



2nd International
MORTAR DATING
Workshop

17-21 September 2012 Mallorca-Spain

drawing of a lead pectorial from a Mallorcan quicklime burial



program & abstracts



Program

Monday 17 September

- 18:30 bus transfer Hotel Felip - Museum of Manacor's History
Opening reception
21:00 bus transfer Museum of Manacor's History - Hotel Felip

Tuesday 18 September

- 9:00 bus transfer Hotel Felip - Museum of Manacor's History

SESSION 1: Chairperson Magdalena Salas

- 9:30-10:30 Damia Ramis
Quicklime Burials in the Balearic archaeological context
10:30-11:15 Mark Van Strydonck
¹⁴C dating of quicklime burials

11:15-11:45 coffee break

11:45-12:30 Guy De Mulder
Cova de Na Dent: a quicklime burial sampled in 2011
12:30-13:15 Roald Hayen
Towards a new approach to separate natural from anthropogenic
lime carbonate

13:15-14:30 lunch

SESSION 2: Chairperson Mark Van Strydonck

- 14:30-15:15 Vincent Debonne
Radiocarbon dating of lime mortars in late medieval buildings
in Belgium. On the accuracy of dating and local pedology
15:15-16:00 Roald Hayen
Radiocarbon dating of lime mortars in late medieval buildings in
Belgium. On the accuracy of dating and mortar characterization

16:00-16:30 coffee break

- 16:30-17:15 Robert L. Hohlfelder
When Dating Ancient Hydraulic Mortar Really Matters and the
Gods Smile: Three Case Studies from the Ancient Harbors of Cosa
(Italy), Alexandria (Egypt) and Soli/Pompeiiopolis (Turkey)
17:15-18:00 Greg Hodgins
Inadvertent Mortars and Plasters: radiocarbon measurements on
carbonates from hearths and pottery

18:15 bus transfer Museum of Manacor's History - Hotel Felip

Wednesday 19 September

9:15 bus transfer Hotel Felip - Museum of Manacor's History

SESSION 3: Chairperson Greg Hodgins

9:45-10:30 Åsa Ringbom
¹⁴C dating of lime lumps in Roman Pozzolana – an improvement of the mortar dating method, or a complementary analysis of an independent material?

10:30-11:15 Alf Lindroos
Lime lumps as a material for mortar dating

11:15-11:45 *coffee break*

11:45-12:30 Fabio Marzaioli
Accuracy evaluation of mortar radiocarbon dating by means of Cryobreaking, Sonication and Centrifugation (CryoSonic) procedure: first two years of experience at CIRCE Centre

12:30-13:15 Irka Hajdas
Results of dating Roman mortar in Vindonissa, Switzerland.

13:15-14:30 *lunch*

SESSION 4: general discussion. Moderator Åsa Ringbom

14:30-15:30 GENERAL DISCUSSION:
Where do we stand?
Plans for the future?

15:30-15:50 *coffee break*

SESSION 5: visits and excursions

15:50-17:00 Magdalena Salas

-Introduction to the excursions
-visit of the museum

17:15 bus transfer Museum of Manacor's History - Hotel Felip

Thursday 20 September

8:45 bus at Hotel Felip

9:30-11:30 Visit of the Museum at Montuïri and the Talayotic village of Son Fornés

11:30-12:45 bus to the Monastery of Lluç

12:45-13:45 Visit of the museum (objects from the quicklime burial of Cometa des Morts) and the monastery

13:45- 14:45 *lunch*

14:45-15:00 Bus to MANUT (Viver Forestal) : visit of a 'forn de calç'

15:00-15:45 bus to Can Picafort

15:45-16:10 coastal walk to Son Real burial site. Good swimmers can have a swim to S'Illa des Porros (burial site)

17:00- 17:25 walk back to the bus

17:25- 18:25 bus back to the hotel

Friday 21 September

9:30 bus at Hotel Felip

10:00-11:30 Pretalayotic & Talayotic site of S'Hospitalet Vell

11.30 - 13.00: bus and walk to Cova de Na Dent. Visit the lime burial of Cova de Na Dent.

13.00- 15.00 *lunch (box)*

If the sea is calm we will take a swim and visit Cave of Colom.

15.00- 17.00 Visit Cova des Pirata.

17:00-17:30 bus back to the hotel

¹⁴C dating of lime lumps in Roman Pozzolana – an improvement of the mortar dating method, or a complementary analysis of an independent material?

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In our experience, ¹⁴C dating of non-hydraulic lime mortars, not damaged by fire, has been relatively straightforward and yielded results in good agreement with analysis of other materials and using other methods. This is especially true of mortars from the Åland Islands, where we have systematically analyzed mortars from nearly all of the medieval churches. Here we have reached a very high percentage of results agreeing with available age control, or results that are conclusive by our definition of different criteria. From other areas in medieval Scandinavia we have similar experience, even if mortar dating has not been applied as systematically. The relevant age has been indicated already by the first CO₂ fraction in a ¹⁴C age profile of five CO₂ fractions from the sequential acid dissolution of the mortar sample.

It has been more complicated when it comes to fire damaged mortars and to hydraulic pozzolana mortars. Here, the interpretation of the age profiles has been difficult. The same structure, i.e. Trajan's Market, can provide three different interpretations of the profiles. For further testing, we have dated lime lumps embedded in the pozzolana in two or three CO₂ fractions. As yet our material is not very extensive, but we have had positive results. The analysis of lime lumps has provided either the assumed true age known from written sources, or it has been in agreement with our earlier estimations of constructions of unknown date. We believe that the lime lumps may provide an important complementary material for ¹⁴C analysis, and a way to determine the age of undated buildings. If successful, it is also a way to cut down on cost since fewer CO₂ fractions need to be radiocarbon dated.

Radiocarbon dating of lime mortars in late medieval buildings in Belgium. On the accuracy of dating and mortar characterisation

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Radiocarbon dating results on lime mortars taken from the parish church of Belsele (Sint-Niklaas) and the former collegiate church of Tongeren yielded in certain cases plausible datings as compared to written sources, stylistic features or dendrochronology results of the roof structures. However, other mortar samples produced aberrant datings, probably related to high percentages of fossil carbonates. A detailed mortar analysis of the different mortar samples is realized to compare the radiocarbon dating results with the presence of fossil carbonates or other stylistic features of the lime mortar samples. The mortar characterization is first of all based on a visual microscopic analysis of thin sections (ZEISS, Axioplan). For each mortar sample, more than one thin section was prepared to obtain a larger inspection area to study the presence of contaminants such as ground limestone, underburned limestone, coal or organic fragments and secondary carbonation along fractures. Most of these contaminants are recognized in the mortar samples but to different proportions. Visual analysis of the thin sections is used as well to identify the sand, including the amounts of glauconite, feldspars and lime shells present within the sand. Especially the presence of lime shells can be an additional source of fossil carbonates. In addition to the visual microscopic analysis, scanning electron microscopic analysis (SEM) on a polished section of the mortar sample is performed to identify the hydraulic properties of the lime binder. These observations are sustained by means of simultaneous thermal analysis (Thermo-Gravimetric Analysis, TGA and Differential Scanning Calorimetry, DSC, STA 449 F3 Jupiter, Netzsch) to identify the amount of water chemically bound within the binder. Furthermore, the thermal analysis provides information about the presence and the amount of gypsum and both magnesium and calcium carbonates with the mortar samples. The thermal analysis specifically proved that the binder of one of the mortar samples taken from the collegiate church of Tongeren, being considerably rich in magnesium carbonate, has been produced by means of a dolomitic lime stone. Finally the volumetric binder/aggregate ratio is estimated based on the dissolution of the mortar samples in hydrochloric acid. The characterization of the different mortar samples is compared with the radiocarbon dating results, evidencing possible pitfalls with regard to the influence of certain contaminants on the realization of an aberrant dating.

Cova de Na Dent, a lime burial sampled in 2011

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Cova de Na Dent is located on the West Coast of Mallorca, close to the village of Cala Romantica. The site is located in a cliff wall overlooking the sea (a so-called 'abrigo') and not in a real cave. The burial was situated in the left corner of the 'abrigo'. The lime burial was partly disturbed by an illegal dig. After cleaning a lime conglomerate of about 40cm could be recognized and sampled. The layer was found on top of a red brownish natural clayey soil.

The lime burial was made up of lime and fragmented bone, only a few small pieces of deteriorated bronze was found between the lime packets. In contrary to other lime burials, no identifiable funerary artifacts or charcoal pieces were present.

In the laboratory 6 different layers could be identified based on the color and consistency of the soil and the aspect and quantity of the bone fragments. These differences could not be recognized in the cave during excavation. From a small carbonate lump a thin section was made for further study. SEM and XRD- analysis were performed on the lime fragments. Each layer of the lime burial was dated. The ¹⁴C-dates (obtained by using the titration method) ranged from 2990±30 BP for the lowest layer till 1950±30BP for the upper part of the lime burial. The lowest layer was dated surprisingly old. Almost 500g of bone was recovered from the excavated lime. The bone fragments represent different parts of the body and originate from different individuals. The differences in colour and shape of the bones is typical for the differences in the degree of incineration, from burned to well cremated. SEM-analysis confirmed the difference in crystallinity between the white well cremated bones and those with a grey or black appearance. FT-IR indicated also that the bones were not uniformly cremated.

Towards a new approach to separate natural from anthropogenic lime carbonate

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Radiocarbon dating of mortars has known some successes over the past decades, certainly where it concerned pure lime putty mortars. Hydraulic lime mortars or lime mortars containing contaminants from a geological provenance, such as (roughly or finely) ground limestone or underburned limestone fragments, or contaminants related to delayed carbonate formation often still prove difficult to successfully obtain an appropriate date. The typical approach exists in dissolving a powdered mortar sample in hydrochloric or phosphoric acid and collecting successive CO₂ increments in function of reaction time. The ¹⁴C ages and stable isotope profiles of the successive dissolution increments are used to model the interfering effects of the different carbonate types. As a clear separation of the different carbonates is not necessarily possible, interpretation and stability of the results is not always straightforward and can sometimes be awkward. In addition the reaction time constants for the different fractions are for some steps very short and subject to small variations. The separation technique by chemical reaction with an acid is based on tiny differences in bonding energy due to different crystallographic properties of the carbonates. Besides chemical attack, heating of the lime carbonates to approximately 800°C is an alternative to release the carbon dioxide. Thermogravimetric analysis (TG, STA 449 F3 Jupiter, Netzsch) is applied to study the release of CO₂ of putty lime and different limestone powdered samples in function of temperature. Freshly prepared putty lime mortar, carbonated under natural conditions, is compared with limestone powder samples from white marble and Gobertange, Balegem, Maastrichter and Tournai lime stones. The thermogravimetric analysis by step heating proves that a separation of the different carbonates is possible under controlled circumstances, while the reaction times are in the order of hours instead of seconds under chemical acid reaction and therefore less subject to variations. In addition, the preliminary test results evidence that the procedure is not influenced by the fineness of the grounded samples. The first attempts to translate the step heating procedure from the thermogravimetric analysis towards the application on a traditional furnace to capture the released CO₂ fractions for stable isotope counting and ¹⁴C dating are presented as well. Further calibration of the test set-up is however necessary.

Radiocarbon dating of lime mortars in late medieval buildings in Belgium. On the accuracy of dating and local pedology

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Scientific dating techniques are increasingly employed in the re-search of medieval architecture in Flanders (Belgium). Dendrochronology is the most frequently used method, though not always feasible. Original timber may be lost or, as is often the case in Flanders, is not datable due to wood of rapid and irregular growth. In such cases, ^{14}C dating performed on atmospheric carbon dioxide enclosed within lime mortar constitutes an alternative.

Said dating technique was first applied in the parish church of Belsele (Sint-Niklaas). The oldest part of the church is the middle nave, still with its original oak roof, dated to 1266-1271 AD by use of dendrochronology. Heightening of the side-aisles in the 16th century has preserved the masonry from both the elements and rejoining during restorations. The lime mortar of the brick masonry contained little fossil carbonate (0,53 %). The mortar sample was dated to 1260-1295 AD (95,4 % prob.). This corresponds with the tree beam dating of the roof, thus confirming the simultaneity of the roof and the masonry underneath.

In the former collegiate church of Tongeren, built between the second half of the 13th century and the beginning of the 16th century, six mortar samples from an equal amount of supposed building phases were dated. Unlike Belsele, referential tree beam datings were not available since the original timber was lost in a fire in 1677. For now, the construction history of the church is partly known out of written sources, with stylistic features offering additional chronological pointers. Three mortar samples yielded plausible datings. The other samples, containing a high percentage of fossil carbonate (up to 51,5%), produced aberrant datings. The limestone used for these mortars may have been heated insufficiently into quicklime or, equally probable, aggregates containing high doses of fossil carbonate were added. This might indeed be the case considering the soil of Tongeren and its surroundings. The area abounds in limestone and marl ('mergel'), a mixture of clay and lime originating from decomposed calcareous rocks. Inversely, the soil in and around Belsele, characterized by clayey sands and large clay deposits, is markedly poor in calcareous content. Here, limestone was only available as import.

Based on the research done in Belsele and Tongeren, it is of importance to associate ^{14}C dating of lime mortar with local pedology, considering the possible impact of ingredients of local origin on the accuracy of datings.

Inadvertent Mortars and Plasters: stable isotope and radiocarbon measurements on carbonates from hearths and pottery.

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The carbonation reaction that is important to mortar and plaster dating occurs in other contexts, and this presentation considers two unusual circumstances: the carbonation of wood ash in hearths, and the decomposition and recarbonation of caliche inclusions in South Western pottery. Both circumstances generate reactive calcium oxide/hydroxide precursors through firing and result in the uptake of environmental carbon into calcium carbonate. Both systems are amenable to experiment and may provide a foil against which questions relevant to conventional mortar and plaster radiocarbon dating might be considered.

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The so-called ‘quicklime burials’ is one of the large variety of funerary practices recorded in the late Iron Age in Mallorca and Menorca. Most of them are probably the result of a process of diversification from more homogeneous inhumation systems found in the early prehistory of the Balearic Islands.

The funerary places become more varied, as well as the rites. Natural and artificial caves continue to be used, but there are changes in the shape of the latter, which tend now to be poly-lobed and often with inner columns. In parallel, open air necropolises are built for the first time. There is a reuse of older structures (funerary or not) as burial places. In some of the deposits the individuals are found in wooden coffins. The traditional inhumations coexist now with cremation rituals. These late Iron Age cultural manifestations show a marked autochthonous character.

And, in this context, the quicklime burials make their appearance. They consist of assemblages of non articulated human bones and archaeological objects found embedded in lime layers. These deposits are found mostly in natural caves and rock-shelters and in rock-cut tombs. The chronology embraces from IVth to IInd centuries BC. The Roman conquest of the archipelago in 123 BC and the following Romanization process will lead to a quick end of this indigenous world.

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Lime burials were supposed to be inhumations in quicklime. Recent studies however revealed that it are cremation burials. The burial rite consists of the cremation of the body of the deceased in contact with crushed limestone. Just like in the preparation of lime mortar the limestone rock powder decomposes into CaO and CO₂ by the heat of the pyre. The formed CaO will react with water to form Ca(OH)₂ and eventually turn into CaCO₃ due to reaction with atmospheric CO₂. In contradiction to the traditional cremations the bones are not datable by radiocarbon because fossil carbon released during the decomposition of the crushed limestone is incorporated in the bioapatite of the bones. If no charcoal from the pyre is present, the lime is the only datable material.

In this study the ‘titration-method’ was used to date different fractions of the lime carbonate from the burials. If possible the dates were compared with radiocarbon dates performed on other materials (charcoal, bone). The best results were obtained by extrapolation of the regression line.

When Dating Ancient Hydraulic Mortar Really Matters and the Gods Smile:

Three Case Studies from the Ancient Harbors of Cosa (Italy), Alexandria (Egypt) and Soli / Pompeiopolis (Turkey)

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Efforts to date ancient pozzolanic mortar collected from concrete cores taken from Roman harbor installations from around the Mediterranean by the Roman Maritime Concrete Study (ROMACONS), which I co-direct, have not been uniformly successful. The protocol for AMS dating of carbon extracted from air bubbles trapped in the mortar, pioneered by Professors Åsa Ringbom, Jan Heinemeier et alii involved in the International Mortar Dating Project, does not appear to provide consistently reliable dates for Roman hydraulic concrete (Rhc), since its curing beneath the sea does not involve CO₂ absorbed from the atmosphere. At the 1st International Mortar Dating Workshop, I did provide an apparent exception to this assertion. The Oxford Research Laboratory for Archaeology and Art History conducted a test of the AMS dating protocol on a mortar sample from a Rhc core extracted from a breakwater at Portus, the ancient emporium of imperial Rome. Surprisingly, it provided a historically accurate date for the construction of this segment of the harbor complex. This successful result was most likely due to fact that the mortar had not cured underwater. The Rhc core from which the mortar sample had been extracted had come from a segment of the breakwater that had never been submerged, and thus it had cured above water with unfettered access to air. Unfortunately, the overwhelming number of concrete cores collected by ROMACONS has come from ancient harbor installations that had been constructed underwater. In these instances, the curing of the mortar of the concrete occurred with the requisite CO₂ supplied by the chemical processes produced by the interaction of components of the ancient mortar itself. The scientific dating of Rhc cores, however, remains critical in many cases where historical evidence does not exist to establish the chronological context of the structures from which the samples had been collected. In the 3 instances involving different Roman harbor installations, organic material was found by mere chance either embedded in the mortar component of the Rhc or extracted from the surviving remains of the wooden formwork in which the concrete had been placed to cure. These samples could be dated by AMS. These wooden fragments collected at Cosa (Italy), the Great Harbor of Alexandria (Egypt) and Soli/Pompeiopolis (Turkey) were critical in establishing the time of construction or renovation for all 3 harbors and thus were instrumental in resolving various outstanding historical issues. The discovery of dateable organic material precisely where fieldwork was being conducted and in circumstances where chronological determination was critical was completely serendipitous. Sometimes the “gods” do smile on archaeologists, but one can hope that secure mortar dating protocols can be developed in the future even for Rhc, thus removing reliance on the chance intervention of the Roman goddess *Fortuna*.

Results of dating Roman Mortar in Vindonissa, Switzerland.

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In this study, mortar samples from Roman monument and a mediaeval building from Windisch (Kanton Aargau, Switzerland) were examined. Mortar samples were taken from the western gate of the roman legionary fortress of ancient VINDONISSA and from a building of the adjacent monastery of KÖNIGSFELDEN. The dating of the Roman gate was not established previously with an accuracy that would allow distinguishing between the two possible construction times, i.e. 1st or 4th century AD. On the other hand, the construction of the monastery - 14th century AD - is well known from the written sources. Therefore, the dated and undated mortar samples from Windisch were expected to be useful for checking the reliability of radiocarbon dating of mortar.

In order to select the amorphous carbonates, which were formed during the binding process, we follow the procedure described by Heinemeier et.al., (2010). The wet sieving of the mortar samples allows selection of fine fraction. This fraction was then dissolved in several steps and two or three samples were prepared from the CO₂ released in consecutive steps.

The first tests with 10 sec dissolution time resulted in radiocarbon ages that were much older than expected from Roman constructions. However, a multiple collection of the CO₂ fraction released during 3 seconds dissolution appears to be close to the expected age (Roman time and one medieval time).

Following the first attempts of dating Roman mortar new samples were taken from mortar of aqua duct excavated in Vindonissa in recent years. Results of this dating will be presented.

References:

Heinemeier J, Ringbom A, Lindroos A, Sveinbjörnsdóttir AE. 2010. Successful Ams C-14 Dating of Non-Hydraulic Lime Mortars from the Medieval Churches of the Åland Islands, Finland. Radiocarbon 52(1):171-204.

Accuracy evaluation of mortar radiocarbon dating by means of Cryobreaking, Sonication and Centrifugation (CryoSoniC) procedure: first two years of experience at CIRCE Centre.

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Mortar represents a potential buffer of atmospheric CO₂ recording, by means of ¹⁴C, the moment of its production and usage. This opportunity offers the potential to date directly constructive artifacts without the need of resort to organic materials found at the study site. Since two years CIRCE ¹⁴C laboratory (Caserta, Italy) is exploring the possibility to accurately radiocarbon date mortars, collecting data on a series of real archaeological samples characterized by well-constrained chronologies. To date, lime lumps, aerial and hydraulic mortars have been analyzed allowing, both, the improvement of the procedure and showing its overall feasibility (i.e. accuracy). In this contribution main results of the activity will be shown highlighting also protocol pitfalls when observed.

Lime lumps as a material for mortar dating.

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Lime mortars have several generations of calcium carbonate and these may have completely different ¹⁴C ages. It is well known, that some of the carbonates reflect the original geological age of the material rather than the building phase in which it was used and others have grown within the mortar centuries after it hardened in response to moisture damage, fires and sudden exposure of still alkaline deeper parts in the constructions. Most of the carbonate was, however, formed in the time period between lime slaking and the hardening of the mortar, which is usually the time we want to date. Several researchers have tested dating of lime lumps with promising results. If the lumps were formed in the slaked lime already before the often problematic aggregate was added then they might be especially well-suited for dating. We have dated a large number of lime lumps and also studied their chemistry and mineralogy. Many lime lumps do indeed yield uniform ¹⁴C ages throughout the dissolution process and the obtained age correspond well with the historical age when this is known. Some lumps yield increasing ¹⁴C ages when the dissolution process is prolonged to several hours. The chemistry and mineralogy of these are discussed. Lime lump ages from Rome, Gotland in Sweden and from SW Finland are presented.

