# Radiocarbon dating of St. Stephen's in Pustijerna church in Dubrovnik, Croatia The Unknown Face of the Artwork

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ABSTRACT: Recent archeaological excavations of the church of St. Stepehen in Pustijerna, Dubrovnik, Croatia, undertaken in 2011/2012, comprised all cultural layers inside the church area and a segment of church cemetary underneath the church courtyard resulting in some important new findings. Several phases of the church development were documented, from the pre-Romanesque period to the destruction of the church in the Big Earthquake in 1667. Radiocarbon analyses 5 human bone samples proved the countinuous existence of the graveyard for several centuries, at least for the period 806 - 1286 cal AD, as well as the existence of the pre-Romanesque church before 806 cal AD. The charcoal samples resulted in the periods 963 - 1219 cal AD and 1296 - 1407 cal AD, and can be related to either burning after eartquakes or to the extension/ rebuilding of the church.

## 1 INTRODUCTION

The city of Dubrovnik (Fig. 1) is the most famous Croatian city in the world and its Old City was inscripted on the UNESCO World Heritage List in 1979. The town of Dubrovnik was created on the high and steep peninsula suitable for the defence that formed well-protected bay convenient for harbour development. The oldest part of the town developed around an old Byzantine citadel built in the first half of the 6<sup>th</sup> century and the new town was named Ragusa (Peković 1998, Regan & Nadilo 2006). Later, this became the first sexterium, i.e., urban area of historic core of Dubrovnik surrounded by thick and high walls. During medieval period, until the 16<sup>th</sup> century, the city walls were upgraded and fortified and today the 2-km-long walls are considered to be among the most massif defense fortifications in Europe. The City Walls protected the freedom of Dubrovnik Republic for centuries.

In the 10<sup>th</sup> century three sexteria (Fig. 2) already existed, as mentioned by the Byzantine emperor Constantine VII Porphyrogennetos in 949 AD (Tomašić 2003, Radić 2009, Peković 2010). In his book *De administrando imperio* he explicitly mentioned the church of St. Stephen in Pustijerna, today one of the most well-known churches in Dubrovnik. Although this book is the first written source which mentioned the church, construction period of St. Stephen's must be earlier according to archaeological excavations, i.e. archaeological finds and architectural remains which indicate to late 8<sup>th</sup> / beginning of the 9<sup>th</sup> century as initial period of this church. The church of St. Stephen in Pustijerna is one of the 24 oldest churches in the historical core of the city of Dubrovnik (Fig. 2) (Peković 1998, 2010).



Figure 1. Map of Croatia and the position of Dubrovnik.



Figure 2. Map of Dubrovnik with the limits of urban areas – sexterii (thick lines). The numbers refer to the following medieval churches: 1 – St. Mary "od Kaštela", 2 – St. Sergie and Bakh, 3 – St. Peter "Klobučić", 4 – St. Simeon, 5 – St. Peter the Great, 6 – St. Fosca, 7 – St. Michael, 8 – St. Stephen "in Pustijerna", 9 – St. Vitus, 10 – The Cathedral, 12 – St. Saviour, 13 – St. Tomas, 14 – St. Theodor, 15 – St. John, 16 – St. Andreas, 17 – St. Martin, 18 – St. Barbara, 19 – St. Michael archangel, 20 – St. Bartolomeus, 21 – All saints, 22 – "Sigurata", 23 – St. Nicholas, 24 – St. Lucas.

The area of Dubrovnik is prone to earthquakes and several earthquakes hit the city during its history. Among them, the most devastating one in 1667 leveled nearly every building in the city and pretty well ended Dubrovnik's golden age (Peković 1998). The St. Stephen's church collapsed in the Big Earthquake in 1667 and it has never been repaired. Since 1667 the church was no longer is use for religious purposes, but its yard was used as a *lapidarium* (a place where stone monuments and fragments of archaeological interest are stored and exhibited) until 1940. The stone fragments might have come from different locations and from different times, so they cannot be used for precise archaeological studies.

Because of its position and history related to early medieval phases of the urban development, the church of St. Stephen in Pustijerna is of great importance for history of the city of Dubrovnik. Recent investigation of the church with the aim of its reconstruction and conservation included also radiocarbon dating analyses in order to determine the time scale of the development of this church.

#### 2 ARCHAEOLOGICAL EXCAVATIONS

### 2.1 Previous excavations

The first excavation lead by Nikola Zvonimir Bjelovučić (Bjelovučić 1929, Gjivanović 1929) was undertaken in 1927, stimulated by the local brench of the Brethren of the Croatian Dragon (in Croatian: *Braća hrvatskoga zmaja*), the Croatian historical and cultural society established in 1905 (Gjivanović 1929, Regan & Nadilo 2006). This investigation revealed the outline of the smaller and older (pre-Romanesque) church beneath the Romanesque-Gothic ruins collapsed in 1667. Several graves were found outside the church, as well as stone plastic fragments which were dated to the end of 8<sup>th</sup> or the beginning of 9<sup>th</sup> century. A plan of the church was prepared and published (Karaman 1929).

Modern archaeological excavations have been performed by Dubrovnik Conservation Department of the Ministry of Culture of the Republic of Croatia in 1997-1998. It was found that the dimensions of the church were much larger than assumed by previous excavation. The majority of the undisturbed south wall was found, as well as fragments of the church fittings and furniture. The remains of an apse of the pre-Romanesque church were found - rectangular from the outside and semicircular inside, in accordance with apses of other churches from the same time (e.g., St. Peter from 10<sup>th</sup> century, no. 5 in Figure 2, and St. John in Pustijerna from 9<sup>th</sup> century, no. 5 in Figure 2), indicating the tradition of such apses from early medieval or even late-Antique period (Peković 2010). Several phases of the development of the church were shown: the first church belongs to pre-Romanesque style (8<sup>th</sup> - 10<sup>th</sup> century), while the second one, longitudinally extended, was built in Romanesque style of 11<sup>th</sup> or 12<sup>th</sup> century. In later periods (Gothic, Renaissance, Baroque) some interventions in church architecture were made, mostly after numerous earthquakes that hit the city. Such intervention/rebuilding was documented after the earthquake in 1348 (Peković 2010).

#### 2.2 Present excavation

Unfortunately the site of the church and its courtyard was never completely explored and documented by earlier excavations. Due to this fact and in order to reconstruct the church it was necessary to carry out new excavations which were undertaken by Omega Engineering d.o.o. in 2011/2012 (Topić et al. 2012). The new excavation comprised all cultural layers till bedrock inside the church area (trenches 1 and 3) and segment of church cemetery (trench 2) situated underneath the courtyard which spreads along north external wall of the church. Archaeological excavations revealed an earlier, pre-Romanesque church under the existing Romanesque one. A series of various building phases are also documented (Fig. 3), along with the constructed tombs which were incorporated into the floor of the later church. Research in the courtyard of the neighbouring building exposed remains of the chapel and street which used to pass behind the apse of the church. A graveyard which encircled both of the churches was also found. New excavations resulted in some important new findings – about 30 graves, coins, numerous pottery and glass fragments. Some pottery shards indicate the existence of Slavic or even prehistoric elements.

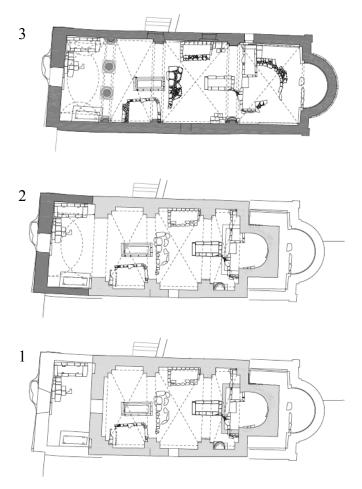


Figure 3. Development of the lay-out of the church of St. Stephen in Dubrovnik through the history. 1) pre-Romanesque period,  $8^{\text{th}} - 9^{\text{th}}$  century, 2) early Romanesque upgrade,  $11^{\text{th}}$  century, 3) Romanesque church,  $12^{\text{th}} - 13^{\text{th}}$  century.

This excavation (2011/2012) is very significant as it represents the first excavation of cemetery which had surrounded the church of St. Stephen. The graves inside the church were excavated in earlier campaigns, apart from some graves and ossuary situated on western side of the church which were not found or documented before. There are 6 grave stone constructions coated with plaster, explored in earlier campaigns which were found inside the

church and 34 new documented graves, mostly found in courtyard along northern external wall of the church and a few inside the church. The graves are mostly simple, only several of them were built with stone constructions (one in the western courtyard and seven inside the church). The existence of graves which bear witness on continuity of burials from early medieval to early modern period is proved. All floor levels found inside the church confirm that the church was in use in the same period as graveyard. This large multiphase cemetery outspread around the whole church on the area where houses were built later, relying to the church walls. More phases of the cemetery provide excellent insight to relative chronology of the site: early medieval Byzantine / pre-Romanesque, Romanesque, late medieval, and early modern periods. This excavation demonstrated that the first church of St. Stephen had adjoint graveyard, which was partially destroyed when Romanesque church was built. The foundations of Romanesque church were used as ossuaries, i.e., as lodgement of bones found during digging of foundations through pre-Romanesque cemetery. The same situation is proved alongside the apse and the western wall of Romanesque church. The Romanesque church also had own graveyard which continued to be in use in later periods. According to stratigraphy of the cemetery the church was extended in  $11^{th}$  or  $12^{th}$  century.

In trench 2 (courtyard along northern wall of the church, Figure 4) about 30 defined graves were found, with approximately ten burials in each phase, mostly orientated in direction E-W (with head on the west) and N-S (with head on the south); with negligible number of exceptions concerning the orientation. We found fragments of bone combs, pottery shards, fibula in layers close to earlier burials, and some grave offerings: coins, dices, belt-joints, bronze hoops, buttons connected with later phase of graveyard; but graves also gave some finds like pottery shards and glass fragments which were thrown as garbage into layers.

## **3** RADIOCARBON DATING

## 3.1 $^{14}C$ Method

The radioactive carbon isotope  ${}^{14}C$  is produced in the Earth's atmosphere by reaction of neutrons from cosmic rays with  ${}^{14}N$  nuclei. After oxidation to CO<sub>2</sub> it enters both the biological and the geochemical carbon cycles together with other carbon isotopes. Carbon is a constituent component of all living organisms, in which the equilibrium between the decay and replenishment of  ${}^{14}C$  is established. However, following the death of an organism, <sup>14</sup>C decays without replenishment and this is the basis of <sup>14</sup>C use for dating applications. Thus, by measuring the remaining concentration of the <sup>14</sup>C activity in organic materials, one can determine the elapsed time since the death of the organism. The half-life of <sup>14</sup>C is conveniently long (5730 years, giving the maximum age that can be determined by the <sup>14</sup>C method of approximately 60 000 years) to cover the period of development of human civilization. <sup>14</sup>C dating method is one of the most well-known radiometric methods of absolute dating and it can be applied for dating materials of biogenic origin, such as wood, charcoal, bones, grains, etc.

Radiocarbon dating of 2 charcoal samples and 5 human bone samples was performed in order to determine the time scale of the church history. Positions of samples within the church area are given in Figure 4 and description of samples in Table 1. Apart from radiocarbon analyses, the skeletons will also be anthropologically analyzed, because information on their habits and way of nutrition is also necessary and helpful.

# 3.2 Sample preparation

After pre-treatment of charcoal samples by the acid– base–acid method, the samples were combusted in a stream of pure oxygen. The produced  $CO_2$  was used for benzene synthesis (Horvatinčić et al. 2004) and the <sup>14</sup>C activity was measured by liquid scintillation counter (LSC) Quantulus 1220.

The collagen from the bone samples was obtained by the modified Longin method (Longin 1971, Sironić et al. 2012). The extracted collagen was combusted to CO<sub>2</sub> at 850°C over night in pre-baked (at 500°C for 8 hours), evacuated and sealed quartz tubes in the presence of CuO (BDH chemicals, wire form 2-4 mm) and Ag (Merck, purity 99.9% for elemental analysis). Amount of CO<sub>2</sub> corresponding to 1.5 mg of carbon is then converted/reduced to graphite in reaction with Zn on 450°C and iron powder at 550°C as a catalyst. The graphitization yield was high (>95%) for all four graphitization units and no memory effect was observed (Krajcar Bronić et al. 2010, Sironić et al. 2012). The prepared graphite targets were sent for <sup>14</sup>C measurement to the AMS facility in East Kilbride, Scotland, UK.

Standard material samples, both background samples (anthracite, marble) and reference activity sample (NIST SRM4990C Oxalic Acid II) were used for graphite preparation without chemical pretreatment (Krajcar Bronić et al. 2010).

Age calculation follows the conventional protocol (Stuiver & Polach 1977, Mook & van der Plicht 1999) based on Libby's half-life of 5568 years. The

results are presented as the conventional <sup>14</sup>C ages in years before present (BP, where 0 BP = AD 1950), with 1 $\sigma$  error, and normalized for stable isotope fractionation by using the default  $\delta^{13}$ C values (-24‰) for charcoal. The  $\delta^{13}$ C values of bone samples were measured in SUERC, East Kilbride, and are given in Table 1. All results are stored in the Zagreb Radiocarbon database ZAGRADA (Portner et al. 2010). The ranges of calibrated calendar years (cal AD) are obtained using software OxCal v.4.1 (Bronk Ramsey 2012) and are given with confidence level of 68.2%.

## 4 RESULTS AND DISCUSSION

The conventional radiocarbon ages, the calibrated age ranges and the median values of the calibrated age distributions are presented in Table 1. Plots of calibration for individual samples are shown in Figure 5, while the summary of calibrated ranges is presented in Figure 6.

The earliest bone sample (sample 2, grave 33, Z-4787) was dated to 806 - 882 cal AD, and the latest to 1260 - 1286 cal AD (sample 7, grave 7, Z-4792). These results prove the existence of pre-Romanesque church before 806 AD, because there must be an earlier built church beside the cemetery. The graveyard continuously existed for several centuries, analyses prove period from 806 - 1286 cal AD (graves 7, 8, 13, 22, 33), but we also found burials which belong to early modern period (Renaissance, Baroque) what is confirmed by the accompanied finds and layers in which they were found.

Beside the cemetery, in western part of trench 2 the chapel which collapsed at the end of 19<sup>th</sup> century was discovered. Underneath the chapel, on one side stone grave construction was discovered with the earliest burial dated to 12<sup>th</sup> century (sample 5, Z-4790).

The graveyard around the Romanesque church was in use till late middle ages, while the constructed tombs inside the church were in use since late middle ages till Earthquake in 1667 (Topić et al. 2012).

The charcoal samples resulted in the periods from 963 – 1219 cal AD (trench 1, stratigrafic unit 033, Z-4791) and 1296 – 1407 cal AD (trench 1, Z-4786, end of cultural layer in north half of the church). They can be related to burning during transition of pre-Romanesque to Romanesque period and to Gothic period of the church. The median of the calibrated age of sample 1 (Z-4786, 1351 cal AD) corresponds well to the restoration of the church after the earthquake in 1348 AD, which was documented in the archives of the Republic of Dubrovnik (Peković

2010). However, the context of these charcoal layers is still not completely clear and we expect to present the whole site in larger extent in future papers.

## 5 CONCLUSIONS

The recent systematic stratigraphic investigation of the church of St. Stephen in Pustijerna (Dubrovnik, Croatia) that comprised all cultural layers till bedrock resulted in uncovering some unknown facts of the history of church. Several phases of the graveyard in the courtyard were documented, early cultural layers below the pre-Romanesque object were discovered with the material that can be dated to late Antique, early medieval period and early Byzantine period (7<sup>th</sup> – 9<sup>th</sup> century). Further evaluation of numismatic, ceramic and osteologic material found in cultural layers will enable full insight in the church history.

The first church of St. Stephens was built in pre-Romanesque period. It must have existed already in  $8^{th}$  century, at least before 806 cal AD because in the early  $9^{th}$  century there is developed adjoint graveyard existing continuously through  $10^{th}$  and  $11^{th}$  centuries, as proven by radiocarbon dating of human bones. In the next phase, the Romanesque church  $(11^{th} - 12^{th}$  century) was longitudinally elongated maintaining the same width as the older church and incorporating earlier outer tombs into the church interior. This church also had a new semicircular apse. Rebuilding continues though developed and late medieval periods (as well as later during the Gothic and Renaissance periods), and the graveyard continues developing along the new church. The graveyards can be followed until 13<sup>th</sup> century (until 1286 cal AD, according to <sup>14</sup>C dates) outside the church, and then until the end of 17<sup>th</sup> century (the end of functioning of the church, until the Big Earthquake) in tombs inside the church. The area around the church has not been completely investigated yet, and it would be important to do it in future.

The project of restoration of the archaeological site of the church of St. Stephen in Pustijerna foresees presentation of various historical layers by different types of folding stony slabs.

There are many archival data in Dubrovnik that helped saving numerous historical documents. However, it may be surprising there are still so few precise and verified historical data on the city development and on the history of numerous churches in the city core. Therefore, there are still many new archaeological investigations to be performed in future.

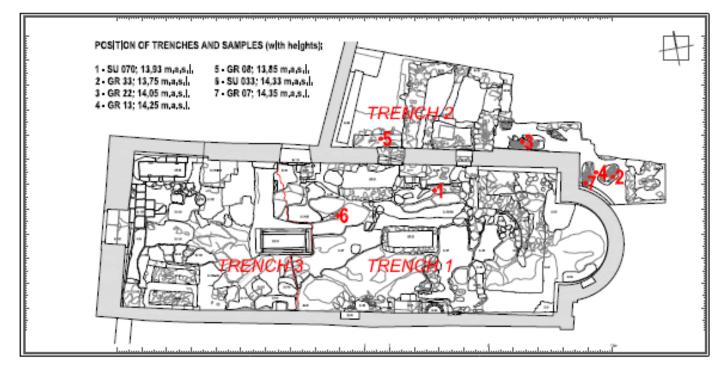
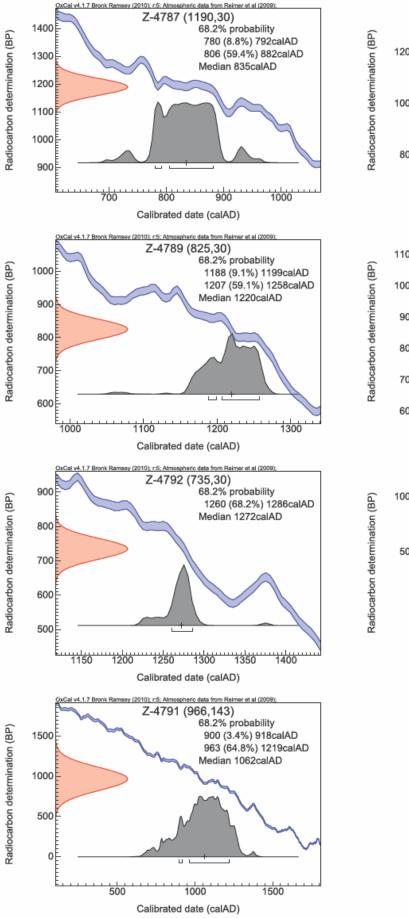


Figure 4. Detailed layout of the church of St. Stephen in Pustijerna with the position of samples for radiocarbon dating.



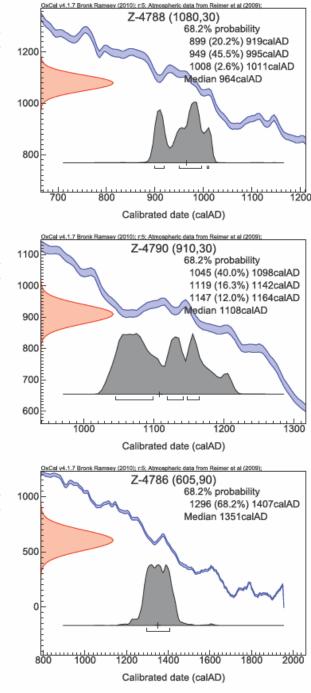


Figure 5. Individual calibration plots for all samples from the church of St. Stepehn in Pustijerna dated by <sup>14</sup>C. Radiocarbon determination in conventional age (BP) is shown on ordinate and the calibrated age ranges (cal AD) on abscissa.

Table 1. Sample details and results of radiocarbon dating of 2 charcoal samples and 5 human bone samples from the church of St. Stephen in Pustijerna, Dubrovnik. Samples were taken during excavations in 2011. Conventional radiocarbon age is defined in text. Calibrated age ranges and median values are obtained by OxCal 4.1.

No.	sample number	Z, B, A, GU codes*	description	δ <sup>13</sup> C ** (‰ VPDB)	conventional <sup>14</sup> C age (BP)	calibrated range (cal AD)	median (cal AD)
1	10	Z-4786 B875	Layer of charcoal in trench 1, bot- tom of cultural layer in north half of the church (stratigrafic unit SU 070)	-24.0	$605 \pm 90$	1297 – 1407	1351
2	88	Z-4787 A393 GU25913	Human bone – forearm, trench 2, grave 33 – the oldest grave	-20.0	$1190 \pm 30$	806 - 882	835
3	67	Z-4788 A381 GU25906	Human bone, trench 2, grave 22 – bottom of older graveyard level – with construction	-20.0	$1080 \pm 30$	899 – 919 949 – 995	946
4	36	Z-4789 A385 GU25909	Human bone, trench 2, grave 13 – upper graveyard level	-19.8	$825\pm30$	1188 – 1199 1207 – 1258	1220
5	79	Z-4790 A394 GU25914	Human bone, original burial, trench 2, grave 8 – synchronous with construction of chapel floor	-18.0	$910 \pm 30$	1045 – 1098 1119 – 1142 1147 – 1164	1108
6	03	Z-4791 B876	Layer of charcoal in trench 1, on a rock (stratigrafic unit 033)	-24.0	965 ± 145	963 - 1219	1062
7	25	Z-4792 A386 GU25910	Human bone, trench 2, grave 7 – upper graveyard level	-19.5	$735 \pm 30$	1260 - 1286	1272

\* Z-number is the identity code of a sample in the Zagreb Radiocarbon Laboratory, and B- and A-codes are the numbers of the benzene synthesis and graphite target, respectively (Portner et al. 2010). GU code number represents the identification of the sample at the AMS facility in East Kilbride.

\*\*  $\delta^{13}$ C values of bone samples were measured, while for charcoal a default  $\delta^{13}$ C value (-24 ‰ VPDB) was taken.

# Dubrovnik - St. Stephen in Pustijerna

DxCal v4.1.7 Bronk Ramsey (2010); r:5 Atmospheric data from Reimer et al (2009);

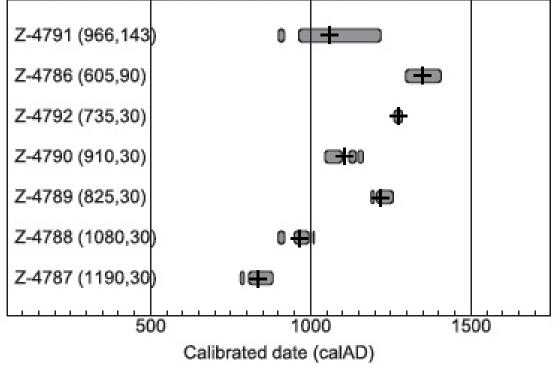


Figure 6. Multiplot of calibrated date ranges (cal AD) for all samples from St. Stephens church in Pustijerna. Median values are marked by "+" signs.

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### REFERENCES

- Bjelovučić, N.Z. 1929. Crvena Hrvatska i Dubrovnik. Zagreb: Matica Hrvatska.
- Bronk Ramsey, Ch. 2012. OxCal 4.1. on-line version <u>https://c14.arch.ox.ac.uk/oxcal/OxCal.html</u>
- Gjivanović, N. 1929. O otkopanim razvalinama crkve sv. Stjepana. *Revija Dubrovnik* 1(8): 278-281.
- Horvatinčić, N., Barešić, J., Krajcar Bronić, I. & Obelić, B. 2004. Measurements of low <sup>14</sup>C activities in a liquid scintillation counter in the Zagreb Radiocarbon Laboratory. *Radiocarbon* 46: 105-116
- Karaman, Lj. 1929. Iskopine u sv. Stjepanu u Dubrovniku. *Revija Dubrovnik* 1(8): 269-273.

- Krajcar Bronić, I., Horvatinčić, N., Sironić, A., Obelić, B., Barešić, J. & Felja, I. 2010. A new graphite preparation line for AMS <sup>14</sup>C dating in the Zagreb Radiocarbon Laboratory. *Nuclear Instruments and Methods in Physics Research B* 268: 943-946.
- Longin, R. 1971. New method of collagen extraction for radiocarbon dating. *Nature* 230: 241-242
- Mook; W.G. & van der Plicht, J. 1999. Reporting <sup>14</sup>C activities and concentrations. *Radiocarbon* 41: 227-239.
- Peković, Ž. 1998. Dubrovnik Beginning anf development of Middle Ages town. (In Croatian: Dubrovnik – nastanak i razvoj grada. Katalozi i monografije 5. Split, Muzej hrvatskih arheoloških spomenika.pp. 146.
- Peković, Ž. 2010. Conservation study report for the Church St. Stephen in Pustijerna in Dubrovnik. (In Croatian: Crkva Sv. Stjepana – Konzervatorski elaborat). Internal publication. Dubrovnik: Omega engineering d.o.o.
- Portner, A., Obelić, B. & Krajcar Bronić, I. ZAGRADA The new Zagreb Radiocarbon Database. *Radiocarbon* 52(2-3): 941-947.
- Radić, I. 2009. Crkve u gradovima bizantske Dalmacije opisane u "*De administrando imperio*" Konstantina Porfirogeneta. Bachelor thesis. Zagreb; University of Zagreb.
- Regan, K. & Nadilo, N. 2006. Crkveno graditeljstvo: Ranoromaničke sakralne građevine dubrovačkog područja (II.). (In Croatian). *Građevinar* 58: 231-242.
- Sironić, A., Krajcar Bronić, I., Horvatinčić, N., Barešić, J., Obelić, B. & Felja, I. 2012. Status report on the Zagreb Radiocarbon Laboratory – AMS and LSC results of VIRI intercomparison samples. *Nuclear Instruments and Methods in Physics Research B.* doi:10.1016/j.nimb.2012.01.048
- Stuiver, M. & Polach, H. 1977. Discussion: Reporting of <sup>14</sup>C Data. *Radiocarbon* 19(3): 355-363.
- Tomašić, N. 2003. Constantine VII Porphyrogennetos *De administrando imperio* Translation into Croatian. Zagreb.
- Topić, N., Radić, I. & Peković, Ž. 2012. Arheološko izvješće. Dubrovnik: Omega engineering d.o.o.