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OPTICALLY STIMULATED LUMINESCENCE DATING USING QUARTZ: REMARKS AND A PLEA FOR FAIRNESS

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ABSTRACT

The optical stimulated luminescence (OSL) dating is a steadily developed method in the fields of archaeology, geoarchaeology and earth sciences. The trapped electrons in lattice defects of suitable minerals during irradiation by natural radioisotopes throughout the time and the emitted luminescent light after agitation by optical radiation in the lab (or by daylight), are physical processes which determine the total equivalent (to a laboratory dose) dose (in Grays). The intentional or accidental light exposure to light and the thermal agitation sets luminescence clock to zero for a mineral within a material to be dated. Excellent publications (either articles or books) and reviews on the OSL dating have been made. The objective coverage of essential novel applications and basic research accompanied by an ethic unbiased pace is a prerequisite for scientific integrity of every publication. A recent Nature Reviews Method Primers on OSL dating of quartz was the stimulus for the present constructive and supplementary information and serves also as a plea for fairness.

KEYWORDS: OSL, luminescence, archaeology, loess, monuments, dunes, sediments, fluvial, impact, dose, dose rate, disequilibrium, partial bleaching, SAR, SAAD, exposure, errors, monuments, quartz.

1. INTRODUCTION

It is indubitable the rapid progress in OSL dating and the world publications per annum; hence, strenuous to update the advancement. The recent *Nature Reviews Method Primers* review article on OSL dating using quartz was intended at users of luminescence, and at students and academics intending to implement the method in their work (Murray et al., 2021).

Though any review is most welcome, yet, the selective bias reports of basic functions, techniques uncertainties as well as misses of important multi-referenced articles and special methodologies of the OSL renders the value of this at any rate worthy review oftentimes as deficit and unfair. A review is entitled to refer with fairness to the basics, the pioneering work and subsequent major refinements and applications, notwithstanding mention and credit should be given to major research centers web sites which upload their publications. This way the objectivity is at most achieved.

The chosen aspects of OSL dating of quartz by this Primer cover reviews and respected encyclopedia entries on the touched luminescence dating versions and materials which have already been published in the last 10 years but not mentioned (Urbanova et al., 2020; Smedley & Wintle, 2018; Zhang et al., 2015; Wallinga and Cunningham 2015; Wallinga 2002; Wintle and Adamiec 2017; Liritzis et al., 2013a; Liritzis 2011; Preusser et al., 2008).

A series of misfortunate missing of important literature in the OSL dating of quartz as well as of selective aspects of the wide applications, specialized development and current trends of the reported subject ultimately require a supplementary attention for the benefit of young researchers, but academia too.

It is not uncommon for multi-cited authors in literature to read articles without describing equivalent dose determination or dose rate evaluation in detail, as it is considered for granted such attention from a renowned authorship. As a result, no reproducibility is possible; while statistical processing and meta-analyses and assumptions made are presented as panacea, yet a false impression, without discussing practical errors involved for all assumptions.

A good practice in every measurement (attached with a due error at 65% and 95% probability, as with C-14) has been seen in various publications but not in all. The inculcation in several limited information data lies always with the chosen reviewer and the well-informed editor. Criteria of recuperation, dose recovery, fading, recycling, sensitivity change must be provided, as well as characteristic signal curves for fast and other components, dose protocol curves for each sample and not representative ones except if they are similar. In particular, the dose rate must be

analytically presented for each radiation component in the proper context, which is usually complex and moreover takes into consideration fallout effects (Liritzis 1987). The complex dosimetry in building up the radiation dose through centuries and millennia is lacking in most publications.

Overall, in the conventional age equation (1)

$$\text{Age} = D_e / d_r \quad (1)$$

where D_e (Grays) is the total equivalent dose from U, Th, K, Rb and alpha, beta and gamma radiation including cosmic rays, and d_r the annual dose rate (mGy/yr), it must be stressed that: a) the U-disequilibrium is not considered properly, and, b) the 5 to (overoptimistically) 10% error has not been improved since its initial applications (first as TL and later as OSL) during 70-80s and its reliable extension to older ages not yet routinely achieved (e.g. for single grain see, Anechitei-Deacu et al., 2018).

Last, but not least in the Murray et al., (2021) review, out of the reported 395 citations in the main article about 140 (35%) and in the supplement 36%, belong to the authors, which appears the subject has been covered by World half a dozen scientists. This is not true and beyond the present appeal for fairness any reader can locate original works on TL/OSL about the basics, the involved equations, the first attempts the applied materials.

Some issues which need attention by readers of the SCIENTIFIC CULTURE and the Nature Reviews Method Primers on the 2021 review article are the following.

2. REGARDING BLEACHED LUMINESCENCE

That OSL is more readily reset by daylight exposure while thermoluminescence bleaches much more slowly and is never completely reset is not explored sufficiently. A major detailed experimental work, not reported, on cautionary remarks on the bleaching, dose growth, phototransfer and preheating in various types of sedimentary and volcanic rock types for OSL dating, has already been published (Faershtein et al., 2019, 2020; Liritzis et al., 2013a; Liritzis et al., 2008).

3. QUARTZ FROM LIMESTONE

The introduction of a new quartz OSL dating of limestone is often overlooked. A novel technique has shown that quartz (and feldspar as well) separation is possible and the expected single aliquot OSL ages can be more accurate than TL of limestone. In fact, single aliquot by blue OSL was applied and indicative ages were given. This pilot study indicated safely an overcome of problems with the luminescence dating of limestone buildings, which are associated either with great uncertainties in TL or inability to getting an OSL

signal and encourages applications of surface dating (Liritzis et al., 2010b).

4. TOTAL/PARTIAL BLEACHING

The ubiquitous assertion of the potential to date almost any surficial deposit (fluvial, marine, alluvial, archaeological floor) during daylight exposure of sediment grains during the sedimentary cycle of erosion, transport and deposition is poorly explored due to the uncertainties involved from a variable partial to total bleaching (Smedley et al, 2019); in contrast to loess an extremely well-studied aeolian origin material.

5. PORTABLE LUMINESCENCE READER

The usefulness of portable luminescence reader in situ measurements of sedimentary profiles is missing to report a most updated review on the subject, in particular the basic principles, constraints and case studies, which elucidate cryptostratigraphic variations in sedimentary sequences for geomorphological applications and future directions (Munyikwa et al., 2020).

6. STONE SURFACE LUMINESCENCE DATING

The issue of daylight exposure of rock surfaces (of quartz bearing rocks or quartz traces extracted from limestones) which includes rock art (petroglyphs) and surface luminescence dating of monuments is an increasingly developed field and the principle of dating progressed in two phases. This is insufficiently covered by the Primer review, but three reminiscence reports of 1997-2012. **First**, regarding the total bleaching of surficial luminescence in masonry (rock types including quartz, and other minerals) (Fig.1), an initiation of this novelty on ancient stone surfaces ushered first in 1994 (Liritzis 1994), then reviewed in 2011 (Liritzis 2011) and successfully applied to various case studies (Liritzis et al., 2010a, 2014, 2015; 2018d). A **second phase** concerns the inverse problem of dating the length of daylight exposure of a rock surface i.e., the bleaching of stone surfaces by daylight as a function of time exposure and depth, for various rock types, in rock art and in constrained OSL chronology, are omitted (Liritzis et al., 2013b, 2015; 2017, 2018; Laskaris and Liritzis 2011; Bednarik 2021).

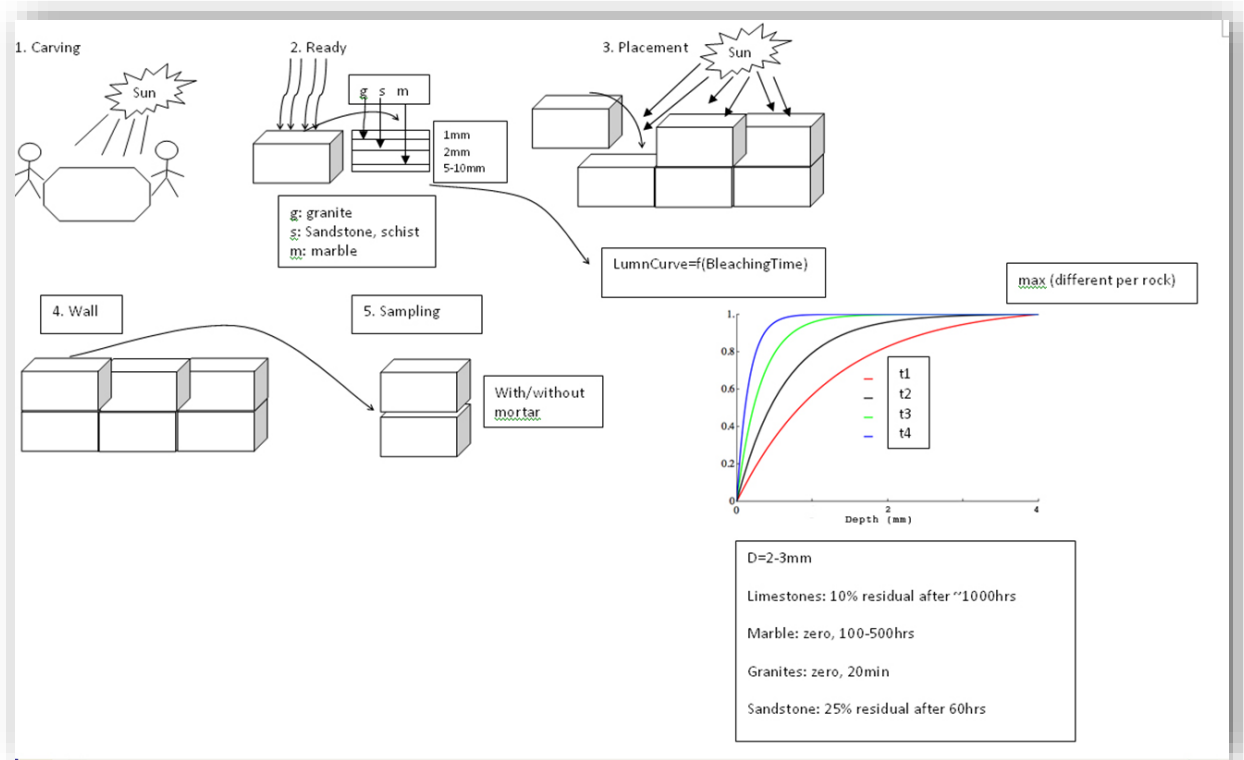


Figure 1. Schematic diagram of carving stone blocks (1), exposed to daylight (2) with different penetration for granite, sandstone and marble/limestone, until placed on the wall (3,4), and sampling between two firmly overlies blocks (5). Removed surface powder in depths of a few millimeters and bleached luminescence as a function of sun exposure time (t1-t4) and depth below surface are shown at the right plot of colored curves. NB: The granite with quartz, feldspar and biotite (e.g. in Mycerinus) bleaches slower than granite with its two-grain phases, mainly feldspar with little quartz and biotite (e.g. in Osirion). (see sampling video: https://www.youtube.com/watch?v=ADaMcaJAnz0&list=PLIwkBdcSy-yiTVqi_xD2kL1RqT6DqNz9g8&index=1, in 13 parts) (also Liritzis & Vafiadou, 2014).

The method of stone surface dating in monuments was first tested with simulated data in the laboratory, then with known-age ancient structures (5th c BC Apollo Temple in Delphi, 11th c BC Mycenae wall, 6th c BC Efpalinion trench in Samos, pyramidal and other masonries; see Liritzis 1994; Liritzis & Vafiadou 2005, 2014) (Fig.2). Applications on different rock types with quartz include limestones (OSL of calcite or traces of quartz therein), granites, basalts, sandstones in Greek and Egyptian monuments (Liritzis et al., 2008; Liritzis & Vafiadou, 2014; Liritzis et al., 2016, 2010a).

Some characteristic snapshots of masonry and sampling is provided in the **APPENDIX**.

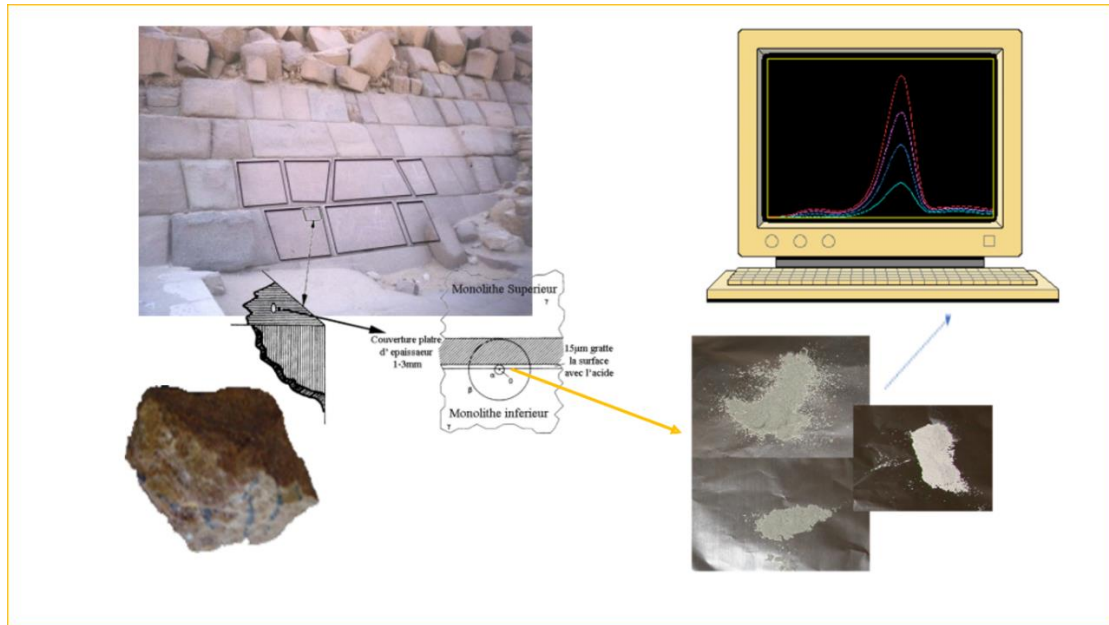
6.1 OSL/TL of calcites

The issue of luminescence dating of calcites presumes amongst others the dose growth of TL and OSL. In luminescence dating of calcites (marble, limestone) it has been documented the use of TL and accordingly for OSL (Ugumori and Ikeya, 1980; Liritzis 1994; Liritzis & Vafiadou, 2014; Theocaris et al., 1997). Further OSL of calcites has been reported. Thus, for example, luminescence around 515 nm wavelength (2.41 eV) from limestone stimulated by pulsed light of 370 nm wavelength (3.36 eV) is found to decrease with increasing radiation dose.

Liritzis (1994) proposed a method for dating the construction of megalithic limestone buildings, based on the latent thermoluminescence of the surface of a limestone building block being bleached by exposure to light prior to incorporation in the building and then, in the inter-block surfaces from which light is excluded, growing again with the passage of time; in a manner akin to the well-known methods of dating sediment deposition using quartz or feldspar extracts (for example Wintle and Huntley, 1980). The method has since given an age for the Temple of Apollo in Delphi consistent with the historical age (Liritzis et al., 1997a delph), and has been applied to determine the age of two Greek pyramidals (Theocaris et al., 1997). Liritzis and Bakopoulos (1997a) observed the decrease in the thermoluminescence peak at 280°C with exposure to sunlight for several samples of Greek limestone. However, a substantial residual signal was found after 100 hours of exposure. Just as the use of optically stimulated luminescence rather than

thermoluminescence is advantageous with quartz or feldspar when dating sediments (e.g., Huntley et al., 1985), the same advantage, namely the absence of residual signal from bleached material, could be hoped for if optically stimulated luminescence could be used with limestone. This bleaching and residual luminescence has been treated with different rock types by TL and OSL (Liritzis et al., 2008) and to circumvent limestone slow bleaching as well as over masking signals of quartz and CaCO₃, Liritzis et al., (2010b), introduced the quartz extraction from the limestone powder removed from surface stone blocks (see below).

Wintle (1997), in a review of luminescence dating procedures, drew attention to the report by Ugumori and Ikeya (1980) of the optical stimulation of luminescence from CaCO₃ and noted that no further work on the topic had been reported. Ugumori and Ikeya (1980) observed luminescence (a broad band around 430 nm, 2.9 eV) stimulated by light from a N₂ laser (337 nm, 3.68 eV) from natural calcite, both crystalline and a piece of stalactite. The potential for archaeological dating was illustrated by an increase in luminescence intensity with increasing distance from the surface into the stalactite. Exposure to the laser light altered the thermoluminescence glow curve, reducing the peak at 347°C, increasing the peaks at 287°C and 237°C, and creating a peak at 57°C. The work reported by Galloway (2002) was developed independently from the study of the bleaching and phototransfer properties of the 286°C peak in the thermoluminescence glow curve from limestone (Bruce et al., 1999). This is the dominant peak in the thermoluminescence glow curve from limestone and the peak used for dating megalithic buildings (Liritzis, 1994; Theocaris et al., 1997). However, Bruce et al., (1999) found that the bleaching of the 286°C peak by light in the wavelength range 350 – 600 nm was more rapid for shorter wavelengths of light, 350 – 400 nm being most effective and wavelengths longer than 500 nm having little effect. Galloway (2002) using ultraviolet light a Nichia LED type NSHU590Ea light emitting diode (LED) with peak emission at 370 nm (3.36 eV) as stimulating light source, concluded that limestone shows useful optically stimulated luminescence and there is an indication of a way forward.



(A)

(B)



(C)

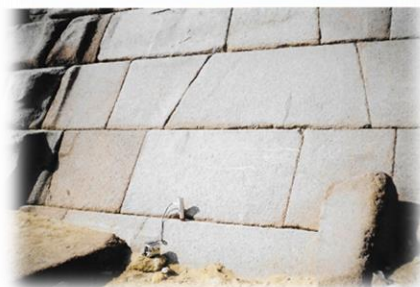


Figure 2. A Upper) Sampling from the east face of Mycerinus red granite casing stones. B) Lower: the Mycerinus pyramid eastern side with granitic casing at the lower rows, C) right: the context of Mycerinus and Chephren pyramids with detail of the granitic casing and the gamma-ray Geiger meter and a detail in between the two adjacent in contact stone blocks. Although most of the surviving casing stones of this pyramid are rough-hewn on their outer surfaces portions of the faces are smoothed as may be seen in photo (B). Sampling in (A) was made from a smoothed portion as it is easier to get at the join between the blocks when the outer surfaces are smoothed. The Pyramid of Menkaure on the Giza Plateau, Cairo, Egypt, is the smallest of the three main Pyramids of Giza. It is thought to have been built to serve as the tomb of the fourth dynasty Egyptian Pharaoh Menkaure. approx. 2500 BC. The OSL age was found equal to 3450 ± 950 BC by Blue OSL, SAAD, inclusion dating (Liritzis and Vafiadou, 2014).

7. U-DISEQUILIBRIUM EFFECTS ON DOSE RATE AND VARIABLE GAMMA-RAY DOSE-RATES IN SPELEOTHEMS

Gamma rays dose rate versus sediment cover introduces two uncertainties, a) the variable and unknown sedimentation rate of covered sediments, and, b) the U-disequilibrium in sediments. The errors involved have been earlier discussed (Liritzis & Galloway 1980; 1981; Liritzis 1989a; Danali-Cotsakis and Liritzis, 1985) but are absent from the Review by Murray et al., (2021).

7.1 U-disequilibrium on Age

The state of equilibrium of ^{238}U decay series can be a serious source of error in dose rate evaluation *in lieu* of their assertion that in most cases, disequilibria have rather small effects on the OSL age. In fact, in chronological studies of dating of speleothems, which consist a considerable part of quaternary geoarchaeological dating, with associated human implements the TL/OSL/ESR and U-disequilibrium series require another age model where total dose D_{tot} is a function of time.

In earlier studies (Liritzis 1989a), for the OSL/TL of calcites in speleothems (travertines, stalactites, stalagmites, flowstones), the calcite has been considered in two cases: a) *pure*, and b) *contaminated by partial detrital sediment* washed in during their formation. Thus, a methodology has been introduced, where the total dose D_{tot} in the luminescence dating equation consists of four components corresponded to the U-238, Th-232, Th-230 and Ra-226 decays (eq.2):

$$D_{\text{dis,tot}} = A*\Delta_1 + B*\Delta_2 + \Gamma*\Delta_3 + \Delta*\Delta_4 \quad (2)$$

where $A = r*\lambda*t$; B, Γ , Δ the activities of the four decay isotopes:

$^{238}\text{U} \rightarrow ^{206}\text{Pb}$, $^{234}\text{U} \rightarrow ^{206}\text{Pb}$, $^{230}\text{Th} \rightarrow ^{206}\text{Pb}$ and $^{226}\text{Ra} \rightarrow ^{206}\text{Pb}$

The Δ_{1-4} coefficients (given in Tables, Liritzis 1989a) represent the effective doses for α , β , γ -radiation components due to these four decays, which include the present and initial concentrations respectively of these isotopes in atoms/g. The total D then from U, Th, K, cosmic rays become:

$$D_{\text{dis,tot}} = (D_{U,\text{sed}} + D_{U,\text{detr}} + D_{U,\text{Calc}}) + D_{\text{Th+K(sed+calc+detr)}} + D_c = f(t) \quad (3)$$

D_U , D_{Th} are the doses of alpha, beta, gamma radiation from the U & Th-disequilibrium series for sediment, calcite and detrital calcite, D_K from potassium and D_c cosmic radiation (Note that for pure calcite Ra-226 and Th-230 concentrations are almost zero).

Serious age deviations are noted when age equation (1) is compared with the conventional age equation (3), due to wrong evaluation of dose-rates.

The D_{tot} for calcite is measured by ESR/OSL/TL; and the isotopic activity (U, Th) and other radionuclidic U-components of eq.2,3 by alpha spectrometry and potassium by conventional techniques (Liritzis et al., 2011). For "partially contaminated calcites" the total luminescence dose from the detrital phase should be added to that of "pure calcite" (as long as uniformly distributed U-compounds in a stratigraphic growth horizon is the case, neglecting any "zoning effect").

A theoretical simulation has been made assuming different ages of 50, 100, 200 and 400 Kyr as "actual" ages (Kyr=1000 years). That is, D_{tot} of eq.3 is calculated (which is in practice measured in lab by TL/OSL/ESR) for respective assumed ages [Example: for D_c of 0.3mGy/yr x50Kyr gives $D_c = 15$ Gy and so on for the other components].

From eq.1 the "conventional" age equation the obtained age is for annual dose-rate based on present day activity ratios of surrounded sediments and the speleothem itself for, a) U-equilibrium, b) U-disequilibrium in sediments. Figs 3, 4 prove the deviation of the "actual" and "conventionally" obtained ages for such dose-rates in (a) and (b).

The liability of the proposed model is that no mobilization (through recrystallization) of isotopes that determine disequilibrium is assumed, thus conventional eq.1 must be used with due care.

The U-Disequilibrium in cave sediments and in the incorporated minerals on calcite lead to ages by eq.1 considerably different from the expected from disequilibrium formula of eq. 3.

The next remarks are made:

- the present dose-rate values for U-equilibrium is closer to the expected dashed line up to 300 Kyr.*
- the age deviations for disequilibrium for "pure calcite" (+) are around 40% lower and for "partially contaminated calcite" (●) ~30% lower for the age span 50-300 Kyr.*
- the "pure calcite" (+) approaches expected age for $T \geq 400$ Kyr. Hence, for some cases of U-series disequilibrium in cave sediments the eq.1 can be used safely giving accurate ages with eq.3.*

Therefore, OSL/TL/ESR of speleothems must be delt with caution regarding use of iso-TL/OSL/ESR methods of eq. 3 instead of the conventional eq. 1.

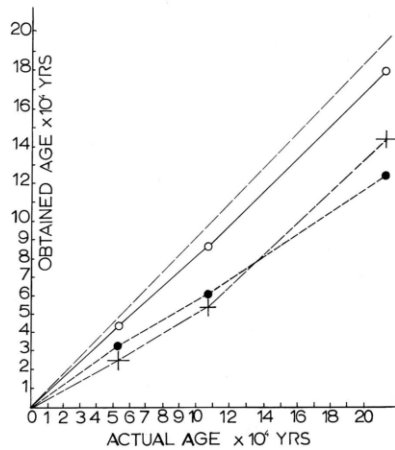


Figure 3: Actual ages (eq.3) versus obtained conventionally by eq.1. The symbols are: (+) U_{diseq} for pure calcite and (*) U_{diseq} for partially contaminated calcite; (o) are for equilibrium in surrounding infinite matrix cave sediments.

Simulated ratios of disequilibrium state of sediments: $^{234}U/^{238}U=0.8$, $^{230}Th/^{234}U=0.8$, $^{226}Ra/^{230}Th=1.5$. For pure calcite $U_{pure}=1$ ppm; for partial contaminated calcites: $^{238}U_{cont}=0.06$ ppm, $^{232}Th=0.2$ ppm, $K_2O=0.1\%$; isotopes for sediments: $^{234}U=6$ ppm, $^{232}Th=17$ ppm, $K_2O=2\%$, a-factor=0.5.

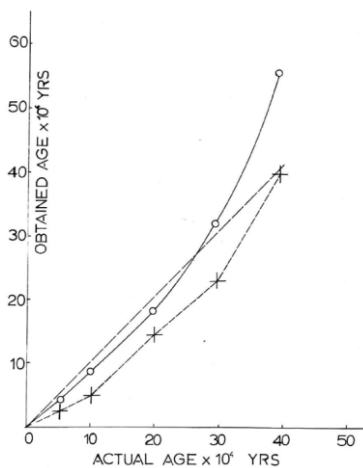


Figure 4: Actual ages (eq.3) versus obtained conventionally (by eq.1) for pure calcite; symbols (+) are for U_{diseq} in sediments calculated dose rates, and (o) equilibrium in surrounding infinite matrix cave sediments. Extension of Fig 3 to 400K yrs.

This way the proposed dating method overcomes several difficulties associated with the precise and accurate determination of the age of speleothems related to geoarchaeological, paleoanthropological investigations (Liritzis 1989).

Actual ages (eq.3) versus obtained conventionally (eq.1) are shown in Fig.4.

7.2 Gamma ray dose rates evaluation in calcitic slabs

Environmental gamma ray dose rates are important in low internal U, Th content of calcites. The

gamma-ray dose-rates from surrounding sediment is usually a small percentage of the total dose received by calcites. But for ground travertines and stalagmites with $< 1\pi$ counting geometry (environmental γ -ray), the gamma-ray contribution is even lower in the total D received by calcite, due to attenuation through these deposits.

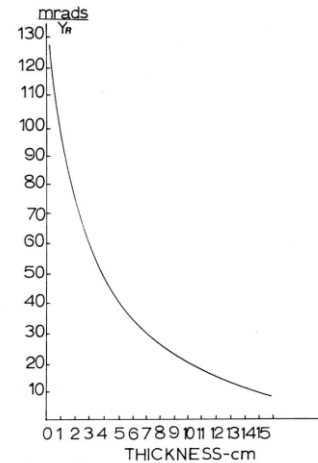


Figure 5. Gamma ray dose rate attenuation through an infinite half-space of calcite in contact with an infinite and homogeneous radioactive layer of sediment. Simulated assumed values for sediment: $U=6.2$ ppm, $Th=17.4$ ppm, $K=2\%$, $SiO_2=70\%$. Backscatter contribution is taken into account for 12-13% for K-40 gamma radiation, 15% for U and 13-14% for Th for $\rho_{calc}=2.71$ g/cm³. (Liritzis, 1989a).

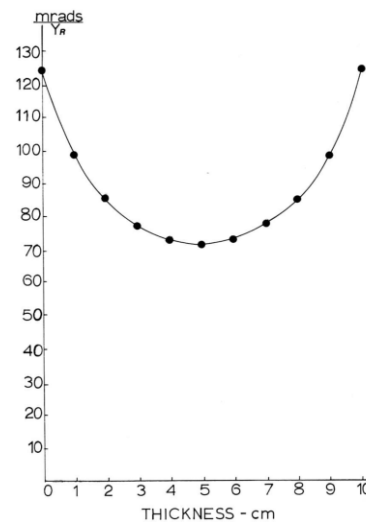


Figure 6. Gamma ray dose rates attenuation through a 10 cm calcite slab with sediments on either sides (assuming: density $\rho_{soil}=1.6$ g/cm³, $\rho_{calc}=2.71$ g/cm³ and $U=6.2$ ppm, $Th=17.4$ ppm, $K_2O=2\%$, $SiO_2=70\%$ sediment). (Liritzis 1989a)

Fig.5 gives the gamma-ray dose rate variation through 10 cm calcitic slab. For a sandwiched calcitic layer with sediments in both sides (Fig.6) the variation of gamma dose rate is evident, e.g in the middle of the slab the dose rate is reduced by about half with respect to surface.

The Figs 5, 6 have been calculated with codes GAMP 1/FGX (using the GAMMA-BANK system through Nuclear Energy Agency, NEA, using Boltzmann photon transport equation (for details see, Liritzis 1989a).

8. ISOTOPIC ACTIVITY TO DOSE RATE CONVERSION FACTORS

Relevant conversion factors of (grays per ka)/(becquerel per kilogram) are summarized in their Supplementary Table 2, and are said that these are regularly compiled and updated, yet promoting the one of the co-author article in Ancient TL (Guerin 2011). However, their Primer review misses to quote the most updated dose rate conversion factors (Liritzis et al., 2013c; Cresswell et al., 2018).

9. PARTIAL BLEACHED MATERIALS AND DOSE EVALUATION

Although, for exposed to daylight sedimentary sufficient net daylight exposure completely removes previously stored energy otherwise it is only partially reset, the total and partial bleaching of quartz has been partially discussed (see also Bailey et al., 1997), and the analogous partial reset in stone surfaces is missing in the Nature Review.

This partial bleaching is met with sediments and for calcitic (limestone) masonries, because due to their slow bleaching rate a residual signal remains which defines the “zero level”. Such a residual signal is met with artificial bleaching by SOL of limestone containing quartz and quartz, feldspar too, for TL and blue OSL (Liritzis et al., 2008).

In fact, for luminescence dose and signal determination by TL the D_e dose is determined for successive

temperature intervals between 200 °C and 350 °C, applying the plateau test (Aitken, 1985; Liritzis et al., 1997a). The constructed growth curve follows the additive procedure of multiple aliquots. The principle of subtraction and the use of a dose plateau are based on the notion that the effect of bleaching causes readings to surpass the equivalent unbleached readings on TL curves by percentages that depend on the length of exposure. Calcite, unlike quartz and feldspar, is not an easily bleached mineral, and in most limestones the unbleachable residual is reached after a prolonged period of some dozens of hours (Habermann et al., 2000; Liritzis and Bakopoulos, 1997a).

Whatever the unknown unbleachable residual TL is, it serves as the ‘non-zero clock’ level upon which subsequent radiation builds up. This principle can also effectively distinguish a recent from an ancient build stone structure (Liritzis et al., 2020).

However, the bleaching is not reduced proportionately for various time exposures for all temperature ranges of a TL curve. Thus, the plateau obtained for different residuals are of variable length. The starting non-zero residual TL is determined as the residual TL level which, when subtracted from the additive dose growth curve, produces the longest plateau in the temperature-dose plateau test (Liritzis and Galloway, 1999; Liritzis et al., 1997a).

The bleached TL curves were subtracted from the natural and N + beta TL curves, after which a dose-temperature plateau (Fig. 7) and a built-up growth curve are constructed (Fig. 8). The longest plateau represents the original (ancient) TL curve, from which the environmental dose builds up. Experimental simulations elsewhere, have shown this plateau (Liritzis and Bakopoulos, 1997; Liritzis et al., 1997a). This maximum plateau length was found for some hours of sun exposure.

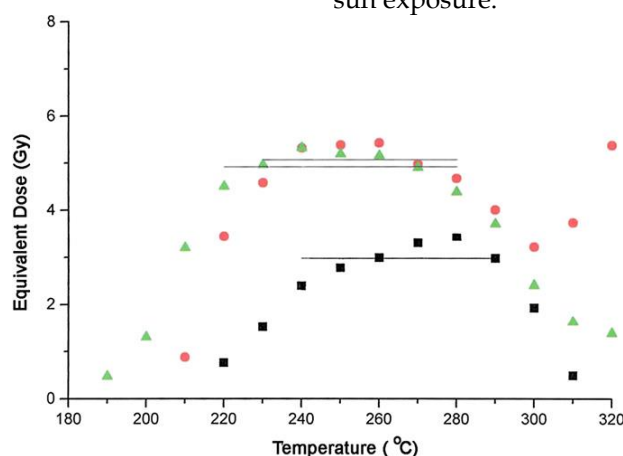


Figure 7. Dose-temperature plateau test. The symbols refer to natural (squares), 12 h (circles), 24 h (triangles) and 36 h (circles) of outdoors sun bleaching. The longest plateau was for the 24–36 h exposure giving an average ED of 5 ± 0.2 Gy (Liritzis 2010).

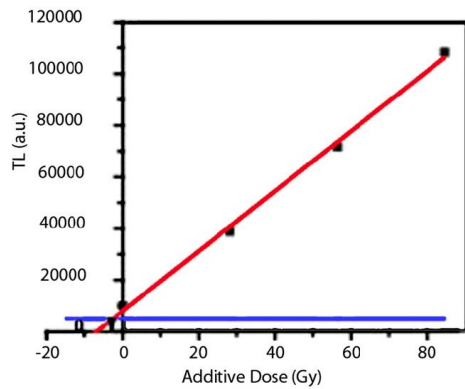


Figure 8. Built-up curve of natural TL. A representative dose-response curve (filled squares) plot for the temperature of 270 °C of the glow curve for the sample STR1. Horizontal line indicates the residual TL level after 24 h of bleaching. The arrow shows the equivalent dose of 4.91 Gy while the equivalent dose plateau yielded 5 Gy (Liritzis 2010).

10. SAR and SAAD protocols

The single aliquot regeneration (SAR) technique has been widely discussed, in spite of the accounting of multiple complications, assumptions and criteria applied. Indeed, using SAR protocol (s), several tests, checks and modifications are recommended to ensure reliable D_e values (Wintle and Murray, 2006; Singhvi et al., 2010; 2011; Ballarini et al., 2007; Ankjærgaard et al., 2006; Xie and Zhang, 2011; Groza-Săcaciuc et al., 2020), and several have been covered by the review, yet a tabular schematic overview of the basic stimulation detection and protocol types of OSL dating has already been given (Liritzis et al., 2013a, table 2.1). Notwithstanding quality of SAR measurements is continually improving (Ballarini, 2006), but applications of SAR in archaeology, palaeoseismology, sedimentary deposits, mortars and other materials, beyond the present authors of the *Primer Review*, are known (a few examples e.g., in cave sediment, Liritzis et al., 2021; Kurecic et al., 2021; Mercier et al., 2012; Clark-Balzan et al., 2012; palaeoseismology, Tsodoulos et al., 2016; loess, Groza-Săcaciuc et al., 2020; ceramics, Sun et al., 2021 and Benea et al., 2007; lake sediment, Zander and Hilgers, 2013; mortar and surface dating, Panzeri 2013).

But the single aliquot added dose (SAAD) is first incompletely presented and second reported as first work by the present first author article in Murray et al., (1997) instead of Duller (1991) (Duller 1991, 1994a, b) was the first to demonstrate how this could be undertaken in practice, and these methods have been widely developed into a single aliquot method for D_e determination by administering additive doses to single aliquot extracts. Same year and later a number of luminescence workers have suggested that it would be feasible to make all the measurements necessary to

calculate a palaeodose on a single aliquot using green or blue OSL (Liritzis et al., 1994; Galloway 1996; Liritzis, et al., 2001, 2002).

This SAAD technique for quartz (and feldspar) requires correction for sensitivity change during read outs as has been described elsewhere and has been further improved and produced fundamental results (Duller 1994 a, b; Liritzis et al., 1994; 1997b; 2001, 2002; Duller & Murray, 2000).

Here we briefly describe this SAAD technique, in which a single aliquot (disc) -either quartz or feldspar- is measured with consecutively administering beta doses and reading the OSL by short shining from diodes at certain wavelength. The signal growth is fitted by appropriate functions. The essential information for the correction of SAAD is provided by the decay curve giving the factors, $f(n)$, which is the exponential fit to loss of signal by successive preheats and by which the stable luminescence signal is reduced at the n th preheating and reading of the aliquot and that the $f(n)$ values are essential dose independent. For example, the stimulation of quartz by blue light, the factors $f(n)$ show an exponential dependence:

$$f(n) = e^{-b(n-1)} = r^{(n-1)} \quad (4)$$

$$\text{where, } r = e^{-b} = f(n)/f(n-1) \quad (5)$$

that is r is the ratio of any factor to the immediately preceding factor. The correction curve of SAAD by IR due to signal loss of quartz and feldspar followed either the α -relation, $1 - \alpha \ln(n)$, n is the number of cycles, or the power law p -relation, n^{-p} , n is the number of cycles (see Liritzis & Vafiadou, 2014, Fig. 12). One consequence of the exponential decay for the correction of single aliquot additive dose measurements is that the correction equations become the same regardless of whether or not the decay of each added component of Luminescence is regarded as being independent of the others. The correction required is simply:

$$\text{corr}L(D_n) = \text{meas}L(D_n) - r \text{meas}L(D_{n-1}) + \text{corr}L(D_{n-1}) \quad (6)$$

where $\text{corr}L(D_n)$ is the corrected value of luminescence resulting from the n^{th} dose D_n and $\text{meas}L(D_n)$ is the measured value of luminescence. The distinction between the first and second correction methods of Duller (1991) which was all important for the stimulation of feldspar by infrared disappears for the stimulation of quartz by blue or green light. Further, the decay factors, $f(n)$ required to correct single aliquot measurements are replaced by r , which can be determined directly from the ratio of any sequential pair of preheating and luminescence readings with no added dose between (although of course better accuracy may come from averaging a sequence of measurements). Thus, unlike the situation with the infrared stimulation of feldspar, the decay factors can be determined directly (without the iterative process de-

scribed by Galloway (1996) from decay measurements made on the same aliquot after the additive dose measurements (Figs. 9, 10). The SAAD is a well-documented technique, though little attention has been drawn, and offers apparent advantages as described elsewhere and with some comparison with SAR; the latter is recommended as a thoughtful application, if all criteria of reliability are applied for each mineral type (Liritzis et al., 2008).

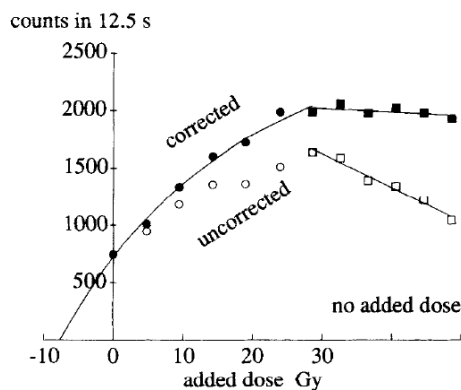


Figure 9. Single aliquot additive dose data (uncorrected and corrected) for B42 : I. with saturating exponential fit to the corrected points. Also shown, to the right of the figure, are the repeated preheat and read cycles which give the mean correction ratio r and the result of correcting these points by the mean ratio, which should ideally give a horizontal line. Liritzis et al., 1997b).

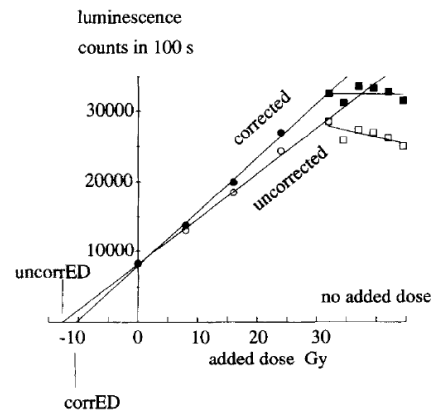


Figure 10. Single aliquot additive dose data (uncorrected and corrected) for B25-3. with linear fit to the corrected points and to the uncorrected points for comparison. Also shown, to the right of the figure, are the repeated preheat and read cycles which give the mean correction ratio r and the result of correcting these points by the mean ratio, which should ideally give a horizontal line (from Liritzis et al 1997b)

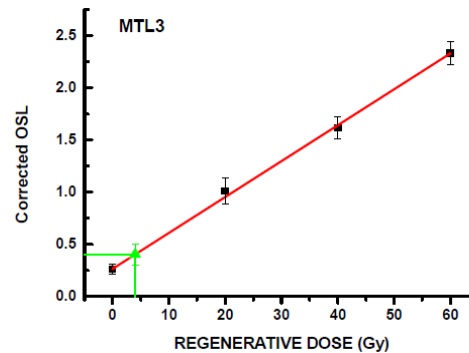
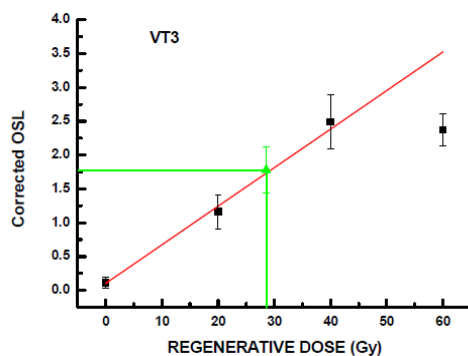


Figure 11. SAR protocol of quartz extracted from limestone, A) of the Valley Temple building (sample No ST3), B) from the southern Wall at Mycenae (sample MTL3) (from Liritzis et al., 2008, 2010b, 2014).

Some further issues worthy of attention are the following. The prolonged exposure to infrared has no adverse influence on quartz additive dose measurements; tests were carried out on material that was free from feldspar (as confirmed by infrared stimulated luminescence testing of another aliquot of the same material). The tests consisted of measuring several points of an additive dose curve, then exposing the quartz sample to the infrared emitting diode system

At any rate SAR and SAAD have been applied successfully in different materials (ceramics, stones with major quartz content or quartz extracted from limestone).

Fig.11 shows examples for the D determination from quartz extracted from limestones in archaeological sites.

for 3000 sec and then continuing the additive dose measurements. The additive dose curve continued smoothly, undisturbed by the exposure to infrared (Fig. 12). It has been earlier concluded that prolonged exposure to infrared could be used to effectively "clean" a quartz sample from ceramics slightly contaminated with feldspar prior to stimulation by green light without undesirable influence on the additive dose curve.

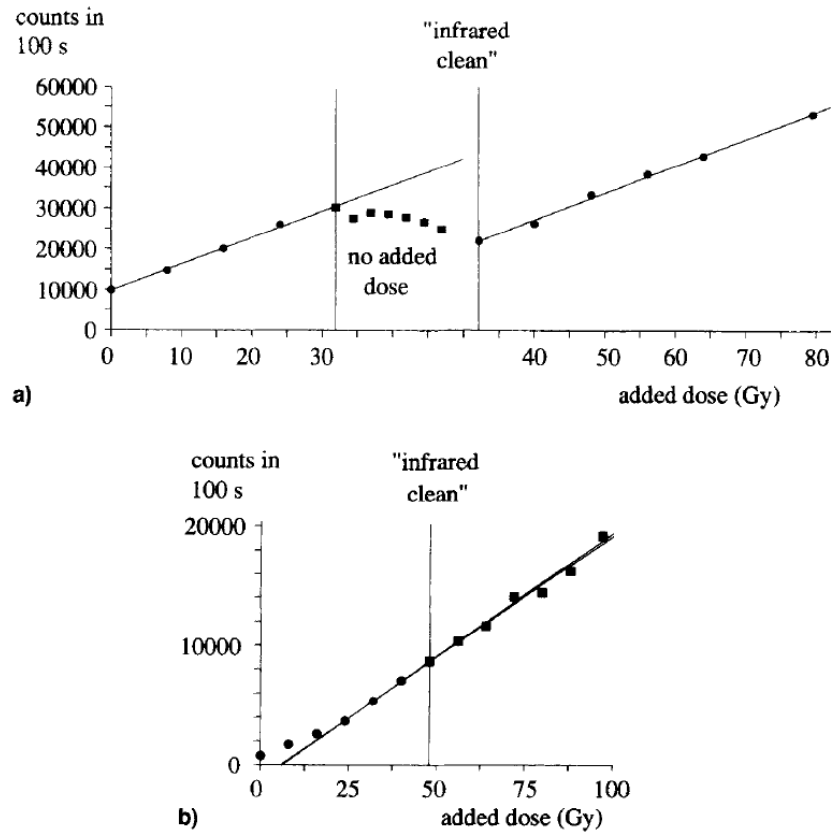


Figure 12. Quartz from ceramics. Two additive dose growth curve measurements on quartz interrupted, but not affected, by 3000 sec exposure to IR. In (a) uncorrected single aliquot additive dose data are presented from a ceramic sample (B25-3, Heliki ancient settlement Peloponnese, Greece). The initial additive dose measurements up to 33 Gy are followed by seven repeated preheating and reading cycles without added dose, typical of the single aliquot procedure. The sample was then exposed to infrared for 3000 sec and the green stimulated additive dose procedure continued. The two additive dose sections show the same sensitivity. 650 ± 20 counts per 100 sec per Gy and 660 ± 30 counts per 100 s per Gy, respectively, unaltered by the IR exposure. Corrected data for sample No K2 (which is supra-linear below 25 Gy added dose) is shown in (b), with the infrared exposure at 48 Gy having no detectable influence on the quartz sensitivity (207 ± 3 counts per 100 s per Gy before, and 203 ± 12 counts per 100 s per Gy after the IR exposure) (based on Liritzis et al., 1997b)

Sensitivity change in quartz follows bleaching. It could however be of interest to see whether, sensitivity apart, the additive dose response curve after bleaching could indicate the shape of curve appropriate to the determination of the equivalent dose, that is. to confirm linearity, or to help determine the appropriate parameters to describe supra-linear or saturating response. The responses of three aliquots of the same material are compared in Fig. 13: for the K2 ceramic sample from Heliki site, Greece, one aliquot

was bleached by Edinburgh daylight for 6 h and shows a supra-linear response; another aliquot was bleached; for 1 h in a Home SOL-2 "solar simulator" and shows a saturating response with increased sensitivity compared to the daylight bleached material; the third was bleached by heating at 500°C for 1 min and also shows a saturating response with even higher sensitivity (Liritzis 1980, 1982). This does not encourage the use of a simple laboratory bleaching method to determine the dose response.

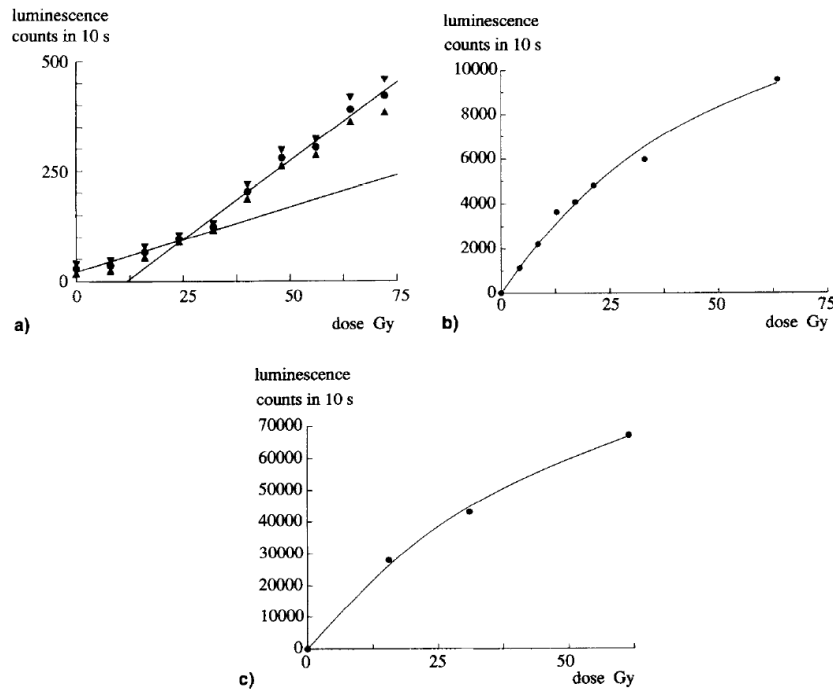


Figure 13. The response of sample K2 after different bleaches: (a) by daylight for 6 h. showing a supralinear response at low dose and with an approximation to the response by two straight lines. The small non-zero intercept, if statistically significant, may be due to the recuperation phenomenon associated with bleaching and preheating. The result following bleaching for 1 h in a Holne SOL-2 solar simulator is shown in (b) along with a saturating exponential fit to the measurements of luminescence and dose, D . luminescence = 12 120 $(1 - \exp(-0.024 \cdot D))$. The response following bleaching by heating at 500°C for 1 min is shown in (c) along with a saturating exponential fit to the data, luminescence = 88 800 $(1 - \exp(-0.023 \cdot D))$. The fit has essentially the same shape (exponent) in both (b) and (c). with (c) showing a higher luminescence sensitivity to dose. (Liritzis et al., 1997b).

11. VIOLET SL

The Violet stimulation luminescence (VSL) in optical dating has been also missing the recent development using a single aliquot regenerative dose (SAR) protocol and a multiple aliquot additive dose (MAAD) protocol (Sontag-González et al., 2019) of a relatively young sample (~40 ka) which was successfully determined. The significant underestimations observed for older samples with higher doses indicated the need for further development of the measurement protocol to date high-dose natural samples. Worth is the exploration of multiple-aliquot methods for quartz violet stimulated luminescence dating (Ankjærgaard, 2019).

12. WATER UPTAKE

The presence of water reduces the dose rate, and thus increases the calculated age. It is therefore important to estimate the average water content of the deposit over the burial period. During sampling, one should consider the water content history of the site and not only resting to collecting samples on which to measure the present-day water content (Liritzis &

Galloway 1981). The Box 1 for Water content estimation of Murray et al., 2021 gives a thorough approach to this correction, but there is no a universal rule for water uptake correction and each case is considered separately; the humidity (and temperature) history of the context must be approached from proxy data and average values between dry and saturated states with attached error bar is most prudent. Moreover, their quoted "...because a 1% increase/decrease in lifetime average water content typically leads to a 1% increase/decrease in derived age..." is reported in Jacobs (2017, 554).

13. BETA-LUMINESCENCE: A SATISFACTORY FIRST APPROXIMATION LUMINESCENCE DATING VERSION

A quite approximate and satisfactory result with around $\pm 15\%$ error is obtained with the *beta luminescence* dating version (initially called beta-TL, Liritzis 1989b), making use of the geochemical relationship between the U, Th, K isotopes in sedimentary rocks for inclusion dating of ceramics and sediments and the reported linear variation between D_β dose rate and potassium content (Fig.14). (Liritzis 1985).

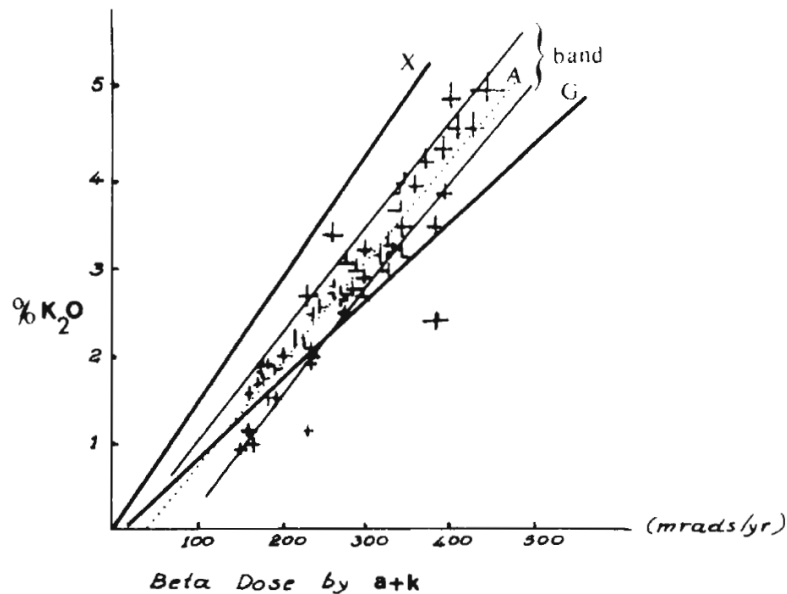


Figure 14. Beta dose rates versus K_2O for sediments and ceramics. Curve A is the fit of all data, T curve is for the data from TLD beta doses, and X curve for the depletion of Th, U giving lower boundary of expected uniform distribution. G curve is the best fit for the geological data. Band indicates 95% inclusion of data points (from Liritzis 1985)

The age is calculated from two parameters, the potassium content (K) and the total dose (D). The concept in brief is as follows:

From the luminescence age equation (7) for inclusions:

$$\text{age} = D_e / [aD_\beta + D_\gamma + D_c] \quad (7)$$

it has been shown a liner relationship between potassium ($K_2O\%$) and D_β dose rates in $\text{mrad/s}=10^{-5}$ Gy in various sediments.

$$K = 0.35 (\pm 22\%) + 0.77 (\pm 3\%) * D_\beta * 10^{-2} \quad (8)$$

the denominator (for $a=1$) of eq.7 can be written as:

$$B = (D_\beta + D_\gamma) / D_\beta \quad (9)$$

As the D_β is a major contributor in annual dose-rate and also carries a weight in eq.9, and due to the geochemical relationship between the U, Th, K isotopes in sedimentary rocks (Fig.14), a linear variation has been observed between the logarithm of factor B and the potassium content (Fig.15).

From eq.8, 9:

$$\text{Log } B = 0.244 (\pm 4\%) - 0.024 (\pm 3.6\%) * K \quad (10)$$

and eq.7, from eq.8, 9 becomes eq.(11).

$$\text{OSL/TL-age} = 0.0077 * D / [B * (K - 0.35) * a + D_c * 0.77 * 0.01] \quad (11)$$

Where, D the total OSL/TL/ESR equivalent dose in $\text{Rads}=10^{-2}$ Gray= 10^3 mrad, K the $K_2O\%$, (a) is the beta attenuation through the grains, B is obtained from potassium of eq.10 and D_c (mrad/s/yr) is the cosmic ray dose-rate.

The water uptake correction (from Zimmerman's formulas, Zimmerman 1970) seems to have a low error in the age of this dating version, because the B factor becomes:

$$B = 1 + [D_g (1.25w - 0.25) / [D_b (1.14w - 0.14)] \quad (12)$$

where w the water weights (%) of saturated to dry/natural state of the sample. For 80% w the error to B is 5% and for lower water uptake values the error even insignificant. The induced respective age error is negligible. Due to principal involvement of D_β dose rates and K content (a major beta contributor) the method was called **beta-TL/OSL**.

Overall, the age errors lie between 10-20% with a mean of 12-15%; for inclusion data the 90% of the TL and respective β -TL obtained ages were compatible to within 0-20% of the respective quoted TL errors.

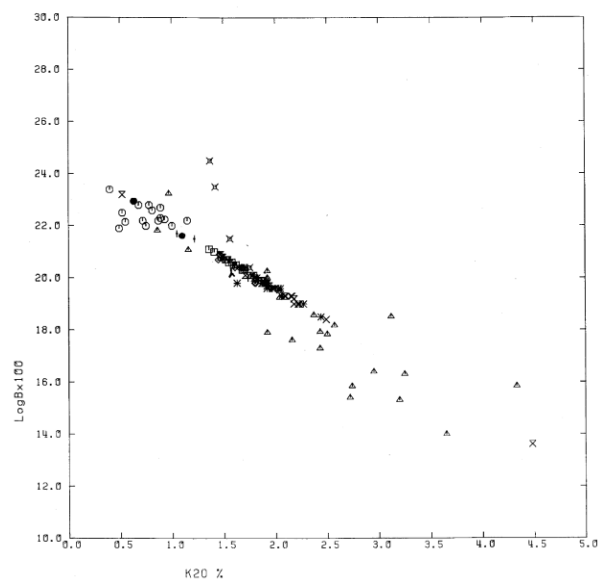


Figure 15. Plot of Logarithm of (B) against $K_2O\%$ for various sediment types (cave, soils, ocean, subaerial, loess and dunes, beach sands, granite, see Liritzis 1989).

13. DUNES AND LOESS & TT-OSL

New optically stimulated luminescence (OSL) applications for dunes have been omitted one e.g on stabilization chronologies for two complexes of fully stabilized, parabolic dunes along the eastern coast of Lake Michigan with the goal of clarifying the timing and consistency of dune stabilization along the shoreline (Fulop et al., 2019). Also, on fluvial sediments for dating Clovis in Texas (Rodrigues et al., 2016), but aeolian sediments too making use of the thermo transfer (TT) OSL signals, something that the review omits regarding also the TL, OSL, and TT-OSL samples from a stratigraphic section in southern Tasmania, Australia. There, Neudorff et al., (2019) provide information on their signal characteristics, their utility as chronometers, and give insight into the bleaching histories of the deposit. Important age results between 20 ka (MIS 2) to 180 ka (MIS 6) were obtained. TT-OSL ages from older sediments in this region, combined with further stratigraphic studies have the potential to determine the climatic history of Tasmania over multiple glacial periods (with an improving TT-OSL SAR approach, see, Adamiec et al., 2010). Kinnaird et al (2016) used luminescence dating methods to provide a chronological framework to interpret landscape processes and human-environmental interactions over this timescale and coupled with landscape studies in the Vasilikós and Dhiarizos valleys in Cyprus, during the Neolithic and the Chalcolithic period, where the underlying geology, geomorphology and environment contributed to the choice of site. The luminescence chronologies, reported, *“suggest that modifications in the first-order catchment hydrology occurred over timescales in excess of 10^3 years. It has been shown that the present-day topography in Cyprus was initiated in the latest Pliocene-Pleistocene, as a result of pronounced uplift of the island and the environmental conditions which prevailed, and that only minor modifications to this first-order topography have occurred since, with the re-working, and re-deposition of Early - Middle Pleistocene sediments over timescales of both 10^{2-3} and 10^{4-5} years”* (Kinnaird et al 2016). Pertinent to archaeological surveys is the value of OSL in distinguishing ancient from more recent structures in an archaeological landscape (Liritzis et al., 2020).

There are numerous other important World case studies which should not be missed out in any academic review, and the unavoidable limited space in articles, could be compensated prudently by citing their respective websites (e.g. <https://theglowcurve.org/>).

Also, Thermally-Transferred Optically Stimulated Luminescence (TT-OSL) from quartz is an extended-

range luminescence dating technique, with an assumed potential to date sediments as old as early Pleistocene (0.8–2.6 Ma) (Faershtein et al., 2018)

At any rate the TT-OSL is still a more complex phenomenon still needs further development.

14. AGE CALCULATION SOFTWARE & OTHER MISSED NOVEL APPLICATIONS

Concerning the age calculation quoted based on one of the few widely available software (see eg <https://CRAN.R-project.org/package=Luminescence>), it should be stressed that may be unorthodox and occasionally misleading due to the complicated dosimetry involved on each occasion and age should be calculated per se (examples see: Liritzis & Vafiadou 2014; 2018), and not resting entirely on these costumed contexts.

A recent book covers comprehensive applications of R to the general discipline of radiation dosimetry and to the specific areas of luminescence dosimetry, luminescence dating, and radiation protection dosimetry and Monte Carlo methods, which are used to simulate the luminescence processes during the irradiation, heating, and optical stimulation of solids, for a wide variety of materials (Pagonis 2021). Also, the interesting study on the intrinsic accuracy and precision of luminescence dating techniques for fired ceramics (Pagonis et al., 2011); the new dating of fossil root cast (Ertek et al., 2015); the meteoritic impact in Bavaria as a destruction factor to 1st millennium BC culture there (Liritzis et al., 2010c), the basic investigations on the relative response of TL and component-resolved OSL to alpha and beta radiations in annealed sedimentary quartz (Polymeris et al., 2011) and the potential of OSL dating of painting ground layers (Polymeris et al., 2013) and alternative measurement of total dose by green light (Liritzis 1995).

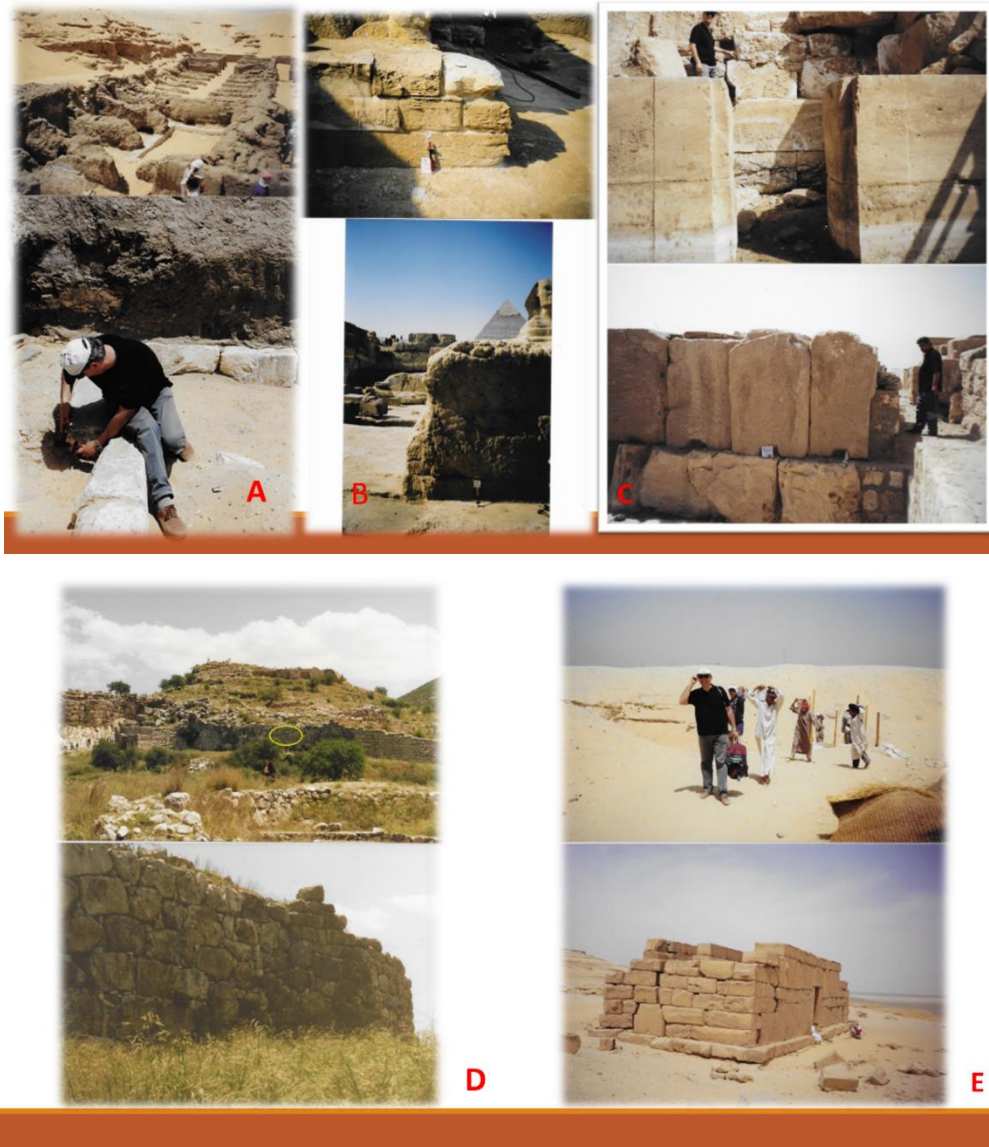
15. CONCLUSION

Any reviews on OSL dating, and any scientific field are most welcome and should refer to all initiatives, unique applications and novelties on a representative and objective geographical and authorship range. The Murray et al (2021) OSL dating review using quartz is a biased to selective narrow case studies and ignore major impacted works. That saying it could be the chosen style of the authors and overlooked by reviewers hence it requires a necessary consummation. Various aspects of total dose and dose rate evaluation and alternative sound protocols have been recalled in the present editorial due to their absentia from the review in Nature primer. Useful protocols and approaches but significant recent references too, fill the missing gap.

ACKNOWLEDGEMENTS

I thank Dr G.Polymeris for useful discussion.

APPENDIX (characteristic photos of masonries and sampling)



Photos. A) Sampling at the pharaoh's Khasekhemwvy rectangular tomb with his mud made "rooms"; a complex covered by desert sand. Photo A5. Sampling at Sphinx Temple (lower, sample ST 1) and at the Valley Temple sampling (upper, sample VT8), C) At Abydos southern Egypt in Seti I Temple and Osirion tomb. Sampling at Osirion wall (sample No 6) and at the ceiling of Seti I (sample No Seti I- 4th). D) Mycenae west wall of Lions gate and location of sampling (with the aid of late prof. S.Iacovides), E) Temple at Qasr-el-Sagha is a small temple and without inscriptions, about 8 km north of the lake Birket Qarum, the front end of an horizontal plateau about 34 m above sea level in the northwest of the Fayum; and head on towards the temple for sampling (in 2001) (for dating results, see: Liritzis & Vafiadou 2014).

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