**Dating Ancient Mortar**

Although radiocarbon dating is usually applied to organic remains, recent work shows that it can also reveal the age of some inorganic building materials.

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Even more than digging implements, archaeologists need tools for finding the age of the objects they study. After all, many sites and remains—in caves, in deserts, on the sea floor—require no excavation, but all must be dated. When archaeologists of the future write the history of their dis-

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discipline, the second half of the 20th century will stand out for the development of many scientific methods for ascertaining the age of artifacts. This article is an account of how our Scandinavian-American team, which includes a nuclear physicist, a geologist, an art historian, and two archaeologists, developed the means for dating ancient building materials that contain lime mortar.

In the early days, archaeologists attempting to make age determinations often depended on information supplied by others. Principally, they relied on historians, who knew the chronologies of literate societies of the past five millennia, with their written inscriptions on seals, records, tombs, monuments and coins. Archaeologists also relied on geologists, who could sometimes make age determinations based on the association of human remains with geological features of known age.

Unfortunately, this dependence on historical dates and geological associations left large areas of the human past untouched. But beginning in the late 1940s, a new world opened with the development of radiocarbon dating for organic remains, tree-ring dating for wood, thermoluminescence for fired clay and potassium-argon dating for volcanic materials. Of these, radiocarbon dating had the most universal importance for archaeology. So vital was its discovery that the pioneer of the field, Willard F. Libby, was awarded the Nobel Prize for Chemistry in 1960.

Radiocarbon 101

The underlying principles of radiocarbon dating are straightforward. Libby and his coworkers realized that cosmic rays impinging on the upper atmosphere create a steady supply of the radioactive isotope of carbon: carbon-14 (or 14C). Plants absorb traces of the 14C during photosynthesis. Animals in turn absorb 14C by eating plants. Initially, the ratio 14C to normal carbon in plant and animal tissues equals the roughly constant atmospheric concentration. But after an organism dies, radioactive decay reduces the original amount of 14C by half every 5,730 years. This phenomenon provides a built-in clock for dating most human foods and many raw materials for tools, weapons, ornaments and buildings. Libby confirmed the validity of his dating method using wood fragments of known age, including heart-wood of a stump of a California redwood tree almost 3,000 years old and the deck board from the funeral boat of the Egyptian pharaoh Sesostris.

Two subsequent developments greatly enhanced the value of 14C dating. Investigators made radiocarbon measurements on the yearly growth rings of long-lived bristlecone pines, which provided an annual record of the varying concentrations of 14C in the earth’s atmosphere over the past four millennia. These results made it possible to account for slight variations in the atmospheric concentration of 14C and thus to construct a calibration curve that could translate “radiocarbon ages” (those determined using only a simple calculation based on radioactive half-life) into true calendar ages. Equally important was the introduction of particle accelerators to separate carbon isotopes and count directly the 14C atoms in the sample, a technique that came to be known as accelerator mass spectrometry or AMS. This advance drastically reduced the amount of material needed: Only one milligram of carbon is required for AMS analysis,
whereas the traditional procedure (the so-called “conventional” radiocarbon method), which involves the counting of particles emitted in the slow radioactive decay of $^{14}$C, requires several grams of carbon to produce a date.

Even with these advances, the study of buildings and other structures presented special problems. Direct dating of an edifice usually required that it was made (at least partially) of wood and that its original timbers were preserved so that they could subjected to $^{14}$C analysis or examined to determine characteristic patterns in the tree rings the wood contains.

Even when such an analyses provides precise dates, an inherent uncertainty remains because the wood tested could be older than the building itself—or it could be younger, if material from later repairs was misidentified as original. In the case of buildings made of mud brick, stone, mortar or cement, these methods cannot be applied at all. In such situations, archaeologists often dig through vast areas around ancient structures—and in consequence irretrievably disturb or destroy material—in search of coins, inscribed objects, fragments of charcoal (which contain carbon) or other datable items that might lie buried in the builders’ trenches or sealed in the walls or floors.

This reliance on secondary dating, aside from its wastefulness in time and effort and archaeological resources, is vulnerable to serious error. Older coins, for example, might find their way into a new building; later objects too might be introduced long after the main structure was erected. Even the largest elements of the structure may cause confusion. For example, the monumental columned porch of the famous Pantheon in Rome bears a prominent inscription proclaiming that it was made by Marcus Agrippa during the reign of the first emperor, Caesar Augustus. But the stamps on the bricks in the great dome prove that everything visible today was built during the reign of Hadrian, more than a century later.

Archaeologists must find ways to overcome these difficulties, for it is of the primary importance in many cases to know exactly when a building was constructed. The complex cultural, technological and economic systems that lie behind all large-scale buildings can provide important clues to the nature of the particular culture and period in question. Whether the archaeologist is dealing with a decorated pyramid in Mexico, a Moorish palace in Spain or a Roman market, the study loses much of its value if the time of construction cannot be pinpointed.

In the 1960s investigators in France attempted to extend $^{14}$C dating to certain inorganic substances. In particular,
they knew that all building materials based on lime—mortar, concrete, plaster, whitewash—absorb atmospheric carbon dioxide as they harden. In this way 14C is fixed in all these lime-derived substances at the exact time of construction. And from that moment the 14C clock begins ticking, just as it does for the remains of any plant or animal immediately after its death. Thus if 14C analysis could be applied to mortar, the radiocarbon clock could be rewound to the point in time when the building came into existence.

The principle was simple enough, but its application proved surprisingly difficult. Although Robert L. Folk and Salvatore Valastro, Jr., of the University of Texas at Austin established many of the prerequisites for this technique in the 1970s, in general the results were so poor that after a few more years, work on this particular application of 14C virtually ceased. One investigator who persisted was Mark van Strydonck of the Royal Institute for Cultural Heritage in Brussels. He found that conventional 14C dating could at times yield accurate results on mortar samples, the process was both complicated and unreliable. The main difficulty was the presence of impurities in all lime-derived building materials—impurities that could seriously affect the outcome of the analysis. Van Strydonck recommended that 14C traces in mortar, or in wood or charcoal fragments embedded in the mortar, might be dated by the AMS method. The difficulty with analyzing charcoal fragments is that they (just like the timbers used in construction) could come from old wood and thus could be anywhere from a few years to several centuries older than the building in which the mortar was found. Direct analysis of lime mortar avoids this problem.

**Lime Is Key**

Lime is created by heating limestone or marble in a kiln to a temperature of 900 degrees Celsius, well above the temperature reached in open wood fires. Charcoal or forced air are thus prerequisites for the making of lime. When the heat reaches 900 degrees, carbon dioxide is completely released, leaving quicklime (calcium oxide) behind, a substance much whiter and more powdery than the original stone.

The quicklime is slaked with water to produce building lime (calcium hydroxide, the source of whitewash and plaster), which absorbs carbon dioxide...
from the atmosphere as it sets. Unfortunately, most lime samples contain impurities in the form of incompletely burned limestone fragments or particles. Because this limestone derives from fossil carbonate deposits, even small levels of contamination will make the sample appear far too old when subjected to \(^{14}\text{C}\) dating.

An additional source of contamination may be introduced when the builder decides to make mortar rather than plain lime. This is done by adding to the quicklime an aggregate—typically sand, gravel or crushed ceramic material—along with the water. Any of these substances can affect the \(^{14}\text{C}\) analysis of the resulting mortar, with the limestone often found in beach sand being perhaps the most troublesome.

Whether pure lime or mortar is used, the chemistry remains the same. The building lime (calcium hydroxide) reacts with carbon dioxide in the atmosphere to form calcium carbonate. But even in the hardening process there are potential problems. Mortar lying on the inside of walls or behind stone facings may take years or even decades to solidify, thus yielding a date that is too young for the building as a whole. Also, mortar exposed to rain may recrystallize, thus resetting the radiocarbon clock long after the original hardening, making the sample appear too young.

Such complications probably dissuaded many people from attempting to determine \(^{14}\text{C}\) ages for mortar. But it sometimes happens in the course of scientific research that an illusory initial success leads to a genuine advance. Those involved must then attribute part of their progress to a strange combination of error and luck. Such was the case with the more recent efforts to develop a reliable method for dating building lime and mortar.

In the late 1980s archaeologists and physicists from the Åland Islands (a Swedish-speaking autonomous province of Finland) and from Finland proper were seeking to date a medieval Franciscan monastery on the remote island of Kókar, on the edge of the Åland archipelago. This island had been important since the Bronze Age, when seal hunters from Germany and Poland established a hunting and oil-processing station there. Traditional dating placed the construction of the monastery and its church in about the year 1450.

Archaeologist Kenneth Gustavsson of the Åland Museum in Mariehamn and physicist Högne Jungner of the Helsinki University Radiocarbon Laboratory took large samples of mortar from the masonry of medieval ruins surrounding the church at Kókar and submitted them for conventional \(^{14}\text{C}\) dating. Gustavsson and Jungner were astonished when the laboratory reported a date of about 1280—more than a century and a half older than expected. And they were further surprised when Gustavsson’s subsequent excavations in and around the church yielded coins and jewelry of types that supported such an early date. Later, thermoluminescence dating of roof tiles from the church’s outbuildings also indicated that they had been built in the 13th century. Thermoluminescence dating (a procedure that uses the small amount of light released during heating to measure the dose of natural radioactivity a ceramic sample has received since it was fired) has its own built-in uncertainties, but the agreement with the radiocarbon determination was compelling. The extraordinary value of mortar-dating for archaeology seemed to have been proved.

Only long afterward did these investigators realize how lucky they had been. Although Kókar and the rest of the Åland islands are made mostly of granite, some of this bedrock has been overlaid since the Ice Age with blocks of limestone deposited by glaciers. Erosion of this glacial cover contributed limestone particles to most Åland beaches, so the builders of the medieval stone churches on these islands typically introduced fossil limestone into the mortar they used when they added beach sand as aggregate to their quicklime. The little island of Kókar, however, is different: It has beach sand and gravel composed almost exclusively of quartz and feldspar. The medieval masons who constructed the Franciscan monastery there used the local beach sand, with the result that the aggregate in their mortar did not throw off Gustavsson and Jungner’s \(^{14}\text{C}\) analyses.

This promising start led to a new project intended to date the eight great medieval “Mother Churches” scattered through the Åland islands. For that Jungner joined forces with two of us (Ringbom and Lindroos). Lindroos, being a geologist, was well prepared to study the physical, mechanical and chemical properties of the various carbonate minerals in the mortars, including the contaminants. Ringbom, in addition to being an art historian, was drawn to the project because she had a family interest in mortar: Her father had been a cement engineer.

The Åland churches are important repositories of medieval sculpture, painting and manuscripts, but no records survive documenting the erection of the buildings themselves. Mod-
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When Ringbom, still working on the Åland churches, learned of the promising results from the Newport Tower, she resolved to abandon the earlier approach and start all over again using only AMS \(^{14}C\) dating. After doing so, the age determinations proved plausible and consistent. Mortar dating indicated that the naves of all eight churches had been completed during a very short interval, from 1280 to 1300, matching the age that the monastery at Kökar was established. Studies of the tree rings in timbers found in the bell tower of one of these churches (at Jomala) dated the structure to 1281. Five samples of mortar from that tower yielded \(^{14}C\) dates of 1279 to 1290—the most remarkable bull’s eye yet achieved with the newly developed method.

Indeed, AMS-based mortar dating appeared to yield a full history for these previously enigmatic structures. The bell tower at Jomala was later copied in the other parishes. Hammarland church got its west tower in 1310 and Lemland in 1316. Then after a long gap, towers were added to the other churches between 1381 and 1467. Porches were added later still. Thus earlier conflicts about the ages of the churches could be explained in part by...
incremental building, a practice fully revealed by AMS dating of the mortar.

Initially it seemed surprising that all these churches should have been established in one great burst of concentrated energy, considering the costs, effort and expertise involved. But Ringbom found a possible explanation. In about 1280, these islands began to enjoy an economic boom as the Alандers supplied timber and lime mortar for the building of two new cities: Stockholm to the west in Sweden and Åbo (Turku) to the east in Finland. The financial fruits of this windfall seem to have found their way into the eight monumental churches, symbols of the Alандers’ communal pride and pious gratitude.

This work on the Åland churches brought important refinements to the mortar-dating method. For example, finer meshes than had previously been used aided the mechanical separation of pure fired lime from contaminants, as did adding the steps of dry and wet sieving. And a technique called cathodoluminescence—essentially bombarding a sample with electrons and viewing the light given off—allowed impurities that could affect the date to be made readily visible. Also, it proved worthwhile to produce a sequence of subsamples of the carbon dioxide released from the mortar after the application of an acid so as to test the consistency of dates derived from various fractions. It turned out that for most of these samples the very first gas fraction came from rapidly dissolving carbonate in the hardened lime, thus yielding the correct date of the building. The second gas fraction was contaminated with carbon dioxide from slowly dissolving fossil limestone, thus giving an erratic result that tended to be too old.

With the promising results in from Kökar, the Newport Tower and the Åland churches, the mortar-dating method was securely established. But from an archaeological point of view, the work was just beginning. Ahead lay the application of this method to mortar samples from different periods and environmental settings (including underwater structures) and the development of precise procedures for collecting the samples. It was already clear that success might require site visits by a number of specialists to verify the original position and condition of each sample: where it lay in the structure, whether it remained chemically pristine, what the local sources of raw materials and potential contamination were and so on.

Beginning in 1999, we formed an interdisciplinary team to test this method on mortars from more ancient sites. Our group includes a physicist (Heinemeyer), an art historian (Ringbom), a geologist (Lindroos) and two archaeologists (Hale and Lancaster). Our focus has been on the Mediterranean and the development of precise procedures for collecting the samples. It was already clear that success might require site visits by a number of specialists to verify the original position and condition of each sample: where it lay in the structure, whether it remained chemically pristine, what the local sources of raw materials and potential contamination were and so on.

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Figure 6. Radiocarbon measurements are easily cast in terms of radiocarbon years before present, using the known half-life of carbon-14. Careful measurements of tree rings have, however, shown that such determinations do not correspond exactly with calendar ages, a result of the slight variation over time in the concentration of atmospheric carbon-14. The calibration between radiocarbon age and calendar age (solid black line, left) varies from the simple relation that would result had the concentration of atmospheric carbon-14 stayed constant (dashed red line). The authors’ analysis of the mortar dates they obtained from the Roman amphitheater in Mérida (center) shows that a bell-shaped error distribution on the radiocarbon measurements (red) corresponds to a rather wide and erratic distribution for the possible calendar age of this structure (green). Their analysis of the mortar dates for a medieval stone church of the island of Jomala (right) produced a much narrower distribution of possible calendar ages, in part because the radiocarbon calibration curve for this interval is quite steep.

When in Rome...

The city of Rome lies between two extinct volcanic systems. As a result, its builders had access to extensive deposits of pozzolana, an unconsolidated volcanic ash that is very rich in silica and alumina. By the first century BC, Romans were improving their mortar by adding this local material to the mix. When combined with builders’ lime, the silica and alumina in the pozzolana cause a chemical reaction that creates a mortar that is eight to ten times stronger than mortar made with quartz sand.

Like modern Portland cement, pozzolana mortar will harden under water, because it can react with dissolved carbon dioxide. By chance or experimentation, Roman builders discovered that a similar mortar with hydraulic properties could be produced without pozzolana, by adding crushed terracotta as an aggregate. In this case, the fragments of fired clay from old tiles and pots introduced silica and alumina into the mortar. Less porous than pozzolana, the crushed terracotta tended to be less chemically reactive and
Figure 7. Sampling of mortar from the Roman amphitheater in Mérida helped to solve a mystery: Archaeologists had uncovered an inscription there indicating that the structure was erected in 7 BC; yet the similarity of this amphitheater with the 1st-century AD Colosseum in Rome suggested that their construction was contemporaneous. Here mortar dating indicated that the amphitheater in Mérida was indeed built in the first century AD, indicating that the inscription represents older material incorporated into the structure by its builders.

correction was 7 AD, making inferences about the correct age of the structure, such as a depiction of the Pantheon. In each of these cases, the “historical” evidence gives an incorrect date.

Our work within the old Roman province of Lusitania did not end in Merida. Nearby were many large farms or villas, where the construction and expansion projects over the centuries provide a barometer of Roman economic prosperity. The largest of these villas was discovered in 1947 at Torre de Palma in eastern Portugal, which was excavated by a team from the University of Louisville under the direction of Stephanie Maloney, starting in 1983. The villa at Torre de Palma included a richly decorated house for the owner, slave quarters, barns, granaries, bath houses, stables, work shops, a wine press and an olive press—not one of which could be dated by inscriptions or other documentary evidence. Much excavation was carried out simply in the hope of finding artifacts that might provide clues to the age of the structure, such as a late Roman coin sealed in a floor where it had been dropped during the pouring of the concrete. The most important building on the site was the early Christian church or basilica, with an adjoining baptistery and cemeteries. German art historians had dated the complex on stylistic grounds to the 6th century AD, when Visigothic kings had taken over the rule of Lusitania and the rest of Iberia. But during the first season of the Louisville excavations, 10 small bronze
coins were found in the mortar under the marble floor near the altar, all of them minted in the middle of the 4th century AD during the time of the sons of Constantine, the first emperor to convert to Christianity. Measurements of the basilica showed that it had been laid out on a grid of Roman feet, and the high quality of the masonry there seemed to support the notion that it had been constructed during the years before the fall of the Roman empire.

Here, as with the Åland churches, mortar dating by AMS analysis was able to reveal the complexities hidden under the archaeological surface. The sanctuary around the altar was indeed constructed during the time of Constantius II in the mid-4th century AD, as was the central part of the baptistery with its unusual “double-cross” shaped pool. But much of the church had been built long after the fall of imperial Rome, after the Visigoths took over control of Iberia in the 6th century AD. A great building project in about 580 AD raised the walls of the nave, with their heavily mortared masonry. From this it follows that in the depths of what are conventionally called the “Dark Ages,” this remote corner of Portugal supported active quarries, lime kilns, marble cutters and polishers, stone masons, architects and contractors. Such elaborate works could only be carried out in a healthy economy. The mortar dates for the basilica of Torre de Palma thus provide important clues about the survival of Roman technology and social order in the centuries after the fall of the last emperor.

The potential benefits of the new mortar-dating method are great. At a time when archaeologists try to dig less and less in an effort to preserve the world’s archaeological heritage for future generations, the method offers the possibility of learning a great deal before excavation is even attempted. In an optimal situation, remains of ancient buildings, whether as isolated ruins or incorporated in later structures, can be dated from samples of no more than a few grams of mortar. An archaeologist carrying out a field survey may be able to determine the age of a building that once stood there simply by collecting fragments of mortar from ancient walls or floors. Buildings with complex histories of expansion and repair can have their stories unfolded. And art works such as frescoes and mosaic pavings can be dated not only on their artistic style but also by determining the moment when the mortar hardened. The results should be significant not only for the history of technology but for human history as a whole.

**Bibliography**


**Links to Internet resources for further exploration of “Dating Ancient Mortar” are available on the American Scientist Web site:**

[http://www.americanscientist.org/articles/03articles/hale.html](http://www.americanscientist.org/articles/03articles/hale.html)