DATAREA RADIOCARBON A UNUI EŞANTION DE MORTAR HIDRAULIC DE LA VAMA DIN POROLISSUM ROMÂNIA
RADIOCARBON DATING OF A SAMPLE OF HYDRAULIC MORTAR FROM POROLISSUM CUSTOMS ROMANIA

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This paper presents the results of AMS radiocarbon dating for a sample of hydraulic mortar taken from foundations of the Roman customs Porolissum - Moigrad, Romania. We test the feasibility of the radiocarbon dating, namely that it delivers an age for the analyzed fraction which belongs to the historical period in question, and is not a result of late or recent interventions. The present case study provides a detailed description of the pre-treatment method of the sample, the stage of obtaining the calcite fraction originating from the moment of mortar hardening in the masonry, the stage of separation and purification of carbon dioxide resulting from the chemical digestion of the final analyte. Some original aspects were developed at RoAMS Laboratory in Magurele. The reduction of carbon dioxide to carbon in the presence of iron catalyst (graphitization), as well as the spectrometric measurement, calculations and calibration are also briefly described. Calibrated result and its interpretation in historical and archeological context recommend the use of this mortar sample in future multidisciplinary analyzes regarding the original recipe and possible sources of raw materials involved in the construction.

Acest articol prezintă rezultatele datării cu radiocarbon prin metoda AMS pentru un eşantion de mortar hidraulic prelevat din fundaţiile vămii romane Porolissum - Moigrad, România. Datarea cu radiocarbon dă încredere în eşantionul care urmează a fi prelevat, și anume că acesta aparține perioadei istorice în cauză, și nu ca urmare a unor intervenții tardive sau recente. Studiul de caz oferă o descriere detaliată a metodei de pretratare a probei, stadiului de obținere a fracției de calcit provenit din momentul întăririi mortarului în zidărie, stadiului de separare și purificare a dioxidului de carbon rezultat în urma digestiei chimice a analitului final. Unele aspecte originale au fost dezvoltate la Laboratorul RoAMS, Măgurele. Reducerea dioxidului de carbon la carbon depus intim pe catalizatorul de fier (grafitizare), precum și etapa de măsurare AMS, calcule și calibrare sunt de asemenea descrise pe scurt. Rezultatul calibrat și interpretarea acestuia în context istoric și arheologic recomandă utilizarea acestui eșantion de mortar în viitoare analize multidisciplinare privind rețeta originală și posibilele surse de materii prime implicate.

Keywords: *ancient hydraulic mortar, radiocarbon dating, Roman era*

1.Introduction

Roman lime / hydraulic mortar, as a material, continues to arouse interest due to its special properties and the impact produced over time on human society, both from a socio-cultural point of view and from a technical-scientific point of view. Determining the "manufacturing recipe" is important both in the processes of restoration - rehabilitation and enhancement of historical monuments and archaeological sites, and for its adaptation to current uses in certain types of construction.

The Roman lime / hydraulic mortar, as archaeological material, was used in present research to test and initiate a strategy for future archaeometry studies, useful for the Romanian community working in the field of cultural heritage - archaeological sites, but also to expand knowledge on this type of construction material, in general. One of the main issues with this type of material is the reliability of the ^{14}C dating and especially the representativeness of the datable fraction, e.g., the extent of reaching the equilibrium with the atmospheric ^{14}C content.

Specifically, the present study refers to a series of investigations carried out on fragments of Roman mortar with a binder role, taken from Porolissum wall foundations (Today Moigrad, Sălaj County, Romania). The relics are located in the northern Carpathian Mountains in the province of *Dacia Porolissensis* (Porolissum).

Porolissum is the name of a former Dacian fortification, later taken over by the Roman settlement, which had its beginnings as a military *vicus* near the Roman camp at Pomet, Mirșida commune, Sălaj County. The oldest military diploma discovered at Porolissum, was dated August 11, 106 AD, the date on which the second Daco-Roman war would end, the new province of Dacia already existing [1].

In the years 117-118 AD, after the death of Emperor Trajan (117 AD), the free Dacians and their allies, the Sarmatians of Roxola, attacked the new Roman province of Dacia, affecting even the Roman

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camps in the northern part of the province. The new Emperor Hadrian personally came to Dacia to stabilize the military situation and he divided the Trajan's province into two parts: *Dacia Superior* (which included western Oltenia, Banat, central and southern Transylvania, nowadays) and *Dacia Inferior* (which included eastern Oltenia and southeastern modern Transylvania). Later, a new province of *Dacia Porolissensis* was created in the northern province of *Dacia Superior*, as evidenced by the Military Diploma of November 12, 123 AD.

Between 106-117 AD, a wooden fortification and clay furrows (*murus cespecitici*) functioned on Pomet Hill. During the reign of the emperors Hadrian - Antoninus Pius, the enclosure of the camp was rebuilt using stone walls. The second phase of the restoration of the camp can be linked to an inscription from the year 213 AD [1]. Its position on the northern border of the Roman province of *Dacia Porolissensis* favored the development of a civilian settlement, which prospered based on trade relations with the inhabitants outside the borders of the Roman Empire. The development of this settlement led to its recognition as a city with the rank of *municipium* during the reign of Emperor Septimius Severus [1].

The civil settlement developed around the Roman camp at Pomet towards the west and north, along the imperial road that crossed the province of Dacia. The first information on civil settlement appeared in the first decade of the 20th century. Later, until the middle of the 20th century, the temple of Liber Pater, an amphitheater, a building with *thermae*, south of the Roman fortification, were identified. The temple of Jupiter Dolichenus was discovered in the 1980s [2].

Between 1986 and 1987, at approx. 300 m west of the Roman camp, a rectangular building was discovered, with sides of 35x47 m and having two square towers. This building was considered by Nicolae Gudea as a Roman customs [3], and by other researchers as a small Roman castle (*burgus*) [1]. The first phase was made of wood and clay, and the second phase was made of stone bound with mortar [4]. Next to this building, in the second phase, two rooms were attached, among which one was an office (*tabularium*). This indicates the existence at Porolissum of a *statio portoria*, as evidenced by two inscriptions discovered between the road and the fortification (Fig. 1). Both inscriptions were dedicated to the Emperor Commodus (177-192 AD) by imperial slaves (*vilici*), whom they called *restitutori commerciorum* [1, 2]. In the frame of restoration works in 2014, two sections and a cassette were practiced on the east side of the building (building FH 3), in the area of the former section S II from 1986 (Fig. 2) [5]. With this occasion, several mortar samples (mortar and associated lime nodules) were taken from the foundations. We supposed that these samples come from a context that is probably part of the second phase of building, respectively starting with the period of the Emperor Commodus, as revealed by the inscriptions discovered, mentioned above.

2.Experimental research

In order to establish the raw materials that make up the mortar and their possible origin, the antique lime-based mortar and the hydraulic antique mortar need basic definitions. Both materials are a product of ancient experience / knowledge. The ancient lime-based mortar is a

Fig. 1 - Roman building (customs or *burgus*) from Porolissum, second phase wall construction, east side (Photo: E.E. Sabo, 2014)
Edificiu roman (vamă sau burgus) de la Porolissum, construcţia de zid din faza a doua, latura estică (Foto: E.E. Sabo, 2014)

Fig. 2 - Roman building (customs or *burgus*) from Porolissum (FH 3); detail on the eastern wall in the area close to Section II, where samples of mortar and lime nodules were taken (Photo: E.E. Sabo, 2014) / *Edificiu roman (vamă sau burgus) de la Porolissum (FH 3); detaliu asupra zidului estic din zona apropiată Secţiunii II, în care s-au prelevat mostrele de mortar şi noduli de var (Foto: E.E. Sabo, 2014)*

binder formed, according to certain "recipes", from sand / quartz, lime, water and other possible inorganic or even organic ingredients. For constructions in areas with harsh, humid climate, but also for underwater foundations and constructions, the Romans used various assortments of hydraulic mortar. For this, they combined the above ingredients with a material that gave hydrophobicity properties and increased resistance by the very presence of water and moisture. At the beginning, they used pozzolana (a special material of a volcanic origin exploited right from the underground Rome, next to the volcanic tuff) for the construction of the ancient fortress. At the expansion of the borders of the Roman Empire, especially after the 1st century AD, the effervescence of constructions was supported by new and new ideas, both to improve the properties of existing materials, to diversify them, but also to find sources of raw materials or their substitutes in the newly conquered territories, far from Rome.

In this particular case, of mortar from Porolissum customs foundations, the preliminary physico-chemical analyzes (Particle Induced X-ray Emission, X-Ray Diffraction, Differential Scanning Calorimetry, Thermo Gravimetric Analysis, laboratory chemical analyzes) led to the idea that it is a hydraulic mortar and, most likely, instead of pozzolana and kaolin rather quartz-kaolinite was used. Limestone and other raw materials: sand, water, quartz-kaolinite, including wood as combustible for lime kilns came from local, neighboring areas or regional sources, depending on the distance from the place where the Roman building was erected. Most likely the sources of water, sand, wood were the local ones. Depending on the quantity needed to obtain the mortar, but also on its desired final quality, the Romans expanded their choice, orienting themselves towards more distant sources for some raw materials. Unlike kaolin / quartz-kaolinite, where small amounts were needed, the mortar was an industrial product. So, the limestone had to be brought from a maximum radius of 30-50 km away. This procedure was generally used for selecting the sources of raw materials, both as a building stone for covering or for creating ornaments, but also for obtaining binders, was limestone.

The whole process of selection and transformation that led to mortar, unearthed at the moment of sampling, involved: (1) selection of local, zonal and / or regional raw materials; (2) obtainment of quicklime from limestone, metakaolin from quartz-kaolinite, possibly sand by sorting; (3) manufacturing of raw mortar according to a "recipe"; (4) hardening of mortar over time by drying and adsorption / chemosorption of atmospheric carbon dioxide by the lime in excess; (5) aging over time of mortar components, and transformations that took place in equilibrium with the elements from soil, water, air. Basically, this last phase can be characterized directly by archaeometry while the others can be deducted directly or indirectly. Among these, the mortar technology and "manufacturing recipe" are the most important.

A first step in initiating archaeometry research for identifying the present-day composition of the mortar (Porolissum Lime / Hydraulic Mortar) is radiocarbon dating of a representative sample (Porolissum Lime Nodule). The effort is justified to establish their authenticity and corroborate the results with other physico-chemical analyzes that were applied to them, and also to compare with different other samples that were used to estimate the sources of raw materials.

Nodule represents, according to DEX Online – geology, a “Small mass of stone-like material in a less hardness zone" [Pl. and: (n.) nodule] – from fr. nodule, lat. *nodulus*. In the particular case of the lime nodule, it represents a small mass of carbonated lime, compact, homogeneous, contained in a less hardness part of the sample, represented in this case by mortar. DEX Online also offers a popular alternative, namely: piátră-de-vár (in Romanian) (calcite, the most stable polymorph of calcium carbonate) (pia-tră-) s. f., g.-d. art. piétrei-de-vár. Although the English language has the variant "nodule" or "concretion", a neologism borrowed from French is "lump", which in Romanian translation would be "Bulgăre". In the case of small and very small formations embedded in the mortar mass, the English term "lumps and bumps" is used. These formations that include only carbonated or re-carbonated lime after hardening under construction are mainly sought for radiocarbon dating [3, 4].

At the beginning, 3 sub-samples of 7 g each of Porolissum Lime Nodule sample, associated with Porolissum Lime / Hydraulic Mortar were pre-treated in parallel, according to a working procedure adapted for our laboratory, based on the recommendations given in the scientific literature [6 - 16]. Thus, after mechanical cleaning of the surface, breaking of the nodule and selection of some parts inside it, inspection, scraping and manual grinding followed. These fragments formed 3 sub-samples of 7 g each, which were further separately suspended in Berzelius glass beakers, in approx. 50 mL ultrapure cold water MilliQ8™.

An original aspect that was introduced was the maintenance of the temperature around 10 - 12°C not only during the ultrasonic separation, but also throughout the pre-treatment, due to the inverse variation of water solubility of the extracted calcium carbonate, with temperature [15].

In the first stage of the pre-treatment, there was a coarse separation of the heavy, insoluble fractions from the light and partially soluble fractions in cold water, by ultrasound for 15 minutes / round, the operation being repeated in 3 consecutive rounds. Next, the approx. 50 mL / round / sample, recovered successively after each of these consecutive rounds, containing the light weight and partially soluble fractions in cold water, are transferred one by one into a 50 mL centrifuge tube, and centrifuged for 10 minutes at 3000 rpm, the temperature being kept at 10 - 12°C. At the end, the resulting supernatant is retained each time, by separation from the solid fraction. After another 2 extractions in ultrapure MilliQ8™ cold water added to the Berzelius beakers over the initial sub-samples, and ultrasound (a total of 3 rounds of extraction for each of these 3 sub-samples), the supernatant fractions obtained were pooled into 3 storage glass containers. These 3 containers were kept in refrigerator until the temperature returned to approx. 10 - 12°C. The collected supernatant, after shaking the bottle before each centrifuge step until the solution in the containers was consumed, was immediately transferred by portioning into another clean centrifuge vials of the same capacity. Finally, by further centrifugation at 1000 rpm for 3 minutes, at 10 – 12°C, a very fine, sub-micronic fraction was obtained in the upper part of the centrifuge tube, together with the fractions partially soluble in cold water and corresponding, in the case of a lime nodule, to the calcium carbonate / calcite fraction - originating from the beginning of the lime hardening until the end of this process (the hardening process can take sometimes up to 200 years). They were separated from the total mass of liquid by pipetting the first 15 mL of supernatant from each tube at the end of each round. All portions of 3x3x15 mL belonging to these 3 sub-samples were pipetted on the same watch glass with approx. 160 mL liquid storing capacity. The watch glass was placed in a *vacuum* oven for 6 hours at 60 °C for complete drying. The white solid that remained behind contains the light weight sub-micron fraction and parts of the original sample partially soluble in cold water, extracted from the lime nodule sample.

After 12/24 h of *vacuum* drying and equilibration at ambient temperature, the white deposit was scraped with a slide and immediately placed in a special borosilicate glass vial. The initial mass of the final solid analyte was approximately 125 mg in this case (10 times the minimum amount required for the reaction, as a safety measure to achieve optimal graphitization parameters). The remaining solid product recovered from the watch glass, a few mg, was placed into another identical vial and used for purging the graphitization system and for isotopic balancing prior to collection of carbon dioxide resulted from the decomposition of the calcium carbonate in the reaction vial, before graphitization step.

The overall yield of the pre-treatment process, starting from 21 g of raw material selected from the lime nodule, was 0.619%. The pre-treatment strategy avoided increasing the overall yield by contamination with exogenous calcite fractions that may not have corresponded to the mortar hardening interval in the masonry. This aspect was urgently pursued because of the context in which the samples were taken (see "Introduction") and because, during the release of carbon dioxide from the final analyte, only one common gaseous fraction was collected. According to the results of the Thermo Gravimetric Analysis (the results will be published separately), if we report the amount of final analyte to the real one, as total mass of calcium carbonates in the lime nodule, than the percentage would be 0.656%. The estimation of the calcite content originating from the moment of hardening the mortar, so that we have a specific recovery yield, in order to appreciate how efficiently it was extracted from the lime nodule, respectively the efficiency of the method used in pre-treatment step, was estimated accordingly. The total calcium carbonate content established by Thermo Gravimetric Analysis was 94.36%, so the nodule was indeed a representative calcite accumulation, in terms of radiocarbon dating. These two values of global recovery yields give an idea on the percentage of original calcite recoverable from the sample taken after 2,000 years in safe conditions, but also on some subsequent phenomena of carbonation / re-carbonation by interaction over time with the environment, as was considered from the beginning.

The pre-treatment of the mortar samples does not always take place as described above in the target preparation unit of the RoAMS Laboratory at IFIN-HH or in other laboratories in the world. It adapts on a case-by-case basis and develops over time [16]. The variant used in this present case study was considered optimal since a well-preserved lime nodule, with a percentage > 94% in carbonates was used.

The digestion of calcite, separated from the initial sample into the sub-micron fraction and / or the partially water-soluble fractions (depending on the temperature regime used in the pre-treatment) which are considered representative of the mortar hardening interval, and the emergence of the carbon dioxide take place in Carbon Handling System™ unit (CHS, IonPLus, Switzerland). In this case, after purging with helium and isotopic balancing into the borosilicate glass vial, the chemical digestion of carbonates from the entire mass of the final analyte takes place. Polyphosphoric acid, for approx. 30 minutes, at a temperature of 80^0C is used for this purpose. The carbon dioxide formed is subsequently non-selectively carried out, using helium, dried on Sicapent™, and directed to the AGE 3™ unit (ETH Zürich, Switzerland). It is retained on zeolite, introduced into the reaction vial, and reduced with hydrogen on the iron catalyst at 580^0C and a pressure of approx. 1.4 atm, up to natural carbon [17 - 19]. An admixture is obtained which is essentially the “AMS target” material.

The elemental carbon deposited intimately on iron was finally introduced, through a guiding system, into the carousel of the accelerator coupled with a mass spectrometer i.e., 1 MV Cockroft-Walton Tandetron HVEE™ Netherlands. The measurement process is optimized and the ^{14}C / ^{12}C, ^{13}C / ^{12}C, ^{14}C / ^{13}C isotopic ratios are determined [20]. The next step is the calculation of the absolute age using the Bats 4.0 program, which delivers the CRA value = Conventional Radiocarbon Age value, measured in BP years, amended with a measurement accuracy expressed as standard deviation, and corrected for total isotopic fractionation using the AMS measured $\delta^{13}C$ value [21]. CRA calculations were performed considering the data obtained, in the same batch of graphitization / AMS measurement, for the reference materials used as Graphitization Process Blank (carbon dioxide reduction at CHS™ / AGE 3™), respectively IAEA-C1, and as modern radiocarbon level HOxII standard (NIST designation SRM 4990 C), using EA™ / AGE 3™ system (EA, Elementar™, VarioMicroCube, Germany) [22, 23].

The transition from CRA to calibrated age (in historical years) was done using OxCal v.4.3 and the IntCal 13 calibration curve, provided online by the Radiocarbon Unit of the Oxford UK Laboratory. Reimer et al., 2013
https://c14.arch.ox.ac.uk/oxcal.html [24].

3.Results and discussion

Age determination of calcite in mortar, more precisely in the lime nodule, using absolute chronology, presents a certain degree of complexity, sometimes the results being inconclusive. There are a number of factors that determine this, the method having advantages, disadvantages and limitations, as stated before [16]. Using of a lime nodule, well preserved and delimited by mortar itself, in a direct connection with it, from an area as "safe" as possible in terms of the date of construction and subsequent interventions (from the foundations), determines the success of the experiment, obtaining an interval in calibrated radiocarbon years, consistent with the historical and archaeological context.

In this particular case, the percentage of 94.36% calcium carbonates, having different origins and co-existing in the composition of the lime nodule at the date of analysis, certifies that it is a convenient and suitable material for radiocarbon dating. The correspondence in calcium oxides is only 52.80%, compared to the value determined by the Particle Induced X-ray Emission method (the results will be published separately), of 74.37%. The smaller ratio 74.37: 52.80 = 1.408x100:94.36 = 1.492 towards to 100:56 = 1.786 (theoretical value of pure calcite) shows the purity of the nodule and the existence of chemical forms in which calcium is involved, other than free calcite. This information is useful in assessing the chemical components separated from mortar in the laboratory (not presented here) and in establishing the pre-treatment strategy for radiocarbon dating.

The obtained radiocarbon date was: CRA = 1775 ± 37 years BP. The calibrated age is shown by intervals of probabilities in the histogram below (Fig. 3).

For the sub-interval $\sigma = 2$ of the probability distribution, in 95.4% of the cases, the true value is included in this sub-interval delimited on the histogram (Fig. 3). It can be stated with certainty that the mortar and the lime nodule samples come from the material used to raise the eastern perimetral wall and prove that they do not belong to later periods (restorations, interventions, etc.). Thus, the results of chemical and compositional analyzes for these materials, but also for samples from possible sources of raw materials, deserve to

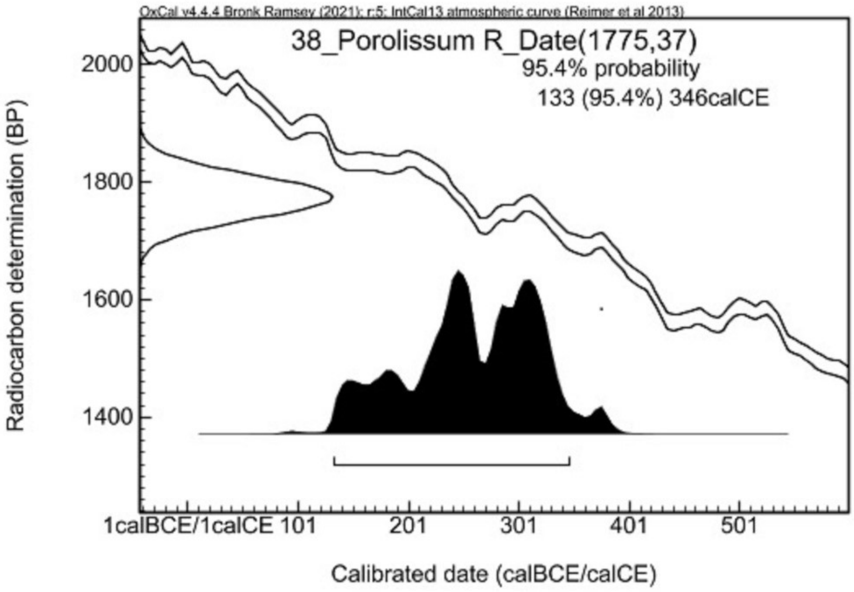

Fig. 3 - Interval in calibrated radiocarbon years for Porolissum Lime Nodule sample (according to the histogram generated Online; https://c14.arch.ox.ac.uk/oxcal.html; 2017) / *Intervalul în ani radiocarbon calibraţi pentru proba de nodul de var de la Porolissum (conform graficului generat Online; https://c14.arch.ox.ac.uk/oxcal.html; 2017)*

be considered in archaeometry studies up to come. Moreover, the inter comparison of chemical and compositional results, and the conclusion on raw materials analyzes can lead to the mutual validation of the "retroversion" towards to the "manufacturing recipe".

On the other hand, the result agrees with the historical and archaeological interpretations presented in detail in "Introduction". It certifies the construction of the perimetral wall, at least on the eastern part of the building, at the same time with the two bastions on the south side or in a later period, close in time. According to this assumption, dating of the stone wall in the second phase of customs construction was assumed, until this study, only on the basis of the architecture of the construction and the association with some archaeological materials, so it could not be clearly specified (stratigraphic information could not determine the time erection of the stone construction, establishing only the anteriority of the wooden fortification and clay furrows to the stone wall). Only the bastions of the access gate can be dated, with safe analogies after the second half of the second century AD, either towards the end of Hadrian's reign (117 – 138 AD) or towards the end of Antonius Pius's reign (138 - 161 AD) [3].

Although the histogram shows several peaks of different intensities / probabilities in the range calCE 133 - 346, and the following mediated values of the 6 identified peaks could be taken into account: average (calCE 276); median (calCE 245); weighted average (calCE 259); the center of gravity of the figure (calCE 260); and the interquartile range (calCE 170), according to the literature recommendations on calibrated radiocarbon data, the safest remains the interquartile range, calCE 170 in this particular case, which will substantiate the second peak, starting from the left part of histogram [25]. Based on these interpretations, in terms of absolute chronology, the eastern wall could be rather associated with the rebuilding of enclosure camp using stone walls started with the reign of the emperors Hadrian - Antoninus Pius, and ended before 213 AD [1].

CE = Common Era is equivalent with AD = Anno Domini, frequently used by the archaeologists.

The dating of the second stone phase of the building, from which the Porolissum samples were taken, can be linked to two inscriptions, which bring specific information about the restoration / transformation of the building, probably starting with the period of the Emperor Commodus (177 - 192 AD). This may be a second hypothesis of dating which restricts the previous historical interval or at least attributes some segments of restorations up to 213 AD, to this Roman emperor.

From the point of view of contact chronology, which integrates relative chronology and absolute chronology, the value of calCE 170 is a good result, at least as a *terminus post quem* date (the date from which the mortar began to harden into the foundations, in this case).

However, if we generate the histogram using the latest radiocarbon calibration curve, IntCal 20, the range calCE 211 – 401 will be obtained. There is a shifting in the whole range of 95.4% probability, to younger ages. A third scenario of the construction moment appears, namely during the Severus Alexander reign, around the historical date of 240 AD. This date is substantiated by a sharp peak which begins to separate clearly on the histogram, especially if the AMS measurements would have been made at better accuracy intervals, respectively of ± 25 years BP or ± 20 years BP. This third date overlaps very well above the value of the median obtained using IntCal 13.

Indeed, on the territory of the Roman edifice of Porolissum were discovered monetary issues from Vespasian (69 - 79 AD) to Severus Alexander (222 - 235 AD) [3]. However, monetary issues prior to the Roman conquest of Vespasian cannot be taken as dating criteria. Instead, those of Severus Alexander indicate the use of construction in the second stage, offering a new perspective on the circumstances of the construction / transformation of this part of customs.

4.Conclusion

This study managed to date for the first time by absolute chronology the stone phase of the Roman customs from Porolissum Romania. This building has a special importance, being the first of its kind recorded in the Roman Empire. The dating of the hydraulic mortar from the customs foundations from Porolissum was made by analyzing the archaeological context combined with the data obtained from radiocarbon measurements, highlighting a good concordance between relative chronology and absolute chronology. The studied samples and their associated archaeometric data will be a benchmark for the selection and comparison of similar results obtained on various materials, proposed as being sources of raw materials.

In order to verify the radiocarbon result and to ensure the time sequence of the stone stage building phase of construction, a systematic analysis of as many samples as possible from several areas of the construction could refine these preliminary results i.e., if there were several stages that followed each other at short intervals, related to a succession of Roman emperors and their decisions, or there was a single restoration from wooden fortification and clay furrows palisade, to stone walls.

After 2020, upon completion of restoration works of the Roman edifice, when similar modern materials were used, such an investigation could be limited / hindered by the exchange that has already occurred between the modern carbon-14 introduced by the new materials and the old original one. This is why this current radiocarbon dating and upcoming analyzes certify the correct sampling of the materials used in construction of the Roman edifice from Porolissum, before the latest interventions.

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