



ADVANCES IN SURFACE LUMINESCENCE DATING: NEW DATA FROM SELECTED MONUMENTS

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ABSTRACT

In the present study, an attempt is being made to date samples from three archaeological sites in the Mediterranean using surface luminescence dating techniques. The methods are well established and this study is an effort to apply it to monuments that have not being dated with these methods before. Megalithic structures are eligible for absolute dating using OSL approaches in routine-based procedures. The structures that were chosen for dating are Osirion and Seti A' Temples in Abydos, Egypt, and a precipitate from a Saudi Arabian rock art site.

OSL ages obtained for the Saudi Arabia monument refer to geological episodes that ascribe at the Middle Bronze Age (middle 2nd mill. BC) while for the two Egyptian monuments, the results report for ages during the Middle Kingdom Age and of a younger date, spanning from the beginning till the end of 2nd mill. BC indicating original or later architectural phases.

KEYWORDS: optically stimulated luminescence, surface luminescence dating, Mediterranean, sandstone, megalithic monument

INTRODUCTION

Optically Stimulated Luminescence (OSL) is used to determine the time elapsed since certain minerals, such as quartz and feldspar, were last exposed to daylight. It is used to date geological sediments (Liritzis, 2000; Murray and Olley, 2002; Polymeris *et al.*, 2009) and has a plethora of applications in archaeology and anthropology (Galbraith *et al.*, 1999; Liritzis *et al.*, 2013a). OSL dating is based on the fact that those minerals preserve a record of irradiation dose received through time, by means of charge trapped at crystal defects, which is stable over long periods. When dating sediments, it is assumed that any previously trapped charge has been removed by exposure to daylight during transport and deposition; a process known as resetting, zeroing or bleaching. The age of the sediment is given by the following equation:

$$\text{AGE} = \text{equivalent dose (ED)} / \text{annual dose-rate (ADR)}$$

Where, the ED (Gy), measures the total exposure to radioactivity accumulated by the sample, and the 'dose-rate' is the (assumed constant) annual rate of exposure.

Most analyses now use so-called single aliquot methods (Duller 1995; Liritzis *et al.*, 1994; 1997; Murray *et al.*, 1997), where all the measurements needed to determine a dose are made on one subsample (aliquot) of the material to be dated. A single grain method may also be valuable in case of sparse mineral content and mixtures of grains containing different equivalent doses (Murray and Roberts, 1997).

The same rationale applies for the age determination of stone structures and artefacts by OSL, such as stone tools, monoliths, buildings, cairns, field walls etc. During construction of buildings or tools by using carved rocks, the surface of these rocks is exposed to light. Subsequently, soil, mortar or other rock covers this surface. Given the construction techniques used by ancient people, it is considered likely that many stone surfaces in archaeological monuments will have been exposed to sufficient daylight to bleach their geological luminescence to a near zero value, providing thus a reasonably accurate chronometer (Laskaris & Liritzis, 2011; Aitken, 1998;

Greilich *et al.*, 2002; 2005, Liritzis *et al.*, 2008). Resetting of the OSL signal to a depth of about 2 to 15 millimetres during light exposures from minutes to hours duration, makes it possible to provide a direct method for dating the time of construction for different prehistoric monuments comprising carved rocks of varied types ranging from granite to basalt and sandstone (Liritzis & Galloway, 1999; Habermann *et al.*, 2000; Greilich *et al.*, 2005; Vafiadou *et al.*, 2007; Sohbaty *et al.*, 2011). Recently, surface luminescence dating on several types of rocks, limestones, granites, sandstones, has been well documented, while recently several megalithic monuments and stone surfaces have been dated using OSL techniques. For an extended review on surface luminescence dating, the readers could refer to Liritzis (2011).

The present work is an attempt to apply surface luminescence dating to rock samples from two new archaeological sites. The first sample was collected from the petroglyph site Ain Jamal in the eastern Jabal Qara near Bir Hima, north of Najran, in southern Saudi Arabia (Fig. 1a). It is of sandstone covered by a re-precipitated calcite lamina that indicates the last wet conditions in the Arabian desert prior the onset of desertification. The rock art is clearly connected with a period of ample water availability. Large pools of calcite-rich water resulted in thick re-precipitated carbonate skins wherever sandstone was submerged by supersaturated solutions. Dating efforts were focused on surface grains of the coarse-grained sandstone, which experienced sunlight until they became concealed by the calcite lamina approximately 3 mm thick. For ancient walls made by limestone/marble the solar penetration can reach depths of 5–10 mm, a useful sampling depth for dating of face wall, provided that sampling is made properly during excavation avoiding exposure to sunlight. Otherwise, sampling from internal contacts between two overlaid blocks, solar penetration ensures complete zeroing only in the first 1–3 mm from surface. Incomplete bleaching from variable solar exposure may be determined applying the dose-plateau test. For granites, the complete bleaching of luminescence in top layers of rocks varies with the attenuation coefficient and light exposure time, and at any rate this depth seems to lie between 1 and 5 mm de-

pending from the particular rock opaqueness (Laskaris and Liritzis, 2011). The archaeological estimated age of most of the region's rock art encompasses the broad interval 1500–3000 BP. The last samples come from Osirion and Seti A' Temples at Abydos, Egypt (Fig. 1b, 1c, 1d). The

archaeological estimated ages span from XIX Dynasty (1295-1185 BC) to Middle Kingdom (XI to XIII Dynasties – ~2030 to 1640 BC) (Liritzis and Vafiadou, 2014). None of these monuments-samples was previously dated using luminescence techniques.

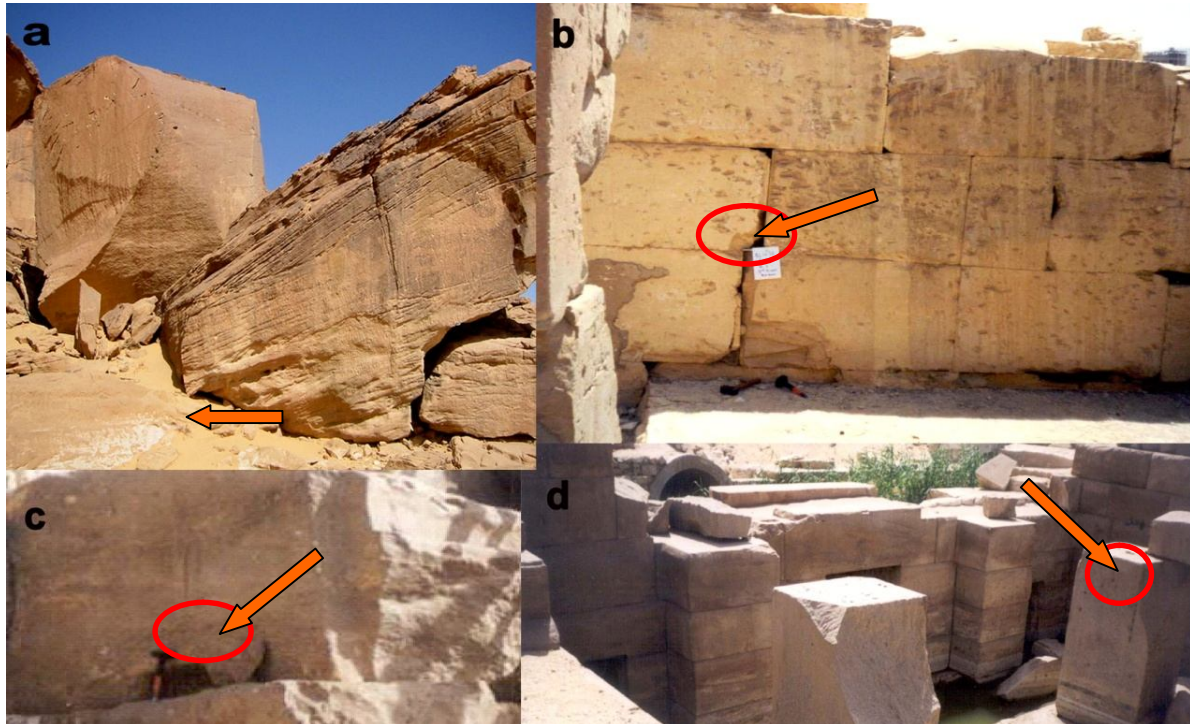


Figure 1: Sampling: (a) Sandstone complex from Saudi Arabia, covered with calcite lamina; (b) Seti Temple Upper room; (c) Osirion, sandstone wall; (d) Osirion, the pillars in the imaginary Osiris island. The sampling points are indicated with arrows.

SAMPLES AND SAMPLING PROCEDURES

The Abydos rock samples were removed from the megalithic monuments with the aid of a hammer and a chisel from the firm contacts between two blocks. The hits were gentle and efficient so that a small piece could be removed, without damaging the original block surface and the rest of the wall. The samples were mostly taken from the lower blocks of the monuments or from part of the wall with no indication of disturbance, and with care to avoid light exposure (Liritzis et al., 2010).

The outsides of the rock samples were washed with 10% HCl and two sets of H₂O₂ (5% and 10%) to remove carbonates and help remove organic material respectively. The grain extraction for ED measurements was achieved by gently rubbing the rock surface. Due to low amount of grains the OSL on polymineral ali-

quots was applied without any more grain separation treatment.

For the sample collected from Saudi Arabia, the calcitic cover (~3 mm) was gently removed with a knife to reveal the sandstone bedrock. The zero time for this sample (RHO-247) is the moment that re-precipitated calcite started to cover the sandstone surface, preventing further exposure to sun.

The samples that were chosen from the Egyptian temples for OSL measurements were of granite and sandstone (Liritzis et al., 2007). For the carved stones time starts from the moment of their placing to the wall and covered by other large carved cobbles. Table 1 provides a summary of the contextual information relevant to the dating of the samples, including the event that caused protection from further light exposure.

Table 1: Details for the samples chosen for OSL dating, such as code names, origin, description as well as composition.

CODE	Origin	Material	Description
RHO-247a	Ain Jamal, north of Najran, Saudi Arabia	Calcite	Precipitate from supersaturated lake on bedrock fragment
RHO-247b	Ain Jamal, north of Najran, Saudi Arabia	Sandstone	
RHO-138	Osirion (OS6)	Sandstone, ~100% quartz	Outer wall
RHO-139	Osirion (OS7)	Granite 40% qrt, 25% biotite and phlogopite, 25% albite, 5% microcline - K-feldspar and 5% amphibole	Pillars
RHO-1073	Seti's A' Temple, Abydos, Egypt	Sandstone	Lower blind room, eastern 2
RHO-1075	Seti's A' Temple, Abydos, Egypt	Sandstone	Lower blind room, north 3

EXPERIMENTAL PROCEDURES AND PROTOCOLS

Three different OSL readers were used. ED measurements on samples collected from Saudi Arabia were performed using a RISØ TL/OSL reader (model TL/OSL-DA-15) located at NCSR Demokritos, Athens. It is equipped with a high-power blue LED light source (470, FWHM 30 nm) delivering 30 mWcm⁻² at the sample position and a 6.41 Gy min⁻¹ ⁹⁰Sr/⁹⁰Y beta source. The samples from Osirion were measured using OSL with a RISØ TL/OSL reader system (also model TL/OSL-DA-15) located at RS ATHENA, Xanthi. It is equipped with a similar high-power blue-LED light source, delivering at the sample position approximately 36 mWcm⁻² and a 0.085 Gy s⁻¹ ⁹⁰Sr/⁹⁰Y beta ray source. In both cases, the readers are fitted with an EMI 9635QA PM Tube, while the detection optics consisted of a 7.5 mm Hoya U-340 optical filter transmitting at wavelengths of the range 260-390 nm (Bøtter-Jensen *et al.*, 2000). For every heating, a heating rate of 1°C s⁻¹ was used in order to avoid significant temperature lag.

The ED for samples RHO-138 and 139 was determined using a home-made OSL (Laboratory of Archaeometry, University of Edinburgh) system comprising of blue, green and IR diodes shining on the sample disk in the middle of the sample holder (Galloway, 1992; Galloway *et al.*, 1997). The samples were sieved and grains of sizes between 80 and 150 µm were chosen. Sample RHO-139 was measured using Single Aliquot Additive Dose protocol and infrared stimulation for 1 s (Galloway, 1996).

In all cases, aliquots were prepared with standard procedure. The single-aliquot regenerative-dose (SAR) protocol, introduced by Murray and Wintle (2000), was used for the rest of the samples (RHO-138, 247b, 1073 and 1075) in order to estimate the equivalent dose. The blue OSL signals were measured in the continuous wave OSL (CW-OSL) mode for 50 seconds at 125°C with the laser held at 90% power. The background OSL levels measured after 45-50 seconds exposure were subtracted from the initial luminescence intensity (0-1 seconds) of the decay curves obtained. Each disc was exposed to infrared radiation for 100 seconds at 125°C before of the blue stimulation, in order to reduce

the malign influence of feldspars grain to the signal. The procedure is similar to the double SAR procedure of Banerjee et al. (2001), containing additional SAR steps in order to minimize the need for chemical separation. The post-IR OSL signals resulting from polymineral grains are believed to be dominated by the quartz signal. The series of increasing regeneration doses delivered to each aliquot in order to obtain a growth curve for each one was varied between samples according to the expected ED. The regenerative sequence included a zero-dose check for the extent of recuperation (Aitken, 1998) and a repeat dose point in order to examine the adequacy of the test dose sensitivity-correction, which was achieved using a fixed test dose. In order to choose the proper preheat temperature, 20 aliquots of both samples RHO-1073 and RHO-1075 were divided into 5 groups of 4 aliquots each. Five different preheat temperatures, ranging from 130 to 260°C, were applied during the SAR procedure, one for each group.

Dose rates were determined from the concentrations of natural U, ^{232}Th and ^{40}K . These were determined by using a Low Level alpha counter type 7286, and Scanning Electron Microscopy (SEM). For the former uranium-235 (and consequently U-238) and thorium-232 were measured by alpha counting employing the pairs tech-

nique assuming U-equilibrium. The alpha counter is a 7286 Low Level Alpha Counter, Littlemore Sci. Eng Co Oxford with an EMI 6097B tube. Measurements were calculated by two similar counting systems calibrated in standards following devised conversion factors as well as relevant computations (Liritzis and Vafiadou, 2012).

The SEM was coupled with energy dispersive spectrometry (EDS) analysis (Philips FEI-Quanta INSPECT with SUTW detector and coupled with EDS PV7760) for ^{40}K (Liritzis et al., 2011b). Analyses were performed in high voltage of 25 keV with 35° take-off angle. Quantitative analysis was performed by using software EDS-Genesis with errors made via ZAF correction. Detection limits are in the range of 100 – 300 ppm while most reliable measurements are those for which the yielded concentration is >0.1%wt. Besides providing an estimate of bulk ^{40}K concentration, SEM-EDS analysis provided information regarding its topographical distribution within the scanned areas; in all samples ^{40}K was found relatively homogenous within the sample matrix. For samples RHO-138 and 139, ^{40}K content was measured by X-Ray Fluorescence (XRF) using a TN-Spectrace 9000 analyzer by Thermo, equipped with an HgI₂ detector (Liritzis and Zacharias, 2011).

Table 2: Details for the chemical content, the calculated ED, annual dose rate and age of the samples. For all samples U and Th were determined using alpha counting while for K use of SEM was made samples RHO247a, b, RHO-1073,1075 and use of XRF for the samples RHO-138, 139.

Sample	U (ppm)	Th (ppm)	K (%)	Water uptake (%)	ED (Gy)	ADR (Gy ka ⁻¹)	AGE (y BC)
RHO-247a	1.11±0.08	1.39±0.25	0.87	20±10	4.9±0.3 (2 aliquots, SAR, Blue, quartz)	1.49±0.045	1630±250
RHO-247b	0.67±0.07	1.12±0.22	2.08				
RHO-138	0.66±0.05	0.96±0.15	0.012	0	1.78±0.16 (3 aliquots, SAR, Blue, quartz)	0.57±0.08	1300±570
RHO-139	2.18±0.31	8.27±0.97	2.89	0	10.75±1.34 (9 aliquots, SAAD, IR, feldspar)	2.70±0.07	1980±110
RHO-1073	0.88±0.1	1.13±0.12	0.079	0	33.56±0.55 (3 aliquots, SAR, Blue, quartz)	-	-
RHO-1075					1.08±0.09 (3 aliquots, SAR, Blue, quartz)	0.35±0.035	1070±400

All radioisotope geochemistry data were transformed to dose rates using the conversion factors of the samples using Liritzis et al.

(2013b). The isotopic and age data are presented in Table 2.

METHODOLOGICAL TESTS - RESULTS

Osirion and Seti's A' Temple

XRD analysis on the several samples from Seti's A' temple the blocks from the upper blind room consist of ~86% limestone, 7% ankerite, 7% montmorillonite and traces of quartz and halite; from the upper corridor of the Seti A' Temple they consist of ~88% quartz, 10% kaolinite and 2% (K-fels). The surrounding wall is sandstone (~90% quartz) (Fig. 1c) and the pillars in the imaginary Osiris island are granitic Fig. 1d).

The results for the preheat plateau tests for samples RHO-1073 and RHO-1075 are presented in Figs 3–5. The dependence of the equivalent dose (A), the recuperation (B) and the recycling ratio (C) on the varying preheat temperature is presented in Fig. 2 for the case of RHO-1075 and in Fig. 4 for the case of RHO-1073. In both cases, equivalent doses form a plateau; nevertheless, this plateau is formed for low preheat temperatures up to 200°C in the former case, while for higher temperatures there is a substantial decrease of the measured ED, along with a simultaneous increase in its dispersion. Recuperation and recycling ratio tests are also satisfactory for the preheat plateau region 130–190°C.

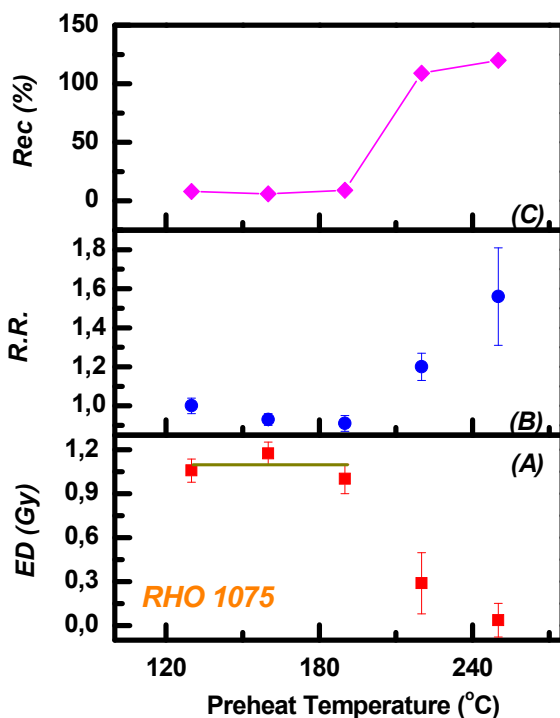


Figure 2: Seti Temple. Preheat plateau test for the sample RHO 1075 (Lower Blind Room northern wall), indi-

cating the ED (plot A), the recycling ratio (plot B) and the recuperation value (plot C) versus preheating temperatures. Five different preheat temperatures were applied. The equivalent dose for the sample was estimated as the mean value (1.08 ± 0.09 Gy) of the plateau region for preheat 130–190°C, horizontal line in plot A.

Typical OSL decay curves for the natural signal are shown in Fig. 3, along with the three regenerative doses (R.D), (1st, 2nd and 3rd R.D. respectively), the repeat dose point (R.R.) and the recuperation afterwards (Rec), for the first 20 s of stimulation. In the inset diagram the SAR growth curve from an aliquot is presented from the sample RHO 1075 after preheating at 190°C. Equivalent dose value is provided by interpolation of the natural normalized OSL signal onto the growth curve resulting from the fit to the results of the measurement sequence. Filled star represents the recycle point value; the SAR ED value yielded (arrow) is 1.06 Gy.

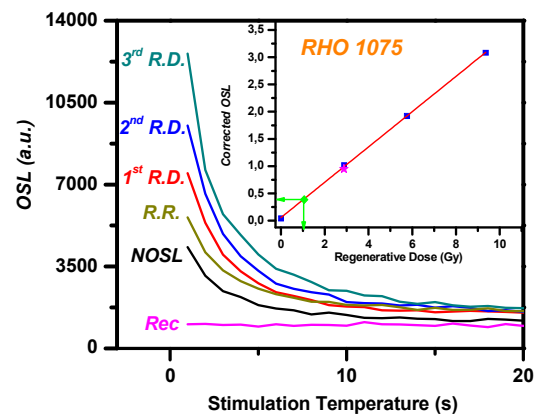


Figure 3: Seti Temple: Typical OSL decay curves for the natural signal (NOSL), the three incremental regenerative doses, (1st, 2nd and 3rd R.D. respectively), the repeat dose point (R.R.) and the recuperation afterwards (Rec), for the first 20 s of stimulation. Inset: SAR growth curve, measured for an aliquot from the sample RHO 1075 after preheating at 190°C. Equivalent dose value is provided by interpolation of the natural normalised OSL signal (filled diamond) onto the growth curve (line) resulting from the fit to the results of the measurement sequence (filled squares). Filled star represents the recycle point value; the SAR ED value yielded (arrow) is 1.06 Gy.

A similar approach for the case of RHO-1073 sample indicated different results, namely different proper preheat plateau temperature region (200–260°C), recycling ratio values with large errors as well as large ED values (Fig. 3). In

both cases the equivalent dose for each sample was estimated as the mean value of the ED values corresponding to the specific preheat plateau region. The results for the sample collected from the eastern wall of the lower blind room indicated a geological dose.

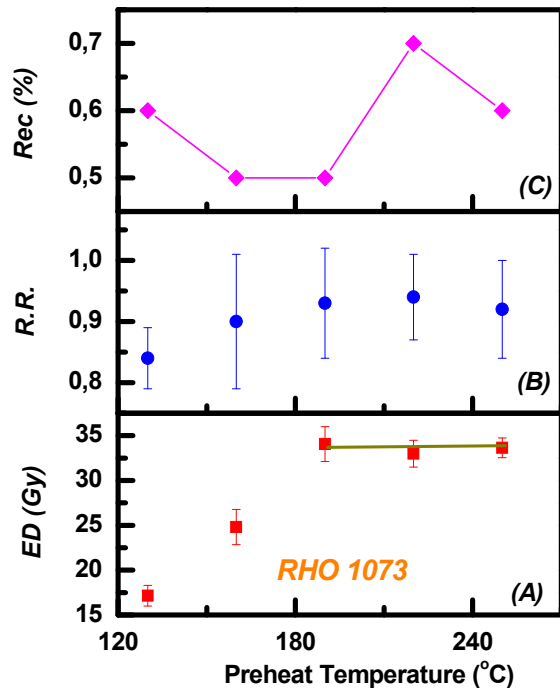


Figure 4: Preheat plateau test for the sample RHO 1073 (Lower Blind Room eastern, sandstone), indicating the ED (plot A), the recycling ratio (plot B) and the recuperation value (plot C) versus preheating temperatures.

The same five different preheat temperatures were applied. The equivalent dose for the sample was estimated as the mean value ($33,56 \pm 0,55$ Gy) of the plateau region (190 – 250°C, horizontal line in plot A).

This implies geological dose.

For Osirion, the gamma dose rate from the surrounding sand was measured using Geiger-Muller detector. For sample RHO-138 the original ED was $1,96 \pm 0,16$ Gy (Fig. 5) and the ED from the surrounding sand during burial period of the 1400 years $0,182 \pm 0,10$ Gy.

The final ED is $1,78 \pm 0,16$ Gy and the estimated age of the sample 1300 ± 570 BC. For sample RHO-139 the original ED was $11,24 \pm 1,34$ Gy, the ED during the burial period $0,49 \pm 0,02$ Gy, the final ED is $10,75 \pm 1,34$ Gy and the estimated age of the sample 1980 ± 110 BC. Fig. 6 shows an ED estimation of an aliquot for sample RHO-139 (more in Liritzis & Vafiadou 2014, forthcoming).

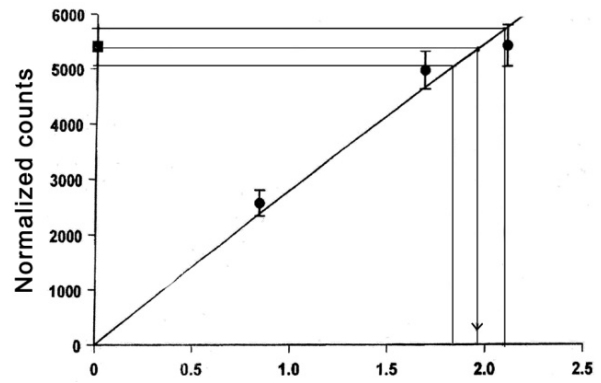


Figure 5: ED estimation for sample RHO-138 (Osirion).

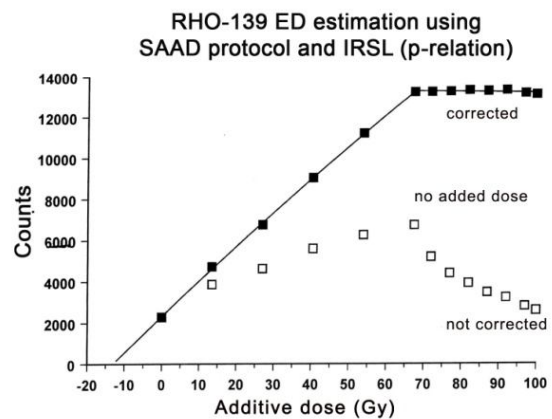


Figure 6: ED estimation of an aliquot for sample RHO-139. The aliquot has been irradiated with IR for 1s after preheating at 220°C for 300s. ED= $13,2 \pm 0,9$ Gy (p-relation).

Saudi Arabia

Several aliquots were measured from this sample. The preheat applied was 220°C. Four aliquots provided ED values above 40 Gy which represent geological luminescence dose from material sampled from deeper layer of the sandstone. Two more aliquots gave EDs of 5.50 and 4.3 Gy. For the age estimation of the sample the average of these latter values was used, i.e. $4,9 \pm 0,3$ Gy. The age from the interface between the sandstone and the calcite encrustation was 1630 ± 250 BC (Table 2). Tests on recuperation, sensitivity changes and repeatability were satisfactory. The age indicates the last time the bedrock surface was exposed to daylight, at the commencement of the deposition of the calcite accretion. This carbonate was precipitated from a lake then existing where today there is only sand desert, and the aquifer, generally of Pleis-

tocene water dated by ^{14}C , can be up to 70 m below the plain (Edgell, 2006).

The sample was collected about 1 m above the present plain, immediately below the rock art site. Although it does not directly refer to the numerous petroglyphs, most Arabian rock art concentrations seem to relate to periods of significantly wetter conditions than today. The implication is that the Ain Jamal petroglyphs, and the numerous faunal depictions in the region, should refer to the most recent wet period. The OSL date indicates a reasonable sequential event and thus conforms well with the only direct date for Jabal Qara rock art, from the nearby site Ta'ar. This is a microerosion date of E2109+250/-540 years (prefix E for erosion, stands for the microerosion relative dates of poor precision but very high reliability, Bednarik, 1992) secured from an anthropomorph and calibrated against an inscription of known age at Ain Jamal (Bednarik and Khan, 2005). Given the residual geological signals observed in the other aliquots from this sample, new date could be an overestimate, but it suggests that the last wet phase in the interior of the Arabian Peninsula commenced about 3600 years ago and began to diminish one and a half millennia later, with rapidly falling aquifer levels ushering in precipitous desertification between 500 BC and AD 500.

DISCUSSION

For the sample from the northern wall of Seti A' the estimated age was 1070±400 BC. The archaeological age estimation is Middle Kingdom (XI to XIII Dynasties, ~2030 to 1640 BC), the lower age implies possibly later re-use.

For Osirion samples, RHO-138 and 139, we have to take under consideration the fact that the base of the structure was covered by sand for 1400 years. Thus a dose must be subtracted from the calculated one. Thus the age of both samples are compatible with the archaeological age attribution of XIX Dynasty (1295–1185 BC) and the Middle Kingdom (1800 BC) respectively (Kemp, 1968; Brand, 2000; Mariette, 1869).

Regarding the Saudi Arabia sample the OSL date and the microerosion date refer to two different events. The first refers to the commencement of the carbonate deposition from a lake.

Although the date is based on 2 aliquots the consistent values obviates the concern of having geological signals. The second refers most probably to the end of the wet phase, because most rock art in deserts dates from times of environmental stress, when people sought to influence nature through supplication. Also, human conflict with swords is depicted in the dated image, and we know from the frequent depiction of camels of identical patination that desertification had already set in. Therefore the two dates are mutually complementary: one marks the beginning of the last pluvial, the other very probably its end.

In all, the surface dating methodology is comparable to independent contextual dating evidence and this reinforces its wide use and applicability. The surface sampling produces, in most cases of small piece acquisition, a small quantity and thus a low number of aliquots. Care should be exercised in removing surface layers and the determination of lower completely bleached depth. This is a recommended procedure, time consuming, but worth checking to differentiate between archaeological and geological luminescence ages. The need for dose reconstruction i.e. at Osirion is a parameter that must be considered in similar desert environments. Finally, the contextualization of the Saudi results are important but cautiously considered and further work is underway reconfirming the presently obtained OSL age.

CONCLUSIONS

In the present study, new surface luminescence dating data were presented for three selected, previously undated monuments. For the sample collected from Saudi Arabia the moment that re-precipitated calcite started to cover the sandstone surface disclosing further exposure to sun is cautiously dated in the middle of 2nd mill. BC. For the Osirion samples, both ages indicate a rather lower than Middle Kingdom Age. This result probably implies later use or occupational phases.

Megalithic structures are eligible for absolute dating using OSL approaches in routine-based procedures. In many cases the geological dose of the materials was revealed, indicating that the

collection procedure remains the most critical step for successful dating of building materials. Nevertheless, following present observations and past investigations, the seemingly complex topic can be a straightforward procedure, much like with other archaeomaterials and geological settings. The combined use of at least two, supplementary, OSL measurement protocols are favorable to provide cross-checking – an inter-calibration practice – of the ED values.

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