# Applying OSL for dating mortar, plaster and soil samples at the archaeological site underneath the Dubrovnik cathedral

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# Abstract

The 20th century has brought unexpected archaeological discoveries under the Baroque Cathedral in Dubrovnik (the UNESCO World Heritage Site). In the 1980s, a church complex older than the historically recorded Romanesque basilica was excavated. The dating of these finds is crucial, not only for understanding the historical changes of the Cathedral site but also for the earliest development of the historical centre of the city of Dubrovnik. After the archaeological research of the 1980s, in 2018, the first, more extensive processing of all findings was initiated through the cooperation of five Croatian scientific institutions (the project *Discovering the Old Dubrovnik Cathedrals* – DiODuCat). Within the framework of the IPERION HS platform, the DiODuCat 2022 collaboration was established with the CENIEH institute in Burgos (Spain) and OSL dating was applied to mortar and plaster collected from the internal layers of key structures in the sanctuary of the “first church”, as well as to the soil collected within its nave. The resulting data supplemented radiocarbon dating (AMS $^{14}C$) of plaster samples applied in 2021 and 2022. The new data combined to indicate a somewhat different chronological development of the "first church" than was suggested by previous scholarly works, implying that it had been constructed by adapting an earlier, Late antique edifice while giving a more precise chronology to the church's refurbishments during the early Middle Ages. These data complement the already formulated scientific assumption of the *DioDuCat* research team based on the marble sculpture analysis and, primarily, the observations of the *in situ* layers of plaster in the church's sanctuary.


# INTRODUCTION

# History of the research

The Dubrovnik Cathedral, as it stands today, is a remarkable Baroque structure erected between 1671 and 1713 on the grounds of its predecessor, destroyed during the 1667 earthquake (1). After the 1979 seismic event, structural reinforcement works were undertaken on the Cathedral’s foundations, including archaeological investigations (2, 3). Commencing in 1981 within the Cathedral interior, the research extended to the adjacent square (Bunićeva Poljana), ultimately encompassing an area of approximately 1,200 m² (Fig. 1) (3, 4, 5, 6, 9).[1]

As anticipated, the inquiry unveiled the foundation walls of the former Romanesque cathedral, whose existence is corroborated by the written sources records, historical depictions in paintings and graphics, as well as by the sculptural representations of the city of Dubrovnik (4, 9, 10, 12).[2] The cataclysmic earthquake of 1667, followed by fire, obliterated its remains, but aside from its foundations, among other archaeological findings, numerous architectural elements, fragments of stone furnishing and sculpture (4, 9, 7, 10, 11), and, based on recent studies, various fragments of wall paintings were found (4, 21).

A surprising discovery emerged when the remnants of an even earlier church were uncovered within the foundations of the Romanesque basilica, at some 2,40 to 2,60 meters beneath the baroque Cathedral pavement level. The investigation revealed the well-preserved architectural assembly of its sanctuary, the

adjoining nave, and the sections of southern and northern aisle. On the sanctuary apsidal wall, stone-built and plastered two-levelled *subselia* with central bishops' cathedra, as well as remains of a painted scene depicting the Church Fathers were still well preserved (Fig. 2) (4, 9, 10, 19, 21).

As the research further indicated, at the time when the construction of the Romanesque cathedral was well advanced, the interior of the 'first church' was filled with rubble and construction waste that served as the foundation for the new pavement. The rubble contained numerous fragments of liturgical stone furniture and sculpted architectural elements of late antique and medieval period, including secondarily carved stone and fragments of pottery and ceramic ware, glass material, and diverse metal objects (4, 7, 9, 19, 22). Among these findings, the most abundant were fragments of wall paintings, which can be classified in different historical and stylistic periods and attributed to different workshops and workshop circles (8, 10, 21).

Given the extraordinary discoveries, in 1983, the archaeological investigation expanded to the adjacent square (Bunićeva Poljana). Noteworthy findings included remnants of a defense wall, which bounded from the west the area encompassing the architectural remains of the 'first church', and those of the newly excavated quatrefoil building, situated next to the church's façade. The latter has been identified as a *memoria* in its early stages, with the hypothesis that it may have later served as a baptistery (4, 9, 19). In different stratigraphic strata numerous burials that encircled the entire church complex were further discovered (4, 20). Finally, at various zones within the site, ruins of medieval dwellings were identified, and the remains of the late medieval bell tower-baptistry, which had been dismantled during the planned extension of the square in the 19th century, were uncovered (4, 9, 13, 19). By the end of the archaeological investigation, it was obvious that the excavated complex represented one of the most compound and layered ensembles of the Eastern Adriatic Coast, whose further research would significantly enrich the knowledge and the understanding of the earliest development of the historic city of Dubrovnik.

Consequently, during the final research campaigns (1987–1988), the site was covered with reinforced concrete slabs, allowing the restauration of the Baroque Cathedral to continue, while keeping the accessibility of the archaeological site for future exploration (3, 5, 19, 23). Unfortunately, at that time all further research had to be stopped due to the war in Croatia (1991–1995), and the majority of the movable artifacts remained stored in the attic and the underground chambers of the Cathedral for almost thirty years, where they were at risk from the war damage and later exposed to unfavorable microclimatic conditions.[3] Following that period, only in 2018, a comprehensive revision of the 1980s research was initiated through the project *Discovering the Old Dubrovnik Cathedrals*, and the systematic processing of all movable finds finally began, inevitably including re-examination of the *in situ* masonry structures, while respecting data given by the archaeological documentation and the written records from the 1980s (16, 17).[4]

## Dating of findings

Due to the scarcity of historical records, the dating of the masonry structures within the “first church”, which was named by the first researchers as Byzantine basilica (3, 4, 9), relied greatly on the stylistic features of the fragmented architectural sculpture and liturgical stone furnishing, as well as *in situ* preserved wall paintings (7, 8, 10, 11). Nevertheless, the first published reports elucidated greatly its phases, as they included the analysis of the spatial and structural relationships between excavated walls and/or defined architectural units, also indicating traces of rebuilding and reconstruction activities, even some less discernable architectural modifications (3, 4, 5, 9, 19).

However, a broader dating range for the initial phases of the ‘first church’ was suggested, spanning from the early-Christian period of the 6th century, to different periods in the early Middle Ages (from the middle of the 7th century to the middle of the 9th century) (3, 4, 5, 7, 9). Additionally, at least four different phases of adaptations were proposed, notably visible in the well-preserved sections of the sanctuary and the eastern portions of the side aisles. Noteworthy alterations would include two different stages of the masonry clergy bench (*subselia*) in the sanctuary apse that was at one time extended to a double bench, and four stages of the bishop’s throne, which would later be expanded and elevated. Rich stratigraphy on the apse wall was identified, as layers of various plasters, limewashes, and wall paintings, spanning an extensive timeframe, were preserved. Significantly, the researchers also pointed out the later additions of columns and T-pylons in the nave and side aisles for the construction of the dome and vaults, as well as changes in stone furnishing (10th century). The persistence of the ‘first church’ culminated at the time when the construction of the Romanesque cathedral was initiated, when, in researchers’ opinion, the “Byzantine basilica” was first spatially reduced, and then partly demolished and partly covered with rubble (4, 9, 19).

Based on its construction techniques, an unearthed segment of a defense wall at Bunićeva Poljana was dated to Late antiquity (the 5th -6th century). The quatrefoil building posed a dating challenge. While its elevated position suggested an early Medieval origin, with discernable later modification (4, 9, 19), an alternate theory proposed its status as the oldest building within the complex. This hypothesis drew parallels with more reliably dated monuments from the broader Mediterranean area, as well as its proximity to the oldest excavated burials, which would typologically correspond to Late antiquity (24, 9). [5] However, more recently performed 14C analyzes of osteological material from one of those graves, as well as the burning layer under the quatrefoil building, point to the 7th to 9th centuries (14, 20), which together open up several research questions.

These considerations were later augmented by the authors delving into a more detailed analysis of the architectural evolution and the alterations of the church predominately based on the ornamented fragments of stone liturgical furnishing, including those from the broader area of Dubrovnik’s historic center (7, 10, 25, 26). But movable archaeological finds of the site in total, stored in the Cathedral’s attic, that could have played a pivotal role in enhancing the contextual understanding of the discoveries – more than 3000 pieces of diverse stone fragments and tens of thousands of wall painting fragments and ceramics, with numerous metal and glass objects – were until 2018 in greater extent excluded from all scientific considerations.[6] As they remained largely unprocessed for more than thirty years, the

collaborative effort was made through the project DiODuCat to initiate the first-ever expert analysis, systematization and cataloguing of all the finds, while including proper storage procedures (Fig. 3) (16, 17, 21, 22).

As the research of the stone fragments and the fragments of wall paintings advanced, a re-examination of the architectural structures of the ‘first church’ was prompted, considering that different layers of plaster and murals, as well as built foundations of the liturgical furniture, including the altar, were preserved in situ. Structural transformations, in parts, evidence of micro adaptations of the church furnishing have been newly identified. Additional late antique and early Christian marble sculptures, complemented by many heterogeneous marble fragments, which could be dated to the 4th to 6th centuries, were subjected to a more detailed analysis (16, 17, 22).[7] On the other hand, by correlating the stratigraphy of wall painting fragments with that of in situ murals, efforts were made to categorize them based on different types of plaster, revealing a relatively diverse array. Six samples of plaster layers were investigated more thoroughly to determine the type of aggregates and binders. The mineralogical-petrographic composition of the samples was determined by optical microscopy and scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS). In addition, one sample was analyzed by X-ray powder diffraction and thermogravimetry/differential thermal analysis (TG/DTA) to verify the presence of hydrated phases (21).[8] These analyses represented the base on which further methods of plaster dating were later planned.

At the Ruđer Bošković Institute in Zagreb, we conducted analyses using the 14C AMS method.[9] These results not only confirmed earlier hypotheses but also provided a new set of data that allowed us to identify numerous remains of wall paintings from the Romanesque cathedral that were previously unidentified. The most significant aspect for our discussion, however, was the results of the examinations of the samples of plaster from the sanctuary apse wall of the ‘first church’, connected with the oldest phase of its wall paintings, as well as of the coal from the remains of the lowest preserved floor level in its sanctuary (18).

Although the results largely corroborated some of the assumptions and hypotheses of the 1980s researchers, they also offered more detailed insights into the chronological relationships of certain masonry structures. Notably, as the applied radiocarbon analysis shows, the grey plaster associated with one of the earliest phases of the sanctuary, featuring a single clergy bench, could be dated to the second half of the 7th to the early 8th century (18). However, a noteworthy observation of the sanctuary apse structure indicates that the presumed oldest layer of grey plaster, which would correspond to the phase prior to the construction of the clergy bench, was applied later than previously considered. Namely, as we have established, the initial layer of the wall levelling in the apse interior comprised surface plaster resembling mortar used in the masonry of the apse wall (21).

Despite these intriguing discoveries, hypotheses proposing earlier structures in the ‘first church’ area still lacked support from natural science research.

To fill this gap, we turned to the IPERION HS consortium's additional dating methods. The project activities involved the application of the OSL for dating plaster and soil, along with the analysis of soil samples (red earth) using palaeomagnetism (archaeomagnetism).[10] OSL yielded effective results, with the outcomes of these analyses presented in this text.

# METHODOLOGY

**Sampling** The objective of employing dating methods on the building materials within the site is to precisely determine the construction timelines of specific elements, enriching the historical narrative of the complex. These data serve to further complement investigations and as supplementary to existing dating methods, including AMS 14C analysis on surface plaster samples and coal fragments as well as formal stylistic analyses of the discovered debris and equipment.

Careful consideration of sampling sites was undertaken collaboratively with the IPERION teamwork project leader – DioDuCat 2022 (Suzana Damiani), project coordinator (Maja Zeman), and other project collaborators, including an expert from the CENIEH Institute in Burgos, Spain (Alicia Medialdea), responsible for sample collection and analysis. Field work and sampling were conducted under the supervision of the Conservation Department in Dubrovnik, Croatian Ministry of Culture and Media (conservator-archaeologist Marta Perkić). Samples had to meet criteria such as representativeness, size, and availability, with a focus on ensuring the surface was unpainted and devoid of significant features like graffiti. Representativeness allowed for later interpretation, and the structure from which the sample was taken had to be clearly defined concerning the entire stratigraphy (Fig. 4).

The mineralogical-petrographic analysis of the plaster (18) identified quartz within the grey plaster on the first church's walls and the mortar within the building structure. This material is, therefore, suitable for Optically Stimulated Luminescence (OSL) dating. This technique estimates the time when quartz grains were exposed to sunlight for the last time. Given that the mortar, once mixed with lime and integrated into the wall structure, remains shielded (except for the surface) from the sunlight, this technique makes it possible to date the moment of construction. Sand, containing quartz, was found under the masonry structures, and accumulated at the walls, most likely transported from a flood. In both cases, again, the last time that quartz grains were exposed to sunlight was right before being deposited and buried inside the structure.

Within the main nave, beneath masonry structures at and below the first church's floor level, a layer of red earth and sand, likely serving as cushioning for building stone elements, was identified. This sandy soil, rich in quartz, was deemed suitable for OSL analysis. Soil samples were taken from beneath the main nave's central structure from two locations (samples 1 and 2) and under the foundation wall of the Romanesque cathedral (sample 3). Dating these samples, coupled with archaeomagnetic analysis of the iron-rich red earth, aimed to provide an insight into when this substrate was applied for construction initiation (thus indicating the earliest construction date for these structures).

To target the time of construction, a sample was collected from the structure of the apse wall beneath the initial masonry *subselium*, representing the earliest construction phase in the first church (sample no. 4). Simultaneously, a sample of the surrounding soil was taken (sample no. 5).

The sample from the "grey plaster" was also extracted (sample no. 6). In this case, the sample size posed a challenge as it is a relatively thin layer (less than 8 mm) on the first church's walls, especially in the crucial apse area under the wall paintings. A compromise was reached, extracting a sample from the southern perimeter wall next to the existing damage.

It's crucial to highlight that the presence of the operator, responsible for data collection and interpretation, is paramount when determining sampling locations. Factors such as estimating sample size, material characteristics, and skilled sample collection in conditions without white light contribute significantly to the success and credibility of results (Fig. 5). Additionally, consensus among all stakeholders and interdisciplinary collaboration enhances the overall success of the investigation.

# Experimental procedures

Quartz grains of sizes 90–180 μm were extracted from each of the sample for optically stimulated luminescence measurements, following standard procedures (27). From the piece of plaster collected (sample no. 6), it was necessary to remove the outer 2 mm layer before any further treatment, to guarantee that only the shielded-from-light grains were extracted. This removing process was carried out by the Conservation and Restoration Laboratory at CENIEH. The burial age was based on the relation

$$Age\ (ka) = \frac{Burial\ dose\ (Gy)}{Dose\ rate\ (\raisebox{1ex}{\$Gy\$}/\raisebox{-1ex}{\$ka\$})}$$

where the burial dose is based on the OSL response and the dose rate is based on the radionuclide concentration in the matrix surrounding each sample (28). Ages are calculated in kiloyear (ka).

OSL was measured using automated Risø readers. The derived dose distributions were used to estimate the burial dose applying the Central Age Model (CAM, [29]). Dose rates were based on the radionuclide concentration of the surrounding matrix, determined by high-resolution gamma spectrometry. Heterogeneity of each sample matrix was taken into account; this refers to the cases in which there were mortar blocks or slabs next to the collected sand sample and in the case of the plaster, only the radionuclides in the wall behind it were contributing to the beta and gamma dose rates.

## Results

The OSL signals of the extracted quartz are fast-component dominated, with a good signal-to-noise ratio (Fig. 6a) leading to well-defined dose-response curves (Fig. 6b). The derived dose distributions have overdispersion values of 15%.

The estimated ages for four samples (numbers 1, 3, 4 and 5) of the six analyzed, gave consistent results (k = 2). The age of the plaster provided a more recent age than the other four samples. Results from sample 2 indicate a burial age of over 8 ka, therefore it cannot be related to any construction phase. This anomalous age is most likely related to the sand not being exposed to sunlight long enough to reset its luminescence signal before being used in the construction. This sand would have been bleached when it was last naturally transported, most likely by a flood (30). All ages are summarized in Table 1.

Table 1
Summary of results for luminescence dating including the dose rate of the matrix, the estimated burial dose and derived age. Errors are reported at k = 1.

| Sample | Lab code CENIEH | Dose rate (Gy/ka) | | | Burial dose (Gy) | | | Age (ka) | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **22-DBK-01** | FIX5LM-01 | 0.97 | ± | 0.03 | 1.39 | ± | 0.03 | 1.44 | ± | 0.06 |
| **22-DBK-02** | FIX5LM-02 | 1.36 | ± | 0.05 | 11.37 | ± | 1.01 | 8.39 | ± | 0.80 |
| **22-DBK-03** | FIX5LM-03 | 1.14 | ± | 0.04 | 1.80 | ± | 0.06 | 1.58 | ± | 0.08 |
| **22-DBK-04** | FIX5LM-04 | 0.92 | ± | 0.05 | 1.50 | ± | 0.05 | 1.64 | ± | 0.11 |
| **22-DBK-05** | FIX5LM-05 | 0.92 | ± | 0.05 | 1.34 | ± | 0.04 | 1.46 | ± | 0.11 |
| **22-DBK-06** | FIX5LM-06 | 0.65 | ± | 0.03 | 0.72 | ± | 0.03 | 1.12 | ± | 0.07 |

Except for this unrelated date of sample 2, the estimated ages provide key information to understand the evolution of the Dubrovnik cathedral site.

Following the initial expectations placing the apse's construction in an age older than that indicated by the analysis of the so-called grey plaster (dating to the late 7th and the early 8th century), the OSL analysis dated the apse structure (sample 4) from the late 3rd to the late 5th century.

In the inner part of the main nave (sample no. 1), the analysis indicated that the foundational construction occurred between the first half of the 6th and the second half of the 7th century. Surprisingly the soil beneath the foundations of the Romanesque cathedral (sample no. 3) was dated to the period from the second half of the 4th to the first half of the 6th century. This suggests that these foundations might rest upon significantly older structures, possibly related to those in the apse.

The analysis of the grey plaster (sample no. 6) revealed it to be an intervention dating from the mid-9th to the mid-10th century. This implies that this section of the church was plastered approximately a century later than the very similar layer of plaster in the sanctuary was applied (as indicated by the results of the AMS $^{14}C$ analysis, dating it from the late 7th to the early 8th century).

The archaeomagnetism method, intended for comparative purposes with samples 1–3 taken from the same locations, proved ineffective. Despite the iron-rich composition, these samples lacked the

representativeness required for accurate dating.

# CONCLUSION

The analyses detailed here shed light on the origins of the elements comprising the sanctuary apse structure and the soil beneath the foundations of structures in the main nave of the oldest church at the site. These findings suggest a timeframe from the end of the 3rd century to the middle of the 7th century. But, the time ranges for specific components (the apse 270–490; soil around the apse structure 450–670; the nave under Romanesque foundations 360–520; structure in the center of the nave 520–640) direct the research to further examine whether the first church was built earlier than previously considered – possibly already in the late 5th century – or whether its construction began with the adaptation of an older, late antique edifice, with the earliest so far established date in the period of the late 3rd to the late 5th century. While these data certainly do not claim to offer definitive conclusions about the earliest history of the Dubrovnik cathedral complex, they do hold the potential to reshape its historical narrative, and first of all direct the next research activities to more specific areas of the site, as well as to more specific research questions. Also, the findings presented here show that OSL dating could play a key role in establishing not only the chronology necessary to understand the evolution of Dubrovnik in its formative years but also its spatial development, pointing to a location of its primary ‘urban core’.

Figure 6. a) Representative OSL decay curve of quartz grains from sample 4; b) dose-response curve showing the natural luminescence signal and the corresponding burial dose.

# Abbreviations

**OSL -** Optically stimulated luminescence

**CENIEH -** National Research Centre on Human Evolution

**IPERION HS -** integrating Platforms for the European Research Infrastructure ON Heritage Science

**DiODuCat 2022** – Discovering of Old Dubrovnik Cathedrals 2022

**INFN-LABEC (INFN-CHNet network)** – Istittuto Nazionale di Fisica Nucleare - Laboratorio di tecniche nucleari per l'Ambiente e i Beni Culturali, Cultural Heritage Networ

# Declarations

This manuscript has not been submitted to, nor is under review at, another

journal or other publishing venue

**Availability of data and materials**

Sequence data that support the findings of this study have been deposited in CENIEH's system, Gestilab, under the code Fixlab_05_Iperion_DioDuCa-F (codes FIX5LM-01 to 06)”

### Competing interests

The authors have no affiliation with any organization with a direct or indirect

financial interest in the subject matter discussed in the manuscript. They all

declare that they have no competing interests


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### Authors' contributions

S.D. – conception and design, sampling assistance, drafting, writing and revising

A.M. – sampling, analysis, interpretation of the data, writing and revising

M.Z – historical and art historical data, writing and revising


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## Footnotes

1. A detailed overview of the research, which includes data from the so far unpublished fieldwork documentation of the 1980s archaeological research – stored in the Archive of the Institute of the Art History in Zagreb, the Archive of the Conservation Department in Dubrovnik, and the Archive of the Institute for the Restoration of Dubrovnik – is only recently given by Marta Perkić (19).
2. The construction of the Romanesque cathedral lasted from approximately the middle of the 12th century to the middle of the 14th century. Although the first known protomagister – *Eustasius* from Trani in Puglia (Italy) – is documented only in 1199, the tomb of the archbishop Andrea of Lucca (†1158), described in the written sources as built in the outer wall of its sanctuary, would indicate earlier date for the initial stages of its construction. For the *terminus post quem* stands the tomb of

the archbishop *Gerardus* of 1132, the last clergy member buried within the premises of the earlier church(4, 9, 10, 11, 19).

3. During the siege of Dubrovnik in 1991, when the artillery fire was launched on the historic center of the city, the Cathedral was hit by three shells that significantly damaged its roof. The finds stored in the attic were exposed to different weather conditions for twenty years, until the Cathedral's roof was renovated in 2012(19, 16, 23). Also, during the years of the excavation in 1980s and after the war in Croatia, some of the finds from the attic, including some stored in underground chambers, were moved(19, 21, 22).
4. The activities were first initiated as the research and educational workshops for the art history students of the Faculty of Humanities and Social Sciences University of Zagreb, in cooperation with the proprietor, the Parish Church of Our Lady in Dubrovnik (2015–2017) (representatives of the Faculty and the proprietor: T. Turković and I. Viđen; collaboration coordinator: M. Zeman; workshop leaders: N. Maraković, A. Marinković, T. Turković, I. Viđen, M. Zeman).The research and scientific project *DiODuCat* has been launched in 2018. It is carried out by the Faculty and the Parish Church, with the collaboration of the Academy of Fine Arts of the University of Zagreb, the Institute of Art History, and the Institute of Archaeology (project coordinator: M. Zeman, proprietor's representative: I. Viđen; team leaders: S. Damiani, A. Marinković, I. Ožanić Roguljić, D. Zelić, M. Zeman).
5. These are vaulted masonry graves with covers in the form of a two-pitched roof made of *tegulae*(4, 20). This type of graves in the Eastern Adriatic coast is usually dated to the Late Antiquity.
6. Only the finds of coins, medallions and metal crucifixes were systematically processed in the years after the 1980s research, and were recently published by Ivan Mirnik (27). Since 2016, as part of the external cooperation with the Faculty of Humanities and Social Sciences of the University in Zagreb and the Parish Church in Dubrovnik, the processing of glass objects has been initiated, and led by Nikolina Topić. The publication is expected in 2025.
7. hese new considerations could be complemented with the published data on the coins from the site, among which are the Roman examples spanning from the time of Tiberius (14–37 AD) to that of Theodosius I (379–395), while the Byzantine coins are dated, at the earliest, from the reign of Justinian I (518–527) (27). Furthermore, as the systematization of ceramics and pottery, conducted within *DioDuCat* activities, has revealed, the earliest findings of amphorae originate to the period of the 4th– 6th centuries (*Spatheion* 1, North Africa). The analysis has also established a greatest statistical presence of the so-called Byzantine and late Byzantine amphorae of the 7th to 13th centuries on one land archaeological locality (15).
8. This is a report No. 1026/20 480 1 on the analyses of preparatory plaster layer samples of wall paintings from the archaeological site under the Dubrovnik Cathedral, conducted by the Department for Materials, Laboratory for Cements, Mortars and Ceramics, Slovenian National Building and Civil Engineering Institute, Ljubljana.
9. Test report No 017–693/1/2021 of the relative specific activity of14C/age, Ruđer Bošković Institute, Division of

experimental physics, Laboratory for low-level radioactivities, Zagreb in collaboration with Center for Applied Isotope Studies, University of Georgia, Athens, USA.

10. Samples were taken in 2022 and were taken to CENIEH, Burgos. As a part of the same project, bones from graves from the area of the same site were also analyzed. The graphite balls were measured by accelerator mass spectrometry (AMS) at the dedicated beamline of the Tandem accelerator at the INFN-LABEC laboratory in Florence (INFN-CHNet network).

# Figures

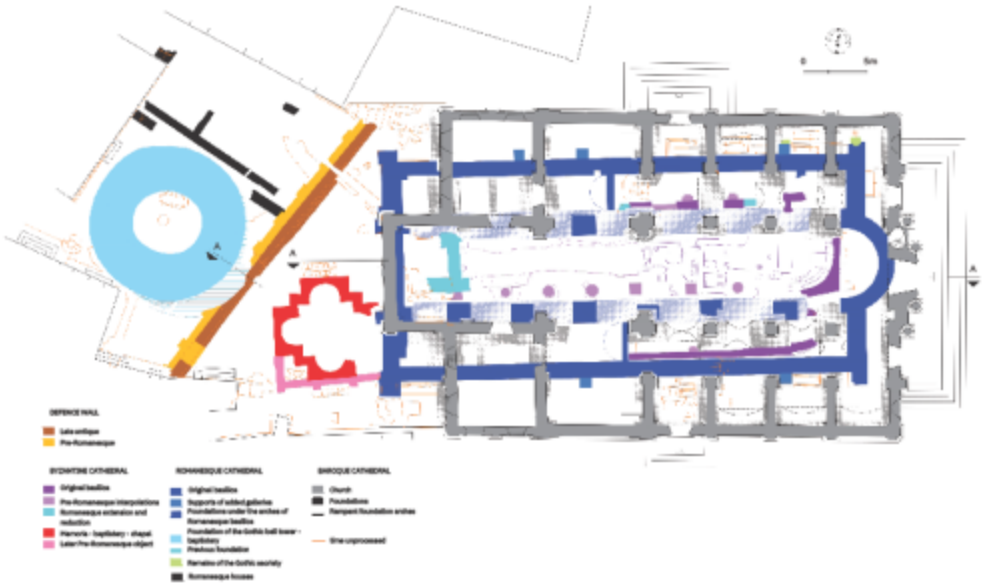

**Figure 1**

***Cathedral and Bunić Square, a schematic plan representing the chronological sequence of building structures***

Figure 2

*Remains of the apsidal wall with the wall painting fragment depicting the Church Fathers*

Figure 3

*Storage box with the fragments of wall paintings*

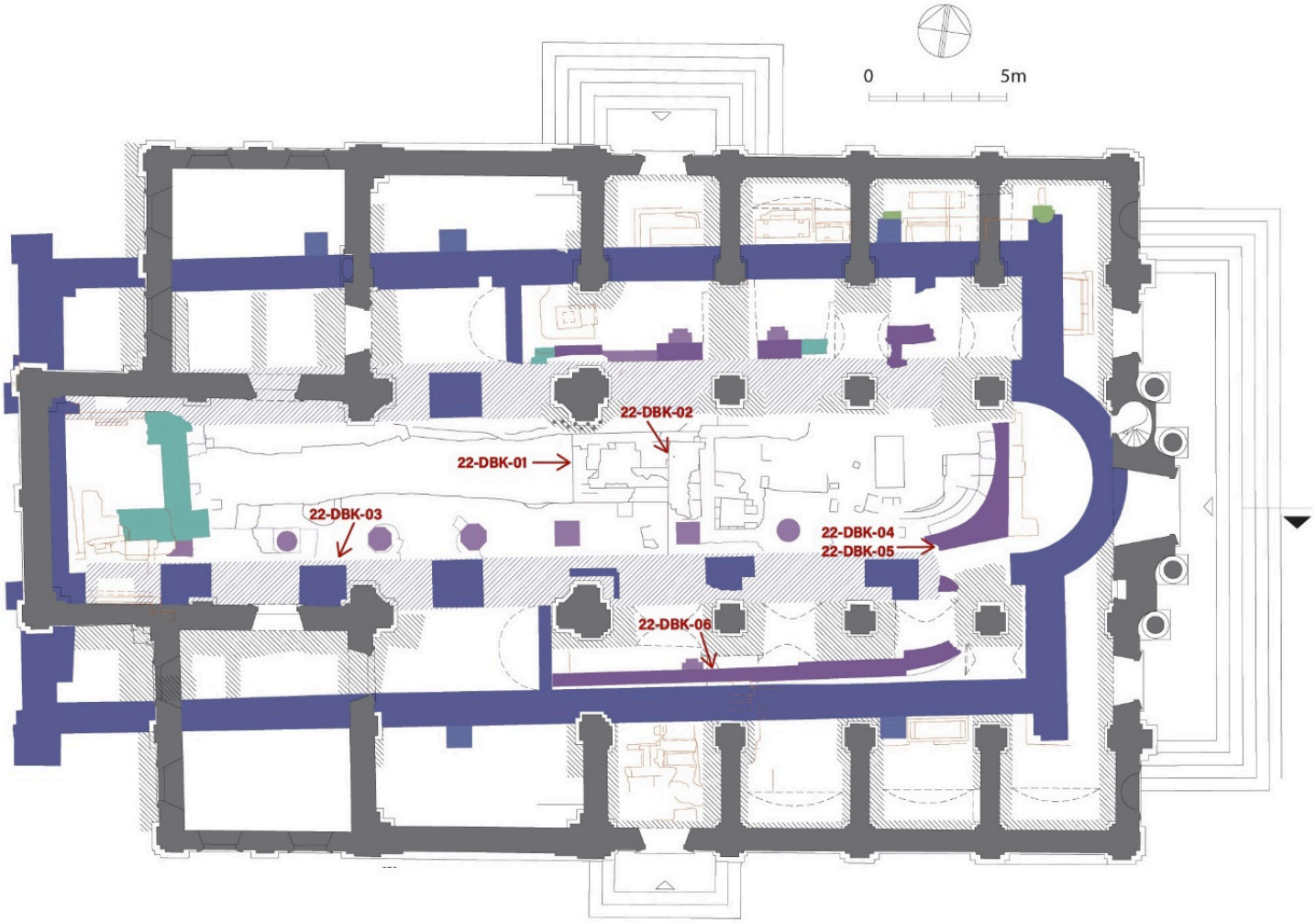


Figure 4

*Floor plan with the sample locations*

*22-DBK-01,02,03 – soil samples, 22-DBK-04 – mortar sample, 22-DBK-05 – sediment surrounding sample 4, 22-DBK-6 – plaster sample*


**Figure 5**

*One of the sample locations in the main nave*

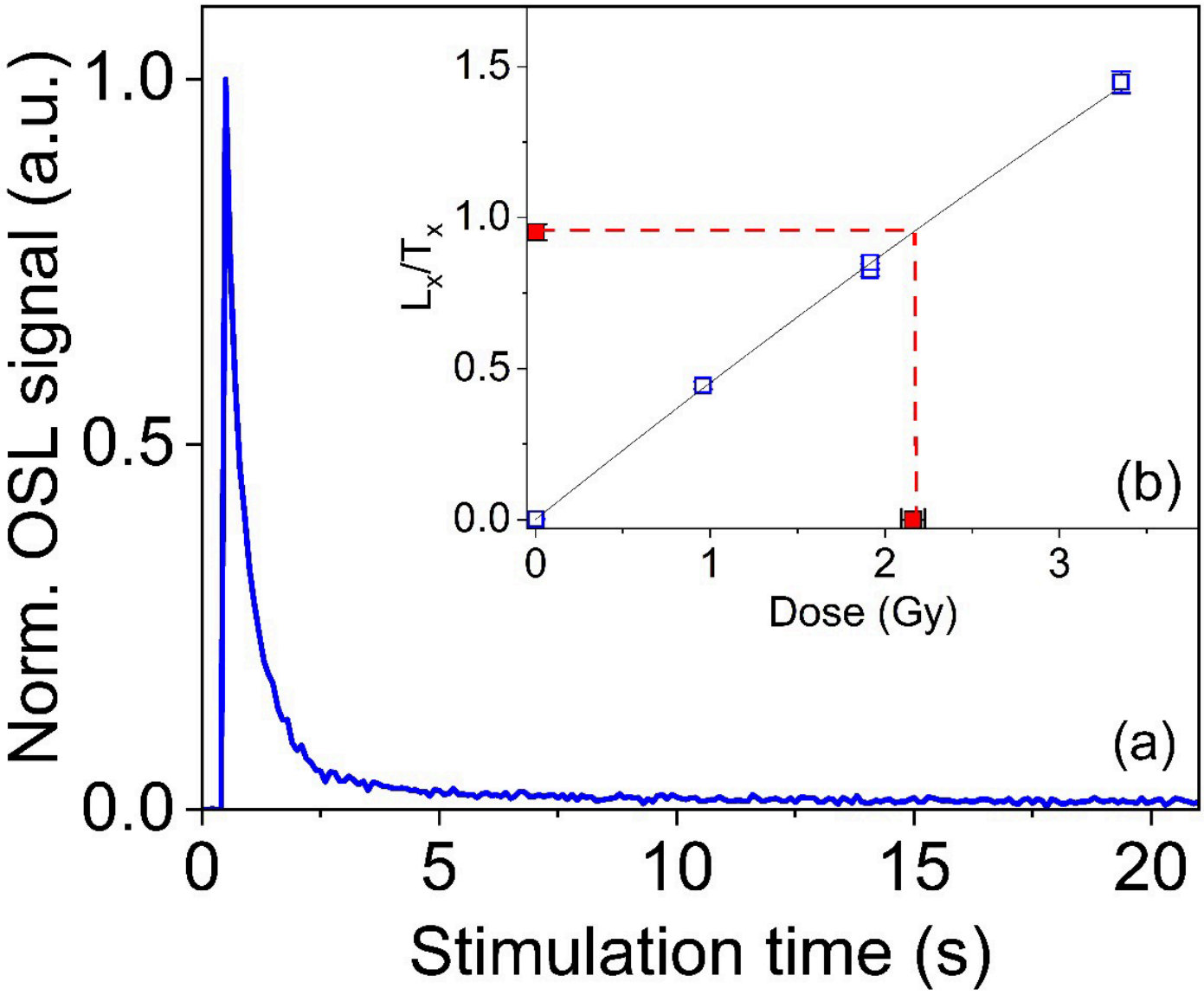


Figure 6

*a) Representative OSL decay curve of quartz grains from sample 4; b) dose-response curve showing the natural luminescence signal and the corresponding burial dose.*