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DEVELOPMENT OF ^{14}C DATING OF MORTARS AT ETH ZURICH

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ABSTRACT. The ages of mortars and plaster can help reveal the history of monuments, their construction, or restoration times. However, these anthropogenic carbonates pose a challenge when it comes to separation of the atmospheric radiocarbon (^{14}C) signal of the CO_2 fixed in the mortar at the time of consolidation, i.e., the time of binder formation. The variety and heterogeneity of mortars require individual assessments of each sample and ^{14}C results. Here we present our current preparation method and summarize experience based on results collected during the last 20 years of mortar dating at the ETH laboratory.

KEYWORDS: archaeology, intercomparison, mortar, radiocarbon AMS dating.

INTRODUCTION

The radiocarbon (^{14}C) dating method, which was developed 70 years ago (Arnold and Libby 1949) from its early days, revolutionized two main research fields: archeology and past climate studies (Olsson 2009). During the last seven decades the method underwent numerous developments both in preparation and measurement techniques, which have substantially improved the precision and accuracy of mortar radiocarbon chronologies. As a consequence, the spectrum of applications expanded to different disciplines, including objects of cultural heritage. A significant portion of tangible cultural heritage is ruins and buildings of sacral and monumental constructions. The very prominent ones, such as castles and cathedrals, have historic evidence revealing their dates, but often their construction phases or modifications, or even their minor buildings still have uncertain chronologies.

Dating the time of the construction or reconstruction is of interest to archaeologists, art and development historians, architects, and conservators. Occasionally, organic remains can be found in walls, floors, or foundations, or the wood of architectural elements can be dated. However, it is the mortar that has the potential to be the most suitable material for dating the construction. In the process of mortar production, the burnt carbonate (limestone, dolomite, marble) (1) is mixed with water and aggregates (2) and can bind the atmospheric CO_2 (3).

The binding process (3) seems to be comparable to the photosynthetic fixation of CO_2 in plants; therefore, an ideal process for incorporating ^{14}C in mortars.

(1) $CaCO_3 \rightarrow$ thermal decomposition $\rightarrow CaO+CO_2$
(2) $CaO+H_2O \rightarrow Ca(OH)_2$
(3) $Ca(OH)_2+CO_2 \rightarrow CaCO_3+ H_2O$

Also, concerning the ^{14}C analyses, the release of carbon from the mortar samples is not sophisticated because carbonates can be easily dissolved in acid or thermally decomposed. Thus CO_2 can be collected for ^{14}C analysis. For this reason, it is not surprising that mortars were among the first materials of interest for radiocarbon dating. The early studies (Labeyrie and Delibrias 1964) used a thermal decomposition at 900°C and obtained an excellent agreement for all four studied samples. But later, other researchers realized that the success of the radiocarbon dating depends on the type and characteristics of a mortar

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(Baxter and Walton 1970; Folk and Valastro 1976). Relevant to ^{14}C dating is the origin and extent of burning the source rock, such as limestone, dolomite, or marble. Moreover, too-old ages of the mortar could result from the presence of old carbonates, for example, added to the lime as aggregates as the sand often contains shells and limestone. The apparent too-old ^{14}C age of mortars was the main problem at the time when a large quantity of carbon was needed for beta particle counting (conventional method).

Following these first attempts of dating, in the 1960s and 1970s, mortar received little attention in ^{14}C dating. One must say, it has not been due to the lack of material or need for dating mortars. More probably, the few early attempts of dating mortars were evaluated as not successful, because expected ages were in disagreement with archaeological information (Stuiver et al. 1965). In effect, decades of a skeptical approach to radiocarbon dating of mortars followed. However, the very first attempts were also helpful and provided valuable information for the later development of the method.

The different solubility of amorphous carbonates, which are formed when the slaked lime binds carbon dioxide, has been recognized by Baxter and Walton (1970), who have shown the effect of different dissolution time on the age of dissolved mortar. Even though they used grams of mortar, significant differences between "fast" and "slow" fractions of nearly 1000 ^{14}C years were observed in the studied samples. The resulting ages were still older than the expected age, but the age of the first (a fast-dissolving) fraction was significantly closer to the predicted age than the age of the whole (bulk) samples of the mortar. The promising trend has been then explored by Ambers (1987) and developed further by Van Strydonck et al. (1986, 1989, 1992, 2011), who modified the method of the sequential dissolution by using titration: a dosage of acid for release of CO_2 from portions of mortar.

An application of the AMS technique to the measurements of the ^{14}C concentration of mortars (Heinemeier et al. 1997) in the 1990s was a turning point in the development of mortar dating. The AMS technique allows counting ^{14}C atoms ($^{14}C/^{12}C$ ratio is measured at 10^{-12} to 10^{-15}); therefore, a smaller amount of material is needed, i.e., tens of mg of mortar or few mg of a pure lime lump. The downscaling from the previously required 300–400 g of sieved mortar (Folk and Valastro 1976) clearly expands the potential for more efficient separation of mortar CO_2. It is now possible to measure minute amounts of carbon (1 mg to tens of μg of C), allowing for testing various fractions of mortar, which is crucial for mortars because they are a very heterogeneous material on a small scale. The Scandinavian team (Heinemeier et al. 1997; Sonninen and Jungner 2001; Lindroos et al. 2007; Heinemeier et al. 2010; Ringbom et al. 2011, 2014) developed the sequential dissolution method, and it has been applied to date numerous monuments. Their criteria for sampling and preparation provide a basis for the development and modifications of the protocol by other laboratories (Hayen et al. 2017).

During the last years, other methods have been developed, focusing on physical (mechanical) "purification" of the binder. The first use of cryo-breaking of mortars was proposed by Nawrocka et al. (2005) and further developed by Marzaioli et al. (2011). An ultra-sonication to obtain the pure binder from suspension (the CryoSonic protocol) was proposed by Marzaioli et al. (2013) and Nonni et al. (2013). Michalska et al. (2017) describe the latest modification of the CryoSonic protocol, involving sequential dissolution, i.e., separation of fast and slow fractions of carbonate as proposed by Heinemeier et al. (1997) and Lindroos et al. (2007).

The first intercomparison performed on mortar samples (Hajdas et al. 2017; Hayen et al. 2017) has shown the need for petrographic and geochemical analysis of the ^{14}C samples. Here we describe the development of the preparation method for ^{14}C dating. For the characterization of the mortars, we rely on the expert evolution of our collaborators. They characterized the samples using polarized light microscopy (PLM), X-ray powder diffraction (XRPD), scanning electron microscope (SEM), and an energy-dispersive X-ray spectrometer (EDS) (Michalska et al. 2017; Caroselli et al. 2020 in this issue).

Samples of Mortar from Various Monuments

Various types of mortar have been submitted to the ETH laboratory for radiocarbon dating of organic material (charcoal, wood) enclosed in the binder. The very first sample from a medieval church in Germany[1] was submitted to the laboratory in 1997. It contained charcoal inclusions, which allowed for the independent control of the age of the mortar. Results of dating the first mortar sample have not yet been published due to the lack of background information on the mortar. In the following years, another project pushed the development of mortar dating at the ETH laboratory. It included mortars from Roman and medieval sites (blinded submission) and triggered the whole set of trials, mostly experimenting with dissolution time. Because the Roman mortars were highly contaminated with the old carbonates, the method was pushed to the physical limit of 3-sec intervals. These mortars from Koenigsfelden (medieval) and Vindonissa (Roman) were essential for the continuous development of the method (Hajdas et al. 2012). In the years that followed, exchange with other laboratories and close collaboration was established in the frame of the mortar dating project lead by Åsa Ringbom (www.mortardating.com). The initiative to perform the first MOrtar Dating Inter-compariSon (MODIS) resulted in an intensified exchange of knowledge and practice shared by seven laboratories (Hajdas et al. 2017; Hayen et al. 2017). The material that was chosen for MODIS included some challenging examples (Basel and Roman mortars) (Michalska et al. 2017). Among other samples submitted to the ETH, laboratory were some mortars of the Roman age (Slovenia and Vindonissa) and medieval mortars from different locations. The mortar from Nitra, Slovenia, was a part of the collaborative study (Povinec et al. in prep.) as was the dating of the remains of an early church known as Hohenrätien, Switzerland (Hajdas et al. forthcoming) and the foundation of the Fraumünster in Zurich, which is not yet completed as this mortar sample is particularly complicated. The mortars of the Müstair monastery, Switzerland, are subject to studies in the frame of a multidisciplinary project. Dolomitic mortars from the extensive archives chosen from this unique UNESCO site were a subject of characterization and radiocarbon dating (Caroselli et al. 2020 in this issue).

Here we discuss the ETH results obtained as a part of the MODIS intercomparison exercise (Hajdas et al. 2017; Hayen et al. 2017). Mortar samples from four sites were chosen and samples were distributed to seven laboratories. The samples included a wall's bedding mortar from the church of Nagu in the Åboland archipelago (Finland), a lime conglomerate from a burial site at Cova S'Estora (Son Pellisser) on the island of Mallorca (Spain), the remains of a medieval mortar mixer from Basel Cathedral Hill (Switzerland) and a bedding mortar/infill from the lower part of a Roman wall excavated in the city of Tongeren (Belgium) (Hayen et al. 2017). The results are published together with those of other laboratories. Here the specific aspects of our results are discussed.

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[1] Tracking down the original location and information about the church failed.

Sequential Dissolution Method “SDM-3sec”

The method development at the ETH laboratory during the last 20 years followed the work of the Scandinavian team: Åsa Ringbom, Jan Heinemeier, and Alf Lindroos (Heinemeier et al. 1997; Lindroos et al. 2007, 2011; Ringbom et al. 2014). The very first attempts made in 1998 to date sample ETH-18245 were based on the observation made by early studies and successfully explored by the Scandinavian team that the anthropogenic carbonate (binder) is more soluble than the geogenic carbonates (Baxter and Walton 1970; Hormes et al. 2015).

Therefore, the method of sequential dissolution targets the fast-dissolving component of the binder. In the early years of using this method at ETH Zurich, wet sieving was used, and fraction chosen for dating was < 32 μm (Hajdas et al. 2012). Currently, the dry sieved mortar fraction of grain size 43–63 μm is used, a change introduced following an exchange and discussions related to the MODIS intercomparison project. The lower limit on the grain size is to remove the potential contamination with fine-grain limestone, which will also dissolve faster and therefore pass the next separation step. Moreover, prior to applying the sequential procedure, lime lumps are sampled and measured. After removal of charcoal (or other organic fragments) and lime lumps, the sample is crushed in a ceramic mortar and sieved to collect fraction 45–63 μm. In the next step, the whole bulk of the chosen fraction is dissolved in acid and graphitized for the AMS analysis. This step is used to estimate the carbon content of each sample. Moreover, it allows measuring the range of the ages of the mortar. For example, mortars of Roman age can be easily detected in this step because the bulk ages can be as old as 8–10 ka. Measurement of bulk is considered exploratory, although, dependent on the type of mortar, this fraction has the potential of providing the accurate ages.

For sequential dissolution, subsamples containing ca. 30–50 mg of powder (dependent on carbonate content as estimated from bulk analysis) is placed in one of the chambers of the special duel chamber glass vessel. The second chamber is filled with 10 mL of concentrated phosphoric acid (85% H_3PO_4). The vessel is then closed and evacuated at room temperature before pouring of acid to the chamber containing mortar. This process is timed, and freezing of purified (passing through a water trap) CO_2 in liquid nitrogen (LN) is performed in sequence: 4 consecutive fractions are collected after 3 sec each. No measurement was performed on the rest of CO_2 that formed after 12 sec. The carbon content of each collected fraction is measured, and 10–100 μg of C is trapped in a 4-mm tube to be flame sealed for analysis using Gas Ion Source (GIS) AMS facility at ETH Zurich. Samples containing more than 150 μg of carbon can be graphitized and measured using the MICADAS (Synal et al. 2007). Table 1 provides information on the recovered amount of carbon. Solid and gas formed samples are analyzed together with the corresponding size of standard (OxA-II) and background samples (C-1, IAEA), and secondary standard (C-2, IAEA).

Assigning the ^{14}C Ages of Mortar Using SDM-3sec

Following Heinemeier and Lindroos (Heinemeier et al. 1997; Lindroos et al. 2007) who developed the age plateau determination of radiocarbon age, the SDM-3sec method described above targets the most soluble fraction of mortar. The first “3sec” interval (named “1–3sec”) is the fastest that can be achieved presently with our system. The age of mortar is assigned based on the age of this fast fraction. The ages of slower fractions (4–6sec, 7–9sec, 10–12sec) are considered as supportive, i.e., if the 2nd or even 3rd and 4th fractions form an age plateau (Lindroos et al. 2007; Heinemeier et al. 2010) the age of the first fraction is considered to be

Table 1 Overview of samples analyzed by the ETH laboratory as a part of the MODIS inter-comparison (Hajdas et al. 2017). The starting mass of the samples is on the order of 30–50 mg.

Lab code	Sample code		Grain size	Dissolution fraction*	mg C	^{14}C age BP	±1σ
ETH-62275	1	Finnish mortar	46–75 µm	1	0.119	495	50
				2	1	395	50
				3	1	569	59
				4	1	535	57
ETH-70605	2f<500µm	Mallorca burial	45–63 µm	1	0.016	2345	144
				2	0.09	2354	78
				3	0.094	2827	79
				4	0.09	3241	82
ETH-62278	3	Swiss mortar mixer	Original	Charcoal	0.989	1314	23
ETH-62278	3	Swiss mortar mixer	45–63 µm	1	0.216	1746	50
				2	0.418	1966	51
				3	0.455	2864	66
				4	0.184	3773	67
ETH-62278	3	Swiss mortar mixer	<45 µm	1	0.13	1767	50
				2	0.39	2065	51
				3	0.403	2774	63
				4	0.188	3655	66
ETH-62279	4	Roman cocciopesto mortar	32–63 µm	1	0.165	844	49
				2	0.26	879	48
				3	0.332	1025	59
				4	0.123	877	59
ETH-62279	4	Roman cocciopesto mortar	<32 µm	1	0.055	946	57
				2	0.227	896	49
				3	0.255	998	59
				4	0.109	1030	58

*Fraction 1: 0–3 sec; 2: 4–6 sec; 3: 7–9 sec; 4: 10–12 sec

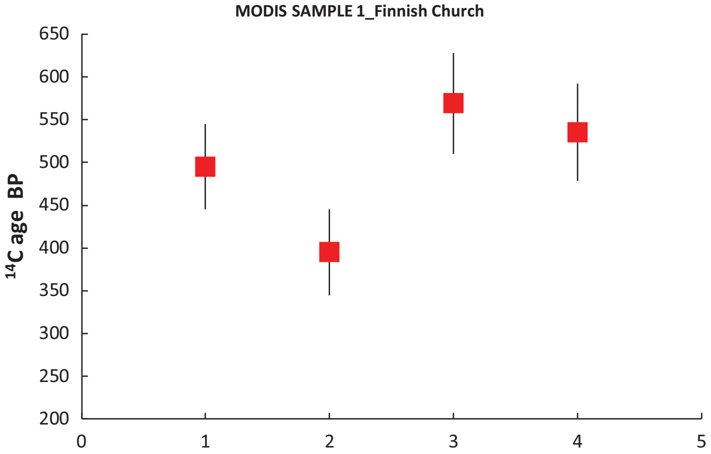

Figure 1 Results obtained by the ETH laboratory for Sample 1 in the MODIS intercomparison (Hajdas et al. 2017). The numbers indicate fractions: 1 = 1–3 sec, 2 = 4–6 sec, 3 = 7–9 sec, and 4 = 10–12 sec dissolution time window. The fine fraction of 46–75 µm was used. The combined value of all 4 fractions is 491 ± 21 BP (χ^2-test: df = 3, T = 6.0 [5% 7.8]) was calculated using the OxCal Combine model function (Ramsey 2009).

free of contamination with old carbon. This is also illustrated in Figures 1–4, showing the results of dating the MODIS samples obtained by the ETH laboratory (Hajdas et al. 2017). In the case, the ages are in 2-σ range agreement a combined radiocarbon age is calculated using the Combine function of OxCal (Ramsey 2009).

MODIS—Discussion of ETH Results

Four samples distributed to seven laboratories participating in the MODIS intercomparison were of different origin and structure of mortar (Hayen et al. 2017). Results are summarized in the supplementary material of Hajdas et al. (2017). Table 1 shows only the information related to the ETH results.

The origin and type of mortar predestined the results of radiocarbon dating as it has been shown that type of mortar sample that reflects the way it was crafted, and its mineral composition is a decisive factor for the success of radiocarbon dating. The results of MODIS intercomparison were published in two complementary papers (Hajdas et al. 2017; Hayen et al. 2017). Our discussion here focuses only on results from the ETH laboratory but with the input of the knowledge gained by the intercomparison collaborative effort of all the labs involved (Hajdas et al. 2017; and Hayen et al. 2017; Michalska et al. 2017).

The first MODIS sample from a medieval Finnish church (Nagu) was chosen for the intercomparison exercise because it had a well-established, independent chronology. The results provided by all the laboratories (including ETH) were in good agreement. They could be combined into one radiocarbon age of 505 ± 8 BP (χ^2-test: df = 16, T = 20.6 [5% 26.3]), which is in accord with the radiocarbon age of wood 515 ± 15 BP (Hajdas et al. 2017). Figure 1 shows all four fractions obtained for fraction 46–75 µm, which resulted in coherent

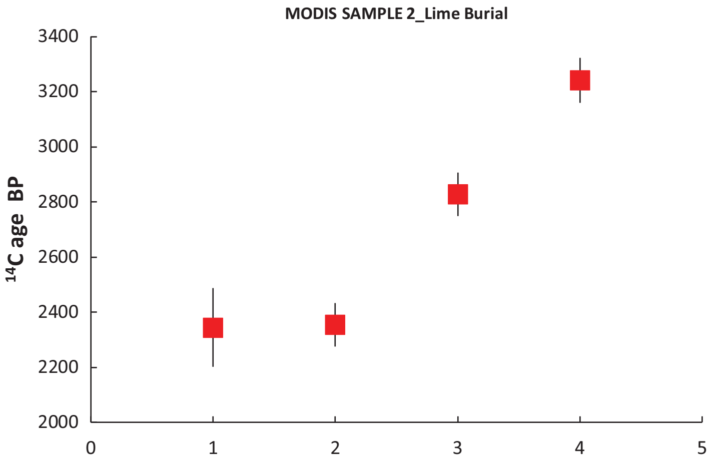

Figure 2 Results obtained by the ETH laboratory for Sample 2 in the MODIS intercomparison (Hajdas et al. 2017). The numbers indicate fractions: 1 = 1–3 sec, 2 = 4–6 sec, 3 = 7–9 sec, and 4 = 10–12 sec dissolution time window. The fine fraction distributed in MODIS exercise was grain size <500 µm but fraction 45–63 µm was used for dating. The first fraction had very low carbon content resulting in high uncertainty. The combined value for the first and second fractions is 2352 ± 69 BP.

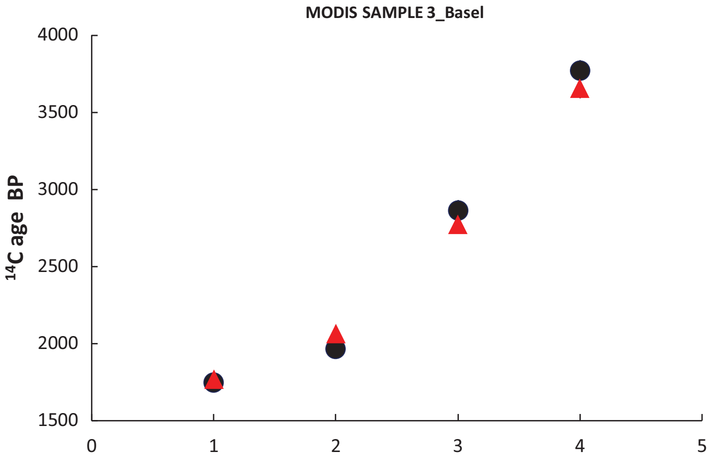

Figure 3 Results obtained by the ETH laboratory for Sample 3 in the MODIS intercomparison (Hajdas et al. 2017). The numbers indicate fractions: 1 = 1–3 sec, 2 = 4–6 sec, 3 = 7–9 sec, and 4 = 10–12 sec dissolution time window. Two preparations were performed: one on a fraction 45–63 µm (circles) and one on fraction <45 µm (triangles). In addition, the charcoal sample found in the mortar was dated at 1313 ± 22 BP. Note the significant change of radiocarbon ages with the increasing fraction number (dissolution time).

radiocarbon ages. The combined value is 491 ± 21 BP (χ^2-test: df = 3, T = 6.0 [5% 7.8]), which is in excellent agreement with results obtained by other laboratories and shows the potential of radiocarbon chronologies based on mortars binder for archaeology, monuments construction, and conservation history.

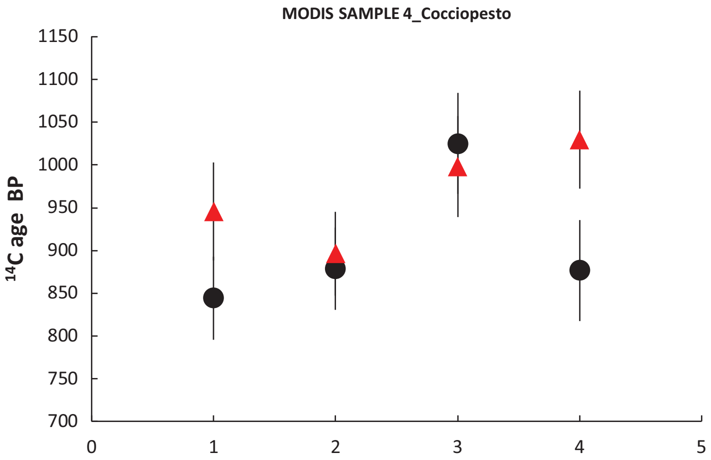

Figure 4 Results obtained by the ETH laboratory for Sample 4 in the MODIS intercomparison (Hajdas et al. 2017). The numbers indicate fractions: 1 = 1–3 sec, 2 = 4–6 sec, 3 = 7–9 sec, and 4 = 10–12 sec dissolution time window. Two preparations were performed on fraction 32–63 µm (circle) and on a fraction smaller than 32 µm (triangles). Note that the three first fractions of both preparations agree in the 2-σ range.

The second sample distributed in MODIS exercise was an archaeological sample from lime burials found at Mallorca (Van Strydonck et al. 2011). Material pre-sieved to fraction smaller than <500 µm was analyzed. The first results obtained on fraction 45–63 µm show that the first (1–3 sec) fraction could be reproduced in the second (4–6 sec) fraction, and the age is combined to 2352 ± 69 BP. When compared with the age of charcoal found in the lime 2336 ± 30 BP and the age of the human bone 2442 ± 30 BP (Van Strydonck et al. 2017) this result is coherent with the chronology of the site. Both charcoal and human bone could still predate the burial (old carbon and marine diet) therefore, dating the lime can provide a cross-check.

The medieval sample from a mortar mixer excavated in Basel was expected to be dated to medieval age, which is indicated by the age of charcoal found in the mortar sample and dated by ETH lab and by Poznan lab 1313 ± 22 BP and 1345 ± 30 BP, respectively (Hajdas et al. 2017; Michalska et al. 2017). However, neither of the laboratories succeeded in separating such a medieval age fraction of this mortar. The repeated preparation at the ETH laboratory reproduced the first results (Figure 3). A substantial change towards old ^{14}C ages in the 3rd and 4th dissolution fractions suggests the presence of an old carbon source. However, the reason for such discrepancy between the results of MODIS (age close to Roman time) and the expected archaeological dating (medieval) is not yet understood. The disparity between mortar and charcoal is not the only difference. The present radiocarbon ages on charcoals and a bone associated with this sample are not as coherent as the results on charcoal found in the MODIS sample (Hajdas et al. 2017; and Hayen et al. 2017). A recycling of the Roman mortar has been suggested as the medieval construction is located near previous Roman structures (Hayen et al. 2017).

The fourth of the samples selected for MODIS intercomparison originated from the 4th-century Roman wall of *Aduatuca Tungrorum*, today Tongeren, Belgium. Already at the time of the first characterization of this sample, the so-called cocciopesto mortar was considered a problematic sample as described previously by Ringbom et al. (2011) and Michalska and Czernik (2015). The results of radiocarbon dating have confirmed this point. As shown in Figure 4, the radiocarbon age of all the fractions is much younger than expected 1700–1800 BP. Such a result was consistently obtained by all the radiocarbon laboratories and discussed in detail by Michalska et al. (2017). At the ETH laboratory analysis was performed twice. The repeated analysis of this sample shows an agreement (at 2-σ level) obtained for the first 3 fractions. The 4th fraction shows a difference, which can be explained by the heterogeneity of the sample.

The above summary of the ETH results obtained in the MODIS intercomparison shows that the results obtained using the SDM-3sec method were highly compatible with the expected ages of the two datable samples (Samples 1 and 2). Results obtained for the two other samples were consistent with observations made by other laboratories (Hajdas et al. 2017; and Hayen et al. 2017).

CONCLUSION AND OUTLOOK

The success of a radiocarbon dating mortar is highly dependent on the type of mortar. Some mortars, mostly Roman mortars, have a large portion of an old component that is difficult to separate with our SDM-3sec method. Nevertheless, most of the medieval monuments show a larger degree of homogeneity and lower content of added limestone aggregates, unlike many Roman monuments in Alpine regions. The results of MODIS are highly encouraging and highlight the importance of investigating the geochemical composition of mortars.

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