance. The radial plots show that the grains have $D_e$ values with a large amount of scatter.

The $D_e$ overdispersion calculated by the author (LL) for sample TH47-1 is 40 ± 4%. The majority of grains (79%) are self-consistent with the weighted mean $D_e$, which was calculated using the Central Age Model of Galbraith et al. (1999). Data consistent with the mean at $2\sigma$ falls within the grey band. The same range of $D_e$ values, and the same overdispersion value, was calculated by ZJ for sample TH47-1. Samples TH47-2 and TH47-3 are from the same stratigraphic layer, and present similar scatter patterns, as can be seen in Fig 3.7. TH47-2 has an overdispersion value of ~70% whereas TH47-3 has an overdispersion value of ~50%. The extent of over-dispersion is similar for both operators; therefore this difference is sample-specific and not operator-specific. The same is true for sample TH47-4 which produced a range of similar $D_e$ values and overdispersion estimates (37 ± 4%) from both operators (Fig 3.7).

The decomposed Ghazal deposit contains calcrete blocks within the sediments (see section 3.2) which may account for some of the higher $D_e$ values. Calcrete nodules may
account for some of the lower $D_e$ values resulting in a higher $D_e$ distribution. In view of these possible sources of contamination, which may explain the higher and lower $D_e$ values in each of the distributions, the burial ages of the Ghazal samples were calculated from the majority of grains that fall within the grey bands. The Central Age Model (Galbraith et al., 1999) was used to calculate the weight mean $D_e$ for age determination. This model takes into account the measured overdispersion and reflects this spread in the uncertainty on the mean $D_e$. The proportion of grains that fall within each of the grey bands are 79% (TH47-1), 59% (TH47-2), 67% (TH47-3) and 83% (TH47-4); this was established by the author.

3.8 Environmental dose rates

Beta, gamma and cosmic-ray dose rates were estimated using the techniques described in section 2.9 along with the conversion factors from Guèrin et al. (2011).

The beta dose rates were measured using GM-25-5 beta counting for all four OSL samples (see section 2.14.3). This technique was used to provide a more accurate and direct measurement for the beta dose rate (Rhodes, 2011). The dry dose rate was adjusted for field moisture content to allow for the water content over the period of sample burial (see section 2.11.1). For individual beta dose rates, see Table 3.5.

The gamma dose rates were measured using an in situ gamma spectrometer for all four OSL samples. This is the preferred method for calculating the gamma dose rate, as well as any spatial non-uniformity of gamma emitters within 30 cm of the sample. The gamma dose rates were adjusted to account for the field water content for each sample. The gamma dose rate values are shown in Table 3.5. Gamma dose rates may be calculated using HRGS, TSAC and GM 25-5 beta counting measurements made for these samples. A slight difference between the lab based (HRGS and TSAC) and field measurements is shown in Table 5.4. This is indicative of the presence of calcrete cappings, and therefore field measurements are preferred. Gamma dose rates have been calculated using HRGS and TSAC and GM-25-5 beta counting; this can be seen for all four samples in Table 3.4. By calculating the gamma rates this way, we may determine if there is any variability present between the lab-based (TSAC and HRGS) and field measurements. The latter is preferable
due to the presence of weathered limestone which may affect the overall dose rate of each sample.

The cosmic-ray dose rates were calculated following Prescott and Hutton (1994) and adjusted for water content (Readhead, 1987). The roof of the rock shelter was included in the final cosmic-ray dose rate calculation as it obscured a clear view of the sky. The dose rate value for all OSL samples was $0.17 \pm 0.02$ Gy/ka. This assumes that the deposition in the rock shelter accumulated rapidly, so that the time-arranged burial depths of the samples are similar to their present burial depths.

### 3.8.1 Total dose rate

The total dose rates for OSL were calculated using the sum of the beta, gamma and cosmic-ray dose rates. Adjustments were made for grain size and moisture content of each sample and an internal alpha dose rate of $0.03 \pm 0.01$ Gy/ka was also included.

The total dose rates for samples TH47-1 to TH47-4 are listed in Table 3.5. In general, the total dose rates increase with depth from $0.66 \pm 0.03$ Gy/ka at 20 cm (TH47-4) to $1.39 \pm 0.07$ Gy/ka at 56 cm (TH47-2). High-resolution gamma-ray spectrometry (HRGS) data indicates that this increase is not because of time-dependant disequilibrium in the OSL $^{238}$U or $^{232}$Th decay chains, but most likely reflects the specific compositions (textural and mineralogical) of the different units from which the OSL samples were collected; see Appendix 1.

### 3.9 OSL ages

The final ages were calculated using the age equation in section 2.13. The age calculated for sample TH47-4 (GH1) is $7.1 \pm 0.5$ ka, which gives a minimum age for the NLT within the underlying Unit GH2. Sample TH47-1 is dated to $7.5 \pm 0.5$ ka; this gives a maximum age for Unit GH2 and a minimum age for Unit GH4, which also contains lithic artefacts. The age for sample TH47-3 is $9.7 \pm 0.7$ ka, and TH47-2 has an age of $8.2 \pm 0.7$ ka. The latter two OSL samples are more than 2 m apart laterally and provide a maximum age of
~10 ka for the lithic artefacts located in Unit GH4. Thus, the lithic artefacts of Ghazal fall in the narrow time range of around 7-10 ka ago, according to the pseudo single-grain analyses carried out in this study.

The pseudo single-grain ages for samples TH47-1 and TH47-3 can be compared to the previous single-aliquot ages of 10.5 ± 0.9 ka (TH47-1) and 9.2 ± 0.9 ka (TH47-3). There are a number of reasons why these multi-grain ages are older than the pseudo single-grain estimates, with the most obvious one being the incorporation of decomposed roof fall grains in the multi-grain aliquots. The presence of weathered limestone (calcrete cappings) within the rock shelter may have broken down over time causing calcrete nodules to become mixed in with the surrounding sediments; this essentially may filter down the stratigraphic profile. This may explain the increase in the spread in the data, essentially producing either an under- or over-estimate of the true burial dose.

### 3.10 Chapter summary

An outline of the site’s depositional and previous dating history has been given to provide contextual information on the site, which was used to explain the need for single-grain analysis. All four OSL samples were analysed by two different operators; this showed that the results were consistent between LL and ZJ and that the resulting $D_e$ distributions are not an outcome of the decisions and assumptions made by different operators. Results indicate that the samples contain many grains that have self-consistent $D_e$ values. Each sample burial age was calculated containing some younger and older grains. The older grains are thought to be derived from decomposed sediments from limestone rocks; this accounts for the slightly older ages obtained using multi-grain aliquots. The presence of contaminant grains are found on each aliquot, resulting in a slightly inflated age when compared to the pseudo single-grain results.
Fig 3.1 Rock shelter Ghazal and site topography (N 17.314483° E54.056617°) (Courtesy of Yamandu Hilbert).
Fig 3.2 a) Stratigraphic drawings of the site showing the locations of the OSL samples. Arrows indicate units where the NLT was found. b) Topographic landscape around Ghazal rock shelter (modified from Hilbert et al., 2012).
Fig 3.3  a) and b) indicate two examples of Leptolithics located at the site, showing methods of blade production; parallel blade formation rather than the typical convergent blade formation. Refit of core reduction modalities showing reattachment to a single platform (modified from Hilbert et al., 2012).
Fig 3.4a) Dose response curve for an aliquot from sample TH47-1 and TH47-3 and the inset OSL decay curve of an accepted bright aliquot. Taken from single-aliquot measurements that were re-analysed by author.

Fig 3.4b) Radial plot showing single-aliquot $D_e$ values; TH47-1 (8.4 ± 0.6%) and TH47-3 (11.2 ± 0.9%) overdispersion. The overdispersion is lower compared to the pseudo single-grain $D_e$ distributions shown in Fig 3.7.
Fig 3.5 a) Dose response curve for a grain from sample TH47-3 and the OSL inset decay curve of an accepted, bright grain (TH47-4). b) OSL decay curve of a typical dim grain from TH47-2.

Fig 3.6 Distribution of OSL signal intensity induced by a 20 Gy test dose from the accepted grains of all samples from Ghazal. Data are plotted as the proportion of the total light sum that originates from the specified percentage of luminescent grains (Duller et al., 2000).
Fig 3.7 Comparative study of $D_e$ values displayed in radial plots. The grey bands are centred on the Central Age Model estimates of mean $D_e$. OSL samples analysed by the author have overdispersion values of 40 ± 4% (TH47-1), 72 ± 5% (TH47-2), 51 ± 4% (TH47-3) and 37 ± 4% (TH47-4). The same values for the data analysed by ZJ are 40 ± 4%, 69 ± 5%, 50 ± 4% and 28 ± 4%, respectively.
Fig 3.8 Stratigraphic sequence showing the pseudo single-grain OSL ages for Ghazal samples: 7.1 ± 0.5 ka (TH47-4), 7.5 ± 0.5 ka (TH47-1), 9.7 ± 0.7 ka (TH47-3) and 8.2 ± 0.7 ka (TH47-2).
### Table 3.1 Geological units of Ghazal rock shelter, including archaeological findings and OSL samples (Based on Richard Roberts’ field notes).

<table>
<thead>
<tr>
<th>Units</th>
<th>Depositional history</th>
<th>OSL samples</th>
<th>Lithic evidence</th>
</tr>
</thead>
</table>
| GH 1  | Calcrete capping roof of rock shelter  
Light brown, sandy silt with granules (poorly sorted, not imbricated) | OSL TH47-4 | NLT stone tools |
| GH2   | Light brown, sandy silt with coarse material | OSL TH47-1 | NLT stone tools |
| GH3   | Light brown, silty sand with fine to medium grains | OSL TH47-2 + TH47-3 | - |
| GH4   | Light brown, sandy silt with coarse material | - | - |
| GH5   | Cream, fine grained silty sand; well sorted | - | - |
| GH6   | Brown sandy silt with granules. | - | - |
|       | White limestone bedrock. | - | - |
### Rejection Criteria applied for Ghazal grains

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Grains measured</th>
<th>Criterion 1</th>
<th>Criterion 2</th>
<th>Criterion 3 IR</th>
<th>Criterion 4 Recuperation</th>
<th>Criterion 5 removal of Recuperation</th>
<th>No. grains rejected 2+ criteria</th>
<th>Total no. rejected</th>
<th>Total no. accepted</th>
<th>Final no. grains</th>
<th>% finalised</th>
</tr>
</thead>
<tbody>
<tr>
<td>TH47-1</td>
<td>1000</td>
<td>626</td>
<td>293</td>
<td>337</td>
<td>412</td>
<td>-</td>
<td>668</td>
<td>857</td>
<td>143</td>
<td>132</td>
<td>13%</td>
</tr>
<tr>
<td>TH47-2</td>
<td>1000</td>
<td>566</td>
<td>281</td>
<td>281</td>
<td>426</td>
<td>-</td>
<td>554</td>
<td>840</td>
<td>160</td>
<td>146</td>
<td>15%</td>
</tr>
<tr>
<td>TH47-3</td>
<td>1000</td>
<td>584</td>
<td>369</td>
<td>326</td>
<td>396</td>
<td>-</td>
<td>675</td>
<td>825</td>
<td>175</td>
<td>166</td>
<td>17%</td>
</tr>
<tr>
<td>TH47-4</td>
<td>1000</td>
<td>416</td>
<td>287</td>
<td>523</td>
<td>1000</td>
<td>0</td>
<td>2226</td>
<td>802</td>
<td>198</td>
<td>191</td>
<td>19%</td>
</tr>
<tr>
<td>Total</td>
<td>4000</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4000</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Table 3.2** Summary of grains rejected according to the formal rejection criteria of (2003, 2006a, 2006c) for Ghazal (TH47) samples.

1. OSL signals are weak (T_N signal less than 3 times the instrumental background)
2. Recycling ratio is poor (i.e. more than 2 standard errors away from unity)
3. Infrared simulation causes significant loss of OSL signal (i.e. OSL-IR depletion ratios smaller than unity by more than two standard errors)
4. Recuperation is high (L_0/T_N for 0 Gy dose is greater than 5% of L_0/T_N)
5. No. of grains rejected following the removal of recuperation.
6. Total no. rejected grains plus total no. accepted grains will equal the no. of grains measured.
7. Total no. of accepted grains for dating.
8. Percentage of finalised grains used for age determination.
### Operator Variance

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Lauren Linnenlucke</th>
<th>Zenobia Jacobs</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$D_e$ (Gy)</td>
<td>Age (ka)</td>
</tr>
<tr>
<td>TH47-1</td>
<td>$6.00 \pm 0.30$</td>
<td>$7.5 \pm 0.5$</td>
</tr>
<tr>
<td>TH47-2</td>
<td>$11.40 \pm 0.80$</td>
<td>$8.2 \pm 0.7$</td>
</tr>
<tr>
<td>TH47-3</td>
<td>$11.80 \pm 0.6$</td>
<td>$9.7 \pm 0.7$</td>
</tr>
<tr>
<td>TH47-4</td>
<td>$4.65 \pm 0.19$</td>
<td>$7.1 \pm 0.5$</td>
</tr>
</tbody>
</table>

**Table 3.3** Operator variation showing ages and $D_e$ values obtained by two analysts on samples from Ghazal (TH47) by LL and ZJ.

### Gamma Comparison

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Gamma (in situ)</th>
<th>Gamma (HRGS)</th>
<th>Gamma (TSAC + GM 25-5 Beta counting)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TH47-1</td>
<td>$0.24 \pm 0.01$</td>
<td>$0.23 \pm 0.01$</td>
<td>$0.28 \pm 0.02$</td>
</tr>
<tr>
<td>TH47-2</td>
<td>$0.48 \pm 0.01$</td>
<td>$0.41 \pm 0.02$</td>
<td>$0.48 \pm 0.01$</td>
</tr>
<tr>
<td>TH47-3</td>
<td>$0.24 \pm 0.01$</td>
<td>-</td>
<td>$0.47 \pm 0.02$</td>
</tr>
<tr>
<td>TH47-4</td>
<td>$0.20 \pm 0.01$</td>
<td>-</td>
<td>$0.19 \pm 0.02$</td>
</tr>
</tbody>
</table>

**Table 3.4** Gamma comparison of field gamma measurements (column 2) against laboratory gamma measurements (column 3) based on high resolution gamma spectrometry and a combination of thick source alpha counting and GM-25-5 beta counting (column 4).
<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Sample location (Lat. Long.)</th>
<th>Sample depth (cm)</th>
<th>Field water content (% dry mass)</th>
<th>γ dose rate (Gy/ka⁻¹)</th>
<th>β dose rate (Gy/ka⁻¹)</th>
<th>Cosmic-ray dose rate (Gy/ka⁻¹)</th>
<th>Total dose rate (Gy/ka⁻¹)</th>
<th>Dₑ (Gy)</th>
<th>σₑ (%)</th>
<th>Age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TH47-4</td>
<td>N 17° 31’ 44.83” E 54° 05’ 66.17”</td>
<td>20</td>
<td>3.0 ± 1.5 (0.8)</td>
<td>0.20 ± 0.01</td>
<td>0.25 ± 0.02</td>
<td>0.17 ± 0.02</td>
<td>0.66 ± 0.03</td>
<td>4.65 ± 0.19</td>
<td>37 ± 4</td>
<td>7.1 ± 0.5</td>
</tr>
<tr>
<td>TH47-1</td>
<td>N 17° 31’ 44.83” E 54° 05’ 66.17”</td>
<td>35</td>
<td>3.0 ± 1.5 (0.1)</td>
<td>0.24 ± 0.01</td>
<td>0.35 ± 0.02</td>
<td>0.17 ± 0.02</td>
<td>0.80 ± 0.04</td>
<td>6.00 ± 0.30</td>
<td>40 ± 4</td>
<td>7.5 ± 0.5</td>
</tr>
<tr>
<td>TH47-3</td>
<td>N 17° 31’ 44.83” E 54° 05’ 66.17”</td>
<td>40</td>
<td>3.0 ± 1.5 (2.6)</td>
<td>0.24 ± 0.01</td>
<td>0.77 ± 0.04</td>
<td>0.17 ± 0.02</td>
<td>1.21 ± 0.06</td>
<td>11.80 ± 0.60</td>
<td>51 ± 4</td>
<td>9.7 ± 0.7</td>
</tr>
<tr>
<td>TH47-2</td>
<td>N 17° 31’ 44.83” E 54° 05’ 66.17”</td>
<td>56</td>
<td>3.0 ± 1.5 (2.1)</td>
<td>0.47 ± 0.01</td>
<td>0.71 ± 0.05</td>
<td>0.17 ± 0.02</td>
<td>1.39 ± 0.07</td>
<td>11.40 ± 0.80</td>
<td>72 ± 5</td>
<td>8.2 ± 0.7</td>
</tr>
</tbody>
</table>

Table 3.5 Table of OSL ages for sediment samples from Ghazal (TH47), together with the supporting dose rate and equivalent dose data.

1 Field water content with measured water content in brackets.
2 From field gamma spectrometry measurements and adjusted for field moisture content.
3 Corrected for beta-dose attenuation and adjusted for the field moisture content.
4 From Prescott and Hutton (1994), assigned relative uncertainties of ± 10%, and adjusted for the field moisture content. Time-averaged burial depth assumed steady sediment deposition.
5 Includes an assumed internal alpha dose rate of 0.03 ± 0.01 Gy/ka.
6 Mean ± standard (1σ) error, determined using the Central Age Model (Galbraith et al., 1999).
7 $D_e$ overdispersion (i.e. the spread in the $D_e$ values after taking all measurement uncertainties into account).
8 Mean ± total (1σ) uncertainty, with the latter calculated as the quadratic sum of the random and systematic uncertainties.
Chapter 4
Chapter 4        Khumseen (TH50)

4.1 Introduction

This chapter will discuss the geological and archaeological history of Khumseen, and provide contextual information. Previous dating efforts, specifically single aliquot OSL dating, will be discussed to provide a comparison with the pseudo single-grain analysis made in this study. Data will be presented in the form of OSL decay curves, dose response curves and radial plots of $D_e$ distributions. The $D_e$ values were obtained following the application of well-established rejection criteria, and the $D_e$ distributions were interpreted before applying the Central Age Model. The latter was used to combine the individual $D_e$ values obtained for each sample into a single representative $D_e$ value to calculate the burial age of each sample. The environmental dose rate results will be included in this chapter for Khumseen.

4.2 Background information

Khumseen is a large overhang located approximately 2 km west of Al-hatab and Ghazal (Fig 4.1). The rock shelter lies at the bend of a prominent tributary channel feeding into the Wadi Dawkah. The site was excavated by DAP in 2010 to produce a 2 m trench. The trench was divided into 1 m units in a grid pattern, and all sediments were sieved using a 5 mm mesh (Hilbert et al., 2012). The site occupies a favourable position, with proximity to resources that are still enjoyed by the local Bedouin community. This group reoccupied the site in 2011 which prevented further investigations.

The 2 m trench is considered one of the deepest stratigraphic sequences in Oman; this is thought to be Pleistocene-Holocene in age due to Leptolithic stone tools located within the base unit GH5b and the overlying Unit GH5a (Hilbert et al., 2012).
4.2.1 Lithic analysis

Leptolithic tools located in Units GH5a and GH5b were manufactured from local Gahit limestone. The blade morphology indicates a similar modality to the NLT of Al-hatab Level 2 (GH5 to GH7) (see section 1.3.1). The Leptolithics at Khumseen are characterised by a blade tradition (i.e. blade twice as long as wide) of the Late Nejd Leptolithic (Rose and Usik, 2009). When compared to other NLT sites, the assemblage located at Khumseen indicates that the archaeological levels span the duration of the Holocene and possibly extend into the Terminal Pleistocene (Hilbert et al., 2012). This proposition will be tested by the use of pseudo single-grain dating. See Fig 4.2 for examples of blades found at Khumseen.

4.2.2 Previous dating

Previous dating was done at this site using AMS $^{14}$C dating on hearth ash from Unit GH4b, giving an age of 6845 ± 105 cal BP (Hilbert et al., 2012). This age will be discussed later in the chapter in conjunction with other dating techniques. Two other hearth fires were located in Units GH2 and GH3 as a result of their prominent dark/grey black colour. Upon closer inspection, however, no microscopic charcoal fragments could be found. Evidence of multiple hearth fires throughout the stratigraphic sequence may indicate human occupancy of the rock shelter on multiple occasions.

4.2.3 Stratigraphy

The rock shelter appears to be formed from weathered limestone and from the erosion of calcrete. The calcrete cappings have formed a roof over the rock shelter, with exposed calcrete below. The rock shelter is located on a gentle downhill slope, with the excavation situated on the eastern side of the hill. The stratigraphy consists of seven geological Units (GH1 to GH5b) as described in Table 4.1 and shown in Fig 4.8. Two of the
strata contain artefacts belonging to the NLT (GH5a and GH5b) and hearth ash was located within three of the strata (GH2, GH3 and GH4b).

### 4.3 OSL dating of samples

#### 4.3.1 Sample collection

Four OSL samples were collected from geological horizons GH4b, GH5a and GH5b using the same procedure as outlined in section 2.4.1. Two OSL samples were collected from the same geological horizon (GH5a) to check for chronological consistency (Fig 4.3). The four OSL samples are as follows:

- TH50-4: 83 cm depth below surface level, Unit GH4b
- TH50-3: 104 cm depth below surface level, Unit GH5a
- TH50-2: 118 cm depth below surface level, Unit GH5a
- TH50-1: 165 cm depth below surface level, Unit GH5b

A bag of sediment was collected at each OSL sample location for beta counting and moisture content. Two additional bags of sediment were also collected, located approximately 0-15 cm and 15-30 cm from the OSL tube holes. These bags of sediment were used for thick-source alpha counting to determine the U and Th concentration for gamma dose rate determination, as field gamma spectrometry measurements were not available due to an instrument malfunction in 2010.

#### 4.3.2 Single-aliquot measurements

Previous dating was done using single aliquots by Richard Roberts from the University of Wollongong for four samples. TH50-1 from basal unit (GH5b) was dated to $9.4 \pm 0.8$ ka and TH50-2 from overlying unit (GH5a) was dated to $7.1 \pm 0.4$ ka (Hilbert et al., 2012). TH50-3 and TH50-4, following measurement and analysis by Roberts, will be re-analysed using the data.
The resulting spread in the $D_e$ values (Fig 4.4a) are reflected in the overdispersion values of 56 ± 18% (TH50-3) and 19 ± 6% (TH50-4). Due to the paucity of aliquots that passed the rejection criteria it would be hazardous to place too much confidence in these re-analysed data points, especially given the range in $D_e$ values. Therefore it would be more plausible to compare ages against TH50-1 and TH50-2.

The single aliquots seen in Fig 4.4b are dominated by the fast component, with the decay curves for samples TH50-3 and TH50-4 showing the two brightest accepted aliquots. The aliquots that were rejected were dominated by either the slow or medium component, as there was too much background signal present to produce a decay curve for dating purposes.

4.3.3 Dose recovery data

Dose response tests were not done using samples from Khumseen; instead measurements were estimated from previous dose recovery data by the author (ALH-2) and by Roberts (TH47-1), see section 3.4.3 and 5.7.

4.4 Single-grain measurements

4.4.1 OSL decay curves

The OSL signal was measured using the same preheat and cutheat temperatures as Ghazal and Al-hatab. Two typical OSL decay curves are shown for a bright grain and a dimmer grain over a stimulation period of 2 s. The decay curve of the bright grain (Fig 4.5a) is dominated by the ‘fast’ component in quartz (see section 2.7) (Bailey et al., 1997).
4.4.2 OSL dose response curves

All OSL samples were measured using the following sequence of regenerative doses: [13.3, 6.7, 20, 40, 0 and 13.5 Gy] together with a test dose of 20 Gy. After applying the rejection criteria outlined in section 2.6.2, the data for the accepted grains were fitted with a saturating exponential function or a saturating exponential function with an extra linear term (Fig 4.5a).

A cumulative light sum plot was used to display the distribution of signal intensity for grains that passed the rejection criteria. Fig 4.6 indicates that, out of all four samples, 50% of the grains produce >90% of the OSL and therefore the $D_e$ distributions are unlikely to be dominated by the $D_e$ values of a few bright grains. This study uses pseudo single grains, where up to 3 to 5 grains may occur in a hole. As a result of this there is an increased probability of each hole containing at least one luminescent grain.

4.5 $D_e$ determination

The grains that passed the rejection criteria show the typical characteristics of a bright grain; Fig 4.5a. Initially 3400 grains were measured from all four samples, of which 3010 were rejected (see Table 4.2, for accepted grains). The majority of grains in samples TH50-1, TH50-2 and TH50-4 were rejected based on the OSL signal being too weak to discern above the background.

Sample TH50-3 had all grains rejected after applying the recuperation criterion, but by removing this criterion a total of 56 grains were able to be accepted for a final $D_e$ estimate. For individual sample outcomes on rejected grains, refer to Table 4.2.
4.6 Analysis and interpretation of $D_e$ distributions

Two of the pseudo single-grain $D_e$ data sets were analysed by two operators: the author (LL) and Zenobia Jacobs (ZJ). Radial plots of all $D_e$ distributions are shown in Fig 4.7. The radial plots will be examined in regards to the amount of spread within each sample.

Sample TH50-1 has an overdispersion value of $37 \pm 5\%$ when analysed by LL, and an overdispersion of $26 \pm 4\%$ when analysed by ZJ. The two analyses (of the same data set) show minor differences, but in both cases the majority of grains fall within the grey band, which is centred on the weighted mean $D_e$ as calculated by the Central Age Model.

Sample TH50-2 has an overdispersion of $47 \pm 5\%$ (LL) and $36 \pm 4\%$ (ZJ) with some differences in the $D_e$ values and associated precisions of certain grains. These are found especially in those measured with high precision, which may account for these differences.

Sample TH50-3 has a $D_e$ overdispersion of $52 \pm 9\%$ (LL), and TH50-4 has an overdispersion value of $53 \pm 5\%$ (LL). For all samples the majority of grains fall within the grey bands, indicating that most grains have $D_e$ values consistent (at 2σ) with the weighted mean values determined using the Central Age Model. The latter were used to calculate the OSL ages of the Khumseen samples.

Samples TH50-2 and TH50-3 are from the same unit (GH5a), and show a similar range of $D_e$ values when analysed by either LL or ZJ. Changing operators may change the overdispersion and weighted mean $D_e$ value slightly. The age estimates used in the study are based on the $D_e$ values obtained by the author, but essentially similar ages are obtained using the $D_e$ values of ZJ (see Table 4.3).

4.7 Environmental dose rates

The beta and gamma dose rates were calculated using TSAC and GM-25-5 beta counting techniques (see section 2.9), together with the conversion factors of Guèrin et al. (2011).
The beta dose rate was measured directly using a GM-25-5 beta counter for all four OSL samples (see section 2.14.2). The measured (dry) dose rates were adjusted for field moisture content and these values are listed in Table 4.4. The beta dose rates decrease with increasing depth, but the HRGS data for TH50-2 and TH50-4 (see Appendix 1) shows that this change is not due to disequilibrium in the $^{238}$U or $^{238}$Th decay chains.

The gamma dose rates were measured using a combination of TSAC and beta counting, with the latter being used to determine the $^{40}$K activity by subtraction for all four OSL samples. Each gamma dose rate was adjusted to account for the field water content. The gamma dose rates are listed in Table 4.4 and decrease in value going down the stratigraphic profile. No field gamma measurements were possible for this site, however gamma dose rates for two of the four samples (TH50-1 and TH50-4) based on HRGS (see Appendix 1) resulted in consistent estimates.

The cosmic-ray dose rates were calculated following Prescott and Hutton (1994) and adjustments were made for water content using Readhead (1987). The roof of the rock shelter was not included in the calculations as it did not impede a clear view of the sky. For more information on cosmic-ray dose rates, see section 2.12.

### 4.7.1 Total dose rates

The total dose rates were calculated as the sum of the beta, gamma and cosmic-ray contributions, after adjustments were made for grain size and moisture content. An internal alpha dose rate of $0.03 \pm 0.01$ Gy/ka was also included in the total dose rate. The total dose rates (Table 4.4) decrease with increasing depth from $0.88 \pm 0.04$ Gy/ka (TH50-4) to $0.56 \pm 0.03$ Gy/ka (TH50-1).

### 4.8 OSL ages

The age of each sample was calculated by dividing the sample $D_e$ (as determined by the Central Age Model) by the total dose rate. The resulting age for sample TH50-4 was 5.9
± 0.5 ka from Unit GH4b. Samples TH50-3 and TH50-2 gave ages of 5.6 ± 0.7 ka and 7.8 ± 0.7 ka, respectively. These two samples are located in the same geological horizon (Unit GH5a) that the NLT assemblage was found. Sample TH50-1, from the basal Unit (GH5b), is dated to 10.0 ± 0.8 ka which gives a maximum age for the entire sequence and for the NLT recovered from this unit. The ages of TH50-3 and TH50-2 differ at 2σ; TH50-2 is considered the more reliable of the pair, as the $D_e$ distribution is less overdispersed than that of TH50-3 and the weighted mean of the latter distribution appears to underestimate the $D_e$ values measured with the highest precision (Fig 4.7).

The OSL ages discussed above can now be compared to previous single- aliquot and AMS $^{14}$C ages. Sample TH50-1 produced a multi-grain age of 9.4 ± 0.8 ka, and TH50-2 an age of 7.1 ± 0.4 ka. These are statistically indistinguishable from the pseudo single-grain ages listed above, with the multi-grain ages only slightly younger than the pseudo single-grain ages. AMS $^{14}$C dating of hearth ash from layer GH4b yielded an age of 6845 ± 105 Cal BP (Hilbert et al., 2012), which is consistent with the OSL age for the overlying layer (5.9 ± 0.5 ka) and with the OSL age of TH50-2 from the underlying Unit (7.8 ± 0.7 ka). The other sample from this unit, TH50-3, has an age (5.6 ± 0.7 ka) that is consistent at 2σ with $^{14}$C age.

4.9 Chapter Summary

This chapter provided background information on Khumseen, and previous dating results from the site were discussed and compared to the pseudo single-grain ages. The latter are discussed in terms of site interpretation in chapter 6, along with the ages for Ghazal and Al-hatab.
Fig 4.1 Topography and site location of Khumseen rock shelter (N 17.31.3517˚ E 54.042111˚)

Fig 4.2 Refitted core from Khumseen, indicating the stage of refitting. Ten blades were struck from this one core (taken from Hilbert et al., 2012).
Fig 4.3 Diagram of the stratigraphy at Khumseen. The diagram shows the positions of the four OSL samples and their corresponding units, along with the three ash hearths (modified from Hilbert et al., 2012)
**Fig 4.4a**) Radial plot showing $D_e$ values for single aliquots samples of TH50-3 (56 ± 18%) and TH50-4 (19 ± 6%).

**Fig 4.4b**) Decay curves from samples TH50-3 and TH50-4; are taken from single aliquot measurements re-analysed by author.
Fig 4.5 a) Dose response curve of a bright grain from TH50-1 which did pass the rejected criteria. The inset plot shows the OSL decay curve for the same bright grain. b) Typical OSL decay curve of a dull grain over an interval of 2 s optical stimulation. This grain would not have passed the rejection criteria.

Fig 4.6 Distribution of signal intensity from single grains that passed the rejection criteria of all four samples from Khumseen. Data are plotted as the proportion of the total light sum that originates from the specified percentage of grains (Duller et al., 2000).
Fig 4.7 Radial plots of $D_e$ values for pseudo grains. The grey bands are centred on the weighted mean $D_e$ values calculated using the Central Age Model. The $D_e$ overdispersion values for these distributions are as follows: TH50-1, 37 ± 5% (LL) and 26 ± 4% (ZJ); TH50-2, 47 ± 5% and 36 ± 4% (ZJ); TH50-3, 52 ± 9% (LL); and TH50-4, 53 ± 5% (LL).
Fig 4.8 Stratigraphic sequence depicting pseudo single-grain OSL ages and AMS $^{14}$C date age for hearth ash.
<table>
<thead>
<tr>
<th>Geological Unit</th>
<th>Geological description</th>
<th>Archaeological description and age control</th>
</tr>
</thead>
<tbody>
<tr>
<td>GH1</td>
<td>Dark brown, coarse sand (predominately gravel)</td>
<td>Hearth ash; no microscopic charcoal fragments</td>
</tr>
<tr>
<td>GH2</td>
<td>Light brown, silty sand with gravels and cobbles</td>
<td>Hearing ash</td>
</tr>
<tr>
<td>GH3</td>
<td>Yellowish-brown, medium-coarse sand</td>
<td>TH50-4 and AMS $^{14}$C dating (hearth ash)</td>
</tr>
<tr>
<td>GH4a</td>
<td>Light brown, medium-coarse sand with silt, gravels and cobbles</td>
<td>TH50-2 and TH50-3; NLT artefacts</td>
</tr>
<tr>
<td>GH4b</td>
<td>Yellowish-brown, medium to coarse sand with gravels and cobbles</td>
<td>TH50-1; NLT artefacts</td>
</tr>
<tr>
<td>GH5a</td>
<td>Yellowish-brown, medium to coarse sand with gravels and cobbles</td>
<td></td>
</tr>
<tr>
<td>GH5b</td>
<td>Yellowish-brown, medium to coarse sand with gravels and cobbles</td>
<td></td>
</tr>
</tbody>
</table>

*Table 4.1* Geological units of the Khumseen site, with the represented archaeological units and the OSL samples (based on field notes by Richard Roberts).
Rejection Criteria applied to Khumseen grains

| Sample Name | Grains measured | Criterion 1 Brightness | Criterion 2 RR ratio | Criterion 3 IR ratio | Criterion 4 Recuperation | No. grains rejected | Total no. rejected | Total no. Accepted | Final no. grains | %  
|-------------|----------------|------------------------|----------------------|----------------------|-------------------------|-------------------|------------------|------------------|------------------|------
| TH50-1      | 1000           | 558                    | 346                  | 415                  | 500                     | 819               | 888              | 112              | 107              | 11%   
| TH50-2      | 900            | 572                    | 437                  | 400                  | 356                     | 865               | 813              | 87               | 84               | 9%    
| TH50-3      | 500            | 225                    | 198                  | 151                  | 500                     | 0                 | 1074             | 443              | 57               | 11%   
| TH50-4      | 1000           | 618                    | 359                  | 347                  | 456                     | -                 | 780              | 866              | 134              | 13%   
| Total       | 3400           |                        |                      |                      |                         |                   | (=3400)          |                  |                  |       

Table 4.2 Summary of grains from Khumseen samples rejected according to the formal rejection criteria of Jacobs (2003, 2006a, 2006c).

1 OSL signals are weak (T_N signal less than 3 times the instrumental background).
2 Recycling ratio is poor (i.e. more than 2 standard errors away from unity).
3 Infrared simulation causes significant loss of OSL signal (i.e. OSL-IR depletion ratios smaller than unity by more than two standard errors).
4 Recuperation is high (L_X/T_X for 0 Gy dose is greater than 5% of L_N/T_N).
5 No. of grains rejected following the removal of recuperation.
6 Total no. rejected grains plus total no. accepted grains will equal the no. of grains measured.
7 Total no. of accepted grains for dating.
8 Percentage of finalised grains used in CAM.
## Operator Variance

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Lauren Linnenlucke</th>
<th>Zenobia Jacobs</th>
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<tr>
<td></td>
<td>$D_e$ (Gy)</td>
<td>$D_e$ (Gy)</td>
</tr>
<tr>
<td>TH50-1</td>
<td>5.57 ± 0.31</td>
<td>5.08 ± 0.22</td>
</tr>
<tr>
<td>TH50-2</td>
<td>5.60 ± 0.40</td>
<td>5.81 ± 0.30</td>
</tr>
</tbody>
</table>

*Table 4.3* Operator variation showing ages and $D_e$ values done by two analysts on samples from Khumseen (TH50) by LL and ZJ.
<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Sample location (Lat. Long.)</th>
<th>Sample depth (cm)</th>
<th>Field water content (% dry mass) $^1$</th>
<th>$\gamma$ dose rate $^2$ (Gy/ka$^{-1}$)</th>
<th>$\beta$ dose rate $^3$ (Gy/ka$^{-1}$)</th>
<th>Cosmic-ray dose rate $^4$ (Gy/ka$^{-1}$)</th>
<th>Total dose rate $^5$ (Gy/ka$^{-1}$)</th>
<th>$D_e$ (Gy) $^6$</th>
<th>$\sigma_e$ (%) $^7$</th>
<th>Age (ka) $^8$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Khumseen</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>TH50-4</td>
<td>N 17˚ 31' 35.17&quot; E 54˚ 04' 21.11&quot;</td>
<td>83 cm</td>
<td>3.0 ± 1.5 (0.8)</td>
<td>0.24 ± 0.01</td>
<td>0.42 ± 0.02</td>
<td>0.19 ± 0.02</td>
<td>0.88 ± 0.04</td>
<td>5.20 ± 0.30</td>
<td>53 ± 5</td>
<td>5.9 ± 0.5</td>
</tr>
<tr>
<td>TH50-3</td>
<td>N 17˚ 31' 35.17&quot; E 54˚ 04' 21.11&quot;</td>
<td>104 cm</td>
<td>3.0 ± 1.5 (1.4)</td>
<td>0.24 ± 0.01</td>
<td>0.36 ± 0.02</td>
<td>0.19 ± 0.02</td>
<td>0.82 ± 0.04</td>
<td>4.60 ± 0.50</td>
<td>52 ± 9</td>
<td>5.6 ± 0.7</td>
</tr>
<tr>
<td>TH50-2</td>
<td>N 17˚ 31' 35.17&quot; E 54˚ 04' 21.11&quot;</td>
<td>118 cm</td>
<td>3.0 ± 1.5 (1.6)</td>
<td>0.17 ± 0.01</td>
<td>0.33 ± 0.02</td>
<td>0.19 ± 0.02</td>
<td>0.72 ± 0.04</td>
<td>5.60 ± 0.40</td>
<td>47 ± 5</td>
<td>7.8 ± 0.7</td>
</tr>
<tr>
<td>TH50-1</td>
<td>N 17˚ 31' 35.17&quot; E 54˚ 04' 21.11&quot;</td>
<td>165 cm</td>
<td>3.0 ± 1.5 (0.9)</td>
<td>0.14 ± 0.01</td>
<td>0.21 ± 0.01</td>
<td>0.19 ± 0.02</td>
<td>0.56 ± 0.03</td>
<td>5.57 ± 0.31</td>
<td>37 ± 5</td>
<td>9.9 ± 0.8</td>
</tr>
</tbody>
</table>

Table 4.4 Table of OSL ages for sediment samples from Ghazal (TH47), together with the supporting dose rate and equivalent dose data.

1 Field water content, with measured water content in brackets.

2 From TSAC and GMBC measurements, and adjusted for field moisture content.

3 Corrected for beta-dose attenuation and adjusted for the field moisture content

4 From Prescott and Hutton (1994), assigned relative uncertainties of ±10%, and adjusted for the field moisture content. Time-averaged depth assumed rapid deposition of sediment to present depth.

5 Includes an assumed internal alpha dose rate of 0.03 ± 0.01 Gy/ka.

6 Mean ± standard (1σ) error, determined using the Central Age Model (Galbraith et al., 1999).

7 $D_e$ overdispersion (the spread in the $D_e$ values remaining after taking all measurement uncertainties into account).

8 Mean ± total (1σ) uncertainty, with the latter calculated as the quadratic sum of the random and systematic uncertainties.
Chapter 5
Chapter 5  Al-hatab (ALH)

5.1  Introduction

In this chapter, both the geological and archaeological history of Al-hatab will be given as cause for the use of pseudo single-grain OSL analysis in Area 3. Using a set of well-established, standardised procedures, data from two samples (ALH-1 and ALH-2) will be given in the form of decay curves, dose response curves and radial plots of the $D_e$ distributions. The $D_e$ distribution will be interpreted and an appropriate statistical model (i.e. Central Age Model) applied to combine the individual $D_e$ values into a single representative $D_e$ value to calculate the final age of each sample. The environmental dose rate results will also be provided. The final ages will be compared against previous dating methods in order to determine site stratigraphy that will be later discussed in chapter 6.

5.2  Background information

Al-hatab was first discovered by the Central Oman Research Project team (CORP) between 2002 and 2008. OSL dates were provided for Area 1 (the original pit) and lithic assemblages (surface collections) were located from Area 2 (Fig 5.1). The focus for this thesis is on OSL samples taken from Area 3 by the Dhofar Archaeological Project (DAP) during the 2010 and 2011 expeditions. This was led by Jeffrey Rose and his team from the University of Birmingham, in conjunction with Richard Roberts from the University of Wollongong.

Areas 1, 2 and 3 are located in front of a partially collapsed rock overhang that is situated inside a small tributary in the upper courses of the wadi of Wadi Dawkah National Park (Hilbert et al., 2012). The site was found behind a wide terrace approximately 15 m above an active channel. Due to the seasonal monsoonal weather, sampling of the site was carried out during the dry season. All excavations in Areas 1, 2 and 3 were conducted in a
systematic manner, following grid patterns (Rose and Usik, 2009; Hilbert et al., 2012). A discussion will be provided below giving background information of each individual Area, including previous dating techniques and ages listed, as well as lithic analysis. This will provide the background context to site stratigraphy and the interpretation of OSL age determinations made in the current study.

5.2.1 Lithic analysis

The lithic assemblage from Area 1 was recovered from the lower archaeological unit of Al-hatab, Level 2 (Table 5.1). The assemblage was manufactured from similar high quality chert found within close proximity to the site. The Leptolithic assemblage has been bracketed to date between 14 and 7 ka using AMS $^{14}$C and OSL ages from Area 1 (Hilbert et al., 2012; Rose and Usik, 2009).

OSL ages were not obtained from Area 2, with only Leptolithic tools collected from a surface collection. The Leptolithic assemblage was collected to represent the variation in Nejd Leptolithic Tradition (NLT) from both the Terminal Pleistocene and Early Holocene (Hilbert et al., 2012). The lithics were compared against those collected at other Nejd plateau sites located near Al-hatab (TH.124b and TH.67) (Hilbert et al., 2012).

5.2.2 Previous dating

1) Area 1: Original trench

The sedimentary deposits of Area 1 mainly consisted of colluvial gravels and aeolian sand deposits in an interstratified sequence from the Terminal Pleistocene and Early Holocene. The excavation trench was divided into five sedimentary units, labelled A to E (youngest to oldest); see Fig 5.3a (Rose and Usik, 2009). Two OSL samples were collected from unit B and the upper portion of unit C. These were dated using the SAR procedure on multi-grain aliquots. All previous dating for this site was done by another laboratory. The
OSL sample from Unit B was dated to $13.7 \pm 2$ ka, and the OSL sample from Unit C produced an age of $13 \pm 1.1$ ka; Fig 5.3a (Rose and Usik, 2009).

AMS $^{14}$C dating was done on a large number of shells of the non-burrowing terrestrial snails ‘Euryptyxis latireflexa’ (Fig 5.3b), which were located within Unit A among the upper colluvial horizons (between 10 - 30 cm from the surface) (Rose and Usik, 2009). The specimens were dated to $10,430 \pm 140$ cal BP, although this may not be representative of a true age. The $^{14}$C reservoir effect can make the age appear too old by ~1000 years (Rose and Usik, 2009; Hilbert et al., 2012). The presence of terrestrial snails of this age in Unit A suggests sedimentation from the Early Holocene (Cremaschi and Negrino, 2005; Rose and Usik, 2009).

2) Area 3: Current trench

Previous dating was done for Area 3 by a different laboratory using AMS $^{14}$C on hearth ash remains located within the first archaeological level (GH2b); see Table 5.1. The ash is dated to $6845 \pm 105$ Cal BP (Rose and Usik, 2009; Hilbert et al., 2012). Located within the ash is evidence of a sandy gravel mix from the reworking of the upper portion of Unit A, that consists of a colluvial horizon approximately 10 to 30 cm in depth (Rose and Usik, 2009; Hilbert et al., 2012). Therefore the AMS $^{14}$C age would seem to be too young due to sediments from the overlying portion of the unit seeping into the ash, resulting in possible contamination.

5.3 Area 3

5.3.1 Stratigraphy

The stratigraphy of the area, specifically Area 3, was described by Mike Morley from Oxford Brookes University in conjunction with DAP. As a result, the description of the geology of the surrounding landscape and Area 3 will be discussed in this thesis using the information provided by Morley; see Appendix 2. The site mainly consists of colluvial
deposits with an interstratified sequence of gravel and silt-dominated sediments that span the Terminal Pleistocene and Early Holocene. The deposit has been divided into eight stratigraphic layers (GH8-GH1) with evidence of anthropologically reworked chert observed throughout the sequence.

Unit GH8 (Fig. 5.2a) represents the base of the sequence (0.4 m thick) and is an accumulation of silt and fine sand, with the occasional coarser components. This unit is well-cemented and could represent deposition in a hyper-arid phase during the Terminal Pleistocene.

Units GH7 and GH5 consist of fine gravel which is not recorded elsewhere in the exposed profile. A mixture of fine grained gravels and larger cobbles are located within unit GH3, indicating imbricated gravels transported downslope from the plateau to the rock shelter. The imbricated gravels also appear in Area 1 (adjacent to Area 3), indicating that the gravels may have been deposited in the wadi during an ephemeral flood event. Archaeological material from Area 1 indicates that the flood may have occurred during the Upper Palaeolithic, or it may correlate with a period of episodic valley re-activation that relates to a brief Late Pleistocene pluvial episode (Parker 2009). Interpretations indicate that it is possible that more than one of the short-lived wadi re-activations may be differentiated in the stratigraphic log as a narrow, channel-like profile. It should be emphasised that units GH7-GH5 do not solely represent fluvial sedimentation, but show fluvial deposition of fine gravel over a wadi-bank proximal area at the front of the rock shelter. All leptolithic stone tools for this area were located between units GH7 to GH5.

Unit GH6 is ambiguous, as it may have formed from the division between the fluvial and colluvial episodes represented by units GH7 and GH5, or it could be the eroded remnants of a depositional event similar to unit GH4, which has been heavily truncated by the deposition of unit GH5.

Unit GH4 consists of silts and sands that are moderately compacted. This unit was most likely deposited rapidly, suggesting an episode of windblown activity. Evidence of fine gravel interspersed within this fine-grained matrix further suggests downhill movement from an upslope source. The absence of coarser material suggests that it is unlikely that there has been a long depositional hiatus.
Unit GH3 represents a colluvial deposit (10-30 cm thick) derived from the reworking of broken down material from the rock shelter’s roof and walls. This unit consists of a poorly sorted diamict of both fine and coarse sediments.

Units GH1 and GH2 show signs of deposition from the mid to late Holocene, due to reworking of sediments and hearth ash being present within unit GH2b. The hearth ash was dated using AMS $^{14}$C to an age of $6845 \pm 105$ Cal BP.

### 5.3.2 Archaeology

The archaeological levels are split into two sections spanning four geological units: Level 1 consists of units GH3 and GH2b (see section 5.2) and Level 2 consists of units GH7 to GH5.

The Early Nejd Leptolithic assemblages have been recovered from GH7-GH5. This shows a similar archaeological sequence to that found in Area 1. The artefacts reside within colluvial horizons that indicate oscillating wet to dry conditions, and are separated by GH4 showing a sandy layer devoid of artefacts (Hilbert et al., 2012). One of the OSL samples (ALH-1) was collected from unit GH4, due to the presence of finer sediments and fewer rocks that are more conducive to OSL dating; this unit separates the two archaeological horizons.

The lithic assemblage from Level 2 is composed of 194 pieces, with no refits possible due to the secondary position of the sediments and small sample size (Hilbert et al., 2012). This is important as it can be taken as evidence for mixing of sediments, which will be discussed in the context of the $D_e$ values. The lithic assemblage belongs to a bladed technology (see section 1.3); for examples of blades found at Al-hatab from Area 2, see Fig 5.4.
5.4 OSL dating of samples from Area 3

5.4.1 Sample collection

Sample collection followed the same procedure as outlined in chapter 2. From Area 3 a total of two sediment samples were collected for OSL dating. The samples were taken from geological Units GH4 (ALH-1) and GH8 (ALH-2) (Fig 5.2a). The archaeological layers of interest are GH2b to GH3 and GH5 to GH7. ALH-1 separates these two Levels and ALH-2 will give a maximum age for Level 2.

The collection of each sample was done twice with two separate plastic tubes; see section 2.4.1. These were collected from the same sample location on the north eastern side of the trench. The two plastic tubes for ALH-1 were taken approximately 14 cm apart and 26 cm below the surface. ALH-2 also consisted of 2 plastic tubes, approximately 12 cm apart and 69 cm below the surface. The samples were collected from these levels because they consisted of finer sediment and were devoid of larger objects. Both samples had bags of sediment taken for beta counting and moisture content. Field gamma-ray spectrometry measurements were made in the empty tube holes at both sample locations.

5.5 Single-grain measurements

At Al-hatab, only pseudo single-grain measurements were made. Single aliquots were not used, based on the results obtained from Ghazal and Khumseen (see chapters 3 and 4).

5.5.1 OSL decay curves

OSL decay curves show inherent grain brightness or regenerated signal intensity luminescence over a stimulation period. Representative OSL decay curves for a typical bright grain and a typical dull grain from the site are shown in Fig 5.5. A cumulative light sum plot
is also shown to illustrate the range of signal intensities and the relative distribution of ‘bright’ and ‘dim’ grains for all grains measured from the two samples.

A cumulative light sum graph of both samples (ALH-1 and ALH-2) is employed to represent the total distribution of signal intensity of single grains that have passed the rejection criteria (Fig 5.6). The data are plotted to show the proportion of the total light sum that originates from the specified percentage of grains. The difference in grain brightness can be identified in the cumulative light distribution to identify the proportion of grains from which reliable $D_e$ estimates can be obtained, and also to recognise samples that contain a small proportion of very bright grains (Duller et al., 2000). Pseudo single-grain analyses will be dominated by these few grains, and any grain-to-grain variability in $D_e$ will be visible even at that scale of analysis (Duller et al., 2000). For samples with a larger proportion of their grains contributing significantly to the total light sum, pseudo single-grain analyses will average out and mask any grain-to-grain variability (Duller et al., 2000).

Fig 5.6 shows that 40% of the Al-hatab quartz grains that passed the rejection criteria produce 90% of the OSL. Compared to many other samples, therefore, a greater proportion of luminescence comes from more accepted grains than is the case at, for example, Blombos Cave (Jacobs et al., 2003b). The likely reason for this is that, as discussed before, we have measured pseudo single-grains (i.e. more than one grain per hole) (e.g. Arnold et al., 2012).

As every grain does not decay at the same rate or to the same extent, all grains were measured following a range of regenerative doses (see section 5.6.3) and a fixed test dose of 20 Gy. The shape and range of OSL decay curves confirms that the quartz grains measured for samples ALH-1 and ALH-2 are dominated by the ‘fast’ component (Bailey et al., 1997). All grains must exhibit prominent fast components to pass the rejection criteria. It should be noted that, for Al-hatab, only 135 grains out of 1000 originally measured of sample ALH-1 passed the rejection criteria, and 136 grains of sample ALH-2. This is a fairly typical percentage return for quartz grains measured from a range of different archaeological sites globally (e.g. Duller et al., 2000; Jacobs et al., 2006).
5.5.2 Signal integration range

Varying the size of the integration range can potentially lead to an increase or decrease in the overdispersion of \( D_e \) values, so it is important to choose an appropriate integration time interval.

For single-grain analysis the first 0.2 s of OSL decay is often used, with the background signal obtained from the last 0.3 s of decay (Jacobs et al., 2006). The background signal is then subtracted from the initial signal to obtain the ‘net’ OSL signal used for dating. This signal range is tuned to the fast component of the quartz signal, which is required for the SAR procedure (Wintle and Murray, 2006). The signal integration range of 0.2 s was used for all ten samples in this thesis regardless of grain-to-grain variation, as the decay curves are dominated by the fast component.

5.5.3 OSL dose response curves

The OSL signals from pseudo single grains extracted from ALH-1 and ALH-2 were measured using two different regenerative-dose sequences, owing to their different natural OSL intensities. The sequence for ALH-1 consisted of regenerative doses of [40, 20, 40, 80, 10, 0 and 40 Gy] and a test dose of 20 Gy. Sample ALH-2 was measured using regenerative doses of [40, 20, 40, 80, 120, 0 and 40 Gy] (see Fig 5.5). These doses were used to construct the dose response curve for pseudo single grains, which was fitted using a saturating exponential function, or a saturating exponential function with an extra linear function.

Dose response curves may exhibit a wide range in OSL characteristics (Murray and Wintle, 2003). The accepted grains used in this study produced signals that increased steadily with each dose, as opposed to those that showed a limited growth as saturation was approached. The latter was not a problem because of the low \( D_e \) values and, hence, the need to use only relatively small regenerative doses.
5.6 $D_e$ determination

The pseudo grains accepted for sample $D_e$ and age determination were obtained on the basis of the criteria discussed in section 2.6.2. The majority of grains measured from ALH-2 (48%) were rejected based on the recuperation criterion. Recuperation is seen as the transfer of electrons from a light-insensitive trap to a light sensitive trap during the preheat stage, and it may be a particular problem for young samples. Recuperations can also cause age over-estimation (Feathers et al., 2010).

The majority of grains for ALH-1 (a total of 87%, with some of the grains rejected based on more than one criteria, see Table 5.2) were rejected on the basis of the IR depletion ratio (i.e. exhibiting contamination by feldspar inclusions), and only 1.8% of grains were rejected on the basis of recuperation for ALH-1. A total of 14% of the measured grains were accepted for $D_e$ determination of samples ALH-1 and ALH-2.

5.7 Dose recovery data

Dose recovery tests were only run using sample ALH-2. The measured dose used preheat and cutheat temperatures of 200°C/10 s and 160°C/5 s, respectively; this is seen to have resulted in an underestimation. Essentially if the measured values were consistent with the given dose then the majority of the values will fall within the grey band that is centred on the given dose of 400 sB.

The 180°C/10 s and 180°C/5 s preheat and cutheat combination resulted in a more satisfactory range of measured dose. As can be seen in Fig 5.7a, the majority of the values fall within the grey band, but there is a conspicuous string of lower outer dose values that is not currently understood. A similar set of results were previously observed by Gliganic et al. (2012).

All measurements for $D_e$ determination was subsequently made using the 180°C/10 s and 180°C/5 s temperature combination.
5.8 Analysis and interpretation of $D_e$ distributions

Pseudo single-grain $D_e$ distributions were displayed as radial plots to examine them for any patterns (Fig 5.7b). A comparative study was also done using two different operators; the author of this study and Zenobia Jacobs (ZJ). This comparison was done to check that the final age estimates were not dependent on the particular operator. Changing operators might change the $D_e$ values and their overdispersion, and the interpretation if operators analyse the data in different ways. Ideally, individual $D_e$ values should be consistent at 2σ for different operators. In this operator test, the author (LL) and ZJ analysed the same data set independently of each other.

Sample ALH-1 showed an overdispersion of $48 \pm 5\%$ when analysed by LL, with $D_e$ values varying from $\sim 1$ to 7 Gy. Jacobs (ZJ) obtained a $D_e$ distribution that is mostly consistent with that produced by LL, but with a slightly lower, but statistically consistent, overdispersion value of $41 \pm 5\%$. In both data sets, a large proportion of the individual $D_e$ values (88\% for LL and 87\% for ZJ) fall within the grey bands, indicating that these $D_e$ values are self-consistent at 2σ with a minority of data points (12\% and 13\%, respectively) falling outside these bands.

Sample ALH-2 has a higher percentage of grains falling outside the 2σ grey band (24\% for LL and 21\% for ZJ), although the $D_e$ values are less overdispersed than in ALH-1; LL calculated an overdispersion of $30 \pm 3\%$ and ZJ an overdispersion of $36 \pm 3\%$. The $D_e$ values range from $\sim 6$ Gy to $> 40$ Gy. The Central Age Model was used to combine the individual $D_e$ values for both ALH-1 and ALH-2, as the sediments of both are considered to have been well bleached prior to deposition. The overdispersion values are, however, high for both samples (>20\%); this may be an indication of grain mixing. The number of contaminated grains, however, is still only a small fraction of the total number of accepted grains; it is unlikely that this will significantly change the age of the sample. Further work should be done on ‘true’ single grains to see whether grain mixing can be resolved using the finite mixture model.
5.9 Environmental dose rates

The environmental dose rates were estimated using the methods described in section 2.10 and the conversion factors from Guérin et al. (2011).

The beta dose rates were measured using a GM-25-5 beta counter for both Al-hatab samples (see section 2.14.3). This technique provides a more accurate measurement of the beta dose rate than the parental U and Th measurements using INAA or ICP-MS (Olley et al., 1996, 1997). The dose rate was adjusted for field moisture content (see section 2.11.1), resulting in beta dose rates of $0.58 \pm 0.03$ Gy/ka for sample ALH-1 from unit GH4, and $0.37 \pm 0.02$ Gy/ka for ALH-2 from unit GH8 (Table 5.5).

The gamma dose rates were measured using an in situ gamma spectrometer for both OSL samples. This is the preferred method for calculating the gamma dose rate, as it accounts for any spatial non-uniformity of radioactivity within 30 cm of the sample. The gamma dose rate was also adjusted to account for the field water content for each sample. Sample ALH-1 has a gamma dose rate of $0.23 \pm 0.01$ Gy/ka and ALH-2 has a gamma dose rate of $0.26 \pm 0.01$ Gy/ka (Table 5.5). Gamma dose rates can also be calculated from the HRGS, TSAC and GM 25-5 beta counting measurements; refer to Table 5.4. The difference between the lab based (HRGS and TSAC) and field measurements for ALH-1 suggest that the presence of limestone gravel may affect the dose rate substantially; as a result of this field measurements are therefore preferred.

The cosmic-ray dose rates were calculated following Prescott and Hutton (1994) and adjusted for water content (Readhead, 1987). The burial depth of the samples was estimated from the top of the trench to the level of the sample. The roof of the rock shelter was approximately 2 m from the trench; therefore taking into account the thickness of roof was not necessary when calculating the cosmic-ray dose rate. The cosmic-ray dose rate for samples ALH-1 and ALH-2 were calculated at $0.19 \pm 0.02$ Gy/ka and $0.17 \pm 0.02$ Gy/ka, respectively. The grain size, moisture contents and dose rate contributions are presented in Table 5.5.
5.9.1 Total dose rates

The total dose rates for samples ALH-1 and ALH-2 were calculated using the sum of beta, gamma and cosmic-ray dose rates (Table 5.5). Adjustments were made for grain size and the moisture content of each sample, plus the internal alpha dose rate, for which an assumed value of 0.03 ± 0.01 Gy/ka was used based on Jacobs et al. (2006). The field water content was fixed at 3.0 ± 1.5%, which reflects the long-term water content (i.e. averaged over the entire period of sample burial) incorporating the likely variation in monsoonal rainfall to the Nejd Plateau region. The OSL ages increase by ~1% for each 1% increase in water content. The total dose rate for ALH-1 is 1.03 ± 0.05 Gy/ka and 0.82 ± 0.04 Gy/ka for ALH-2 (see Table 5.5). The total dose rates and final $D_e$ values are discussed below to obtain the final ages for the samples from Al-hatab.

5.10 OSL ages

The final ages were obtained by dividing the sample $D_e$ values by the total dose rates, using the age equation outlined in section 2.3. The age obtained for sample ALH-1 is 2.3 ± 0.2 ka, which gives a maximum age for the artefacts in units GH2b and GH3. Sample ALH-2 is dated to 19.5 ± 1.2 ka, which gives a maximum age for the artefacts in units GH5-7. A minimum age for the latter of ~2-3ka is provided by ALH-1.

The final ages for ALH-1 and ALH-2 (Area 3) can now be compared to previous dating results from Area 1. The samples from Area 1 were collected from the top three stratigraphic layers (Units A-C in Fig 5.3a). These units are roughly equivalent to Units GH8 to GH5 in Area 3 (Fig 5.2a). Sample TH29-1 was dated to 13.7 ± 0.2 ka (Unit B) and TH29-2 was dated to 13.0 ± 1.1 ka (Unit C) (Fig 5.3a). The AMS $^{14}$C ages obtained from Unit A gave an age of 10,430 ± 105 Cal BP. Together the upper two thirds of Area 1 was dated to between ~10 and 14 ka. These ages differ from the two OSL ages produced in this study for Area 3: 2.3 ± 0.2 ka (Unit GH4) and 19.5 ± 1.2 ka (Unit GH8). The AMS $^{14}$C age of 6845 ± 105 Cal BP for Unit GH2b from Area 3 was interpreted by Hilbert et al. (2012). The AMS $^{14}$C age would seem to be too young due to sediments from the overlying portion of the unit.
seeping into the ash and causing possible contamination. This would thereby indicate post-depositional mixing from layer GH2b into underlying layer GH4. Nonetheless, the three ages are in stratigraphic order; however differ significantly from the ages obtained in Area 1. Until the two areas are correlated through excavation, it will remain uncertain about whether the ages for Area 1 may be in error due to possible contamination and the effects of averaging, when comparing single aliquots to pseudo single grains. The final ages will be discussed in relation to leptolithic assemblages found in Levels 1 and 2 of Area 3; this will be addressed in chapter 6.

5.11 Chapter summary

The site’s depositional and previous dating history has been used to provide reasons as to why single-grain OSL analysis is necessary for the most recent trench (Area 3). The results for samples ALH-1 and ALH-2 were achieved using a set of well-established, standard procedures to ensure that results were obtained systematically; operator variance uncertainties on the $D_e$ values was also examined and found to be insignificant. Results indicate that the samples were probably well bleached, but that some post-depositional mixing likely took place, giving rise to $D_e$ distributions that are overdispersed by between 30 and 50%. This range of values is larger than commonly accepted for well-bleached grains that have remained undisturbed since burial; but the majority of grains in each sample give self-consistent $D_e$ values from which their ages were calculated.
Fig 5.1 Rock shelter Al-hatab and site topography. The two circles indicate Area 1 and Area 3. (N17.313417°E54.061050°).
Fig 5.2 a) Stratigraphic drawing of Area 3, representing individual geological horizons and indicating the positions of OSL samples dated in this thesis, and AMS $^{14}$C age b) Topographic landscape of Al-hatab rock shelter, with previous pits shown also (modified from Hilbert et al., 2012).
Fig 5.3 a) Stratigraphic diagram of sequence from Area 1, (the original trench) showing units A to E. OSL TH29-1 (13.7 ± 2 ka) and OSL TH29-2 (13 ± 3.1 ka) (Taken and modified from Rose and Usik, 2009). b) Image of the *Euryptyxis latireflexa*, non burrowing terrestrial snail and located in Unit A of section Area 1 and AMS $^{14}$C dated to 10,430 ± 140 cal BP (Rose and Usik, 2009). The same species of non-burrowing terrestrial snail was located in Area 3; AMS $^{14}$C dated to 6845 ± 105 Cal BP (Rose and Usik, 2009; Hilbert et al., 2012).
Fig 5.4 Drawings of Leptolithic examples collected from Area 2, Al-hatab (Taken from Hilbert et al., 2012).
Fig 5.5 a) Dose response curve of a typical bright grain from ALH-2, with the inset showing the decay curve for the same grain. b) OSL decay curve for the dimmest grain accepted from Al-hatab samples.

Fig 5.6 Distribution of signal intensity using pseudo single grains from both ALH-1 and ALH-2, that passed the rejection criteria. Data are plotted as the proportion of the total light sum that originates from the specified percentage of the brightest grains (Duller et al., 2000).
#1  PH = 200°C/10 s  
    CH = 160°C/5 s  
    Given dose = 400 sβ  
    Measured dose = 334 ± 6 sβ  
    OD = 13 ± 2%

#2  PH = 180°C/10 s  
    CH = 180°C/5 s  
    Given dose = 400 sβ  
    Measured dose = 363 ± 7 sβ  
    OD = 13 ± 2%

Fig 5.7a) Dose recovery tests done on sample ALH-2: #1 Preheat of 200°C/10 s with cutheat of 160°C/5 s and #2 Preheat 180°C/10 s with cutheat 180°C/10 s.
Fig 5.7 b) $D_s$ values presented as radial plots showing the extent of $D_s$ overdispersion. OSL data analysed by the author (LL) are shown for: ALH-1 (48 ± 5%) and ALH-2 (30 ± 3%). The same data analysed by Zenobia Jacobs (ZJ): ALH-1 (41 ± 5%) and ALH-2 (36 ± 3%). The grey bands are centred on the Central Age Model (CAM) with estimates of the $D_s$ for each sample.
Fig 5.8 Stratigraphic column depicting OSL ages for samples ALH-1 (2.3 ± 0.2 ka) and ALH-2 (19.5 ± 1.5 ka), as well as AMS $^{14}$C age (6845 ± 105 Cal BP) from hearth ash. NLT assemblage was located between units GH5 and GH7.
<table>
<thead>
<tr>
<th>Geo Units</th>
<th>Arch Units</th>
<th>Archaeological findings</th>
</tr>
</thead>
<tbody>
<tr>
<td>GH1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>GH2a</td>
<td>Level 1</td>
<td>Hearth ash AMS $^{14}$C 6845 ± 105 Cal BP.</td>
</tr>
<tr>
<td>GH2b</td>
<td>Level 1</td>
<td>Iron Age Pottery layer</td>
</tr>
<tr>
<td>GH3</td>
<td>Level 1</td>
<td>Sandy layer devoid of artefacts (OSL ALH-1)</td>
</tr>
<tr>
<td>GH4</td>
<td>Level 2</td>
<td>Early NLT</td>
</tr>
<tr>
<td>GH5</td>
<td>Level 2</td>
<td>Early NLT (Terminal Pleistocene)</td>
</tr>
<tr>
<td>GH6</td>
<td>Level 2</td>
<td>Early NLT (Terminal Pleistocene)</td>
</tr>
<tr>
<td>GH7</td>
<td>Level 2</td>
<td>Early NLT (Terminal Pleistocene)</td>
</tr>
<tr>
<td>GH8</td>
<td>Level 2</td>
<td>Early NLT (Terminal Pleistocene)</td>
</tr>
</tbody>
</table>

Table 5.1 Geological units, archaeological units, and the archaeological findings at Al-hatab, Area 3 (2010-2011 excavations) (Using data collected from Mike Morley, 2011).
Table 5.2 Summary of grains rejected according to the formal rejection criteria of (2003, 2006a, 2006c) for Al-hatab (ALH) samples.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Grains measured</th>
<th>Criterion 1</th>
<th>Criterion 2 RR ratio</th>
<th>Criterion 3 IR ratio</th>
<th>Criterion 4 Recuperation</th>
<th>No. grains rejected ≥ 2 criteria</th>
<th>Total no. rejected</th>
<th>Total no. Accepted</th>
<th>Final no. grains</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>ALH-1</td>
<td>1000</td>
<td>478</td>
<td>272</td>
<td>557</td>
<td>18</td>
<td>325</td>
<td>862</td>
<td>138</td>
<td>135</td>
<td>14%</td>
</tr>
<tr>
<td>ALH-2</td>
<td>1000</td>
<td>450</td>
<td>225</td>
<td>465</td>
<td>480</td>
<td>620</td>
<td>852</td>
<td>148</td>
<td>136</td>
<td>14%</td>
</tr>
<tr>
<td>Total</td>
<td>2000</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>=2000</td>
<td></td>
<td></td>
<td>14%</td>
</tr>
</tbody>
</table>

1 OSL signals are weak (T_N signal less than 3 times the instrumental background)
2 Recycling ratio is poor (i.e. more than 2 standard errors away from unity)
3 Infrared simulation causes significant loss of OSL signal (i.e. OSL-IR depletion ratios smaller than unity by more than two standard errors)
4 Recuperation is high (L_X/T_X for 0 Gy dose is greater than 5% of L_N/T_N)
5 Total no. rejected grains plus total no. accepted grains will equal the no. of grains measured.
6 Total no. of accepted grains for dating.
7 Percentage of finalised grains used in CAM
### Operator Variance

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Lauren Linnenlucke</th>
<th>Zenobia Jacobs</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$D_e$ (Gy)</td>
<td>Age (ka)</td>
</tr>
<tr>
<td>ALH-1</td>
<td>2.87 ± 0.15</td>
<td>2.3 ± 0.2</td>
</tr>
<tr>
<td>ALH-2</td>
<td>18.60 ± 0.20</td>
<td>19.5 ± 1.2</td>
</tr>
</tbody>
</table>

*Table 5.3* Operator variation showing ages and $D_e$ values obtained by two analysts on samples from Al-hatab (ALH) by LL and ZJ.

### Gamma Comparison

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Gamma (in situ)</th>
<th>Gamma (HRGS)</th>
<th>Gamma (TSAC + GM 25-5 Beta counting)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ALH-1</td>
<td>0.23 ± 0.01</td>
<td>0.33 ± 0.01</td>
<td>0.37 ± 0.02</td>
</tr>
<tr>
<td>ALH-2</td>
<td>0.25 ± 0.01</td>
<td>-</td>
<td>0.25 ± 0.02</td>
</tr>
</tbody>
</table>

*Table 5.4* Gamma comparison of field gamma measurements (column 2) against laboratory gamma measurements (column 3) based on high resolution gamma spectrometry and a combination of thick source alpha counting and GM-25-5 beta counting (column 4).
### Table 5.5 Table of OSL ages for sediment samples from the Al-Hatab (ALH), together with the supporting dose rate and equivalent dose $D_e$ data.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Sample location (Lat. Long.)</th>
<th>Sample depth (cm)</th>
<th>Field water content (% dry mass) $^1$</th>
<th>$\gamma$ dose rate $^2$ (Gy/ka$^{-1}$)</th>
<th>$\beta$ dose rate $^3$ (Gy/ka$^{-1}$)</th>
<th>Cosmic-ray dose rate $^4$ (Gy/ka$^{-1}$)</th>
<th>Total dose rate $^5$ (Gy/ka$^{-1}$)</th>
<th>$D_e$ (Gy) $^6$</th>
<th>$\sigma_d$ (%) $^7$</th>
<th>Age (ka) $^8$</th>
</tr>
</thead>
<tbody>
<tr>
<td>ALH-1</td>
<td>N 17° 31’ 34.17” E 54° 06’ 10.5”</td>
<td>26 cm</td>
<td>3.0 ± 1.5 (0.4)</td>
<td>0.22 ± 0.01</td>
<td>0.58 ± 0.03</td>
<td>0.19 ± 0.02</td>
<td>1.03 ± 0.05</td>
<td>2.87 ± 0.15</td>
<td>48 ± 5</td>
<td>2.3 ± 0.2</td>
</tr>
<tr>
<td>ALH-2</td>
<td>N 17° 31’ 34.17” E 54° 06’ 10.5”</td>
<td>69 cm</td>
<td>3.0 ± 1.5 (0.2)</td>
<td>0.25 ± 0.01</td>
<td>0.37 ± 0.02</td>
<td>0.17 ± 0.02</td>
<td>0.82 ± 0.04</td>
<td>18.60 ± 0.20</td>
<td>30 ± 3</td>
<td>19.5 ± 1.2</td>
</tr>
</tbody>
</table>

1. Field water content with measured water content in brackets.
2. From field gamma spectrometry measurements and adjusted for field moisture content.
3. Corrected for beta-dose attenuation and adjusted for the field moisture content.
4. From Prescott and Hutton (1994), assigned relative uncertainties of ± 10%, and adjusted for the field moisture content. Time-averaged burial depth assumed steady sediment deposition.
5. Includes an assumed internal alpha dose rate of 0.03 ± 0.01 Gy/ka$^{-1}$.
6. Mean ± standard (1σ) error, determined using the Central Age Model (Galbraith et al., 1999).
7. $D_e$ overdispersion (i.e. the spread in the $D_e$ values after taking all measurement uncertainties into account).
8. Mean ± total (1σ) uncertainty, with the latter calculated as the quadratic sum of the random and systematic uncertainties.
Chapter 6
6.1 Introduction

This chapter discusses the timing and possible causes of the hunter-gatherer to pastoralist development within western Oman. By obtaining pseudo single-grain ages established in this study, the specific aims, provided in section 1.7 will be addressed individually along with previous dating and contextual information which is provided in the site-specific chapters.

6.2 Timing and possible causes

This study has provided interpretations for OSL ages and applied them to individual site stratigraphy for the three sites (Ghazal, Khumseen and Al-hatab). All sites will be discussed in relation to one another with relevance to the NLT, and this will be applied to my proposed aims:

1) Using pseudo single-grain analysis to provide accurate ages for Ghazal, Khumseen and Al-hatab, we have been able to place them within an appropriate time period.
2) This will enable the leptolithic assemblages located at the three sites to be placed on a suitable timescale or to be adjusted from previous dating ages given.
3) Doing this will enable the sites and NLT assemblages to be discussed collectively in regards to the two proposed models; whether the leptolithics in Oman represents the development of an in situ human population with unique stone tool industries and genetic signatures.
4) Whether it was the result of a population expansion from the western Mediterranean (the Levant) and/ or an expansion out of Africa (Bab al Mandab Strait). Therefore, the transitional period from hunter-gatherer to pastoralists within western Oman will be established in this study.
The first two aims will be addressed within each site interpretations for Ghazal, Khumseen and Al-hatab, and the final two aims will be discussed within migration patterns.

6.3 Site interpretation

6.3.1 Ghazal

Interpretation of Ghazal was done through analysing the $D_e$ distribution and OSL ages, and applying them to site depositional information. Four OSL ages were obtained in order to date the site, and thereby providing a time range for the Leptolithic assemblages located within Units GH2 and GH4 (see Fig 3.2a).

The four samples gave ages of between 7 and 10 ka, based on pseudo single-grain analysis. Sample TH47-4 was dated to $7.1 \pm 0.5$ ka, providing a minimum age for the NLT within unit GH2. TH47-1 gave an age of $7.5 \pm 0.5$ ka, resulting in a maximum age for Unit GH2 and a minimum age for Unit GH4. Unit GH5 gave two ages of $9.7 \pm 0.7$ ka (TH47-3) and $8.2 \pm 0.7$ ka (TH47-2), providing an approximate age of 8.5 ka for the NLT. The pseudo grains indicate a $D_e$ distribution of mixed sediments; this was due to in situ decomposed blocks of weathered limestone which were located within the rock shelter. A mixed $D_e$ is indicated through levels of high overdispersion values ranging from $37 \pm 4\%$ to $72 \pm 5\%$ (see Fig 3.7). Localised annual monsoonal periods may have resulted in the breakdown of calcrete nodules throughout the sediments, which may have caused the finer sediments to shift through the profile.

The NLT assemblages located within the stratigraphic profile of Ghazal have been narrowed down to between 7 and 10 ka; this has enabled the assemblages to be placed within a Late Leptolithic time period.

6.3.2 Khumseen

Khumseen was interpreted using depositional information provided by applying $D_e$ distribution and OSL ages to date the NLT assemblage and the site. Four OSL ages were
obtained using pseudo single-grains; these were compared against two single-aliquot dates for the lower units, and an AMS $^{14}$C hearth ash age.

The NLT assemblage located within Units GH5a and GH5b has been dated to between ~8 to 10 ka. The pseudo single-grain ages for four OSL ages are provided in order to date the assemblages, giving more information regarding the transitional period from hunter-gatherer to pastoralists. The sample TH50-4 is dated to 5.9 ± 0.5 ka for Unit GH4b. Samples TH50-3 and TH50-2 are located within the same unit, giving ages of 5.6 ± 0.7 ka and 7.8 ± 0.7 ka, respectively. TH50-2 is considered the more reliable of the two ages, due to less overdispersion. TH50-1 provides a maximum age for the sequence of 9.9 ± 0.8 ka. The $D_e$ distribution suggests well bleached sediments that have been undisturbed since deposition (Fig 4.7). This is indicated by the spread located within the radial plots, as no presence of contamination was found in other layers or part of the rock shelter itself (weathered limestone). The combination of AMS $^{14}$C ash age from GH4b is dated to 6845 ± 105 Cal BP and is consistent at 2σ with sample TH50-3. Khumseen differs to the other two sites as there is no evidence of decomposed sediments from weathered limestone throughout the stratigraphic profile. Al-hatab and Ghazal are approximately 500 m apart whereas Khumseen is 2 km and appears to be less weathered, thereby suspecting a perhaps more sheltered environment.

The NLT at Khumseen was dated to around 8 to 10 ka, indicating a Late Leptolithic assemblage; this Late Leptolithic period indicates that the rock shelter was occupied by humans during the transition from hunter-gatherer to pastoralists. Three hearth ash fires were located throughout the stratigraphy between Units GH5a to GH4b, and two were located between Units GH3 and GH2. Humans may have occupied the site from ~7 ka and onwards; evident by AMS $^{14}$C. This would imply that the Leptolithic stone tools are older (8-10 ka) than the hearth ash (~7 ka). There is no evidence of Leptolithics located within the same layers containing ash. This may be due to the movement of humans leaving the rock shelter as a result of the climatic conditions of the Indian Ocean Monsoon System. Further speculation suggests that the development of Leptolithics later than ~7 ka may not have occurred at this site.
6.3.3 Al-hatab

The interpretation of Al-hatab was done through applying site depositional information to $D_e$ distribution and OSL ages. Al-hatab had been previously dated using single aliquots. The single-aliquot dates gave older ages than suspected; therefore it was considered necessary to date Area 3 using pseudo single-grains. Two ages were obtained using pseudo single grains; a maximum age of 19.5 ± 1.2 ka (ALH-2) and a minimum age of ~2 to 3 ka (ALH-1) for the NLT.

The $D_e$ distribution of ALH-1, as well as stratigraphic integrity of the site observed through field observations made by Roberts, indicate evidence of weathered limestone blocks within the rock shelter. There was also evidence of sediment mixing, observed by the indistinct boundary of GH2 into the underlying layer GH4 of the stratigraphy. Either of these may have been the cause of an increased overdispersion and $D_e$ distribution indicating post-depositional mixing between Unit GH2 and Unit GH4. Therefore the age of ALH-1 may not be accurate as a result of contamination.

Pseudo single-grain ages from Al-hatab have indicated that the Leptolithic assemblages belong to both early (ALH-1) and late (ALH-2) phases. By doing pseudo single-grain dating, the sediments within the profile have been dated, and applied to the NLT, thereby allowing us to extend the time period of both the minimum and maximum ages of Al-hatab by approximately 10 ka. This includes the time period suggested for the NLT from Ghazal and Khumseen, however represents a much broader age range. As discussed above, the $D_e$ distributions suggest contamination that can only be further resolved using true single-grain analysis. Thus, this broader range may be an artefact of inaccurate ages. As such, from a site formation and stratigraphic viewpoint, these ages are at least informative in regards to accurately pinpointing the age duration of any NLT within western Oman.

6.4 Nejd Leptolithic stone tools

The NLT located at Ghazal and Khumseen are both classified as Late Leptolithics and are dated to the same time period. This would imply that the Leptolithics located at Ghazal
are the same tools found at Khumseen; Ghazal and Al-hatab are within close proximity to one another as compared to Khumseen. The ages given at Khumseen are the only ages to not show any form of contamination, unlike Ghazal which indicates a possible mixing of sediments. The core reduction modules of the NLT indicate that Ghazal and Khumseen do not share the same Modalities (refer to section 1.3.1), however both are in the same time bracket. Khumseen shows characteristics specific to Modality two and three, whereas Ghazal is found to contain features of Modality one; however this is also found to be present within Modality two. We can infer that the Leptolithics from Ghazal and Khumseen may show similar characteristics, and therefore cannot dispute the ages based on the contaminations of mixed sediments.

The stone tools for Al-hatab are dated to incorporate the entire age range of all three sites; therefore it is likely that the leptolithics show signs of changes in human development, as humans left the rock shelter and returned. Al-hatab indicates features from all modalities, incorporating features similar to Ghazal and Khumseen; this would imply that the pseudo single-grain ages match the outcomes of the core reduction modalities established in section 1.3.1.

6.5 Migration Patterns

The NLT proposes three initial core reduction modules which categorise the presence of Leptolithics within western Oman, essentially allowing variations to be applied to the NLT. By providing OSL ages for Al-hatab, Ghazal and Khumseen, the NLT assemblage within each site has now been categorised into early or late. This now allows the sites to be examined collectively in regards to the two proposed models: whether or not the presence of NLT assemblages represents an in situ population development of hunter-gatherer to pastoralists, or was a result of a population expansion from the western Mediterranean (the Levant) and/ or an expansion out of Africa (Bab al Mandab Strait).

The first model is the most likely scenario for a number of reasons. An in situ human population with unique signatures is indicated by the modalities described within section 1.3.1 for the NLT. There is also no connection of the NLT outside of western Oman,
indicating specific development of toolkits during this period by developing pastoralists. Another reason for the development of hunter-gatherer to pastoralists as an *in situ* population is the results of the OSL ages. The pseudo single-grain ages obtained in this study indicate that humans occupied all three sites either across the Holocene or the Pleistocene-Holocene boundary. As western Oman receives annual monsoonal weather, it is plausible to propose that humans during the wet season would move into higher regions e.g. the Dhofar Mountains, and then settle back into the same area thereby resulting in a development of unique stone tool industries (NLT).

Two of the migration patterns out of the second model are less plausible for the following reason. The bladed technology of the NLT differs from the Levantine variations in blades, thereby refuting a population expansion from the Mediterranean, i.e. River Nile and the interior of Arabia (Hilbert et al., 2012), where humans brought their toolkits with them from outside. Leptolithic assemblages of western Oman also bear little resemblance to eastern African toolkits, thus suggesting that movement from the Horn of Africa is less likely.

The first model is the most plausible scenario due to the influence of annual monsoonal activity as a result of the Indian Ocean Monsoon System. This may be attributed to the changes in climatic conditions over the Quaternary that resulted in human populations of developing pastoralists to seek refuge in highland regions during monsoonal fluctuations. Also the development of NLT through core reduction modalities is seen to have no or little connection outside of other Leptolithic assemblages, thereby making this toolkit unique to western Oman.

6.6 Conclusion

This study has accomplished four main aims. Firstly it has provided accurate pseudo single-grain ages for three sites on the Nejd Plateau in western Oman. Next, using these ages we have estimated narrower time frames for NLT assemblages within these sites. Thirdly the proposed model indicating an *in situ* population has been discussed in relation to current research on palaeoclimatology, NLT comparisons and accurate OSL ages. This
indicates that humans during the development from hunter-gatherer to pastoralists occupied the intended sites during the Pleistocene-Holocene boundary. Finally the second model has been disputed due to current research on palaeoclimatology (MIS 5) and stone tool taxonomic variation.

Future work should focus on applying ‘true’ single-grain analysis to the dating of the sediments. This will allow us to untangle statically any mixing that will then result in the improved accuracy of the age determinations.
References
References


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Appendices
Appendix 1 - “High-Resolution Gamma-Ray Spectrometry (HRGS)”

The radionuclide activities determined by HRGS are listed in Table A.1, together with the corresponding beta and gamma dose rates that have been adjusted for water content at a rate of 3.0 ± 1.5%. The data presented in Table A.1 shows that the $^{232}$Th decay chain are in a state of secular equilibrium (the only exception being sample ALH-1); this is not unusual for terrestrial sediments (Olley et al., 1996, 1997). This is a direct result of radon gas leaking into the atmosphere which causes the $^{238}$U decay chain to have a deficit of $^{210}$Pb relative to $^{226}$Ra in samples. The HRGS beta dose rates show similar measurements directly related to GM-25-5 beta counting, however these are systematically lower. For consistency with the other samples, not examined using HRGS, the OSL ages have been calculated using the beta dose rates determined by GM-25-5 beta counting.

The HRGS gamma dose rates values are similar to the field gamma-ray spectrometry measurements for samples TH47-1 and TH47-2; this also includes the values estimated from a combination of beta counting and thick-source alpha counting for samples TH50-2 and TH50-4. Again the sample ALH-1 differs, as it shows a HRGS gamma dose rate 1.5 times that measured in the field. This can be explained by the consequence of high density limestone rocks in the Al-hatab deposit. These low-radioactivity rocks will have influenced the field measurement of the gamma dose rate, whereas the laboratory (HRGS) measurements were made on the sedimentary matrix, which is more radioactive than the limestone. The calculation of OSL ages, as done in this study, should be done using field measurements of the gamma dose rates when within close range to limestone rocks.
<table>
<thead>
<tr>
<th>Radionuclide activities (Bq/kg)</th>
<th>TH47-1</th>
<th>TH47-2</th>
<th>TH50-2</th>
<th>TH50-4</th>
<th>ALH-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^{238}$U</td>
<td>15.02 ± 0.83</td>
<td>28.24 ± 1.74</td>
<td>12.80 ± 1.33</td>
<td>13.95 ± 1.25</td>
<td>12.81 ± 1.22</td>
</tr>
<tr>
<td>$^{226}$Ra</td>
<td>18.05 ± 0.26</td>
<td>28.54 ± 0.48</td>
<td>11.31 ± 0.24</td>
<td>15.08 ± 0.30</td>
<td>15.71 ± 0.30</td>
</tr>
<tr>
<td>$^{210}$Pb</td>
<td>15.46 ± 1.00</td>
<td>19.53 ± 2.07</td>
<td>7.94 ± 1.11</td>
<td>10.92 ± 1.61</td>
<td>13.25 ± 1.51</td>
</tr>
<tr>
<td>$^{228}$Ra</td>
<td>4.61 ± 0.22</td>
<td>11.12 ± 0.56</td>
<td>3.42 ± 0.29</td>
<td>4.22 ± 0.38</td>
<td>8.61 ± 0.45</td>
</tr>
<tr>
<td>$^{228}$Th</td>
<td>4.36 ± 0.17</td>
<td>10.74 ± 0.41</td>
<td>2.92 ± 0.22</td>
<td>4.22 ± 0.38</td>
<td>8.61 ± 0.45</td>
</tr>
<tr>
<td>$^{40}$K</td>
<td>65.56 ± 1.97</td>
<td>157.29 ± 5.54</td>
<td>81.40 ± 2.95</td>
<td>91.88 ± 3.80</td>
<td>136.97 ± 4.50</td>
</tr>
<tr>
<td>HRGS beta dose rate (Gy/ka)</td>
<td>0.33 ± 0.02</td>
<td>0.66 ± 0.03</td>
<td>0.31 ± 0.02</td>
<td>0.36 ± 0.02</td>
<td>0.51 ± 0.02</td>
</tr>
<tr>
<td>GM-25-5 beta dose rate (Gy/ka)</td>
<td>0.35 ± 0.02</td>
<td>0.71 ± 0.05</td>
<td>0.33 ± 0.02</td>
<td>0.42 ± 0.02</td>
<td>0.58 ± 0.03</td>
</tr>
<tr>
<td>HRGS/ GM-25-5 Beta dose rates</td>
<td>0.94 ± 0.08</td>
<td>0.93 ± 0.08</td>
<td>0.94 ± 0.08</td>
<td>0.86 ± 0.06</td>
<td>0.88 ± 0.06</td>
</tr>
<tr>
<td>HRGS gamma dose rate (Gy/ka)</td>
<td>0.23 ± 0.01</td>
<td>0.41 ± 0.02</td>
<td>0.16 ± 0.01</td>
<td>0.21 ± 0.02</td>
<td>0.33 ± 0.01</td>
</tr>
<tr>
<td>Other gamma dose rate (Gy/ka)</td>
<td>0.24 ± 0.01</td>
<td>0.47 ± 0.01</td>
<td>0.17 ± 0.01</td>
<td>0.24 ± 0.01</td>
<td>0.22 ± 0.01</td>
</tr>
<tr>
<td>HRGS/ other gamma dose rates</td>
<td>0.96 ± 0.06</td>
<td>0.87 ± 0.05</td>
<td>0.94 ± 0.08</td>
<td>0.88 ± 0.09</td>
<td>1.50 ± 0.09</td>
</tr>
</tbody>
</table>

Table A.1  High-resolution gamma-ray spectrometry (HRGS) measurements of radionuclide activities in the $^{238}$U and $^{232}$Th decay chains, and of $^{40}$K, in five dried and powdered samples, and the corresponding beta and gamma dose rates calculated at 3% field water content. Also listed are the beta dose rates obtained by GM-25-5 beta counting and the gamma dose rates measured by field gamma-ray spectrometry (or from beta counting and thick-source alpha counting for the TH50 samples), all adjusted to a field water content of 3%.
Appendix 2 - Geological Survey of Area 3 (Al-hatab)

Geological survey provided by Mike Morley, Geologist from the Oxford Brooks University, whose field observations were conducted during DAP excavations in 2010-2011. These interpretations were adopted into Chapter 3 for site interpretations and geological depositional information.

1. Al-hatab Rock shelter, Southern Nejd Plateau

Al-hatab has already been described in terms of its archaeological and geoarchaeological sequence, and the current work follows on from this research. In order to generate more archaeological data and to gain a better insight into the sedimentation processes occurring at the site (and the provenance of these sediments) a new test pit was opened approximately 2 m southwest from the previous test pit.

Area 3: Stratigraphic succession

The sequence is outlined in Table 1 and in very general terms comprises a minerogenic suite of gravel- and silt-dominated sediments with negligible included material except that related to human use of the rock shelter (i.e. anthropogenically worked chert). Units 1 and 2 most likely relate to deposition during the mid to late Holocene, and certainly the lower part of 2 (2a) appears be evidence of the use of fire at the site in the form of a hearth. The upper part is the reworked (‘raked out’) portion of this hearth which is intermixed with surface sediments.

Unit 3 is a poorly sorted diamict, containing a very wide range of fine and coarse components, which in its landscape context suggests a colluvial deposit probably deriving from the mechanical and chemical breakdown of both the walls and roof of the rock shelter, possibly with some additional input from the plateau above the shelter. Certainly the sediments thicken towards the northwest which suggests that the material originates from the area of the rock shelter and not from the small tributary wadi in which Al-hatab is formed.
Silt and sand make up the sediments of unit 4 and these sediments are moderately compact and homogeneous suggesting deposition in a relatively rapid event, most likely as an episode of windblown activity. Some fine gravel interspersed within the otherwise fine-grained matrix is likely to have rolled downhill from upslope sources and as there is not a discrete lens of coarser material it is unlikely that there has been a sufficiently long break during this period of deposition.

Units 5 and 7 are particularly notable as the coarse component comprises fine gravel which is not recorded elsewhere in the exposed profile. Whereas imbricated gravels in unit 3 indicate movement downslope from the rock shelter (and plateau above), the fine gravel in these units can be seen to bank up against larger cobbles, and imbricated gravels in the adjacent section suggest that they were deposited by the wadi, possibly during an ephemeral flood event. There may be more than one of these short-lived wadi re-activations as narrow (0.5 to 1.2 m) channel-like profiles can be differentiated within these units. Given the Pleistocene dates for contiguous units in the adjacent test-pit, along with the archaeological material which has Upper Palaeolithic affinities, it is just possible that this period of episodic wadi re-activation relates to a brief Late Pleistocene pluvial episode broadly correlated with the Bolling-Allerod (BA) interstadial (refs in Parker 2009, p. 45) at 15 – 13 ka BP. It should be emphasised that this unit does not solely represent fluvial sedimentation, but is fluvial deposition (most likely as a wash) of fine gravel over a wadi-bank proximal area at the front of the rockshelter. Colluvial sedimentation is likely still the dominant geomorphological process during this period.

Unit 6 is ambiguous, and may just form the division between the fluvial/colluvial episodes represented by units 5 and 7. However, it could be the eroded remnants of a depositional event similar to that related to unit 4 which has been heavily truncated by the deposition of unit 5.

At the base of the sequence is unit 8, which is a thick (0.40 m where seen in section) accumulation of silt and fine sand which contains occasional coarser components. This unit is well-cemented and could represent deposition in a hyper-arid phase in the terminal Pleistocene.
<table>
<thead>
<tr>
<th>GeoArch unit</th>
<th>Description</th>
<th>Depositional environment/interpretation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Mod compact, v. pale brown (10YR 7/4), fine to medium gravel in a sandy silt matrix. Freq modern organics and charcoal and occ to mod worked chert</td>
<td>Recent and sub-recent sediments</td>
</tr>
<tr>
<td>2</td>
<td>Weakly to mod compact, light yellowish brown (10YR 6/4) sandy silt with freq fine to medium gravel. Contains low quantity of finely divided charcoal powder</td>
<td>Mixture of aeolian sand, bedrock attrition and reworked ash and charcoal. Most likely Holocene</td>
</tr>
<tr>
<td>2a</td>
<td>Weakly to mod compact, light brownish grey (10YR 6/2) fine silt with high quantity of finely divided charcoal powder. Only present as a discontinuous band</td>
<td>This unit is dominated by ash and charcoal and is thought to represent a mid to late Holocene (?IA) hearth</td>
</tr>
<tr>
<td>3</td>
<td>Weakly to mod compact, v. pale brown (10YR 7/4), poorly sorted, sub-a to sub-r, fine to medium limestone gravel in a sandy silt matrix (10YR 7/4 v. pale brown). Includes mod quantities of worked chert</td>
<td>Colluvial and aeolian surface sediments</td>
</tr>
<tr>
<td>4</td>
<td>Mod to densely compact, v. pale brown (10YR 7/4), homogeneous sandy silt with occ to mod, sub-a to sub-r, fine to medium limestone gravel</td>
<td>Aeolian sands, most likely deposited in a relatively short continuous event</td>
</tr>
<tr>
<td>5</td>
<td>Weakly compacted, sub-a to sub-r, fine limestone gravel in a sandy coarse silt matrix (10YR 7/4 v. pale brown). Contains occ to mod quantities of medium sub-a to sub-r gravel, and v. occ large limestone clasts which fine gravel is banked up against on the SE (wadi-side). Imbricated gravel aligned southwest-northeast observed in adjacent NW-facing section</td>
<td>Second phase of fluvial sedimentation which occurs in tandem with continued colluvial sedimentation, and most likely some aeolian input</td>
</tr>
<tr>
<td>6</td>
<td>Essentially the same as unit 4 in physical characteristics</td>
<td>Possibly heavily truncated aeolian sand deposition</td>
</tr>
<tr>
<td>7</td>
<td>Essentially the same as unit 4 in physical characteristics</td>
<td>First phase of fluvial sedimentation which occurs in tandem with continued colluvial sedimentation, and most likely some aeolian input. Bolling-Allerod interstadial fluvial sediments??</td>
</tr>
<tr>
<td>8</td>
<td>Well cemented, v. pale brown (10YR 7/4) sandy silt with occ to mod sub-a to sub-r, fine to medium (medium dominated) limestone gravel. Thick and homogeneous unit with massive structure</td>
<td>Sedimentation in a stable environment, possibly Terminal Pleistocene</td>
</tr>
</tbody>
</table>

**Table A.2** Lithological characteristics of southwest-facing section in Al-hatab Area 3
(Provided by Mike Morley, Oxford University, 2010-2011)