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^{14}C DATING MORTAR IN IRELAND

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ABSTRACT. I have developed a method of dating early medieval Irish buildings using charcoal encased in mortar. Due to the inclement weather over centuries, timbers do not preserve well in these structures, leaving little suitable material for ^{14}C dating. Initially, several buildings of known age were analyzed to verify the mortar charcoal technique. Then, a series of buildings for which no definite architectural-historical dates existed, *e.g.*, churches, houses, oratories and round towers, was successfully tested. I discuss here the results of this dating approach, and provide architectural historians with a firmer understanding of the origin and antiquity of early Irish buildings.

INTRODUCTION

Until recently, the origin of a considerable number of historical buildings and sites listed in the "Guide to the National Monuments in the Republic of Ireland" (Harbison 1982) was not well understood. Details of construction dates, such as would appear in reliable literary accounts or characteristic styling elements, were largely lacking. A method for ^{14}C dating timber of medieval buildings had been worked out earlier for Europe and England (Berger 1970). However, a new technique was needed for Ireland, as most of the structures no longer contain original timbers because of the harsh Irish climate and historical events, which rule out conventional ^{14}C dating. Thus, I have developed such a method using mortar, which typically outlasts wood. With varying success, several ^{14}C scientists have tried to date construction mortars using either the inorganic or organic fraction of mortar (Delibrias, Guillier & Labeyrie 1964). Yet Stuiver and Smith (1965) encountered disappointing discrepancies in their study of similar material. Other investigators studied organic matter used in mortar preparation (Kedar & Mook 1978). Baxter and Walton (1970a) and Folk and Valastro (1979) also studied the feasibility of ^{14}C dating mortar samples with inorganic fractions. Most recently, Van Strydonck, Dupas and Keppens (1989) investigated the isotopic fractionation of oxygen and carbon in mortar, demonstrating that neither $\delta^{13}C$ or $\delta^{18}O$ can provide any information on the fossil carbonates in mortar.

THE GEOCHEMISTRY OF MORTAR PRODUCTION

The raw material used in mortar production is chiefly limestone, but occasionally sea shells. For convenience of handling, limestone is broken down into centimeter-sized pieces and bedded together with charcoal in a lime kiln. During heating (or calcining), CO_2 is liberated from the carbonate raw material, producing "burned lime."

$$\underset{\text{limestone}}{CaCO_3} \rightarrow \underset{\text{burned lime}}{CaO} + CO_2 \quad . \tag{1}$$

If the original limestone is not heated sufficiently, some source carbonate remains from the time when the rock was originally formed. Because this typically dates into a 1 Ma+ range, no ^{14}C is detectable in limestone.

After the calcining, the kiln is emptied, and burned lime is quenched in water, releasing much heat in an exothermic reaction to yield "slacked lime." This process is allowed to go to completion in a matter of days or weeks, depending on the batch size, to ensure complete reaction and homogeneity of the formerly lump-sized material.

$$\underset{\text{burned lime}}{CaO + H_2O} \rightarrow \underset{\text{slacked lime}}{Ca(OH)_2} \quad . \tag{2}$$

To produce mortar for construction, slacked lime is mixed with sandy aggregate. At this stage, other carbonate-containing material of different geologic age may be introduced, if the sand is not composed of pure silicate, as is usually the case. Thus, two sources of geochronological contamination exist: 1) carbonate from a partially burned parent material with its potential age; and 2) some other carbonate from the aggregate of yet another age. In practice, however, both these ages are older than the ^{14}C-dating limit, because all of the isotope has decayed over geological time. Therefore, the joint contamination is no longer measurable in ^{14}C dating equipment, and is said to be "dead." The resulting diluted ^{14}C concentration will produce a false age. In such a hypothetical combination of historical and geological carbon, the date obtained would be much too old. Therefore, accurate ^{14}C dating demands that the ratio of these sources of carbon is precisely known, in order to calculate the correct age.

Once mortar has been placed between stone courses in a building, it begins to set by reacting with CO_2 from the Earth's atmosphere, which contains cosmogenic ^{14}C

$$\underset{\text{slacked lime}}{Ca(OH)_2 + {}^{14}CO_2} \rightarrow \underset{\text{mortar carbonate}}{Ca^{14}CO_3 + H_2O} \quad . \tag{3}$$

In this manner, most of the hydrated or slacked lime is converted to calcium carbonate crystals, locking aggregate and stones into place. This reaction is the step in which ^{14}C from the contemporary atmosphere is introduced into the mortar mix. If no "foreign" carbonates are present, a reliable mortar-carbonate date is possible. The problem is knowing if a geological carbonate is present, or if it is, precisely how much.

Methodology

My research on this problem centered on testing the reliability of mortar-carbonate dating on early medieval Swiss churches of known age. However, this led to generally unsatisfactory results, even though I used an oxygen isotopic correction, and observed that the differences in $^{18}O/^{16}O$ in mortar and $^{18}O/^{16}O$ in most carbonates of sedimentary rocks or their sand-like erosion products are too small to act as accurate indicators of the historical/geological carbon ratio. Hence, accurate dating corrections cannot be made. During these tests, I noted that many charcoal particles remained in the mortar residue. This charcoal came from the original fuel used to produce burned lime, but was not totally consumed in the heating process.

The first Irish early medieval samples were selected as a source of historical carbon in the form of charcoal enclosed in mortar. Two dangers still existed: 1) if isotopically dead coal had been used, only infinite dates could be expected; 2) peat, as a fuel, would also yield only very old dates, as well as remnants of wood, such as bog oak, if they had been used in manufacturing this mortar in the samples.

Thus, the next tests had to involve mortar of known age, *i.e.*, from buildings of established origins, in order to check the reliability of the method. On balance, the list of well-dated structures is not as extensive as one might like, but these results show that mortar-charcoal dating provides the best chronology for medieval structures in Ireland.

Whenever possible, samples were collected from behind masonry blocks, which were removed and later reset by experts from the Board of Works. Gross contaminants, *e.g.*, roots, insect remains,

were mechanically removed in the field and in the laboratory. To obtain datable charcoal, mortar was treated with cold dilute hydrochloric acid until all inorganic carbonate had been destroyed and liberated as CO_2. This process lasted from days to weeks per sample, depending upon the integrity of the mortar being dissolved. The resulting residue was washed with distilled water, treated with weak (1N) carbonate-free sodium hydroxide to exclude humic acids, washed again with distilled water of mildly acidic pH and dried at 110°C. This mixture of charcoal particles and carbonate-free inorganics was then burned to CO_2 in a stream of pure oxygen. CO_2 was purified by passage through solutions of silver nitrate and then chromic acid to exclude halogens and to ensure complete oxidation. Also, any CO was converted to fully oxidized CO_2 by treatment with hot copper oxide. As a last purification step, all electronegative contaminants, such as trace amounts of oxygen, were eliminated by repeated passage of CO_2 through pure copper at 650°C. Radon ($t_{1/2}$ = 3.8 days) resulting from trace amounts of uranium found in the samples, was allowed to decay completely for one month, equivalent to about eight half-lives.

After this chemical and physical purification process, all samples were assayed for ^{14}C content in gas proportional counters, the largest of which (7.5 liters) needs 4 g of carbon for a complete fill and has a statistical counting error of as little as ± 20 yr, in the time range of the Middle Ages. A smaller 200-ml unit, for samples of 100 mg or less, can measure dates to within ± 100 yr over several days of counting time. These counters are routinely calibrated with the oxalic acid standard provided by the U. S. National Institute of Standards and Technology (NIST, formerly NBS), with radioactively dead CO_2 from marble and with known-age samples.

Results for known-age buildings

The most architecturally outstanding Romanesque church building is Cormac's Chapel on the Rock of Cashel in County Tipperary, Ireland (Fig. 1). This structure has been securely dated by historical documents to AD 1127–34 (Leask 1987). The least disturbed area appeared to be the chapel croft, from which two mortar samples (UCLA-2582 and 2584) were removed and dated (Table 1).

TABLE 1. Historical and ^{14}C Dates of Irish Buildings of Known Age

UCLA-no.	Building	^{14}C age (yr BP)	Cal age AD*
2582	Cormac's Chapel–12th c.	900 ± 40	1038–1183
2584	Cormac's Chapel–12th c.	925 ± 35	1030–1166
2713B	Cashel Round Tower–11th c.	990 ± 90	960–1160
2713C	Cashel Round Tower–11th c.	975 ± 110	960–1180
2714A	St. Columb's House–9th c.	1270 ± 125	650–890
2714B	St. Columb's House–9th c.	1295 ± 80	654–786
2715	Kilmalkedar Church–12th c.	910 ± 60	1024–1183

*Stuiver and Pearson (1986) calibration

Two samples were also selected from the Cashel Round Tower, dating from a time somewhat earlier than Cormac's Chapel, based on architectural-historical expectations. Both samples (UCLA-2713B and 2713C) came from the interior of the Round Tower from behind surface mortar or stone masonry. Sample UCLA-2713A contained too little charcoal for a meaningful date. Table 1 lists the results, which are in general agreement with archaeological and architectural-historical considerations.

St. Columb's House in Kells, County Meath is thought to have been built shortly after AD 804 (Leask 1987). Two samples, UCLA-2714A and UCLA-2714B, were removed and dated. Calibrated

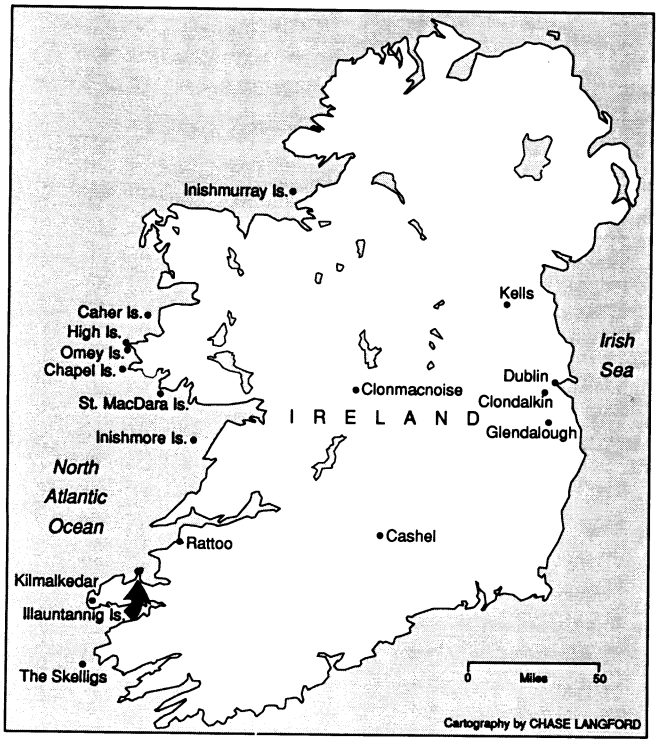

Fig. 1. Ireland: map of site locations

radiocarbon dating places this building into the 7–9th century AD (Harbison 1982), making it one of the oldest mortared structures in Ireland designed as an oratory.

UCLA-2715 came from the 12th century Romanesque church at Kilmalkedar, County Kerry (Table 1). Archaeological experts recognize both design and ecclesiastical ties between Kilmalkedar and Cormac's Chapel in Cashel (Killanin, Duigan & Harbison 1989; Harbison 1982). The sample was obtained from behind interior stones and dated to 910 ± 60 (UCLA-2715), virtually identical with the dates from Cormac's Chapel. Table 1 shows that these mortar charcoal dates compare well with historically expected ages (Berger 1990).

Buildings of Undetermined Age

Described briefly below are the structures of previously undetermined age, dated by the mortar-charcoal method. Table 2 summarizes the ^{14}C results.

County Dublin

Clondalkin Round Tower is located in an early monastery attributed to St. Cronan (Harbison 1982). Round towers are thought to have been erected between the 10th and 12th centuries: Clondalkin Round Tower is one of the earlier examples of this uniquely Irish form of architecture. A mortar sample (UCLA-2749) was dated to 1050 ± 50 BP.

County Galway, Aran Islands

Temple Benen on Inishmore, a small oratory of cut stones, overlooks Killeany Bay. Sample UCLA-2731A was dated to 965 ± 65 BP. As this "minichurch" was expected to date as early as the 8th century, the ^{14}C result places it much later in time (Harbison 1982; Killanin, Duigan & Harbison 1989) probably the 11th century as explained below.

TABLE 2. Mortar Dates from Buildings of Previously Undetermined Origin

UCLA-no.	Building	^{14}C age (yr BP)	Cal age AD*
2570	High Island Oratory	1389 ± 195	430–852
2727B	Clonmacnoise, Temple Ciaran	1245 ± 55	681–881
2738D	Skellig Michael, St. Michael's Church, Phase I	1250 ± 25	688–777
2725D	Inishmurray, Teach Molaise	1215 ± 45	724–736
2792	High Island Oratory	1090 ± 105	810–1020
2725C	Inishmurray, Men's Church	1040 ± 160	810–1170
2751D	Caher Island Oratory	1100 ± 50	886–1000
2727A	Clonmacnoise Round Tower	1080 ± 60	891–1012
2749	Clondalkin Round Tower	1050 ± 50	897–1025
2722D	Skellig Michael, St. Michael's Church, Phase II	975 ± 75	985–1160
2822	Omey Island, St. Feichin's	825 ± 200	1010–1310
2750	Rattoo Round Tower	980 ± 40	1012–1039
2731A	Inishmore, Temple Benen	965 ± 65	1012–1160
2823	St. MacDara's Island, Church	875 ± 100	1020–1260
2748A	Glendalough, St. Kevin's	910 ± 60	1024–1217
2821	Chapel Island, St. Caillin's	860 ± 90	1030–1260
2819	Inisheer, St. Cavan's	900 ± 55	1030–1217
2731C	Inishmore, Temple MacDaugh	890 ± 60	1030–1226
2824	Inisheer, Kilgobnet	860 ± 60	1047–1093
2725B	Inishmurray, Women's Church	460 ± 80	1406–1475
2773AA	Illauntannig midden**	4030 ± 60	2510 BC†
2773A	Illauntannig midden**	1340 ± 40	654–682
2751C	Caher Island, Priest's House‡	2470 ± 140	350 BC–AD 20§
2773B	Illauntannig midden‡	1975 ± 45	400–460§
2571A	Caher Island Oratory‡	1540 ± 370	500–1260§

*Stuiver and Pearson calibration (1986)
**Charcoal sample
†Pearson *et al.* calibration (1986)
‡Marine shell sample
§Stuiver, Pearson and Braziunas calibration (1986)

Temple MacDuagh on Inishmore is a small pre-Romanesque church southwest of Kilmurvey on the westernmost isthmus of the island. A mortar sample, UCLA-2731C, dated to 890 ± 60 BP. From an architectural-historical point of view, both Temples Benen and MacDuagh should antedate Cormac's Chapel of the 12th century. Because ^{14}C dating cannot distinguish between the 11th and 12th centuries, these Aran temples could actually date to the 11th century (Harbison 1982; Killanin, Duigan & Harbison 1989).

Kilgobnet Church (Fig. 2) on Inishere is a small oratory from which a mortar sample, UCLA-2824, dated to 860 ± 60 BP.

St. Cavan's Church on Inishere is a small church entrenched in a sand dune. Mortar was removed from the older section of the church and dated to 900 ± 55 BP (UCLA-2819). As Harbison (1982) points out, the Aran Islands churches were somewhat difficult to date by conventional means, so that calibrated dates provide a more secure temporal framework.

Fig. 2. Kilgobnet Church on Inishere, Aran Islands

County Galway

High Island Monastery has been associated with St. Feichin of Fore, who died in AD 664 (Harbison 1982). A sample collected in 1984 dated to 1395 ± 195 BP (UCLA-2570) and calibrated to AD 430–810 or 5th–9th centuries. The most recent limit, *i.e.*, the 9th century, is the most acceptable in terms of our present understanding of first use of mortar in Ireland. A second sample collected in 1987, UCLA-2792, dates to 1090 ± 105 BP and calibrates to AD 810–1020.

St. Macdara's Island Church, a monastic site in County Galway, is said to have been founded by St. MacDara in the 6th century (Harbison 1982). A sample was collected in 1987, UCLA-2823, which dated to 875 ± 100 BP and calibrates to AD 1170-1280.

St. Caillin's Church, Chapel Island. A mortar sample collected in 1987, UCLA-2821 dated to 860 ± 90 BP which is calibrated to AD 1030–1260.

St. Feichin's Church, Omey Island. A small sample, UCLA-2822, dated in the 200-ml counter to 825 ± 200 BP (calibrated AD 1020–1320). Legend reports that seven brothers, St. Feichin, St. MacaDara, St. Caillin, *etc.*, sons of the King of Leinster, built churches in bell-sound of each other, which these dates seem to support. However, different design features rule out the same architect-builder.

County Kerry

Illauntannig is a monastery that St. Senan is reported to have founded on this island in the 6th or 7th centuries. A massive stone wall surrounding the monastery may have been a ring-fort (Marshall 1989; Killanin, Duigan & Harbison 1989; Harbison 1982). None of the structures in this site contain mortar, so samples of shell and charcoal were collected from a midden. UCLA-2773A was composed of coarse charcoal and dated to 1340 ± 40 BP. Another charcoal sample, UCLA-2773AA, was much older, 4030 ± 60 BP. While the first may relate to the construction of the wall, the older one either refers to older organic material or an early prehistoric site upon which the monastery was constructed. The third sample was shells, UCLA-2773B, dated to 1975 ± 45 BP.

This date converts to the 5th century AD (Stuiver, Pearson & Braziunas 1986) and agrees with the 7th century date of UCLA 2773A suggesting an early Christian time frame.

Rattoo Round Tower is part of the remains of a little known monastic foundation. A late medieval cemetery is located nearby (Killanin, Duigan & Harbison 1989; Harbison 1982). A sample was collected in 1984, UCLA-2750, dating to 980 ± 40 BP. This compares well with other dates for round towers, *e.g.*, Cashel (Table 1) and Clondalkin (Table 2).

Skellig Michael, off Port Magee near Caherciveen, was founded supposedly by St. Finan. Historic records list a Viking raid in AD 823 and other funerary dates of AD 950 and 1044 (Harbison 1982). The monastery continued to be used into the Middle Ages, and is still the aim of pilgrimage today. Sample UCLA-2738D was collected from St. Michaels Church (Fig. 3) and dated to 1250 ± 25 BP. The later expansion of the original 8th century church permitted us to select a sample, UCLA-2722D, from a newer construction phase which dates to 975 ± 75 BP. Thus, Phase I of St. Michael's Church dates to the 8th century, and phase II to the 11/12th. Literature on Skellig Michael includes Horn, Marshall and Rourke (1990), Killanin, Duigan and Harbison (1989), Harbison (1982) and Lavelle (1976).

Fig. 3. St. Michael's Church on Skellig Michael, County Kerry

County Mayo

Caher Island is a small island containing an ancient monastic enclosure with a small church ruin. The mortar of this building contains large amounts of shells, some of which were collected in 1984 by J.-W. Marshall of UCLA. UCLA-2571A dated to 1540 ± 370 BP. A mortar-charcoal sample was dated in 1989 (UCLA-2751D) at 1100 ± 50 BP agreeing with the first shell date.

Interestingly, the ruin called the "Priest's House" provided a shell date of 2470 ± 140 BP (UCLA-2751C), which is calibrated to the second century BC. Either earlier living shells are incorporated into the dry stone wall or a much earlier structure existed well before the Christian era.

County Offaly

Temple Ciaran, Clonmacnoise (Fig. 4). Reportedly, St. Ciaran, who founded Clonmacnoise in AD

548-549, is buried here. Also, an 11th century crozier was found in the oratory. In 1984, a mortar sample, UCLA-2727B, was dated to 1245 ± 55 BP, one of the oldest oratories.

Fig. 4. Clonmacnoise: Temple ciaran in foreground and O'Rourke's Round Tower in the background to the left

Another mortar sample, UCLA-2727A, from a round tower said to have been built by Fergal O'Rourke in the 10th century (Killanin, Duigan & Harbison 1989; Harbison 1982) dated to 1080 ± 60 BP, agreeing with expectations. The date shows that the base of the tower still contains original mortar, even after restoration in the 12th century. This is the oldest tower dated of four in Ireland.

County Sligo

St. Molaise Inishmurray is a monastery founded by St. Laisren in the early 6th century (Killanin, Duigan & Harbison 1989). A massive wall surrounding the site was probably used for defense (Marshall 1989), similar to Illauntannig, described above. Inside the enclosure are two main buildings, a small oratory called Teach Molaise and the Men's Church. Outside this center is the Women's Church. All these buildings were mortar charcoal-dated. From Teach Molaise, UCLA-2725D, dated to 1215 ± 45 BP, calibrated to about AD 800. This is one of the oldest mortared buildings in Ireland today.

Mortar from the Men's Church (UCLA-2725C) dated to 1040 ± 160 BP. Because the amount of charcoal recovered from the mortar was too small to give a more precise date, a larger sample will be analyzed in the future. Stylistically, however, Teach Molaise clearly antedates the Men's Church.

The Women's Church dated to 460 ± 80 BP (UCLA-2725B). From an architectural-historical point of view, this building is distinctly later than the previous two (Marshall 1989).

County Wicklow

St. Kevin's Church, Glendalough. St. Kevin founded this ecclesiastical center in the 6th century (Killanin, Duigan & Harbison 1989). A mortar sample from the croft (UCLA-2748A) dated to 910 ± 60 BP, agreeing with general expectations. Inasmuch as the building was modified at times, more

sampling of the oldest portions may be advisable to see if the original church dates to the 11–12th centuries or earlier.

Conclusions

Tables 1 and 2 show that the earliest use of mortar in Ireland dates to the 7/8th centuries as confirmed by St. Columb's at Kells, St. Michael's on Skellig, Teach Molaise on Inishmurray and Temple Ciaran at Clonmacnoise. Yet none of these buildings is as old as the introduction of Christianity into Ireland in the 5th century. Apparently, mortar building technology was not yet available in Ireland at this time. In general, Irish mortared structures are better preserved than the earlier dry-wall buildings attesting to this improvement in construction which makes them last longer. Another group of buildings dates back to the 9th century, such as the oratories on Caher and High Islands on the west coast including the Men's Church on Inishmurray north of Sligo.

A third group of structures stems from as early as the 11th century or somewhat later. This includes the Connemara churches of St. Feichin's on Omey Island, St. MacDara's on St. MacDara's Island, St. Caillin's on Chapel Island; the Aran Island churches on Inishmore, Temple Benen (suspected earlier to be 8th century) and Temple MacDuagh, on Inisheer, St. Canan's and Kilgobnet; and last not least, on Skellig Michael, St. Michael's Church Phase II, and on the mainland at Glendalough, St. Kevin's.

A late building on Inishmurray is the Women's Church outside the monastic enclosure, dating to the 15th century, when separation of the sexes at worship was still very much practiced.

All the round towers dated fall between the 9th and 12th centuries, with O'Rourke's Tower at Clonmacnoise and Clondalkin among the earliest (9–11th centuries), Rattoo belonging definitely to the first half of the 11th century, and the Cashel Round Tower fitting into the 10–12th century range. The general time frame of tower construction encompasses the period of many Viking raids, which supports the view that round towers were not only landmarks but also defensive structures.

On the whole, the dates obtained not only confirm the generally suspected age range of these early medieval buildings but place them into tighter chronological context. They provide the Irish Board of Works-National Monuments with better conservation priority planning needed to preserve their heritage and to promote tourism, an important source of national income.

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