ESR in the 21st Century:

From Buried Valleys and Deserts to the Deep Ocean and Tectonic Uplift

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1.0 Abstract

Electron spin resonance (ESR) dating can date many materials, including hydroxyapatite in enamel and some fish scales, aragonite and calcite in corals, molluscs, some travertine and calcrete, and quartz from ash, fluvial deposits, and some flint. Dating studies using these materials have numerous potential applications in many varied Quaternary settings. ESR dating uses signals resulting from trapped charges created by radiation in crystalline solids. Ages are calculated by comparing the accumulated radiation dose in the dating sample with the internal and external radiation dose rates produced by natural radiation in and around the sample and produced by cosmic radiation. When compared to other dating techniques, age agreement has been excellent for teeth, corals, molluscs, and quartz. Recent improvements have included using a more complex modelling technique to calculate the cosmic dose rates and more detailed modelling techniques for dealing with variable external dose rates. Methods in development include using quartz from buried fluvial valleys to date geomorphic surfaces and using the signals in barnacles and benthic foraminifera for dating fossils or their associated sediment. New chronometer applications recently developed include using coral and mollusc dates to build sealevel curves and to monitor volcanic activity and tectonic uplift, using tooth and mollusc dates to assess water availability in deserts, and using isochron data to assess U uptake processes into teeth. When coupled with other geochemical and geomorphological techniques, ESR can provide the chronometric control to build paleoclimatic and other paleoenvironmental records. Many other applications are possible, from heating studies for artefacts to dating sulphates and other minerals on distant planets.

Key words: ESR dating; Quaternary applications; archaeological applications; paleontological applications; sealevel change, paleoenvironmental studies; hominin fossil dating; Paleolithic artefact dating.

2.0 Introduction

Electron spin resonance (ESR) dating can provide chronometric (absolute) dates over a substantial time range, from as young as 0.5 ka to about 2-5 Ma, currently with 2-8% precision. Like thermo- (TL), optically stimulated (OSL), and radio- luminescence (RL), ESR detects trapped charges in minerals induced by radiation, but it provides several advantages over rival methods. For example, unlike TL, OSL, and RL, ESR does not need signals that have been completely zeroed for most applications. Moreover, ESR signals, unlike some TL, OSL, and RL signals, do not suffer anomalous fading. None of these other trapped charge methods can date samples older than 200-500 ka (Figure 1). While TL and OSL have become very popular for dating sediment particularly in aeolian sediment, they cannot date fossils. Consequently, by limited to dating the Late-Middle Quaternary at the best, TL and OSL provide less useful archaeological and paleontological data over a much shorter time range than ESR.

Compared to the radiometric methods, ESR also provides several advantages. ESR can date fossils much older than the ^{14}C dating limit. ^{14}C dates organic material only as old as $\sim 40\text{-}50$ ka, and cannot date non-organic material (Figure 1). ESR does not require a handy volcano to produce datable rocks like $^{39}\text{Ar}/^{40}\text{Ar}$ needs, because ESR can date fossils and sediment directly. $^{39}\text{Ar}/^{40}\text{Ar}$ can provide precise ages for samples as young as about 0.2-0.5 Ma, but rarely can date sediment or igneous rocks in the later Quaternary. Unlike the uranium (U) series methods, ESR can date most mollusc species accurately, as well as authigenic cements, some clays, and aeolian sediment. Although $^{230}\text{Th}/^{234}\text{U}$ can date speleothems, coral, and molluscs from 1-5 ka until about 500-600 ka, it cannot date vertebrate fossils with precision due to U uptake issues (see below). Again, ESR can date a range ~ 10 times longer than $^{230}\text{Th}/^{234}\text{U}$. The U-Pb methods, $^{238}\text{U}/^{206}\text{Pb}$, $^{235}\text{U}/^{207}\text{Pb}$ and $^{232}\text{Th}/^{208}\text{Pb}$, all can date igneous rocks or uraniferous materials that are much older than ESR can, but they can rarely produce precise ages for Quaternary materials less than 1.0-2.0 Ma.

ESR's potential to date a wide variety of sample types will undoubtedly continue to make it an important research tool in late Cenozoic settings. ESR can date many materials that are commonly encountered in geological, paleontological, and archaeological contexts, and new applications are constantly being tested (e.g., Debuyst *et al.*, 2001; Dibble *et al.*, 2013a, 2013b; Fukuchi, 2001; Godfrey-Smith *et al.*, 2003; Gotze & Plotze, 1997; Lee & Schwarcz, 2001; Molod'kov, 2001; Patnaik *et al.*, 2009; Potter *et al.*, 2004; Schellman & Radtke, 2001; Rink *et al.*, 2003; Shih *et al.*, 2002; Skinner *et al.*, 2001b, 2005a; Tani *et al.*, 1998; Ulusoy 2003, 2004; Whitehead *et al.*, 2002; Woda *et al.*, 2001). In archaeological contexts, ESR has dramatically changed our understanding of human origins and cultures (e.g., see references in Blackwell, 2001, 2006; Falguères, 2003). Here, we review the development of several new materials suitable for ESR dating and several recent applications of standard techniques to new geological and archaeological problems.

3.0 Principles of ESR Analysis

When natural radiation, such as α , β , γ , or high-energy cosmic radiation, hits minerals, such as hydroxyapatite, aragonite, calcite, or quartz, the minerals gradually accumulate trapped unpaired electrons and positively charged "holes" (Figure 2a), which each produce characteristic ESR signals detectable with an ESR spectrometer. Most minerals acquire several ESR signals, many of which result from defects in the crystalline lattice associated with trace contaminants. If the ESR signal height (intensity) for a radiation-sensitive signal can be converted into an accumulated dose (Figure 2b) and if the radiation dose rates experienced by the mineral from different sources around and in the mineral during its geologic history is known or can be modelled (Figure 3a), an ESR date can be calculated. ESR dates can be obtained for any material with a radiation-sensitive ESR signal (e.g., Figure 3b), provided it satisfies the following criteria,

1. At the time of interest, the mineral's ESR signal was 0.0 (i.e., no detectible peak existed), or its initial value can be estimated or modelled.
2. The signal lifetime, τ , exceeds the site age by at least two orders of magnitude.
3. The ESR signal is not saturated (i.e., the accumulated dose, $\mathcal{A}_\Sigma$, can still grow if more radiation were added).

Although ESR has been routinely applied for more than 25 years to teeth and molluscs, new applications of the method appear regularly in the literature.

By applying a microwave signal with a variable field and a strong, externally applied, magnetic field that together resonate with the tiny magnetic fields generated by the unpaired electrons or the holes, an ESR spectrometer can produce a spectrum that displays several signals simultaneously. Lande's factor, called the g value, is a dimensionless number that uniquely describes the ESR characteristics for any peak on the spectrum. Most geological and archaeological applications are currently done in the X band, but pulsed X-, K-, L-, and Q-band ESR may ultimately prove to be useful for identifying and detecting small or interfering signals (e.g., Skinner *et al.*, 2000; Kinoshita *et al.*, 2004). In the X band, spectra are analyzed at 1-10 mW power using microwave frequencies near 8-10 GHz under a 100 kHz field modulation, and thus, most geologically or archaeologically interesting ESR signals fall within $3 < g < 1.9$ (see Blackwell, 1995, Table 2).

When an ESR signal zeroes, its intensity becomes indistinguishable from background levels. Most newly formed minerals have no measurable ESR signals. Once a measurable signal exists, heating to temperatures above 125-500°C, depending on the mineral, will zero most ESR signals (Figure 4b; e.g., Bensimon *et al.*, 2000). Although some signals in a few minerals, especially in quartz, will zero when exposed to light (Figure 4a; e.g., Bartoll *et al.*, 2000), most radiation-sensitive signals in most minerals do not zero easily in light. In faults, high pressure or shearing can partially or fully reset some signals, as can the strain developed during comminution during an earthquake or grinding for sample preparation (Figure 5). During remineralization and diagenesis, new minerals may form whose radiation-sensitive signals will be 0.0 (i.e., no detectible peak heights), unlike the minerals already present in the sample. Should the original and new minerals have ESR signals with similar g

values, the resultant complex signal will not give an accurate age determination. If, however, the two signals do not interfere, as is true for tooth enamel, only the dating signal's intensity is reduced, thereby, reducing the discriminatory range and dating limits for the technique (Skinner *et al.*, 2000).

The particular signal's thermal stability partially defines the method's reliability. Signals which zero at typical Earth surface temperatures rarely are used for dating, but may provide other information, such as surface reactivation. The mean signal lifetime, τ , must exceed the desired sample dating range by about 2-3 orders of magnitude for reliable ages. In tooth enamel, for example, $\tau \leq 10^{19}$ y (Skinner *et al.*, 2000), a sufficiently long time, that in theory at least, that one could date anything within the history of the universe. In most circumstances, however, the signal saturates at somewhere between 2 and 5 Ma (Figure 2b). Unlike TL and OSL, no datable ESR signal appears to suffer anomalous fading. In practice, because no crystal has an infinite number of traps, most ESR signals actually have a finite saturation limit defined in terms of added radiation dose. Beyond that limit, no new electrons can be trapped, because all the available traps are filled. Many minerals also have a steady state dose, usually at somewhat lower radiation dose than their saturation dose, caused by minor electron loss and retrapping (Figure 2b). While the mean signal lifetime and the steady state dose or the saturation dose define the maximum datable limit, the ESR spectrometer's ability to discriminate between the dating signal and its surrounding background noise sets the minimum dating limit. Both limits differ depending on the signal selected, the mineral housing the signal, and the mineral's habit in the material to be dated. The radiation dose rates experienced by the sample determine how those dose limits translate into actual age limits. If samples experience low radiation dose rates, the minimum datable age will be relative high, but so will its maximum datable age, whereas high radiation dose rates will translate into lower minimum and maximum age limits.

The ESR signal height (Figure 3b) measures the number of trapped charges at that lattice site, and, therefore, the total accumulated radiation dose, A_{Σ} , that the material has experienced. The ESR age, t_1 , the time that has elapsed since the mineral formed and began to accumulate charges, then derives from Equation 1,

$$\mathcal{A}_{\Sigma} = \mathcal{A}_{\text{int}} + \mathcal{A}_{\text{sed}} + \mathcal{A}_{\text{cos}} = \int_{t_0}^{t_1} D_{\Sigma}(t) dt = \int_{t_0}^{t_1} (D_{\text{int}}(t) + D_{\text{sed}}(t) + D_{\text{cos}}(t)) dt \quad (1)$$

- where
- $\mathcal{A}_{\Sigma}$ = the total accumulated dose in the sample,
 - $\mathcal{A}_{\text{int}}$ = the internally derived accumulated dose component,
 - $\mathcal{A}_{\text{sed}}$ = the externally derived accumulated dose component from sedimentary sources,
 - $\mathcal{A}_{\text{cos}}$ = the externally derived accumulated dose component from cosmic radiation sources,
 - $D_{\Sigma}(t)$ = the total dose rate,
 - $D_{\text{int}}(t)$ = the total dose rate from internal sources: U, its daughters, and any other radioisotopes,
 - $D_{\text{sed}}(t)$ = the total dose rate from the external environment: sedimentary U, Th, and K,
 - $D_{\text{cos}}(t)$ = the total dose rate from the external environment: the cosmic dose,
 - t_1 = the sample's age,
 - t_0 = today.

For samples in which the total dose rate, $D_{\Sigma}(t)$, is constant, this reduces to

$$t_1 = \frac{\mathcal{A}_{\Sigma}}{D_{\Sigma}(t)} \quad (2)$$

4.0 ESR Methodology

An ESR age calculation requires measuring or modelling some 30 different parameters, which affect the accumulated dose, A_{ex} , the internal, $D_{\text{int}}(t)$, sedimentary, $D_{\text{sed}}(t)$, and cosmic, $D_{\text{cos}}(t)$, radiation dose rates. Although improved spectrometers and ancillary equipment have sped the process and improved the precision, the basic ESR dating protocols were well established by 2000. Standard analytical protocols for all mineralogies require powdered samples for the ESR component. Although some ESR labs have developed “non-destructive” analyses for tooth enamel (e.g., Robertson & Grün, 2000; Miyake *et al.*, 2000), even these cause some sample destruction.

4.1 Determining the Accumulated Dose, $\mathcal{A}_{\Sigma}$

For each sample, the accumulated dose, $\mathcal{A}_{\Sigma}$, is determined with the additive dose method (Figure 6a), which requires a sample mass about 0.2-1.0 g (Table 1) in order to provide 10-15 aliquots of pristine, powdered, well homogenized mineral. Using fewer than 10 measurements for the additive dose calculation significantly lowers its precision. Except for one aliquot, a high-intensity radioactive source is used to irradiate each aliquot to a different precisely known added radiation dose. Normally, ^{60}Co γ or ^{137}Cs β are used as radiation sources. The added doses used usually range from 0.1-10 Grays for the lowest added dose to 1-25 kGy for highest, depending on the sample's A_{ex} . Since the added doses affect the curve fitting statistics and the $\mathcal{A}_{\Sigma}$'s precision for enamel (e.g., Lee *et al.*, 1997), and presumably, for other minerals also, samples with higher $\mathcal{A}_{\Sigma}$'s generally get higher added dose ranges. In the ramping technique, only 3-8 aliquots are used, but half are used to calibrate the spectrometer for each new set of ESR measurements, and the others are successively irradiated to ever higher added doses (Blackwell, 2001). While this method takes much longer, it can provide dates for very small samples, like benthic foraminifera (see below).

After measuring the ESR signal heights for both the naturally and artificially irradiated aliquots, the signal intensity (peak height) is plotted against the added dose to yield a growth curve (Figure 6a). Usually, the points are weighted inversely with intensity ($1/I^2$). In some materials, however, accurately measuring a peak height requires using signal subtraction to isolate the dating signal from interference peaks (Figure 6b). Despite controversies over measurement protocols, derivative spectra actually provide better resolution (Lyons & Tan, 2000). Most evidence also suggests that deconvolution is not necessary for many dating peaks (e.g., Skinner *et al.*, 2001a). The accumulated dose, $\mathcal{A}_{\Sigma}$, required to produce the observed natural ESR signal intensity equals the x -intercept on the growth curve. Within some materials, such as travertine, calcrete, and caliche, crystals may vary greatly in their $\mathcal{A}_{\Sigma}$. If some regions are at or near saturation, while others are younger, and hence not saturated, age underestimation may also occur, because the dose response becomes nonlinear near the saturation dose (Figure 2b). In tooth enamel, however, the linear behaviour persists to large doses (Brennan, 2000).

Generally, $\mathcal{A}_\Sigma$ can be measured with 0.8-5% precision, depending on the spectrometer's calibration (Nagy, 2000), the radiation source calibration (Wieser *et al.*, 2005a, 2005b), the sample's age and diagenetic state (e.g., Blackwell *et al.*, 2005a, 2005b, 2007b; Skinner, 2007a; Skinner *et al.*, 2005a, 2005b).

4.2 Determining the Internal Dose Rate, $D_{\text{int}}(t)$

Measuring the internal dose rate, $D_{\text{int}}(t)$, requires measuring the radiation sources (all U, Th, K, etc.) within the sample (Figure 3b), usually using neutron activation analysis (NAA) or any geochemical technique that can assess elemental concentrations with precisions ≤ 20 ppb. Then, $D_{\text{int}}(t)$ is derived from theoretical calculations. For samples with significant U or Th, those calculations must include the increased radioactivity due to ingrowth of the U or Th daughter isotopes (Figure 7a) over time. $D_{\text{int}}(t)$ calculations also must consider the radiation attenuation by water within the sample, β and γ dose attenuation due to mineral density, and radon (Rn) or other daughter isotope losses for U- or Th-rich samples (Figure 7b; e.g., Brennan *et al.*, 2000).

In samples, such as tooth enamel, bone, and fish scales, where the internal dose rate derives solely from U absorbed during its burial history, the calculated ESR age must account for p , the U uptake parameter: Either the sample must be dated by coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ or coupled ESR-U/Pb analysis, which both allow a unique p to be calculated, or p must be assumed. In the latter case, four models are commonly used (Figures 7c, 7d):

Early uptake (EU), $p = -1$, assumes that the sample absorbed all its U soon after burial, providing the youngest age given the accumulated dose, $\mathcal{A}_\Sigma$, and external dose rate, $D_{\text{ext}}(t)$. This model only applies to the dentine in very young teeth and quartz sediment.

Linear uptake (LU), $p = 0$, assumes that the sample absorbs U at a constant rate throughout its burial history, giving a median age. Most dating applications have found that this model works well for molluscs, the dentine and enamel in many teeth younger than 300-500 ka.

Recent uptake (RU), $p > 0$, assumes U uptake very late in the sample's burial history, which reduces its internally generated dose rate, $D_{\text{int}}(t)$, to a minor contribution compared to $D_\Sigma(t)$. This gives the maximum possible age. Teeth older than 500 ka often have experienced RU uptake in both enamel and dentine.

Coupled uptake (CU) assumes that the enamel, dentine, cementum, and any attached bone associated with the tooth absorb U at different uptake rates. LU is sometimes used for the enamel and EU for the dentine, cementum, and any attached bone, yielding ages somewhat younger than strict LU, but older than strict EU, models.

Other models have also been suggested (e.g., Ikeya *et al.*, 1997). CU only applies to materials like teeth where two different phases absorb U at different rates. In teeth, LU or CU ages often agree most closely with ages determined by other means for samples between than 80 ka and 500 ka, but, within a site, the uptake model can vary, since it depends strongly on microenvironmental conditions (e.g., Blackwell *et al.*, 2001a, 2001b, 2002a). Although using a coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ calculation will constrain the U-uptake history in teeth (e.g., Eggins *et*

al., 2003), for teeth older than ~ 500 -600 ka, their $^{230}\text{Th}/^{234}\text{U}$ ratios become indistinguishable from the secular equilibrium value, making it impossible to assess p . U/Pb can date some uraniferous samples older than 1-2 Ma, but it has rarely been applied to calculate p . U leaching or secondary U uptake may also present problems for some samples, and hence, require more complex models (Figure 7c; Blackwell *et al.*, 2005b; Hoffman & Mangini, 2003). The precision for $D_{\text{int}}(t)$ depends strongly on the measurement precision for the U concentration. Any other technique that can measure the U at a detection limit ~ 1 -10 ppb with ~ 1 -10 ppb precision can provide sufficient discrimination to yield reliable ages. Delayed neutron counting (DNC) NAA can routinely provide good precision for $D_{\text{int}}(t)$, whereas instrumental NAA averages ~ 200 ppb for precision and ~ 1000 ppb for detection limits, which makes it inapplicable for measuring U in samples younger than ~ 200 ka. Coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ methods can usually detect ≤ 0.1 ppb. Hand-held X-ray fluorescence (XRF) machines have not reached the necessary precision and detection limits to make elemental concentrations for the $D_{\text{int}}(t)$ measurements.

4.3 Determining the External Dose Rate, $D_{\text{ext}}(t)$

The external dose rate, $D_{\text{ext}}(t)$, comprises the sedimentary dose rate, $D_{\text{sed}}(t)$, and the cosmic dose rate, $D_{\text{cos}}(t)$. $D_{\text{ext}}(t)$ strongly affects a sample's calculated ESR age (Figure 8a), especially for samples with low internal dose rates, $D_{\text{int}}(t)$. Particularly, in limestone or dolomite terrains, where $D_{\text{int}}(t)$ is often low, $D_{\text{ext}}(t)$ may be as much as 10-200 times higher than $D_{\text{int}}(t)$, making it critical to measure $D_{\text{ext}}(t)$ correctly to ensure accurate ages. Samples from open-air sites and other environments tend to have larger $D_{\text{int}}(t)$ values, but both $D_{\text{sed}}(t)$ and $D_{\text{cos}}(t)$ also can be more variable over the long term. Sites need to be examined carefully to understand all the dynamic processes that affect $D_{\text{ext}}(t)$.

Many geological and archaeological sites have inhomogeneous sediment (i.e., they are "lumpy"; Figure 8c), which generates inhomogeneous radiation dose fields. In particular, caves suffer this problem, where sedimentary inhomogeneity generally is the rule, rather than the exception. In sediment, α particles penetrate $\sim 20 \mu\text{m}$, β particles, ~ 2 -3 mm, and γ radiation, ~ 30 cm (Figures 8a, 8b). The sedimentary α dose is usually ignored by the simple expedient of removing the outer 20-30 μm from the sample before ESR analysis. The sediment immediately attached to, or surrounding, the dating sample is usually analyzed by a β detector or neutron activation analysis (NAA) to calculate the sedimentary β radiation dose rate. Due to the size of the γ dose sphere that surrounds the sample, the sediment contributing to the γ dose rate requires a bit more work to determine the time-, and volumetrically-, averaged $\bar{D}_{\text{sed},\gamma}(t)$ (e.g., Blackwell *et al.*, 2000a).

To derive the external dose rates, $D_{\text{ext},\gamma}(0)$, four methods can be used:

1. TL dosimeters emplaced at least 30 cm deep in the sediment measure the modern external dose rate, $D_{\text{ext}}(0)$, from sedimentary and cosmic sources for 0.25-2.0 years (e.g., Guibert *et al.* 2009).
2. A γ spectrometer sitting in a hole at least 30 cm deep measures the modern dose rate, $D_{\text{ext},\gamma}(0)$, from

sedimentary γ and cosmic sources over 0.5-2.0 hours. $D_{\text{ext}\beta}(0)$ must be measured separately, using a β detector or NAA, to analyze the sediment within 3 mm of the sample.

3. Bulk geochemical analysis, often by NAA, using powdered sediment collected along with the sample (Blackwell, 1989) is analyzed the U, Th, K, and other significant radioisotope concentrations in any layers which may have contributed to the sample's $D_{\text{sed}}(t)$ (Figure 8c). The radioisotope concentrations are used to mathematically calculate (e.g., Adamiec & Aitken, 1998) the current dose rates, $D_{\text{sed},\gamma}(0)$ and $D_{\text{sed},\beta}(0)$.

A time-averaged cosmic dose rate, $\overline{D}_{\text{cos}}(t)$, is also measured or calculated (Figure 9; e.g., Deely *et al.*, 2011) for samples buried < 20 m. In sites with thinly layered deposits or inhomogeneous sediment, $D_{\text{ext}}(t)$ calculations ideally should consider each unit or sediment component individually by determining volumetrically averaged dose contributions (Figure 8; Blackwell *et al.*, 2005a; Brennan *et al.*, 1997).

For all methods, the time-averaged sedimentary water concentration is used to correct for radiation attenuation by water in the sediment (Figure 8d). For adjacent U-rich or Th-rich layers or sediment components, the measurement or calculation is corrected for possible long-term U uptake, U daughter isotope ingrowth, and potential Rn or other isotopic losses (e.g., Blackwell & Blickstein, 2000; Blackwell *et al.*, 2001a, 2002a; Guibert *et al.*, 2009 see below). Changes in water depth and/or sedimentary cover thickness must also be modelled to estimate $\overline{D}_{\text{cos}}(t)$ (e.g., Deely *et al.*, 2011; see below).

Recent comparisons between different dosimetric methods have shown that external dose rates, $D_{\text{ext}}(t)$, measured from the three commonly used methods agree within errors, once they have been corrected for sedimentary diagenesis, changing sedimentary water concentrations, and changes in the water and sediment cover depth with time (e.g., Dibble *et al.*, 2013a, 2013b). Nonetheless, $D_{\text{ext}}(t)$ that have been assessed using two or three methods from among sediment geochemistry, *in situ* γ or TL dosimetry provide better modelling for the long-term changes in the sedimentary and cosmic dose rates. All three methods have drawbacks. Effective TL dosimetry requires that area within 3 m of the dosimeter insertion site be unaffected by further excavation or deflation for 6-12 months. Thus, TL dosimetry is usually done before sample collection and rarely does the dosimetry sample fall anywhere near or within the 30 cm γ dose sphere around the sample. While not problematic for samples in homogeneous, thickly bedded sediment, reasonable estimates for $D_{\text{ext}}(t)$ for samples from inhomogeneous or thinly bedded sediment then require modelling and extensive mathematical corrections for variability in the dose field. In caves where restricted access can prevent tampering or theft of TL dosimeters, any dosimetry method usually works well. In open-air sites, however, TL dosimeters rarely survive undisturbed for the needed time.

With sedimentary geochemistry, the external dose field can be mathematically modelled reasonably accurately. When using γ or TL dosimetry, this sediment must still be analyzed geochemically to provide the external γ dose rate. Several sediment samples may need to be analyzed to represent the γ dose sphere around the dating sample.

Assuming that $D_{\text{sed}}(t)$ remained constant with time, as many early studies did, proved naïve. Changing sedimentary water or radioactive element concentrations (Figure 8d; e.g., Olley *et al.*, 1997), increasing burial depth (Figure 8b), or reworking, among others, can all affect $D_{\text{sed}}(t)$ acquired by the sample. Hence, $D_{\text{sed},\gamma}(t_0)$ and occasionally $D_{\text{sed},\beta}(t_0)$, must be modelled to correct for such significant differences. At sites where sedimentary water concentration changed dramatically, or where sediment accumulation or deflation can alter the depth of sediment cover, these considerations cannot be ignored (e.g., Deely *et al.*, 2011; Skinner *et al.*, 2013).

In using geochemical analysis (e.g., NAA) at "lumpy" or thinly bedded sites, the inhomogeneity in the dose field (Guibert *et al.*, 1998) requires volumetric analysis to calculate an accurate $D_{\text{sed}}(t)$. The $D_{\text{sed},i}(t)$ contribution from each component or layer, i (Figure 8c), is weighted based on its contribution to the volume within the β and γ "dose spheres". That still, however, does not consider the potential changes in $D_{\text{sed}}(t)$ due changes in radioisotopic concentrations within the sedimentary components (e.g., Guibert *et al.*, 2009).

In lumpy sites, sedimentary components that may absorb U (e.g., peat, teeth, bones, mollusc shells) can constitute a significant sedimentary fraction. If they can absorb U, $D_{\text{sed}}(t)$ will probably have changed with time, because,

1. Components, like teeth and bone not only absorb U, but the daughters ingrow later (Figure 6a).
2. When the U uptake occurs early in the sediment's depositional history, its effect is greater than if the uptake was recent. This means that sedimentary U uptake must be modelled analogously to that into teeth (see $D_{\text{int}}(t)$ models above; Figure 7c).
3. Leaching can remove U or other soluble daughters, or diffusion can remove Rn (Figure 5b), from these components, requiring modelling to assess the effect on $D_{\text{ext}}(t)$ (e.g., Pike & Hedges, 2001; Figure 7c).
4. More than one discrete uptake or loss event may have affected these components (Figure 7c).

These sedimentary processes can produce significant differences in the calculated $D_{\text{sed}}(t)$ (or $D_{\text{ext}}(t)$) and ages (Blackwell & Blickstein, 2000). Isochron analyses can often help to identify these problems (see below).

Precision for the ESR dates depends on the method used to measure $D_{\text{sed}}(t)$ (or $D_{\text{ext}}(t)$) and how it compares to $D_{\text{int}}(t)$. For all three methods, precision tends to average 3-10%, but the more inhomogeneous or thinly bedded the site and/or the more complex its depositional history, the larger the error both in $D_{\text{sed}}(t)$ (or $D_{\text{ext}}(t)$) and the ages.

Where both $D_{\text{int}}(t)$ and $D_{\text{sed}}(t)$ are low, such as in marine carbonate systems, $D_{\text{cos}}(t)$ may present a significant source of dose for the samples. $D_{\text{cos}}(t)$ depends on the site's latitude and altitude (Figure 9a), but since $D_{\text{cos}}(t)$ also varies significantly with time as water depths change and sediment cover builds up or erodes away above the sample (Figure 9b), it becomes necessary to model time-averaged cosmic dose rates, $\bar{D}_{\text{cos}}(t)$. Precise water depths and sediment cover thicknesses must be estimated from current paleontological and geological constraints in order to build a model for the changing $D_{\text{cos}}(t)$ with time (e.g., Figure 9c, 9d). In testing how water

depth and sedimentary cover in San Salvador varied with time, Deely *et al.* (2011) showed that $\bar{D}_{\text{cos}}(t)$ were robust enough that the ages did not change significantly with small changes in their model assumptions, such as changing the water depth from 5 to 10 m for the sample's initial deposition, or changing the timing for the platform emergence by a few thousand years. If the $D_{\text{cos}}(t)$ model assumes plausible geological and paleontological constraints, its associated accuracy can be high and its uncertainty can be as low as 3-10%.

Reworking a fossil alters both its $D_{\text{sed}}(t)$ and $D_{\text{cos}}(t)$ (Blackwell, 1994; Blackwell *et al.*, 2012). In the new location, different sediment produces a different $D_{\text{sed}}(t)$, and a different burial depth means a new $D_{\text{cos}}(t)$. Calculating an ESR date requires estimating the original $D_{\text{sed}}(t)$ and $D_{\text{cos}}(t)$. In marine environments, where ESR is used to date molluscs, foraminifera, and corals, reworking is often the rule, especially on the coastal plain. How prevalently reworking occurs in the deep ocean remains uncertain. To determine if fossils have been reworked, 4-5 randomly chosen subsamples should be dated. For small molluscs, this means taking grab samples from a bag with many individuals, in the hopes that each subsample will grab a slightly different combination of several populations from different times. For larger fossils, it means dating 4-5 individual fossils. If their ages, A_{Σ} , or U concentrations differ significantly, the sample must be assumed to contain reworked fossils. Samples found in life position are most reliable, because they have not likely been reworked (e.g., Blackwell *et al.*, 2010a, 2012; Florentin *et al.*, 2014; Skinner *et al.*, 2013).

4.4 Uncertainties and Calibration

Normally contributing 1-5% uncertainty, the uncertainties in the A_{Σ} , $D_{\text{sed}}(t)$, and $D_{\text{cos}}(t)$ calculations introduce the largest sources of error (uncertainty) into the ESR ages. In teeth, uncertainties in A_{Σ} tend to average 1-3% for well preserved teeth, but poorly preserved teeth may have A_{Σ} uncertainties up to 5-8%. When a tooth contains high Fe or Mn concentrations, Fe or Mn signal interference will reduce both the accuracy and precision in its A_{Σ} determination. Uncertainty for $D_{\text{sed}}(t)$ calculations can be as low as 3-5%, but sites that contain inhomogeneous layers can have higher uncertainties that arise from the volumetric analyses. Where these have been comparable using different dosimetry analyses, the three using agree well within errors (e.g., Dibble *et al.*, 2013b). Uncertainties associated with time-integrated $D_{\text{sed}}(t)$ and $D_{\text{cos}}(t)$ depend on the reliability with which the geological history for deposition, deflation, inundation, emergence, uplift, and other sedimentary or tectonic processes that affect the sediment coverage thicknesses or water depths (see below for examples).

Contamination can cause other errors. If U-rich bone, dentine, or cementum were to contaminate the enamel, which contains much less U than the other tissues, the enamel's U concentration would be overestimated, causing its internal dose rate, $D_{\text{int}}(t)$, to be overestimated, and thus, the tooth's age to be underestimated. If the outer 20 μm of enamel affected by α dose is not totally removed, then $D_{\text{int}}(t)$ will be underestimated.

Age accuracy depends on the accuracy for the ESR spectrometer's calibration, the radiation source's calibration, and the sample's diagenetic state (Blackwell, 2001). In two past interlaboratory calibration tests

found, the radiation sources were intercalibrated and unknown samples were dated (e.g., Barabas *et al.*, 1993; Wieser *et al.*, 2005). Each laboratory using γ sources also calibrates its own radiation sources against their national standards and calibrated daily against an independent dosimeter that is also calibrated regularly. Calibration for β sources are also tested, but the protocols and frequency vary more among laboratories.

For detecting the U, Th, K, and other elemental concentrations, standard samples are regularly tested. For NAA, accuracy and precision are ensured by calibrating each run against a known standard, such as NIST 1633B or other standards, which are inserted and analyzed with each NAA run (e.g., Blackwell, 1989).

4.5 Isochron Analyses

Isochron analyses have been used mainly for teeth (Figure 10), but also for quartz from fault gouge and stalagmites. In theory, since a large tooth must have the same $D_{\text{ext}}(t)$, subsamples that have different U concentrations will have different accumulated doses, $A_{\text{ext},i}$, for each subsample i due to variations in their $D_{\text{int},i}(t)$ values (Figure 10a). In a large sample, ≥ 5 -8 subsamples are analyzed by the standard ESR protocol. If the accumulated doses, $A_{\text{ext},i}$, plotted against the time-averaged internal dose rate, $\overline{D}_{\text{int},i}(t)$, for each subsample, i , yield a straight line, its slope equals the sample's age, t_1 , while the y-intercept yields the accumulated dose due to external sources, A_{ext} . From A_{ext} , one can derive the time-averaged external dose rate, $\overline{D}_{\text{ext}}(t)$.

In teeth, the method yields a family of lines that converge on A_{ext} , but whose ages and $\overline{D}_{\text{ext}}(t)$ each depend upon the U uptake model used to calculate $\overline{D}_{\text{int},i}(t)$ (Figures 10b, 10c). If the isochron has a high regression coefficient, R^2 , the slope gives ages that agree with other dating methods (Blackwell *et al.*, 2002a). The isochron method only works with samples having ≥ 2 ppm U, and those with $D_{\text{int}}(t) \approx D_{\Sigma}(t)$. If samples have lost U or gained U in more than one event, however, the isochron analyses may yield erroneous ages and/or $\overline{D}_{\text{ext}}(t)$ values (Figure 10d; Blackwell *et al.*, 2001a, 2001b). Since isochron ages are almost always less precise than standard ESR analyses, their biggest value is not in yielding ages, but their ability to identify samples that have lost or gained U multiple times. For isochron analyses, precision for the ages and A_{ext} can range as low as 3-4%, while minimum uncertainties for $\overline{D}_{\text{ext}}(t)$ tend to be ~ 5 -6%.

If an independent method (e.g., TL or γ dosimetry) can be used to measure $D_{\text{ext}}(t)$, and if $D_{\text{ext}}(t)$ can be shown to have been constant throughout time at the site by geological studies or an independent date, the isochron method can instead determine the U uptake history. Since the isochron calculation gives A_{ext} , which must equal the product of the age, t_1 , with $\overline{D}_{\text{ext}}(t)$, the isochron's slope that matches this age represents the “correct” isochron and uptake model for the sample (e.g., Figure 10d).

4.6 ESR Microscopy and Other New Techniques

An ESR microscope is a spectrometer modified to scan across a solid mineral surface in order to measure the spin concentrations for a preset signal. Specialized analytical programs allow 2D, 3D, and 4D imaging, some of which are combined with other systems, such as electrically stimulated luminescence (ESL), NMR, and CT (e.g., Miki *et al.*, 1996; Omura & Ikeya, 1995; Mizuta *et al.*, 2002). ESR microscopy is still being fine-tuned, but it shows great potential for understanding fossil diagenesis, and mapping crystal growth and defects, among other applications. Currently, it works best for materials with very strong ESR signals, such as tooth enamel (e.g., Oka *et al.*, 1997), bone (Schauer *et al.*, 1996), coral, gypsum (e.g., Ulusoy, 2004), mollusc shells, aragonite, sulphates and barite (e.g., Whitehead *et al.*, 2002).

At radiation accidents, portable ESR dosimeters and spectrometers can assess radiation releases (e.g., Oka *et al.*, 1996). In the field, such technology could help to recognize reworked fossils, to aid in selecting the best dating samples. Portable XRF has almost reached the sensitivity to assess site inhomogeneity. Eventually, such technology may even allow preliminary age estimates while still in the field or for use in on site dosimetry for radiation accidents (e.g., Onori *et al.*, 2000).

Other advances in ESR spectroscopy itself are being developed, such as pulsed ESR (e.g., Kohno *et al.*, 1996; Grün *et al.*, 1997) and new techniques to examine spectra with existing machines (Grün, 2002). Several machines that use different bands, such L and Q bands, have been appeared recently, although their applications have been limited thus far in geology or archaeology. Most recently, several tabletop X band ESR systems have also come onto the market. While not as precise for dating young samples as the much more expensive large X band models, these new small machines when coupled with a small irradiator do provide the possibility of taking these machines into the field, if methods to purify the samples can also be developed for the field. Simply just being able to estimate ages in the field has advantages in developing more detailed, cost-effective sampling strategies.

5.0 Developing New ESR Chronometers

New applications are under development for determining the cosmic dose rate using a coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ method in corals as well as for dating benthic foraminifera, fluvial sand, and barnacles with X band machines.

5.1 Determining the Cosmic Paleodoses in Corals from the Florida Platform, USA

Because radiation is a transitory event, measuring past radiation dose rates can be extremely difficult. One of the major questions that plagues ESR and the other luminescent (trapped charge) dating methods is how the cosmic dose rate, $\overline{D}_{\text{cos}}(t)$, may have changed in the past. For areas where the amount of sedimentary or water cover over a sample may have changed dramatically, having an age calculation protocol that does not depend on knowing $\overline{D}_{\text{cos}}(t)$

would remove one source of uncertainty.

Although coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ dating has become routine for dating uraniferous teeth (see references in Blackwell, 2001, 2006; Falguères, 2003; Blackwell *et al.*, 2010b), it had never been used to date corals before (Blackwell *et al.*, 2007a). Because the $^{230}\text{Th}/^{234}\text{U}$ dates do not depend on $\bar{D}_{\text{cos}}(t)$, but the ESR ages do, by coupling the two methods, the age and $\bar{D}_{\text{cos}}(t)$ can be determined simultaneously. This is analogous to the application in teeth where the age and U uptake parameter, p , are determined simultaneously.

Florida sits on a very stable carbonate platform on the edge of North America. Corals grow in the fringing reefs that rim the platform edge along the Florida Keys (Figure 11a). Corals here grow or cease growing primarily in response to sealevel change. Some platform erosion does denude the outer platform when sealevel is low, and some deposition occurs when sealevel is high (Blackwell *et al.*, 2007a).

For stable platforms, like Florida, $\bar{D}_{\text{cos}}(t)$ as determined from the coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ analysis for corals can be used to estimate a water depth profile that can be compared to $\bar{D}_{\text{cos}}(t)$ values estimated from model sealevel curves. If the two $\bar{D}_{\text{cos}}(t)$ determinations agree well, this provides independent validation for the sealevel model that produced the good agreement. For six corals from the Florida platform (Figure 11b), their $\bar{D}_{\text{cos}}(t)$ values and ages from the coupled analyses agreed well with the sealevel curve from Waelbroeck *et al.* (2002). This study demonstrated the general applicability for the coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ dating method. More precise measurements for water depth and coral cover thickness would improve the precision in fully testing the sealevel curves. Where one large coral head can be sampled at several points, or a reef can be sampled over a transect, the precision for the predicted $\bar{D}_{\text{cos}}(t)$ would increase dramatically for testing sealevel curves. Theoretically, a similar analysis could be applied to molluscs, barnacles, or other carbonate fossils datable by both the ESR and $^{230}\text{Th}/^{234}\text{U}$ methods (Blackwell *et al.*, 2007a).

5.2 Dating Benthic Foraminifera from the Ceará Rise, Central Atlantic

Benthic foraminifera (forams) are ubiquitous protozoans in deep ocean environments. Having carbonate tests, these species are often well preserved in oceanic sediment (Corliss, 1985). In some cores, they may be the only datable material. While planktonic foraminifera have been dated by ESR in the past (e.g., Hoffman *et al.*, 2001), benthic foraminifera have only been attempted recently (Blackwell *et al.*, 2010c).

In the equatorial Atlantic Ocean at 3-8°N 38-48°W, the Ceará Rise sits in water depths of 2800-4700 m between the 4° and 8° fracture zones, roughly as far from the Mid-Atlantic Ridge as its sister ridge, the Sierra Leone Rise. Basaltic basement underlies the central Ceará Rise at 4.56-5.5 km depth. Primarily during Pleistocene lowstands, turbidity currents carried biogenic and terrigenous clay-rich sediment from the Amazon River onto the Ceará Rise. During the interglacial highstands, as is true now, hemipelagic sediment with abundant benthic foraminifera dominated the sedimentation (McGreary & Damuth, 1975; Kumar & Embley, 1977). Wood's Hole Oceanographic Institute's (WHOI) piston core, EW9209 1JPC, was drilled on northeastern

Ceará Rise flank at 5°N and 43°W in 4056 m of water depth, in an area where deep waters from the Southern Atlantic dominate, but the North Atlantic Deep Water contributes little flow. On average, the sedimentation rate in 1JPC exceeds 4 cm/ky, but in glacial intervals, reached 5 cm/ky when input from the Amazon Fan increased the noncarbonate fraction. Core EW9209 was sampled at 1 to 3 cm spacing. Thus, each sample represents ~ 300 years of accumulation with a median sample spacing of ~ 600 years (Curry & Oppo, 1997; Blackwell *et al.*, 2010c).

Due to the water depth, $D_{\text{cos}}(t) = 0.0$ mGy/a, but $D_{\text{sed}}(t)$ must be done using sedimentary geochemistry (Table 2). Due to the small numbers of benthic foraminifera recovered from the sediment, the ramping method was used measure an accurate $\mathcal{A}_E$ (Blackwell *et al.*, 2010c).

At depths 479-489, BF1 yielded an EU age of 120 ± 38 ka (with seven points), while the sample from depths 561-570 cm, BF2, yielded an EU age of 175 ± 19 ka (11 points). Comparing the ESR ages with the known ages for those layers shows that while these samples still need more ramping doses (i.e., reirradiation and reanalyses) to improve their precision (Figure 12), their age accuracy suggests that ESR method can indeed be applied to date benthic foraminifera, with the same precision as planktonic foraminifera (Hoffman *et al.*, 2001; Blackwell *et al.*, 2010c).

5.3 Dating Quartz Sand, Bytham River System, England

Many fluvial deposits lack concentrations of mollusc or other shells datable by ESR. Thus, very few methods exist that can date fluvially deposited sediment, particularly if that sediment is > 500 ka, the limit for both TL and OSL dating. Because quartz resists erosion, and is easily transported, accurately dating its deposition time at a new location requires that any preexisting ESR signals from earlier depositional events must have been zeroed out. In quartz, the ESR signals can be reduced by sunlight. If the quartz grains did not see enough light when being transported by the river, they would retain a signal from their previous history. This studied dated fluvially deposited quartz grains associated with England's Cromerian Forest Beds.

In England during the Early Pleistocene from 2.6-0.78 Ma (MIS 103-19), the Bytham Rivers drained central Britain, with a catchment area extending from Wales, across East Anglia, into the North Sea (Figure 13). By the early Middle Pleistocene, flowing eastwards from the Midlands to the present North Sea, the Bytham River had extended its catchment area to become one of the largest rivers in Britain, draining the area where the Cromerian Forest Beds were being deposited. Early hominid archaeological sites have recently been found in the Cromerian Forest Beds, dating from ~ 500-700 ka (Rose, 2009). Parfitt *et al.* (2010) reported even older archaeological evidence for hominin occupations in paleomagnetically reversed sediment that must date from the later part of the Matuyama Chron, ~ 0.99-0.78 Ma. The Bytham and Thames River valleys apparently acted as major routes along which hominins moved across Britain. In the Anglian Stage ~ 450 ka (MIS 12), ice moved south to cover the British Isles, filling in the older paleochannels and displacing the ancient drainages south toward the modern Thames channel (Rose, 2009). When OSL and radiometric dating methods failed to give

conclusive results, quartz grains were tested with ESR to see if they could date these middle Pleistocene sites from the former coastal plain associated with the Cromerian Forest Beds more precisely. Samples from four drill cores collected from river sediment in a small tributary to the Bytham Paleochannel were dated (Skinner *et al.*, 2012).

Quartz contains several paramagnetic centers, such as the Ge ($g = 1.9936\text{--}1.997$), Al ($g = 1.9922\text{--}2.0026$), Ti ($g = 1.91\text{--}1.98$), E' ($g = 2.001$), and OHC ($g = 2.0008\text{--}2.0023$) signals. Because pressure during earthquakes easily zeroes the OHC, Ge, Al, and E' signals, they can date fault gouge, and thus, fault movements (see references in Blackwell, 2006). Since heating will completely zero the E' , Al, and Ti signals, but γ irradiation will regenerate them, these signals can date volcanic rocks and ash (e.g., Toyoda & Ikeya, 1991), burnt flint and chert (see references in Blackwell, 1995). The Ti-Li peak has been used to estimate ages for aeolian samples and marine terraces (Yoshida, 1996; Tanaka *et al.*, 1997). Where light acts as the zeroing agent, however, the Al and Ti signals are the two main signals used for dating quartz grains (Tissoux *et al.*, 2008; Skinner *et al.*, 2012).

The complex Al center is an impurity center that comprises two signals, one bleachable and the other unbleachable (Toyoda *et al.*, 2000; Voinchet *et al.*, 2003), but both are stable at ambient temperatures (Tissoux *et al.*, 2007). Toyoda *et al.* (2000) also identified three Ti centers, Ti-H, Ti-Li, and Ti-Na. Because each quartz signal bleaches and anneals at different rates, they can theoretically monitor bleaching during sediment transport: Agreement between the accumulated doses from the Ti-Li, Ti-H, and/or Ti-Na peaks would signal that complete bleaching had likely occurred. Insufficient bleaching leaves the accumulated dose for the Ti-Li peaks exceeding those for the Ti-H and/or Ti-Na signals (Toyota *et al.*, 2000; Yoshida, 1996; Rink *et al.*, 2007). To assess their bleaching rate, pure dry quartz sediment from the Bytham River Palaeochannel was exposed to sunlight in clear glass tubes. After ~ 350 hours, the sunlight exposure had reduced the Al signal by $\sim 14\text{--}64\%$, and the Ti signals, by $43\text{--}89\%$ (Table 3). In fluvial systems, the grains would likely require much longer to bleach fully, due to turbidity and reduced water clarity (Skinner *et al.*, 2012).

To prepare the quartz for ESR analyses, all the minerals other than quartz were removed by hand picking any contaminant minerals and treating with hydrofluorosilicic acid. The latter treatment also removed the outer $20\text{ }\mu\text{m}$ from the grains to remove the need for an α attenuation calculation. Associated sediment analyses showed that both the bulk marine sediment and the purified quartz contained a little U, Th, and K producing a low $D_{\text{sed}}(t)$ (Table 4). In some samples, the difficulty in measuring the Ti-H(1) and the Ti-Li(1) peaks made it impossible to determine A_{Σ} values (Table 5). For some other peaks, associated errors are high for the same reason. Because the regional history is not well known yet, the ages were calculated with both a modelled $\bar{D}_{\text{cos}}(t)$ and $\bar{D}_{\text{cos}}(t) = 0$. The latter value sets the maximum possible age for the site, while the former value gave a median estimate for the actual age. The ages (Table 5) should be considered as preliminary, because as the core histories are understood better, the ages might change somewhat. Across the cores, the ages vary considerably, and in no case do all the ages from the five peaks agree (Skinner *et al.*, 2012).

The ESR ages (Table 5) are larger than those expected from the site geology, $\sim 0.78\text{--}0.87$ Ma (Parfitt *et*

al., 2010), even with the time-averaged $D_{\cos}(t)$. Only the Ti-H(1) signal for CQ12 gave an age that matched the geology. Since Ti-H(1) is one of the fastest bleaching signals, this suggests that the quartz grains were completely bleached, at least for Ti-H(1) in CQ12, during transport. That other signals were not so completely bleached sheds light about the sediment recycling in the Bytham River System. The last time that all the grains were totally bleached was $> 4.9\text{--}6.8$ Ma. These ages must be considered a minimum possible age, because some peaks have been partially bleached from these samples, indicating that all peaks should have been bleached partially. Seven ages range from 1.6–2.5 Ma, suggesting that most grains were also strongly, but not totally, bleached between 1.6 and 2.5 Ma during the early Pleistocene, and hinting that much of the sediment in the Bytham system has been recycled since the early Quaternary. All this hints that most of the Bytham River must have been too turbid for effective sediment bleaching at 600–900 ka (Skinner *et al.*, 2012).

Changing the sedimentary water concentration, W_{sed} , did not significantly affect the calculated ages, nor did changing $D_{\cos}(t)$. This indicates that the errors in the ages are so large that errors in W_{sed} and $D_{\cos}(t)$ will not add negligible discrepancies to the calculated ages. When ESR dating on quartz becomes refined to give ages with lower errors, the choice of the time-averaged W_{sed} and $D_{\cos}(t)$ may then become significant (Skinner *et al.*, 2012).

This study on the Bytham River System showed that this dating method can give geologically reasonable ages for fluvial sediment. If the ages from all five peaks agree bleaching has been complete. The lack of agreement between these peaks here suggests that these quartz samples have not been bleached completely during their most recent fluvial transport. A bleaching event at $\sim 5\text{--}7$ Ma did completely zero all the quartz signals, suggesting that sediment in the Bytham River System has been recycled since the Miocene. Other partial bleaching events may have occurred at $\sim 1.2\text{--}1.6$ Ma, and ~ 360 ka (Skinner *et al.*, 2012).

By using an ESR microscope to prescan and separate the grains into different populations, the method may have better success at finding well bleached grains. Given the problem of mixed quartz grain populations, the final solution likely lies in doing single grain dating, which is under development (e.g., Beerten *et al.*, 2003). These methods would allow researchers to pick out grains with differing degrees of zeroing for further examination.

5.4 Dating Barnacles from the Khyex River, British Columbia

In rocky coastal settings, barnacles plastered on the cliffs may prove the only indicators about past sealevel positions. Dating these positions would enable one to build sealevel curves and understand paleoenvironmental conditions more completely. For barnacles older than 40 ka, however, no good dating method has been available. This study illustrates a method for dating barnacles with ESR.

Located at $54^{\circ} 14' \text{ N}$ and $129^{\circ} 48' \text{ W}$ in northwestern British Columbia, Khyex River is 35 km east of Prince Rupert (Figure 14). During the Late Quaternary, this area experienced rapid sealevel changes due to tectonic uplift, crustal subsidence and warping, in addition to glacially driven sealevel changes. In November,

2003, a landslide displaced $4.7 \times 10^6 \text{ m}^3$ of glaciomarine silt and clay (Figure 15). In the displaced sediment, a giant *Balanus nubilus* was recovered from the glaciomarine clays, while several *Balanus crenatus* were found in growth position attached to the recently exposed bedrock valley wall, and hence, not reworked. In the overlying layer, *in situ* barnacles, bivalves, and gastropods were discovered. Their presence suggested a cold, turbid, brackish, intertidal environment. This paleoshoreline sits $\sim 30 \text{ m}$ above the current sealevel. In the massive sand above the fossil layer, woody debris was ^{14}C dated to $10.29 \pm 0.03 \text{ ky BP}$ (uncalibrated), while the barnacles themselves were dated to $11.57\text{--}11.80 \pm 0.03 \text{ ky BP}$ (uncalibrated). Lumpy, boulder-rich colluvium overlies the massive sand (Blais-Stevens *et al.*, 2006; Blackwell *et al.*, 2010a).

B. nubilus and *B. crenatus* were both analyzed by ESR using the peak at $g = 2.0053$ (Blackwell *et al.*, 2010a), which was absent in modern barnacles. Although marine calcitic fossils can absorb U during diagenesis, the barnacles from Khyex River had almost no U (Table 6). Hence, the ESR ages did not depend on the U uptake rates, and potential Rn loss could not be a source of uncertainty. The three subsamples from the giant *Balanus nubilus* yielded a weighted mean age of $15.1 \pm 1.2 \text{ ka}$, while several *Balanus crenatus* dated at $15.0 \pm 2.1 \text{ ka}$. Since their $\mathcal{A}_2$ and ages did not differ at the 95% confidence limit (2σ), the ESR signal in the two *Balanus* species does not appear to depend on the species. Their mean age, $15.0 \pm 1.0 \text{ ka}$, for the sealevel stand exposed at Khyex River, agreed well with the uncalibrated ^{14}C dates for the barnacles and the overlying wood (Figure 15), and estimates for the glacial ice removal from the region (Dyke & Prest, 1987; Dyke, 2004; Blackwell *et al.*, 2010a). The difference between the uncalibrated ^{14}C and ESR ages suggests that the calibration in this area should be $\sim 3.3\text{--}3.4 \pm 1.3\text{--}2.3 \text{ ky}$.

Some *Balanus* shells, however, contain significant Mn contamination that precludes their dating, as occurred at Norridgewock, Maine. Moreover, while barnacle shell hashes may prove datable, multiple random subsamples will need to produce the same ages to ensure that reworking has not mixed two noncontemporaneous barnacle sources, just as is tested for other shell hashes now when using molluscs. More fossil *Balanus* from other sites with different known ages should be ESR dated to demonstrate the method's accuracy across a broader time range. Since these are the first *Balanus* dates and the signal lifetime, τ , for $g = 2.0053$ is unknown, a stability test is needed to determine τ , which would also indicate the potential dating range. If the signals in the barnacles have a similar thermal stability to those in molluscs, however, their dating range could approach 2.5 Ma (Blackwell *et al.*, 2010a).

6.0 Field Applications to Solve Geological Problems with ESR Dating

The more established ESR chronometers, such as shells and teeth, are now being routinely applied to date volcanic eruptions, tectonic uplift, sealevel change, water availability, paleoclimatic change, and of course, archaeological and paleontological sites. These results answer geological questions ranging from determining tectonic uplift rates and building sealevel curves to determining paleoenvironmental conditions and tracking hominin and cultural migration patterns.

6.1 Dating the Kos-Nisyros Volcanic Complex, Greece

Most magma feeding volcanoes contains plenty of K to allow $^{39}\text{Ar}/^{40}\text{Ar}$ dating, but for those that lack K, dating them requires more innovative approaches. Many marine molluscs, especially those that live as infauna and filter their food from the water, cannot survive in water clogged with ash, while other marine organisms, like algae that deposit carbonate shells, cannot tolerate the low sunlight that accompanies large volcanic eruptions. Ergo, ESR can date the molluscan shells and algal carbonates, and thus, date the quiescent periods when eruptions must have been absent or very small.

The Kos-Nisyros volcanic field comprises the islands Kos, Pyrgoussa, Pahia, Yali, and Nisyros. Located in the Hellenic Arc, Nisyros is a truncated volcano 8 km in diameter with a central caldera ~ 4 km wide, that reaches ~ 100 m above sealevel. Several eruptions throughout Nisyros' history have deposited pumice found on nearby islands Yali and Pyrgoussa (Sachpazi *et al.*, 2002; Sykioti & Briole, 2003; Tibaldi *et al.*, 2008). With extremely low K concentration in the magma, applying $^{39}\text{Ar}/^{40}\text{Ar}$ to date the eruptions has been largely futile.

In Nisyros' first extrusive eruption at ~ 161 ka, a submarine vent between Kos and Nisyros erupted > 100 km³ pumice and ash that fell across > 3000 km². This deposit, the Kos Plateau Tuff (KPT) reaches > 20-30 m thick on Kos, Nisyros, Yali, and Pyrgoussa. Pyroclastic lahars and tephra deposited calcalkaline pumice, tuff, and lava flows. By ~ 150 ka, frequent eruptions had formed a large stratovolcano, whose summit later eruptions destroyed to form a large flooded caldera between Yali and Nisyros. From ~ 60-45 ka, several massive eruptions deposited the Emporios Lava, the Lower Caldera Pumice, and Upper Caldera Pumice on Nisyros, and formed the current caldera. Since the Nikia Lavas were deposited in the caldera at ~ 32 ka, hydrothermal eruptions have continued sporadically building several domes within the caldera. A magmatic intrusion ~ 5 km deep flowing from SE toward the central dome probably produced the intense seismic unrest in 1995-1998 (Fytikas & Vougioukalakis, 2005; Francalanci *et al.*, 2007; Tibaldi *et al.*, 2008).

Yali, the youngest volcanic centre in the Kos-Nisyros field, sits 5 km NW of Nisyros. Late Quaternary eruptions formed Yali's core. Today, two hills joined by a low isthmus make Yali, an island 5 km long, 1.5 km wide, and up to 180 m high. Between ~ 145 and ~ 55 ka, the Yali Lower Pumice (YLP) erupted from a shallow submarine vent, depositing up to 160 m of white rhyolitic pumice. Its KPT pumice xenoliths show that the YLP must postdate 161 ka. Overlying the YLP and now sitting up to 120 m amsl, Yali's marine terrace (YMT) averages 0.5-1.0 m thick (Figure 16). This shallow marine sandy-silty limestone contains some pumice, plus bivalves, molluscs, coralline algae, and numerous worm burrows deposited during a fairly quiescent period. The YMT lacks the *Strombus bubonus* typically found in Tyrrhenian beds (MIS 5). Also formed during a quiescent period, Yali's Oldest Paleosol (YOP), a reddish-brown terra rosa 40-50 cm thick grew above the YMT. The paleosol has shells derived from the underlying YMT. At ~ 32 ka, a shallow submarine vent between Yali and Kos erupted the grey dacitic Yali Upper Pumice (YUP). At ~ 24 ka, obsidian and perlite domes and flows formed. The overlying paleosol, Yali's Neolithic Paleosol (YNP), developed during a quiescent period, and contains Neolithic pottery sherds and obsidian tools. Above that, eolian dunes host two more thin paleosols and

are topped by the modern paleosol. Since ~ 140 ka, uplift due on E-W and NW-SE trending normal faults has raised all the units above sealevel, but lowered their surfaces by > 160 -350 m (Francalanci *et al.*, 2007; Stiros & Vougioukalakis, 1996; Blackwell *et al.*, 2011).

To the west of Nisyros lies Pyrgoussa. Its basal lava flows dating to ~ 2 Ma resemble those on Kos. They underlie the 161 ka KPT ash and pumice. Above that sits the PMT, a marine terrace ~ 2 m thick. The preservation of molluscs, coralline algae, and tube worms in the PMT without silica hints that Pyrgoussa may have had more clear water and less volcanic activity than the other islands. Later, porphyritic calcalkaline lava partially covered the marine terrace. On the NW shore, the grey Kyra Tephra reaches 1 m in thickness. Likely due to the prevailing winds or later erosion, Pyrgoussa lacks all the later pumice beds that cover Yali and Nisyros, except the YUP (Francalanci *et al.*, 2007). House foundations, terraces, pottery, and obsidian tools recall the Neolithic-Roman occupations (Blackwell *et al.*, 2011).

To date the tectonic activity associated with Nisyros, nine mollusc samples from Yali's oldest paleosol, 11 from Pyrgoussa's, and one from Yali's, marine terrace were dated. To determine $D_{\text{sed}}(t)$, 12 sediment samples were analyzed by NAA. Then, time-averaged sedimentary, $\bar{D}_{\text{sed}}(t)$, and cosmic, $\bar{D}_{\text{cos}}(t)$, dose rates were calculated. The amount of ash and other volcanic contaminants in the sediment resulted in fairly high variation between individual analyses, which translated into large associated errors on the $\bar{D}_{\text{sed}}(t)$, and thus, on the ages (Table 7). The molluscs all had U concentrations < 1.25 ppm (Blackwell *et al.*, 2011).

All 12 samples from Pyrgoussa were collected from the marine terrace (PMT) at ~ 100 m amsl on the NW coast. With the wide spread of A_{Σ} 's (Table 7c), the ages ranged from 45 ± 14 ka to 121 ± 31 ka. Thus, the terrace apparently grew during MIS 5e, 5c, 5a, 4, and mid MIS 3. Since several species preserved in the terrace would not have survived any significant ash deposition, and the samples contained no quartz, probably little volcanic ash reached Pyrgoussa at these times. Thus, the dates suggest that Nisyros was quiescent in MIS 5e through mid MIS 3. Because all the species would have preferred water depths > 10 m, the dates suggest that this terrace did not begin to uplift until after ~ 45 ka. Yali's single marine terrace sample dated at 52 ± 15 ka is also in mid MIS 3, which agrees well with the age for the last period for the PMT formation, suggesting that these likely at least partially coeval (Blackwell *et al.*, 2011).

Nine YOP samples were collected from the SW quarry wall at ~ 100 m amsl. Again, the wide spread of A_{Σ} 's, hints at several periods of deposition. Since the YOP developed on the YMT, the ages, which cluster at 50 ka, 39-42 ka, 18-24 ka, and 10 ka, suggests that the YMT probably developed mid MIS 3 until mid MIS 2. Alternatively, the YMT may have grown until ~ 39 ka or even 18 ka, before the YOP began to develop, whereupon it incorporated molluscs from the YMT into its base. The youngest mollusc, FM54, may have been reworked from the Neolithic paleosol, YNP, above. Dating the YNP might solve the question about FM54's original location. The lack of thick ash units in the YOP suggest that Nisyros-Kos was relatively quiescent until sometime after ~ 18 ka, when the terrace had been uplifted above sealevel. More dates from the YMT would help to clarify when its fossils were growing. Dating the algal fronds from the PMT & YMT remains to be

completed (Blackwell *et al.*, 2011).

While these dates have fairly large uncertainties, they do provide a better glimpse into the late Pleistocene history of the Nisyros-Kos complex than $^{39}\text{Ar}/^{40}\text{Ar}$ dating could give for this low K volcanic complex.

6.2 Dating Marine Terraces and Tectonic Uplift in the Antakya Graben, Hatay Province, Turkey

In temperate to tropical waters around the world, marine terraces exist that can provide clues about coastal uplift and sealevel changes. For more than 30 years, ESR has been used to date molluscs and corals from limestone terraces to determine tectonic uplift rates (see references in Blackwell, 1995, 2006). In this example, however, molluscs from clastic marine terraces were dated to examine the tectonic uplift within the local graben.

During the late Middle Miocene, the Arabian Plate collided with the Anatolian Plate (Bozkurt, 2001). Initially, the Arabian Plate moved northward along the sinistral Dead Sea Fault (DSF) at 17.8 ± 1.1 mm/y (Reilinger *et al.*, 2006), creating a compressional tectonic regime in the Anatolian Plate, but post-collisional compression caused the Anatolian Plate to thicken and then to slide westward along the dextral North Anatolian (NAF) and sinistral Eastern Anatolian (EAF) faults during the Pliocene, aided by back-arc extension in Aegean Plate (Bozkurt, 2001; Flerit *et al.*, 2004; Mantovani *et al.*, 2006; Aktug *et al.*, 2012). During this westward movement, the Anatolian Plate broke into several continental segments with different tectonic characters (İşseven & Tüysüz, 2006) and several small grabens developed, into which Pliocene and Quaternary sediment was deposited (Şengör *et al.*, 2004). The study area occurs in the fault-bounded Antakya Graben (Figure 17), one such area between Samandağ on the Mediterranean coast and Antakya (formerly Antioch on the Orontes River) in the east (Tari *et al.*, 2013). Sinistral fault movements on the DSF initially controlled the graben's development, but later, normal faulting dominated. Extensional movements, which still occur in the region, controlled the Quaternary tectonic evolution of the Antakya Graben. Thanks to strain still accumulating along the DSF and at the Karliova Triple Junction, large earthquakes still plague the area (Akyüz *et al.*, 2006; Karabacak *et al.*, 2010; Ahmet *et al.*, 2012; Karabacak & Altunel, 2012; Mahmoud *et al.*, 2012). Along with the tectonic movements, sealevel changes have strongly shaped the Quaternary geomorphological changes in the Antakya Graben.

During the Quaternary since 2.5 Ma, climatic changes produced by orbital forcing (Milanković cyclicity) have caused glaciers in the Northern Hemisphere to wax and wane. This altered the oceans' volume, and caused sealevel to seesaw > 50 times, by up to 100-120 m every few thousand years, exposing or submerging coastal areas (McCulloch & Esat, 2000). As Quaternary glaciers in the interior high plateaux, especially in the Tauride Mountains to the north, melted, floods eroded much sediment from the area, providing clastic material that could be reworked by coastal processes into beaches and marine terraces along the Mediterranean coast (Figure 18). Coupled with the sealevel change, the substantial vertical tectonic uplift on the faults marking the southern edges of the Antakya Graben has stranded and stacked several Quaternary marine terraces high above modern sealevel on the Hatay seacoast, both north and south of the Asi River Delta. Near Mağaracık, the marine terraces extend

along section for 3-5 km. Dating these terraces can help to understand the local geomorphic development, to identify faults, and to constrain the timing for local tectonic movements (e.g., Bekaroğlu, 2012; Doğan *et al.*, 2012; Florentin *et al.*, 2014).

Thus, molluscs and associated sediment for ESR dating were collected from 11 locations near Mağaracık and Samandağ along the northern side of the Antakya Graben (Figure 17, 18). After analyzing 44 sediment samples by NAA, $\overline{D}_{\text{sed}}(t)$ and $\overline{D}_{\text{cos}}(t)$ were calculated for each shell-bearing horizon. Differences in the U concentrations indicated that many shells were reworked from older deposits (Florentin *et al.*, 2014).

At six localities in the 30 m terrace near Mağaracık, the 19 ESR ages (Table 8a-f) also suggest that this terrace accumulated shells from older outcrops in the area, with the youngest ages being 56 ± 6 ka, but the oldest 1.1 Ma. Shells from MIS 3, 4, 5, 6, and 31 were all found buried in that terrace. At the base of the section at Mağaracık IV, *Lithophagus* burrows still containing shells *in situ*, such as FM65a, cross the unconformity between the Quaternary marine beds and the underlying Pliocene Samandağ Fm. clay, indicating that the deposit had to have been emplaced for them to burrow across the boundary. Thus, an age of 63 ± 6 ka for that *Lithophagus in situ* (Table 8c) sets the minimum possible age for the terrace's deposition. From Mağaracık Streamside, AM15H had the youngest age at 56 ± 6 ka (Table 8d), which sets the maximum possible age for this terrace's deposition. Statistically, these ages also agree with the youngest ages for shells at Mağaracık II and Mağaracık III. Therefore, the 30 m terrace was most likely deposited at 60.5 ± 2.6 ka, which correlates with the MIS 3/4 boundary. This suggests that this block is being uplifted at 0.54 ± 0.13 m/ky.

On the 40-50 m terrace, three samples were dated from two locations (Table 8g-h). With only two samples that agreed both at 116 ka (MIS 5e) plus one at 58 ka (MIS 3/4), the high likelihood of dating older reworked samples make it impossible to ensure that either date actually constrain the age of the terrace.

On the 60-70 m terrace, one sample dated from Mağaracık I (Table 8i). AM06a dated *Ostrea* (oysters) that grew on a limestone block found in a trough crossbedded sandy unit, likely on the shoreface associated with a tidal channel, but the block had likely been reworked from a higher terrace. AM06a dated at 82 ± 8 ka (MIS 5a).

At Mağaracık Dump (Figures 17, 18), ~ 103 m amsl, *Ostrea* (FM60, AM49) and other shells were found cemented in growth position on the Miocene Sebenoba Fm. limestone boulders outcropping there. The shells dated in this study all sat ~ 1-2 m above a wave-cut notch. FM15 dated at 84 ± 14 ka, while FM60 dated at 91 ± 13 ka (Table 8j), which both correlates with MIS 5a-5b. AM49, however, dated to 256 ± 44 ka, which correlates with MIS 8. Likely, the boulder rolled to its current location from a higher terrace where AM49 was originally deposited. If two young ages accurately reflects the age for this outcrop and its associated wave-cut notch just below the boulder, the fault block on which the Mağaracık Dump sits has been uplifted at 1.18 ± 0.12 m/ky, since MIS 5d. Since this fault block appears to be uplifting twice as fast as the fault block on which the 30 m Mağaracık terrace locations sit, a fault probably cuts between the Mağaracık Dump and the other Mağaracık locations (Florentin *et al.*, 2014).

At Samandağ Kurtstream (Figures 17, 18), several molluscs were deposited in a large tidal channel. Although the shells were broken, they were not rounded or abraded, suggesting that they had likely moved only a short distance, and may date the actual channel deposit. FM68A-D all gave consistent ages, averaging 116 ± 5 ka, and thus probably dating to late MIS 5e. These dates also suggest that the Kurtstream fault block has uplifted at 0.33 ± 0.02 m/ky since the MIS 5e boundary. Therefore, a fault also must exist between the Mağaracık Dump and Samandağ Kurtstream locations. The ages also suggest that another fault likely lies between the other Mağaracık locations on 30 m terrace locations and the Samandağ Kurtstream site (Florentin *et al.*, 2014).

In summary, uplift in the Antakya Graben has continued to uplift the north side of the Antakya Graben at rates of 0.33-1.18 m/ky. At least two faults likely exist between Mağaracık Dump and Samandağ Kurtstream that have produced uplift, but have not yet been mapped in outcrop or seismic surveys (Florentin *et al.*, 2014). Thus, ESR can provide uplift rates for both clastically and carbonate- dominated marine terraces. That data can also indicate where faults may exist that have foiled earlier mapping projects.

6.3 Dating Sealevel Change on San Salvador Island, The Bahamas

In developing sealevel curves, the lack of dates for terrestrial sequences often hamper efforts to understand the complexity of the sealevel changes with time. ESR has been used for more than 30 years to date the marine units associated with sealevel changes (see references in Blackwell, 1995, 2006), but few studies have used ESR to date the terrestrial sequences.

Located at 24°00'N 74°30'W, San Salvador is an isolated carbonate platform in the Bahamas archipelago (Figure 19). On San Salvador, glacioeustasy creates transgressive and regressive phases as sealevel fluctuates, creating sedimentary cyclothems. During the lowstand between the transgressive and regressive phases, watertables are very low, allowing terra rosa paleosols and caves form on the exposed platform and buried units to become cemented. During the transgressive phase as the platform floods, high wave activity builds beaches, which form the main source for the early transgressive eolianites. Meanwhile, reefs fringe the platform forming lagoons where new oolites begin to form and waves attack the transgressive eolianites. During the highstand, the coral reefs reach sealevel. Oolite production reaches its maximum. During the regressive phase as the sealevel falls, waves and wind mobilize the accumulated sediment from the lagoons to form regressive eolianites. At the lowstand, the next cyclothem begins (Mylroie, 2007).

In the Quaternary units exposed on San Salvador, the Rice Bay Formation lacks a terra rosa paleosol overlying it. In places, the Rice Bay Fm. sits atop an older terra rosa paleosol, which separates it from the Pleistocene Grotto Beach Formation. Only formed after the sealevels drop off the platform in the regressive phase, terra rosa paleosols mainly postdate the regressive phase dunes. The Late Pleistocene Grotto Beach Fm. shows transgressive, stillstand, and regressive phases: The French Bay Member includes the

transgressive phase, while the Cockburn Town Member contains the highstand and regressive phases. Underneath the Grotto Beach sits the Owl's Hole Formation, which contains only marine eolianites. Likely, the terrestrial facies were either eroded away, or have been covered by the Grotto Beach Formation (Mylroie & Carew, 2008).

Deely *et al.* (2011) completed dates for several corals (Table 9) and molluscs (Table 10) from the stillstand phases in the reef and lagoonal sediment. They also reported the first molluscan ESR ages for regressive dunes. To calculate $\overline{D}_{\cos}(t)$ for each sample, they used sedimentological, geomorphological, and paleontological indicators to estimate past sediment cover thicknesses and water depths in order to build ramped box models for both the cosmic dose rates (e.g., Figure 9c) and the sedimentary dose rates. For the corals, such as RC42 (Figure 20), any geologically reasonable combination of water depth and sediment cover thickness still produced an age that correlated with MIS 5e. When cosmically corrected, seven analyses for corals found *in situ* in the Cockburn Town Reef yielded ages ranging from 127 ± 6 to 130 ± 5 ka (MIS 5e; Table 9c), which all agree well with $^{230}\text{Th}/^{234}\text{U}$ ages for the reef (Mylroie, 2007). Three loose corals from local beaches all correlated with MIS 1 (Table 9a), while three corals from the rubble zone associated with the Cockburn Town Reef all gave older ages (Table 9b), suggested possible reworking from older units.

Today, the mollusc *Codakia* today lives at 20-50 cm under the sediment surface in water 1-5 m deep in lagoonal settings. For *Codakia* samples from 1 m above modern sealevel at Moon Rock Pond, their ages ranged from 82 ± 10 ka to 114 ± 11 ka, and correlated with MIS 5a, 5c, and 5e (Table 10c), after assuming the most geologically likely scenarios for their deposition. These data agree with Kindler *et al.* (2010), who found evidence for an MIS 5a highstand near modern sealevel on Eleuthera, and Shackleton (2000), who reported highstands at or above modern sealevel for MIS 5a and 5c. Marine molluscs from the Cockburn Town Reef also gave ages that correlated with MIS 5a (Table 10b), suggesting that sealevel was close enough to modern levels to permit molluscs to live in the reef (Deely *et al.*, 2011).

For the terrestrial mollusc *Cerion* from the regressive-phase dunes, $\overline{D}_{\cos}(t)$ depends strongly on the thickness of the dune that covered the molluscs during their history and how rapidly the dunes covered the fossils. Using geologically reasonable assumptions, Deely *et al.* (2010) derived ages that correlated with MIS 3/4, 5a, and 5c (Table 10a). While the presence of *Cerion* does not really constrain the dune's formation time, *Cerion* inhabit tropical vegetation that prefers sandy substrates with elevated fresh groundwater tables. Therefore, the *Cerion* dates appear to corroborate the idea that moderately high sealevels drowned part of the San Salvador platform in MIS 5a and 5c (Deely *et al.*, 2011).

Therefore, corals, terrestrial and lagoonal molluscs all gave consistent ages. They strongly suggested that San Salvador has preserved fossils from MIS 5a and 5e high seastands, as well as possibly, an MIS 5c high seastand. More dates are needed to confirm these data, especially for the MIS 5a and 5c phases (Deely *et al.*, 2011). Nonetheless, this study demonstrated that ESR can date the regressive dune sequences and the lagoonal

sediment to lock down the past elevations for sealevel during the highstands and the subsequent regression.

6.4 Dating Water Availability at Kharga Oasis, Egypt

Deserts have the nasty habit of depositing and then winnowing away the sediment, thus removing whatever record the sediment may have preserved. Bone and other softer tissues are usually destroyed by the weathering and exposure. Thus, determining paleoenvironmental conditions becomes much more difficult. Unlike TL and OSL, which can only date the sediment's initial deposition if it remains buried, ESR signals in carbonates and tooth enamel are not destroyed by light exposure. Thus, ESR can date materials exposed on the desert surface to date the desert's history. Understanding the hominid history in such ephemeral habitats, however, requires that one use proxy data. This study used ESR dating on snails to assess water availability, and thus, the potential for hominid habitation.

While today the hyperarid Western Desert, Egypt, gets ~ 0.01-0.07 mm/y precipitation, mostly as nocturnal condensation, evaporation rates average > 2 m/y. That, coupled with extensive groundwater pumping for local agriculture, ensures that area now lacks naturally occurring standing water (Smith *et al.*, 2007). In earlier wet climatic phases, however, this area may have received as much as 30 cm/y of net precipitation (Hawkins *et al.*, 2001; Osmond & Dabous, 2004).

Kharga Oasis had surface water repeatedly during the Pleistocene, as demonstrated by the lacustrine sediment and tufa deposits that form the large carbonate mounds dotting the Libyan Escarpment edge. These mounds, including at Matana and Midauwara (Figure 21), occur at nickpoints for the local groundwater aquifer where slope breaks occur along the escarpment. Where tufa dams block water flow, small basins form within the tufa deposits. At Kharga these basins range up to 1-5 km² in size. In order for basins to trap, and hence, preserve lacustrine silt or clay deposits, those basins must contain standing water for several decades to millennia. Freshwater snails, like the *Melanoides tuberculata*, *Gyraulus* sp., *Lymnaea stagnalis*, and *Planorbis planorbis* which occur in all the mounds near Kharga, can only live and breed when abundant fresh water fills lakes. Moreover, hominids and many ungulate species, also require relatively fresh surface water to drink daily. During higher rainfall events, the water retained in the ponds is likely to be fresher than if solely derived from groundwater (Adelsberger & Smith, 2010). During wetter phases in the Pleistocene, water was sufficiently abundant to support a large, relatively diverse ecosystem that included hominins who left Paleolithic artefacts scattered across the landscape ranging from Oldowan choppers to LSA settlements. Later peoples left Neolithic hearths and beads, while the ancient Egyptians and then the Romans left their agricultural deposits, pottery, temples, and waterworks (Smith *et al.*, 2004a, 2004b; Nicoll *et al.*, 1999, 2001; Churcher *et al.*, 1999; Mandel & Simmons, 2001; Kleindienst *et al.*, 2008, 2009).

While molluscs indicate times when surface water existed, only rarely do the archaeological artefacts occur within any sedimentary context that can be correlated with datable materials. Furthermore, intermittent fluvial scouring and the pervasive winds have removed many paleoenvironmental indicators and eroded the

context for many fossils and artefacts, leaving them to form a desert lag that contains debris mixed from several different geological periods. Thus, dating proxy materials becomes imperative for understanding both the geohydrology and the hominin occupations.

Unfortunately, datable materials are rare in the Western Desert. Although $^{230}\text{Th}/^{234}\text{U}$ can sometimes date the well preserved tufas ≤ 400 ka (Kleindienst *et al.*, 2008; Smith *et al.*, 2004a, 2004b, 2007, 2012), colluvial and eolian silt and sand with high concentrations of detrital ^{230}Th can easily contaminate the tufa. Commonly, the porous travertines and lake carbonates contain multiple episodes of calcite deposition rimming the pores that preclude accurate dates with any method. During the wet phases, the same precipitation source that fed the Ethiopian Highlands, the source for the Nubian Aquifer, probably also fed surface ponds and lakes in the Western Desert. Likely derived from airmasses originating over the Atlantic Ocean, this rainfall produced low $\delta^{18}\text{O}$ ratios in the tufa, as well as in deep and shallow ground water in the Nubian and Libyan Aquifers. It also maintained a small recurrent paleolake, and hence, a stable water source at Midauwara (Smith *et al.*, 2004a; Osmond & Dabous, 2004; Kieniewicz, 2007; Kieniewicz & Smith, 2007, 2009).

Several localities at Midauwara had temporally mixed populations of *Melanoides tuberculata*, some of which presented visibly distinguishable characteristics. In some cases, however, neither the gastropod species, size, nor their preservation state uniquely distinguished the various subpopulations (Blackwell *et al.*, 2012).

In open air desert sites, modelling both $\overline{D}_{\text{sed}}(t)$ and $\overline{D}_{\text{cos}}(t)$ is essential to account for the fossil's initial deposition, later sedimentation over the fossil, possible deflation and reworking, in order to constrain the fossil's age. Figure 22 illustrates such an analysis for snails from Matana 3, where a tufa unit above the snail-bearing silts suggests that the water ponded here for some time (Blackwell *et al.*, 2012). Using $\overline{D}_{\text{sed}}(t)$ and $\overline{D}_{\text{cos}}(t)$ for AM07b yielded an age of 69 ± 7 ka, which does not differ significantly from 63 ± 6 ka, the age calculated assuming continuous exposure at the surface. The age with the time-averaged dose rates also does not differ significantly from 83 ± 8 ka, the age calculated assuming burial under > 20 m of sediment. While clearly an impossibly low $\overline{D}_{\text{cos}}(t)$, the latter conditions constrains the absolute minimum age. The reality lies somewhere between these extrema, but does not produce any significant change in the calculated ages for the snails at Matana 3 (Figure 22), nor for those at Matana 2. At Matana 2, two subsamples gave an age of 28 ± 2 ka (Table 11; MIS 2/3 boundary), while at Matana 3, the two subsamples averaged 65 ± 4 ka (MIS 4). At Bulaq, a mound hugging the Escarpment edge south of Matana (Figure 21), Skinner *et al.* (2013) reported an ESR age of 24 ± 3 ka (MIS 2) for a single mollusc sample, which may be contemporaneous with stone-block structures found at a similar elevation by Caton-Thompson (1952).

At the Lazy Beach outcrop at Midauwara (Figure 23), a stratified section with a lightly cemented silt, rich in snails, is capped by another ~ 5 m of intercalated silt and tufa units. The snail bearing horizon sits ~ 4 m above the basin floor. A single sample analysis for snails weathering from the snail-bearing horizon here produced an age of 2.29 ± 0.38 Ma (Table 11). Substantially older than any other tufa outcrops found elsewhere and older than any archaeological materials found nearby, the estimated age hinted that should artefacts be found

here, they should be Oldowan. During the next field season, when trying to find enough shell for a confirming date, Oldowan artefacts were found within weathered debris at the base of this outcrop. Unfortunately, no artefacts were visible *in situ* within the tufa unit or on the cliff face, nor was enough shell found to allow another ESR date. Nonetheless, this association hints that the units here do date to the Early Pleistocene. Thus, climates were relatively wet during the Matuyama Chron at MIS 87, possibly making the Western Desert a viable route for early hominins to migrate across North Africa (Skinner *et al.*, 2013).

Four site clusters occur on the eastern side of the Midauwara mound. At Railway 1, nine subsamples all yielded consistent ages averaging 134 ± 8 ka, which correlates with a warmer blip in late MIS 6 or very early MIS 5, as does the age from Snail Trail 1 (Table 11). Where the largest *Melanoides tuberculata* seen in the area occur at Big Snail Gully, 10 subsamples all averaged 104 ± 5 ka, which correlates well with MIS 5d, as do two other sites. While analyzing multiple samples should occur at every site, for most sites, the small size of the deposits prevented more than one analysis. With > 50 analyses from 16 sites, ages cluster at the MIS 5e/6 boundary, in MIS 5d, 5a, and 4, at three different periods within MIS 3, at the MIS 2/3 boundary, and at the MIS 1/2 (Figure 24). Unexpectedly, no dates occurred for the coldest times during MIS 6 and only two in the colder parts of MIS 2, when $\delta^{18}\text{O}$ values were ~ -0.8 . Instead, the ages agreed best with the cusps between major glacial/interglacial swings, like times when the global climate was warming up toward MIS 5e or was cooling down from MIS 5e. Dates for standing water in MIS 5 correlated with slightly cooler periods when $\delta^{18}\text{O}$ values approached zero (Figure 24). While these data indicates that water occurs at particular times, some water that fed these ponds likely derived from the local aquifer, while some likely came from local precipitation (Smith *et al.*, 2004a). What that residence time might have been is currently unknown, but given the size of the aquifer, it probably would not lag the precipitation events by more than a few hundred years. Nonetheless, the data here do show that fresh surface water occurred more frequently and in more locations during MIS 5 and 3. That fresh water was available for use by hominins and their prey as they inhabited the Western Desert or crossed it to invade new territories (Blackwell *et al.*, 2012; Skinner *et al.*, 2013).

Thus, the great stability of the ESR signals provides dates that other chronometers cannot. Studies like this could be applied in other deserts and in more humid localities to help understand their geohydrological and hominid histories.

6.5 Dating Paleoclimate Change and the Hominid Occupation at Divje Babe I, Slovenia

Paleoclimatic data is only as good as the dates that allow that data to be correlated with similar data from other areas. This study details a method to measure proxies for paleohumidity and paleotemperature recorded by cave sediment. In this particular cave, ESR was the only method that provided reasonable ages for the whole of the excavation.

In caves, condensation moisture generally increases if both regional temperatures rise and meteoric precipitation increases, resulting in increased vegetational cover, higher potential evapotranspiration, higher

subsurface watertables, increased regional karstic erosion, more cave formation, more speleothem deposition, and more secondary sedimentary cementation. A short time lag can occur between the start of condensation corrosion and the secondary sedimentary cementation. When regional temperature drops with no change in meteoric precipitation, the potential evapotranspiration drops, but the relative atmospheric humidity rises both inside and outside the cave, causing more condensation corrosion, slower speleothem precipitation rates, and more surface weathering (Turk *et al.*, 2002; Blackwell *et al.*, 2008a).

Divje Babe I Cave contains a Late Pleistocene Paleolithic site, situated at about 450 m elevation overlooking the Idrijca River valley, about 50 km east of Ljubljana. In addition to acting as a cave bear den during the late Pleistocene, Divje Babe is also the richest Late Pleistocene site in Slovenia for fauna, charcoal, and pollen, which all indicate an alpine and predominantly temperate forest near the cave, similar to today's environment with occasional boreal elements, but that slowly degraded near the top to colder conditions (Culiberg & Šercelj, 1998; Toškan, 2003).

Within Divje Babe, roof collapse, mass wasting, cryoturbation, carbonate and phosphate corrosion, strong post-depositional weathering, including dissolution and reprecipitation, and faunal and hominid occupation have all combined to produce a complex sequence of largely inhomogeneous (lumpy) sedimentary layers. In 3-4 major sedimentary units (facies), 26 stratigraphic layers were identified, all of which contained significant quantities of dolomitic *éboulis* in dolomitic silty sands. While Layers 17a2, 17a3, and 20 contained sporadic flowstone, Layer 2 had experienced massive secondary carbonate deposition, and Layer 17/18, secondary calcite precipitation to form small aggregates, while several others had secondary phosphatic depositional cements, encrustations, and crusts. The abundant fossil remains coupled with periodically abundant water caused phosphatic dissolution throughout the sequence and secondary reprecipitation that post-depositionally cemented all Layer 8a and parts of Layers 7-8, 10-11, 16, 18a, 19, 20, 22, and 23. The phosphate mobilization in Layers 8, 10-14, and 16 suggests that these layers were waterlogged for some significant time. In Layers 2-5a significant cryoturbation and dry *éboulis* deposition in pockets indicate periglacial conditions. Layers 16-17 also had significant cryoturbation and locally massive *éboulis* deposition (Turk *et al.*, 2001, 2002, 2003, 2006; Blackwell *et al.*, 2008a).

The cave also periodically sheltered Paleolithic peoples, including a short-lived Aurignacian occupation in Layer 2. In Layers 21 through 3, several Mousterian occupations left behind lithic artefacts that are most concentrated in Layers 4, and 10-14, and 20. Well defined hearths with associated tools occur in Layers 5, 6, 8, 10-13, and 20. Most archaeological layers yielded small Mousterian artefact assemblages (*sensu lato*), with some bone awls and fragmented bone points, scrapers and denticulates, that all show heavy wear, including fracturing on one or more edges. In Layer 8 and deeper layers, numerous borers or *becs* were discovered that had lost their tip of the *bec*. Unambiguous evidence for human worship of cave bear skulls also occurs during deposition of Layer 8, analogous to the much younger example from Chauvet (Brodar, 1999; Bastiani *et al.*, 2000; Turk and Bastiani, 2000; Blackwell *et al.*, 2008a).

The site is most famous, however, for its "Neanderthal flute" found in Layer 8c. Human visits were

limited in duration, and likely seasonal or sporadic. Since most bears apparently died during hibernation (Debeljak, 2002), humans would probably not have risked visiting the site then. The abundance of cave bear bone available for tools, fat and marrow extraction may have drawn people to the site (Kunej & Turk, 2000; Turk *et al.*, 1995a, 2005; Blackwell *et al.*, 2008a).

Despite the preservation at Divje Babe I, which has even yielded cave bear hair, most ^{14}C dates exceeded the upper dating limits, while several others had contamination. Only the ages for the Aurignacian in Layer 2, at 35.3 ± 0.7 ky BP, and the bone from the Mousterian in Layer 6, at 43.3 ± 1.4 ky BP, should be considered reliable. ESR ages were completed for 28 teeth (Figure 25a), while 140 sediment samples were analyzed to determine volumetrically averaged sedimentary dose rates. On site, the volume of *éboulis* and aggregates were measured, and combined with details about corrosion etching, aggregate formation, subsurface weathering rates, and frost wedging (Table 12) to assess paleohumidity and paleotemperatures and to build paleoclimatic curves (Figure 25b; Turk *et al.*, 1995b, 2002; Blackwell *et al.*, 2008a).

The ages showed internal consistency when plotted stratigraphically (Figure 25a). The dates and paleoclimatic analyses suggest that four sedimentation cycles had occurred within Layers 2 to 23. In Layers 13 to 23, ESR ages ranged from 80 ± 8 to 116 ± 12 ka, indicating deposition in MIS 5. In Layers 8a-10, dates ranged from 59 ± 3 to 67 ± 4 ka (MIS 4), suggesting that the flute probably dates to $\sim 60 \pm 4$ ka. Ranging from 40 ± 5 to 50 ± 6 ka for Layers 2-7 (MIS 3), the ESR ages agree within their uncertainties with the ^{14}C ages for Layers 2 and 6. Nonetheless, the consistent offset suggests that, at this age range, the calibration between the ^{14}C ages and calendric ages may average ~ 3 -5 ky (Blackwell *et al.*, 2008a).

Sedimentation likely occurred continuously from the initiation of Layer 23 at ~ 116 ka in the warm, dry Phase DB21 (see Figure 25b) through a warm, moderately humid climatic phase in Layer 20 (~ 110 ka) and Layer 17-18 boundary (the flowstone; Phase DB15), until ~ 102 ka, when the lower Layer 17a2 (warm, dry Phase DB14) was deposited. These correlate with the final phase of MIS 5e and Dansgaard-Oeschger (DO) Event 25. This deposition rate averages 0.02 cm/y, which is typical in a cave actively inhabited by humans and bears, who carry in sediment as they enter and who leave behind large masses of bone. The cementation events that cement the “breccias” in Layers 18a (DB17), 20/21 (DB19), and 23 (DB21) appear to correlate with marginally cooler, but more humid periods during MIS 5e-5d, and with somewhat cooler phases on the GRIP curve. DB Phases 16-14 correlate respectively with DO Events 24 and 23 (= Brørup Interstadial in MIS 5c; Figure 25b; Blackwell *et al.*, 2008a).

Then, a drastic decrease in the sedimentation rate producing a hiatus in deposition lasted until ~ 85 ka when more rapid sedimentation resumed (Figure 25a). From ~ 85 ka in upper Layer 17a2 until 78 ka in Layer 13, during MIS 5a, the sedimentation rate averages 0.036 cm/y. The cementation in Layers 15/16a (= cool wet Phase DB11) appear to correlate with another marginally cooler, but more cooler, more humid period at the beginning of MIS 5a on the GRIP curve, while Layer 13-14, the warm, relative dry Phase DB10, is likely coeval with DO Event 21, the Odderade Interstadial (Figure 25b; Blackwell *et al.*, 2008a).

The deposition of Layer 13, a rich archaeological layer, precedes a hiatus that lasted from ~ 78 to 70 ka and correlates with the MIS 4/5a boundary and the early glacial maximum (= Würm 1/Early Weischelsian/Early Wisconsinan Glaciation) starting at about 76 ka, a time that further north saw rapid extreme climatic oscillations (Barron *et al.*, 2003). At this hiatus, all sedimentary characteristics, including the frequency of aggregates, corrosion, and post-depositional clast cryoclasts in all classes, changes. Pollen production also shifted as typically occurs at the MIS 5a/4 boundary (Culiberg & Šercelj, 1998; Blackwell *et al.*, 2008a)

Rapid deposition at ~ 0.012 cm/y resumed, depositing Layers 12-10 through 8a, from ~ 70 to 55 ka (Figure 25b), a period that correlates with the later part of MIS 4 and matches a slightly warmer phase in the pollen records from Castiglione, Italy (van Andel, 2003), but further north appears to have been quite cold. Layers 12-10 contain the richest Paleolithic unit, Horizon E, which included "advanced" elements, such as *becc* and bone points. Despite the materials here having Upper Paleolithic affinities, they are clearly Mousterian, as is evident from the extensive use of Levallois reduction strategies and the abundance of scrapers and denticulates. Layer 12 probably correlates with the end of DO Event 20, and the cool, humid climatic Phase DB9 in upper Layer 11 likely with DO Event 19 at ~ 68 ka. At the boundary between Layers 8b and 10, the cold, very humid climatic Phase DB8, probably correlates with the cold phase from 62-65 ka in MIS 4, between DO Events 18 and 19, while upper Layer 8b was deposited during the warm, but less humid climatic Phase DB7, suggesting that it correlates chronostratigraphically with DO Event 18 at ~ 61 ka. Divje Babe Layers 8c-8a were deposited between ~ 60 and 58 ka (Figure 25b) during the cold, humid Phase DB6. The warm, humid Phase DB5 correlates directly with DO Event 16, aka the Oerel Interstadial, and also, likely corresponds to Zone 11 in the Lago Grande di Monticchio pollen record (Allen *et al.*, 2000). After deposition of Layer 7 (DB5), sedimentation slowed or possibly stopped at ~ 55-50 ka, which correlates well with the cold phase between the Oerel and Glinde-Moerschoofd Interstadials in Northern Europe and the short phase at Castiglione with low arboreal pollen (van Andel *et al.*, 2003; Blackwell *et al.*, 2008a)

From ~ 45 to 40 ka, sedimentation began again, depositing Layers 6 to 2, at a mean rate of about 0.04 cm/y (Figure 25a). This period correlates with several warmer phases, the Dansgaard-Oeschger Events 14-8, in mid MIS 3 (Figure 25b; Huntley and Allen, 2003). Having the cold, dry Phase DB4, Layer 6 likely correlates with the cold period between the Hengelo and Glinde Interstadials and also corresponds with the time of the lowest winter temperatures seen in the Grand Pile, Les Echets, and Lago Grande di Monticchio pollen records (Allen *et al.*, 1999). Since Layers 5a1-5 were deposited under the warmer, somewhat drier Phase DB3, they probably correspond chronostratigraphically to DO Event 12 at about 42-44 ka, which equates with the Hengelo Interstadial (Dansgaard *et al.*, 1993; Weissmüller, 1997). In Layers 5a-4, the cold, moderately humid Phase DB2, probably correlates with the cold, humid phase between the Hengelo and Les Cottés Interstadials. At 38-39 ka, Layers 3 and 2 were deposited in the warm, dry Phase DB1, that most likely correlates with DO Event 8, and from the pollen with the Les Cottés Interstadial on the GISP2- $\delta^{18}\text{O}$ curve (Blackwell *et al.*, 2008a).

In central and western Europe, the Middle-Upper Palaeolithic transition occurred during the Hengelo to

Les Cottés Interstadials (i.e., ~ 44-34 ka). Divje Babe Layers 5-2 (Phases DB3-DB1) correlate with this period archaeologically, chronologically, and paleoclimatically, but house nothing special archeologically due to the sparse finds. By Layer 4, however, the occupants had abandoned the Levallois tradition, that had, among other things, characterized the earlier Mousterian horizons. Assuming uniform sedimentation rates in Layer 5-2, the MP-UP transition occurred about 40 ka at Divje Babe I (Blackwell *et al.*, 2008a).

This profile from Divje Babe I is the first sedimentological analysis from a Slovenian cave to permit such detailed paleoclimatic interpretations and correlations on a regional, continental, or global scale. Unfortunately, correlations with other nearby Mousterian and Aurignacian sites are currently impossible due methodological differences (Turk *et al.*, 2006; Blackwell *et al.*, 2008a). Nonetheless, this study stands as the most extensive use of ESR dating within a single site and one of the few where the paleoclimatic data have been fully exploited to build a detailed history.

6.6 Dating the Naisiusiu Beds, Olduvai Gorge, Tanzania

Even when volcanoes abound in an area, archaeologists cannot always obtain reliable $^{39}\text{Ar}/^{40}\text{Ar}$ dates. Like every method, preservation of the right samples for $^{39}\text{Ar}/^{40}\text{Ar}$ must occur before they can be dated. Also, $^{39}\text{Ar}/^{40}\text{Ar}$ frequently cannot provide ages in the range between 20 and 200 ka, where its lower dating limit usually falls.

At Olduvai Gorge, Tanzania, the lower sequence in Beds 1-IV that houses the early Pleistocene hominin sites has been well dated, but the younger beds dating from the later Pleistocene, like the Naisiusiu Beds, have not been well dated, despite the volcanic horizons there. In 1969, Mary Leakey found Later Stone Age tools, lithic production waste, teeth, and fragmentary bones, including *Equus birchelli* at MDL Hill (Leakey *et al.*, 1972; Skinner *et al.*, 2003).

MDL Hill, the type section for the Naisiusiu Beds (Figure 26), sits about 110 m W of the Second Fault. At MDL Hill, the lower Naisiusiu Beds constitute a sequence of intercalated sandstones, conglomerates, and cemented tuffaceous units overlying a basal basalt. Deposited in an alluvial-fluvial setting, this sequence has two fossiliferous units that yielded teeth for ESR dating. These lower beds are capped by the aeolian upper Naisiusiu Beds, of which the Cattle Trail site is a typical outcrop. Cattle Trail also yielded teeth whose age sets the upper limit for the Naisiusiu Beds' deposition (Skinner *et al.*, 2003). Modern C contamination coupled with their age falling close to the upper dating limit prevented ^{14}C dating at both the MDL Hill and Cattle Trail sites. Meanwhile, MDL's age falls close to the $^{39}\text{Ar}/^{40}\text{Ar}$ lower limit, and its secondary cementation hampered attempts with $^{39}\text{Ar}/^{40}\text{Ar}$ (Hay, 1976; Manega, 1993). Amino acid racemization analysis on bone proved equally problematic, and has since been shown to be more dependent on diagenesis than chronometric age (Bada, 1981; *cf.*, Blackwell, 1987; Blackwell *et al.*, 2000b, 2005b). Ambrose (1998) suggested that the Middle-Late Stone Age transition occurred before 46 ka.

The Cattle Trail tooth averaged 39 ± 2 ka for the youngest of the Naisiusiu Beds (Table 13). Since the

ESR ages for the three teeth from MDL agreed well within their errors, it is unlikely that all three were reworked. Therefore, these data suggested that the Middle-Late Stone Age transition had already occurred by 62 ± 5 ka in MIS 4. This is the oldest date yet published for the Middle-Late Stone Age transition. Again, ESR had proven its worth for dating sites with archaeological significance.

6.7 Dating the Hominid at Obi-Rakhmat, Uzbekistan

Hominids have the bad habit of wanting to live in many places where the radiometric methods like $^{39}\text{Ar}/^{40}\text{Ar}$ cannot be applied for lack of a volcanic ash source. Their accumulated waste, particularly bone, often makes it impossible to date associated travertine or speleothem by $^{230}\text{Th}/^{234}\text{U}$. Many sites also exist in that zone where the site exceeds the ^{14}C upper limit. Yet archaeologists and human paleontologists demand dates for their sites. This study exemplifies a situation where both $^{230}\text{Th}/^{234}\text{U}$ and ^{14}C could not provide dates for the hominids or their tools.

Central Asia provided a potential corridor for anatomically modern humans migrating from Africa and the Levant into northern and eastern Eurasia during the late Pleistocene (e.g., Wells *et al.*, 2001; Kuzmin & Keates, 2006). Having little reliable chronological data for the central Asian Paleolithic sites hampers reconstructions for hominin settlement patterns in, and migrations through, the area. Research at archaeological sites in this region, such as Obi-Rakhmat, can also help to elucidate regional paleoenvironments.

Located 100 km northeast of Tashkent, Uzbekistan, the site in the Obi-Rakhmat abri contains 22 geological layers, mainly comprising sandy silts cemented by carbonate (Figure 27). Layers 11, 12, 14, and 15 contain high concentrations of limestone *éboulis* up to 40 cm in diameter derived from stoping and roof collapse. All layers contain some *éboulis*, but below Layer 16, *éboulis* are less common, hinting that the climate then may have been warmer than that during the Layer 11-15 deposition (Krivoschapkin & Brantingham, 2004; Skinner *et al.*, 2007a).

More than 40,000 Paleolithic artefacts and > 3,000 animal bones were found along with a lithic industry, rich in blades, that displays a combination of Middle and Upper Paleolithic traits (Derevianko *et al.*, 2001; Krivoschapkin & Brantingham, 2004). The animal taxa suggested a mixture of species preferring mountainous forest or steppes (Wrinn, 2004), while the pollen analyses indicated that a dry shrub-forb steppe dominated the late Pleistocene landscape (Kul'kova, 2004). Layer 16 yielded six hominid teeth and many hominin cranial fragments (Glantz *et al.*, 2004), that constituted a partial cranium with strong Neanderthal affinities (S.E. Bailey, pers. comm., 2007).

For Obi-Rakhmat, ^{14}C ages ranged from 9.8 to 48.8 ka, but did not increase consistently with depth. Yet for many of the same layers, $^{230}\text{Th}/^{234}\text{U}$ dates for stalagmitic travertine from a spring mound at the back of the abri ranged from 87.1 to 97.1 ka, but with relatively little variation with depth (Wrinn *et al.*, 2004; Figure 28). Moreover, recent ^{14}C analyzes at other archaeological sites older than 35 ka often have yielded ages that were dramatically inconsistent with the site stratigraphy (e.g., Conard & Bolus, 2003). ESR analyses were used to

date eight bovid teeth from Obi-Rakhmat to attempt to resolve the dating discrepancy. Dentinal U concentrations ranged up to 155 ppm, which suggested saline to hypersaline groundwater in the cave, and a dry climate outside (Skinner *et al.*, 2007a).

Coupled ESR- $^{230}\text{Th}/^{234}\text{U}$ ages and isochron analyses indicated that using the U uptake parameter, $p = 0$, was the most accurate for this site. Moreover, no teeth showed secondary U uptake or leaching. For three teeth, the points scattered somewhat around the isochron, suggesting that some minor, very recent U remobilization had affected these teeth (*cf.* Blackwell *et al.*, 2002), but not enough to reduce the accuracy of the ages (Skinner *et al.*, 2007a).

For eight teeth from four layers Obi-Rakhmat, dates ranged from 57 ± 2 ka to 87 ± 4 ka (Figure 28). These ages correlate with MIS 5a-4, when world climates were cooling. Assuming constant sedimentation in Layers 21.2 to 13, the Layer 16 hominids date to 74 ± 5 ka. ESR dating teeth from Layers 10, 14, and 17 would yield more accurate sedimentation rates, improving the age estimates for the hominids and other Paleolithic cultural deposits at Obi-Rakhmat. Nonetheless, this provides the first reliable date for the Obi-Rakhmat hominid and the archaeological materials there (Skinner *et al.*, 2007a).

This is only one of many sites where the radiometric chronometers, like ^{14}C , $^{230}\text{Th}/^{234}\text{U}$, and $^{39}\text{Ar}/^{40}\text{Ar}$, have not yielded reliable ages for hominids or their artefacts (see references in Blackwell, 1995, 2006), due to the nature of the preservation, the site geology, or other problems.

7.0 Conclusions

Other new chronometers, such as ESR dating for shark and marsupial enamel (e.g., Blackwell *et al.*, 2002b, 2004) and the Pb centre in amazonite (e.g., Mittani *et al.*, 2004), are under development. Dentine and bone dating seems to be plagued with problems of secondary U remobilization on a much greater scale than in enamel, which when coupled with the problem of the organic $\dot{\text{C}}$ radical seen in the spectra, has not allowed these materials to be used reliably yet (e.g., Blackwell *et al.*, 2002c). Given the strides made in the development of ESR methods in the last few years, no doubt the next 20 will see equally important improvements.

Its accuracy and wide range of datable materials makes ESR an important chronometer for dating Quaternary sites and solving geological, archaeological, and paleontological problems. Nonetheless, ESR cannot be used as the sole chronometer in any site where other methods can be also applied. To be assured that an age from any chronometric method is accurate, it should be corroborated with a second and even a third age from (an)other chronometer(s). Only by getting consistent ages from several methods that have different underlying assumptions, and that depend on different geochemical processes can one be certain that the ages are correct. Therefore, ESR remains an important weapon in the Quaternary geochronologists' arsenal of methods to date sites when no other methods will work or to corroborate other dates.

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Figure Captions

Figure 1. The age ranges for Pleistocene materials.

ESR can date a longer range within the Pleistocene than most other date methods, including ^{14}C , TL, OSL, $^{230}\text{Th}/^{234}\text{U}$ and U/Pb.

Figure 2. ESR signal production.

With increased irradiation, the ESR signal's intensity grows, eventually reaching saturation (adapted from Blackwell, 2006):

- After absorbing energy from incident radiation, excited electrons jump to, and move through, the conduction band. Although most return to the ground state, a few get trapped in charge site defects that have specific energies above the ground state. Such traps often occur where trace element have substituted into the crystal lattice. With irradiation, such trapped charges accumulate. Each trapped unpaired electron and the empty hole it left behind in the valence band generate a magnetic field with a characteristic ESR signal.
- When naturally irradiated, an ESR signal eventually saturates at its maximum accumulated dose, $\mathcal{A}_{\Sigma,\text{sat}}$, or at a steady state accumulated dose, $\mathcal{A}_{\Sigma,\text{ss}}$, where signal loss due to fading equals signal production.

Figure 3. ESR signals in teeth.

With increased irradiation, the ESR signal's intensity grows, eventually reaching saturation (adapted from Blackwell, 2006):

- For any sample, any possible radiation sources exist to create the ESR signal. In teeth, U in the enamel itself helps to create the signal, but U in the dentine and other mineralized tissues also emits radiation. U, Th, K, and other radioactive elements in all the sedimentary components also emit radiation.
- Except for teeth that have been irradiated in a nuclear accident, the hydroxyapatite in live teeth has no signal. If a live tooth experiences irradiation, a measurable signal appears after ~ 0.01 Grays exposure, making it a useful signal for monitoring exposure during nuclear accidents. In fossil teeth, a measurable hydroxyapatite (HAP) signal is present after ~ 0.5 -20 ka, depending on the total dose rate that the tooth experiences. When any tooth is artificially irradiated, the HAP signal will enlarge.

Figure 4. Zeroing in quartz and chert.

All signals can be zeroed with heat, but more irradiation will regenerate the signal. Quartz signals can be zeroed with (adapted from Blackwell, 2006):

- UV radiation or sunlight can zero the Ge signal and partly bleach the Al, Ti, and OHC signals.

- b. In this chert, the E' signal and its accumulated dose, $\mathcal{A}_\Sigma$, was zeroed by heating to 350-450°C.

Figure 5. The effects from shear strain on ESR signals in quartz.

In quartz, shear strain will reset most ESR signals (adapted from Blackwell, 2006):

- a. As the strain rises, the differences in ESR intensity between different grain size fractions drop.
- b. At a normal stress of 10 MPa, the measured accumulated dose, $A_{\square}$, drops with decreasing grain size for both the E' and Al signals, until at a small grain size, the two signals give equal $A_{\square}$ values.

Figure 6. Determining the accumulated dose, $\mathcal{A}_\Sigma$.

The additive dose method is used to calculate the accumulated (or environmental) dose, $\mathcal{A}_\Sigma$:

- a. Plotting the signal intensity *versus* the added radiation dose produces a growth curve. The x -intercept for this curve gives $\mathcal{A}_\Sigma$. This bovid tooth from Treugol'naya Cave, Russia, has a large $\mathcal{A}_\Sigma$, as expected for a Middle Paleolithic site deposited during Marine Isotope Stage (MIS) 11 (Blackwell *et al.*, 2005a).
- b. For signals with interference, signal subtraction can reduce its effect (adapted from Blackwell, 2006):
 - Curve 1: A pure Al signal is unaffected by interference signals.
 - Curve 2: An organic radical signal, $\dot{C}$, interferes with the Al signal.
 - Curve 3: Unidentified interference signals affect the Al signal.
 - Curve 4: The Al and $\dot{C}$ signal in a natural archaeological sample.
 - Curve 5: The same sample as Curve 4 heated for 10 minutes at 320°C to zero the Al signal.
 - Curve 6: Subtracting Curve 5 from Curve 4 gives a signal that shows the hyperfine splitting typical for the Al signal (see inset).

Figure 7. Factors affecting the internal dose rate, $D_{\text{int}}(t)$.

For minerals having, or capable of absorbing, U, such as teeth and molluscs, U uptake must be measured or modelled, as must any loss of U or its U daughters via leaching or degassing:

- a. After an initial EU uptake of 10 ppm U but no Th or Pa, the U daughter isotopes ingrow causing $D_{\text{int}}(t)$ to rise (adapted from Blackwell, 2006).
- b. When created by U decay, radon (Rn) gas easily escapes during diagenesis or fossilization, causing $D_{\text{int}}(t)$ to fall. This can reduce the age accuracy, if the Rn loss goes unrecognized. Assuming 0% Rn loss will not contribute significant errors to the age calculation for most samples, except those with high U concentrations. In this mammoth molar from Üröm, Hungary, the selected U uptake model significantly affects the age calculation, because the dentine contains relatively high U concentrations, creating significant differences in the different calculated model ages (adapted from Blackwell, 2006).

- c. The early uptake (EU) model assumes that the fossil absorbed all its U rapidly after death, but the recent uptake (RU) model assumes that the tooth absorbed little U until just before the end of Event 1. In the linear uptake (LU), the fossil absorbs U continuously and constantly throughout Event 1. Analogously, linear leaching (LL) assumes continuous, constant U loss throughout Event 2. Under early leaching (EL), the fossil loses U in a geological instant some time before the fossil's recovery, whereas under recent leaching (RL), the loss occurs just before recovery. More complex models can be devised by combining several uptake and/or leaching events (adapted from Blackwell, 2006).
- d. For T76en1, from Yarımburgaz, Turkey, all the calculated model ages fell as the apparent U concentration rose. The shaded fields around each line show how the associated errors changed with U concentration (adapted from Blackwell *et al.*, 2010b).

Figure 8. Factors affecting the sedimentary dose rate, $D_{\text{sed}}(t)$, reaching dating samples.

Radiation can reach the dating sample from radioactive decay occurring in sediment up to 30 cm away:

- a. α particles deliver a variable component to $D_{\text{sed}}(t)$ and $D_{\text{int}}(t)$. Since β particles penetrate ~ 1 -2 mm, roughly the thickness of tooth enamel or a mollusc shell, $D_{\text{sed}}(t)$ and $D_{\text{int}}(t)$ calculations must include β attenuation in the sample and the sediment, but the latter rarely need to include more than two or three sedimentary units (adapted from Blackwell, 2006).
- b. In sediment, γ radiation penetrates ~ 30 cm, and can come from several sedimentary units or components, which can each yield different dose rates (adapted from Blackwell, 2006).
- c. In “lumpy” sites, those with inhomogeneous or thinly bedded sedimentary units, different mineral clasts often have different radioactive element concentrations, which deliver dose at very different rates. $D_{\text{sed}}(t)$ calculations must volumetrically average the individual dose rate from each stratigraphic unit or sediment type relative to its volumetric contribution and location within the sphere around the sample (adapted from Blackwell, 2006).
- d. Sedimentary water attenuates $D_{\text{sed}}(t)$ reaching a sample. As the sedimentary water concentration, W_{sed} , rose for this mollusc from Hatay, Turkey, the calculated ESR age also increased. Here the age does change significantly regardless of W_{sed} . For most samples, changing W_{sed} by < 10 wt% does not significantly affect the calculated age, especially for samples where $D_{\text{sed}}(t)$ represents a small percentage of the total dose rate. For some samples, if W_{sed} changes by > 10 wt%, the model ages will exceed the reported values by more than the 2σ uncertainty (after Florentin *et al.*, 2013).

Figure 9. The effect on ESR ages from the cosmic dose rate, $D_{\text{cos}}(t)$.

$D_{\text{cos}}(t)$ depends on several variables:

- a. At higher altitudes and higher latitudes, $D_{\text{cos}}(t)$ increases (adapted from Blackwell, 2006).

- b. As water or sediment cover depth increases over a sample, $D_{\cos}(t)$ drops quickly, while the calculated age rises. For samples covered by > 40 m of water, the contribution from $D_{\cos}(t)$ is negligible compared to errors in the total dose rate from other sources (adapted from Blackwell *et al.*, 2007a).
- c. To calculate $\bar{D}_{\cos}(t)$, a ramped box model was constructed using instantaneous $D_{\cos}(t)$ values integrated over time. At 125 ka, RM79 sat under 10 cm of sediment in 5 m of water with $D_{\cos}(t) = 127 \mu\text{Gy/y}$. With 50 cm of sediment by 115 ka, $D_{\cos}(t)$ fell to $98 \mu\text{Gy/y}$. After sealevel dropped 5 m rapidly, $D_{\cos}(t)$ rose to $194 \mu\text{Gy/y}$. Erosion reduced the sediment to 10 cm today, while $D_{\cos}(t)$ fell to $252 \mu\text{Gy/y}$ (adapted from Deely *et al.*, 2011).
- d. For this Turkish mollusc, the calculated age dropped exponentially. A $100 \mu\text{Gy/y}$ rise in the measured $D_{\cos}(t)$ would drop the age by > 20 ky, while a $50 \mu\text{Gy/y}$ rise drop in $D_{\cos}(t)$ would raise the calculated age by > 15 ky. Both would produce significant differences compared to the 2σ age uncertainties (after Florentin *et al.*, 2013).

Figure 10. ESR isochrons.

If a large tooth can be broken into several subsamples with variable amounts of U, then an isochron can be built:

- a. When the total accumulated dose, $\mathcal{A}_{\Sigma,i}$, for each subsample, i , is plotted *versus* the time-averaged internal dose rate for that subsample, $\bar{D}_{\text{int},i}(t)$, the slope of the regression line yields the sample's age, t , while the y-intercept equals the externally derived component of the accumulated dose, $\mathcal{A}_{\text{ext}}$ (adapted from Blackwell *et al.*, 2002a).
- b. A isochron for a molar from Bau de l'Aubesier, Provence: In practice, each U uptake model produces a line, which all converge on $\mathcal{A}_{\text{ext}}$. Comparing the $D_{\text{ext}}(t)$ known from the sedimentary geochemical analyses with that from the isochron showed that this tooth experienced U uptake with uptake parameter, $p = 0.5$ (adapted from Blackwell *et al.*, 2002a).
- c. If a sample, such as this tooth, has experienced U leaching or a secondary U uptake event, the isochron's intercept can be negative. Here, the secondary uptake event must have occurred recently, because the isochron age agrees well with $^{230}\text{Th}/^{234}\text{U}$ ages for adjacent stalagmitic horizons (adapted from Blackwell *et al.*, 2002a).
- d. For this tooth from Pradayrol, France, diffusional remobilization has affected parts of the tooth, shifting their points for three subsamples off the isochron. Ignoring the affected points gives an age of 327 ± 24 ka, an age that agrees well with the ages for the other three teeth found in this layer.

$\bar{D}_{\text{int},i}(t)$, t , and $\mathcal{A}_{\text{ext}}$ all depend on the U uptake model selected after the first iteration of this technique.

Figure 11. Coupled ESR- $^{239}\text{Th}/^{234}\text{U}$ dating for corals from the Florida platform and Isla de Mona.

To test the sealevel curve that best applies to the Florida platform (adapted from Blackwell *et al.*, 2007a),

- Six corals from the Florida Keys and Florida Bay were dated by coupled ESR- $^{239}\text{Th}/^{234}\text{U}$ dating.
- The coupled ESR- $^{239}\text{Th}/^{234}\text{U}$ dating analyses yielded depth profiles for the five corals dating to MIS 5 that matched better with the curve by Waelbroeck *et al.* (2002). One sample from Isla de Mona fell well above any sealevel curve suggesting that it had been uplifted at some time.

Figure 12. Benthic foraminiferal ages, Cear Rise, Central Atlantic.

For the EW9209 1JPC core, AMS ^{14}C ages for the core between 0 and 1.5 m depth were determined with planktonic forams. For greater depths, the $\delta^{18}\text{O}$ data were correlated with the SPECMAP curve to calculate the ages. BF1, benthic foraminifera from 478-492 cm dated at 118 ± 38 ka, which agrees well with the known core age at this depth. Although the exact age at depth 575 cm is unknown, its higher sedimentation rate plus its higher clastic sediment content and the low foraminiferal abundance hints that this level was deposited during a glacial interval. Benthic forams from 560-570 cm dated at 149 ± 17 ka, which agrees well with the age at ~ 145 ka extrapolated from the known ages higher in the core (core data from Curry & Oppo, 1997; ESR data from Blackwell *et al.*, 2010c).

Figure 13. Early-Middle Quaternary Palaeochannels in East Anglia.

Before the Anglian Glaciation (MIS 12) at ~ 450 ka, the Bytham River drained south-central England, emptying into the North Sea in Norfolk (map adapted from Rose, 2009). Quartz was collected for ESR analysis from cores in channels in the Bytham River system. These are associated with the terrestrial Cromerian Forest Beds.

Figure 14. The Khyex River landslide site, British Columbia.

The landslide occurred along the Khyex River, 35 km east of Prince Rupert, in northwestern BC. The landslide formed in varved glaciomarine silt and clay. During the Late Wisconsinan Glaciation, ~ 11 -12 ka, these beds were deposited, when the Pacific Ocean invaded the Skeena River estuary as the alpine glaciers were melting (adapted from Blackwell *et al.*, 2010a).

Figure 15. The glaciomarine stratigraphy exposed by the slide at Khyex River, BC.

At the exposed profile at Khyex River, glaciomarine rhythmically laminated silty clay coarsens upward to sand. These underlie Holocene colluvium. *Balanus crenatus* (RM33) were anchored to bedrock and *Balanus nubilus* (RM32) sat in the displaced sediment block. The barnacles and other fauna define a marine paleoshoreline sitting at 30 m amsl. Wood from the base of the sand unit gave an uncalibrated ^{14}C age of 10.29 ± 0.03 ky BP. Two small *B. crenatus* and the giant *B. nubilus* gave uncalibrated ^{14}C dates of 11.57 ± 0.04 ky BP and 11.80 ± 0.03 ky BP respectively. By ESR, RM33 dated at 15.0 ± 2.1 ka, while RM32 dated at 15.1 ± 1.2 ka. Both ages agree well with the uncalibrated ^{14}C ages (adapted from Blackwell *et al.*, 2010a).

Figure 16. The stratigraphy on Yali, near Nisyros, Greece.

The oldest paleosol here (YOP) reaches ~ 50 cm thick above Yali's marine terrace (YMT). Samples 2009YAL12 and 2009YAL14 were dated by ESR (after Blackwell *et al.*, 2011).

Figure 17. Raised terraces and tectonic features in the Antakya Graben, Hatay Province, Turkey.

Several large normal faults mark the southern rim of the Antakya Graben. Many have compressive stress in a north-south direction, which has uplifted the south side relative to the north side. Coupled with Quaternary sealevel fluctuations, the uplift has created several marine terraces that rim the graben along the coast (after Florentin *et al.*, 2013).

Figure 18. The raised marine terraces near Samandağ and Mağaracık, Hatay Province, Turkey.

Quaternary sealevel fluctuations combined with tectonic uplift on the faults along the Antakya Graben have produced several stacked marine terraces on the coastal plain near Mağaracık and Samandağ. ESR dating samples were collected from six marine terraces near Mağaracık and Samandağ (after Florentin *et al.*, 2013).

Figure 19. San Salvador's Quaternary surficial geology.

Quaternary regressive and transgressive dune sequences cover the San Salvador platform, while coral reefs from the Cockburn Town Member of the Grotto Beach Formation rim much of the island. Molluscs and corals from the Grotto Beach Formation were dated in order to build a sealevel curve for the island (after Deely *et al.*, 2011).

Figure 20. Modelling the time-averaged cosmic dose rate, $\overline{D}_{\text{cos}}(t)$, for RC44, San Salvador, Bahamas.

This coral sat in the Cockburn Town Reef. Assuming that its possible depth during growth ranged from 1 to 10 m, and its maximum sediment cover did not exceed 2 m more than its current cover of 1 m yields ages that range from 121 to 128 ± 8 ka. All these ages correlated with MIS 5e (adapted from Deely *et al.*, 2011).

Figure 21. The Kharga Oasis Depression.

Near Kharga, large tufa mounds dot the escarpment marking the edge between the Libyan Plateau and the Kharga Oasis Depression. Within Kharga, lacustrine silts containing freshwater snails were sampled at Midauwara, Bulaq, and Matana to assess when fresh water might have been available for hominin and other vertebrates to drink (after Skinner *et al.*, 2013).

Figure 22. AM07b's age vs. external dose rate, $D_{\text{ext}}(t)$.

As $D_{\text{ext}}(t)$ rises, the ages drop and the differences between the different model ages decrease. Considering the 2σ errors (95% confidence limit), the ages calculated with a cosmic dose rate, $D_{\text{cos}}(t) = 0 \mu\text{Gy/y}$, did not differ significantly from those calculated using the time-averaged external dose rate, $\overline{D}_{\text{ext}}(t)$, which was modeled for changes in both $D_{\text{cos}}(t)$ and $D_{\text{sed}}(t)$ with time. The ages calculated using the modern $D_{\text{cos}}(0)$, $292.9 \mu\text{Gy/y}$, do not significantly differ from those ages calculated with $\overline{D}_{\text{ext}}(t)$ (after Blackwell *et al.*, 2012).

Figure 23. The tufa deposits at Midauwara, Egypt.

At least four generations of spring-fed tufa deposits form the Midauwara mound. The tufas dammed ponds or small lakes which collected carbonate-rich lacustrine silt and freshwater gastropods, such as *Melanoides tuberculata* and *Gyraulus*. Gastropods were collected from 16 localities clustered in six areas (after Skinner *et al.*, 2013).

Figure 24. ESR dates showing the times for standing water at Kharga Oasis, Egypt.

Molluscan ESR dates show that fresh standing water sat at Kharga Oasis during Marine Isotope Stage (MIS) 87 in the Early Pleistocene. Water was also present at several localities in Midauwara during MIS 5e, 5d, and 5a, as well as during MIS 4, 3, and 2. Matana had water during MIS 4 and at the MIS 2/3 boundary, while Bulaq had water in early MIS 2 (after Skinner *et al.*, 2013).

Figure 25. Paleoclimatic analyses at Divje babe I, Slovenia.

Divje Babe I's Late Pleistocene shows major climatic fluctuations which correlate well with other Late Pleistocene records:

- a. By comparing the Holocene frequency of aggregate and congelifactor formation against that seen in Pleistocene cave sediment, relative paleotemperature (dotted line) and paleohumidity (solid line) profiles were constructed.
- b. The GRIP (summit) record for Pleistocene climate.

This strong correlation in OIS 3 and 4 suggests relatively continuous sedimentation. At the Layer 12/13 boundary dated at ~ 80 ka, a short depositional hiatus occurred. A short hiatus or depositional slowdown may have also occurred between Layers 6 and 7, and possibly, within Layer 17a2 (adapted from Blackwell *et al.*, 2008a).

Figure 26. The stratigraphy for the Naisiusiu Beds, Olduvai Gorge, Tanzania.

At MDL Hill, the Naisiusiu Beds constitute a sequence of intercalated sandstones, conglomerates, and cemented tuffaceous units deposited in an alluvial-fluvial setting, overlying a basal basalt. The sequence includes two fossil rich layers. Teeth from both MDL Hill and Cattle Trail were dated (adapted from Skinner *et al.*, 2003).

Figure 27. The stratigraphy on the western wall of the excavation at Obi-Rakhmat, Uzbekistan.

Many layers, especially the lower layers, contain large amounts of *éboulis*, limestone derived from roof stoping or collapse. Layer 15 contains *éboulis* from a large roof collapse. Layer 16 yielded several hominin teeth and cranial fragments (adapted from Skinner *et al.*, 2007a).

Figure 28. Dates for Obi-Rakhmat, Uzbekistan.

The ^{14}C ages scatter and bear no relationship to the stratigraphy. The $^{230}\text{Th}/^{234}\text{U}$ ages hardly vary with depth, which suggest that the samples come from a stalagmitic flowstone that drapes an older stalagmite. Both must have been deposited before the sediment that bears the artefacts. The ESR ages consistently increase with depth. Using the mean sedimentation rate, Layer 16 dates to ~ 74 ka (after Skinner *et al.*, 2007a).

D.

A.

B. ESR, TL, or OSL Accumulated Dose Growth with Time

B. T87, Layer H2
Bau de l'Aubesier

C. QT11, Layer H2 Bau de l'Aubesier

A.

- Multer & Hoffmeister 1961-1967
- Perkins 1977
- RH Harrison et al. 1984
- △ McNeill et al. 1996
- Toscano & Lundberg 1997

All other cores indicated
are from USGS drilled during
the 1990s

— cross sections
- - - subsea contours in meters

1 Lazy Beach
 2 Parking Lot Basin sites
 3 Railway sites

4 Big Snail Gully sites
 5 New Sites 1 & 2
 6 Wind Wadi

Marine Isotope Stages (MIS) 1-7

Sites

1. Lazy Beach	5. Railway 1	9. New Site 1	13. Parking Lot Basin NW Cliff
2. Johanna's Section	6. Railway 2	10. New Site 2	14. Matana 2
3. Snail Trail	7. Railway 3	11. Carpark 2	15. Matana 3
4. Big Snail Gully	8. Wind Wadi	12. Parking Lot Basin Mound	16. Bulaq

MDL Hill, Olduvai Gorge