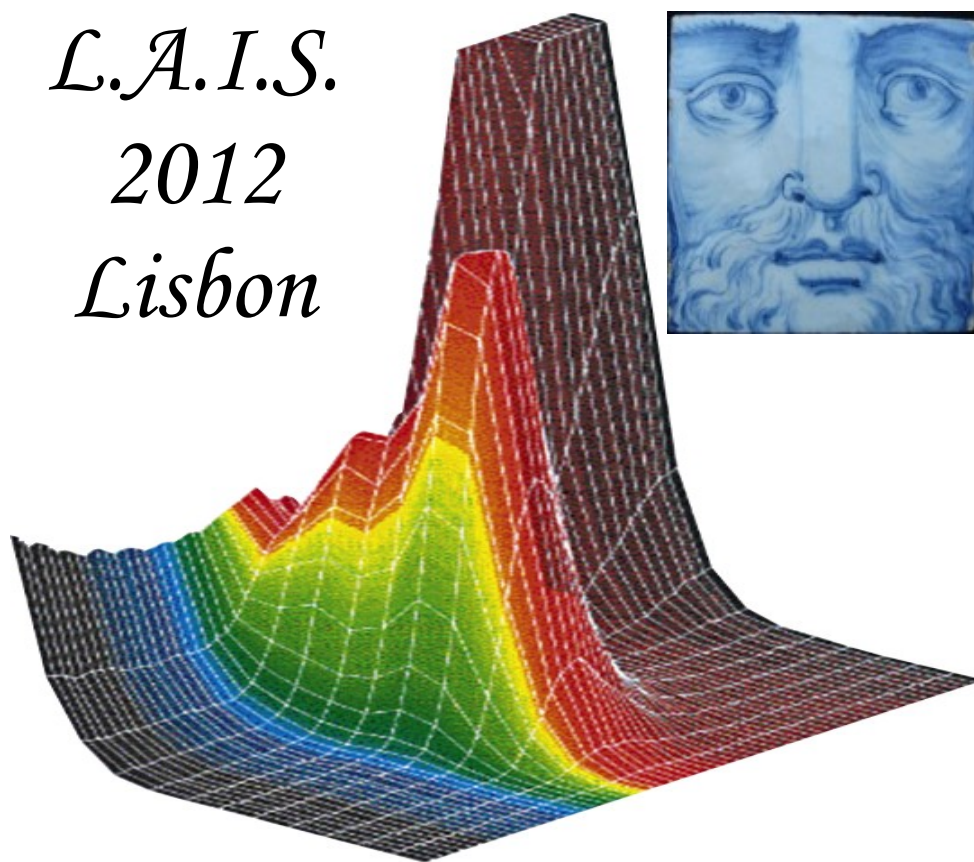


2nd Luminescence in Archaeology International Symposium

5th - 7th of September, 2012.

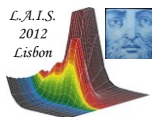
IST/ITN, Instituto Superior Técnico, Universidade Técnica de Lisboa

*L.A.I.S.
2012
Lisbon*



*Exploration of the potentialities and limitations of luminescence and
related analyses for materials and questions of archaeological and
cultural heritage significance.*

Book of Abstracts - Final



2nd Luminescence in Archaeology International Symposium

5th - 7th of September, 2012.

Book of Abstracts - Final

With participants, program and abstracts revised to reflect last minute changes

Hosted by:

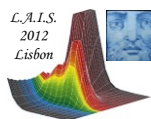
Grupo de Geoquímica Aplicada & Luminescência no Património Cultural (GeoLuC)

Unidade de Ciências Químicas e Radiofarmacêuticas (UCQR)

CAMPUS TECNOLÓGICO E NUCLEAR, IST/ITN

Instituto Superior Técnico, Universidade Técnica de Lisboa,

Estrada Nacional 10, 2686-953 Sacavém, Loures-Portugal



Invitation

The 2nd Luminescence in Archaeology International Symposium will be held in Lisbon, Portugal, from the 5th to the 7th of September, 2012. It will be hosted by the Group of Applied Geochemistry & Luminescence on Cultural Heritage (GeoLuC), Instituto Tecnológico e Nuclear (ITN), Sacavém, Portugal.

L.A.I.S. is an international initiative focussing on the use of luminescence for the dating and analysis of materials and questions of archaeological significance; in addition it supports archaeological and archaeometrical communities of the World to further develop and expose luminescence issues.

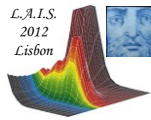
L.A.I.S. 2012 continues the series of symposia initiated in Delphi 2009, which focus on presentation and exploration of the potentialities and limitations of luminescence and related analyses, for materials and questions of archaeological and cultural heritage significance. L.A.I.S. Symposia aim at bringing together experts in the fields of luminescence, archaeology and archaeological materials from around the world for the exchange of knowledge.

Presentations are invited to focus on either contextual, methodological or phenomenological aspects of luminescence studies in archaeology and cultural heritage, including dating and material characterisation. For LAIS 2012 presentations are invited to address archaeological issues related to the following broad themes: megaliths and megalithism; ceramics, glass and vitreous materials; reconstruction of environments in archaeological sites.

Oral and poster presentations based on an integrated multidisciplinary approach are encouraged (i.e. co-authorship by luminescence specialists, archaeologists, archaeometrists, (geo)chemists, geomorphologists, geologists, palaeoenvironmentalists, physicists), as are applications for oral presentations by students.

Articles based on oral and poster presentations will be eligible for submission to a special issue of *Mediterranean Archaeology and Archaeometry* for peer reviewed publication.

C.I. Burbidge,
Inv.Aux.Contr. Física Aplicada,
GeoLuC, UCQR, ITN.
On behalf of
I. Liritzis
L.A.I.S. President



People

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M.I. Dias
M.I. Prudêncio

Secretariat

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D. Franco
G. Cardoso
A. Rodrigues

Website

T. Pires

Logo

“Veronica” Courtesy of Alexandre Pais, Museu Nacional do Azulejo

Local Scientific Committee

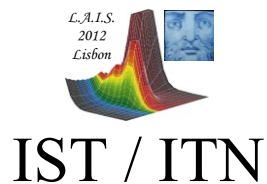
M.J. Trindade, GeoLuC, UCQR, ITN, Portugal.
A. Valera, ERA Arqueologia SA., Portugal
L. Oosterbeek, Instituto Politécnico de Tomar, Portugal
V. Corregidor, U.F.A. ITN, Portugal
J. Sanjurjo Sánchez, IUXIPP, Universidade da Coruña. Spain
V. Correcher, CIEMAT, Madrid, Spain
C.P. Marques, CFNUL, Universidade de Lisboa, Portugal

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G. Wagner, University of Heidelberg, Germany



IST/ITN is a research institute with 50 years of contribution to the development of knowledge in collaboration with the scientific community in Portugal and internationally.



IST/ITN is based in a scenic 10 ha science park next to the town of Bobadela at the doorway to Lisbon and is well served by main road, rail and air networks.



Auditorium

Oral presentations are in the fully equipped 156 seat auditorium located in the library building of IST/ITN.



Poster presentations will be made in the “VIP Room” of the IST/ITN Canteen.

Refreshment

Luncheon and refreshments during breaks between sessions will be served in the “VIP Room” of the IST/ITN Canteen, adjacent to the Library Building.



Locations and Transport

Transport to and from Lisbon Airport

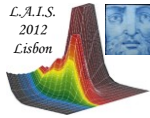
Lisbon International Airport is located approximately 8 km from ITN and 7 km from Lisbon city centre. Transport to or from the airport is by metro, taxi (around €5 to Roma-Areeiro or €20 to IST/ITN) or bus (22 to Roma Areeiro, 44 to Oriente/Parque das Nações, or Airport Express to City Centre or Oriente/Parque das Nações). A new Metro line extension has just opened between Lisbon Airport and the hotel locations. This now offers the best combination of simplicity and value for transport to and from the Airport, unless you are travelling in a large group, when a taxi may still be slightly more economic.

Transport to and from ITN

Transport to and from ITN is best accomplished by means of the suburban train network. ITN is 3 minutes' walk from Bobadela train station. 6 trains per hour run to/from Bobadela at peak times (07:00-09:30, 17:00-19:30), 2 at off-peak times. To get directly to Bobadela from Roma-Areeiro (journey time approx. 20 min), use trains with destinations Alverca and Azambuja (plus destination Castanheira do Ribatejo from Oriente or Santa Apolonia). To get directly to Roma-Areeiro from Bobadela, use trains with destinations Sintra and Alcantara Terra (plus destination Santa Apolonia to Oriente or Santa Apolonia). For those staying in the centre of Lisbon, the metro system is inter-linked with the suburban train network.

The same rechargeable ticket, "Via Viagem", can be used on public transport in greater Lisbon (bus, tram, metro, suburban trains: not tourist services or airport express). To mix freely between transport modes, "Zapping", it must be charged with a cash value rather than with a number of journeys using a particular mode. All tickets must be validated on entry to stations/lines/busses by swiping a detector, subject to a fine. Note that line changes on the metro make part of the same journey (so long as you stay in the station), but changing from the metro to the suburban train network still constitutes 2 journeys.





Visits and Conference Dinner

Visits

On Thursday afternoon, 6th of September, participants will be taken by bus directly to the Sé (See) of Lisbon in the city centre.

A functioning place of worship under the auspices of the Patriarcado de Lisboa the Sé is the oldest church in the city, having been founded on the site of an earlier mosque following the Christian re-conquest of Lisbon in the 12th Century. In addition to the history, architecture and materials of the church and cloisters, it contains an exhibit of historic ecclesiastical articles in halls lined with painted canvases. The cloisters contain an area of open archaeological excavations exhibiting sequences of occupation from the Iron Age onwards.

From the Sé, narrow streets of richly tiled Pombaline buildings lead, via the preserved excavations of a Roman Theatre, to the Castelo São Jorge. While focussed on the 11th century Moorish fortification, with alterations until the 19th century, the castle and its grounds encompass a museum, an open excavation of Prehistoric and Moorish occupation levels, the ruins of the former Royal Palace of the Alcáçova. They also offer fine views over the city and bay, as well as opportunities for refreshment.

Conference Dinner

RESTAURANTE_CERVEJARIA_BALEAL

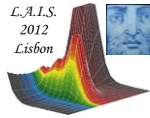
Rua da Madalena 265 - 1100 Lisboa, Portugal

The conference dinner will be held at the Restaurante Cervejaria Baleal in the “Baixa de Lisboa” (downtown Lisbon), following the visits. It is a traditional Portuguese restaurant serving quality dishes and specializing in sea-food, located just downhill from the Sé-Cathedral and Castelo São Jorge, close to Rossio and Baixa-Chiado metro stations.



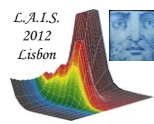
Visits and Conference Dinner



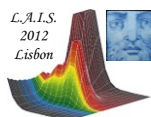


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A.J.C. ZINK	Palais du Louvre	France	antoine.zink@culture.gouv.fr	48

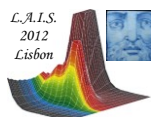


Program



Wednesday, 5th of September 2012

09:00	Registration	
09:20	PRESIDENT'S ADDRESS	
Session 1	Dose Rate.	Chair: C.I. BURBIDGE
09:40	Plenary Presentation: <u>I. LIRITZIS</u> , K. STAMOULIS, C. PAPACHRISTODOULOU AND K. IOANNIDES - UPDATING OF RADIATION DOSE-RATE CONVERSION FACTORS: A CRITICAL RE-EVALUATION	24
10:00	<u>C. T. MICHAEL</u> , A. HEIN AND E. DOTSIKA - ISOTOPIC DATING AND ENVIRONMENTAL MICRODOSIMETRY USING THICK SOURCE ALPHA PARTICLE SPECTROSCOPY	28
10:20	<u>A. BOUVIER</u> , P. GUIBERT AND C. SAPIN - DATING BRICKS FROM THE CRYPT OF THE CHARTRES' CATHEDRAL BY OSL: THE IMPORTANCE OF INTERNAL RADIOACTIVITY OF QUARTZ GRAINS	5
10:40 – 11:20	Coffee break	
Session 2	Materials and Equipment.	Chair: P. GUIBERT
11:20	<u>M. L. CHITHAMBO</u> - LUMINESCENCE LIFETIMES IN THE '600 -700°C' TRANSITION REGION FOR QUARTZ	11
11:40	<u>V. K. ASFORA</u> , P. L. GUZZO, A. M. PESSIS, S. WATANABE, H. J. KHOURY – SELF-CONSISTENT PROCEDURE TO DATE BURNT PEBBLES USING THERMO-LUMINESCENCE AND ELECTRON PARAMAGNETIC RESONANCE	1
12:00	K. DORNICH, M. KRBETSCHKE, A. RICHTER AND <u>D. RICHTER</u> - A NEW MEASUREMENT SYSTEM FOR LUMINESCENCE DATING – LEXSYG	14
12:20	<u>D.C.W.SANDERSON</u> – LINKING RADIOMETRIC MAPPING AND REMEDIATION: UK AND EUROPEAN EXPERIENCE	<i>Impromptu Presentation</i>
12:40 – 14:40	Lunch	
14:40		
Session 3	Posters	
15:40		
15:40 – 16:20	Coffee break	
Session 4	Prehistoric Contexts 1.	Chair: D. RICHTER
16:20	<u>T.C. KINNAIRD</u> , J.E. DIXON, E. PELTENBURG AND D.C.W. SANDERSON GEO-ARCHAEOLOGICAL INVESTIGATIONS AT SOUSIKOU-LAONA (CYPRUS): PLACING THE CHALCOLITHIC SETTLEMENT AND CEMENTARIES IN A LANDSCAPE CONTEXT	21
16:40	<u>D. RENZELLI</u> , F. SIRIANNI, A. OLIVA, P. BARONE AND V. PINGITORE TL DATING OF NEOLITHIC and MEDIEVAL CERAMICS from LAMEZIA, CALABRIA (SOUTH ITALY)	34



Thursday, 6th of September 2012

09:00 **Registration**

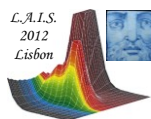
Session 5	Palaeolithic Contexts.	Chair: N. ZACHARIAS
09:20	<u>D. RICHTER</u> , D. E. ANGELUCCI, C. I. BURBIDGE, M. I. DIAS, M. A. GOUVEIA, M. I. PRUDÊNCIO AND J. ZILHÃO - HEATED FLINT FROM GRUTA DA OLIVEIRA (PORTUGAL): COMPARISON OF TL-DATING RESULTS WITH RADIOCARBON AND U-SERIES DATING	35
09:40	A. BRODARD, <u>P. GUIBERT</u> , C. FERRIER, E. DEBARD, B. KERVAZO AND J.-M. GENESTE - THERMAL CHARACTERIZATION AND TL-DATING OF REDDENED WALLS OF THE CHAUVET CAVE (ARDÈCHE, FRANCE)	6
10:00	<u>M. FROUIN</u> , N. MERCIER AND C. LAHAYE - CHRONOLOGY OF NEANDERTHAL BURIAL SITES IN SOUTH-WEST FRANCE: PRELIMINARY IRSL DATING RESULTS	15
10:20	<u>M. HERNANDEZ</u> AND N. MERCIER - TOWARDS A CHRONOLOGICAL FRAMEWORK OF MIDDLE PLEISTOCENE HUMAN SETTLEMENTS IN SOUTHWESTERN FRANCE THROUGH THE USE OF THE TT-OSL DATING METHOD APPLIED TO SEDIMENTARY QUARTZ	17

10:40 – 11:20 **Coffee break**

Session 6	Environmental Processes.	Chair: V. CORRECHER
11:20	Invited Plenary Presentation: <u>DAVID C. W. SANDERSON</u> - A DECADE OF LUMINESCENCE PROFILING OF ARCHAEOLOGICAL AND ENVIRONMENTAL SEDIMENTS	37
11:40	<u>J. VAN NIEULAND</u> , D. VANDENBERGHE, F. GELAUDE AND P. VAN DEN HAUTE - THE INFLUENCE OF AEOLIAN ACTIVITY IN THE ENVIRONMENTAL SETTING OF THE CITY OF GHENT (EAST FLANDERS, BELGIUM): A CONTRIBUTION FROM OSL DATING	45
12:00	<u>D. MUTZENBERG</u> , A. M. PESSIS, D. CISNEIROS, H. KHOURY, A. C. B. CORRÊA AND R. LIBONATI - OSL CHRONOSTRATIGRAPHY OF PALEOENVIRONMENTAL EVENTS – LAGOA URI DE CIMA ARCHAEOLOGICAL SITE, SEMI-ARID REGION IN BRAZIL	29
12:20	<u>A. L. RODRIGUES</u> , C. I. BURBIDGE, M. I. DIAS, F. ROCHA, A. VALERA, M. I. PRUDÊNCIO - LUMINESCENCE AND MINERALOGY OF PROFILING SAMPLES FROM NEGATIVE ARCHAEOLOGICAL FEATURES	36

12:40 – 14:40 **Lunch**

14:40	Bus to Sé-Cathedral
15:00	Visit to Sé-Cathedral
17:00	Walk to Castle
17:20	Visit to Castle
19:40	Conference Dinner



Posters

1 Dose Rate

C.I. BURBIDGE, P.M. TELES, M.J. TRINDADE, M.J. REIS, M.I. PRUDÊNCIO, S. ANDREJKOVICOVA, D.C.W. SANDERSON, F. ROCHA, J.V. CARDOSO, G. CARVALHO, J. ABRANTES, R. MARQUES, G.J.O. CARDOSO, Y. ROMANETS, L. SANTOS, D. FRANCO, M.C. SEQUEIRA, M.A. GOUVEIA. **VADOSE - A NEW PROJECT TO INVESTIGATE SPATIAL VARIATION OF DOSE RATES IN SOILS AND SEDIMENTS** 9

M.J. TRINDADE, M.I. PRUDÊNCIO, C.I. BURBIDGE, M.I. DIAS, R. MARQUES, G. CARDOSO AND F. ROCHA. **DISTRIBUTION OF NATURAL RADIONUCLIDES (K, TH AND U) IN AN APLITE DYKE FROM THE BEIRA URANIFEROUS PROVINCE (FORNOS DE ALGODRES, PORTUGAL)** 43

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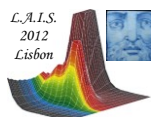
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Posters

4 Prehistoric Contexts

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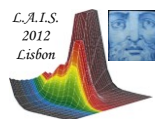
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Abstracts

SELF-CONSISTENT PROCEDURE TO DATE BURNT PEBBLES USING THERMOLUMINESCENCE AND ELECTRON PARAMAGNETIC RESONANCE.

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Introduction: Luminescence techniques for dating heated materials such as pebbles and pottery are based in the same principles. However, quartz pebbles offer extra difficulties as follows: (i) it is not simple to distinguish burnt from unburnt specimens based on crack and color patterns; (ii) the temperature may not be enough to reset the thermoluminescence (TL) signal; (iii) mechanoluminescence can be induced because quartz is harder than ceramics. The problem to date heated materials using quartz grains was already considered. Gösku et al. (1989) used the thermally activated sensitivity change of the 110 °C TL peak to estimate the heating temperature experienced by flints. Falguères et al (1994) proposed that the electron paramagnetic resonance (EPR) signal of E' center can be an indicator of poor initial zeroing in quartz grains found in volcanic samples. Michab et al. (1998) suggested that datable pebbles are those that exhibit in natural TL curves a distinct peak near 300 °C, in addition to that one near 400 °C. Although it is assumed that HF etching removes the near-surface layer of coarse-grained quartz, the consequence of grinding process was not considered in dating protocols of burnt pebbles. Thus, combining TL and EPR techniques, the aim of this study is to improve the methodology to dating burnt pebbles with the option to estimate the temperature in which the specimen was fired and ensuring that the stored signal is not affected by the sample preparation.

Experimental: All pebbles were collected 70 cm below the surface of a remaining stratigraphic block of the Boqueirão da Pedra Furada shelter (São Raimundo Nonato, Piauí, Brazil). This site assumed major importance to South America peopling since the discovery of well-structured hearths associated with lithic tools in levels dated at 25 ka BP (Guidon and Delibrias, 1986). Based on surface signs, the pebbles were classified in three categories: burnt, unburnt and unidentified. Unburnt pebbles were used to create a burn pattern by heating each specimen at temperatures ranging from 200 and 600 °C (2 h) in a muffle furnace. Each pebble was ground with impact strokes using an agate mortar. The grains between 75 and 150 µm were etched in HCl and HF solutions. Each sample was divided into three aliquots that were irradiated with 10 Gy of ⁶⁰Co source. The TL glow curves were recorded with a Harshaw 3500 reader. The EPR measurements were performed with a

Bruker EMX+ spectrometer (X band) at room temperature. The same procedure was applied for burnt and unidentified pebbles.

Results and Discussion: The intensities of TL and EPR signals are shown in Fig. 1. The 110 °C peak is not observed in those pebbles that were heated at temperatures ≤ 400 °C but its intensity increases when the heating temperature is higher than 400 °C. The EPR signal of the E' center decreases with the increase of the temperature and the signal disappears above 450 °C. The intensities of the peaks appearing near 300 and 375 °C are not related to the heating history of the pebble. TL and EPR signals of previously classified burnt and unburnt specimens appear in well agreement with the values related to the burn pattern. It was also possible to reclassify the unidentified specimens. Thus, it is concluded that both TL peak at 110 °C and E' center can be used to identify the temperature range in which the pebble was heated in the past. To verify if the sample preparation procedure was affecting the stored TL signal, an unburnt pebble was sawn into two pieces and one of them was irradiated with 24 Gy. The accumulated dose of each piece was determined by the method of regenerative doses. As a result, the value of natural dose plus 24 Gy was retrieved from the calibration curve with a satisfactory degree of confidence.

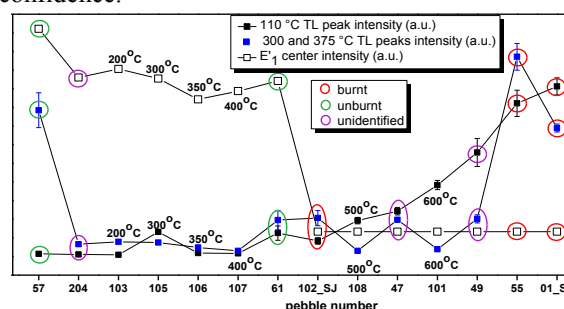
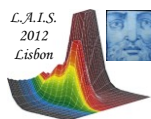


Fig. 1. TL and EPR signals of burnt and unburnt pebbles.

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THERMOLUMINESCENCE DATING OF CERAMIC VESTIGES FROM THE ARCHAEOLOGICAL SITE OF ALDEIA DO CARLOS - PIAUÍ, BRAZIL

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Introduction: The National Park of Serra da Capivara (PNSC) located in the South-East of Piauí State (Brazil) yield almost 400 archaeological sites, many showing parietal paintings dated to the Holocene (Pessis, 2000). The hypothesis supported by the excavators that some sites were occupied during the Pleistocene opened the interest of the international community to this area, which has been the subject of several investigations since 1986. Besides rock paintings, several sites showing fragments of ancient pottery were discovered and some of them were not dated until now. One of these sites is Aldeia do Carlos, where more than 54,000 fragments were collected since the discovery in 2006. The aim of this study is to carry out the thermoluminescence (TL) dating ceramic fragments collected at different depth levels from Aldeia do Carlos site.

Metodology: The accumulated doses of natural radiation were determined by the method of regenerative doses applied to coarse quartz grains (Aitken, 1985). After removing the superficial layer of each fragment, aliquots of quartz grains classified between 75 and 150 μm were prepared using the standard procedures of grinding, sieving, drying and chemical etching with HF and HCl. The suitable conditions of TL reading were established after performing plateau and pre-heat tests in one aliquot of each fragment. The TL response curves were obtained by administering gamma doses ranging from 0.5 to 10 Gy delivered from a Co-60 source with a dose rate of 4.171 Gy/h. The TL glow curves were recorded using a Harshaw 3500 reader with a heating rate equal to 4 $^{\circ}\text{C/s}$. The intensity of TL emission was evaluated by integrating the area under the peaks appearing from 200 and 375 $^{\circ}\text{C}$ and was normalized to the aliquot weight. The annual dose rate of natural radiation was determined considering the external and internal doses. The assessment of external doses was carried out by gamma spectrometry (HPGe detector) estimating the concentrations of U-238, Th-232 and K-40 in sediments collected nearby the fragments. In order to assess the internal doses, the concentrations of these radionuclides were measured

after samples extracted from different ceramic fragments being activated by a flux of neutrons ($10^{12} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$) during eight hours.

Results: All TL response curves showed a linear fitting factor (R^2) better than 0.960. The values determined for the accumulated dose varied from 0.82 ± 0.08 to 3.60 ± 0.15 Gy. The values of annual dose rate varied from 1.93 ± 0.12 to 3.12 ± 0.26 mGy/year. Using these values, it was possible to estimate the ceramic fragment ages, as shown Table 1. These results show that the ages correspond to three periods: 300-400, 600-800 and 1100-1200 years. No clear relationships were found between deepness level and surface finishing with the ages of pottery fragments. These results are in agreement with the ages found in other sites of PNSC in which ancient pottery vestiges were found.

Table 1: Age values.

<i>Ceramic fragment reference number</i>	<i>Age (years)</i>
179703	466 ± 57
179709	1056 ± 103
179713	428 ± 48
179718	1183 ± 101
179720	958 ± 117
179728	627 ± 83
179732	519 ± 47
179733	1175 ± 116
179741	359 ± 35
179743	317 ± 34
179746	730 ± 70

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Pessis, A. M. (2000) Pré-história da Região do Parque Nacional Serra da Capivara. In *Pré-História da Terra Brasileira*, M. C., Tenório (ed.), 61-72, Editora UFRJ, Rio de Janeiro.

AN ATTEMPT TO DATE BY OSL THE IMPLANTATION OF MEGALITHS: THE CASE OF THE STANDING STONES OF LE GÂVRE (FRANCE).

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The site of the stone row of the Pilier is located in the northern area of Le Gâvre forest (Loire-Atlantique, France). 94 monoliths, some of which standing, some others laid down or broken, line up on a 900-meter distance (Fig 1), on a NW-SE main axis with perpendicular structures on the north. The rock blocks are mainly made of white milky quartz. Some of the stones display dark schistose enclaves often cut by a number of quartz stringers. More seldom are the blocks mainly made of grey-green schist but also striated with white quartz veins.



Figures 1 & 2: The line of standing stones in the forest of Le Gâvre; the excavation hole for the sampling.

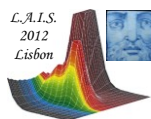
The bottom of some blocks from the western area of the row was sampled with a core-drill bit water-lubricated (Fig 2), during night-time to avoid the exposure to day-light. Sediments from the layout pits were also taken with opaque tubes. Finally a wedge stone was recovered from a pit.

In this study, the event to be dated was the layout of the blocks, i.e. the last day-light exposure of the sediments (during their shuffling) or the bottom of the steles (during their layout in the pit). It was one of the first attempts in France to date the layout of standing megaliths.

The idea of surface dating by OSL – the light penetration being limited, e.g. only few millimetres – is not new (Liritzis & Vafiadou, 2005; Liritzis *et al.*, 2007^o; Liritzis, 2011). However dating by OSL the last sun-light exposure of rocks is still a methodological challenge due to the samples' dependence on the bleaching process (Tribolo *et al.*, 2003). In this study, the aim was to evaluate the feasibility of OSL surface dating in order to date directly the event related to the erection of the megaliths. A standard OSL reader was used to analyse simultaneously the luminescence from several hundreds of grains. However the quartz grains of 3 steles and a pseudo-wedge stone did not provide any natural or regenerated luminescence signal sufficient enough to be used on a dating purpose. Therefore it seems that these particular veined quartz are not workable in OSL and cannot be used for an attempt of surface dating. An alternative was nevertheless performed in dating the sediments deposition of a pit filling, from which the quartz grains emitted an OSL signal. The date range of the sediment ([2040 ; 1680] BC) was in good agreement with the stratigraphic sequence and C14 dating applied on charcoal from a pit ([1752-1628] cal BC).

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LUMINESCENCE AND ELECTRONIC PARAMAGNETIC RESONANCE PROPERTIES OF PREHISTORIC CERAMICS FROM AL-KHIDAY EXCAVATION SITE, SUDAN.

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The archaeologic El Sahla Project funded by IsIAO (Istituto Italiano per l'Africa e l'Oriente) aims at the study of Sudan prehistory, whose testimonies are quite rare in the area. A number of ceramics excavated at the Al Khiday site, located in central Sudan, South of Khartoum, associated to Mesolithic and Neolithic phases, have been submitted to absolute dating.

The dosimetric results obtained using both Thermoluminescence (TL) and Optically Stimulated Luminescence (OSL) are reported. They regard the evaluation of the radiation dose absorbed by ceramics since their manufacture. Analyses were performed by using different techniques [*fine grain* (Zimmermann, 1971) and *inclusion* (Fleming, 1970)] and protocols [MAAD, Multiple Aliquot Additive Dose, Zimmermann, 1971, and SAR, Single Aliquot Regenerative Dose (Murray and Wintle, 2000)]. Such approach was attempted in order to possibly circumvent the complications due to the onset of TL saturation exhibited by most samples.

For the Neolithic ceramics, the results obtained with TL-MAAD protocols were in agreement with those of the OSL-SAR protocol. For the Mesolithic samples, the results obtained with the two techniques were quite scattered. Possible reasons of such disagreement are discussed.

Attempt was also made of using Electronic Paramagnetic Resonance (EPR) as complementary dosimetric technique. EPR is a method commonly used for dosimetric measurement on irradiated materials (Regulla, 2005), applied for age determination of archaeological materials. It is based on the measure of the paramagnetic defects created by natural ionizing radiation and accumulating with time (Jonas, 1997). However, the application of CW-EPR (Continuous Wave EPR) on pottery dating has been prevented by the

superimposition on the weak lines of such defects, of the much stronger Fe(III) broad signal. The application of EDEPR (Echo Detected EPR) has proven to be a suitable tool to spot out radiation induced signal from the iron background. This is possible since Fe(III) signal is characterized by short relaxation times, decaying shortly after the microwave pulses, while the radiation induced defects, with their much longer relaxation times, can still be detected (Zoleo et al., 2011). It was possible to record the natural EDEPR signal of a few samples. An overestimation of the absorbed dose was observed. In order to understand the origin of such overestimation, different tests as chemical treatments and annealing procedure were performed.

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DATING BRICKS FROM THE CRYPT OF THE CHARTRES' CATHEDRAL BY OSL: THE IMPORTANCE OF INTERNAL RADIOACTIVITY OF QUARTZ GRAINS.

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Within the framework of a European research program on ceramic building materials, the crypt of the cathedral of Chartres was studied. The aim of the dating campaign was to establish the chronology of the first building stage, in order to determine if the remains currently preserved were contemporaneous with the first Christian community in Chartres (Heber-Suffrin and Sapin, 2010). Due to lacunas in archaeological and historical data it is difficult to attribute a precise chronological frame (between the 2nd and the 5th century AD) to these preserved remains.

Luminescence dating was applied on bricks. In addition, building archaeology study was performed on masonry structures, providing relative chronological data. A preliminary study based on chemical and mineral analyses (performed with SEM-EDS) and optical petrography, highlighted the predominance of cleaved sedimentary quartz grains. A specific sample preparation for bricks (H₂SiF₆+HNO₃ mixture for matrix digestion followed by HF etching of quartz) was applied to select and treat the 125-200µm quartz inclusions (Bouvier et al., 2011). The equivalent dose measurement was performed using a SAR OSL protocol that derives from the classical ones (Murray and Wintle 2000; Wintle and Murray, 2006) by adaptating preheat and zeroing conditions (optical and thermal drainage of charges) to each sample. Dose rate calculation was obtained by combining gamma spectrometry and field TL dosimetry (CaSO₄:Tm) measurements.

The OSL quartz grains were found to be significantly radioactive. K, U and Th contents of grains were then measured by ICP-MS. The importance of this internal radioactivity is discussed and calculated.

The results obtained showed a noticeable variability of luminescence properties among samples (sensitivity, preheat dependence to thermal transfer ratio) which is probably due to the fact that this material was highly heated during brick making. An important sensitivity to alpha irradiation was observed. Nevertheless, the ED measurements were very precise (around 2%) and the dates obtained allowed us to propose a new chronology for the crypt of the Chartres' cathedral, that is very different to the

initial expectations. Two different brick production periods were identified, one around the 7th-9th century AD and another a later one around the 12th-13th century. It highlighted the partial or complete reconstruction of the crypt, which was unexpected. The crypt appears to be a reconstruction, maybe after one of the numerous fires that destructed the cathedral, using building materials from the former building stages.

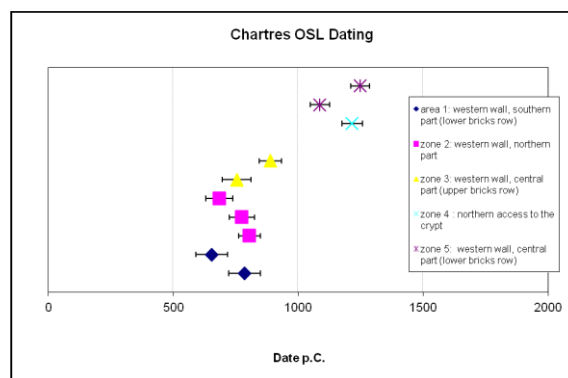
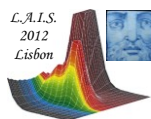


Figure 1: Dates obtained for the study of the crypt of Chartres' cathedral.

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THERMAL CHARACTERIZATION AND TL-DATING OF REDDENED WALLS OF THE CHAUVET CAVE (ARDÈCHE, FRANCE).

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The Chauvet Cave (Department of Ardèche, France) is a world-wide known site for its famous gravettian and aurignacian rock art representations, paintings and engravings (Clottes, 2001). We were interested in the numerous rubefactions and desquamations that are visible in many places of the cave (*Galerie des Mégacéros, Diverticule des Ours, Porche d'Entrée* ...).

We firstly wanted to know if the marks resulted from heating or ambiental temperature chemical alteration. Thus, four fragments of reddened limestone were sampled at different locations. In order to characterize their possible heating state, the thermoluminescence (TL) signals were studied, using the specific experimental conditions for calcite TL (Roque *et al.*, 2001). In a first approach, it was possible to know whether or not a sample was heated by evaluating the state of saturation of its natural TL signal (if the TL signal does not grow after a consequent laboratory irradiation, we can assume that it was not heated in the past). Secondly, reference samples were processed by heating for one hour at various temperatures samples cut in the same Urgonian limestone (a large piece of unheated material was sampled in the debris cone that fills the cave entrance) as the ornamented Chauvet walls.

The comparison of shape and intensity of TL curves of samples and those of thermal references allowed us to estimate the equivalent maximal paleotemperature attained (equivalent temperature depending on the thermal cycle characteristics chosen to make the thermal references). We showed that the reddened samples were heated in the past at more than 300°C, but below 400°C.

Thanks to these results, we could use these samples for TL-dating of the fire event that provoked the reddened marks. So, due to the low temperature archaeological heating of these samples, it was possible to perform a TL protocol that is very similar to the SAR technique generally

used in OSL. On site gamma spectrometry (NaI:TI) measurements allowed us to determine the environmental component of the annual dose. Laboratory measurements by high resolution gamma spectrometry of the limestone fragments being dated permitted us to determine the alpha and beta dose-rates. We observed a high enrichment of fragments by ²¹⁰Pb, witness of a sub-actual large excess of radon-222 inside the cave.

To determine the average annual dose, we used a model of accumulation of ²²²Rn in excess (radon) since the entrance had collapsed 22 ka ago, that gives a minimal age for the heating of walls by prehistoric people. This age is around 30 ka. In addition, a maximal age limit of 40 ka can also be calculated considering that the radon in excess does not account for a significant supplementary irradiation. These age limits are consistent with the calibrated radiocarbon dates (Valladas *et al.*, 2005) of charcoals used for the ornements of the walls and definitely demonstrate that the reddening of walls is the result of heating by prehistoric fires in the cave.

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OSL DATING AT PERDIGÕES ENCLOSURE COMPLEX (REGUENGOS DE MONSARAZ, PORTUGAL).

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The Os Perdigões archaeological complex, in the Evora district, South-Central Portugal, is a focus for multidisciplinary archaeological research. Its major structures are concentric ditches encompassing a shallow natural amphitheatre with an area of c. 24 ha. Initial finds placed Os Perdigões in the late 4th - late 3rd millennia BC (Chalcolithic) but, as investigation has progressed, a wide range of other prehistoric structures have been identified. Luminescence investigations have contributed to this process since 2005.

The preserved archaeological sequences at Os Perdigões are encountered in negative features. These were commonly cut into the friable weathered diorite-gabbro bedrock (in contrast to the granodiorites and tonalites bounding the site) but near the centre of the amphitheatre some were cut into localised areas of deep regolith. Coluvial and slope-wash movement across the site appears important in the accumulation of soils/sediments in the archaeological features.

The present study reports and compares chronological indications from different combinations of luminescence analyses: dating of 17 ceramic sherds and 30 soil/sediment samples, and luminescence profiling of 2 sequences, to make detailed assessments of chronological sequences and for survey of newly uncovered features. These are applied to a range of types and scales of structure in three sectors of the Os Perdigões complex, extending from the centre to the edge of the enclosure.

Absorbed dose estimation for absolute dating was made using SAR OSL on 90-160 µm quartz grains obtained from soils/sediments and ceramic sherds. That for the small soil/sediment samples used in semi-quantitative profiling, was obtained for polymineral and quartz enriched fractions, using IRSL, post-IR OSL and post-IR&O TSL, in a regenerative protocol with a single calibration dose point. The average dose rate to the quartz grains from the dating samples was evaluated based on field NaI gamma spectrometry and instrumental neutron activation analysis at IST/ITN.

The relationship between field gamma and INAA results varies from the edge to centre of the site. Precipitated or weathered quartz grains/agglomerates were observed to produce low absorbed dose estimates. High residual OSL signals were larger in shallow and narrow structures. Different features and layers dated to the mid- and late-Neolithic (4800, 4400, 3400 BC), early- and late- Chalcolithic (2800, 2300BC), Bronze Age (1300BC), and late-Medieval periods (1600AD).

The combination of luminescence dating of soils/sediments and ceramic sherds, with stratigraphically detailed profiling provided the most powerful combination for dating both archaeological assemblages and their contexts of accumulation, and interpreting any differences between these. However, in highly dispersed datasets such as those obtained from Os Perdigões, key information in this respect was obtained by comparing distributions of results from stratigraphically matched samples of soil/sediment and ceramic sherds.

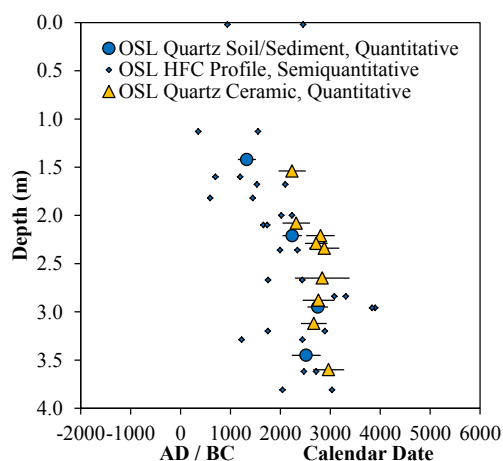


Figure 1. Results of OSL analyses from a section through the stratigraphy of the exterior ditch encircling the Os Perdigões site, in Sector L.

This work was supported by the FCT funded project PTDC/CS-ANT/104333/2008

LUMINESCENCE SIGNALS AND EMISSIONS FROM GRAINS OF QUARTZ PREPARED FROM PORTUGUESE GRANITE AND PEGMATITE.

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Quartz grains have been prepared from Portuguese granite and pegmatite samples, including repeated HF etching. The quartz obtained from the granites is transparent and exhibits clean crystal facets little attacked by the HF. That from pegmatites is milky and exhibits severe pitting.

Large (ca. 1 mm diameter) hand picked grains were measured using IRSL, post-IR OSL and post-IR&O TSL (500°C), in a regenerative protocol. The TSL response of each grain was then measured repeatedly, using different detection filters to observe emissions in the NUV, Blue, Green-Yellow and Orange-Red (Figure 1). The samples were then annealed for 1 hour in air and both measurement sequences repeated, for anneals of 600, 800, 1000 and 1100 °C, to examine changes induced by heating. RL and IL spectra from un-annealed grains of these samples were measured, for repeated X-irradiations and under continuous proton irradiation, to explore their emission characteristics in greater detail and provide indications of radiation quenching effects.

IRSL signals in the NUV were 3 orders of magnitude lower than OSL signals. TSL and RL emissions were strongest in the Blue, particularly for TSL at higher temperatures: lower temperature TSL emissions were also strong in the NUV. Both Blue and NUV RL emissions quenched strongly under X-irradiation. Peaks in IL emission were resolved in the NUV and Blue, but the strongest signals were observed in the Orange-Red: stronger for pegmatitic than granitic quartz. RL in this band was not radiation quenched, but was found to be weaker when a grain of heavily pitted pegmatitic quartz was split to expose un-etched crystal.

The TSL and OSL sensitivity of these geological samples increased by around 1 order of magnitude after rapid heating to 500°C. Initial OSL and low temperature TSL signals in the NUV sensitized by up to 4 orders of magnitude following 1 hr anneals. In granitic quartz the increase tended to be strong at 600°C but stabilised or reversed by 1100°C. In pegmatitic quartz the main activation was at 800°C and continued at higher temperatures. Sensitization of emissions in the Orange-Red was stronger in the pegmatitic quartz than the granitic.

In most emission regions the strongest TSL sensitization related to a peak around 150°C, which came to dominate the 100°C TSL peak in the pegmatitic quartz. Initial OSL signals sensitized more than the 100°C TSL peak, but their pattern of sensitivity change followed a combination of decline in the 300-340°C TSL region for anneals above 800°C, and the continued strong increase of the 150 °C TSL peak. The contribution of centres related to the 150 °C peak was evident as a slightly slower decaying component in the OSL signal, even after the trapped charge from this peak was preheated out.

Relationships between, and changes in, the different types of luminescence response, will be discussed in terms of the structural state of the quartzes and their expected trace element compositions.

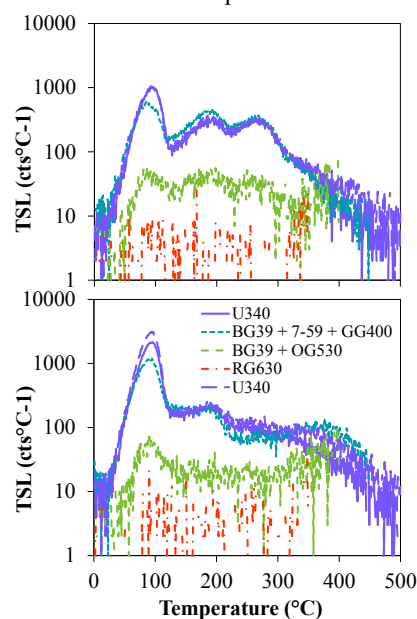


Figure 1. TSL responses of single grains of quartz from a pegmatite (upper) and from a granite (lower) in different detection bands, to 35 Gy beta doses.

This work was supported by the project italia128584682220330 Convénio Portugal (FCT) / Itália (CNR) 2011-2012. Burbidge recognises the support of the project PTDC/AAC-AMB/121375/2010 for dosimetric calibration.

VADOSE - A NEW PROJECT TO INVESTIGATE SPATIAL VARIATION OF DOSE RATES IN SOILS AND SEDIMENTS

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Determination of dose rates from naturally occurring radioactive materials (NORMs) is key to establishing their locations, concentrations and the effects on the environment of the ionising radiation they emit. These effects are applied directly to the luminescence dating of archaeomaterials and their contexts.

Natural materials generally contain heterogeneously distributed mixtures of radionuclides and absorbers of different types, producing complex mixed attenuated radiation fields. Radiometric and geochemical measurements of activity and concentration are conducted under specific experimental conditions using samples and geometries of defined scales. Transfer functions must be applied to assess radionuclide distributions, dose rates and /or implications for different conditions, scales and/or applications.

Established methodologies need to be reassessed experimentally, taking account of advances in instrumentation, understanding of radiation transport processes and computational capacity. The principal aim of VADOSE is to develop improved recipes for the simple, rapid and accurate prediction of dose rates at different scales in soils and sediments, through detailed experimental investigation and Monte Carlo modelling. The implications of this understanding and the information gathered for its development will be assessed in the context of methodologies in environmental dosimetry, and the significance of radiological and geochemical baselines.

VADOSE is an integrated and intensive dosimetric study combining instrumental and computational techniques. Radiologically contrasting environmental settings are being examined in terms of spatial variability of dose rate and differences in the radioactivity of subsamples of different grain size-fractions. Samples will be of well sorted sediments of dune sand and silt, and soils developed on granitic, limestone, siltstone and uraniferous dolerite lithologies. In situ radiometric measurements are being made by NaI gamma spectrometry, and

luminescence enclosure dosimetry. Mineralogy and major and trace element composition including radionuclides will be determined for bulk samples and different grain size fractions, using SEM, XRD, XRF, INAA and HPGe spectrometry, to evaluate radioactivities and radiation absorption characteristics. The experimental determinations will be used, with granulometric distributions, to calibrate radiation transport models of the alpha, beta and gamma radiation environments in the soils and sediments using MCNPX and proprietary codes.

Preliminary work has included the establishment of high resolution gamma spectrometry of non-neutron-activated samples in the GeoLuC group, for uraniferous samples from the study region (e.g. Trindade et al., this volume), and the modelling and testing of source geometries for the calibration of environmental dosimeters (Figure. 1, and Burbidge et al., this volume).

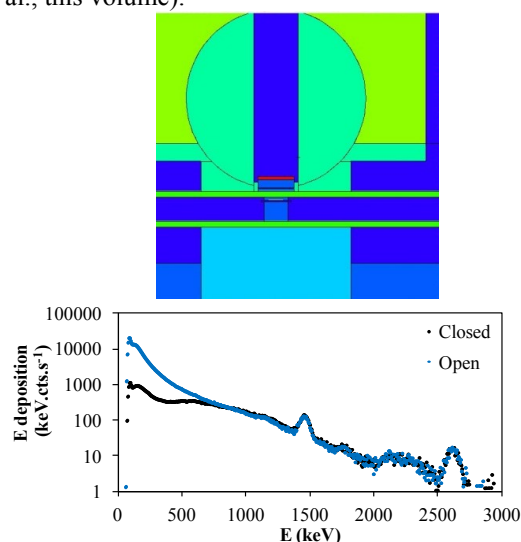


Figure 1. Detail of elements of a Riso automatic irradiator being used for modeling with MCNPX (upper). Measured photon spectra in front of the reader with source open and closed (lower).

This work was supported by the FCT funded project PTDC/AAC-AMB/121375/2010

BUILDING A DATABASE OF ABSOLUTE DATING IN ARCHAEOLOGICAL SITES OF THE IBERIAN PENINSULA: CONTRIBUTION TO THE STUDY OF TIMELINES SEQUENCES.

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Introduction: The construction of a database which allows published absolute dates to be compiled, organized and made available to the scientific community, is an important tool in archaeological research. Specifically designed and engineered for this purpose, the database for the Absolute Dates of the Iberian Peninsula (DAPI), includes the results collected from different dating methods available to the archaeological sites of the Iberian Peninsula.

Metodology:

This database was designed in Access, a program released worldwide and with a simple interface, while still allowing exporting to Excel, simplifying the visualization of data.

Its interface allows the user to view the data already entered by archaeological site or by printing the list of existing dates; view in Google Earth, the geographic location of the site; enables the insertion of new data or even change any erroneous data.

Results: With a new update by May 2012, the database references a total of 994 archaeological sites, 412 in Portugal and 582 in Spain. Currently, 35% are geographically referenced in Google Earth. This set of sites corresponds to 4294 absolute dates, obtained by different methods: radiocarbon (14C and AMS), U-series, paleomagnetism and luminescence (OSL and TL).

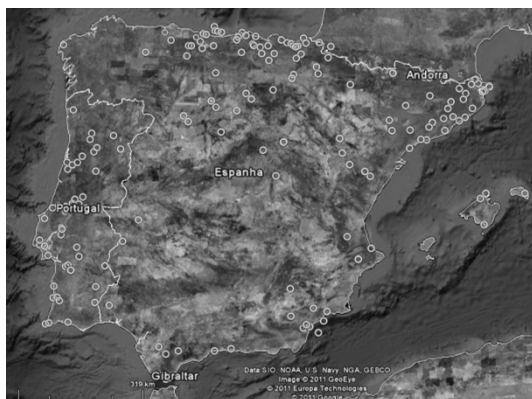


Figure 1: Pin point location of the archaeological sites in the Iberian Peninsula.

The main objective of the database is to facilitate identification and localization of absolutely dated archaeological sites, to contextualize ongoing work and aid the planning of future research. The volume of existing data already permits a degree of geographical and technological intercomparison. To illustrate database outputs, different cultures associated with the absolute dates (according the authors), are presented in a timeline for the most recent prehistory of the Iberian Peninsula, Portugal and Spain. The frequency of their occurrence is presented in intervals of 1000 years using the period of 12,500 BC (middle Dryas), as the "zero point" of the graphics and the midpoint of the calibration range of each absolute dating, presented in publications.

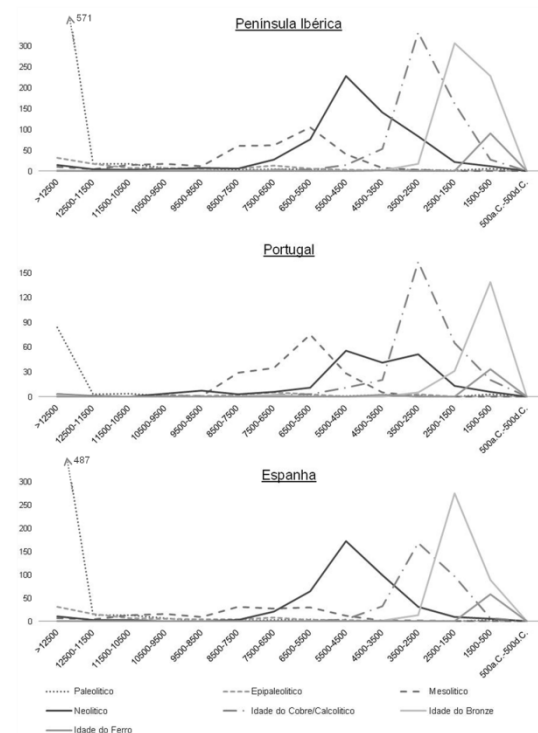
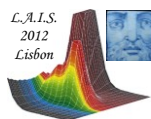


Figure 2: Absolute dates for different technological cultures in the Iberian Peninsula, for the period between 12,500 BC and 500 AD.



LUMINESCENCE LIFETIMES IN THE '600 -700°C' TRANSITION REGION FOR QUARTZ.

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Annealing has a profound effect on both the sensitivity of optically and thermally stimulated luminescence from quartz (Bøtter-Jensen et al., 1995; Rendell et al, 1994;) as well as on luminescence lifetimes determined from its time-resolved optically stimulated luminescence spectra (Chithambo et al, 2008, 2009; Galloway, 2002). In particular, luminescence lifetimes are independent of annealing up to temperatures between 500 and 600°C. The lifetimes then decrease thereafter as the annealing temperature is increased with the transition from the higher to the lower lifetime being more apparent between 600 and 700°C, that is near the first phase inversion point of quartz (Figure 1). We report on the characteristics of the principal and secondary lifetime components in this transition region. In extension of earlier studies, the effect of temperature, irradiation, duration of annealing, preheating, and wavelength of stimulation have been investigated in order to properly understand the phenomenon of the distinct change of lifetime and sensitivity to stimulation with annealing at temperatures close to the first phase inversion point of quartz.

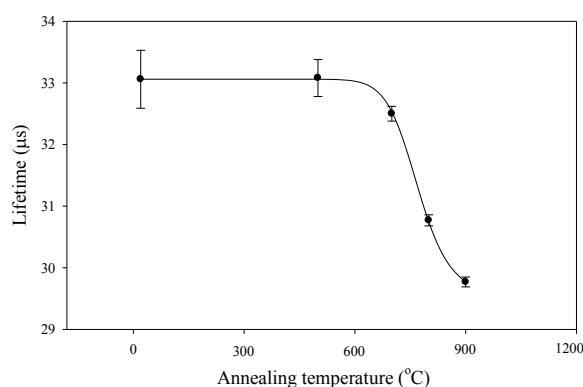


Figure 1: Luminescence lifetimes as a function of annealing temperature for measurements made at 25°C.

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INFLUENCE OF MINERALOGICAL COMPOSITION AND NATURAL RADIONUCLIDE CONTENT ON THE UV-THERMOLUMINESCENCE EMISSION OF BASALTS.

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Introduction: We herein report on the natural thermoluminescence (TL) emission of four lava flows from different volcanic regions that could potentially be useful for dating purposes. All the samples were characterized by means of: (i) x-ray diffraction to determine the components that act as main contributors of the TL response and (ii) gamma-ray spectrometry to identify the natural radionuclides that induce the TL glow curve. The observed changes in the natural TL emission of the samples depend, essentially, on (i) the mineralogical composition, (ii) the elapsed time from the last eruption, i.e. when the last zeroing took place and (iii) the radioactive content.

Content: One of the most interesting features of this material is the presence of mineral phases (mainly plagioclases) exhibiting TL emission. It is possible to appreciate different structures and contrasts associated with the presence of: (Fig 1a) hematite (Hm), dark inclusions into the bulk of the lava that comes from Costa Rica; (Fig 1b) montmorillonite, with a granular structure that has been detected in all samples, this figure corresponds to the Tenerife-2 lava flow; (Fig 1c) pyroxenes (Px), big inclusions close to 400µm observed in the La Palma sample and (Fig 1d) microcrystals of plagioclases (Pg) that appear also in all samples. This figure corresponds to the Iceland sample. In both Fig 1c and Fig 1d one can observe several fractures in the crystals that are a consequence of the very fast cooling rate from high temperature in the magmatic chamber (at 900-1200°C) to environment temperature.

The gamma-ray spectrometry results estimates that the ^{238}U values are between lower values than $6.33 \text{ Bq}\cdot\text{kg}^{-1}$ in the sample from Iceland and the higher value of $30.4 \text{ Bq}\cdot\text{kg}^{-1}$ in the case of La Palma. The ^{232}Th estimations are in a range of $3.06 \text{ Bq}\cdot\text{kg}^{-1}$ (in the Iceland sample) and $51.2 \text{ Bq}\cdot\text{kg}^{-1}$ (for La Palma lava). In the case of ^{40}K the activities are between $191 \text{ Bq}\cdot\text{kg}^{-1}$ in the Tenerife 2 sample and $564 \text{ Bq}\cdot\text{kg}^{-1}$ in the La Palma sample. The ^{238}U and ^{232}Th activity concentration in the samples are determined from the activity values of ^{234}Th and ^{228}Ac respectively. The ratio $^{238}\text{U}/^{226}\text{Ra}$ indicates that the samples show secular equilibrium for the Tenerife-2, Costa Rica and Iceland samples, but La Palma sample is not in

equilibrium as is observed with a value clearly lower than 0.9 (Fig 2).

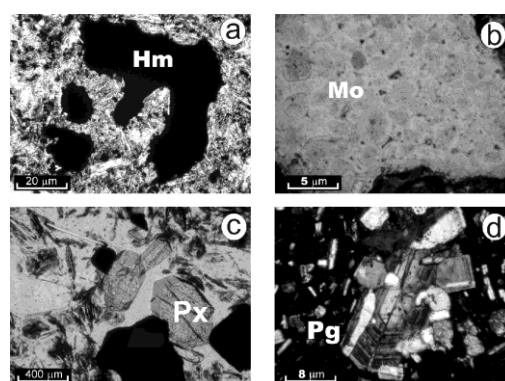


Figure 1. BSE-SEM image of an area of lava flows from (a) Costa Rica, (b) Tenerife, (c) La Palma and (d) Iceland.

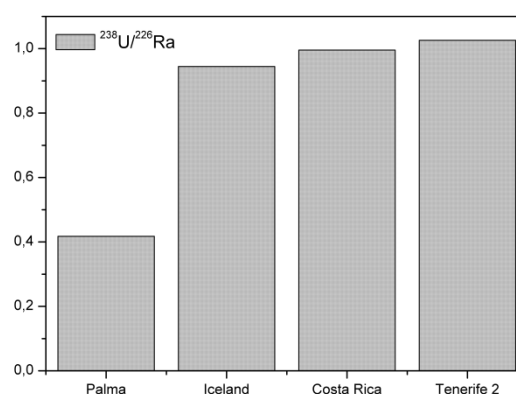


Figure 2. $^{238}\text{U}/^{226}\text{Ra}$ ratio of the La Palma, Iceland, Costa Rica and Tenerife-2 lava flows.

The UV-blue TL emission could be attributed mainly to the presence of plagioclase silicate since is the most abundant in all of the studied basaltic rock samples and, usually, feldspars exhibit the most intense TL glow signals in nature. The strained structure of these materials includes (i) possible non-bridging oxygen, (ii) silicon vacancy-hole centres and (iii) Si-O bonding defects which are the responsible for this typical emission observed in all of the stressed silicates.

COMBINATION OF IBA TECHNIQUES TO STUDY ART-HISTORICAL OBJECTS USING A MICROPROBE.

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The ion microbeam analytical end-station adds a set of valuable analytical techniques for the community involved in the study and conservation of Cultural Heritage. With the ion microbeam it is possible to analyze the elemental composition (in point, line or areal maps modes) and perform structural studies of different objects, large or small, using Ion Beam Analysis (IBA) techniques in open air or helium atmosphere – i.e. without vacuum conditions – and without the need of sampling or any special preparation. In this presentation, the details concerning the external beam set-up and a selection of the results obtained from selected analyzed objects will be presented.

Generally speaking, a beam of high energy particles (1 – 3 MeV) is directed towards the sample under study. Upon interaction in the sample, the beam induces the emission of secondary radiation (X-ray, light, gamma-ray photons and particles): depending on which we choose to explore, there will be associated a specific IBA technique: PIXE, IBIL, PIGE, RBS, etc. Each can provide different information, but what is really remarkable is the information that can be extracted when the techniques are used in combination. PIXE is very sensitive for elements with Z number higher than 12 (to the $\mu\text{g/g}$ level); from RBS spectra we can determine the elemental depth profiles; IBIL is a spectrometric technique where the luminescence signal is induced by the beam. It is sensitive to the local chemical environment of compounds and trace substitutes, very suitable to study the active optical centers usually found in pigments and mineral crystals. PIGE follows a similar mechanism operating at nuclear level.

The applications of IBA techniques to the study of Cultural Heritage objects are as varied as the objects themselves. Until now, different objects were studied using the microprobe (under vacuum and also in external conditions) located at ITN-IST [1]. Some examples are tiles, stained glasses, jewellery,

paintings and ceramics (figure 1) showing the versatility of the set-up since different detectors, software and experimental conditions were used to study each specific case. Also they can be used as complementary technique to the diagnosis of pathologies, for example in ceramic tiles.

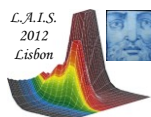
Knowing the composition of an object is extremely important for conservation purposes, dating or attribution processes, etc. considering that along centuries the fabrication procedures and the elements used were modified. Furthermore, the trace element concentration can, in some cases, indicate the provenance or relate it with other objects of the same type. Finally, the possibility to analyze and identify corrosion products is also important, in order to better understand the mechanisms of degradation, which is essential for their preservation for the present and future generations.



Fig. 2: Photograph during the experiments with the proton beam using the external set-up. Object: Roman glass.

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[1] Corregidor, V., Alves, L.C., Rodrigues, P.A., Vilarigues, M. and Silva, R.C., *e-conservation magazine*. 22 (2011), 40-5



A NEW MEASUREMENT SYSTEM FOR LUMINESCENCE DATING – LEXSYG.

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A highly flexible modular system for a variety of luminescence measurements for dating application and luminescence research was developed. The LEXSYG systems provide standard measurement options like Thermoluminescence (TL), Optically Stimulated Luminescence (OSL) and Infrared Stimulated Luminescence (IRSL), but also Infrared Radiofluorescence (IR-RF) and spectral, as well as spatially resolved measurements are possible. A basic measurement chamber can be equipped with a variety of modules for irradiation, luminescence stimulation and detection.

The measurement chamber has a capacity of 80 cups/discs and is designed for vacuum/inert gas use. Samples are stored independent from the measurement section and hence sample change is possible at (nearly) any time, while radiation cross-talk, a common problem affecting the accuracy of SAR luminescence protocols, is avoided. The Beta (Sr-90) irradiation unit provides a homogeneous irradiation field with deviation across the sample area (10 mm diameter) of $< \pm 3$ (about $\pm 2\%$ at 8mm) and allows RF imaging (8mm). Sample heating (TL/preheat) can be performed up to 700°C. There are several possibilities of OSL excitation by lasers and LEDs (850nm / IRSL; 532nm & 475nm / green & blue OSL; 405 nm / UV) which homogeneously illuminate the sample area for the stimulation of quartz, feldspar and other minerals. The excitation units can be used independently/simultaneously in CW or modulated mode. The instrument can hold up to 4 detection positions. PMT (bi-alkaline and an IR-sensitive cooled tubes), cooled EMCCD or CCD

based detection units are available. While the PMTs are used for conventional (small) aliquot measurements, the EMCCD is suitable for spatially resolved (resolution c. 16 $\mu\text{m}/\text{Pixel}$) / single-grain measurements of hundreds of grains at once. Up to two automatic optical filter changing units adapt each relevant PMT or imaging optics (EMCCD) at the same time. Luminescence detection windows can be set from UV to NIR and new optical filter-combinations have been tested for particular emission wave-bands and luminescence types of feldspars and quartz to perform high efficiency luminescence measurements for experimental work and routine dating applications. Furthermore detectors can be changed automatically between detection position, to some extend also within a measurement sequence, allowing a maximum flexibility and high throughput. A solar light simulation unit allows mimicking of different natural bleaching modes with variable wavelength spectra of samples within the chamber.

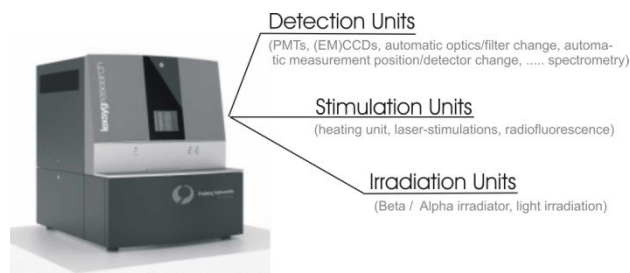
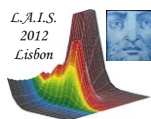


Figure 1: LEXSYG luminescence measurement system.



CHRONOLOGY OF NEANDERTHAL BURIAL SITES IN SOUTH-WEST FRANCE: PRELIMINARY IRSL DATING RESULTS.

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The Aquitaine basin, in SW France, has been known for over a century for the wealth of its Middle Paleolithic sites (300 - 35 ky) which have also delivered a significant assemblage of Neanderthal remains. This quite favourable area for prehistoric and paleoanthropological research relates to the widespread presence in the region of rock shelters and caves hosted in limestone formations which were locally dug and shaped by rivers and then partly filled by Quaternary deposits. This context allows a well preserved archiving for the studies of Paleolithic tools and for studying subsistence strategies of human cultures and their climatic and environmental settings. An absolute chronological framework and a review of the archaeological and environmental contexts would for many sites allow researchers to specify the temporal evolution of Neanderthal cultures in the southwest of France. The purpose of the present study is to establish a new chronology for several major burial sites: Roc de Marsal (Charente), La Quina (Charente) and La Ferrassie (Aquitaine).

The first known European Neanderthal burials belong to the Mousterian culture, around 60-40 ky, according to most commonly used relative dating techniques which also correspond to the age-range limit of ¹⁴C dating method. Furthermore, karst domains and particularly Roc de Marsal are known to be complex with a view to applying luminescence dating method because of microdosimetric heterogeneities (Guérin et al., in press; Guibert et al., 2009). In order to overcome those problems, it was decided to employ the IRSL dating method on feldspars. This has the advantage of being less sensitive to the radioactive heterogeneities due to their internal radiation dose. Currently, the most important challenge in the IRSL dating method is to take into account or to overcome the phenomenon of anomalous fading. In this paper we test procedures proposed for anomalous fading correction by comparing the effectiveness of IR and post-IR IRSL protocols based on the work of Huntley and Lamothe (2001), Buylaert et al. (2009) and Thiel et al. (2011) with an independent age control.

We have used a classical method to calculate the annual dose from radionuclide concentration (U, Th and K) on the sediment samples using a high resolution gamma-spectrometry and from in-situ measurements. Radioactive disequilibria in the U decay chain were accounted for when they were observed.

The results already obtained are encouraging and will be presented at the symposium. They promise an immediate bearing upon our current understanding of Pleistocene chronology and provide a basis for discussion over the issue of the oldest Neanderthal burial in France.

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ATTEMPT OF DATING QUARTZ-RICH MOSAIC GLASS USING THE PRE-DOSE TECHNIQUE

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In a recent study on the XVI century mosaics of the vault of St. Peter's Dome in Rome, (Galli et al., 2011) it was found that the presence of crystals of calcium antimonate and cassiterite dispersed in the glassy matrix allowed to evaluate by luminescence the radiation dose accumulated in the sample during its archaeological life. The accuracy was good enough to identify the XIX century restorations. In that case, the dosimetric evaluations were done using the single aliquot regeneration method (SAR, Murray and Wintle, 2002).

In the present work we show the results obtained from further mosaic tesserae of the walls of the Hosios Loukas monastery, near Athens (Greece), in which the only detected crystalline phases were quartz and cristobalite, the high temperature polymorph of silica (Arletti et al., 2011).

For TL measurements, the samples were sliced and crushed to obtain 4-10 micron diameter grains.

All the samples showed a natural signal with two low temperature emission peaks centered at 150°C and 200°C respectively, plus two emissions at about 300°C and 380°C (Fig.1).

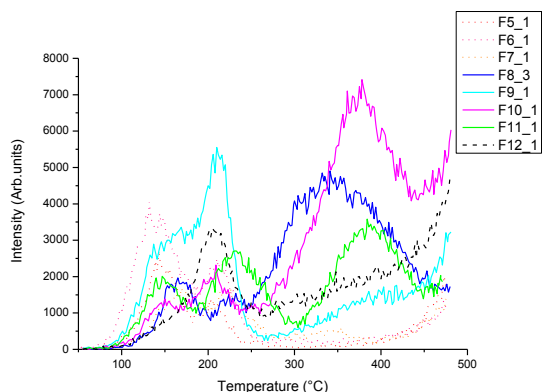


Figure 1: Natural TL glow curves of mosaic samples

Artificially irradiated samples, promptly measured after irradiation, exhibited the glow curves typical of quartz, dominated by an intense TL peak centred at 100°C. To evaluate the equivalent dose, the Pre-dose technique was therefore used. It is a dosimetric technique specifically developed for quartz grains extracted from *young* samples (Bailiff, 1994), exploiting the variation of TL sensitivity of the low

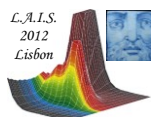
temperature peaks as a function of absorbed dose combined with thermal treatments

Problems arose in correctly calculating such a dose, because of the difficulty in separating the signal due to the vitreous component, strongly affected by anomalous fading, from that of the quartz micro-inclusions. Another difficulty was the low initial TL sensitivity of the 110 °C peak. The TL response to the small dose used as test-dose in the standard protocol (typically a few hundred of mGy) could not be detected. We tried to circumvent this problem using a simplified protocol previously described (Galli et al., 2006), in which the test-dose and the activation dose are unified. Measurements were made reading the TL emissions of the same aliquot subsequent to repeated irradiations with relatively high dose (usually between 0.5 and 1 Gy) until the saturation of the effect. For each sample, several evaluations were made (15-25, depending on the availability of sample), and the distribution of the data was statistically treated to check the reliability of the results. The measured values were in the range 1-3 Gy.

Once measured the radioactivity concentrations to evaluate the annual dose-rates, the dates obtained showed a satisfactory agreement with the know ages.

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TOWARDS A CHRONOLOGICAL FRAMEWORK OF MIDDLE PLEISTOCENE HUMAN SETTLEMENTS IN SOUTHWESTERN FRANCE THROUGH THE USE OF THE TT-OSL DATING METHOD APPLIED TO SEDIMENTARY QUARTZ.

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The southwestern (SW) France has yielded numerous remains attributed to the late Middle Pleistocene period. The analyses of archaeological material revealed a high degree of variability in lithic industries across the region. This raises many questions related to cultural influences and/or adaptation to local lithic raw material sources. Although this documentation is of major importance for understanding Acheulian and early Middle Palaeolithic cultures, those sites suffer from a lack of reliable chronological data (Turq *et al.*, 2010).

Middle Pleistocene archaeological sites in this area are associated with alluvial terraces or karstic contexts. Consequently, radiometric methods such as K/Ar, Ar/Ar, U/Th, could generally not be used because of a lack of suitable material for dating. Moreover, the expected ages of the deposits are beyond the upper limit of the dating techniques usually applied in such contexts (C-14 and luminescence). Nevertheless, in the last few years studies have been undertaken on a thermally transferred OSL signal (TT-OSL) offering the potential to date Middle Pleistocene sediments (Wang *et al.*, 2006). The present study aimed at developing this approach for the dating of four archaeological sequences in SW France: the Vaufray cave in Dordogne, the aven of Coudoulous in the Lot and two open air sites, Duclos and Romentères, in the southern Aquitaine Basin.

In a first step, the reliability of SAR TT-OSL protocols was investigated. These experiments revealed significant sensitivity changes (between the first and following cycles) which cannot be corrected by any normalization procedure (Hernandez *et al.*, *in*

press). Therefore, a broadly applicable protocol based on a regenerative growth curve build up from multiple aliquots previously bleached was implemented. This protocol circumvents the problems induced by SAR, while preserving the advantage to plot numerous natural TT-OSL signals and interpolate them with the growth curve, and thus, get as much equivalent doses (De). The distribution of the De values will be discussed in order to define possibilities for identifying the best bleached grains.

Finally, the bulk of TT-OSL dating results will be compared to chronological informations provided by geomorphology and biochronology studies and/or determined by chronometric methods (ESR and TL).

In an archaeological perspective, this work will shed a new light on the chronology of each of the studied sites, and consequently, on the evolution of lithic industries in SW France during the Middle Pleistocene.

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COMPARISON OF OSL AND ¹⁴C AGES ON THE ARCHAEOLOGICAL SAMPLES FROM KOREA.

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OSL dating on sedimentary quartz grains from two archaeological sites in Korea, called Hahwagye-ri and Hwadae-ri, was undertaken to aid archaeological interpretation for the migratory routes of the Upper Paleolithic and Mesolithic people in Korea. The Hahwagye-ri site is located on a river terrace of the North Han river at Kangwon prefecture and the Hwadae-ri is located on a river terrace of the Imjin river at Gyeonggi prefecture, both in the central region of Korean peninsula. From the results of excavation conducted for recent two years, archaeologists strongly suggest that these areas would be an ideal camping place with relatively long-term residence for the Paleolithic and/or Mesolithic foragers to get food resources by means of collecting, fishing and hunting. In order to check the quartz OSL results on the sediments at those excavation sites, radiocarbon dating was also carried out on a small series of finely layered charcoal samples. The OSL ages obtained showed a good stratigraphic correlation and were found to be in satisfactory agreement with those of the radiocarbon dating, implying that the incomplete bleaching of quartz grains, which is usually observed from fluvially deposited sediments, is not significant for these samples. These consistent ages also suggest that the quartz OSL dating can provide reliable age estimates in case that there are not enough materials suitable for other conventional tools for dating archaeologically important sediment layers. The resultant ages provide important information for the migratory routes of the Upper Paleolithic and Mesolithic people in Korean archaeology.

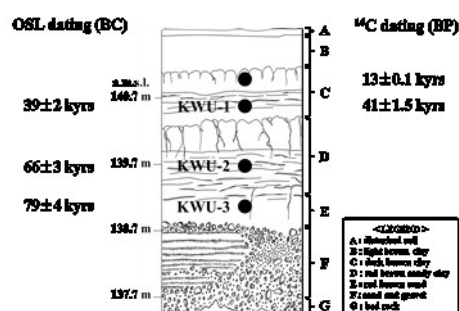


Fig. 1 A schematic diagram for sample collection and showing comparative radiocarbon and OSL ages for the Hahwagye-ri site

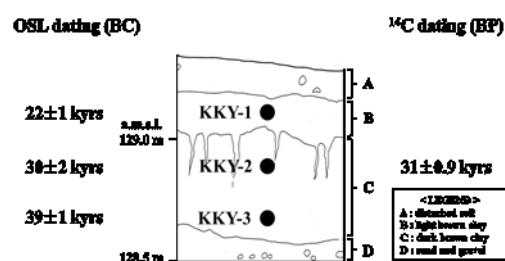


Fig. 2 A schematic diagram for sample collection and showing comparative radiocarbon and OSL ages for the Hwadae-ri site.

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APPROACH OF KOREAN PALEOSOL CHRONOLOGY USING OPTICAL DATING AND RADIOCARBON DATING: CASE STUDIES.

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In Korean Palaeolithic archaeology, most of Paleolithic sites are of open-air sites, so that the chronology of paleosol layers is very important. In this study we collected paleosol samples from two Paleolithic sites located in the central Korea. For the determination of those deposition dates and reliability, optical dating and radiocarbon dating were performed, respectively.

For optical dating, each paleosol quartz sample was chemically extracted and then divided into two groups; very fine sand-size quartz (63~125 μm) and fine sand-size quartz (125~250 μm). Each paleodose was obtained by using single aliquot regenerative dose (SAR) method. The optical bleaching and reliability of the deposit date of each paleosol layer were interpreted from the comparison between two resultant ages.

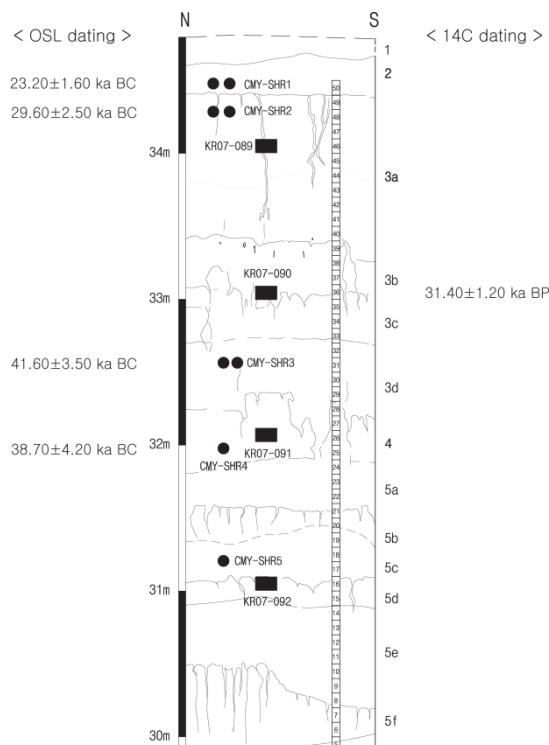


Figure 1: Summary of optical and radiocarbon dates measured at Suheol-ri Paleolithic site, Chunan.

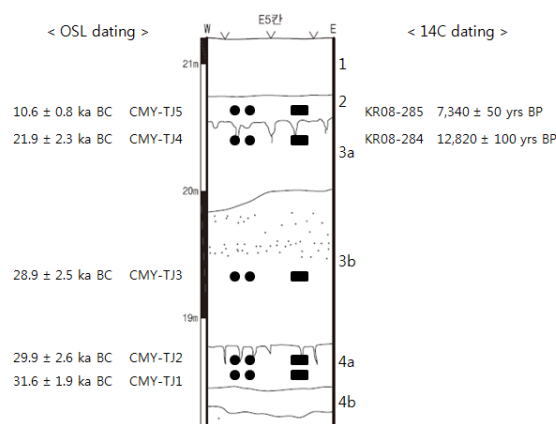


Figure 2: Summary of optical and radiocarbon dates measured at Yongdu-ri Paleolithic site, Asan.

For radiocarbon dating, both fulvic and humic acids were chemically extracted during the standard acid-alkali-acid (A-A-A) treatment. Humic acid was then divided into acid (HA), neutral (HN) and base (HB). After verifying the robustness of this pretreatment by using EA and FTIR analyses, four resultant samples were combusted and finally reduced to graphite for AMS measurement. The stability during the burial period and the reliability of paleosol deposit date were also turned out from the comparison with four resultant 14C ages.

Finally, the chronology of paleosol layers was concluded with the integration of optical and radiocarbon ages, geomorphology interpretations and archaeological contexts.

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AUTHENTICATION TEST OF A VARIETY OF KOREAN CULTURAL ARTIFACTS USING ABSOLUTE DATING METHODS.

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Introduction: In art, antiques and archaeology, authentication test was generally carried out to verify that a given artifact was made in a certain period of history or by a certain person. Apart from expert judgements, many scientific methods have been tried to distinguish the genuine cultural artifact from the modern counterfeit, as like absolute age dating, chemical analysis, provenance study, and so on. In this study we examined a variety of Korean cultural artifacts on authentication using absolute age dating methods and those related analyses.

Methods: Authentication of the cultural artifacts was examined as shown in Figure 1. For the fired clay artifacts in Figure 1 (a) ~ (e), TL/OSL dating was applied. Each quartz sample, grain size $\sim 200 \mu\text{m}$, was extracted using micro-sampling technique and the resultant paleodose was determined by single grain regenerative dose method (SGR). For the wooden artifact in Figure 1 (f), radiocarbon dating based on AMS measurement was performed. After the standard acid-alkali-acid (A-A-A) treatment, the small wood piece was combusted and then reduced to graphite for AMS measurement. For the iron artifacts in Figure 1 (g) and (h), proton induced X-ray emission (PIXE) analysis which is non-destructive method was carried out. The concentration of cosmogenic radionuclide ^{36}Cl was compared between the iron artifacts and a modern nail.

Discussion and conclusion: We studied authentication of several Korean cultural artifacts by using TL/OSL dating, radiocarbon dating and PIXE analysis. Considering the resultant ages and ^{36}Cl concentration, it is concluded that these cultural artifacts are all modern counterfeits, except for the brick related to Muryoung Royal Tomb of Baekje Kingdom.

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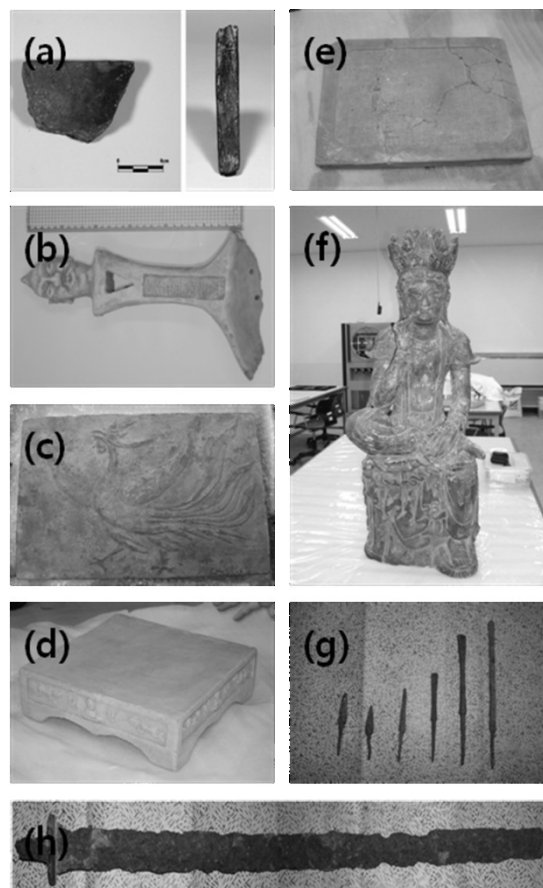
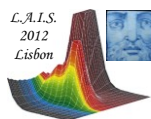


Figure 1: Photographs of cultural artifacts examined on authentication: a brick related to Muryoung Royal Tomb of Baekje Kingdom (a), Taebuhyanggi (b), Red bird birck, one of four guardian gods (c), table of a Buddha statue (d), inscription brick (e), wooden Buddha statue (f), Iron arrowheads (g) and iron sword (h).



GEO-ARCHAEOLOGICAL INVESTIGATIONS AT SOUSIKOU-LAONA (CYPRUS): PLACING THE CHALCOLITHIC SETTLEMENT AND CEMENTARIES IN A LANDSCAPE CONTEXT.

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The Chalcolithic settlement and cemeteries of Sousikou are one of the best known sites of Erimi culture in 4th Millennium BC Cyprus, containing many unique objects and artefacts, and evidence for the manufacture of the cruciform picrolite figurines. The complex is located on a spur formed by the confluence of the Dhiarzos and Argakin Vathin rivers in the southwest of Cyprus, with views west over the Dhiarzos to the Ktima lowlands, east to the Troodos mountains, and south to the Mediterranean Sea. In detail, the complex is dissected by a ravine, cut by the Vathyrkakas stream, which separates the settlement (located on an elevated spur) from the main concentration of graves (located on an elevated plateau to the east). Given its location, between the hilly uplands and interior of the island, and the flat lowlands, the position of the complex here is likely to reflect its environmental and spatial context. The placement of the settlement and cemeteries on opposite sides of the ravine has long intrigued the archaeologists working on this site; they have favored an interpretation in which the ravine was cut following abandonment of the site.

To understand the relationship between site placement and geology, geomorphology and environment, the authors have undertaken a range of geomorphological investigations in the wider region. This has involved the identification and correlation of a number of regional erosion surfaces (Kinnaird et al., 2011) and channel fills (Deckers et al. 2005), and the construction of a relative chronology to unravel the incision histories of the Dhiarzos and Argakin Vathin rivers. OSL dating and magnetostratigraphic studies have provided the chronological framework to this investigation.

The present-day drainage of the Dhiarzos and its tributaries was established by the late Pliocene-early Pleistocene; this 'initial topography' developed in response to progressive uplift of the Troodos Massif, combined with slightly lower base-levels related to eustatic sea-level. From then, through the Chalcolithic

period, to the second century BC, material was regularly flushed through the Vathyrkakas ravine, such that no detritus associated with the settlement is preserved in the axis of the ravine, or any early channel fills. During this period, the slopes of the ravine, on which part of the settlement was located, have evolved, as witnessed by the partial collapse of several structures. The earliest occupational layers in the settlement are intercalated with colluvial layers. The chalcolithic inhabitants carved hollows and pits out of the soft bedrock: hearths and plastered surfaces within these hollows attest to occupation. In this process, they de-stabilised the slopes, generating slope failures.

Initial luminescence profiling, using portable OSL equipment, was used to characterize the stratigraphy through a number of profiles examining these intercalated layers. Preliminary laboratory analysis, using a simplified 2-step SAR procedure, indicated that material in all units shared similar sensitivities and stored dose estimates, attesting to a short chronology. Quartz SAR OSL ages provide the chronology to interpret the late incision history of the Argakin Vathyrkakas ravine.

The geo-archaeological investigations have revealed how the inhabitants dealt and interacted with the geology, landscape and topography of their chosen location.

Deckers, K., Sanderson, D.C.W. and Spencer, J.Q. (2005) Thermoluminescence screening of non-diagnostic sherds from stream sediments to obtain a preliminary alluvial chronology: An example from Cyprus. *Geoarchaeology*, v. 20, p. 67-77.

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GEO-ARCHAEOLOGICAL INVESTIGATIONS OF THE PONT D'AVIGNON (SAINT BÉNÉZET BRIDGE): CHRONOLOGICAL CONSTRAINTS ON THE FLUVIAL DYNAMICS OF THE RHÔNE FROM EARLY MEDIEVAL TIMES.

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OSL investigations provide a chronological framework to reconstruct the fluvial dynamics of the Rhône River at Avignon (south France) through the late Medieval to early modern periods, and support geo-archaeological investigations of the Pont d'Avignon. Diachronic analysis of historic cartographic data has provided a relative chronology for geomorphic changes of the Rhône River, which with historical accounts of the bridge; provide a link between the construction, utilisation and abandonment of the bridge with historic flooding events.

Five boreholes were drilled through the west bank of the Rhône to a depth of c. 8m, in the vicinity of Villeneuve-lès-Avignon, in the position of a former palaeo-channel of the river. Detailed luminescence investigations were carried out on two cores, which contain (from top down) several metres of silty clay, interpreted as post-abandonment fills, several metres of sands, interpreted as late channel fills, and gravels at c. 7.35 m, reflecting the main fill. Initial luminescence profiling, using portable OSL equipment, was used to characterise the stratigraphy in each core (Sanderson and Murphy, 2010). It was possible to correlate between the lithostratigraphic units identified in each core on the basis of their luminescence characteristics (Figure 1).

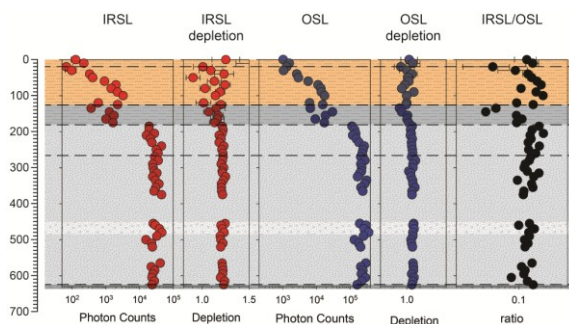


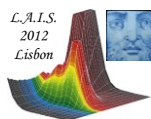
Figure 1: Luminescence intensities-depth profiles obtained using portable OSL equipment for the core recovered from borehole Vill3

Additionally, the field profiles indicated the parts of the cores that may be better suited for dating, and showed that similar signal levels might be expected above and beneath the main sand accumulation, suggesting a high rate of sedimentation and a relative short chronology. Laboratory analysis comprised luminescence screening measurements on 15 selected samples, to assess sensitivities and approximate stored doses, and quantitative OSL SAR analysis on 9 dating samples. The laboratory profiling results implied that the age progression above and beneath the sand accumulation was modest, reinforcing the notion that the chronology was short.

The individual dates fall into the late medieval to early modern period of French history, with statistical combinations pointing to an early to mid 17th century accumulation of sediment.

These dates are consistent with the interpretation that the sediment accumulation is associated with destructive floods, in the period associated with the end of the Little Ice Age and the warming period that followed it. It was during this period that the Pont d'Avignon was finally destroyed and that attempts to repair the broken arches were abandoned. The OSL investigations have indicated a compelling link between historic flooding events which led to the destruction of the Pont d'Avignon and the sediment deposited in over bank deposits as these floods retreated.

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TIMING AND DYNAMICS OF THE FIRST SETTLEMENTS IN AMERICA: NEW INSIGHTS THROUGH OSL DATING OF THE SERRA DA CAPIVARA ARCHAEOLOGICAL SITES (BRAZIL).

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When and how did human beings people the American continent? Were there different phases of peopling, and what were their origins? These questions remain partially unanswered. Our research aims at bringing a chronological precise and reliable framework to these matters.

Different theories have been in contradiction for a long time, and the paradigm of a post-11,500 years BP occupation remains predominant. Nevertheless new data seem to question the initial acceptance of a theory of a migration from Siberia to Beringia, and then from the North to the South of the American continent; these new data question maybe most of all the value of the terminus post-quem imposed by a chronological limit fixed to the Last Glacial Maximum (LGM) (Guidon and Delibrias 1986, Steele and Politis 2009, Valladas *et al.* 2003).

This theory identifies the populations culturally named as Clovis, and present in the North of the continent after ca. 11,500 years BP, as being the first inhabitants of the New World ("Clovis-first" theory). Within the discoveries that question this theory, most have been made in the southern part of the continent, particularly on the actual territory of Brazil. But until now some of the chronological data that have been deduced from these discoveries have not been accepted, because of problems linked to the methods themselves or specific problems of the studied archaeological sites, as for example taphonomic issues, and probably because of an ideological barrier.

In the face of this knowledge and the reasons that led the scientific community to reject some sites, we selected accurately some archaeological sites to bring new chronological data. Besides the imperative quality and reliability of the datings, it also appeared to be very important to study archaeological sites where there is no doubt about the human occupation. We also ensured the reliability of the data that can be deduced, verifying the absence of strong taphonomic problems and that no mixing between the levels could be suspected. For the chosen sites we checked the

absence of post-depositional perturbation and the quality of the sedimentary record.

The sites are all located in the Serra da Capivara National Park (or in the close vicinity) and are excavated by a Franco-Brazilian team within the framework of the project entitled "Research into the Pleistocene and Holocene peopling in Nordeste (Brazil). Spaces and times of the first men in Piauí".

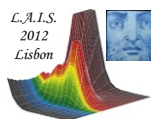
The Toca da Tira Peia for example is a few kilometres from Boqueirão da Pedra Furada (Guidon 1989), the anthropic nature of the assemblage of the site is attested with no doubt (tens of lithic artefacts, knapped in an allochthonous material, no foot of cliff), and the quality of the sedimentary record is attested as well. The dating results from Optically Stimulated Luminescence (OSL) of the two lower archaeological levels containing lithic material are clearly older than predicted by the classic theories. These results and other obtained for different archaeological sites from the same area (like « Vale da Pedra Furada » site, Baxão da Esperança, ...) will be presented and discuss. They all question the generally accepted chronological framework for the first peopling in America.

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UPDATING OF RADIATION DOSE-RATE CONVERSION FACTORS: A CRITICAL RE-EVALUATION.

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It has been about forty years since the dose rate conversion factors in tabular forms commenced to appear and often on a decadal period were updated. Past work was carried out by Aitken and Bowman (1975), Carrievau and Troka (1978), Bell (1979), Aitken (1985), Nambi and Aitken (1986), Liritzis and Kokkoris (1992), Ogoh et al (1993), Adamiec and Aitken (1998), Liritzis et al., (2001) and Guérin et al., (2011).

The present work presents an analytical up-dated tabular form of annual dose conversion factors per individual natural parent and daughter isotope, mostly used for luminescence chronology. Based upon revised atomic and nuclear radiation data regarding energies, intensities, branching ratios and half-lives for the U-238 series, U-235 series, Th-232 series, K-40 and Rb-87, the absorbed dose-rates are recalculated. Furthermore, for the first time, all the individual uncertainties involved in dose-rate calculations are taken into account and the associated total uncertainty is determined by error propagation.

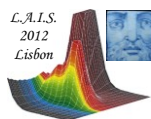
Comparison between all earlier nuclear data (1978 to 2011) and deduced conversion factors and a critical assessment of the decadal updating are made. Exemplary luminescence dating cases, incurred changes and their essential impact on the obtained chronologies by luminescence dating techniques are given for high/ low radioactivity contexts involved. The quoted discrepancies of the order of some percentages are thoughtfully reconsidered. An online electronic software is prepared for automatic calculation of dose rates on future updates of radiation data and derived ages thereof.

In fact, the present data are based on a compilation of earlier continuously updating values. Although the minimizing of systematic errors is a self-evident task, here the meticulous elaboration of dose rate conversions caused by the updating of U-238, U-235, Th-232, Rb-87, K-40 isotopic data, is revisited. The compiled isotopic series data and derived dose rates

for alpha, beta and gamma rays through the updating values are presented as a function of updating year, and their impact on age calculation is critically discussed. The worth of such periodical elaboration is re-evaluated. The present work is a revised overview that future luminescence (and ESR) dating applications should safely rely upon.

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ADVANCES IN SURFACE LUMINESCENCE DATING: NEW DATA FROM SELECTED MEDITERRANEAN MONUMENTS.

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Introduction: In rock surfaces the penetration of solar radiation (UV and optical spectrum) bleaches the optically sensitive electron traps in the carved surface down to a variable depth. In calcitic stones solar radiation can reach depths of 0.5-1 mm and up to 16 mm respectively, depending on the opaqueness, while for other igneous rocks fast bleaching occurs up to 5mm and almost complete in the first 1 mm within ~5 min (Liritzis et al., 2008). The surface dating has been developed and improved on various cases (see review by Liritzis, 2011). Laboratory measurements make use of standardised blue light stimulated, single-aliquot regenerative protocols, SAR, SAAD, MAAD (Vafiadou et al., 2007; Zacharias et al., 2009; Liritzis et al., 1997).

Results: The study reports on methodological advances and newly resulted dates from four sites:

a) *Saudi Arabia at Bir Hima (north of Najran)* a rock art sandstone covered by reprecipitated calcite lamina, which indicates the last wet conditions in the Arabian desert prior the onset of desertification. An age from the inter-face between the sandstone and the calcite encrustation was resulted. The age of sandstone surface indicates the last time was exposed to daylight due to wet conditions, about 1500-3000 BP.

b) *Delphi, Livadi valley, Mount of Parnassus*, a round threshing floor, obviously used by recent farmers but thought to form the foundations of an earlier ceremonial site, built out of large calcitic plates, and a

nearby test excavation that revealed foundations of a building. OSL ages reported on rock surface as well as of ceramic sherds not easily typologically recognised. The ages span indicates Bronze Age.

c) *Nuraghi megalithic monuments* in Sardinia at Sola Russa. In general, the rock types range from basaltic, to fossiliferous limestone with quartz. Preliminary OSL dates are reported during Late Bronze.

d) *Oseirion and Seti I Temple at Abydos, Egypt*. Granite and sandstone was used and the OSL ages span from XIX Dynasty to Middle Kingdom.

Methodological considerations, since the birth (1991) of surface dating, and the evaluation of the present ages of these important structures shall be discussed.

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IRRADIATION DEFECTS PRODUCED IN IBIL ANALYSIS OF SAPPHIRE.

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Introduction: Ion beams find a wide range of applications in materials science, from analytical techniques for composition and structural studies (e.g. RBS, IBIL or PIXE) to processing methods, such as ion implantation.

The nature of the interaction of accelerated charged particles with matter in any of these methods produces side effects in the irradiated materials, generally known as ion beam defects, which are usually undesired, particularly if the optical properties of the material are to be kept. These defects range from electronic imbalance to atomic displacement, and depend upon the characteristics of the irradiated material, the irradiation time and the nature (mass, charge and energy) of the impinging beam and typically present an optical signature, that is, characteristic absorption or emission bands.

In this work we study the time evolution of ion beam defects in sapphire produced by ion beam induced luminescence (IBIL) analysis with two different 1 MeV beams, He^+ and H^+ .

IBIL is the analysis of luminescence produced by solid samples during irradiation with energetic charged particles. In contrast to processes leading to the production of X-rays, in which the inner atomic shells are involved, the luminescence process is related to the transitions of the outermost electrons involved in chemical bonds of atoms or in lattice defects. For this reason, IBIL is sensitive to the local chemical environment of compounds and trace substitutes and to the microstructure of the network. The electronic optical transitions are induced by the secondary electrons scattered into the track by the impinging ion.

Sapphire is a crystalline form of aluminum oxide, an insulator and radiation resistant material. The main defect centers associated with irradiation damage are F-type centers, which are oxygen vacancies occupied by one (F^+) or two (F) electrons.

Figure 1 shows a typical IBIL spectrum of pristine sapphire. The defects signatures occurs in the UV – blue region and are assigned to the above mentioned F and F^+ centers.

As the ion beam probes the sample further defects are introduced. Figure 2 shows the evolution with time of these defects, comparing H and He 1MeV beams. From these analyses the dynamics of the formation of defects is discussed and the lifetime of

these defects is estimated to be around 400 s, in accordance with the work of Morono and co-workers¹.

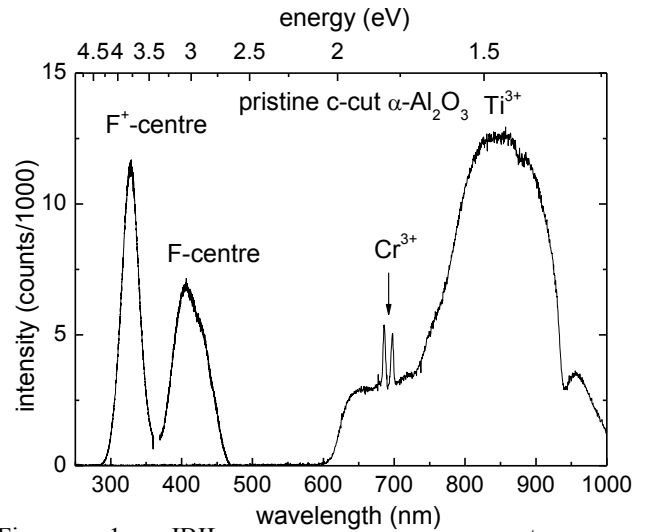


Figure 1: IBIL spectrum of the characteristic features of pristine sapphire samples: F-type centres and Cr^{3+} and Ti^{3+} emission.

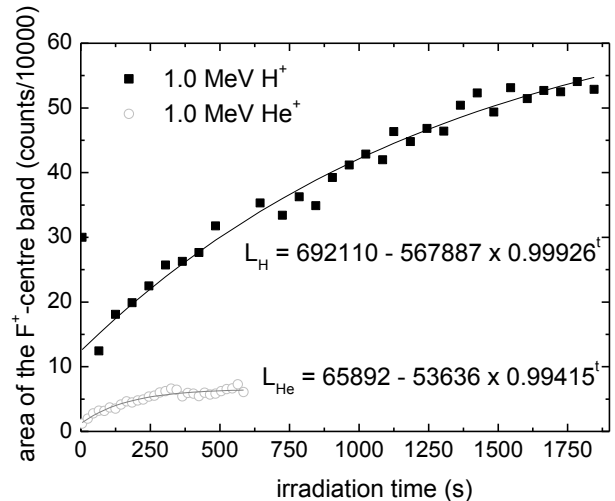
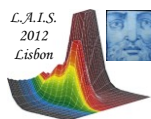


Figure 2: F^+ -centre evolution with irradiation time in pristine sapphire samples for 1.0 MeV H^+ and 1.0 MeV He^+ .

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DATING MORTARS BY OPTICALLY STIMULATED LUMINESCENCE (OSL) AND RADIOCARBON: EMERGING PROBLEMS AND PERSPECTIVES.

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After mortar was recognized as a suitable luminescence dosimeter for the dose reconstruction after nuclear accidents (Bøtter-Jensen *et al.*, 2000), attempts were made to establish reliable OSL dating protocols based on the assumption of a full bleaching of quartz grains during the mortar preparation (Zacharias *et al.* 2002, Gueli *et al.* 2010; Goedicke, 2011). At the same time, because of its high content of carbon, mortar was considered suitable for radiocarbon dating as well, and a large number of relevant experiments have been carried out by means of accelerator mass spectrometry (AMS). But despite noteworthy efforts, mortar dating by AMS has not become a routine method, as the chemical separation of the calcite formed during solidification of lime in historical periods from the geological one remains a problem.

We present the results of a dating study on mortars using both techniques. The samples came from two historical buildings: the church of the medieval abbey of Castelletto Cervo and the outside walls of the Certosa di Pavia, both located in northern Italy. The samples were taken from structures whose bricks had already been dated by TL.

OSL measurements were carried out on both fine (4-10 microns) and coarse (about 200 microns)-grains quartz extracted from mortars. The shine-down curves were characterized by the absence of the so-called *fast component*, as confirmed by Linear Modulated OSL technique, an efficient method for separating constituent OSL components, and for detailed investigation of the luminescence properties of each component (Bulur, 1996). To measure the radiation dose absorbed since the last light exposure, the Single Aliquot Regenerative Dose protocol, SAR, was applied (Murray and Wintle, 2000). The results were highly dispersed indicating an uncomplete bleaching of the quartz grains, more pronounced in the fine fraction (Goedicke, 2003). Single grain analyses were also performed to circumvent this problem.

The separation of calcite from mortars for radiocarbon dating was performed using a new technique based on physical properties, rather than chemical ones (Marzaioli *et al.*, 2011). A Fourier transform infrared spectroscopy (FTIR) analysis has been made on all the samples, in order to evaluate the ratio of the characteristic peaks of calcite: they should be valid

markers of the provenance of the calcite contained in the material (anthropogenic, geogenic; Gueta *et al.*, 2007; Chu *et al.*, 2008). The results coming from this approach are promising (Fig.1), even though further studies must be started to correctly read the changes in FTIR signals related to the composition of mortars, in order to optimize the selection of samples to date.

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ISOTOPIC DATING AND ENVIRONMENTAL MICRODOSIMETRY USING THICK SOURCE ALPHA PARTICLE SPECTROSCOPY.

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Introduction: During the recent years the potential applications of thick source alpha particle spectroscopy to isotopic dating and environmental microdosimetry measurements were investigated. Originally this technique was mainly used for the U and Th concentration estimation. From this estimation the dose rate can be estimated, which is necessary for luminescence (TL, OSL) and ESR dating.

Applications: In comparison to other techniques usually used such as “pairs technique” etc, this new technique gives also the possibility for detecting the disequilibrium in the samples, which results in increasing the accuracy of the calculated dose rates. Apart from this advantage, this possibility can be used for extending the technique to isotopic dating of speleothems and other materials. The application of this technique will be demonstrated with the measurement of a stalactite sample from the Agios Georgios cave (Kilkis, Macedonia, Greece). The stalactite layers were dated through U/Th geochronological method, which places the carbonate precipitation in Middle Pleistocene (630-300ka BP). The activity ratio of $^{230}\text{Th}/\text{U}$ is estimated using the possibility of disequilibrium estimation of thick source alpha counting technique. Furthermore, this technique proved to provide the basis for extending the application towards environmental radioactivity measurements. In the present paper the application in terms of the estimation of the Radon concentration is presented. First measurements for testing the accuracy and for calibrating the results will be presented.

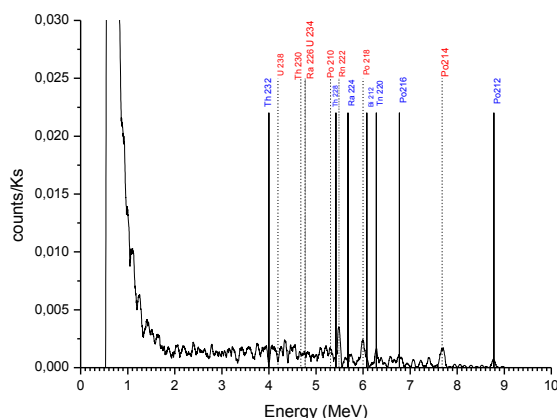


Fig.1: The alpha-particle spectrum taken from a thick sample of sediment Y. The red letters correspond to the energies of the emitted alpha-particles of the U series and the blue of the Th series. The rapid increase of counts below 1.8 MeV is due to β -particles and electronic noise.

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OSL CHRONOSTRATIGRAPHY OF PALEOENVIRONMENTAL EVENTS – LAGOA URI DE CIMA ARCHAEOLOGICAL SITE, SEMI-ARID REGION IN BRAZIL.

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Goals: This study aims the chronological reference of deposition in Lagoa Uri de Cima archeological site, as base for local paleoenvironmental reconstitution. The archeological site is located in the State of Pernambuco, Brazil. Nowadays, the region climate is semi-arid, warm and dry, with an average rainfall below 500 mm per year. In this site, a lake, a considerable amount of lithic artifacts of several raw materials (more than 2000 pieces) was found and also megafauna bones in lower levels. To have a chronological reference on the deposition episodes and archeological site formation, it was used the method of dating by OSL (Optically Stimulated Luminescence).

Importance of research: An approach that relates the pre-historic groups with the environment in which they were inserted enables a better comprehension of the occupational and environmental dynamics in the semi-arid region of Northeast of Brazil, as well as it assists the local and regional paleogeographic studies. This research also rises from the necessity of situating chronologically the archeological remains prior to natural processes which were responsible for their burial, besides checking possible gaps in the archeological site formation.

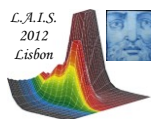
Methodology: Samples were collected from each individual sedimentary layer avoiding the incidence of natural light and the loss of the natural luminescence signal. Samples were chemically treated with HCl and HF. The dating was accomplished by the regenerative protocol. Annual dose of individual samples was obtained by gamma spectrometry. Gamma radiation measurement was conducted over five days, and the concentrations of ²³⁸U, ²³²Th and ⁴⁰K were determined based on photo-peaks obtained from the gamma spectrum. In order to obtain the annual dose (AD), contributions from gamma and beta emissions as well as cosmic rays and radon were taken into account.

Main results: The dating results can be summarized in the following table:

Table 1: OSL Results.

Sample	Accumulated Dose Average (Gy)	OSL Age
H	410 ± 14	124.000±5.200
G	256,6 ± 44,4	77.800±7.800
A	83,9 ± 19	25.400±3.000
C	45,2 ± 2,5	13.700±430
D	26,4 ± 3,2	8.000±570
E	13,03 ± 1,3	3.950±350
F	9,81 ± 0,35	2.980±180

Short interpretation: From the relation between the obtained chronology by OSL and the sedimentological and morphostratigraphic analysis of the profile, it was established a series of formative events of archeological deposition. During the Pleistocene, the lake was half connected to the drainage network, and there was a fluvial environment with high energy, channeled flow, indicating a lowered base level with reworking of disarticulated skeletons of mega-fauna under semi-arid conditions. Afterwards, there was an overlaying of carbonate cementing (calcrete), reaching only the base of deposition unit, indicating a phase of great aridity and lower temperature. There was, after that, a pelitic deposition in an environment with low energy, probably flooded, indicating local conditions of humidity and lower temperature. The Pleistocene-Holocene transition is marked by gravitational flow of non-channeled slope with remobilization of weathering conditions, under persistent torrential conditions. The Early Holocene is marked by gravitational flows of slopes with partial retention of larger-size fenoclasts and predominance of sandy-clay matrix, indicatives of persistent torrential conditions with progressive recovery of vegetation. From the Middle/Upper Holocene, there are intermittent mudflows derived from spasmodic convective torrentiality consistent with the pene-contemporaneous semi-arid conditions. Regarding this sedimentary package, there is a contemporary semi-arid pedogenic system with vertisols formation with expansive clay existence.



THERMOLUMINESCENCE CHARACTERIZATION OF BIOACTIVE MATERIALS; THE CASE OF 58S BIOACTIVE GLASS.

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Bioactive glass-ceramics are synthetic amorphous materials, which upon insertion in the human body attain both chemical and structural changes occurring as a function of time within the glass surface. Due to this specific property, they can be used as implant materials in the human body in order to replace and/or repair diseased or damaged soft and hard tissue in orthopedic, cranio- and maxillofacial and periodontal surgeries as well as a filling material for human teeth. The formation of a carbonated hydroxyapatite (HCAp) layer on the surface of bioactive materials is the main reaction that takes place upon their immersion in physiological fluids. However this formation does not occur instantly upon immersion. To date, all techniques used for the identification of this HCAp formation, such as Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and Fourier Transformed Infrared Spectroscopy (FTIR) are rather time consuming and not well suited to detailed and rapid monitoring of changes in the bioactivity response of the material.

The present work explores the possibility of using thermoluminescence (TL) for the discrimination between different bioactive responses in the case of the 58S bioactive glass. In the present study, the 58S bioactive glass (SiO₂ 60, CaO 36, P₂O₅ 4 in wt %) was prepared by the sol-gel method as described in literature, namely by mixing all reactants and leave the system to become gel. The original samples were grains with dimensions of 20–40 μm, which were immersed in the Simulated Body Fluid (SBF) for various periods of time between 0 and 6 days. 18 different immersion times were selected. All experiments were carried out under solution renewal conditions as it has been reported that this method simulates better the in vivo conditions by maintaining the ionic concentration and pH of the solution constant.

Results so far suggest the use of the 110 °C TL peak using a Hoya U-340 filter, for a number of reasons, namely: (a) this TL peak is present in all the 58S samples, regardless of the immersion time; (b) due to its simplicity and non-composite nature, the 110 °C TL peak does not require deconvolution analysis, while this is not the case for the rest of the glowcurve, which

is dominated by the presence of amorphous components, corresponding to continuous trap distribution; (c) the 110 °C TL glowpeak shape could be yielding a timescale regarding the immersion period for the beginning of some among the initial stages included in the bioactivity sequence; (d) the intensity of the 110 °C TL peak was proven to be very sensitive to the different bioactive responses, indicating a strongly decreasing pattern with increasing time immersion in SBF. In fact, TL can easily identify the loss of silica which takes place. This loss is reflected to the decrease in the 110 °C TL peak intensity versus different immersion times, which appears to be fast even for the shorter immersion times; (e) the 110 °C TL peak intensity plot versus immersion time yields discontinuities, providing thus experimental indications regarding the timescale for both the beginning as well as the end of the procedure of the crystalline hydroxyl-apatite formation respectively; (f) while studying the sensitization of the 110 °C TL peak, spike signatures were monitored in the plot of sensitization versus for immersion times between 20 and 40 min, providing thus an experimental feature indicating the beginning of the crystalline HCAp formation. Consequently, TL could indicate the beginning of crystalline HCAp formation, occurring after 20–40 min of immersion, while for longer immersion times the HCAp phase could be traceable.

For higher temperatures in the glow curve, a system of TL peaks centred at approximately 200 °C could be resolved after 24 h of immersion measured using the combination of Pilkington HA-3 and Corning 7–59 filters only. This group of peaks, being typical for all apatite formations, could possibly be used as a probe for TL bioactivity studies supplementary to the 110 °C TL peak. The suggested protocol consists of two sequential measurements, the first one using the Hoya U-340 filter up to the maximum temperature of 125–150 °C to measure the 110 °C TL peak and the second one using the combination of a Pilkington HA-3 heat absorbing and a Corning 7–59 blue filter up to the maximum temperature of 500 °C to identify the TL peak corresponding to HCAp.

PRELIMINARY RESULTS ON STRUCTURAL STATE CHARACTERIZATION OF K-FELDSPARS BY USING TL AND IRSL

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Introduction: Feldspars stand among the most widely used minerals for luminescence dating, besides certain drawbacks such as anomalous fading. Several studies were carried out concerning their composition and structure, after applying a variety of methods, such as X-Ray Powder Diffraction (XRPD), Fourier Transformed Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), etc. Nevertheless, the use of luminescence has been so far restricted to age assessment.

K-feldspars occur in monoclinic and triclinic forms. Their unit cell formula is of the form MT_4O_8 , where M site is occupied by K and Na cations and the four tetrahedral T (T_10 , T_1m , T_20 , T_2m) sites are occupied by one Al and three Si cations. The probability of Al cation to occupy one of the T sites is expressed by t_10 , t_1m , t_20 and t_2m . Sanidine is the high temperature, completely disordered ($t_10 = t_1m = t_20 = t_2m = 0.25$), monoclinic, $C2/m$ form while microcline is the low temperature, completely ordered ($t_10 = 1.0$, $t_1m = t_20 = t_2m = 0.0$), triclinic, $C\bar{1}$ form. Orthoclase stands as an intermediate form ($t_10 + t_1m > t_20 + t_2m$) yielding monoclinic, $C2/m$ symmetry.

Aim of the work: The aim of the present study is to investigate whether luminescence could be used as a tool for structural state characterization in the case of K-feldspars. Toward that direction, certain TL and IRSL properties, such as:

1. Glow or decay curve shape
2. Sensitivity
3. Sensitization following repeated cycles of irradiation-measurement,

Will be correlated to their unit cell parameters, the probability of Al cation to occupy one of the T1 sites ($\Sigma t_1 = t_10 + t_1m$) and the temperature corresponding to their structural state.

A computerised glow curve deconvolution analysis was performed in the case of TL glow curves. For all samples, mass reproducibility was strictly kept lower than 5%.

Materials and Methods: The samples used are ten naturally occurring K-feldspars from igneous

rocks of Northern Greece. The K-feldspars were separated from the mafic and felsic minerals with the use of Franz (model L-1) magnetic separator and Sodium Polytungstate (SPT) heavy liquid, respectively. The purity of K-feldspars was identified and classified based on XRPD measurements. Their unit cell parameters, as well as the refinements were calculated with CHECKCELL software. The probability of Al-cation to occupy one of the T1 sites ($\Sigma t_1 = t_10 + t_1m$) was calculated using the equations of Kroll & Ribbe (1987). According to the XRPD patterns of the examined samples, they are divided in three species: sanidines, orthoclases and microclines. It is to note that the Σt_1 have the lowest values at sanidines and the highest at microclines, while orthoclases have intermediate values.

Results: All TL glow curves were deconvolved into 5 different individual TL peaks. There is a well distinguished correlation between the intensity of each glow peak and the probability Σt_1 . In the case of four among the five TL peaks, the intensity in terms of the integrated signals after deconvolution is monotonically increasing with probability of Al cation to occupy one of the T₁ sites. Only the TL peak which corresponds to the largest Tmax value is decreasing while this latter probability increases. Furthermore, since this probability indicates the temperature corresponding to their structural state, the intensity can be directly correlated, also, to this temperature.

Typical XRPD measurements can identify the three different groups, namely sanidine, orthoclase and microcline. The preliminary results so far suggest that luminescence could also possibly discriminate between these three phases. Further work is required in order to study more luminescent properties of more samples towards the formation of a calibration curve.

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ON THE FEASIBILITY OF DATING PORTABLE PAINTINGS; PRELIMINARY TL AND OSL MEASUREMENTS.

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Introduction: The analytical study of the ground layers of wood and canvas paintings is a very interesting field as well as an artistic aspect of each specific artifact that has not been thoroughly researched by the scientists so far. These ground layers are complicated mixtures, not recorded precisely by the artists and include a variety of organic and inorganic materials.

Most traditional recipes for the ground layers of paintings on wood and canvas consist of at least one extender with or without filler material along with binding media. An extended review on recipes from 15th to 20th century, revealed the passion that the artists had on making the perfect ground for their painting in order to apply their technique and incarnate their artistic inspiration^[1].

During the past centuries, the recipes for the painting ground layers have remarkably changed, following the artistic needs of the masters of the following ages^[2]. From the traditional gesso grounds (gypsum-CaSO₄ with or without chalk (CaCO₃) combined with animal glue), to the innovative and complicated mixtures of the 18th century (zinc white (ZnO), lead white (2PbCO₃.Pb(OH)₂), kaolinite (Al₂Si₂O₅(OH)₄) with or without pigments (such as ochres) together with organic media), to the light and delicate recipes of the 20th century, recipes seem to be as versatile as the masters that adopted them^[1].

Aim of the work: Identification of the ground layer's composition becomes helpful providing with some useful hints towards indirect dating of the artefact. Nevertheless, direct dating for some among the paintings on wood and canvas by applying luminescence techniques still stands as a challenging task^[1].

In the framework of these two tasks, (identification as well as dating, both by luminescence), the present work provides with preliminary luminescence measurements for some among the main white extenders and fillers as well as yellow ochre.

Methods: TL curves of all aforementioned fillers were obtained. TL sensitivity was studied for two different combinations of detection filters. Moreover, sensitization of the signal after repeated cycles of irradiation-measurement was monitored. Possible lack

of sensitization versus repeated cycles recorded for the glow curve would be indicating the suitability for single aliquot measurement protocols. Furthermore, thermal stability was studied for all feasible luminescence signals.

Although in the case of some Ca-based materials application of OSL is restricted, the bleachability of the luminescence signal of all fillers was studied after applying both OSL and IRSL. The selection of the most effective technique among TL, OSL and IRSL does not depend, only, on the ground layer composition, but also on the expected age, since each technique impose different limitations. Therefore, natural U, ²³²Th and ⁴⁰K concentrations were estimated in order to get some preliminary dose rate assessment.

Results: TL could easily identify most of the fillers, since most of them present TL glow curve with different shapes. One exception stands in the case of zinc white and lead white, because those two materials show identical TL glow curve shape. The TL curves of the yellow ochre resemble much the glow curve of unheated quartz. However, the low sensitization of the material stands as a drawback to be circumvented. Barium sulphate presents strong sensitization, excluding thus the possibility of single aliquot dating protocols. On the contrary, neither gypsum nor kaolinite present sensitization after successive cycles of irradiation and heating. OSL and IRSL curves are presented for most of the materials, along with residual TL curves after bleaching. Kaolinite seems to be a promising material towards luminescence dating. Further work is required in order to establish its usefulness.

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SAR AND MAAD TL DATING OF STRUCTURAL ELEMENTS FROM THREE SITES IN CALABRIA, SOUTH ITALY.

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In this work three types of “*Caroselli*” from different sites in Calabria have been studied. The “*Carosello*” is a structural hollow element made of clay, placed in arches, in domes or even in the walls of buildings such as churches and houses, with the function of relieving vaults and main structures. Diagnostic tests have been conducted on the “*Caroselli*” in order to determine the presence of luminescent material, the final goal being the age assessment of such artifacts using thermoluminescence (TL) techniques. Of course the historical frame of the buildings, to which “*Caroselli*” belong, is known from their style and architectural considerations. The scope of our dating is to supply additional information to clarify whether “*Caroselli*” were used from the very beginning of building construction or employed in subsequent restorations. The dating process has been conducted using two protocols, the Multiple Aliquot Additive Dose (MAAD) and the Single Aliquot Regenerative-dose (SAR), both by applying TL. The correction obtained by the SAR TL method is preferred because it allows a more accurate estimate of the equivalent dose and avoids problems arising from sample sensitivity. The results show that the three “*Caroselli*” can be respectively dated back to 1844 ± 11 , 1825 ± 40 , 1803 ± 30 A.D.. Such results suggest that for two sites (“*Madonna del Buonconsiglio*” church and “*Torre dello Zuino*” colonial house), the “*Caroselli*”, as structural elements, were used since the beginning of the building construction, while for the remaining site (“*Pettoruto*” church) probably the “*Caroselli*” were added in a successive restoration of the building, since the historical context of this building belongs to the fourteenth century.

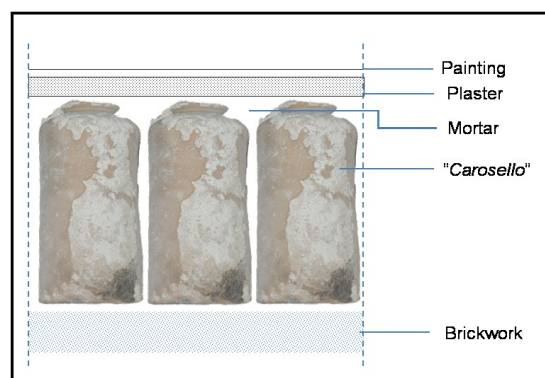


Figure 1: Schematic drawing of a wall cross section showing the spatial allocation of “*Caroselli*”.

TL DATING OF NEOLITHIC AND MEDIEVAL CERAMICS FROM LAMEZIA, CALABRIA (SOUTH ITALY).

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Abstract

In this work some ceramic fragments from the neolithic period have been studied. We show the measurements conducted on six samples of "Stentinelliane" ceramics found in the area of Acconia, in the "Piana di Curinga" (Lamezia Terme, Cz, South Italy), dating back to the V millenium a.C.. This culture, that begun with the Neolithic civilization, is characterized by the presence of imprinted decorations on the artifacts, which are made on the external surface by fingers or by other instruments (shells, pieces of wood, bones, flint stones, etc.). Such decorations are peculiar of the neolithic culture as studied in the Neolithic archeological sites nearby Syracuse (Sicily). The "Stentinello" culture represents an evolution of these decorative techniques, during the diffusion of the Neolithic in the South Italy.

The samples were analysed by both optically (OSL) and thermal (TL) stimulated luminescence techniques, using the Single Aliquot Regenerative-dose (SAR) protocol. The luminescence signals originate from stable traps filled by the natural radiation during the burial.

The SAR method allows an accurate estimate of this absorbed radiation by measuring the equivalent dose (ED) and avoids problems arising from sample sensitivity (fig.1, example of ED by SAR-TL).

Knowing the radiation dose to year (annual dose rate, DR) to which the sample is exposed, the age can be assessed by the simple equation:

$$\text{Age} = \text{ED (Gy)} / \text{DR (Gy / year)}.$$

The so obtained results provided reliable age estimates for the following samples (the Italian labels refer to different archaeological sites in the same valley): "Torrevecchia", 4000 ± 350 a.C.; "Suveretta", 4900 ± 500 a.C.; "Romatizi", 5300 ± 400 a.C..

Comparing the historical-artistic characterization of the samples with the dating results, it is possible to validate the relevant historical period of pottery fragments, which can be dated back to the V millennium a.C.

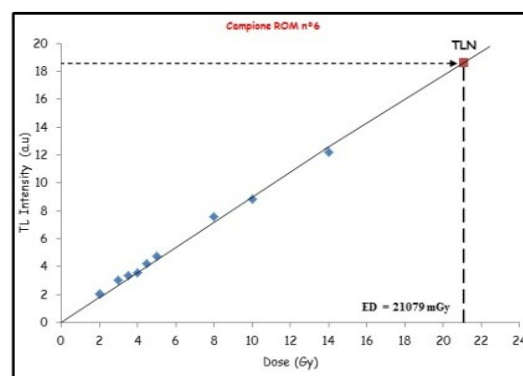
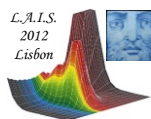


Figure 1: Determination of the Equivalent Dose, based on the Single Aliquot Regenerative-dose (SAR) protocol, using sensitivity corrected TL signals.

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HEATED FLINT FROM GRUTA DA OLIVEIRA (PORTUGAL): COMPARISON OF TL-DATING RESULTS WITH RADIOCARBON AND U-SERIES DATING.

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The Middle Palaeolithic site of Gruta da Oliveira (Almonda, Portugal) contains a stratigraphic succession of sediments with archaeological layers attributed to the Mousterian. The succession is made up, mainly, by slope sediments which accumulated inside the cave from the entrance and from vertical shafts. Individual layers are often separated by calcareous crusts or flowstones that sealed the archaeological record (artifacts and features).

Some of the lower layers (10–14) contain backed microliths, associated with a few prismatic and pyramidal blade and bladelet cores, among otherwise characteristically Middle Palaeolithic assemblages based on discoidal, Levallois, and Kombewa flake production schemes (Zilhão, 2006). These anthropogenic components of the sediments were shown to be in primary position, with some penecontemporaneous biological activities but little post-depositional processes disturbing the context (Angelucci & Zilhão, 2009). This provides a good basis for the assumed association of samples collected for dating with prehistoric human activities.

Radiocarbon dating of organic material from Gruta da Oliveira indicates the late survival of Neanderthals in Iberia around 36 ka (layer 8) and provides a short time span for the Mousterian occupations of layers 9–15 between 42 and 50 ka (Angelucci & Zilhão, 2009). Bones from Gruta da Oliveira layer 8, which contains the uppermost human occupation of the site, were dated to the 34–40 ka range by U-series (Diffusion/Adsorption), which is in good agreement with the available radiocarbon results (Hoffmann et al., 2012). U-series dating of speleothems from the cave constrains the accumulation of the Gruta da Oliveira sediment fill to between 23 ka and at least 102 ka (Hoffmann et al., 2012).

Here we present Thermoluminescence (TL) ages for heated flints from layers 13 and 14 of the Middle Palaeolithic sequence at Oliveira. Palaeodoses were obtained by a standard multiple aliquot additive regeneration protocol (Aitken, 1985), while the alpha sensitivity was determined by a regeneration method. The internal dose-rates were established by Neutron Activation Analysis and the external γ -dose rate with α -Al₂O₃:C-dosemeters (Richter et al., 2010). Because the sediments are situated deep in the cave, which is providing a relatively stable environment, the uncertainties associated with some assumptions to be made in luminescence dating (i.e. sediment moisture and cosmic dose) are smaller than usual for this site. The TL data will be discussed and ages compared with the chronometric data available, which is allowing a verification of the dating methods used.

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LUMINESCENCE AND MINERALOGY OF PROFILING SAMPLES FROM NEGATIVE ARCHAEOLOGICAL FEATURES

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The aim of this work is to relate luminescence behaviour and mineralogy for samples from profiles through four archaeological sequences from fills of negative features cut into calcitic substrate. Samples of c. 10 g each were collected, using small tubes (2 cm diameter, 5 cm length) through the different fill materials that had accumulated in these features, stratigraphic differentiation was made in the field on the basis of their colour and texture. The identification and semi-quantification of crystalline phases was performed by X-ray diffraction. Measurements were made using a Philips X'Pert Pro diffractometer, with CuK α radiation and operating at 45 kV and 40 mA, on non-oriented powder in the 4-70 $^{\circ}2\theta$ range, a 1 $^{\circ}$ divergence slit, and a scan rate of 1 $^{\circ}$ /min. For luminescence studies, the samples were subjected to simple preparation protocols to obtain quartz-enriched material and polyminerals [1], and measured using a simple regenerative sequence to evaluate IRSL, OSL and TL sensitivity, capacity for sensitization, and absorbed dose. Measurements were made using a Risø automatic reader with calibrated β source ($^{90}\text{Sr}/^{90}\text{Y}$) and U340 detection filter. XRD results from the profiles show the mineral assemblages to be predominantly calcite, associated to phyllosilicates, quartz, plagioclase and potassium feldspar, with occasional haematite and dolomite. Patterns of relative mineral concentrations through the sections, often show an increase of calcite content with depth, and a decrease of quartz, phyllosilicates and feldspars, but one of the sites exhibited contrasting behaviour (Figure 1). OSL signals from Cor and Cov sites, indicate a low absorbed dose for modern sediments. Within the the archaeological sequences behaviour is distinct. The Cor profile shows a gradual increase in absorbed dose with depth from 6 to 8 Gy, interpreted as gradual filling of this ditch. The Cov profile produced absorbed doses centered around 6 Gy, with minor variations evident through the archeological stratigraphy. Higher values from the lowermost sample indicated the presence of geological signals, which are also evident in sample from a parallel profile at the edge of the excavation (open circles in Figure 2). Infill of this pit appears to have occurred rapidly. Comparison of luminescence and mineralogical analyses permit understanding of

behavioural patterns in the selected profiles, with regard to sensitization and the absorbed dose of the samples, to distinguish changes in accumulation processes and identify promising layers for dating.

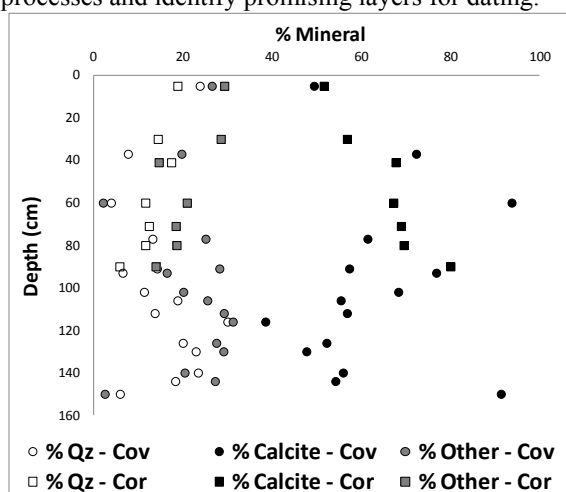


Figure 3: % Mineral vs. Depth, for two different profiles.

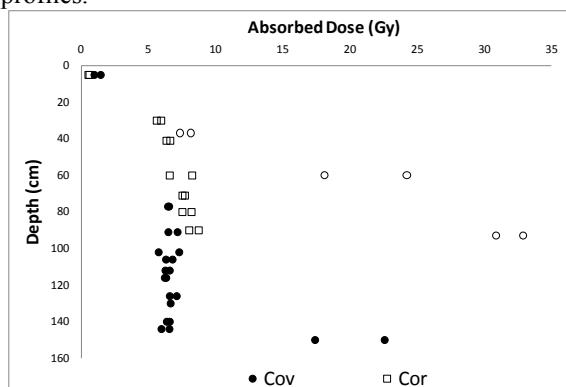
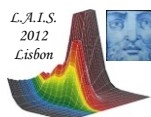


Figure 2: Absorbed Dose (Gy) vs. Depth, for two different profiles, determinate by OSL in Quartz grains, for two different profiles.

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A DECADE OF LUMINESCENCE PROFILING OF ARCHAEOLOGICAL AND ENVIRONMENTAL SEDIMENTS.

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Abstract: This paper considers the development and application of profiling methods to qualify luminescence dating samples and providing additional indicators of depositional conditions and their past variations. It considers some of the case studies in which both laboratory and field profiling have been used to assist environmental and archaeological investigations. It looks at the emergent outputs from the growing communities using simple portable OSL systems in diverse conditions. Finally the underlying physical mechanism for modulation of readily observed luminescence signals will be considered together with a look at the limitations of current approaches, and discussion of additional information which might be gleaned from future developments.

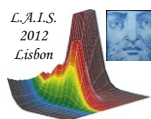
The motivation for developing luminescence profiling methods in the late 1990's arises from one of the fundamental difficulties of sediment dating: qualifying the initial zeroing condition. This long standing problem has been recognised since the pioneering work of Ann Wintle on TL sediment dating. The presence of residual signals was recognised, and "total bleach" and "partial bleach" methods conceived to try to manage them. For practical and methodological reasons these approaches were particularly limited for young sediments, which have only become tractable since the adoption of more readily-bleached photo-stimulation approaches (OSL and IRSL). Nonetheless the problems associated with incomplete or heterogeneous bleaching, with co-deposition of mixed age sediments and with tracking bioturbational or anthropogenic alterations remain.

It is now well established that OSL processes in both quartzes and feldspars result from complex multi-trap signals, with wavelength dependent photo-stimulation cross sections. This results in complex non-exponential bleaching associated with subtle luminescence decay shape changes. Absolute bleaching rates, and the relative bleaching rates of different mineral components within sediments, thus vary widely with the intensities and wavelength distributions of the environmental illumination fields associated pre-depositional illumination. It should therefore be unsurprising that these environmental and physical variables lead to complex and heterogeneously bleached sediments in many systems, which need to be taken into account in dating studies.

Within sample approaches to deal with such issues use dose-distributional analysis (including single-grain analysis) coupled to age modelling to address heterogeneity, or use signal component (or decay shape), to evaluate completeness of bleaching in homogeneous systems. Samples are selected on contextual or sedimentological grounds, generally avoiding dosimetric discontinuities. Under favourable conditions very useful chronometric data can be achieved from a wide variety of contexts and ages. But, as practitioners and users are aware, problematic cases are also encountered, where despite significant efforts, the nature of the material precludes accurate dating.

Profiling approaches exploit the relative sequences of luminescence signals to qualify the most favourable conditions for dating, and to reveal information about depositional conditions and processes. The recognition of discontinuities, incised or erosional surfaces, inverted sequences (due to redeposition without bleaching) and mixed-age layers have all been achieved. Early case studies included the investigation of regional covers sands in SE Asia, where quartz TL-OSL relationships with depth were used to appraise competing diagenetic models, studies of ancient canals in Cambodia, and incised archaeological features (such as ditch fills) where basal stratigraphy and the presence of redeposited material associated with bank collapse have been observed. The relationships between sedimentary stratigraphy, lithic assemblages, palynology and age have been examined on palaeolithic sites. Laboratory profiling, where suites of small mineral samples, subject to rapid equivalent dose determination, are being used in several laboratories to assist with the interpretation of complex sedimentary sequences. Such laboratory studies have also been enhanced by the use of field-portable equipment capable of conducting screening measurements of bulk samples, or prepared material, during fieldwork, and thus assisting with site interpretation and sampling decisions. These systems are now in use by several groups, and the published literature of case studies is expanding rapidly.

Examples of the use of laboratory and field profiling together with quantitative OSL analysis will be discussed from a variety of geoarchaeological and environmental settings.



ESTIMATING MAXIMUM TEMPERATURES ATTAINED DURING BUILDING FIRES BY THERMOLUMINESCENCE.

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Introduction: Fire is one of the main catastrophic weathering agents for building stone. It can be the result of relevant historic events, such as wars. Thus, dating fires and knowing the maximum temperatures attained by rocks during them is important to understand the level of damage a fire could cause in the past and how it affected to the subsequent weathering history after the fire. Mineralogical analyses allow us to roughly assess the maximum temperature attained by stones during fires if some high temperature intervals (above 600°C) are reached (Gómez-Heras et al., 2006). However, accurate methods does not exist.

In the case of quartz-rich materials such as some rocks, the thermoluminescence (TL) sensitivity of quartz to different temperatures could be used, as it is strongly influenced by firing at high temperatures but even to temperatures below 600°C. The TL glow curve (blue emission band: 470±30nm) of quartz shows three main peaks located at 110, 210 and 350°C. The sensitivity of such peaks can be increased if some temperatures are reached during the firing of quartz (Charitidis et al, 2000). Thus, maximum temperatures of fires can also been assessed in the case of stony building materials containing quartz. The 110 peak has been proposed to assess the firing temperature of quartz (Sunta and David, 1982) although it seems that it does not always work (Watson and Aitken, 1985). Other works, proposed the use of the other TL peaks and OSL (Polymeris et al., 2007).

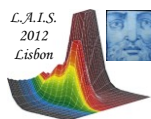
Aim of the work: Laboratory tests have been carried out to assess the maximum temperature reached by a sandstone during past fires by Thermoluminescence (TL). The test rock is the 'Uncastillo sandstone', commonly used continuously from Roman times onwards in the region of Saragossa (Spain). This is a calcitic sandstone with a variety of quartz grain types (mono, polycrystalline and chert). Numerous examples of fired heritage buildings have been constructed with this stone type. The TL of a test sample (fired a few years ago to an unknown temperature) was also measured to assess the temperature reached during a past fire.

Methods: 5 cm cubes of the Uncastillo sandstone were heated in an oven at different temperatures from 200°C to 700°C (in 100°C steps) for 8 hours. Quartz grains were obtained from cubes by soft ginding, sieving and chemical treatment with HCl and HF.

Results: The TL curves of the quartz extracted from the probes were measured and compared with an unheated quartz (erosion of the TL glow curves) and with curves of quartz grains of the test sample. The comparison of the eroded glow curves show that probably the maximum temperature of the fire was near 300°C. The sensitivity of the TL peaks of irradiated quartz from the heated and test samples were also compared. They showed a similar result. A final test was to assess the firing temperature by a full predose protocol (Polymeris et al., 2007) with similar results.

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OSL DATING OF MEDIEVAL ANTHROPOGENIC SEDIMENTS: USE FOR HISTORY OF URBAN GROWTH.

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Introduction: Santiago de Compostela is a city located at the NW of Spain and the old town is part of the UNESCO World Heritage since 1985. During works for the construction of an auditorium in the old town of Santiago (years 2008-2009), a group of hollows filled with sediment was found in the ground. 22 hollows (19 circular and 3 rectangular) were filled with clay-rich sandy sediments. The depth of the hollows was from 3.30 m to 0.8 m (from the deepest to the shallowest one) and the diameter from 1.5 to 1 m. Fourteen of them were excavated and some materials were recovered (sediment, charcoal and fragments of vessel) to date the infill. Hypothetically, the hollows were excavated on rock and filled with water (as some of them are connected by channels), but their function is unknown. It is possible that the hollows were used for leather tanning (where leather was immersed in brine). Such activity was considered as unhealthy or harmful in middle ages, thus the facility was moved outwards the new wall (and the hollows filled) because the expansive growth of the town during the middle ages.

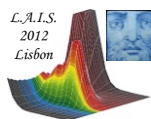
Layout: The aim of this work is to date the anthropogenic sediment infill of the hollows by OSL, as not many samples of this kind have been dated by OSL (Lang et al. 1999). Two sediment samples and a charcoal sample were collected from one of the circular hollows (P-76) and another one of a rectangular one (PL-1). To test the reliability of the OSL ages, both a vessel fragment and a charcoal sample were taken from a next hollow dated by TL and radiocarbon, respectively to crosscheck OSL dates.

Methods: OSL dating was performed in the Luminescence Lab of the University of A Coruña (Spain). Coarse quartz was used for dating. For TL dating a multiple aliquot additive dose protocol was used (Aitken, 1985). For OSL, the SAR protocol was used (Murray and Wintle, 2000) on discs with few quartz grains, after performing preheat tests. The bleaching of the OSL signal of the quartz grains was observed by bleaching tests and LM-OSL (Bulur et al., 2000). To calculate the dose rate, broad energy gamma spectrometry was used to measure the ²³⁸U, ²³⁵U, ²³²Th and ⁴⁰K decay chain activities. ICP-MS was used to measure U, Th and K content of the sherds. Conversion factors of Adamiec and Aitken (1998) were used to calculate dose rates.

Results: Signal tests showed suitable OSL signals (fast signal decay) to get reliable ages. Radial-plots and histograms were used to assess possible partial bleaching. Samples from P-76 showed aliquots with high equivalent doses due to partial bleaching, but some others show low doses (a similar low dose for both samples, ~3.5 Gy). The quartz grains of the sample taken in PL-1 showed lower equivalent doses, but also some aliquots showed evidence of partial bleaching. Thus, a minimum age model (MAM) was used to calculate equivalent doses (Galbraith et al., 1999). The TL indicated a more high equivalent dose (~6 Gy) for the vessel fragment. ²³⁸U and ²³²Th decay chains did not show secular disequilibrium. Thus, ages were calculated from equivalent doses and the calculated dose rate. All the ages (OSL and TL) fell between the 10th and 12th centuries. The results of ¹⁴C of charcoals showed calibrated ages between the same centuries. Thus, OSL ages can be considered as reliable for this kind of sediments if the MAM is used, despite the possible brief sunlight exposure of the sediment before being used as infill in the hollows.

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TOWARDS ANSWERING TECHNOLOGICAL AND PROVENANCE QUESTIONS OF ARCHAEOLOGICAL GLASS MATERIALS BY USING LUMINESCENCE TECHNIQUES.

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Introduction: Two different Islamic glass fragments from Raqqa, Syria, were fabricated under different conditions. Al-Raqqa 40 is a rare example of raw glass imported to the production site of al-Raqqa; al-Raqqa 35 was fused from raw materials at al-Raqqa. The chemical composition and physical properties of glasses suggested that al-Raqqa 35 (plant ash type 1 hereafter) and al-Raqqa 40 (plant ash type 2 hereafter) are compositionally quite distinct, (Henderson et al., 2004). The main aim of the present investigation was to try discriminating between these two types of archaeological glass by applying luminescence techniques.

Experimental: Three pieces from each type were independently crushed and grains with sizes between 80 and 140 microns were selected. The measurements were performed independently to the grains of each one of the 6 aforementioned parts. The aim of this approach was to check homogeneity-reproducibility between the pieces-parts of the same type as well as to discriminate between these two types. Mostly TL was applied to the glass samples, while some preliminary blue LM-OSL measurements were also performed. In all cases a beta test dose of 75 Gy was attributed. The main properties that were studied were the glow curve shape, the sensitivity and last but not least the sensitization following repeated cycles of irradiation-measurement.

Results: The glow curve shape of all 6 parts without any prior artificial irradiation is identical.

In the case of type 1(Raqqa-35), all 3 parts provided similar results. The “110°C” TL glow peak is the only prominent peak. There are also strong indications suggesting the presence of at least two more peaks bellow 200°C. Furthermore, repeated cycles of irradiation and measurement result in sensitization only for the case of the “110°C” TL glow peak. Unfortunately, there is only a qualitative similarity to those three sensitization patterns. In general, besides the quantitative differences in the sensitization patterns of these 3 parts, the rest TL properties suggest homogeneity between them.

The glow curves following repeated cycles of irradiation-measurement resulting from two parts of type 2 (Raqqa-40), present three different prominent peaks discriminated bellow 200°C; moreover, after the first cycle of irradiation-measurement, another prominent peak is also monitored around 250°C. It has to be mentioned that both sensitivity as well as sensitization of each peak differs from part to part. Another two, prevalent features for all three parts of the second glass type are the strong sensitization that is induced to all glow peaks following the first heating and the relative low sensitization induced to the “110 °C” TL glow peak after the second heating.

Conclusions: Luminescence can effectively discriminate between the two specific types of glass of the present study based on the following features:

- The different TL glow curve shape following artificial beta irradiation.
- The different sensitization patterns of the 110°C TL glow peak.
- The different LM-OSL curve shape following artificial beta irradiation.
- There is lack of homogeneity in several TL features for the three parts of glass type 2, such as sensitivity and sensitivity changing patterns. This lack of homogeneity may be a drawback for a luminescence study of the specific glass type; however it stands as another additional criterion for discriminating between these two glass types.

The proposed TL examination is also currently applied, on two sets of archaeological vessel glass samples of the same type (soda-lime based glasses), coloration and archaeological period (Hellenistic Era); TL signals may provide useful data on the way of verifying the potentiality of applying luminescence studies to answer technological and provenance issues of historical and archaeological glass materials, in general.

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SCINTILLATORS APPLIED IN NEUTRON IMAGING TECHNIQUES

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Introduction: The neutron imaging techniques (NIT) are a non - destructive testing techniques commonly employed to inspect the internal structure of objects. Because of the neutron - matter interaction characteristics, this technique is largely employed to inspect light substances even when these are surrounded by metallic layers which would make them invisible under X-ray imaging. Thus, the neutron imaging techniques find applications in the technological fields of the automotive, nuclear and aerospace industries, as well as in medicine, archeology, biology and geology. The radiography is obtained by irradiating the object in a uniform neutron beam and a converter screen transforms the transmitted neutron intensity into ionizing radiation which is able to sensitize a film forming the image. The screens consist of strongly neutron absorbing elements (gadolinium, dysprosium, lithium) and the films are the conventional for X-ray films and the track-etch foils. Alternatively, neutron scintillators are also used as converter and in this case the light emitted sensitize either a film or the CCD of a video camera. In the last case the radiography image can be obtained in real-time. For both cases the radiographs are 2-D projections of the internal structure of the object.

Among the many technical efforts to improve the neutron imaging methods, the employment of digital systems for image acquisition and processing is the best option in terms of image quality. Another very important technical effort which spread the use of the neutron imaging around the world was the development of neutron tomography systems to provide a 3-D view of the internal structure of the object under investigation. Basically these systems consist of an automated rotating table able to rotate an object between 0 and 360 degrees, associated to a real-time neutron imaging system and to softwares for image processing, reconstruction and visualization.

The neutron tomography setup of IST/ITN was installed in 2008 in the horizontal access of the thermal column of the 1MW Nuclear research reactor, and is operational since 2009 and being used for multipurpose applications, especially in samples of interest of the cultural heritage. The IST/ITN tomography system consists of a water cooled CCD digital video camera, which captures an image from LiF scintillator which is reflected by a 45° mirror, with respect to the neutron beam, to keep the camera

out of the neutron beam. The camera is also coupled to a high performance computer and to a rotating table making the system automated. The images are formed by the neutron transmitted by the sample in a LiF converter. In the present two softwares are employed: Octopus and VG Studio for image reconstruction and for 3-D visualization respectively.

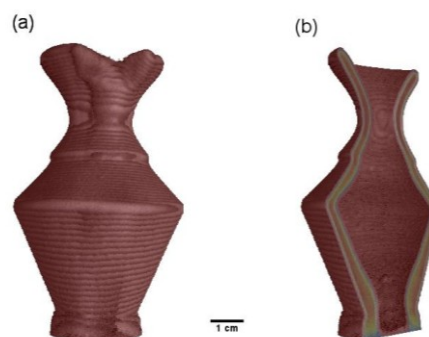
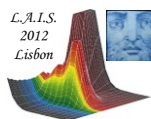


Figure 4: 3-D Ceramic vessel image. Original aspect (a), transverse section (b).

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EPR DATING OF FOSSIL BONE AND TOOTH ENAMEL OF HUMANS FROM RIO GRANDE DO NORTE STATE, BRAZIL.

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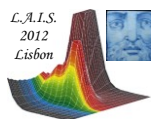
Introduction: The aim of this paper is the EPR dating of fossil bone fragment and tooth enamel found at the archaeological site of Pedra do Alexandre, located in the Rio Grande do Norte state (Pessis, A. M. 2000, Gabriela M, 2008.). This area is located in the Brazilian northeastern, where several underground caves were found. There are some dating studies of charcoal with C-14. Such studies indicated that the period when the burials took place is from 2600 to 9000 BP. The objective of this study is to contribute for the chronological identification of burial through the dating of bones and teeth.

Methodology: For this study a fossil bone fragment and tooth enamel were collected from burial number 27 found in the site with geographical coordinates 6° 32'S and 36° 31'W. Initially, the samples of fossil bone was cleaned with distilled water, crushed and sieved until grains with diameter of 0,080 – 0,180mm were obtained and the tooth enamel samples was mechanically separated and similarly treated as done with the bone fragment (Schwarcz 2002, Ikeya 2002). For dating, an additive method has been used and after the measurement of the accumulated dose signal, the samples were splitted into several aliquots and irradiated with at Co-60 source. Gama spectra of the sediments collected around the bone and tooth analyzed, were obtained using Ge(HP) detector and the concentrations of U-238, Th-232 and K-40 have been determined. For the internal dose rate, neutron activation of the samples was done. The results showed, the ages of 4940BP and 3000BP for fossil bone and tooth enamel respectively.

Results: The results showed hyperfine lines of Mn^{2+} impurities and CO_2^- signal from hydroxyapatite for the bone sample and only the EPR CO_2^- signal for the tooth sample. The results of the accumulated dose were 17.6Gy and 10.8Gy for fossil bone and tooth respectively. To estimate the burial's age, the value of the annual dose rate was estimated taking into consideration the contribution of external and internal radiations. The results showed that the internal dose rate was lower than the detection limit. Based on the accumulated dose and the annual dose rate found, it was possible to estimate ages. The results showed, the ages of 4940BP and 3000BP for fossil bone and tooth enamel respectively, these results are in agreement with the dating studies of charcoal by measurements of the C-14 content, and will contribute for the better understanding the archaeological site.

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DISTRIBUTION OF NATURAL RADIONUCLIDES (K, TH AND U) IN AN APLITE DYKE FROM THE BEIRA URANIFEROUS PROVINCE (FORNOS DE ALGODRES, PORTUGAL).

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Abstract: A uranium-rich aplite dyke from the Fornos de Algodres area (Northern Central Portugal), located in the Beira uraniferous province, is the focus of our study. This dyke intruded in Hercynian granitic rocks is greatly altered showing evidence of spheroidal weathering, consisting mainly of yellowish residual clay with boulders of less altered aplite. The main aim of this work is to investigate the distribution of U and other trace elements in the aplite, considering the variation within the vertical profile, samples showing different degree of alteration, different size fractions, and in different concentric shells of decayed aplite formed during spheroidal weathering.

The goal of this work is to better comprehend the geochemical behavior of uranium and other radionuclides, concerning their distribution in U-enriched rocks and its mobility and redistribution during weathering of granitic rocks.

Chemical and mineralogical composition was determined for the whole rock of six samples of aplite and for the various grain-size fractions (2 mm-63 μm , 63-20 μm , 20-2 μm and <2 μm) of four of the samples distributed along a vertical profile (2 samples of residual clay and 2 samples of soil).

The chemical investigation was performed by instrumental neutron activation analysis (INAA), using the Portuguese Research Reactor at ITN (Sacavém), whereas the mineralogy was studied by X-ray diffraction (XRD), using a Philips X'Pert Pro diffractometer. An in situ gamma-ray measurement

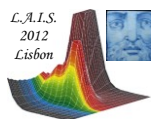
was undertaken using a NaI (2"x2") portable detector to obtain concentrations of the three primordial radionuclides (K, Th and U) that are primarily responsible for terrestrial gamma radiation.

The mineralogical composition is similar for all samples, varying mainly the proportion of the mineral phases. They are essentially composed by quartz and potassium feldspar, which clearly identifies the origin of aplite as granitic. Anatase is common and abundant, except in the two soil samples, and the phyllosilicates observed are mainly of vermiculite, kaolinite, illite and rarely smectite and gibbsite.

Concentrations of uranium vary from 5 to 80 ppm in the studied samples. The higher concentrations of U are found in the: i) more altered samples, ii) coarse fraction (2 mm-63 μm) and clay fraction (<2 μm) of residual clays and just in clay fraction of soil samples, and iii) core of the rounded boulders of aplite formed through spheroidal weathering. The higher concentration of U in levels where clay fraction is more abundant, suggests that U is mainly adsorbed/incorporated on clay minerals. The geochemical behavior of U is generally accompanied by a similar distribution pattern of lanthanides.

Results for the studied aplite from Fornos de Algodres area show that the uranium suffered processes of mobilization, transport and deposition.

This work was supported in part by the FCT funded project PTDC/AAC-AMB/121375/2010.



GEOCHEMISTRY AND FIELD RADIOMETRIC MEASUREMENTS OF NATURALLY OCCURRING RADIONUCLIDES IN SEVERAL LITHOLOGIES OF FORNOS DE ALGODRES AREA (CENTRAL PORTUGAL).

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Abstract: Naturally occurring radiogenic elements (K, Th and U) are present in rocks in concentrations that vary greatly as function of several geologic factors, particularly the lithology. The decay of U produces ionizing radiation and radon emissions, which may have significant radiological environmental impact. This study contributes to the compilation of mineralogical, geochemical and in situ gamma-ray data as a basis to evaluate the radon levels emanated from rocks, by studying the distribution of radiogenic elements in several lithologies from the Sobral Pichorro area (Fornos de Algodres, Central Portugal), located in the Beira uraniferous province.

The geological context of this research is a coarse- to medium-grained porphyroid two-mica granite (“Granito da Muxagata”), aplite and dolerite veins that frequently intersect the granite, and also the schist-greywacke complex (“Complexo Xisto-Grauváquico do Grupo do Douro”). About 30 samples of sediments and soils of the various lithologies were collected and studied, often corresponding to different stages of weathering within a vertical profile.

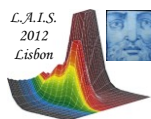
The mineralogical composition of the samples was obtained by X-ray diffraction (XRD). The chemical data was obtained by instrumental neutron activation analysis (INAA), by irradiating samples and standards in the core grid of the Portuguese Research Reactor (ITN, Sacavém) for 1.5 minutes and then for 6 hours, which allowed to obtain the concentration of U, Th and K, among other elements. For selected samples

the abundance of these naturally occurring radionuclides was also determined by in situ gamma spectroscopy, using a NaI (2”x2”) portable detector, to compare with INAA data. The combination of INAA and field measurements enables inferences related to dose rate stability and may help detect disequilibrium among the U chain.

The concentrations of radiogenic elements in the studied area vary strongly, but in general they are higher than the average values estimated for the upper continental crust (2.8% K, 10.7 ppm Th and 2.8 ppm U): i) granite – 1.7-6.4 % K, 7.3-20.9 ppm Th and 3.1-28 ppm U; ii) schist – 1.3-4.9 % K, 10-36.9 ppm Th and 3.4-13.9 ppm U; iii) aplite – 2.7-8.8 % K, 5.7-30.1 ppm Th and 4.5-77.2 ppm U; iv) dolerite – 1.7-6.2 % K, 3.5-25.7 ppm Th and 1.2-33.2 ppm U.

The soil samples present much lower concentrations of U than the granite and aplite substrates, and all soil samples are richer in Th. INAA and field gamma spectrometry results were commonly similar. Slightly lower values from the field measurements are related to in situ water. Occasional large differences in values for uranium are considered to result from spatial variations in U enrichment, consistent with the INAA results.

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THE INFLUENCE OF AEOLIAN ACTIVITY IN THE ENVIRONMENTAL SETTING OF THE CITY OF GHENT (EAST FLANDERS, BELGIUM): A CONTRIBUTION FROM OSL DATING.

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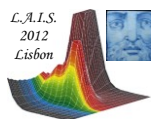
Historical and archaeological studies hold strong evidence that the city of Ghent (East Flanders, Belgium) originated near the confluence of the rivers Scheldt and Lys nearby a hill that represents an erosional outlier of Tertiary sediments (locally known as the Blandijnberg). This setting forced the research to focus on fluvial processes. Although the current course of the Scheldt river has been largely dictated by erosion and sedimentation processes related to Late Weichselian aeolian activity, the influence of these processes on the development, functioning and expansion of the city has rarely been considered.

During the Late Weichselian, strong westerly winds caused sand drifting, coversand deposition, and the formation of inland and river dunes. Localized and discrete phases of pronounced aeolian activity persisted throughout the Holocene until the beginning of the 20th century. As the city of Ghent developed, inhabitants may have been confronted with this late Quaternary windblown sedimentary environment. Historical maps and pictures of Ghent show numerous hills while over 40 “berg” (mountain) toponyms suggest their occurrence in the area. The current urban landscape has been largely flattened as a result of the city growth and development, and only small, short slopes and talus testify to an original relief of windblown dunes and ridges.

By combining evidence preserved in historical records and Quaternary maps with toponymical research, several possible coversand ridges and dunes underlying the area occupied by the city of Ghent, could be identified.

This paper reports on a chronological study of these deposits using quartz-based optically stimulated luminescence (OSL) dating. The main aim of this study is to contribute to an improved understanding and re-appreciation of the history of Ghent in its past environmental setting.

Urban improvement works allowed us to access dune and coversands at four difficult localities and 21 samples were taken for OSL dating. The Quartz OSL signal in the samples investigated so far is dominated by the fast component and procedural checks confirm the suitability of the applied SAR methodology. At the time of writing, analysis is ongoing but our first results already indicate that sand deposition occurred – in the Ghent city area – from ~ 16 ka onwards. As such, our study supports the idea that the hills, depicted in many historical images may have been more important morphological features, than previously assumed. They probably formed an essential component of the landscape, which should not be undervalued when studying the historical development of the city of Ghent from its earliest days to the onset of the 20th century.



LUMINESCENCE ABSOLUTE ANSWERS IN ARCHAEOLOGY: IS THERE A NEED FOR INTER- AND INTRA-(DISCIPLINARY)-APPROACHES?

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Absolute dating in archaeology celebrated its' continuous presence within the 50 years of International Symposia on Archaeometry (ISA 2012, May 27 – June 1, Leuven) and, more important, over 60 years of related research communications (Archaeometry journal special issue 50(2), 2008).

Luminescence dating and authenticity testing are supported by a dozen of acknowledged laboratory protocols, routinely applied to provide absolute decisions on issues ranging from artifacts to geoarchaeological questions.

A key parameter for successful dating applications stands, and shall always be an issue, that is the as much closely as it can be, collaboration between individuals from the humanities and science; this topic is considered as sufficiently covered by scholars

oriented from archaeometry and archaeological science.

Furthermore, and valid only for archaeochronometry scholars there are specific topics and still open research issues all forming two distinct areas: intra- and inter-technique approaches. The term intra-technique (IAT) refers to issues that occupy the range of issues within one individual dating technique (e.g. luminescence) and inter-technique (IET) refers at cases where combined use of more than one technique is to be applied (e.g. radiocarbon and luminescence).

The present paper reviews significant IET case studies from up-to-date European published literature; but also provides space for IAT cases in an effort to highlight the significance of intra-disciplinary data collection for more accurate and successful luminescence dating studies.

CHRONOLOGICAL SEQUENCES FROM THE PALAEOLITHIC CAVE OF KLEISOURA AT ARGOLID, GREECE: DATING AND DOSIMETRY ISSUES.

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Klissoura Cave preserves a long series of Middle Paleolithic, Upper Paleolithic and Mesolithic cultural layers (Koumouzelis et al., 2001; Karkanas, 2010), interrupted by at least three significant erosional hiatuses. The sedimentary features, artifacts and animal remains of the Upper Paleolithic though Mesolithic layers testify a wide range of on-site activities, with complex cycles of feature construction and abandonment.

Recently (Kuhn et al., 2010), reported 29 radiocarbon dates from Middle and Upper Paleolithic layers at Klissoura 1 Cave, all but two obtained from material identified as wood charcoal; ages obtained range from 25 – 60 ka BP.

Within the frames of the present study, a total of 15 sediment samples were collected during a field trip using metal cylindrical tubes placed at depths from 2-6 m; in all sampling spots, a dosimeter capsule was inserted into the inner most of the hole left by the use of the cylinder, containing both LiF and Al₂O₃ phosphors and buried for a four months period before recover. Use of dosimetry phosphors was a demand originated by the scattered presence of ash layers that bracket calcitic and terra rosa sediment layers.

SAR and TT-OSL (Athanassas and Zacharias, 2010, Fig. 1) protocols were applied on quartz aliquots which found to be in agreement and providing ages that span from 40 – 120 ka.

The study reports on the luminescence doses recorded but also on the importance of applying *in situ* dosimetry studies and laboratory statistical analysis for the accurate estimation of surrounding radiation field.

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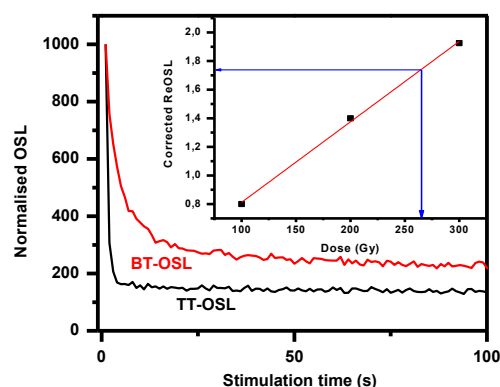


Figure 1. Typical TT-OSL and BT-OSL decay curves for the sediment sample D115 Inset: resulted Re-OSL growth curve yielded a De value (line) of 267.62 Gy.

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DATING TILES : TWENTY-FIVE YEARS OF INVESTIGATION IN THE LABORATORY OF FRENCH MUSEUM.

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For thirty years, ancient tiles are regularly identified during the restoration of historical monuments roofs (Querré et al., 1995). By their dimensions and typologies, they are often identified as medieval. However, their position in situ does not allow to use the usual rules of stratigraphy in order to test these hypotheses. The tiles can be original, as well as in re-used, or correspond to posterior restorations. We must therefore carry out a direct dating of tiles by physico-chemical methods (archaeomagnetism or luminescence dating).

The C2RMF, formerly the Research Laboratory of the Museums of France, has experience of twenty-five years in the luminescence dating of ancient tiles.

In early experiments (Vaury, 1989), the annual dose rate was not measured. However, thermoluminescence measurements allowed, at the time, to clarify the archaeomagnetic dating of tiles from the churches of Bussière-sur-Ouche and Collonge-lès-Bévy (Côte d'Or).

Thereafter, the radionuclides contents of the tiles were systematically measured. The external dose-rate was either neglected (St. Etienne's cathedral of Meaux, Querré et al., 1995), or based on the assumption that the contribution from the vaults and walls is similar to that of the tiles, the space above tiles are inert (St. Etienne's church of Etrechy, Zink and Porto, 2004). For Etrechy, we established a new protocol coupling TL and OSL on three samples per tile, distributed along a diagonal on the inner side.

This protocol was used in a comprehensive study on four churches in the Yonne: the cathedrals of Sens and Auxerre, the abbey of Pontigny, and the church of Vermenton, initiated by the centre for medieval study, Auxerre (Aumard et al, 2009). We worked on dozens of tiles grouped according to their type. The combination of luminescence dating and archaeomagnetic demonstrates the belonging of the tiles from the cathedral of Sens to the reconstruction of the thirteenth century, according to the dating of the structure by dendrochronology AD 1292 (Aumard et al., 2009). The analysis of the other monuments is still ongoing, but shows mainly productions included between the twelfth and the fourteenth century. (Aumard, 2011).

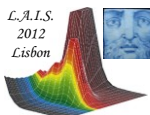
Most recently, we worked on a shipment of tiles found in a wreck in the Somme River, upstream of Abbeville, consisting of small flat tiles with a central hook and hollow tiles (ridge tiles?). The first results of the study (Rieth, 2011), still in progress, show dates from the late seventeenth century, in agreement with the dendrochronological dating of the wreck (*terminus post quem* AD1693).



Figure 1: BSO: Bussière s/Ouche; CLB: Collonge lès Bévy; VER: Vermenton; SET: Auxerre; PONT: Pontigny; ETR: Etrechy; MEX: Meaux; EPA: Epagne-Épagnette

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