Abstract Book

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In this study we date directly, for the first time, an example of a desert kite structure in the southeast of Jordan using luminescence signals from buried rock surfaces. These kites consist of two long low stone-walls lead outward in a funnel-like shape, often with some sort of stone enclosure where the walls meet; they are presumed to be animal traps used by hunters. Little known about the age of these kites because of an absence of attributable artefacts, and the lack of organic matter suitable for carbon dating.

The luminescence samples were taken from recently excavated kite in Jibal al-Ghadiwiyat in the east of al-Jafr (south-east Jordan). One rock sample was excavated from a pit in the kite enclosure; the sample was part of a long upright slab that forms part of the wall of the pit. Sediment samples from the infill of the pit were also collected for single grain measurements. The quartz from both the sandstone construction materials and the infill sediments (accumulated since site abandonment) are very suitable for luminescence measurements (high sensitivity, fast-component dominated).

The luminescence depth-profile through the rock slab shows the history of sequence of burial and daylight exposure for the two surfaces (the inward and out ward of the standing slap) Figure 2. Preliminary results from the fitting model of Sohbatí et al. (2011) and Freiesleben et al. (2015) gives a construction age of \(\sim 6.3 \text{ ka}\) from the exposed and \(\sim 6.5 \text{ ka}\) from buried (during use), indicating that the kite was not in use for a prolonged period.

\(*\) Speaker
Up to now, the methods of OSL measurements, when used for trap investigations, have relied on recording the luminescence decay during the optical stimulation with a constant energy $hn$ and a constant (Continuous Wave-OSL, CW-OSL) or linearly increased (Linearly Modulated-OSL, LM-OSL) flux of stimulation photons $f$. During these measurements the ratio of probabilities of the optical release of electrons from traps ($f = f_s$) that have different optical crosssections $s_1$ and $s_2$ is constant: $f s_1 / f s_2 = \text{const}$, because the $s_1$ and $s_2$ remain constant at constant temperature and constant stimulation energy. This causes that it is hard to separate the signal originating from different traps. In particular, the initial optical emptying of shallower traps before investigating the deeper ones does not bring good results (Kijek and Chruścińska, 2014), contrary to the TL analysis where the initial thermal emptying of shallow traps is a usual treatment when a signal from deep traps is going to be tested. In TL measurements, the ratio of probabilities of the thermal release of electrons from different traps changes in such a way that it is possible to empty the shallow traps effectively without depopulating the deeper traps. As it was shown recently, similar advantageous changes of the probability ratio during the OSL experiments, and in consequence more information about the trap than only the OCS value, can be obtained by inducing the changes of OCSs by increasing the stimulation energy (Variable Energy of Stimulation-OSL, VES-OSL) (Chruścińska, 2014). This method requires a strong tuneable light source that supplies stable flux of photons during the stimulation energy changes and, presumably, because of this the VES-OSL cannot be widely used. Such limitations, however, do not concern the possibility of inducing the OCSs changes by increasing temperature. Such a technique can be realised by means of standard OSL readers after a slight modification.

The method of experiment that is going to be presented here is somewhat related to a measurement called thermo-optical luminescence (TOL) (Hütt et al., 1988; Duller, 1997), however, a difference between the proposed regime of stimulation and TOL should be noticed. In the TOL experiment the OSL signal is stimulated by short (e.g. 0.1 s) pulses every few degrees (e.g. 10°C) during linear heating (a few degrees per second) in order to test the OSL intensity at higher temperature. In this way the thermal assistance effects can be investigated. Here a continuous stimulation is used in order to cause the OCS changes during the stimulation and generate the OSL curve shape that can help to obtain more information about the trap, e.g. the optical depth of trap and the electron-phonon coupling parameters, in the framework of the simplest OSL models. Although it was earlier shown how OCS depends on temperature (Chruścińska, 2010), this work, beside its main aim, gives a deeper insight in the character of this dependence and its relation with trap parameters.

The main objective of this work is to demonstrate what possibilities gives the optical stimulation during heating. The effects that the experimental parameters such as heating rate, stimulation light intensity and stimulation energy have on the OSL curve shape and its position on the temperature axis have been investigated in wide ranges of the relevant parameters. It turns out that all three factors can be the very useful tools for the regulation of OSL curve. By this kind of stimulation one can reach very deep traps that are not detectable by TL below 500°C. The resolution of the OSL signal originating from different traps is remarkable.


AN IONOLUMINESCENCE STUDY OF LUSTER CERAMICS, COMPLEMENTED WITH ELASTIC BACKSCATTERING AND FOCUSED ION BEAM-SCANNING ELECTRON MICROSCOPY

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Luster ceramics is an invention of Iraq Islamic potters dating back to the 9th century AD to decorate glazed ceramics. The main feature of this decoration is a very thin layer of Ag and/or Cu nanoparticles diffused inside the glaze some tens or hundreds of nm below its surface. An important issue when analyzing the technology of these artifacts is the determination of the composition, extend, size and concentration of the metallic nanoparticles forming the luster layer. In this work we report on the complementary use of the analytical techniques ionoluminescence (IL), elastic backscattering (EBS) and focused ion beam-scanning electron microscopy (FIB-SEM) to characterize IX-XVIII AD Islamic luster ceramic samples and to gain insight on the effect of the luster on the emitted IL spectra.

A set of 24 shreds of luster Islamic ceramics were supplied by the Ashmolean museum. Simultaneous EBS and IL was performed with the 5 MV CMAM’s tandem accelerator using a He beam of 3070 keV. IL spectra were recorded with an OCEAN spectrophotometer. A fragment in many of the samples was taken and prepared for FIB-SEM analysis at Center for Research in Nano-Engineering from UPC. EBS is a suitable technique for the study of the luster layer as it can provide quantitative determination of the concentration profiles of the elements Ag and Cu composing the nanoparticles. The information on the composition profile obtained from EBS was compared to the FIB-SEM images. Both results showed an excellent agreement, confirming the suitability of EBS for these studies. In many cases the glaze covering the ceramic may show luminescence induced by the analyzing ion beam, in our case He ions. The spectral composition of the light emitted may depend, among other things, on the chemical environment of the glaze, on the filtering effect of the nanoparticles forming the luster layer as well as on the damage produced by the probing He ion beam.

Relating the ionoluminescence to the composition of the glaze covering the ceramic, we have found, mainly, two different light emission spectra, depending on if the glaze contained or not lead oxide. The main conclusion regarding IL are:

IL from the glaze is mainly due to SiO2, showing a main emission band at 540-550 nm (2.3 eV).

The presence of lead oxide in the glaze, either originally or formed during the luster process, enhances IL emission bands at around 450 nm (2.8 eV) and around 680 nm (1.8 eV).

The presence of copper oxide in the glaze enhances IL emission bands at around 600 nm (2.1 eV).

Silver nanoparticles from the luster layer absorb, probably via surface plasmon excitation, the emission band at 450 nm.

No especial changes of the IL emission has been observed for the Cu nanoparticles luster layer.

It has been observed that the emitted luminescence may change with time, that is with increasing the irradiation dose. The general tendency for the band at around 540 nm is the peak to decrease with increasing irradiation dose. For the specific case of the peak around 680 nm in the case of a glaze containing lead oxide, and in the luster region with Ag nanoparticles, this peak exceptionally increases with irradiation dose.

*Speaker
AN INSIGHT ON SURFACE DATING OF BRICKS

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The routine thermoluminescence (TL) dating technique is widely used to assess the construction chronology of buildings, and to highlight possible restorations and modern remakes. Unfortunately, dating bricks can be misleading, since the dated event is the last heating (> 500°C, generally related to the brick production), that can significantly differ from that of the edification, also due to the practice of reuse. To avoid this problem a new technique, based on optically stimulated luminescence (OSL), has been developed. OSL surface dating uses the last exposure of a brick surface to light as the resetting event of the luminescence phenomenon. - A necessary requirement is that sunshine reduces the OSL signal of the superficial layers to a level at or near zero after short exposure.

The first experiments on luminescence of surfaces were made on marble sculptures using the TL signal of quartz [Polikreti et al, 2002; Polikreti et al., 2003]. Later on, new attempts were tried on granites [Geirlich et al, 2005] and clasts [Sohbati et al., 2012] using OSL signals, which are better bleached than TL ones, giving a higher precision. OSL has been used to date the surface of medieval Uzbek bricks [Vieillevigne et al., 2006 ], with encouraging results. The Surface dating technique is anyway far from being a routine procedure, even if very satisfactory results have been recently obtained on rock art examples from Utah [Chapot et al., 2012].

A further application of surface dating to bricks was recently attempted in a renaissance wall. [Galli et al., 2014] It was possible to distinguish bricks contemporary to the edification from others surely reused. The main requirement for the successful application of surface dating is the assessment of the bleaching effectiveness due to solar exposure. To evaluate the sunlight effects on the surface, small carrots of brick have been exposed to daylight for different periods (from 60 s to 1 year). Measurements of the OSL/IRSL signals as a function of depth showed that after a few hours the signal from the surface was reduced to 20% of the archaeological one. The deeper layers were significantly bleached after one month of exposure. The depth profile of signals depends on the opacity of the material, on the daylight spectrum and on the exposure time. To describe the characteristic form of the OSL /IRSL resetting profile in a brick, an empirical model in proposed, in which a time-dependent exponential term has been added to the exponential function of depth used to describe the penetration of light [Sohbati et al., 2012].

References:
M.S. Chapot et al., Quat Geochronol 13 (2012).
E. Vieillevigne et al., Radiat Meas

*Speaker
Introduction: Shellmounds are found in large number along the Southeast and Southern Brazilian Coast. Their sizes range from about 1 up to 25 meters altitude and from 2 up to 5 meters width. As to their origin there is no unique definition. Originally, it was supposed to be due to sea shore inhabitants that threw remains of shell fish bones in the course of long rivers. More recently, some authors claimed the shellmounds were made as burial site for some people.

Florianopolis Island, Capital of the Santa Catarina (SC) State, is located in Brazil South and it is an island. Around 50 sites containing shellmounds were found on it, most of them are medium and small sizes, containing organic particles, whole or fragmented shells and quartz grain with sediments.

Experimental: Samples of shellmound material from two sites, Ponta das Vílias (PV) and Canto dos Araçás (CA), located in SC State, were collected for dating. The samples were taken from base (B), "middle" point (M), and close to the top (T) of each shellmound. Initially, an aliquot of samples were washed in solutions of H2O2(20%v/v), HCl(conc.), and HF (28%v/v). H2O2 solution eliminates organic material and dark particles reducing almost 85% of the total sample aliquot. Since shells are basically calcite, leaching with HF and HCl promotes its dissolution. At the end, only remains a very small portion of sediments. The chemically treated samples were then sieved to retain grain sizes ranging between 0.080 and 0.180 mm diameter. Thermoluminescence (TL) and EPR were used for the accumulated dose (Dac) evaluation using the additive method. SAR protocol was applied in the OSL technique, however, only T sample was studied by this strategy.

In order to be feasible age determination, two data are needed the accumulated dose and the annual dose (Dan). There are several methods to calculate Dan, in this study the Ikeya [9] tables 4.3 and 4.4 of (1993, pp 109 e110) were used, however for applying these tables U, Th and K concentration are fundamental. U, Th and K concentrations were obtained by ICP-MS (Inductively Coupled Plasma Mass Spectrometry, Elan 6100 Perkin Elmer) analysis. To be possible these analyte determinations, around 50mg of each sample was digested with microwave-assisted technology (DGT 100 plus, Provecto Analitica) using an acid mixture (3 HNO3 (conc.):1.5 HF(conc.)). The ICP-MS analytical program was obtained after the dilution of each mono-element SpexO stock solution, covering the concentration range from 0.5 to 50 ug/L to Th and U, or 10 to 100 ug/ml for K. A series of four GSJ (Geological Society of Japan) certified reference materials (JG1a, JA-3, JB2 and JB-3) and one soil certified reference material (IAEA Soil 7) were also analyzed to check analytical method performance.

The ages obtained for Ponta da Vílias samples were for T sample: a) by TL 4.89±0.51 years B. P., b) by EPR 5.11±0.55 years B. P., c) by OSL 4.21±0.45 years B. P., B sample by TL 7.01±0.65 years B. P.

Results: From CA site 3 shells samples were collected besides sediment. Smaller shells were crushed and sieved for TL and EPR analysis. Accumulated doses (Dac) obtained were: Dac (TL) = 36.46±0.31 Gy, Dac(EPR) = 34±0.30 Gy. ICP-MS measurements enable annual dose as 8.34±0.05 mGy/year. Cosmic rays contribution to Dan was 0.015 mGy/year. Ages calculated were 4360.7±0.4 B. P. (TL) and 4073±0.3 B. P. (EPR).

Table 1: PV Shellmound Age (years B. P. - Before Present)

<table>
<thead>
<tr>
<th>Sample</th>
<th>TL</th>
<th>EPR</th>
<th>OSL</th>
</tr>
</thead>
<tbody>
<tr>
<td>T</td>
<td>4.89±0.51</td>
<td>5.11±0.55</td>
<td>4.21±0.45</td>
</tr>
<tr>
<td>M</td>
<td>5.86±0.55</td>
<td>5.29±0.51</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>7.01±0.65</td>
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<td></td>
</tr>
</tbody>
</table>

Easel paintings contain various pigments dispersed in an organic matrix. From the Renaissance to the 20th century, lead white was the most common pigment, present in the preparation layers as well as in the paint layers, sometimes mixed with other colours. Lead white is composed of two main lead carbonates phases [3], i.e. cerussite PbCO3 and hydrocerussite 2PbCO3.Pb(OH)2. Historical sources reveal that various qualities of this pigment were proposed to the artist, and sold at very different prices. A thorough examination of treatises shows that the main quality marker of the lead white pigment was its optical features: after the pigment was synthesized, several post-synthesis treatments were applied, possibly in order to gain those desired optical characteristics. The optical and luminescence properties of the two phases of interest thus constitute an interesting issue, with applications in the field of art history: the aim is to connect manufacture recipes of the past with those properties. This work proposes new insights on the luminescence of cerussite and hydrocerussite. The two phases were synthesized in the laboratory. Fluorescence studies were performed using a EKPSLA pulsed laser (10 Hz), with excitation energies between 3 and 5 eV, combined with an ICCD camera. The signal was analyzed with an ACTON SP2300 monochromator. This contribution will propose explanations for the various emissions detected. Furthermore, new insights on the ionoluminescence of the cerussite phase will be given, based on irradiation at the AGLAE facility (C2RMF).

Pre-Colonial irrigation canals have long been recognized in the lower Salt River valley in and around Phoenix, Arizona. Results from both radiocarbon and archaeomagnetic dating as well as ceramic typology have been used to indicate that the canals were excavated sometime between ~1500 to 600 years ago by the ancient Hohokam (Howard, 1991; Eighmy and Howard, 1991; Henderson, 1995). In order to reconstruct the evolution of the canals and to better understand Hohokam culture and potentially the reasons for its demise more precise age estimates are needed. This study was undertaken to determine how precisely these canal fills could be dated with single-grain optically-stimulated luminescence methods. We present here results from our pilot study in which we analyzed eight samples collected from depths of 1.0 to 4.2 m from three distinct canal fills. All analyses were conducted on single grains and final age estimates were calculated using a minimum of 131 accepted grains. Our results show equivalent doses are predominantly normally distributed, but that both overdispersion (18-35) and skewness (0.5-4.4) values are moderately elevated. These latter indications would typically suggest that we employ a minimum age model, however, those age estimates which ranged from 480 to 740 years ago, are generally younger than expected for these fills and are not stratigraphically consistent. Final age estimates were instead calculated with the central age model. Those results ranged from 750 to 1360 years ago and better agree with other available age control and are stratigraphically consistent. Two samples collected from one of the older canals identified at the site were dated at 1230 to 1360 years ago, consistent with the dating of the earliest villages in the canal system. The two younger canal segments were dated between 750 to 970 (n = 3 ages) and 990 to 1040 (n = 3 ages) years ago. The former was consistent with ceramic materials incorporated in its fill. These initial results indicate that single-grain OSL dating can be used to successfully distinguish between different aged canal fills in this setting.


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The development of OSL dating in the 1980s was a watershed for archaeological chronology. Yet while widely applied throughout many regions, in Egypt the development of OSL coincided with the introduction of a law that enacted a ban on the export of antiquities. This ban included a clause which continues to prevent the removal of archaeological samples for scientific analysis. Thus, the study of Egyptian archaeology has not benefited from this important chronometric tool. Nowhere is this more evident than in the study of ceramics, which continue to be dated using relative dating techniques only. Recent research undertaken at the Research Laboratory for Archaeology and the History of Art, Oxford, has aimed to bring OSL dating to Egyptian ceramic chronology. Specifically, a new sampling protocol, the minimum extraction technique (MET), has been developed in order to conduct OSL dating on ceramic material housed in museum collections. As the name implies, MET removes only a tiny sample for analysis from the ceramic, thus ensuring that the aesthetic integrity of the museum object is upheld at all times, with minimal damage being inflicted on the vessel.

This paper will discuss MET and its development, and will also present OSL results obtained from Egyptian ceramic material using this sampling protocol. This project focused specifically on dating the end of the Early Dynastic period as it transitions into the Old Kingdom: the beginning of the age of the pyramids and the culmination of Egyptian state formation. Ceramic material obtained primarily from the site of Beit Khallaf, an early Old Kingdom elite burial site, has been dated. By examining the absolute chronology of this material using OSL, our research assigns the first calendrical dates to this transitional phase of Egyptian history, and to the best of our knowledge, we present here the first OSL dates determined for ancient Egyptian ceramics.
Access to water resources was vital for the survival of ancient peoples inhabiting arid landscapes. In such regions, surface water quickly becomes unavailable through evaporation or infiltration into the subsurface. To gain access to the subterranean water supply required new and innovative techniques. One example of this innovation was the development of the qanat. The qanat (also known as Foggara, Falaj, Khettara, Kariz, and many other names) is effectively a water tunnel dug horizontally into the subsurface towards a vertical, water-producing well located in the hinterland that is used to conduct water towards the plains for agriculture and subsistence. Vertical access shafts are dug at regular intervals along the tunnel to both remove the excavated material and provide access during routine maintenance. The material removed from the qanat via the vertical shafts is piled onto the ground surface surrounding the opening to form a mound resembling a ring, and often referred to as a ‘doughnut’.

The dating of the construction of qanat systems, however, is problematic. Although in some instances, historical documentation or association with nearby settlements may help shed light on the age of construction, the frequent absence of suitable organic materials associated with the formation of the mounds has led to a general absence of the testing of the chronology of qanat systems using absolute dating methods. However, recent work conducted on a site in northern Spain (Bailiff et al., 2015) has successfully demonstrated the potential of OSL applied in conjunction with micromorphological analysis to investigate the chronostratigraphy of qanat shaft mounds. Similar work in Iran (Fattahi, 2015) has also underlined the importance of finding minerals with favourable optical bleaching characteristics and the complexities that arise when this is not the case. Given the nature of the deposition of sediments that form the mounds, the generally more rapid optical bleaching characteristics of quartz OSL compared with that of feldspars is a significant advantage. Hence a detailed assessment of the luminescence characteristics of the mineral suite available within the shaft mound deposits at a single grain level is of particular importance.

This paper presents the preliminary results of the assessment procedure applied to a qanat system in southern of Morocco. Basic mineral composition and luminescence sensitivities from coarse grains were assessed using an OSL laser-scanning system (Bailiff and Mikhailik, 2003), with both untreated and HF-etched separates. A measure of the overdispersion of equivalent dose values was obtained by applying a SAR procedure to small single aliquots, together with the application of a bright grain count procedure. Soil micromorphological examination of block samples taken from the mounds will also be applied to evaluate sediment structure and composition to reconstruct the site formation processes. Combined, these two approaches will allow the OSL age determinations to be placed within their proper sedimentological context.

References

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PRELIMINARY CHARACTERIZATION AND THERMOLUMINESCENCE INVESTIGATION OF NORTON COUNTY AND HOLBROOK METEORITES

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Meteorite falls have attracted the attention of scientists earlier than the mid of the 20\textsuperscript{th} century and have been the object of extensive studies up to present. Earlier studies have focused on the composition of the meteorites, but the most widely investigated feature of these materials has been their Thermoluminescence (TL) behaviour. The majority of the published works have been focussed on the natural TL of meteorites, which informs on the extent of the stored charges in the traps and thereby the absorbed dose and ambient temperature. The above can be used to gain an insight into their terrestrial age, orbits and thermal and irradiation history linked to extra-terrestrial exposure. In the same respect, laboratory induced TL of meteorites has also been studied to produce information regarding their mineralogy and crystallography, which can provide further information on the degree of metamorphism, post-metamorphic cooling history, shock and regolith history (Lal 1969; Sears et al. 2013).

In the present study an integrated approach towards the detailed characterization and investigation of the dosimetric properties of two meteorites is attempted combining several advanced techniques. The scope of the work is to enrich our knowledge on the luminescence behaviour of these materials and to test their potentiality to serve as space-probes and dosimeters. The ultimate goal would be to identify equivalent earth materials with similar properties that would allow the implicit study of meteorites and/or other extra-terrestrial materials without the need of their physical presence in the lab. The latter would permit the complete characterization of such rare materials towards the better understanding of space cosmic ray and/or the age of planetary materials.

The bodies and the inclusions of the meteorites were stereoscopically analysed by means of a high-performance stereoscopic microscope to gain an insight into their structure. In addition, their chemical composition was also determined, using non-destructive multi-elemental micro X-Ray Fluorescence Spectroscopy. Finally, their major dosimetric properties (e.g. sensitization, dose response) were also studied by means of TL.

Preliminary results indicate the complexity of the structure and composition of the meteorites (Fig. 1a) due to the existence of various crystal-like inclusions. It is the synergy of the above that contribute to the acquired glow curves during the TL measurements (Fig. 1b) which seem to witness the presence of deep and stable traps in both meteorites studied ascribing very interesting and worth studying dosimetric properties.

![Norton County meteorite: (a) Stereoscopic photomicrograph, (b) TL glow curves.](image-url)

**Figure 1:** Norton County meteorite: (a) Stereoscopic photomicrograph, (b) TL glow curves.

**References**
Ceramics – pottery and bricks were one of the first objects of the thermoluminescence (TL) dating over fifty years ago. Recently, the OSL method, next to the TL method is more frequently applied in the ceramics dating [Guibert et al]. The SAR protocol, which is used in dating, was developed for geological sediments. The parameters of OSL measurements, like the temperature, the optimal stimulation time, and the manner of bleaching the signal after OSL regauged measurements were established. Although in both methods, TL and OSL, quartz is applied, it is commonly known that the luminescent properties of quartz annealed during the pottery production in high temperature differ from the properties of quartz originating from sediments [Kijek el al.]. Therefore, some caution should be maintained in using the same protocol for sediment and ceramics dating. In the standard dating procedure, the OSL signal is read out at 125°C. In order to examine its influence on the dating results, the measurements of the De were carried out for three temperature T OSL during readout of the OSL signal in SAR protocol: 25, 70 and 125°C. Before a proper OSL measurement the preheat test was applied, in order to choose the optimal temperature for each sample. Besides the observation of the dependence of De on the temperature, the repeatability of the results (a recycling test) and the quality of bleaching the OSL signal after a regeneration dose have been monitored. For each OSL measurement temperature, the recovery test was carried out. The investigations were carried out for quartz originating from medieval bricks. The age was determined for the total of 12 samples which were collected in 2010-2013. In the quartz collected from the ceramics, the value of De is strongly dependent on the temperature of the OSL measurements. The greatest changes appears above 90°C. The obtained results of the dependence of De on the temperature of the OSL measurements (Tab. 1.) were used to determine the age of the sample for each temperature of the OSL investigations. For lower temperatures of the OSL measurements the errors of the De measurements, and also the errors determining the age, are generally lower. The comparison of the age assigned to various temperatures allows to conclude that the results for lower temperatures make it possible to obtain better correlation in groups of the samples which represent specified stage of building investigated object. The obtained dating results are actually consistent with the historical knowledge, but also allow to put forward a thesis concerning unknown fate of the church. It turned out that the samples originating from the lower part of the foundations of the church are older than the remaining part of the building, which shows that before the currently existing church there had existed an earlier brick construction at the same place and the brick from its demolition are the foundations of today’s temple.

<table>
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<td>1270.81±19.16</td>
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<td>JAK7</td>
<td>1652.33±35.06</td>
<td>1624.48±26.74</td>
<td>1586.66±34.99</td>
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</table>

Tab. 1. The results of the dependence of De on the temperature of the OSL measurements.
RE-INVESTIGATED: NEW LUMINESCENCE DATING RESULTS FOR THE MOUSTERIAN SEQUENCE LA COMBETTE (BONNIEUX, VAUCLUSE, FRANCE)

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Nowadays, methodological and instrumental advances in luminescence dating gained during the last two decades allow for precise and reliable age estimates up to ca. 250 ka. Even more, recent developments made in the field of optically stimulated luminescence (OSL) dating may demand for a re-investigation of sites examined using thermally stimulated (TL) or infrared stimulated luminescence (IRSL) dating to enhance and prove the previously obtained dating results.

Here, for the first time, we present OSL dating results for the Mousterian sequence La Combette using the fine grain (4–11 µm) quartz and polymineral fraction. The section is located in the Luberon mountain in the south-east of Avignon (France) and is known for its five well-preserved archaeological layers (A to F/G) over ca. 7 m of sediment indicating a late Mousterian occupation (Texier et al., 2003; Texier, 2004). The section has been detailed investigated between 1986 and 2003 using a multidisciplinary approach. Based on IRSL and TL measurements the chronological framework of the section was originally settled between 45 ka and 75 ka (Texier et al., 2003).

In summer 2014 the section was re-investigated for OSL dating using the quartz fine grain fraction. In contrast to (a) TL dating an age overestimation due to insufficient bleaching during transport of the investigated signal is not expected and (b) contrary to feldspars previously dated by IRSL the mineral quartz is not known to suffer from anomalous fading causing age underestimation. Furthermore, in loess environments the fine grain quartz fraction was proved to result in reliable age estimates up to ca. 120 ka (Kreutzer, et al., 2012; Fuchs et al. 2013).

Samples were taken from the loess dominated upper levels A to D (upper main unit) from the fluvial level F/G and the heavily disturbed gravel and block enriched layer E (both lower main unit). With our contribution the new dating results are presented along with a critical discussion on the implication for the interpretation of the occupation history of this important archaeological site.

References:


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searching for mycenean desfina (delphi, greece): luminescence dating of ceramic sherds and other archaeometric support

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Mycenean presence in the area of Delphi is scanty, though there is evidence of prehistoric habitation of Delphi region (province of Phokis) (Liritzis et al, 2015). We have initiated the sub-project of Mycenean Delphi Isotopes Project (MDIP) part of the Delphi4Delphi Program, for a) dating materials related to possible Mycenean habitation, by luminescence and C-14 dating methods, and b) carry out stable isotopes analysis & DNA of human (corroborated by animal bones and local springs) to investigate genetic similarities, diet and origin. The first attempt comes from Desfina village, sitting on a plateau ~680 m asl, across the pleistos river valley. An illicit excavation and robbing of a tomb at Kastrouli in Desfina left back several broken sherds and demolished human bones (Fig.1). Here three ceramic sherds are dated by thermoluminescence (TL) & optical stimulated luminescence (OSL) dating, and the human bone femur by C-14. Strontium, nitrogen and oxygen isotopes are measured for the human bone, as well as, a tooth of a recently died ram nearby the site. XRF composition of sherds was measured, as well as DTA and IR was used for determination of firing temperature of these ceramics. (Xingxiang Zhang et al 2014). OSL Dates fall within 1500-1200 BC. The cal C14 date was around 13th c BC. Isotopic data indicate that the Mycenean person had a mixed diet. Finally the firing temperature of ceramics examined was less than 800°C.

Fig 1 Left is typical ceramic sherds and right the femur bone.


∗Speaker
Among the multiple applications of Light emitting diodes (LEDs), archaeology and earth sciences have benefited too concerning the determination of time (chronology) based on the optically stimulated luminescence (OSL) method of dating artifacts, material culture of archaeological and geoarchaeological significance. Blue Light emitting diodes (LED) is mostly used for stimulation and bleaching of luminescence, in lieu of green and IR LEDs. Ancient inorganic materials made of geological rocks (carving, knapping, mixing, firing) are either fired in antiquity above 450o C or sun exposed for a short time (surface luminescence dating) and thus the electron traps are completely bleached for the “zero time” clock to set up. The importance of blue LEDs in archaeology was the research product of 2014 Nobel Prize in Physics. On the occasion of blue LED invention and Nobel Prize we offer an extended overview of the development of semiconductor physics focused on LEDs (blue, green, IR) and linked to the principles of OSL dating in archaeology.

The Nobel Prize in Physics 2014 was awarded jointly to Isamu Akasaki, Hiroshi Amano and Shuji Nakamura "for the invention of efficient blue light-emitting diodes which has enabled bright and energy-saving white light sources". When theyproduced bright blue light beams from their semi-conductors in the early 1990s (Akasaki et al 1992, 1993, 2014) they triggered a fundamental transformation of lighting technology.


*Speaker
Introduction: The lack of natural embayments along the Eastern shores of the Mediterranean made of Tel Akko a major maritime site with a lengthy history of continuous settlement and ancient trade activity spanning 4,000 years. Rich archaeological evidence has unearthed a strong connection with ancient Egypt (from the early part of the 2nd Millennium B.C.E.), Cyprus, Greece and others. In the 2nd and 1st Millennia B.C.E., Tel Akko has been an important anchorage/harbour site due to its strategic geographical location on the northern end of Haifa Bay, in Northern Israel, a focus of terrestrial and maritime routes. Notwithstanding, the exact locations of the ancient vibrant anchorages of Tel Akko, from the Middle Bronze Age to the Persian Period, are still unknown, as well as the precise demarcation of this ancient Mediterranean coastline.

Content: This multi-disciplinary research, framed within the Tel Akko Total Archaeology Project (a holistic innovative field school and research initiative) integrates various methodologies of the Earth Sciences, Archaeology and History to address not only the evolution of the landscape around this ancient site, in space and time, but also its own relationship with the different environments surrounding it, including the Crusader Akko - St. Jean d’ Acre, a UNESCO World Heritage Site, the Na’aman River, and the present coastline, now located ca. 1.5 km west of Tel Akko.

Besides incorporating a broad range of surveying techniques to understand the morphology and sub-aerial exposure of the tell, including but not restricted to aerial photography, traditional topography, and detailed historical cartographic analyses, all geospatially constrained, the sub-surface has been mapped with Electrical Resistivity Tomography (ERT) and Ground Penetrating Radar (GPR) in an attempt to locate possible ancient sea gateways, harbour structures, base rock, old marshlands and the ancient 1 Millennium B.C.E. period coastline. An extensive sediment collection campaign is also a major focus of the study in order to understand the palaeo-environmental evolution of the site through space and time. For this, long sediment cores have been collected, analyzed by a combination of Sedimentological, Petrophysical and Biological techniques with key units dated with Optically Stimulated Luminescence (OSL). Shallow test pits have been dug in the south area of the tell, in an attempt to understand its present shape, but also to examine the top-most loci and soil units in relation to other probable ancient paths and entrances to the site vis-à-vis the location of the ancient anchorage or sea gate. OSL ages presented herein are the first of their kind obtained for the study area. Other chronological techniques include identification of ceramics and many radiocarbon ages from one reference sedimentological core (i.e. Kaniewski et al., 2013; 2014).

Results indicate abrupt lithological changes along the western and southern faces of present-day Tel Akko. The presence of a marshy / lagoonal environment on the south vicinity of the tell are indicative of a shallow protected area, proper for an anchorage. The west vicinity of the tell shows a sandy coastline dating back at least to -3,000 years ago (OSL age, datum 2014), indicative of an active ancient bay shoreline. Over geologic time, the natural environment surrounding Tel Akko has experienced a progressive marine regression, shown in the latero-vertical progradation of its surrounding coastland.

References:
Since the first experiments conducted by Jain (2009) for probing deep traps in quartz using violet photons, with the aim to extend the dating range accessible with this natural dosimeter, a series of methodological experiments have been made for better characterizing the Violet Stimulated Luminescence (VSL) signal (Ankjaergaard et al., 2013; Hernandez & Mercier, 2015). In particular, it was shown that the VSL signal exhibits a dose saturation level which is about 20 times higher that what is usually observed with the OSL signal of quartz, and that the thermal stability of the VSL signal is excellent with a lifetime of 1011 years. In parallel, the first application of this signal to date geological samples has been realized (Ankjaergaard et al., 2015). In our poster, we report measurements of the VSL signal performed with a Lexsyg Research system (Freiberg Instruments GmbH) conducted for improving a single aliquot regenerative (SAR) dose protocol. First applications to samples of known ages coming from archaeological sites are also reported.

References:
The luminescence emissions of quartz (TL, OSL and RL) 

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The use of quartz as a natural luminescence dosimeter (1) is fundamental in OSL and TL dating. Many features of the luminescence emissions are well known, but at the same time still unknown are the defect centres responsible for the emissions. Also unknown are the dynamics that are at the basis of the changes of sensitivity typical of thermal treatments and/or irradiation sequences. Three main emissions are known to be present in quartz, in the red at around 620 nm, in the blue, around 470 nm, and in the UV at 340-380 nm. These emissions have been detected in many types of luminescence, in TL, OSL and Radioluminescence, RL. Specifically this latter technique, RL, allowed to find out that the blue and the UV emissions are in fact composite (2) and a role of alkali ions has been proposed in the above mentioned changes of luminescence sensitivity typical of quartz, also in the consequences on the activation of Al recombination centres (3-5). Recent results will be reported on the correspondence of the TL and RL emissions in the blue and in the UV, confirming the composite nature of both these emissions. Specifically, the presence of two UV emissions could possibly account for the differences present in the literature in the reported effects after irradiation and heat treatments, with important implications in many dating procedures.

References

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A NEW CONCEPT OF AUTOMATED TL-SET

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The Clermont TL-dating group practices routinely the quartz-inclusion technique with an additive + regeneration protocol (see, e.g., Bassinet et al., 2006). Since the dose-response curves of quartz are not simple, that is neither linear nor saturating-exponential, several different dose-points are needed for reconstructing these curves. Also, because the TL of quartz is usually scattered, it is necessary to measure several distinct aliquots for getting an acceptable uncertainty on a single dose point. For example, an additive dose-response curve will be plotted with a minimum of 5 different added doses, plus the zero-one (i.e., the natural TL), with 15 aliquots per dose. This leads to 90 distinct measurements at least. It is highly time-consuming, all the more since two dose-response curves have to be constructed for one dating. Because no commercially available TL-reader had enough positions for 90 samples when we began this program, we decided to build a specially designed one, with 100 positions. We also took this opportunity to imagine a system which would not need disks, so as to avoid a complementary source of scattering of the TL curves, due to the variation of thermal contacts with the heating element for individual disks. Our protocol allows avoiding two usual requirements for TL-sets: the aliquots are not re-used after measurement and we do not need beta irradiation. Actually, the samples are routinely irradiated with a gamma source, by batches of about 200 mg and measured long after irradiation, i.e., at least several months. This routine is intended at allowing the unstable components of TL to vanish. This is because, like other authors (e.g., Smith and Prescott, 1984) we have observed that the scattering of the TL curves has a tendency to diminish with the time elapsed after irradiation.

The mechanical system was built with the following principles. The sample holder is a carousel carrying 100 glass tubes (L: 25 mm; f: 5 mm) individually able to move around a horizontal pin. These tubes are intended at keeping the different aliquots of grains before they are poured on the heating element - a blade of titanium, 1 cm wide - by mechanical dumping of the tubes. A mobile centering ring allows a uniform spreading of the grains on the surface of the blade. The aliquots are prepared as for a manual TL-set, with a calibrated container which typically selects ~9 mg of grains in the range 200-300 mm. The photomultiplier tube is moved away during the phase of emplacement of the grains on the heating element. After measurements, the grains are eliminated from the heating element and evacuated toward a unique reservoir by a combination of air-pumping and vacuum-breaking.

The thermal regulation system is classic. It enables any programmable heating rate between 0°C/s and 20°C/s and any heating and pre-heating sequence. Interchangeable optical filters are fixed on the mobile photomultiplier holder and cannot be changed during the course of a measurement series.

Tests of reproducibility and stability were performed before a routine application.

Acknowledgements:
The system was designed and made in the technical department of the Laboratoire de Physique Corpusculaire de Clermont-Ferrand and especially by C. Crozatier (mechanics), D. Lambert and R. Chadelas (automation and regulation) and E. Delage (soft interface).

References:


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LUMINESCENCE DATING OF SEDIMENTS FROM ARCHAEOLOGICAL SITE IN JASTRZEBNIK CAVE, THE CZESTOCHOWA UPLAND, POLAND

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Jastrzebnik Cave is situated in the limestone rock located in the southern part of the Krakow-Częstochowa Upland (Poland). It is one of the most interesting Palaeolithic settlements in the region (Cyrek, 2009) and the stratigraphic sequence encompasses middle and upper Pleistocene sediments. Four aliquots were selected for luminescence dating. The OSL method, applying the single aliquots regenerative (SAR) dose protocol (Murray and Wintle, 2000) was used. The Riso reader, model TL/OSL-DA-20 (Bøtter-Jensen et al., 2010) equipped with blue LED light source for stimulation was used for OSL measurements. The newly established equivalent bleaching method (Przegiętka and Chruścińska, 2013) was applied for estimation of equivalent dose. The extended preheat tests were performed in order to choose optimal temperature value for OSL readouts (Kijek et al., 2013). The annual dose rates comprised of beta and gamma radiations, were calculated on the base of gamma spectra measured in laboratory with help of gamma spectrometry (Oczkowski and Przegietka, 1998). The radon 222Rn escape was observed for some samples and this effect is investigated in details.

The luminescence chronology supports archaeological and geological research approaching the cave as a complex archaeo-environmental study. The results of sedimentological, geomorphological, palaeozoological and archaeological investigations accompanied by OSL ages provided the basis for the reconstruction of the history of habitation at the Jastrzebnik Cave, in the background of palaeoenvironmental transformations.

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ITALIAN INTERCALIBRATION OF ALFA AND BETA RADIOACTIVE SOURCES FOR LUMINESCENCE DATING (CHNET PROJECT)

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CHNet is an INFN (Istituto Nazionale di Fisica Nucleare, National Nuclear Physics Institute) network formed by twelve Italian laboratories with acknowledged experience in archaeometry and cultural heritage diagnostics. It aims at supporting, organizing and funding the involved laboratories, and coordinating them to enhance the number and quality of the services available for the public.

For what concerns the luminescence dating techniques, CHNet supports the first Italian interlaboratory calibration of the irradiation by radioactive sources used for TL and OSL dating: 241Am and 90Sr-90Y. The involved laboratory are: Università di Milano-Bicocca, Università di Bari, Università di Torino, Università di Firenze, Laboratori Nazionali di Frascati. In order to improve the accuracy of the accumulated dose assessment in the main TL dating techniques (Zimmermann 1971, Fleming 1970), experiments are carried on both coarse and fine-grained material (180-250 and 4-11 µm respectively).

Each step of the adopted protocols will be described in detail, starting from the material selection through the sample annealing, preparation, packaging and transport, to the final luminescence measurements and data processing.

The beta sources calibration is obtained using the gamma irradiated quartz powders provided by Nordic Laboratory for Luminescence Dating, Aarhus University, Denmark (Hansen et al., 2015). To get calibrated alpha doses, two different irradiation experiments were set up in the tandem accelerator facilities of Naples and Florence While describing in details the irradiation protocols, three main experimental problems will be underlined: the sample positioning on an axis perpendicular to the beam, the beam spatial irradiation uniformity and the need of a constantly dark working environment.

The available preliminary results will be also shown.

References:

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In recent years the application of retrospective dosimetry using quartz extracted from mortar has been developed since Bøtter-Jensen et al. (2000) discovered that mortar is bleached to a considerable degree during manufacturing and thus it is suitable to be used as a dosimeter. After that some attempts were made in order to establish reliable OSL dating protocols (Urbanova et al. 2015, Panzeri, 2013, Goedicke, 2011, Gueli et al., 2010). Mortar dating is in fact unquestionably a better tool for the chronology of buildings than brick because it is made shortly before its use and it is not recyclable, hence it correspond to the time of the construction. Even though, mortar dating by Optically Stimulated Luminescence (OSL) has not become a routine method and its application to samples of known age must be carried out to establish the best measurement protocols.

Earthen mortars from Cremona (Northern Italy) are suitable for OSL due to their high quartz content (Cantù et al. (2015)). Four mortar samples from Palazzo Soldi (late XVIII century) and seven mortar samples from Palazzo Raimondi (late XV century) were analysed. Moreover it was possible to sample “sandwiches” of bricks still stick together with the original mortar.

Dose recovery preheat tests were carried out before OSL measurement in order to evaluate which were the best conditions of preheat temperatures. OSL measurements were performed using the single aliquot regeneration (SAR) protocol (Murray and Wintle, 2000, 2006). Equivalent doses (De) were obtained both on small multi-grain aliquots and on single-grain with the purpose of compare the data and determine which method gives the best results. Data were then treated using the statistical approach proposed by Galbraith et al. (1999) and by Roberts et al. (2000).

Data obtained by small multi-grain aliquots and single-grain aliquots are both highly dispersed (the former less than the latter), which indicates that the samples were not well-bleached during their last exposure to sunlight. The mortars ages estimated from small multi-grain aliquots are higher than expected. So the Minimum Age Model was applied in order to find and select grains that were completely emptied by sunlight during the preparation of mortar. Only for a few samples this model gives good results. In order to obtain reliable results also the Finite Mixture Model was used. The results obtained are compared and discussed.

References:

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Radiation Measurements, 32, 459-465.
In recent years, the direct application of optically stimulated luminescence (OSL) dating to rock surfaces has attracted considerable interest. In archaeology, there are many examples of rock surfaces, rock art, and stone structures whose chronology is of great importance to understanding the way in which people have used the landscape. We have recently developed a new technique of OSL dating that is directly applicable to rock surfaces. Here, we demonstrate the application of this new technique to rock samples from three different archaeological sites of different ages around the world: 1) a Rodedian prehistoric shrine from Israel, 2) a Barrier Canyon Style rock art from Utah, USA and 3) a whetstone from an Iron Age village in Denmark. The advantage of the new OSL rock surface dating technique to that of the conventional OSL sediment burial dating is that rocks can retain a record of their depositional history in their OSL-depth profiles. This allows us to recover multiple burial/exposure events in the history of our samples and, in two cases, estimate the duration of these events using known-age profiles.
The Orkhon Valley Cultural Landscape (Mongolia) is characterized by an extraordinary concentration of cultural remains of various nomadic civilizations including the ancient cities of Khar Balgas and Karakorum, numerous smaller settlements and fortified sites as well as burials. This study investigates the potential of different luminescence methods applied to quartz and feldspar to date young (< 2500 years) samples collected in the Orkhon Valley.
USING THE NEW R PACKAGE FOR TL DATING

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Since the development of the SAR protocol (Murray and Wintle, 2000), most luminescence dating applications use OSL rather than TL. When it comes to dating sediments, as often in Geoscience, using OSL is clearly the best choice. However, for burnt material, e.g., in archaeological context, using TL is still relevant. Moreover, the SAR protocol was shown to be easily adaptable to TL dating (Schmidt, 2013).

R is a programming language and environment for statistical computing and graphics. It provides a wide variety of statistical and graphical techniques and is highly extensible (R core team, 2015). A package specifically designed for luminescence dating is available (Kreutzer et al., 2012). However, it mainly includes functions for the analysis of OSL data.

We developed a new R package specifically designed for TL dating. The structure of this package is based on the existing Luminescence package. However, it treats the uncertainties completely differently. Analysis of TL data needs pre-treatment before any estimation of the equivalent dose. The background signal has to be subtracted and, very often, the peaks have to be aligned. These operations affect the uncertainties. Therefore, an uncertainty matrix, which contains an estimation of the random uncertainty for each data point, was added to the existing signal matrix, which contains the signal intensity/time information. Rather than estimating the uncertainties at posteriori, the new package functions update the uncertainty matrix each time the signal matrix is modified.

The TLdating package is designed to be as easy to use as possible. It includes two functions called script_TL.MAAD and script_TL.SAR, depending on the protocol applied. These functions only need the name of the .binx file, the uncertainty, and the temperature boundary for the integration, in order to provide De estimations. Complementary parameters, like the dose interval used for the growth curve, the fitting method (linear, exponential; weighted or unweighted) or the rejection criteria, help to improve the De estimation. However, if these are not specified, default values are provided. After using the readBIN2R function, from the luminescence package, these script_* functions successively call a series of other functions from the TLdating package, including: (i) Risoe.BINfileData2TLum.FileData, which convert the data in the proper format and create the uncertainty matrix; (ii) mod_extract.TL and mod_remove.preheat which only keep the TL curves and removing those identified as preheat curves; (iii) mod_substract.background which subtracts the background signal from the luminescence signal; (iv) mod_align.peaks which aligns the peaks; and (v) analyse_TL.MAAD or analyse_TL.SAR which provides the equivalent dose estimation using the MAAD or the SAR protocol, respectively. For the SAR protocol, the final result can easily be plotted using plot_AbanicoPlot from the Luminescence package. On the other hand, TLum.fileData2Risoe.BINfileData converts your data backwards, allowing to use all the functions from the luminescence package including writeR2BIN. It is therefore possible to analyse the modified data with, e.g, Riso Analyst. Finally, it has to be noted that the analyse_* functions provide not only a De estimation for the growth curve approach but also for the De plateau approach, allowing a better estimation of the temperature boundary that are used for the integration.

This project was realized in the context of the CRC 806 “Our way to Europe” funded by the German Research Foundation (DFG).


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To be able to date an object, one need to have a clock; that is a physical process, function of the time with an outstanding instant. In luminescence dating, the physical clock is the accumulation of absorbed energy, i.e. the dose, since the last heating. To obtain an absolute dating method, one need to calibrate the clock by a term reflecting the environment. It is made by measuring the dose-rate, or annual dose and by dividing the accumulated dose by the dose-rate. The dose rate received by an object has two components: an internal component (alpha and beta) from the object and an external component (gamma and cosmic ray).

In the case of archaeological artefact, external component can be measured, from soil sample in laboratory or directly using radiation measurements made at the artefact’s location. Such a measurement is not possible for museum object due to the ignorance of it location before it entrance in the collection. This ignorance on the external dose rate contributes largely to the total uncertainty on age.

To reconstruct the local environmental radioactivity, two steps are required.

1. Firstly identify information that is available and which will be used as proxy of the dose rate. This information can be vague and we only have global values. To build our expert knowledge, we built a database of dose rates from a survey of the literature covering the major part of the planet. The information can be explain if we have some idea of the region of origin of the object. We are in this case dependent of the current dosimetrical and geological data on the area.

2. Secondly, quantify the uncertainty on the external dose rate using statistical tools. The recent development of the Bayesian approach and numerical simulations in luminescence dating are a valuable asset for this purpose.

Starting from the experience of 40 years in the dating on museum objects, we will discuss the different solutions used in our laboratory, advantages and disadvantages, as well as future research directions.
**Venue address**
Centre de recherche et de restauration des musées de France
site Carrousel
Palais du Louvre - Porte des lions
14 quai François Mitterrand 75001 Paris
Dinner address
Le Bretagne
Bateau Parisiens
Port Debilly 75008 Paris

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